Supplementary Material (ESI) for Chemical Communications

Enantioselective Organocatalytic Conjugate Addition of α-Aminoketone to Nitroolefins

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Supporting Information Available

I) Experimental section

II) References

III) ^{1}H NMR, ^{13}C NMR and SFC spectra of all compounds

I) Experimental Section

Experimental Data for Compounds

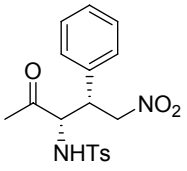
General Remarks. All reactions were not carried out under nitrogen or argon atmosphere and without dry solvents, unless otherwise noted. If needed, solvents were dried by filtration over alumina (activated at 350 °C under nitrogen atmosphere for 12 h). Chloroform was purchased from Acros and used without further purification. Pyrrolidine **6** was distilled at atmosphere pressure prior to use. (S,S)-N-*i*Pr-2,2'-bipyrrolidine **5** was prepared according to the literature procedure.[1] (*S*)-(+)-(1-pyrrolidinylmethyl)pyrrolidine **7** and (S)-(-)-(diphenyltrimethylsiloxymethyl)-pyrroldine **8** were purchased from Aldrich and used without further purification. *Tert*-butyl 2-oxopropylcarbamate **1d** [2,3] and 4-Methyl-N-(2-oxo-propyl)-benzenesulfonamide **1e** [4] were prepared according to the literature procedure. Yields refer to chromatographically and spectroscopically (^{1}H NMR) homogeneous materials, unless otherwise stated. Reactions were monitored by thin-layer chromatography (TLC) carried out on 0.25 mm E. Merck silica gel plates (60F-254) using UV lamp as visualizing agent and $KMnO_4$ solution as developing agents. Flash chromatography was performed using silica gel (particle size 32-63 μm, 60 Å).

^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra were recorded on Bruker AV-400 instrument and calibrated using residual undeuterated solvent as an internal reference. Chemical shift (δ)

are given in ppm relative to tetramethylsilane (0 ppm). Multiplicity is indicated as follows : s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), br s (broad singlet). Coupling constants *J* are reported in Hz. Mass spectra (MS) were obtained by EI (70 eV) or ESI and High resolution mass spectra HRMS by Electrospray Ionisation (ESI) or by electronic impact (EI). IR spectra were recorded on a Pelkin-Elmer Spectrum One FTIR spectrometer with diamond ATR accessory. Optical rotations were measured at 25°C in a 10 cm cell in the stated solvent; $[\alpha]_D$ values are given in 10^{-1} deg.cm^2 g^{-1} (concentration c given as g/100 mL). Enantiomeric excesses were determined by chiral Super Fluid Chromatography (SFC), with appropriated program using a gradient of methanol which are described as follows: initial methanol concentration (%) – initial time (min) – methanol gradient (%/min) – final methanol concentration (°C); retention times (R_T) are given in min.

General Procedure: Addition of α-aminoketones to nitro-olefins catalysed by amines. To a solution of pyrrolidine or chiral aminocatalyst (0.05 mmol, 15 mol%) in chloroform (3 mL) was added at 25°C the nitroolefin (0.34 mmol) and the α-aminoketone (1,68 mmol, 5 eq). The reaction mixture was stirred at 25°C until completion of the reaction whose evolution was monitored by TLC. Then the reaction mixture was hydrolysed with 3 mL of an aqueous saturated solution of NH_4Cl. The layers were separated and the aqueous phase was extracted with CH_2Cl_2. The combined organic phases were dried over Na_2SO_4, filtered and finally concentrated under reduced pressure. Purification by a flash column chromatography on silica gel using a mixture of ethyl acetate and cyclohexane as eluant gave unseparable diasteroisomers.

