Supporting Information

Asymmetric Transfer Hydrogenation Reactions of *N*-Sulfonylimines by Using Alcohols as Hydrogen Sources

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A: General Information

The reactions and manipulations were performed under an atmosphere of argon by using standard Schlenk techniques and Drybox (Mikrouna, Supper 1220/750). ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker-Avance 400 MHz spectrometer. $CDCl_3$ was used as solvent. Chemical shifts (δ) were reported in ppm with tetramethylsilane as internal standard, and *J* values were given in Hz. The enantioselective excesses were determined by Agilent 1260 Series HPLC using Daicel AD-H、AS-H、OJ-H and OD-H chiral columns eluted with a mixture of isopropyl alcohol and hexane. Melting points were measured on X-4 melting point apparatus and uncorrected. High resolution mass spectra (HRMS) were performed on a VG Autospec-3000 spectrometer. Column chromatography was performed with silica gel (200-300 mesh) with petroleum ether and ethyl acetate as eluents.

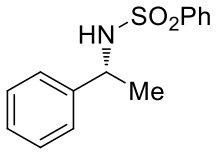
B: Procedure for the reactions

Typical procedure for the preparation of *N*-sulfonylimines[1]**:** To a 50 mL Schlenck flask were charged with benzenesulfonamide (314 mg, 2.0 mmol), acetophenone (1.2 mL, 10 mmol) and $Ti(OEt)_4$ (1.7 mL, 8.0 mmol) in CH_2Cl_2 (30 mL). The resulting solution was heated to reflux and monitored by TLC monitoring until complete consumption of benzenesulfonamide. Then CH_3OH (6mL) and a few drops of $NaHCO_3$ were added. The solution was filtered through anhydrous Na_2SO_4 and washed with EtOAc. The solvent was removed under reduced pressure and the crude product was dissolved in 10 mL CH_2Cl_2 followed by the addition of *m*-CPBA (0.7 mg, 3.0 mmol). After the completion of the reaction, the solution was washed with saturated solution of $NaHCO_3$ and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude product was purified by column chromatography using hexanes and EtOAc (10:1) to obtain the corresponding *N*-sulfonylimine **1a** in 66% yield. Compounds **1a-o** were prepared in similar methods.

Typical procedure for the preparation of cyclic *N*-sulfonylimines[2]**:** Saccharin (367 mg, 2.0 mmol) and THF (20 mL) were added to a Schlenk flask under argon atmosphere. Then phenylmagnesium bromide (4.4 mmol in THF 4.4 mL) was added dropwise at 25 °C. The resulting mixture was stirred overnight before quenching with a saturated aqueous NH_4Cl solution. The aqueous layer was extracted with CH_2Cl_2 and combined organic layer was washed with brine and dried over Na_2SO_4. The solvent was removed under reduced pressure and the crude product was dissolved in toluene (30 mL), followed by the addition of TsOH (200 mg, 1.2 mmol). The resulting solution was heated to reflux for overnight. The solvent was removed and then a saturated aqueous NH_4Cl solution (20 mL) was added. The aqueous layer was extracted with CH_2Cl_2, and the organic extracts were dried over anhydrous Na_2SO_4. After concentration, the residue was finally purified by column chromatography using hexanes and EtOAc (2:1) to afford the cyclic imine **1m** in 93% yield. Compounds **1n** was prepared in similar methods.

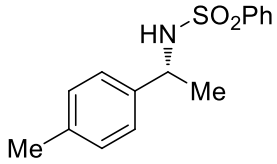
Typical procedure for the asymmetric transfer hydrogenation reaction of *N*-tosylimines: $Pd(OAc)_2$ (2.3 mg, 0.01 mmol), (*R*)-Segphos (7.3 mg, 0.012 mmol) and 1.0 mL DCE were added to a Schlenk tube under argon atmosphere. The resulting mixture was stirred at room temperature for 30 min, then $Zn(OTf)_2$ (7.3 mg, 0.02 mmol) was added and stirred for additional 10 min, followed by the addition of 4 Å Molecular sieves (50.0 mg) and a solution of methanol (40 µL, 1 mmol) in DCE (1.0 mL), and the mixture was stirred for additional 10 min. After the addition of *N*-tosylimines **1a** (51.8 mg, 0.2mmol), a solution of trifluoromethanesulfonic acid (1µL) in DCE (0.1 mL) was added, the mixture was then stirred at 70 °C under argon atmosphere with TLC monitoring until complete consumption of **1a**. The reaction mixture was concentrated, and the residue residue was purified by chromatography on a silica gel column to afford the desired product **3aa** (48 mg, 93% yield).

C: Characterization Data of Products



(*R*)-*N*-(1-phenylethyl)benzenesulfonamide (3aa)[3]

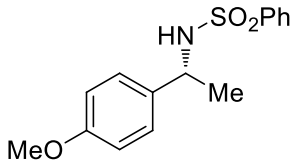
White solid, 93% yield, 98% *ee*. Mp 92-95 °C. $[\alpha]_D^{20}$ = +70.6 (c = 0.68, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.72 (d, *J* = 7.2 Hz, 2H), 7.46 (t, *J* = 7.2 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.16-7.14 (m, 3H), 7.09-7.07 (m, 2H), 5.41-5.34 (m, 1H), 4.52-4.45 (m, 1H), 1.42 (d, *J* = 6.8 Hz, 3H). The *ee* of **3aa** was determined by HPLC analysis using two Daicel Chiralcel OJ-H columns (2 × 25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 70/30, 1.0 mL/min, 240 nm; t_{minor} = 12.5 min, t_{major} = 18.3 min.



3ba

(*R*)-*N*-(1-(*p*-tolyl)ethyl)benzenesulfonamide (3ba)[3]

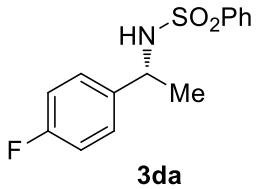
White solid, 85% yield, 97% *ee*. Mp 143-145 °C. $[\alpha]_D^{20}$ = +87.1 (c = 0.76, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.74-7.72 m, 2H), 7.51-7.47 (m, 1H), 7.414-7.37 (m, 2H), 7.00-6.95 (m, 4H), 4.97 (d, *J* =7.2Hz, 2H), 4.48-4.41 (m, 1H), 2.27 (s, 3H), 1.42 (d, *J* = 6.8 Hz, 3H). The *ee* of **3ba** was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =70/30, 0.5 mL/min, 240 nm; t_{major} = 10.4min, t_{minor} = 11.6min.



3ca

(*R*)-*N*-(1-(4-methoxyphenyl)ethyl)benzenesulfonamide (3ca)[3]

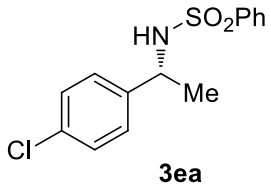
White solid, 82% yield, 98% *ee*. Mp 98-99 °C. $[\alpha]_D^{20}$ = +78.7 (c = 0.82, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.6Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.61 (d, *J* = 8.8 Hz, 2H), 5.25 (d, *J* = 7.2 Hz, 1H), 4.39-4.32 (m, 1H), 3.65(s, 3H), 1.32 (d, *J* = 6.8 Hz, 3H). The *ee* of **3ca** was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 90/10, 0.8 mL/min, 240 nm; t_{major} = 20.0 min, t_{minor} = 23.3 min.



3da

(*R*)-*N*-(1-(4-fluorophenyl)ethyl)benzenesulfonamide (3da)[3]

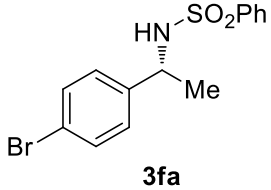
White solid, 81% yield, 99% *ee*. Mp 141-142 °C. $[\alpha]_D^{20}$ = +64.7 (c = 0.68, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.72-7.70 (m, 2H), 7.51-7.47(m, 1H), 7.40-7.35 (m, 2H), 7.07-7.04 (m, 2H), 6.85-6.80 (m, 2H), 5.46 (d, *J* = 7.2 Hz, 1H), 4.48 (m, 1H), 1.39 (d, *J* = 7.2 Hz, 3H). The *ee* of **3da** was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =70/30, 0.5 mL/min, 240 nm; t_{major} = 10.9 min, t_{minor} = 12.3 min.



3ea

(*R*)-*N*-(1-(4-chlorophenyl)ethyl)benzenesulfonamide (3ea)[3]

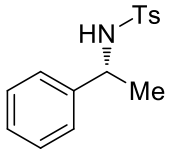
White solid, 76% yield, 98% *ee*. Mp 153-155 °C. $[\alpha]_D^{20}$ = +81.3 (c = 0.62, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.71-7.69 (m, 2H), 7.53-7.49 (m, 1H), 7.40-7.36 (m, 2H), 7.14-7.11 (m, 2H), 7.04-7.01 (m, 2H), 5.25 (d, *J* = 6.8 Hz, 1H), 4.51-4.44 (m, 1H), 1.39 (d, *J* = 6.8 Hz, 3H). The *ee* of **3ea** was determined by HPLC analysis using Daicel Chiralcel OJ-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =70/30, 1 mL/min, 240 nm; t_{major} = 9.9 min, t_{minor} = 11.2 min.



3fa

(*R*)-*N*-(1-(4-bromophenyl)ethyl)benzenesulfonamide (3fa)[3]

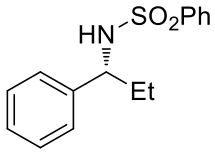
White solid, 65% yield, 99% *ee*. Mp 158-159 °C. $[\alpha]_D^{20}$ = +71.1 (c = 0.54, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.63-7.61 (m, 2H), 7.44 (t, *J* = 7.6 Hz, 1H), 7.31 (t, *J* = 8.0 Hz, 2H), 7.21 (t, *J* = 6.4 Hz, 2H), 6.89 (d, *J* = 8.4 Hz, 2H), 5.22 (d, *J* = 6.8 Hz, 1H), 4.42-4.35 (m, 1H), 1.31 (d, *J* = 6.8 Hz, 3H). The *ee* of **3fa** was determined by HPLC analysis using Daicel Chiralcel AS-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =70/30, 1 mL/min, 240 nm; t_{major} = 13.3 min, t_{minor} = 20.5 min.



3ga

(*R*)-4-methyl-*N*-(1-phenylethyl)benzenesulfonamide (3ga)[2]

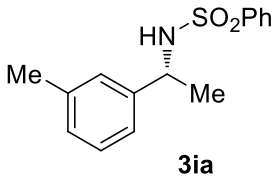
White solid, 90% yield, 99% *ee*. Mp 106-108 °C. $[\alpha]_D^{20}$ = +79.5 (c = 0.86, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.4 Hz, 2H), 7.20-7.14 (m, 5H), 5.23 (d, *J* = 7.2 Hz, 1H), 4.48-4.41 (m, 1H), 2.37 (s, 3H), 1.40 (d, *J* = 6.8 Hz, 3H). The *ee* of **3ga** was determined by HPLC analysis using Daicel Chiralcel OJ-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 70/30, 1 mL/min, 240 nm; t_{minor} = 10.7 min, t_{major} = 16.1 min.



3ha

(*R*)-*N*-(1-phenylpropyl)benzenesulfonamide (3ha)[3]

White solid, 63% yield, 98% *ee*. Mp 101-103 °C. $[\alpha]_D^{20}$ = +66.6 (c = 0.44, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.59-7.57 (m, 2H), 7.37-7.32 (m, 1H), 7.24-7.19 (m, 2H), 7.05-7.03 (m, 3H), 6.92 (dd, *J* = 7.2, 3.6 Hz, 2H), 5.32 (d, *J* = 7.2 Hz, 1H), 1.76-1.60 (m, 2H), 0.72 (t, *J* = 7.6 Hz, 3H). The *ee* of **3ha** was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =90/10, 0.5 mL/min, 240 nm; t_{minor} = 21.6 min, t_{major} = 22.9 min.

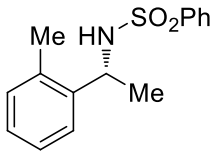


3ia

(*R*)-*N*-(1-(*m*-tolyl)ethyl)benzenesulfonamide (3ia)

Colorless oil, 87% yield, 97% *ee*. $[\alpha]_D^{20}$ = +70.8 (c = 0.76, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.65-7.63 (m, 2H), 7.38 (t, *J* = 7.6 Hz, 1H), 7.29-7.26 (t, *J* = 7.6 Hz, 2H), 6.97 (t, *J* = 7.6 Hz, 1H), 6.80 (dd, *J* = 19.6, 7.6 Hz, 2H), 7.75 (s, 1H), 5.37 (d, *J* = 7.2 Hz, 1H), 4.40-4.33 (m, 1H), 2.10 (s, 3H), 1.33 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ

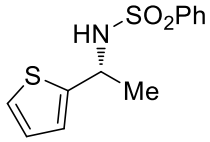
140.7, 139.7, 137.1, 131.2, 127.7, 127.4, 127.1, 125.9, 125.8, 122.1, 52.7, 22.5, 20.2. HRMS calcd for $C_{15}H_{17}NO_2S$ $[M]^+$: 275.0980. Found: 275.0984. The *ee* of **3ia** was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =80/20, 0.5 mL/min, 240 nm; t_{major} = 10.9 min, t_{minor} = 12.2 min.



3ja

(*R*)-*N*-(1-(*o*-tolyl)ethyl)benzenesulfonamide (3ja)

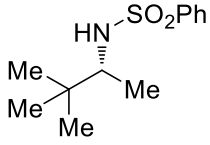
White solid, 73% yield, >99% *ee*. Mp 111-112 °C .$[\alpha]_D^{20}$ = +42.7 (c = 0.66, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73-7.71 (m, 2H), 7.49-7.45 (m, 1H), 7.38-7.34 (m, 2H), 7.14-7.01 (m, 4H), 5.39 (d, *J* = 6.8 Hz, 1H), 4.82-4.75 (m, 1H), 2.22 (s, 3H), 1.42 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.6, 140.1, 134.5, 132.3, 130.4, 128.8, 127.3, 126.9, 126.4, 125.3, 49.8, 23.2, 18.9. HRMS calcd for $C_{15}H_{17}NO_2S$ $[M]^+$: 275.0980. Found: 275.0979. The *ee* of **3ja** was determined by HPLC analysis using Daicel Chiralcel OJ-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =70/30, 1 mL/min, 240 nm; t_{minor} = 6.9 min, t_{major} = 10.5 min.



3ka

(*R*)-*N*-(1-(thiophen-2-yl)ethyl)benzenesulfonamide (3ka)

White solid, 94% yield, 95% *ee*. Mp 93-95 °C. $[\alpha]_D^{20}$ = +63.3 (c = 0.80, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.80 (m, 2H), 7.55-7.51 (m, 1H), 7.46-7.42 (m, 2H), 7.09 (dd, *J* = 5.2, 1.2Hz, 1H), 6.80-6.72 (m, 1H), 6.75-6.74 (m, 1H), 5.30 (d, *J* = 7.6 Hz, 1H), 4.80-4.73 (m, 1H), 1.52 (d, *J* = 6.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 145.9, 140.6, 132.6, 129.0, 127.1, 126.7, 124.8, 124.5, 49.3, 24.0. HRMS calcd for $C_{12}H_{13}NO_2S_2$ $[M]^+$: 267.0387. Found: 267.0391. The *ee* of **3ka** was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =90/10, 1 mL/min, 240 nm; t_{major} = 12.2 min, t_{minor} = 14.2 min.

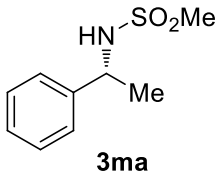


3la

(*R*)-*N*-(3,3-dimethylbutan-2-yl)benzenesulfonamide (3la)[4]

White solid, 80% yield, 96% *ee*. Mp 116-119 °C. $[\alpha]_D^{20}$ = +4.5 (c = 0.44, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91-7.89 (m, 2H), 7.59-7.49 (m, 3H), 4.61 (d, *J* = 7.6 Hz, 2H),

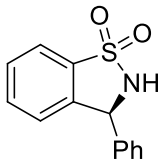
3.11-3.03 (m, 1H), 0.88 (d, *J* = 6.8 Hz, 3H), 0.83 (s, 9H). The *ee* of **3la** was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =95/5, 0.5 mL/min, 240 nm; t_{minor} = 20.1 min, t_{major} = 21.4 min.