4-methyl-N-((2R,3S)-1-nitro-4-oxo-2-phenylpentan-3-yl)benzenesulfonamide 3e:



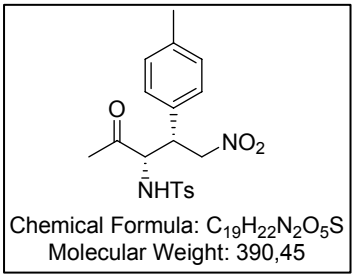
Chemical Formula: $C_{18}H_{20}N_2O_5S$
Molecular Weight: 376,43

Compound **3e** was synthesised starting from 4-methyl-N-(2-oxopropyl)benzenesulfonamide (190 mg, 0.83 mmol, 5 eq.), β-nitrostyrene (25 mg, 0.17 mmol, 1 eq.) and (S,S)-N-*i*Pr-2,2'-bipyrrolidine (3.9 mg, 0.025 mmol, 0.15 eq.) according to *General Procedure* (overnight) to give branched regioisomer as a mixture of inseparable diastereomers (*syn*/*anti* 87:13) as a yellow oil. The crude product was purified by flash column chromatography on silica gel (AcOEt/CH: 30/70) to provide branched regioisomer as a 85:15 mixture with its diastereomers *syn*/*anti* (240 mg, 80%).

FT-IR: ν 3269, 2028, 2924, 1722, 1598, 1552, 1495, 1456, 1423, 1340, 1170, 1090, 987, 908, 815, 672 cm^{-1}. **HRMS** (ESI mode) Calcd for $C_{18}H_{21}N_2O_5S$: 377.1165 $[M+H]^+$, found: 377.1171.

Syn isomer: **^{1}H NMR** (400 MHz, $CDCl_3$): 2.09 (s, 3H), 2.41 (s, 3H), 4.04-4.06 (m, 1H), 4.32-4.34 (m, 1H), 4.72-4.76 (m, 1H), 5.16-5.20 (dd, 1H, *J*= 15.1 and 8.9), 5.40-5.42 (d, 1H, *J*= 8.1), 7.09-7.11 (m, 2H), 7.27-7.31 (m, 5H), 7.62-7.64 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): 21.6 ($1CH_3$), 26.9 ($1CH_3$), 43.7 (1CH), 63.1 (1CH), 75.6 ($1CH_2$), 127.2 (2CH), 129.0 (2CH), 129.2 (1CH), 129.6 (2CH), 130.0 (2CH), 133.0 (C_{quat}), 135.4 (C_{quat}), 144.3 (C_{quat}), 201.7 (CO). The enantiomers were separated by chiral SFC (AD column: 10%-2-1-25% MeOH, 200 bars, 2 mL/min, 30°C, R_T: 5.4 min (*R*,*S*), 5.8 min (*S*,*R*). $[\alpha]_D^{20}$= +63.7 (*c* 1.0, 98% ee, *syn*/*anti* 85:15), $CHCl_3$).

4-methyl-N-((2R,3S)-1-nitro-4-oxo-2-p-tolylpentan-3-yl)benzenesulfonamide 10a:



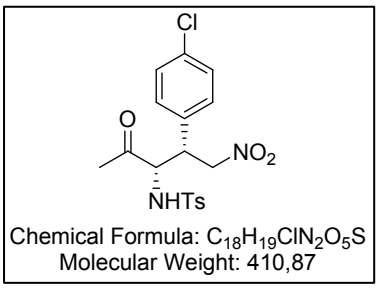
Chemical Formula: $C_{19}H_{22}N_2O_5S$
Molecular Weight: 390,45

Compound **10a** was synthesised starting from 4-methyl-N-(2-oxopropyl)benzenesulfonamide, (E)-1-methyl-4-(2-nitrovinyl)benzene and (*S*)-(+)-(1-pyrrolidinylmethyl)pyrrolidine according to *General Procedure* (overnight) to give branched regioisomer as a mixture of inseparable diastereomers (*syn*/*anti* 86:14) as an orange powder. The crude product was purified by flash column chromatography on silica gel (AcOEt/CH: 30/70) to provide a mixture of diastereomers as a white powder (111 mg, 85%).