3ma

(*R*)-N-(1-phenylethyl)methanesulfonamide (3ma)[5]

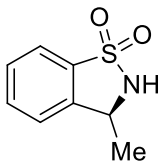
Colorless oil, 81% yield, 99% *ee*. $[\alpha]_D^{20}$ = 52.6 (c = 0.50, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39-7.28 (m, 5H), 5.18(d, *J* = 7.2 Hz, 1H), 4.64 (m, 1H), 2.60 (s, 3H), 1.54 (d, *J* = 7.2, 3H). The *ee* of **3ma** was determined by HPLC analysis using Daicel Chiralcel OJ-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH =70/30, 0.5 mL/min, 220 nm; t_{minor} = 18.7 min, t_{major} = 19.3 min.



3pa

(*S*)-3-phenyl-2,3-dihydrobenzo[*d*]isothiazole 1,1-dioxide (3pa)[6]

White solid, 90% yield, 90% *ee*. Mp 115-118 °C. $[\alpha]_D^{20}$ = +71.5 (c = 0.54, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79-7.77 (m, 1H), 7.53-7.51 (m, 2H), 7.36-7.35 (m, 5H), 7.13-7.11 (m, 1H), 5.71 (d, *J* = 4.0 Hz, 1H), 5.37-5.32 (m, 1H). The *ee* of **3ma** was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 70/30, 0.8 mL/min, 230 nm; t_{major} = 15.3 min t_{minor} = 17.8 min.



3qa

(*S*)-3-methyl-2,3-dihydrobenzo[*d*]isothiazole 1,1-dioxide (3qa)[6]

White solid, 62% yield, 84% *ee*. Mp 95-98°C. $[\alpha]_D^{20}$ =-15.5 (c =0.20, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.6 Hz ,1H), 7.64 (t, *J* = 7.4 Hz ,1H), 7.53 (t, *J* = 7.4 Hz ,1H), 7.40 (d, *J* = 8.0 Hz, 1H), 5.12(d, *J* = 27.6 Hz ,1H), 4.83-4.77 (m, 1H), 1.61 (dd, *J* = 6.8, 2.4 Hz, 3H). The *ee* of **3na** was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 0.8mL/min, 230 nm; t_{major} = 24.6 min, t_{minor} = 31.3 min.

Notes and references:

[1] Q. Yang, G. Shang, W.-Z. Gao, J.-G. Deng, X.-M. Zhang, *Angew. Chem. Int. Ed.*, 2006, **45**, 3832-3835

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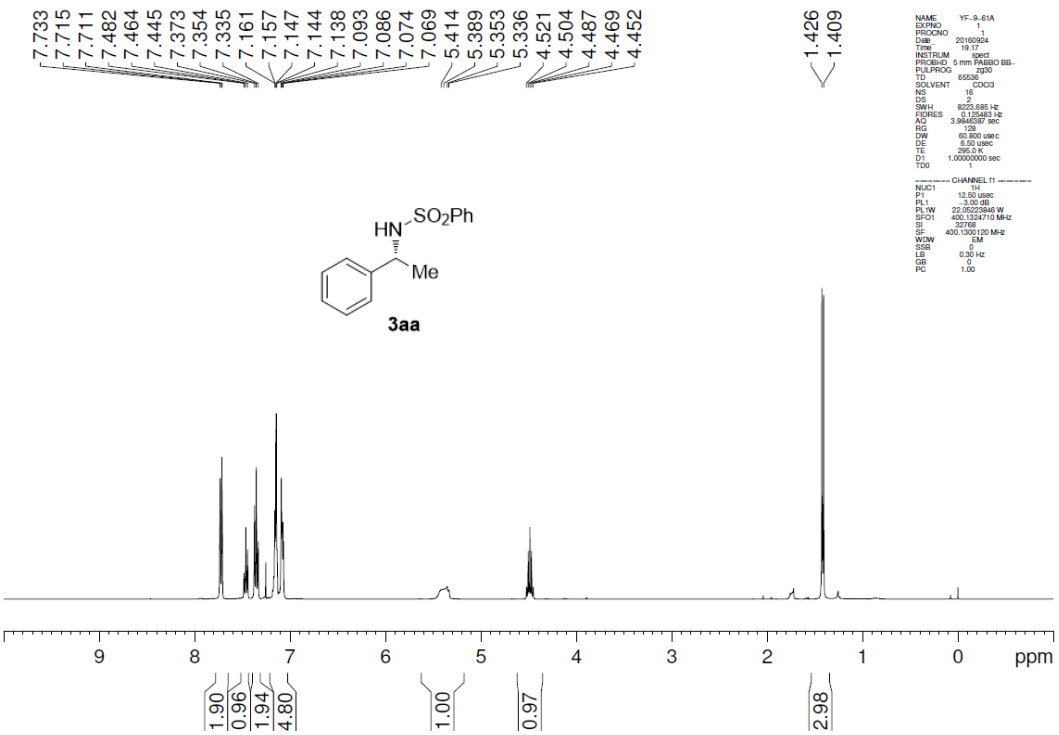
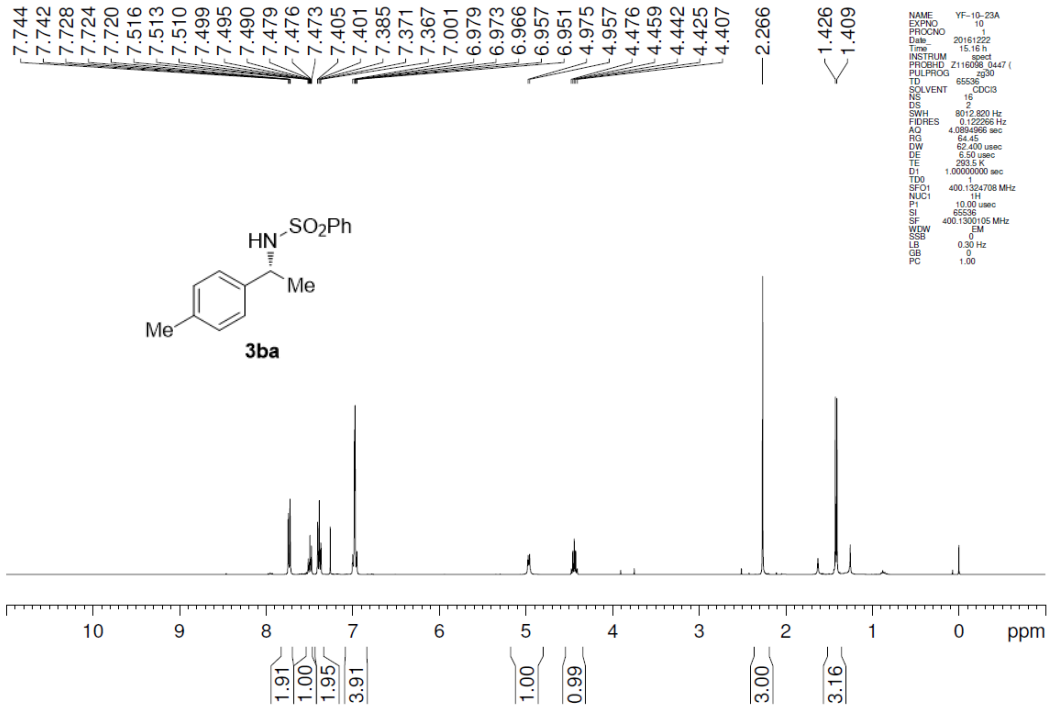
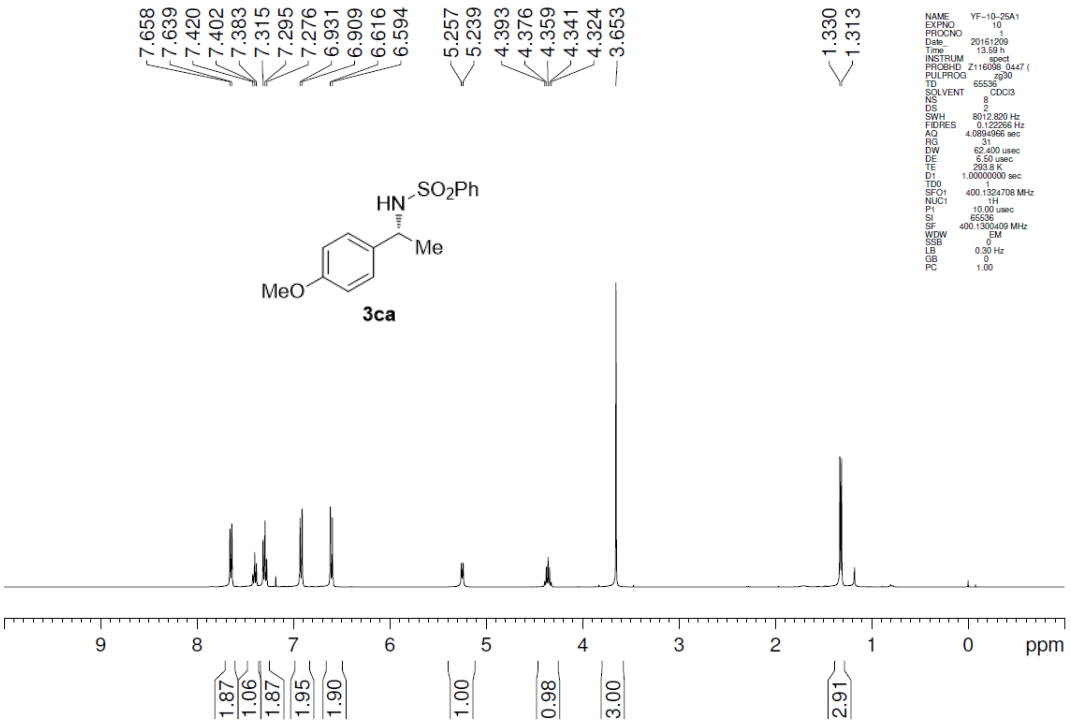
[5] A. D. Gillie, R. J. Reddy, P. W. Davies, *Adv. Synth. Catal.*, 2016, **358**, 226-239.

[6] Y.-Q. Wang, S.-M. Lu, Y.-G. Zhou, *J. Org. Chem.* 2007, **72**, 3729-3734.

D: NMR Spectra of Products



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5.257
5.239
4.393
4.376
4.359
4.341
4.324
3.653
1.330
1.313
HN
SO2Ph
Me
MeO
3ca
ppm
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1.87
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1.90
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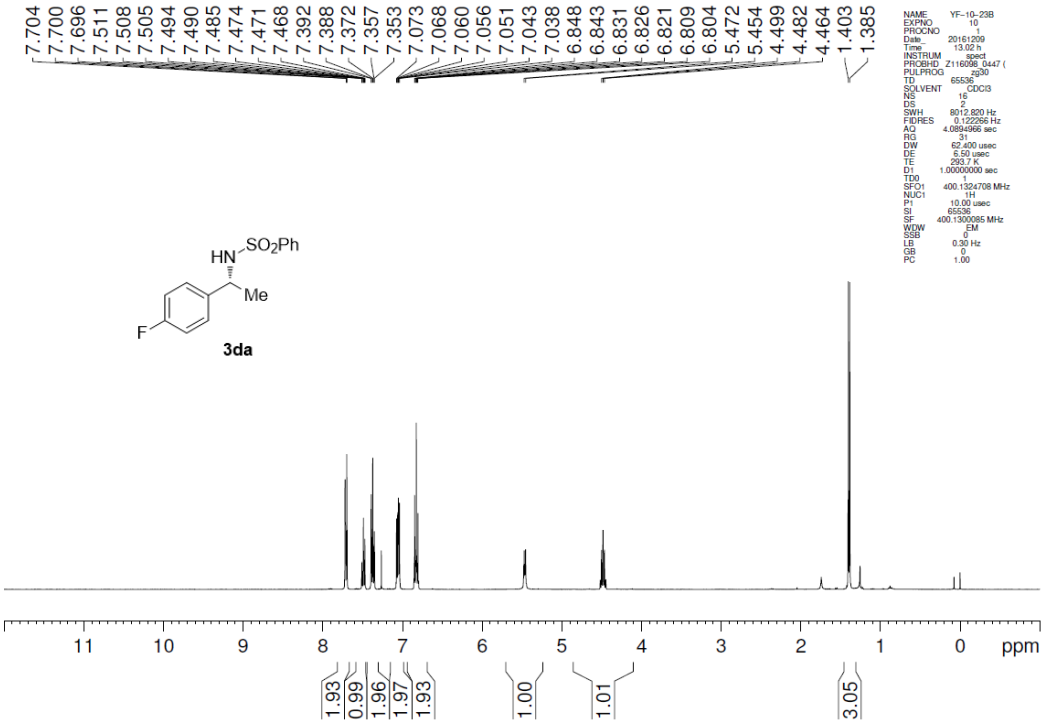
0.98
3.00
2.91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7.392
7.388
7.372
7.357
7.353
7.073
7.068
7.060
7.056
7.051
7.043
7.038
6.848
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6.826
6.821
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1.385
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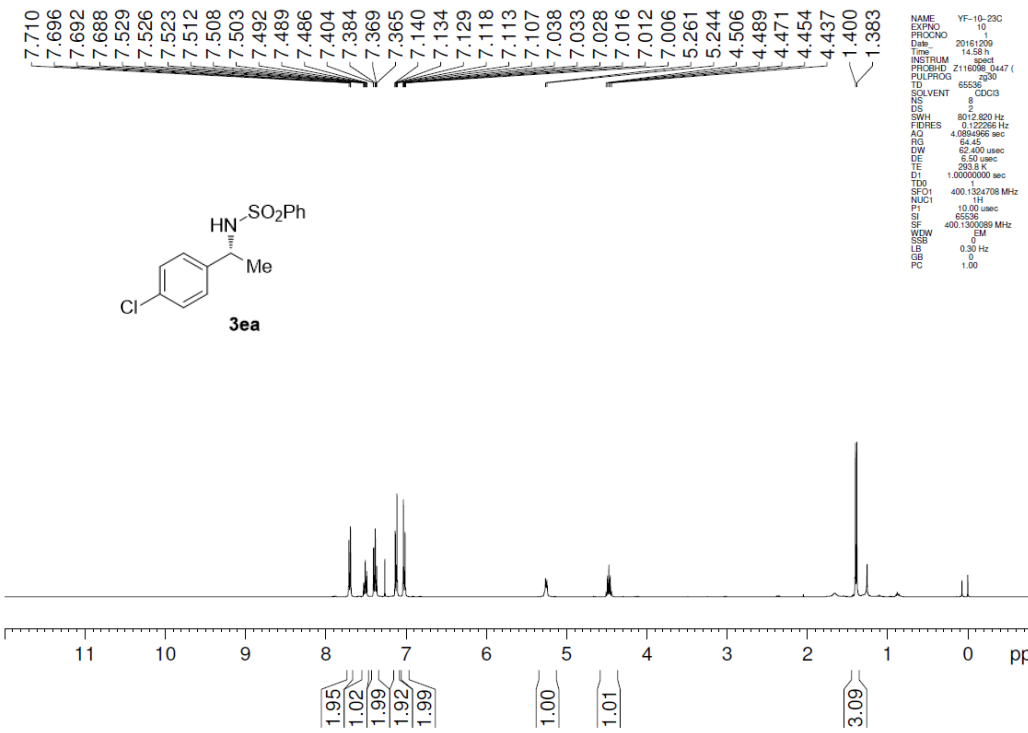
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7.369
7.365
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7.134
7.129
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7.113
7.107
7.038
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Cl
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10
9
8
7
6
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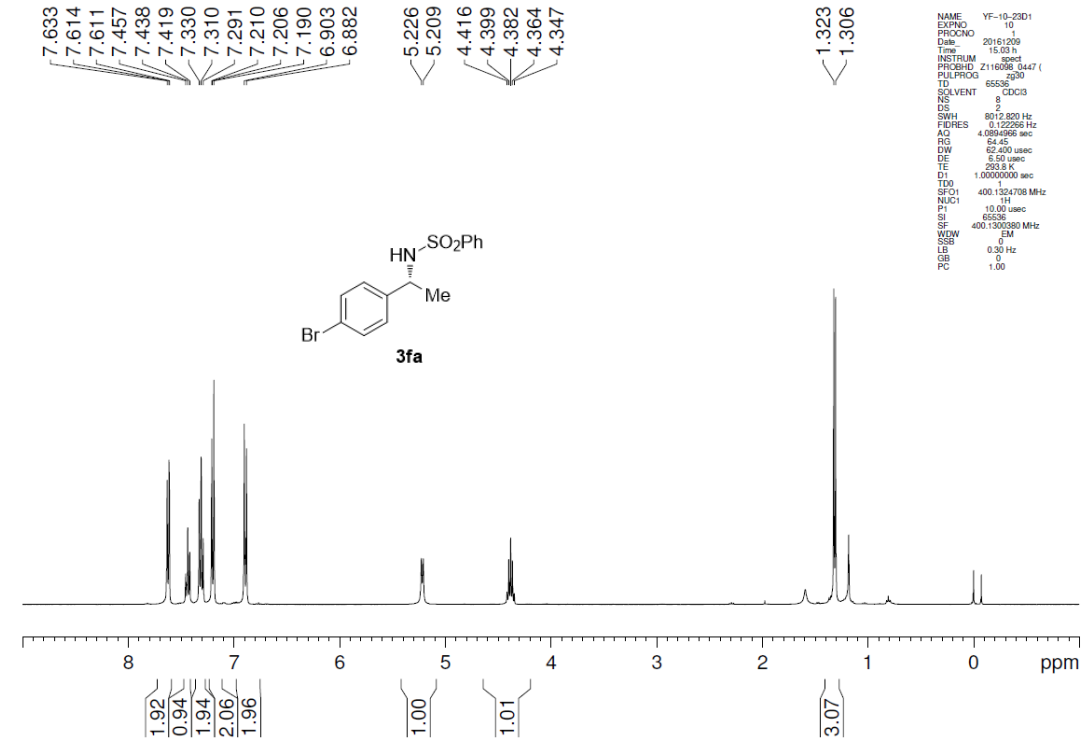
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6.882
5.226
5.209
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4.382
4.364
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SO2Ph
Me
Br
3fa
8
7
6
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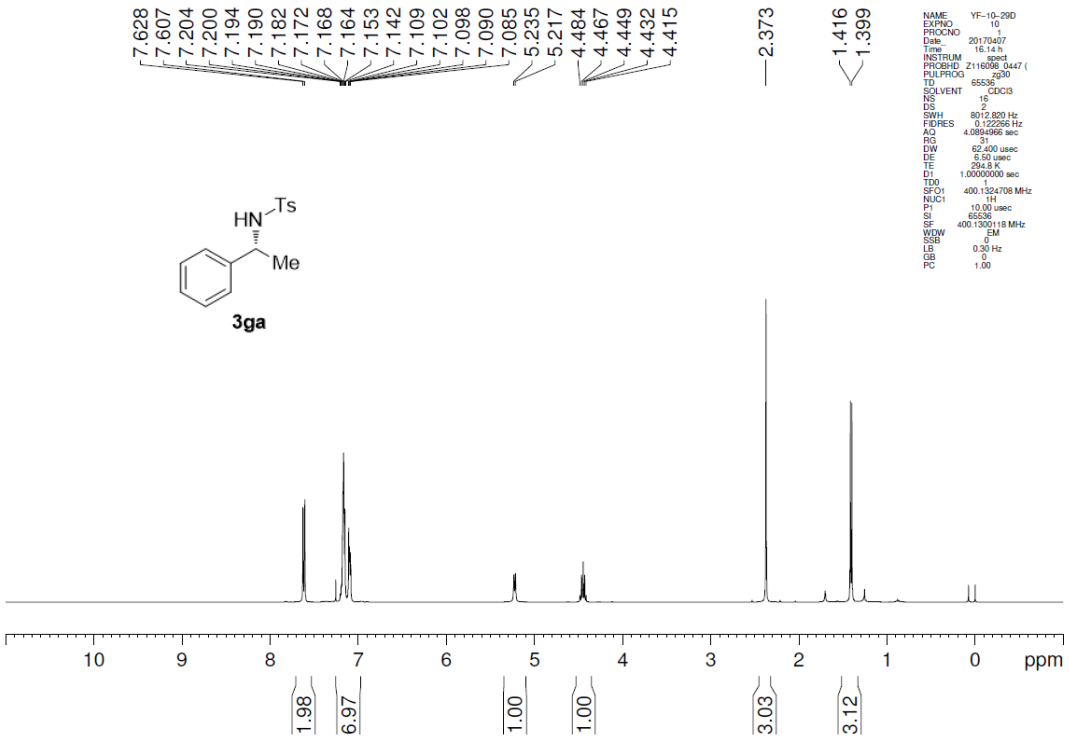
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7.090
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4.432
4.415
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1.399
HN
Ts
Me
3ga
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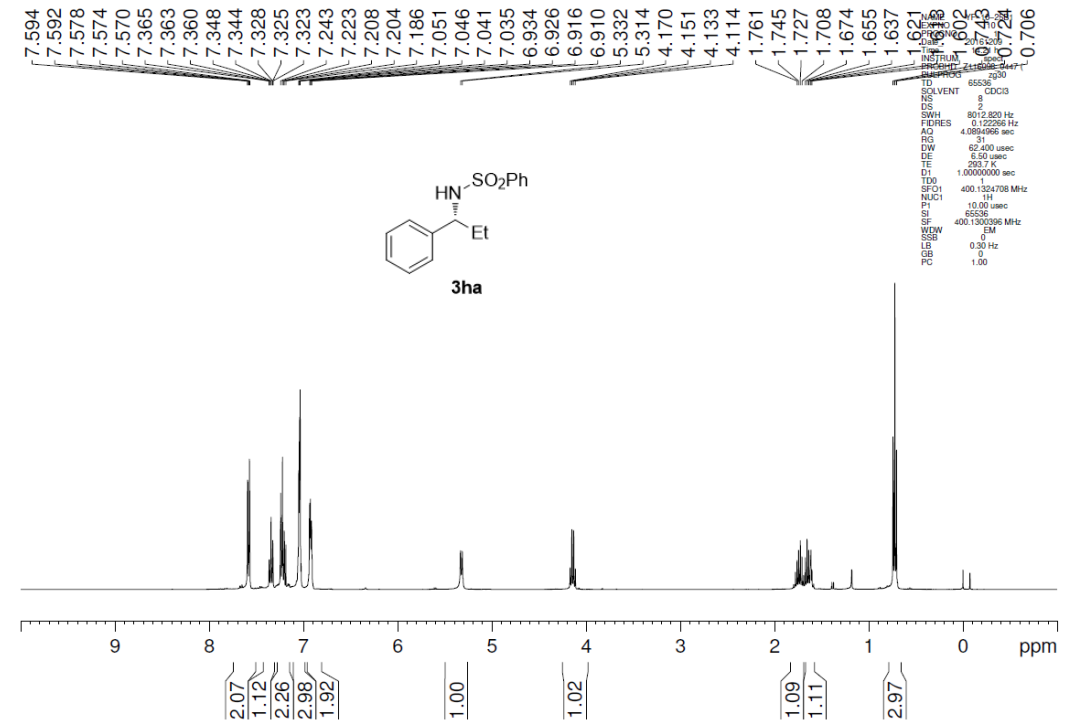
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7.323
7.243
7.223
7.208
7.204
7.186
7.051
7.046
7.041
7.035
6.934
6.926
6.916
6.910
5.332
5.314
4.170
4.151
4.133
4.114
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1.745
1.727
1.708
1.674
1.655
1.637
0.724
0.706
HN
SO2Ph
Et
3ha
9
8
7
6
5
4
3
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1.12
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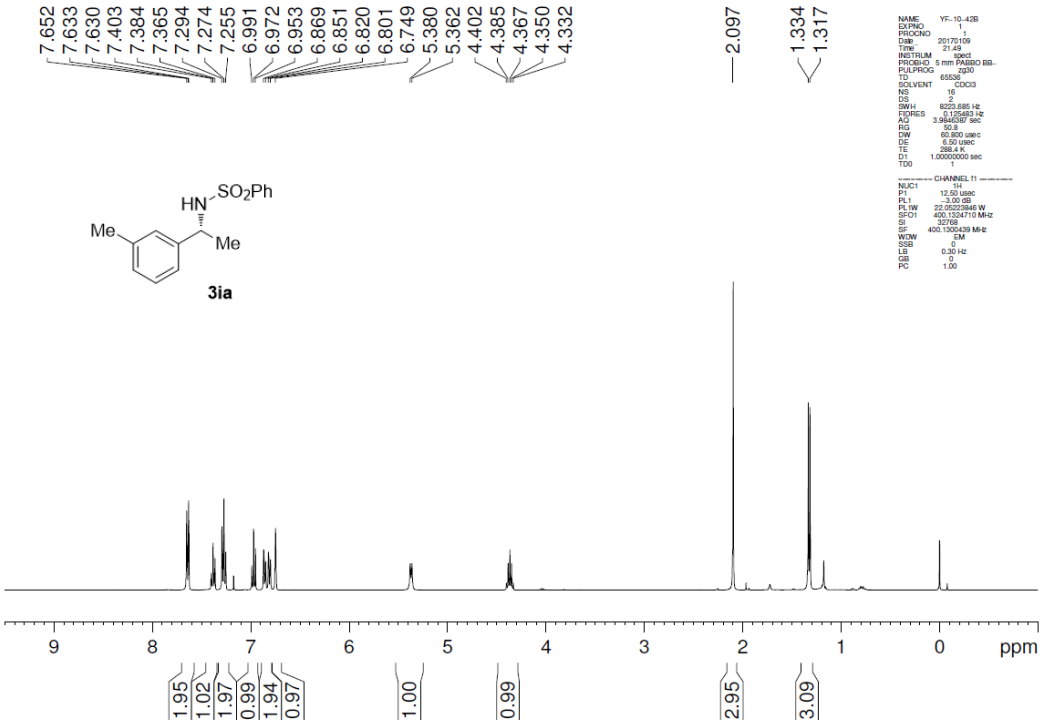
1.00
1.02
1.09
1.11
2.97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6.851
6.820
6.801
6.749
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4.402