FT-IR: ν 3279, 2923, 2854, 1722, 1598, 1552, 1516, 1425, 1378, 1334, 1161, 1090, 988, 815, 735, 671 cm^{-1}. **HRMS** (ESI mode) Calcd for $C_{19}H_{23}N_2O_5S$: 391.1325 $[M+H]^+$, found: 391.1322.

Syn isomer: **^{1}H NMR** (400 MHz, $CDCl_3$): 2.08 (s, 3H), 2.30 (s, 3H), 2.40 (s, 3H), 4.01 (m, 1H), 4.29 (dd, 1H, *J*= 8.3 and 3.0), 4.70 (dd, 1H, *J*= 14.9 and 5.8), 5.14 (dd, 1H, *J*= 14.9 and 8.3), 5.38 (d, 1H, *J*= 8.4), 6.97 (d, 2H, *J*= 8.1), 7.09 (d, 2H, *J*= 7.8), 7.26 (d, 2H, *J*= 7.8), 7.62 (d, 2H, *J*= 8.3). **^{13}C NMR** (100 MHz, $CDCl_3$): 21.2 ($1CH_3$), 21.7 ($1CH_3$), 27.1 ($1CH_3$), 43.5 (1CH), 68.3 (1CH), 75.8 ($1CH_2$), 127.4 (2CH), 128.2 (2CH), 129.9 (C_{quat}), 130.0 (C_{quat}), 130.0 (4CH), 139.0 (C_{quat}), 144.4 (C_{quat}), 202.0 (CO). The enantiomers were separated by chiral SFC (OD column: 10%-2-1-25% MeOH, 200 bars, 2 mL/min, 30°C, R_T: 7.4 min (*S,R*), 7.9 min (R,*S)*).

N-((2R,3S)-2-(4-chlorophenyl)-1-nitro-4-oxopentan-3-yl)-4-methylbenzenesulfonamide



Chemical Formula: $C_{18}H_{19}ClN_2O_5S$
Molecular Weight: 410,87

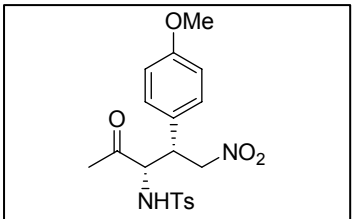
10b: Compound **10b** was synthesised starting from 4-methyl-N-(2-oxopropyl)benzenesulfonamide, (E)-1-chloro-4-(2-nitrovinyl)benzene and (*S*)-(+)-(1-pyrrolidinylmethyl)pyrrolidine according to *General Procedure* (overnight) to give branched regioisomer as a mixture of inseparable diastereomers (*syn*/*anti* 90:10) as an orange powder. The crude product was purified by flash column chromatography on silica gel (AcOEt/CH: 30/70) to provide a mixture of diastereomers as a white powder (109 mg, 79%).

FT-IR: ν 3266, 2926, 2852, 1722, 1598, 1552, 1494, 1423, 1378, 1334, 1160, 1089, 1015, 834, 814, 674 cm^{-1}. **HRMS** (ESI mode) Calcd for $C_{18}H_{20}N_2O_5SCl$: 411.0802 $[M+H]^+$, found: 411.0775.

Syn isomer: **^{1}H NMR** (400 MHz, $CDCl_3$): 2.06 (s, 3H), 2.38 (s, 3H), 4.00 (m, 1H), 4.27 (dd, 1H, *J*= 7.8 and 2.8), 4.68 (dd, 1H, *J*= 14.9 and 6.1), 5.13 (dd, 1H, *J*= 14.9 and 8.3), 5.37 (d, 1H, *J*= 8.1), 7.02 (d, 2H, *J*= 8.3), 7.23-7.26 (m, 4H), 7.58 (d, 2H, *J*= 8.3). **^{13}C NMR** (100 MHz, $CDCl_3$): 21.7 ($1CH_3$), 27.1 ($1CH_3$), 43.3 (1CH), 63.0 (1CH), 75.6 ($1CH_2$), 127.4 (2CH), 129.6 (2CH), 129.7 (2CH), 130.1 (2CH), 131.5 (C_{quat}), 135.3 (C_{quat}), 135.3 (C_{quat}), 144.6 (C_{quat}), 201.6 (CO). The enantiomers were separated by chiral SFC (OD column: 10%-2-1-25% MeOH, 200 bars, 2 mL/min, 30°C, R_T: 6.7 min (*R*,*S*), 7.9 min (*S*,*R*).