4.385
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4.350
4.332
2.097
1.334
1.317
HN
SO2Ph
Me
Me
3ia
9
8
7
6
5
4
3
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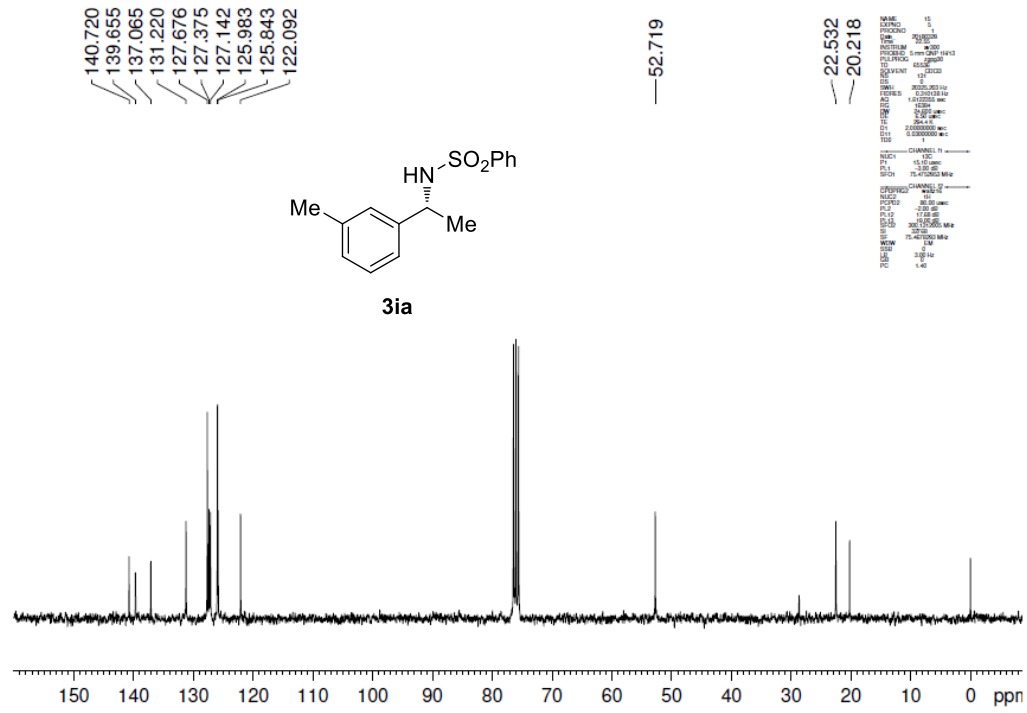
1.02
1.97
0.99
1.94
0.97
1.00
0.99
2.95
3.09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20.218
HN
SO2Ph
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Me
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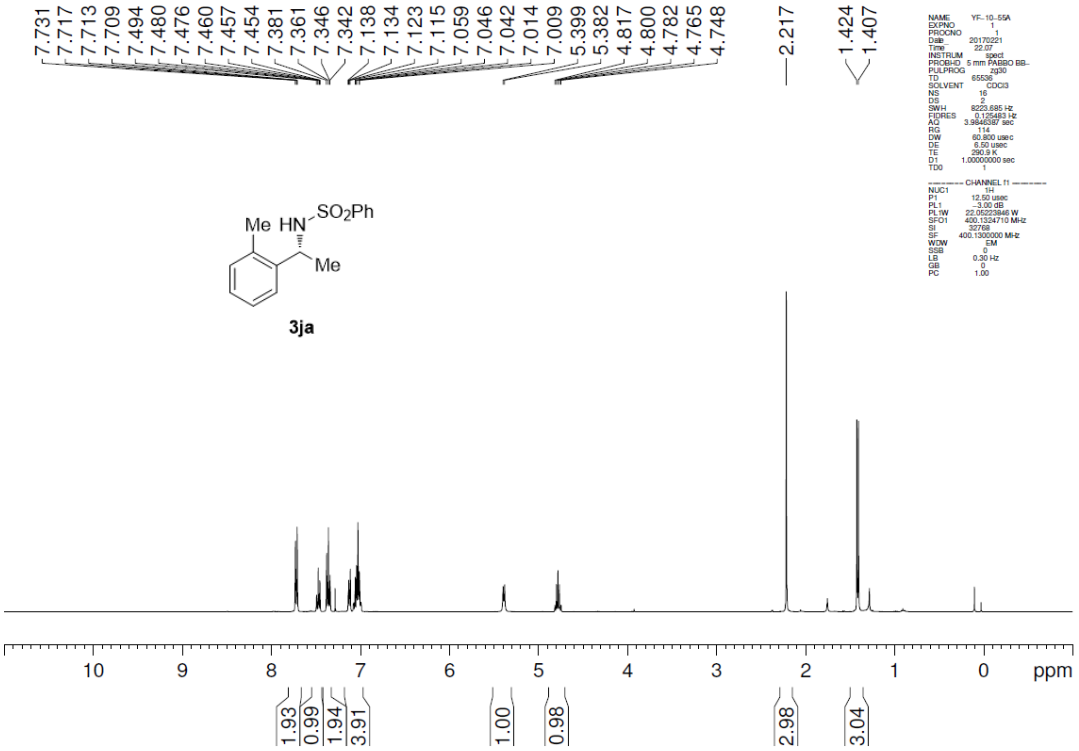
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.115
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7.042
7.014
7.009
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5.382
4.817
4.800
4.782
4.765
4.748
2.217
1.424
1.407
Me HN SO2Ph
Me
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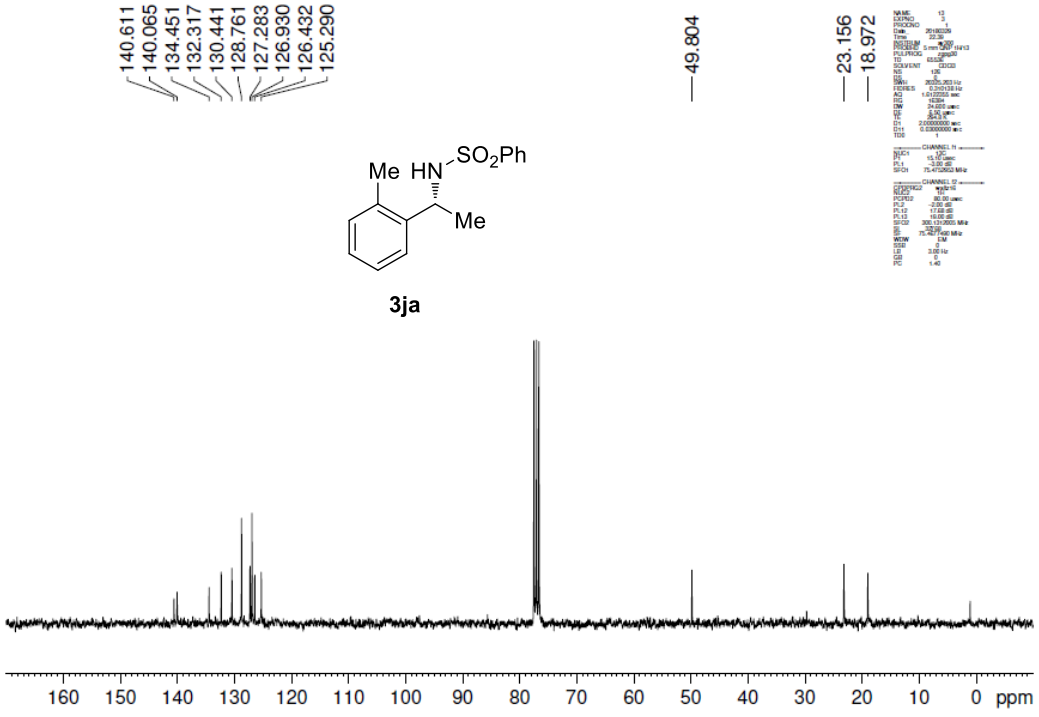
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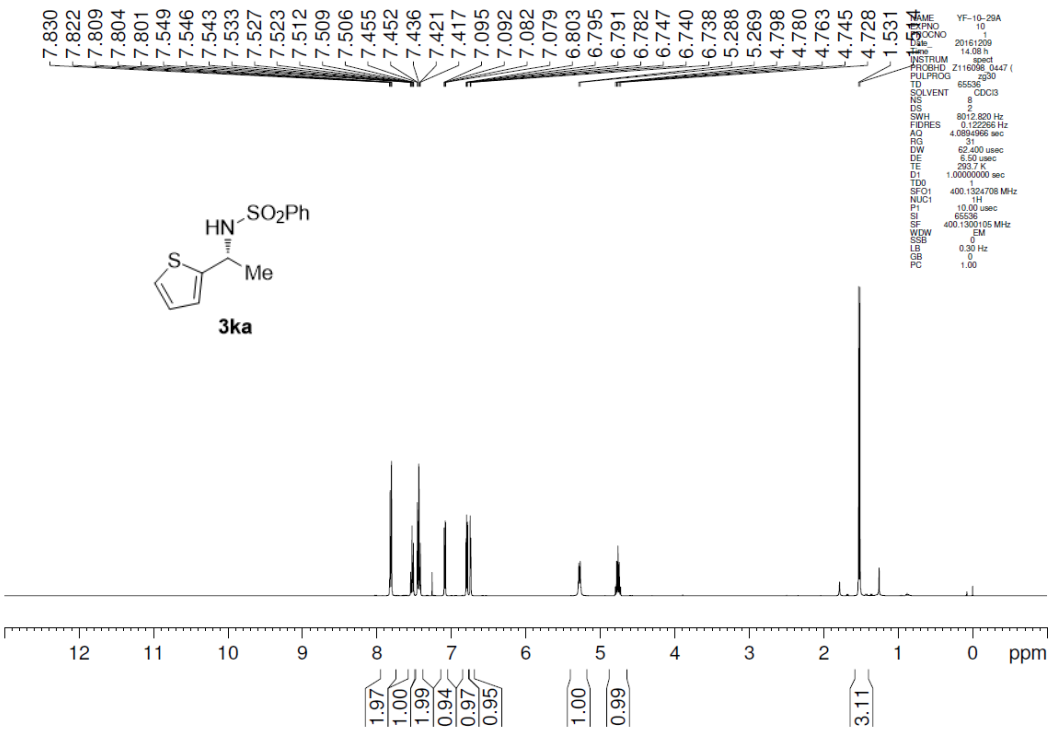
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S
Me
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8
7
6
5
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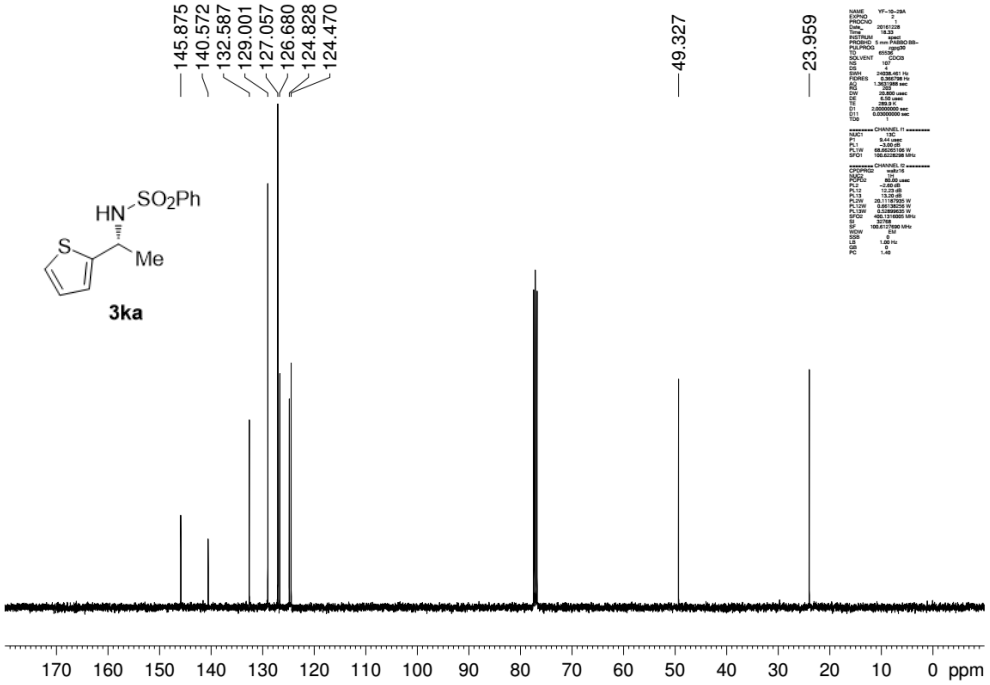
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SO2Ph
S
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130
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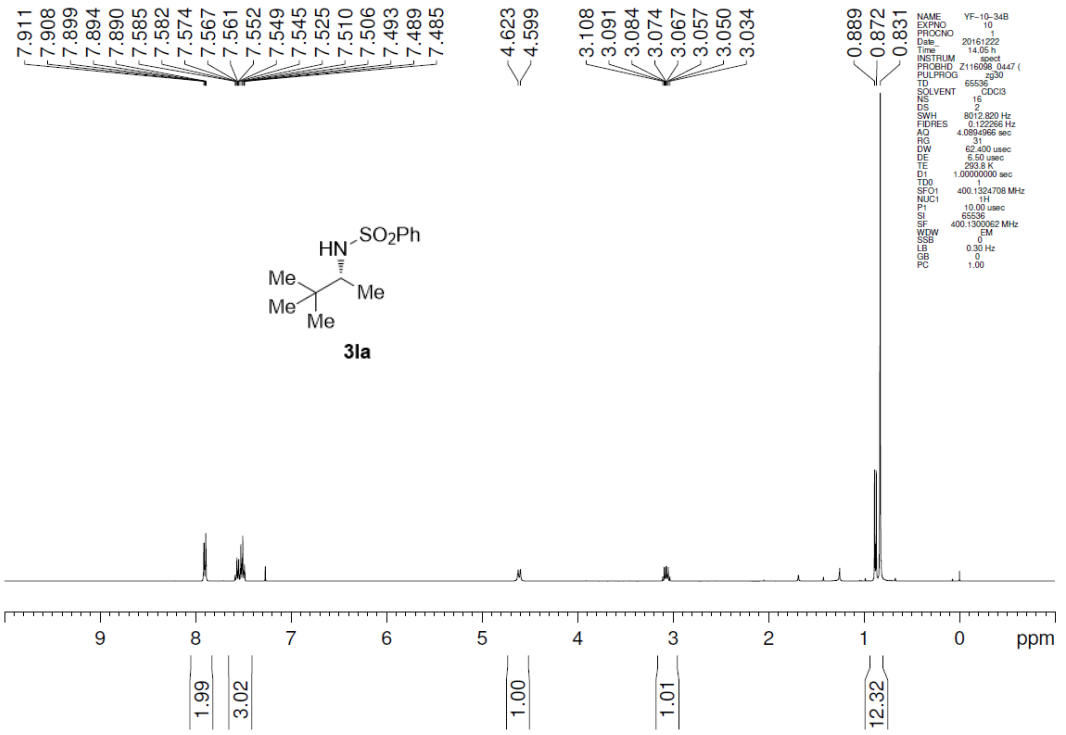
30
20
10
0 ppm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7.549
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7.525
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7.506
7.493
7.489
7.485
4.623
4.599
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3.091
3.084
3.074
3.067
3.057
3.050
3.034
0.889
0.872
0.831
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SO2Ph
Me
Me
Me
Me
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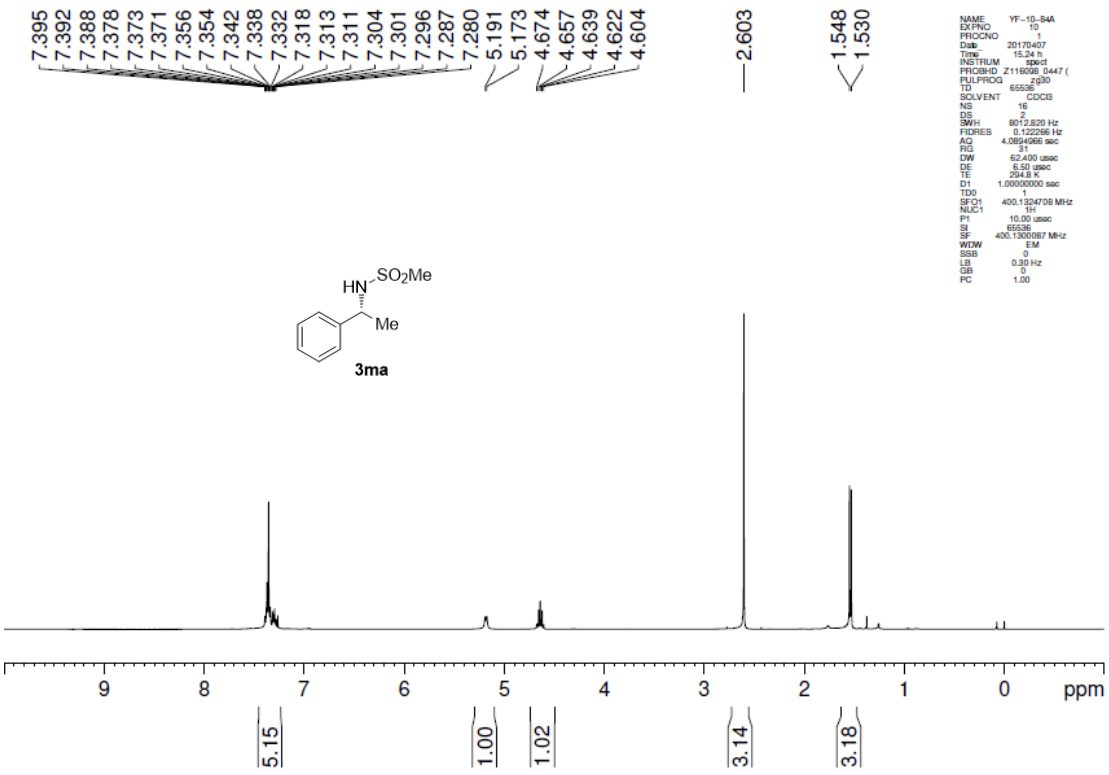
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7.287
7.280
5.191
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4.622
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SO2Me
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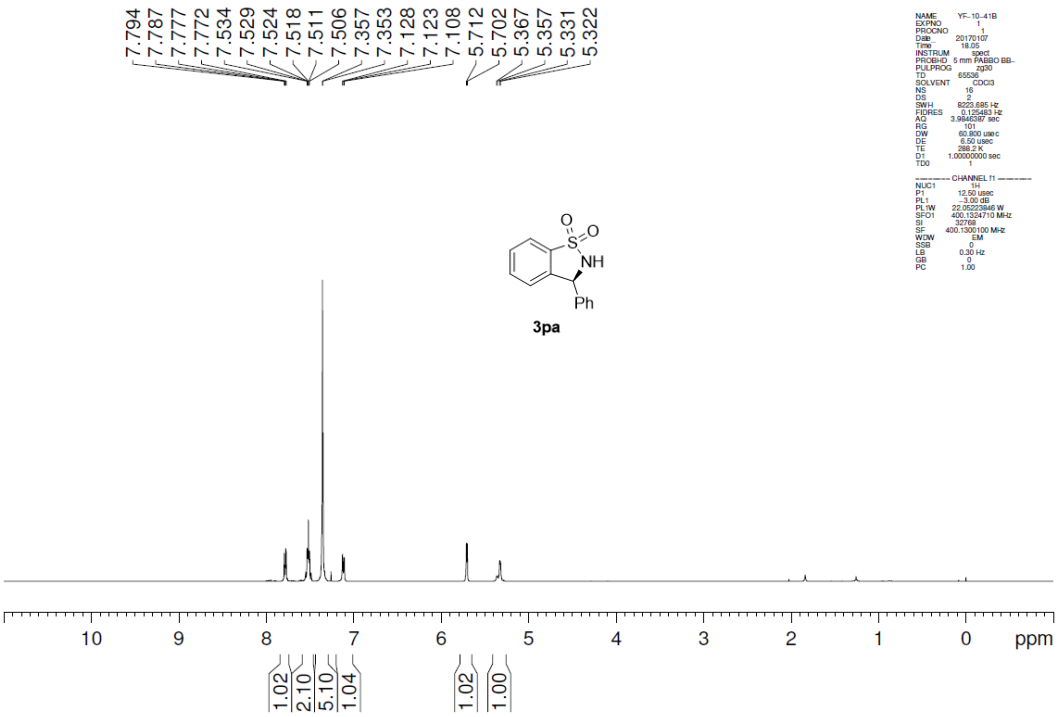
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5.331
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NH
Ph
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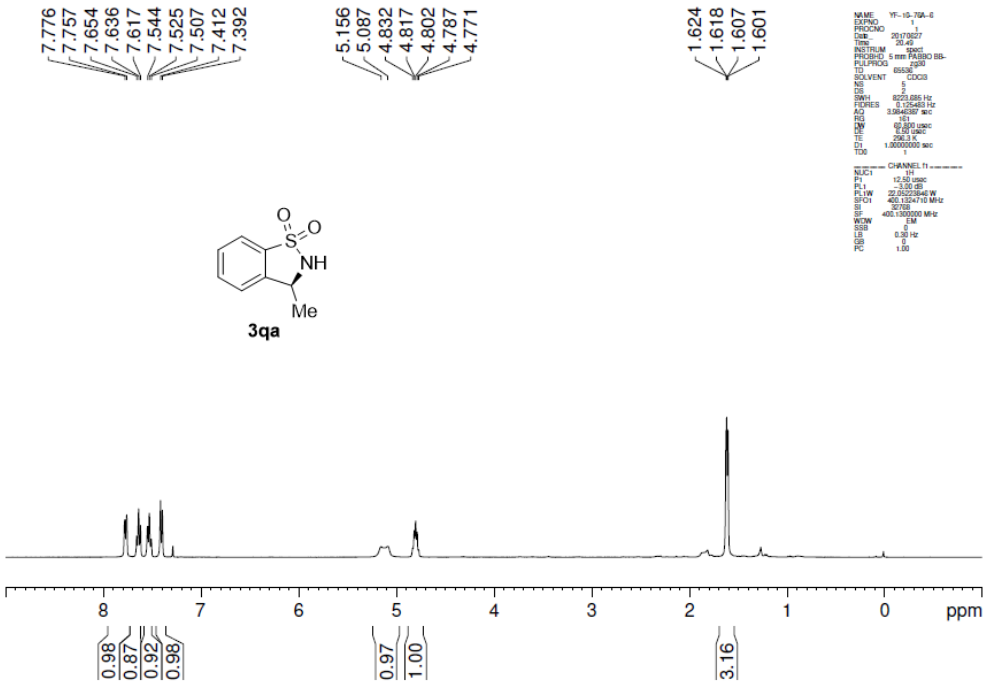
1.00

3pa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7.392
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5.087
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4.771
1.624
1.618
1.607
1.601
NH
Me
8
7
6
5
4
3
2
1
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ppm
0.98
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0.92
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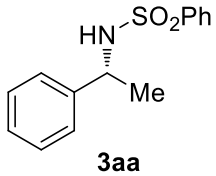
1.00
3.16

3qa

E: HPLC Spectra of Products



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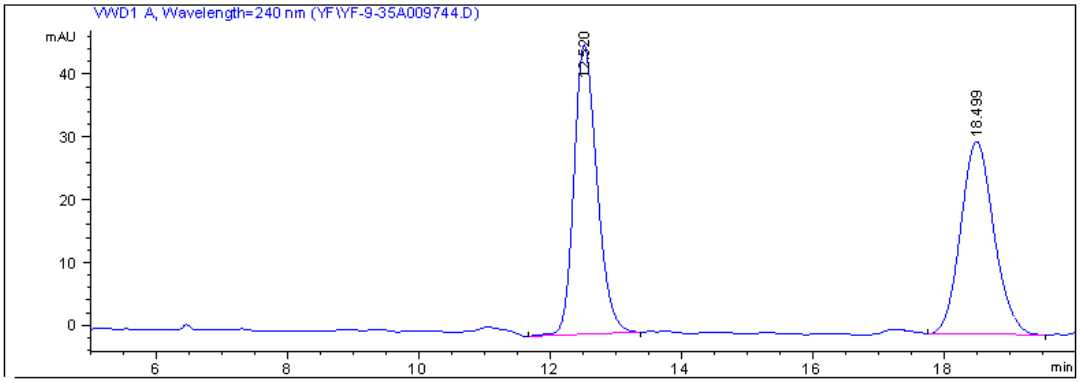

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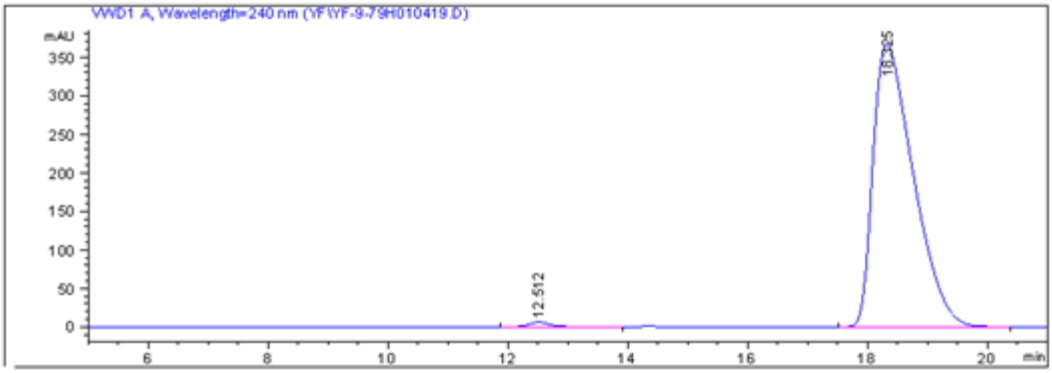
1	12.520	BB	0.3752	1122.27161	46.06736	51.0666
2	18.499	BB	0.5420	1075.39185	30.67477	48.9334



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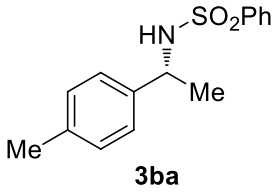
1	12.512	BB	0.4159	163.57930	5.92804	0.9457
2	18.325	BB	0.7137	1.71332e4	366.95953	99.0543



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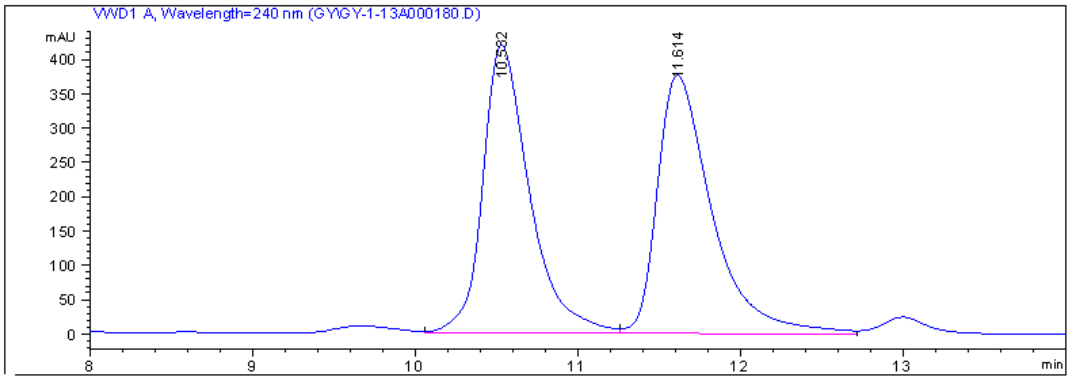
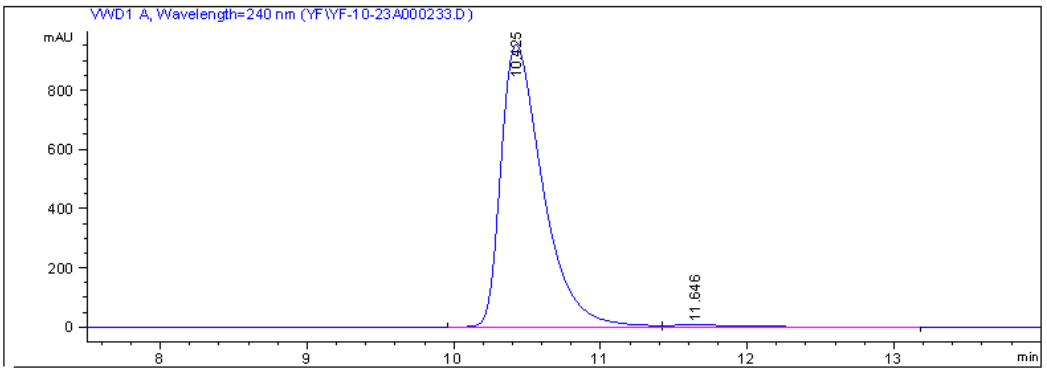


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.532	VV	0.3011	8419.28711	419.61191	48.9716
2	11.614	VV	0.3512	8772.88770	376.03952	51.0284



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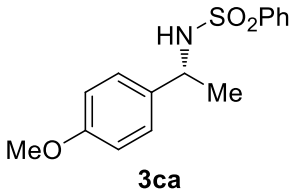
2	11.646	VB	0.4577	270.46820	8.13909	1.4111



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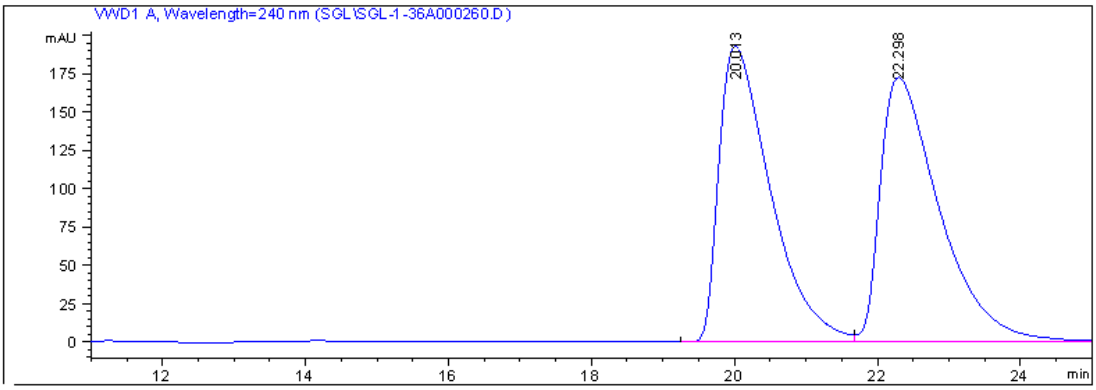
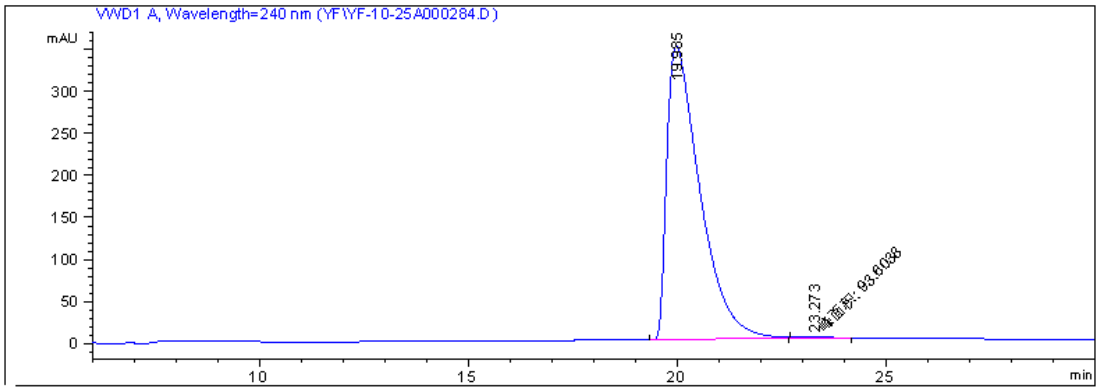


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.013	BV	0.7615	9670.74805	192.68793	49.3074
2	22.298	VBA	0.8735	9942.43750	172.23538	50.6926



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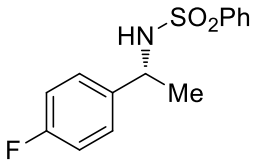
2	23.273	MM	0.8097	93.60379	1.92672	0.4852



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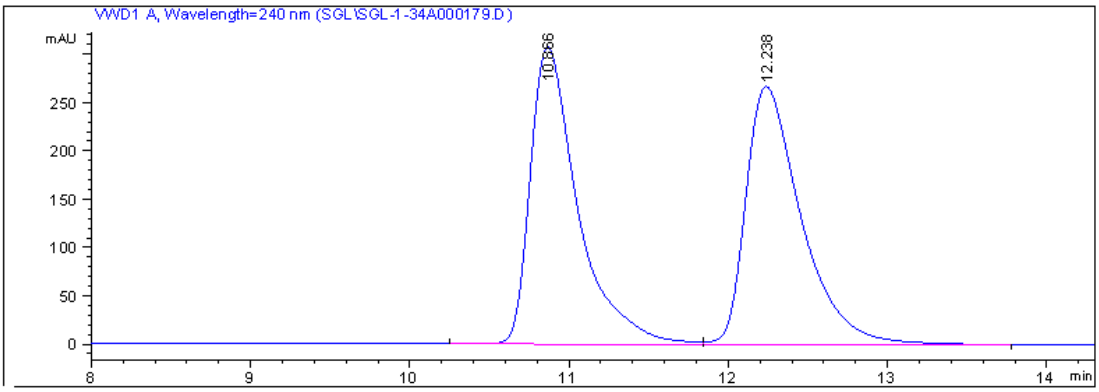

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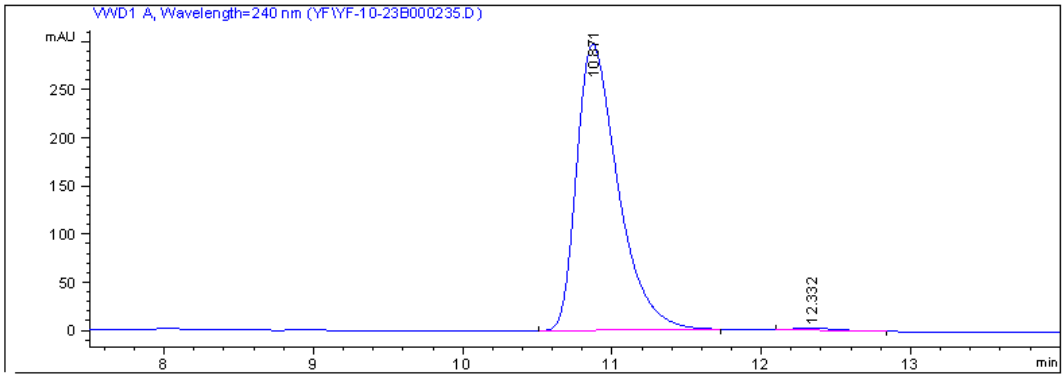
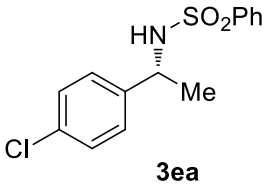
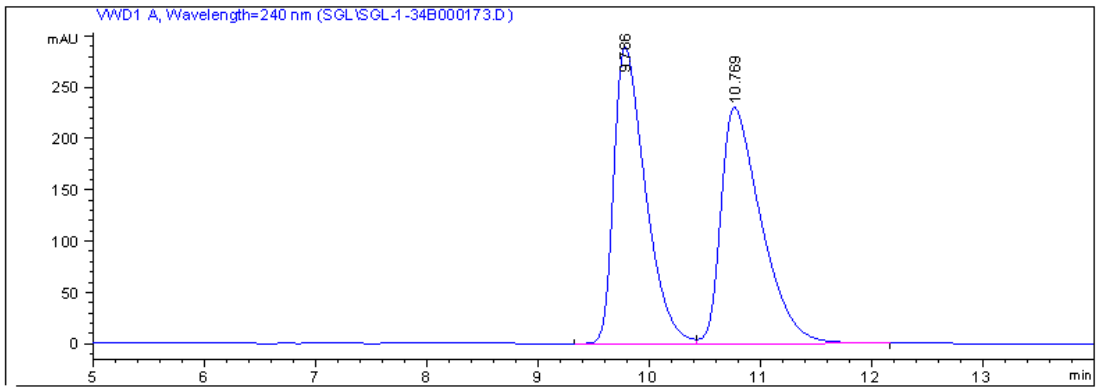
1	10.866	BV	0.3111	6383.38867	307.56976	49.7951
2	12.238	VB	0.3634	6435.93359	266.83139	50.2049