N-((2R,3S)-2-(4-methoxyphenyl)-1-nitro-4-oxopentan-3-yl)-4-methylbenzenesulfonamide

10c: Compound **10c** was synthesised starting from 4-methyl-N-(2-oxopropyl)benzenesulfonamide, ((E)-1-methoxy-4-(2-nitrovinyl)benzene and (*S*)-(+)-(1-pyrrolidinylmethyl)pyrrolidine according to *General Procedure* (36 hours) to give branched regioisomer as a mixture of inseparable diastereomers (*syn*/*anti* 83:17) as an orange powder. The crude product was purified by flash column chromatography on silica gel (AcOEt/CH: 30/70) to provide a mixture of diastereomers as an orange oil (116 mg, 85%).



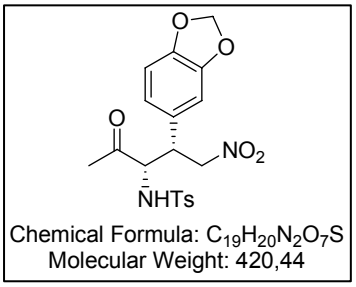
Chemical Formula: $C_{19}H_{22}N_2O_6S$
Molecular Weight: 406,45

FT-IR: ν 3274, 2925, 1723, 1611, 1553, 1515, 1425, 1379, 1337, 1253, 1163, 1090, 1033, 835, 814, 730, 703, 671 cm^{-1}. **HRMS** (ESI mode) Calcd for $C_{19}H_{23}N_2O_6S$: 407.1269 $[M+H]^+$, found: 407.1271.

Syn isomer: **^{1}H NMR** (400 MHz, $CDCl_3$): 2.07 (s, 3H), 2.39 (s, 3H), 3.75 (s, 3H), 4.01 (m, 1H), 4.28 (dd, 1H, *J*= 8.1 and 3.0), 4.70 (dd, 1H, *J*= 14.9 and 6.0), 5.12 (dd, 1H, *J*= 14.9 and

8.3), 5.41 (d, 1H, *J*= 8.3), 6.80 (d, 2H, *J*= 8.8), 7.02 (d, 2H, *J*= 8.8), 7.26 (d, 2H, *J*= 8.1), 7.61 (d, 2H, *J*= 8.3). **^{13}C NMR** (100 MHz, $CDCl_3$): 21.7 ($1CH_3$), 27.0 ($1CH_3$), 43.1 (1CH), 55.4 ($1CH_3$), 63.3 (1CH), 76.0 ($1CH_2$), 114.7 (2CH), 124.8 (C_{quat}), 127.3 (2CH), 129.4 (2CH), 130.0 (2CH), 135.5 (C_{quat}), 144.4 (C_{quat}), 160.0 (C_{quat}), 202.0 (CO). The enantiomers were separated by chiral SFC (AS column: 10%-2-1-25% MeOH, 200 bars, 2 mL/min, 45°C, R_T: 3.7 min (*R,S*), 5.4 min (S,R)).

N-((2R,3S)-2-(benzo[d][1,3]dioxol-5-yl)-1-nitro-4-oxopentan-3-yl)-4-methylbenzenesulfonamide 10d: Compound **10d** was synthesised starting from 4-methyl-N-(2-oxopropyl)benzenesulfonamide, (E)-5-(2-nitrovinyl)benzo[d][1,3]dioxole and (*S*)-(+)-(1-pyrrolidinylmethyl)pyrrolidine according to *General Procedure* (36 hours) to give branched regioisomer as a mixture of inseparable diastereomers (*syn*/*anti* 83:17) as an orange powder. The crude product was purified by flash column chromatography on silica gel (AcOEt/CH: 30/70) to provide a mixture of diastereomers as a yellow oil (121 mg, 86%).