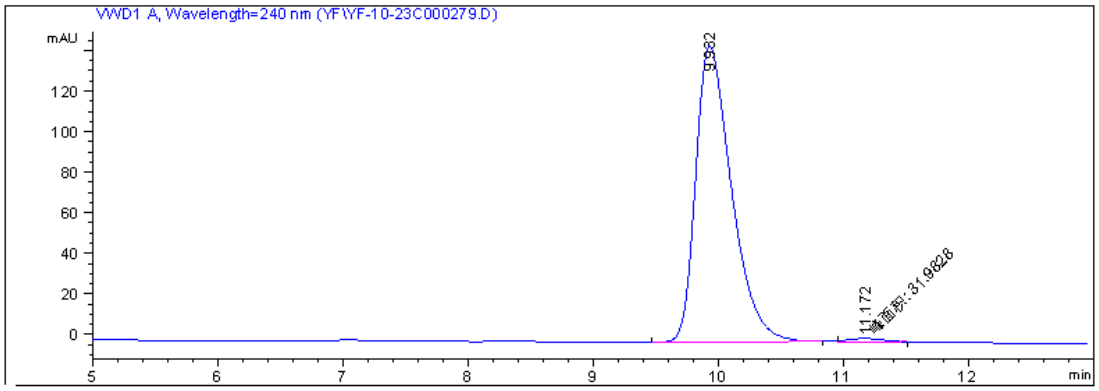
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.871	BB	0.2926	5716.16016	296.90973	99.3561
2	12.332	BB	0.2920	37.04686	1.97327	0.6439



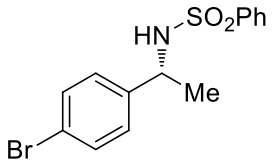
3ea



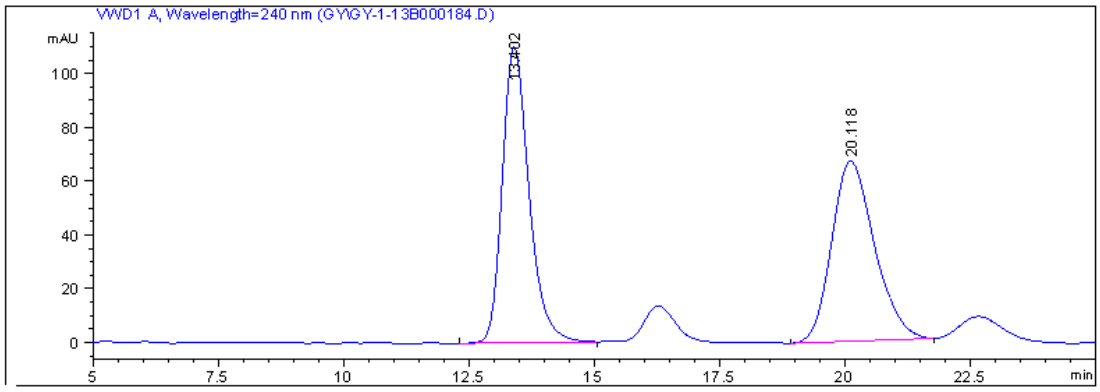
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.786	BV	0.2992	5695.89355	288.72711	49.5854
2	10.769	VB	0.3788	5791.15186	229.94351	50.4146



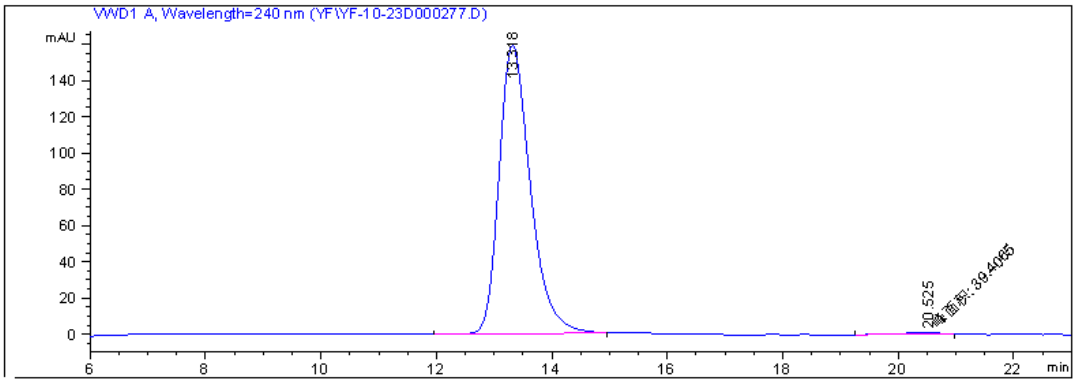
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.932	BB	0.3028	2893.77710	145.66599	98.9069
2	11.172	MM	0.3191	31.98281	1.67054	1.0931



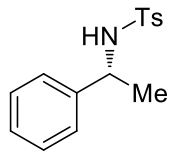
3fa



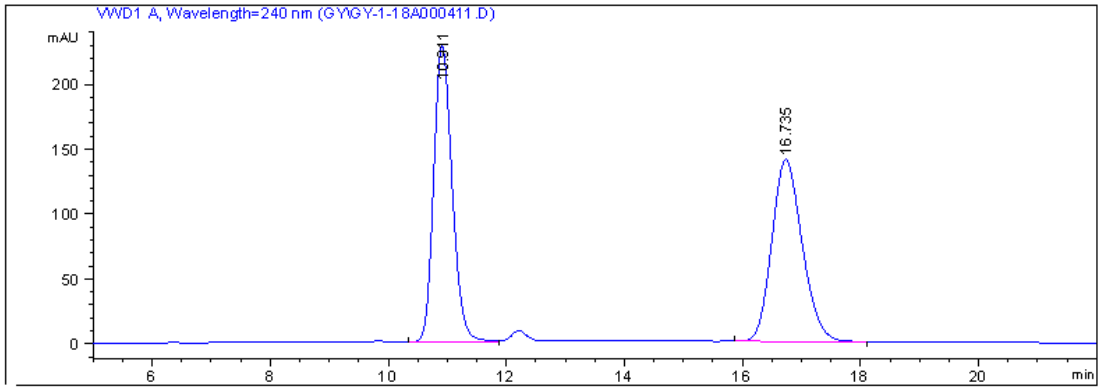
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.402	BB	0.5667	4072.95020	110.07288	50.6292
2	20.118	BB	0.9098	3971.71948	66.98355	49.3708



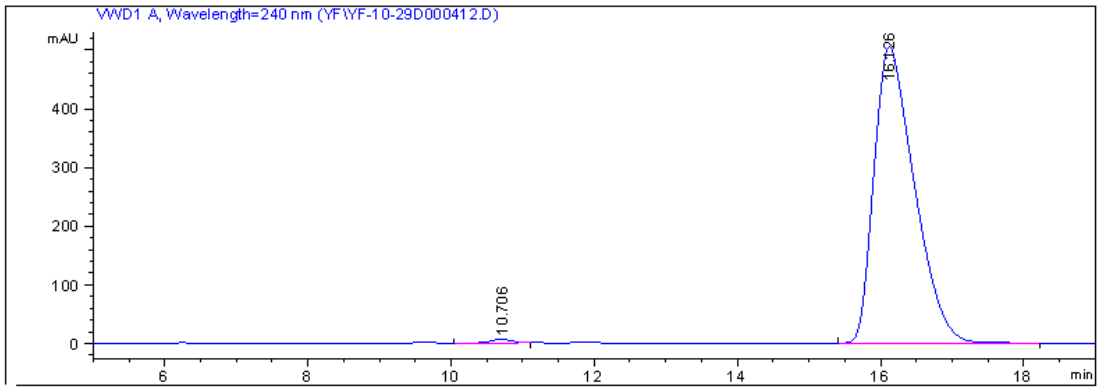
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.318	BB	0.5790	5990.16309	158.83418	99.3464
2	20.525	MM	0.7126	39.40650	9.21684e-1	0.6536



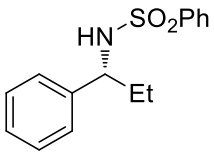
3ga



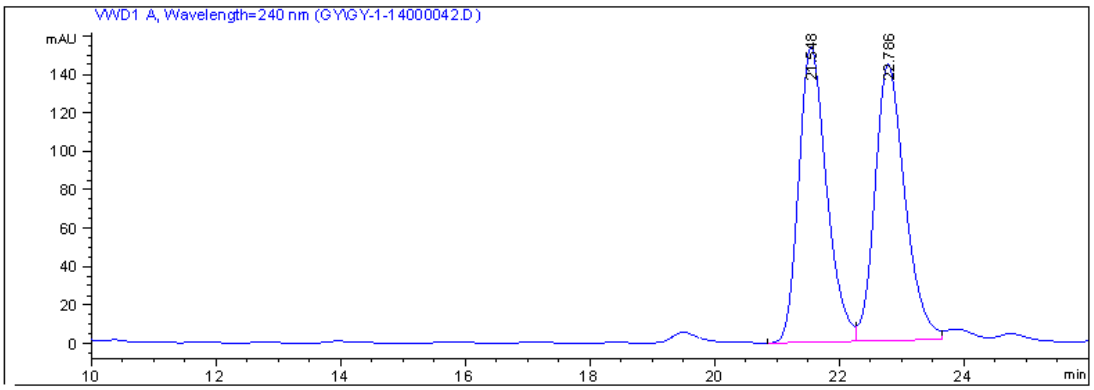
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.911	BV	0.3375	4954.62744	228.06793	49.7489
2	16.735	BB	0.5481	5004.64063	140.33078	50.2511



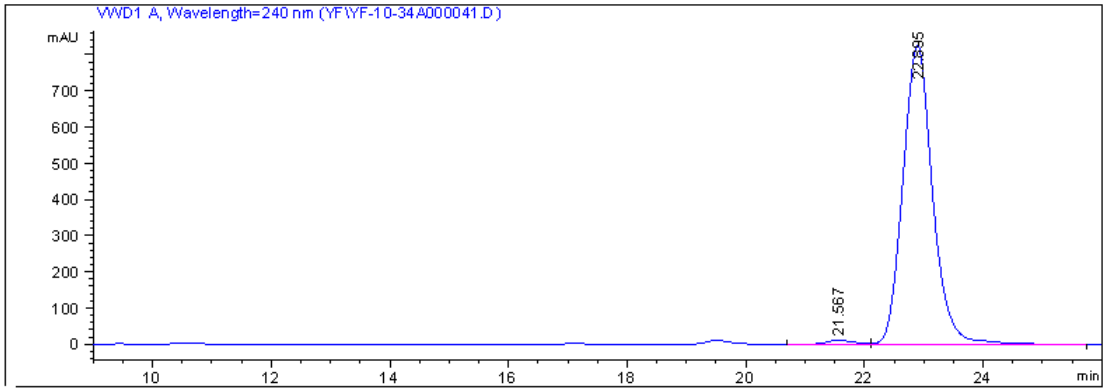
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.706	BB	0.3104	125.07738	6.30638	0.6286
2	16.126	BB	0.6006	1.97736e4	505.24591	99.3714



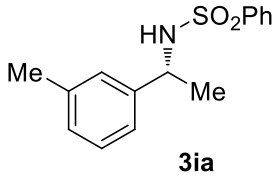
3ha



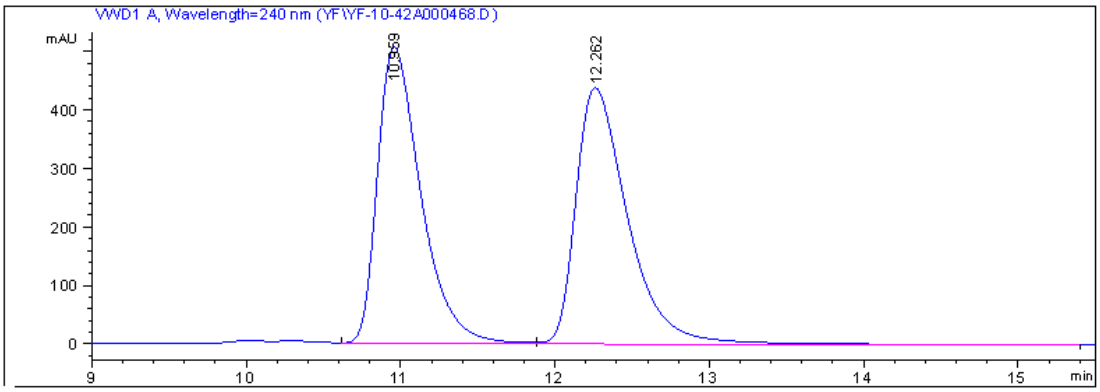
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.548	BV	0.4749	4770.20264	153.33679	49.7812
2	22.786	VV	0.5088	4812.12842	143.67285	50.2188



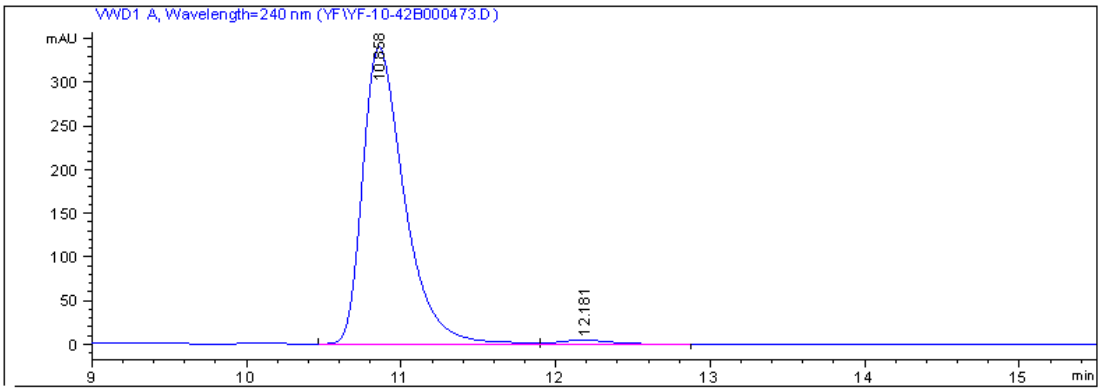
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.567	BV	0.4773	349.90408	11.14547	1.2056
2	22.895	VB	0.5324	2.86729e4	825.13385	98.7944



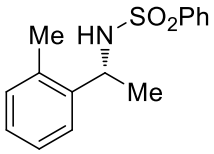
3ia



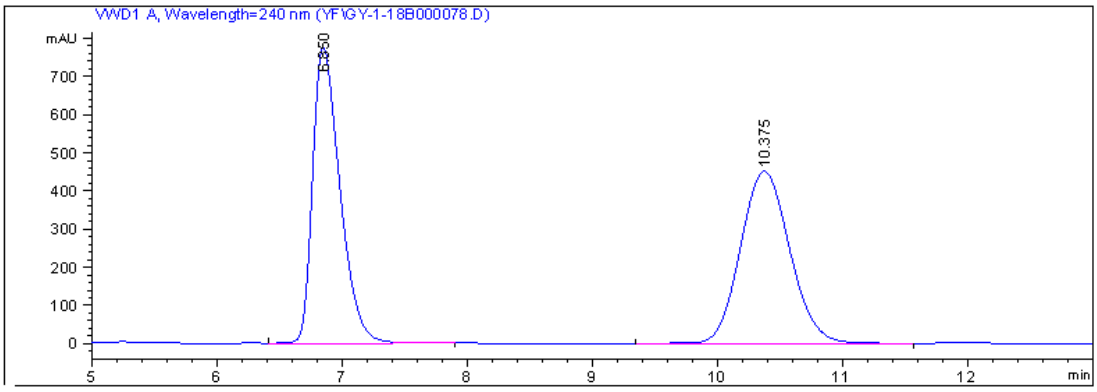
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.959	VV	0.2988	9949.96582	507.33347	49.4062
2	12.262	VB	0.3536	1.01891e4	437.75040	50.5938



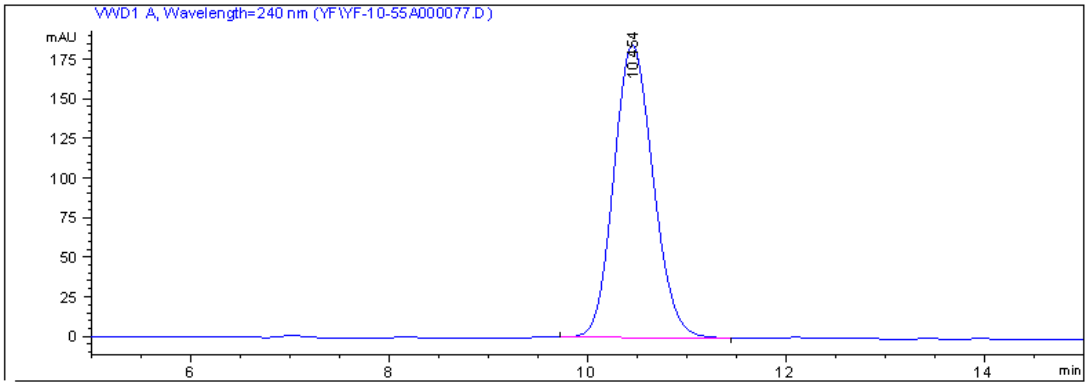
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.858	BV	0.2847	6378.53760	340.29050	98.4078
2	12.181	VB	0.3266	103.20136	4.71044	1.5922



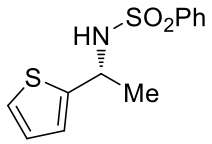
3ja



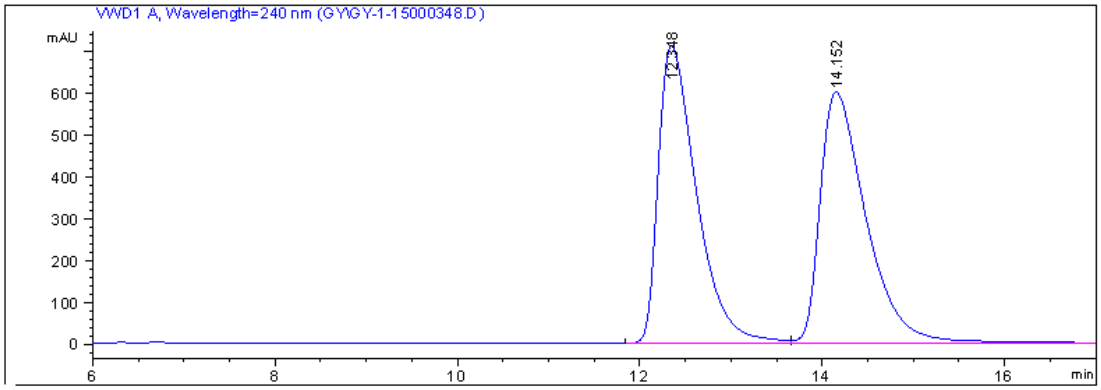
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.850	VB	0.2332	1.18089e4	777.28241	48.9398
2	10.375	BB	0.4291	1.23206e4	450.41965	51.0602



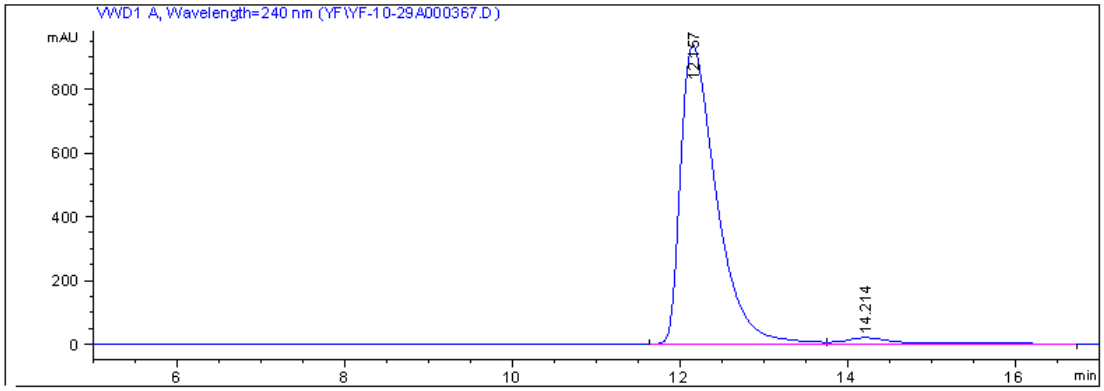
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.454	BB	0.4315	5101.67480	184.53714	100.0000



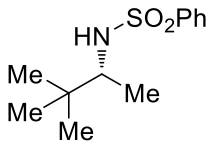
3ka



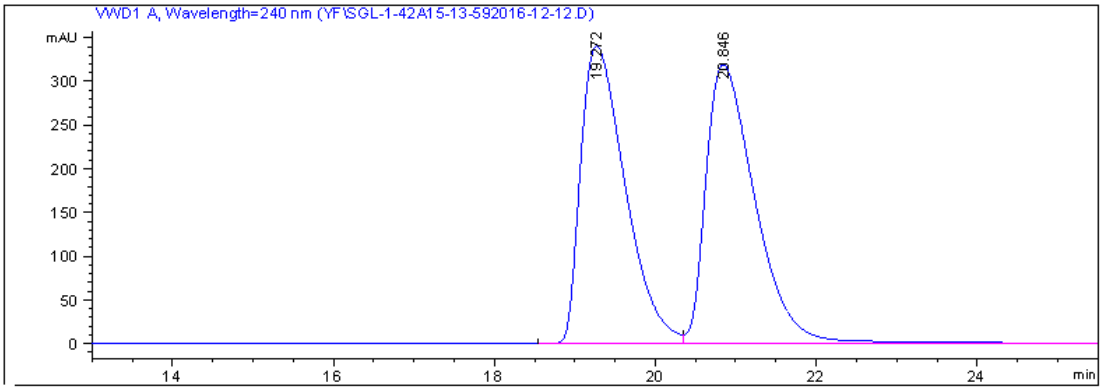
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.348	BV	0.4451	2.06360e4	709.90100	49.7537
2	14.152	VBA	0.5277	2.08404e4	599.23828	50.2463



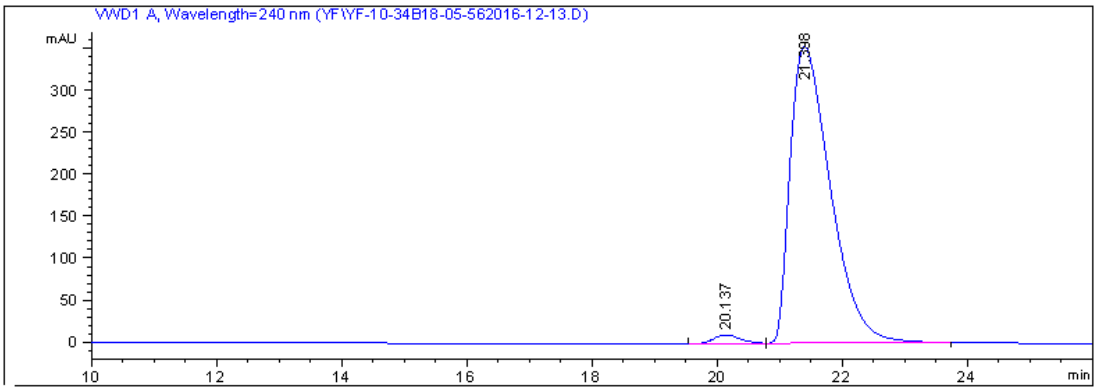
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.157	BV	0.4498	2.77262e4	935.51746	97.4590
2	14.214	VB	0.5733	722.90405	18.36365	2.5410



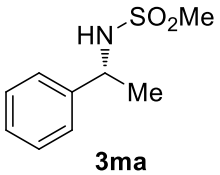
3la



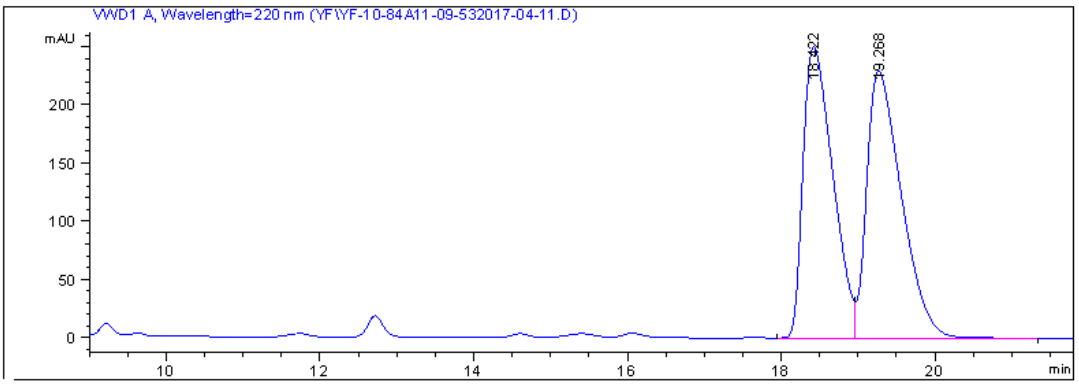
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.272	BV	0.6003	1.30183e4	340.28882	49.1391
2	20.846	VBA	0.6505	1.34745e4	318.57135	50.8609



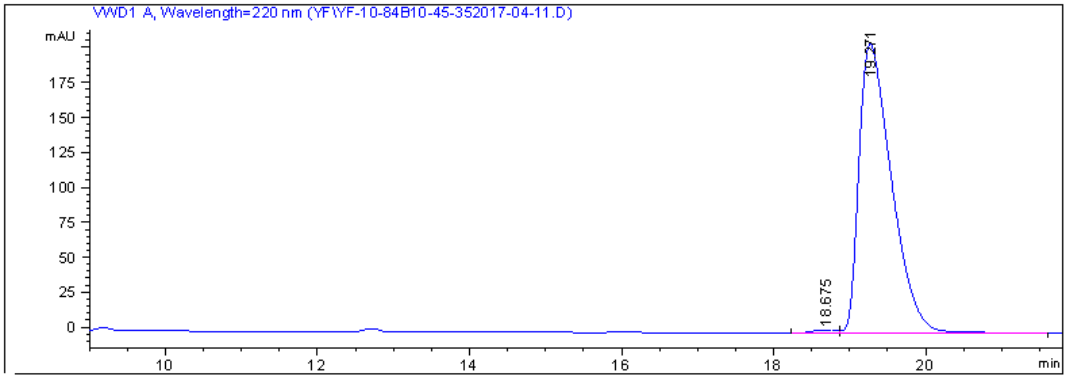
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.137	BV	0.4343	290.09961	10.34066	1.8081
2	21.398	VB	0.6980	1.57540e4	352.79245	98.1919



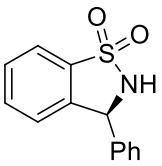
3ma



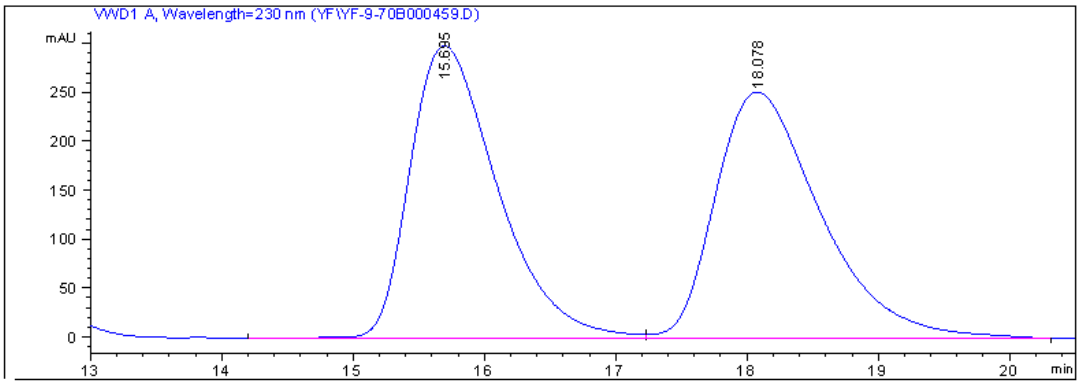
#	[min]		[min]	[mAU*s]	[mAU]	%
1	18.422	BV	0.4242	6731.70996	250.73637	48.3197
2	19.268	VB	0.4817	7199.88379	229.68756	51.6803



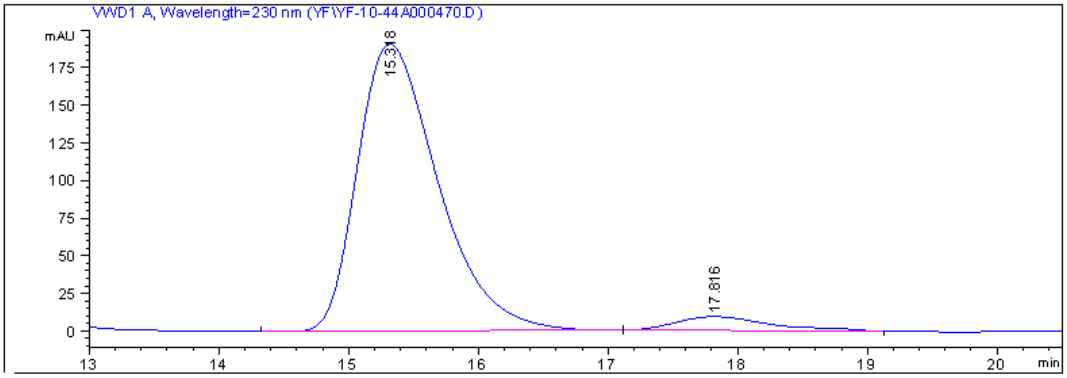
#	[min]		[min]	[mAU*s]	[mAU]	%
1	18.675	BV	0.3042	40.95028	2.10282	0.6632
2	19.271	VB	0.4544	6133.78955	206.60033	99.3368



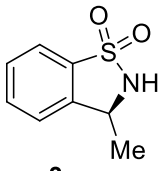
3pa



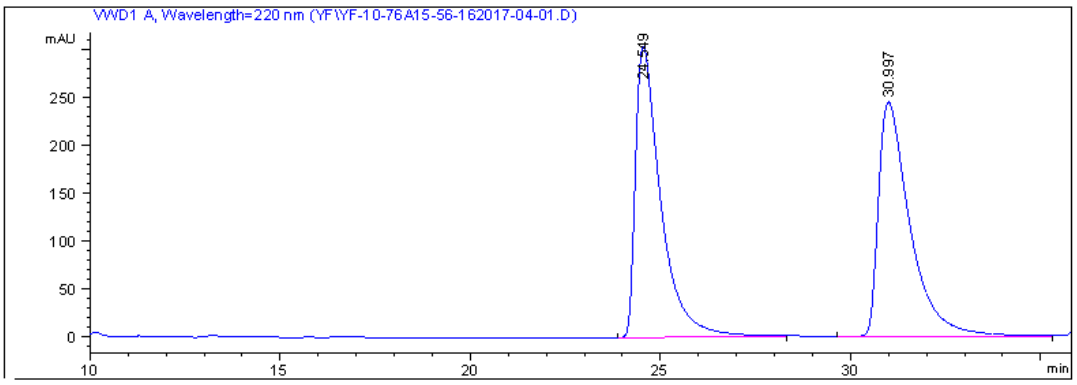
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.695	BV	0.7141	1.37966e4	298.54706	49.7327
2	18.078	VB	0.8524	1.39450e4	251.29665	50.2673



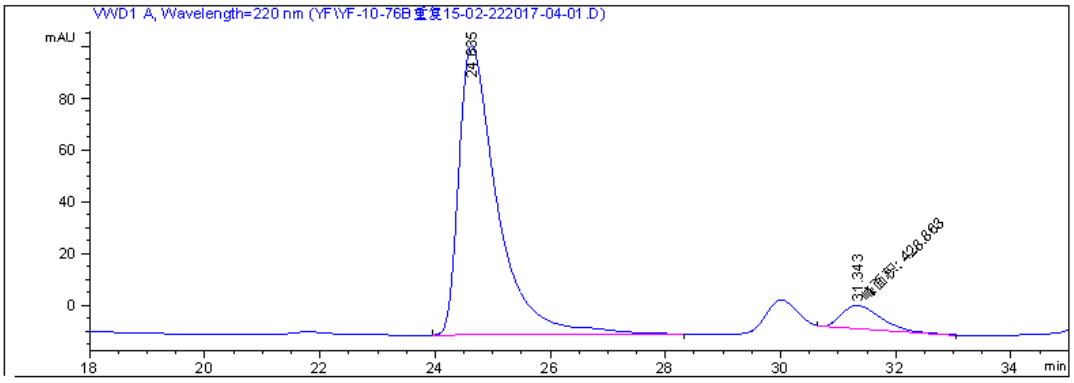
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.318	BB	0.6659	8215.36719	190.59251	94.7649
2	17.816	BB	0.7735	453.83926	9.05881	5.2351