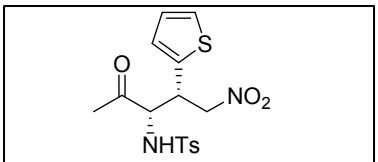


Chemical Formula: $C_{19}H_{20}N_2O_7S$
Molecular Weight: 420,44

FT-IR: ν 3267, 2922, 1721, 1598, 1553, 1505, 1490, 1446, 1339, 1250, 1160, 1089, 1037, 932, 906, 814, 734, 669, 669 cm^{-1}. **HRMS** (ESI mode) Calcd for $C_{19}H_{21}N_2O_7S$: 421.1051 $[M+H]^+$, found: 421.1063.

Syn isomer: **^{1}H NMR** (400 MHz, $CDCl_3$): 2.07 (s, 3H), 2.40 (s, 3H), 3.98 (m, 1H), 4.27 (dd, 1H, *J*= 8.1 and 3.0), 4.68 (dd, 1H, *J*= 13.6 and 6.3), 5.08 (dd, 1H, *J*= 14.7 and 8.1), 5.47 (d, 1H, *J*= 8.1), 5.94 (s, 2H), 6.54 (d, 2H, *J*= 8.1), 6.55 (s, 1H), 6.61 (m, 1H), 7.26 (d, 2H, *J*= 8.6), 7.62 (d, 2H, *J*= 8.3). **^{13}C NMR** (100 MHz, $CDCl_3$): 21.7 ($1CH_3$), 27.1 ($1CH_3$), 43.6 (1CH), 63.3 (1CH), 76.0 ($1CH_2$), 101.6 ($1CH_2$), 108.3 (1CH), 108.9 (1CH), 122.0 (1CH), 126.5 (C_{quat}), 127.4 (2CH), 130.1 (2CH), 135.4 (C_{quat}), 144.5 (C_{quat}), 148.2 ($2C_{quat}$), 201.9 (CO). The enantiomers were separated by chiral SFC (OJ column: 10%-2-1-25% MeOH, 200 bars, 2 mL/min, 45°C, R_T: 6.2 min (*R,S*), 7.1 min (S,R)).

4-methyl-N-((2R,3S)-1-nitro-4-oxo-2-(thiophen-2-yl)pentan-3-yl)benzenesulfonamide 10e: Compound **10e** was synthesised starting from 4-methyl-N-(2-oxopropyl)benzenesulfonamide, (E)-2-(2-nitrovinyl)thiophene and (*S*)-(+)-(1-pyrrolidinylmethyl)pyrrolidine according to *General Procedure* (36 hours) to give branched regioisomer as a mixture of inseparable diastereomers (*syn*/*anti* 88/12) as an orange powder.



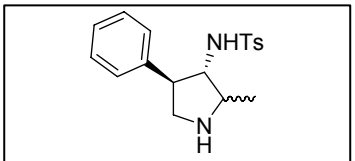
Chemical Formula: $C_{16}H_{18}N_2O_5S_2$
Molecular Weight: 382,45

The crude product was purified by flash column chromatography on silica gel (AcOEt/CH: 30/70) to provide a mixture of diastereomers as a yellow oil (106 mg, 83%).

FT-IR: ν 3286, 2924, 1722, 1598, 1554, 1505, 1424, 1378, 1339, 1160, 1090, 815, 734, 704, 672 cm^{-1}. **HRMS** (ESI mode) Calcd for $C_{16}H_{19}N_2O_7S_2$: 383.0724 $[M+H]^+$, found: 383.0729.