3qa

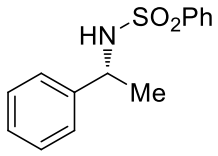


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.549	BB	0.6865	1.41048e4	303.89590	50.0131
2	30.997	BB	0.8585	1.40974e4	244.93741	49.9869



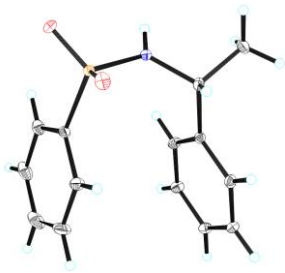
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.635	BB	0.6860	5185.42285	112.02570	92.3612
2	31.343	MM	0.7962	428.86328	8.97720	7.6388

F: X-Ray Crystallography of Compound 3aa



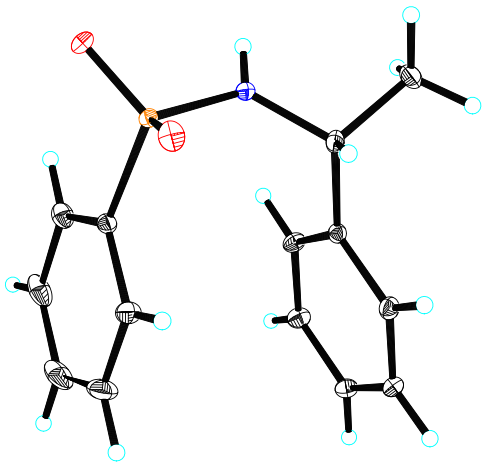
3aa

≡



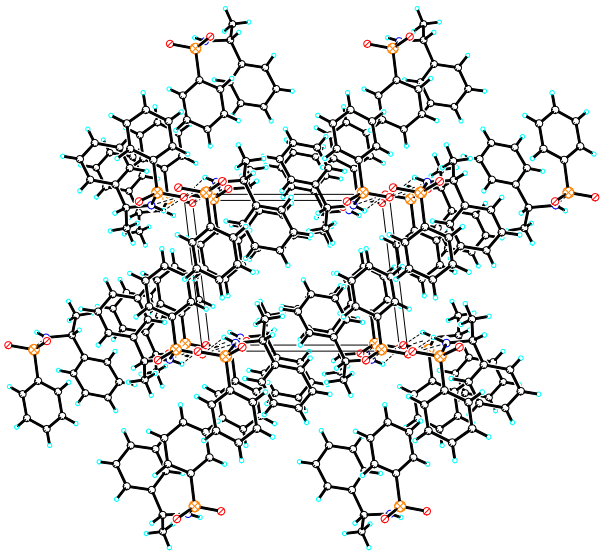
X-ray of **3aa** (CCDC 1820486)

Crystal data for mo_vyf961a_0m: $C_{14}H_{15}NO_2S$, M = 261.33, a = 8.4454(12) Å, b = 7.2057(10) Å, c = 10.9962(16) Å, α = 90 °, β = 96.797(2) °, γ = 90 °, V = 664.47(16) Å^3, T = 100(2) K, space group $P21$, Z = 2, μ(MoKα) = 0.237 mm^{-1}, 7384 reflections measured, 3779 independent reflections (R_{int} = 0.0175). The final R_1 values were 0.0271 ($I > 2\sigma(I)$). The final $wR(F^2)$ values were 0.0710 ($I > 2\sigma(I)$). The final R_1 values were 0.0278 (all data). The final $wR(F^2)$ values were 0.0714 (all data). The goodness of fit on F^2 was 1.077. Flack parameter = 0.029(19).



View of a molecule of vyf961a with the atom-labelling scheme.

Displacement ellipsoids are drawn at the 30% probability level.



View of the pack drawing of vyf961a.

Hydrogen-bonds are shown as dashed lines.

Table 1. Crystal data and structure refinement for mo_vyf961a_0m.

Identification code	mo_vyf961a_0m	
Empirical formula	C14 H15 N O2 S	
Formula weight	261.33	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	a = 8.4454(12) Å	α= 90 °.
	b = 7.2057(10) Å	β= 96.797(2) °.
	c = 10.9962(16) Å	γ = 90 °.
Volume	664.47(16) $Å^3$	
Z	2	
Density (calculated)	1.306 Mg/m^3	
Absorption coefficient	0.237 mm^{-1}	
F(000)	276	
Crystal size	1.200 x 0.270 x 0.250 mm^3	
Theta range for data collection	1.865 to 30.885 °.	
Index ranges	-12<=h<=12, -10<=k<=10, -15<=l<=15	
Reflections collected	7384	
Independent reflections	3779 [R(int) = 0.0175]	
Completeness to theta = 25.242 °	99.8 %	

Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3779 / 1 / 164
Goodness-of-fit on F^2	1.077
Final R indices [I>2sigma(I)]	R1 = 0.0271, wR2 = 0.0710
R indices (all data)	R1 = 0.0278, wR2 = 0.0714
Absolute structure parameter	0.029(19)
Extinction coefficient	n/a
Largest diff. peak and hole	0.386 and -0.398 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for mo_vyf961a_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	114(1)	10025(1)	8729(1)	13(1)
O(1)	-192(1)	10136(2)	9991(1)	21(1)
O(2)	-509(2)	11425(2)	7876(1)	21(1)
N(1)	-628(2)	8050(2)	8253(1)	14(1)
C(1)	5425(2)	9508(3)	8624(3)	37(1)
C(2)	4751(2)	8856(3)	9639(2)	31(1)
C(3)	3126(2)	9052(3)	9690(2)	22(1)
C(4)	2198(2)	9922(3)	8720(1)	15(1)
C(5)	-769(2)	7504(2)	6953(1)	16(1)
C(6)	780(2)	6841(2)	6516(1)	14(1)
C(7)	1908(2)	5814(2)	7268(1)	17(1)
C(8)	3305(2)	5220(3)	6838(2)	20(1)
C(9)	3579(2)	5606(3)	5638(2)	20(1)
C(10)	2452(2)	6599(3)	4878(2)	20(1)
C(11)	1068(2)	7226(2)	5319(1)	17(1)
C(12)	-2055(2)	5999(3)	6766(2)	26(1)
C(13)	2861(2)	10593(3)	7705(2)	23(1)
C(14)	4487(2)	10368(3)	7666(2)	34(1)

Table 3. Bond lengths [Å] and angles [°] for mo_vyf961a_0m.

S(1)-O(2)	1.4339(13)
S(1)-O(1)	1.4435(12)
S(1)-N(1)	1.6166(14)
S(1)-C(4)	1.7632(14)
N(1)-C(5)	1.474(2)
N(1)-H(4)	0.8800
C(1)-C(14)	1.388(4)
C(1)-C(2)	1.393(4)
C(1)-H(1)	0.9500
C(2)-C(3)	1.387(3)
C(2)-H(15)	0.9500
C(3)-C(4)	1.395(2)
C(3)-H(3)	0.9500
C(4)-C(13)	1.393(2)
C(5)-C(6)	1.523(2)
C(5)-C(12)	1.531(2)
C(5)-H(5)	1.0000
C(6)-C(11)	1.395(2)
C(6)-C(7)	1.397(2)
C(7)-C(8)	1.389(2)
C(7)-H(9)	0.9500
C(8)-C(9)	1.395(2)
C(8)-H(8)	0.9500
C(9)-C(10)	1.388(2)
C(9)-H(2)	0.9500
C(10)-C(11)	1.392(2)
C(10)-H(6)	0.9500
C(11)-H(7)	0.9500
C(12)-H(12)	0.9800
C(12)-H(11)	0.9800
C(12)-H(10)	0.9800
C(13)-C(14)	1.388(3)
C(13)-H(14)	0.9500
C(14)-H(13)	0.9500
O(2)-S(1)-O(1)	119.74(8)
O(2)-S(1)-N(1)	108.06(8)

O(1)-S(1)-N(1)	104.65(8)
O(2)-S(1)-C(4)	108.39(8)
O(1)-S(1)-C(4)	107.46(7)
N(1)-S(1)-C(4)	108.04(8)
C(5)-N(1)-S(1)	122.17(12)
C(5)-N(1)-H(4)	118.9
S(1)-N(1)-H(4)	118.9
C(14)-C(1)-C(2)	120.54(18)
C(14)-C(1)-H(1)	119.7
C(2)-C(1)-H(1)	119.7
C(3)-C(2)-C(1)	119.89(19)
C(3)-C(2)-H(15)	120.1
C(1)-C(2)-H(15)	120.1
C(2)-C(3)-C(4)	118.91(18)
C(2)-C(3)-H(3)	120.5
C(4)-C(3)-H(3)	120.5
C(13)-C(4)-C(3)	121.73(15)
C(13)-C(4)-S(1)	119.07(12)
C(3)-C(4)-S(1)	119.04(12)
N(1)-C(5)-C(6)	114.55(12)
N(1)-C(5)-C(12)	107.08(14)
C(6)-C(5)-C(12)	110.95(14)
N(1)-C(5)-H(5)	108.0
C(6)-C(5)-H(5)	108.0
C(12)-C(5)-H(5)	108.0
C(11)-C(6)-C(7)	118.77(14)
C(11)-C(6)-C(5)	119.14(14)
C(7)-C(6)-C(5)	122.06(14)
C(8)-C(7)-C(6)	120.55(15)
C(8)-C(7)-H(9)	119.7
C(6)-C(7)-H(9)	119.7
C(7)-C(8)-C(9)	120.25(15)
C(7)-C(8)-H(8)	119.9
C(9)-C(8)-H(8)	119.9
C(10)-C(9)-C(8)	119.56(15)
C(10)-C(9)-H(2)	120.2
C(8)-C(9)-H(2)	120.2
C(9)-C(10)-C(11)	120.11(15)

C(9)-C(10)-H(6)	119.9
C(11)-C(10)-H(6)	119.9
C(10)-C(11)-C(6)	120.74(15)
C(10)-C(11)-H(7)	119.6
C(6)-C(11)-H(7)	119.6
C(5)-C(12)-H(12)	109.5
C(5)-C(12)-H(11)	109.5
H(12)-C(12)-H(11)	109.5
C(5)-C(12)-H(10)	109.5
H(12)-C(12)-H(10)	109.5
H(11)-C(12)-H(10)	109.5
C(14)-C(13)-C(4)	118.51(18)
C(14)-C(13)-H(14)	120.7
C(4)-C(13)-H(14)	120.7
C(1)-C(14)-C(13)	120.42(19)
C(1)-C(14)-H(13)	119.8
C(13)-C(14)-H(13)	119.8

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for mo_vyf961a_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	12(1)	14(1)	13(1)	-2(1)	2(1)	1(1)
O(1)	22(1)	25(1)	16(1)	-7(1)	7(1)	-1(1)
O(2)	21(1)	16(1)	24(1)	1(1)	-2(1)	3(1)
N(1)	14(1)	15(1)	14(1)	-1(1)	3(1)	-2(1)
C(1)	15(1)	31(1)	65(2)	-17(1)	9(1)	-2(1)
C(2)	17(1)	25(1)	49(1)	-8(1)	-7(1)	2(1)
C(3)	18(1)	20(1)	26(1)	-2(1)	-2(1)	1(1)
C(4)	12(1)	15(1)	18(1)	-4(1)	3(1)	0(1)
C(5)	16(1)	17(1)	13(1)	-3(1)	-1(1)	2(1)
C(6)	15(1)	12(1)	14(1)	-1(1)	1(1)	-1(1)
C(7)	19(1)	19(1)	15(1)	3(1)	4(1)	3(1)
C(8)	18(1)	21(1)	20(1)	2(1)	4(1)	5(1)
C(9)	17(1)	25(1)	20(1)	-2(1)	7(1)	0(1)

C(10)	21(1)	25(1)	14(1)	-1(1)	4(1)	-4(1)
C(11)	19(1)	19(1)	14(1)	1(1)	-1(1)	-1(1)
C(12)	16(1)	30(1)	32(1)	-14(1)	4(1)	-5(1)
C(13)	24(1)	23(1)	23(1)	-2(1)	9(1)	-5(1)
C(14)	27(1)	35(1)	45(1)	-10(1)	22(1)	-10(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for mo_vyf961a_0m.

	x	y	z	U(eq)
H(4)	-958	7276	8788	17
H(1)	6536	9362	8587	44
H(15)	5403	8279	10295	38
H(3)	2652	8600	10374	26
H(5)	-1153	8606	6449	19
H(9)	1718	5518	8080	21
H(8)	4076	4550	7364	23
H(2)	4530	5191	5342	24
H(6)	2626	6852	4056	24
H(7)	312	7924	4798	21
H(12)	-1703	4903	7252	39
H(11)	-2236	5660	5898	39
H(10)	-3049	6470	7029	39
H(14)	2215	11191	7055	28
H(13)	4960	10807	6978	41

Table 6. Torsion angles [°] for mo_vyf961a_0m.

O(2)-S(1)-N(1)-C(5)	42.60(14)
O(1)-S(1)-N(1)-C(5)	171.24(12)
C(4)-S(1)-N(1)-C(5)	-74.47(13)
C(14)-C(1)-C(2)-C(3)	0.6(3)
C(1)-C(2)-C(3)-C(4)	-0.7(3)
C(2)-C(3)-C(4)-C(13)	0.1(3)

C(2)-C(3)-C(4)-S(1)	175.51(15)
O(2)-S(1)-C(4)-C(13)	-21.82(18)
O(1)-S(1)-C(4)-C(13)	-152.55(15)
N(1)-S(1)-C(4)-C(13)	95.04(16)
O(2)-S(1)-C(4)-C(3)	162.69(14)
O(1)-S(1)-C(4)-C(3)	31.96(17)
N(1)-S(1)-C(4)-C(3)	-80.46(15)
S(1)-N(1)-C(5)-C(6)	78.20(16)
S(1)-N(1)-C(5)-C(12)	-158.32(12)
N(1)-C(5)-C(6)-C(11)	-145.80(15)
C(12)-C(5)-C(6)-C(11)	92.83(18)
N(1)-C(5)-C(6)-C(7)	35.9(2)
C(12)-C(5)-C(6)-C(7)	-85.43(19)
C(11)-C(6)-C(7)-C(8)	1.3(3)
C(5)-C(6)-C(7)-C(8)	179.59(16)
C(6)-C(7)-C(8)-C(9)	-1.6(3)
C(7)-C(8)-C(9)-C(10)	0.5(3)
C(8)-C(9)-C(10)-C(11)	0.8(3)
C(9)-C(10)-C(11)-C(6)	-1.1(3)
C(7)-C(6)-C(11)-C(10)	0.0(2)
C(5)-C(6)-C(11)-C(10)	-178.30(15)
C(3)-C(4)-C(13)-C(14)	0.5(3)
S(1)-C(4)-C(13)-C(14)	-174.89(15)
C(2)-C(1)-C(14)-C(13)	0.0(3)
C(4)-C(13)-C(14)-C(1)	-0.6(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for mo_vyf961a_0m [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(4)...O(1)#1	0.88	2.19	2.882(2)	134.6
N(1)-H(4)...O(1)#1	0.88	2.19	2.882(2)	134.6
N(1)-H(4)...O(1)#1	0.88	2.19	2.882(2)	134.6
N(1)-H(4)...O(1)#1	0.88	2.19	2.882(2)	134.6
N(1)-H(4)...O(1)#1	0.88	2.19	2.882(2)	134.6
N(1)-H(4)...O(1)#1	0.88	2.19	2.882(2)	134.6

N(1)-H(4)...O(1)#1	0.88	2.19	2.882(2)	134.6
N(1)-H(4)...O(1)#1	0.88	2.19	2.882(2)	134.6

Symmetry transformations used to generate equivalent atoms:

#1 -x,y-1/2,-z+2