Syn isomer: **^{1}H NMR** (400 MHz, $CDCl_3$): 2.07 (s, 3H), 2.38 (s, 3H), 4.28 (dd, 1H, *J*= 8.1 and 2.8), 4.42 (m, 1H), 4.63 (dd, 1H, *J*= 15.2 and 5.6), 5.10 (dd, 1H, *J*= 15.1 and 8.3), 5.59 (d, 1H, *J*= 7.8), 6.87 (m, 1H), 6.91 (m, 1H), 7,20 (d, 1H, *J*= 5.1)7.23 (d, 2H, *J*= 8.2), 7.61 (d, 2H, *J*= 8.4). **^{13}C NMR** (100 MHz, $CDCl_3$): 21.7 ($1CH_3$), 26.9 ($1CH_3$), 40.0 (1CH), 63.0 (1CH), 76.2 ($1CH_2$), 126.5 (1CH), 127.4 (3CH), 130.1 (2CH), 130.2 (1CH), 134.0 (C_{quat}), 135.4 (C_{quat}), 135.4 (C_{quat}), 144.5 (C_{quat}), 201.2 (CO). The enantiomers were separated by chiral SFC (AS column: 10%-2-1-25% MeOH, 200 bars, 2 mL/min, 45°C, R_T: 4.0 min (*R,S*), 4.9 min (S,R) or AD column: 10%-2-1-25% MeOH, 200 bars, 2 mL/min, 45°C, R_T: 5.4 min (*R,S*), 6.3 min (S,R)).

4-methyl-N-((3S,4R)-2-methyl-4-phenylpyrrolidin-3-yl)benzenesulfonamide 11:



Chemical Formula: $C_{18}H_{22}N_2O_2S$
Molecular Weight: 330.44

Following the procedure described in the literature.[5] A mixture of adduct NHTs/ β-nitrostyrene (0.16 g, 0.42 mmol) and 10% $Pd(OH)_2$ on carbon in 20 mL of methanol was hydrogenated at 60psi for 48h by using a Parr apparatus. The resulting reaction mixture was filtered on celite and an acido/ basic treatment was carried out to get a yellow oil (0.11 g, 76%). **^{1}H NMR** (400 MHz, $CDCl_3$): 1.25 (bs, 1H), 1.30 (d, 3H, *J*= 6.3), 2.33 (s, 3H), 2.93-3.00 (m, 2H), 3.08 (t, 1H, 6.8), 3.33-3.37 (m, 2H), 6.92-6.96 (m, 2H), 6.97 (d, 2H, *J*= 8.1), 7.06-7.14 (m, 3H), 7.37 (d, 2H, *J*= 8.1). **^{13}C NMR** (100 MHz, $CDCl_3$): 18.7 ($1CH_3$), 21.6 ($1CH_3$), 52.8 ($1CH_2$), 53.4 (1CH), 61.4 (1CH), 68.3 (1CH), 126.6 (1CH), 126.9 (2CH), 127.3 (2CH), 128.7 (2CH), 129.5 (2CH), 137.6 (C_{quat}), 141.1 (C_{quat}), 142.9 (C_{quat}). **FT-IR**: *v* 3279, 3060, 2924, 1559, 1495, 1454, 1324, 1266, 1155, 1091, 898, 812, 734, 700 cm^{-1}. **HRMS** (ESI mode) Calcd for $C_{18}H_{23}N_2O_2S$: 331.1474 $[M+H]^+$, found: 331.1488.

II) References

1 (a) A. Alexakis, O. Andrey, *Org. Lett*., 2002, **4**, 3611. b) O. Andrey, A. Alexakis, A. Tomassini, G. Bernardinelli, *Adv. Synth. Catal*., 2004, **346**, 1147.

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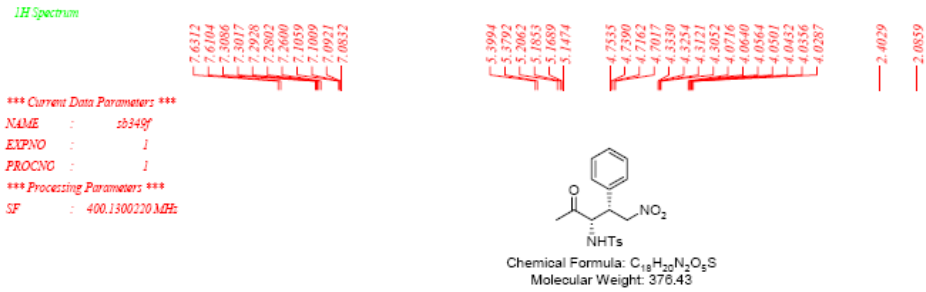
III) 1H, 13C NMR and SFC spectra of all compounds



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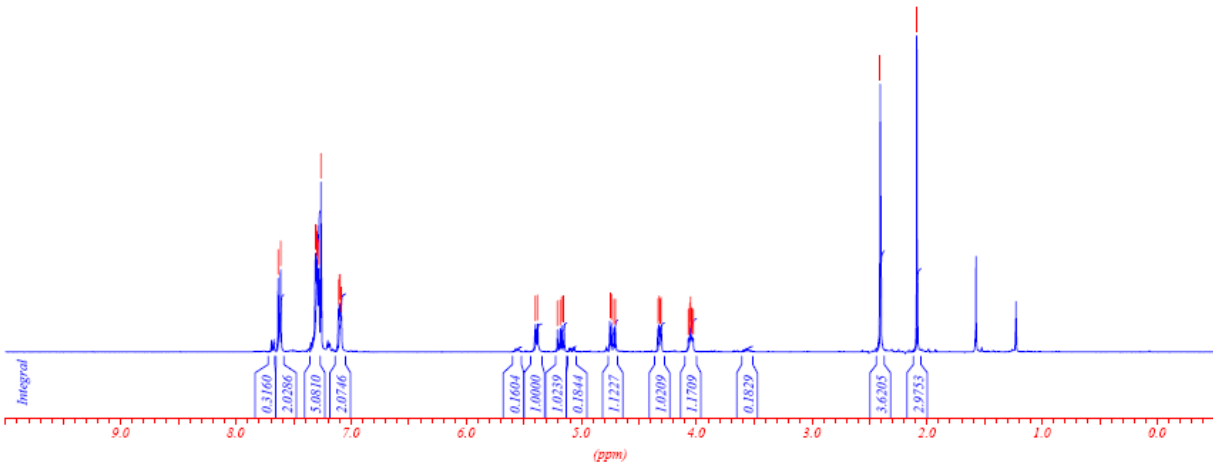

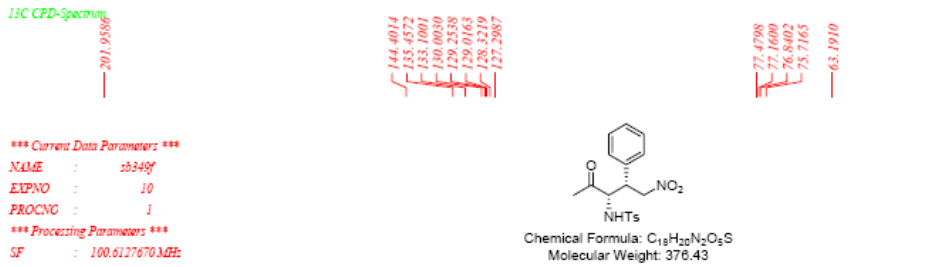

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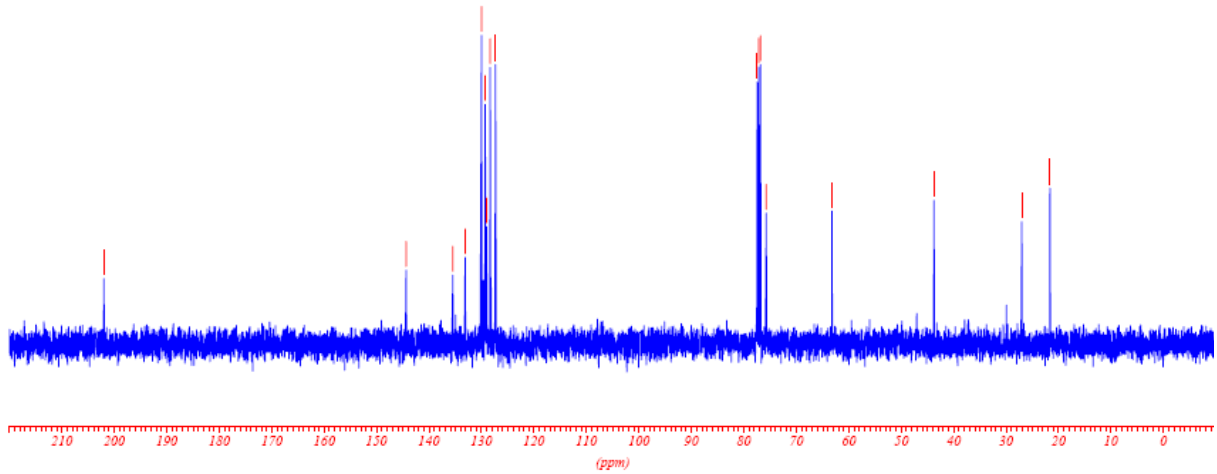

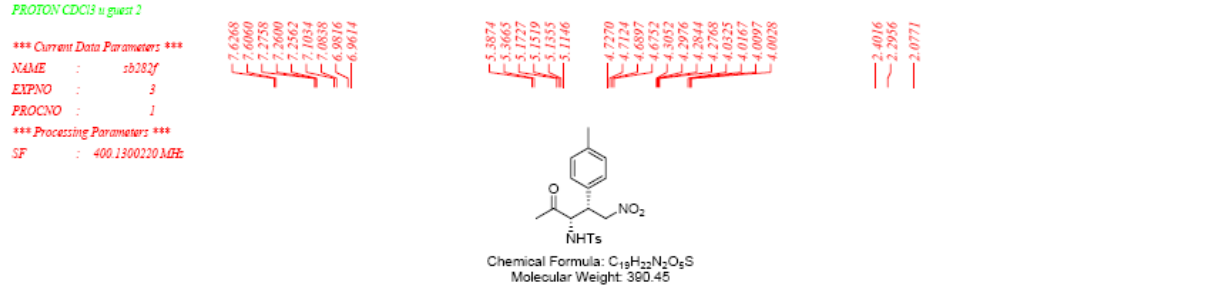
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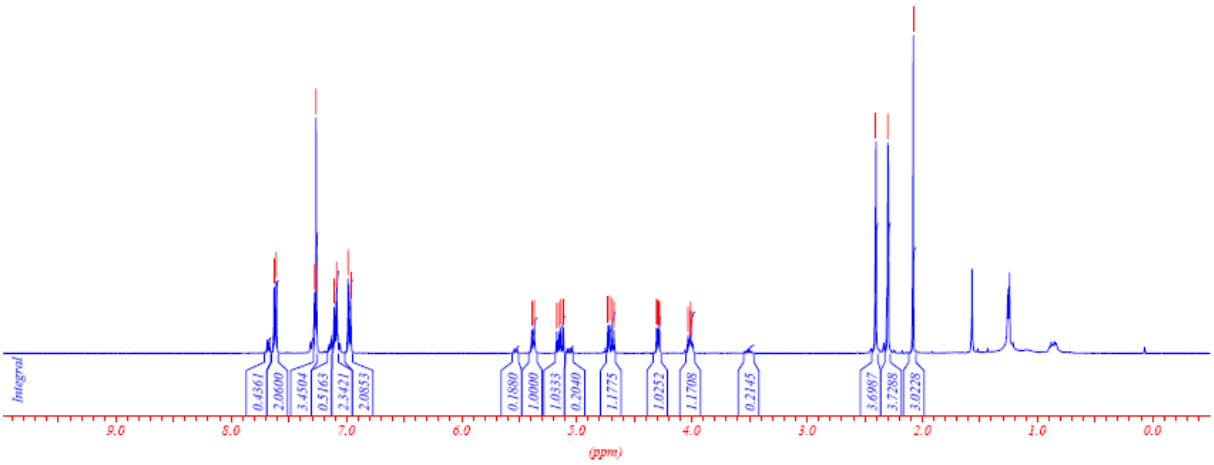
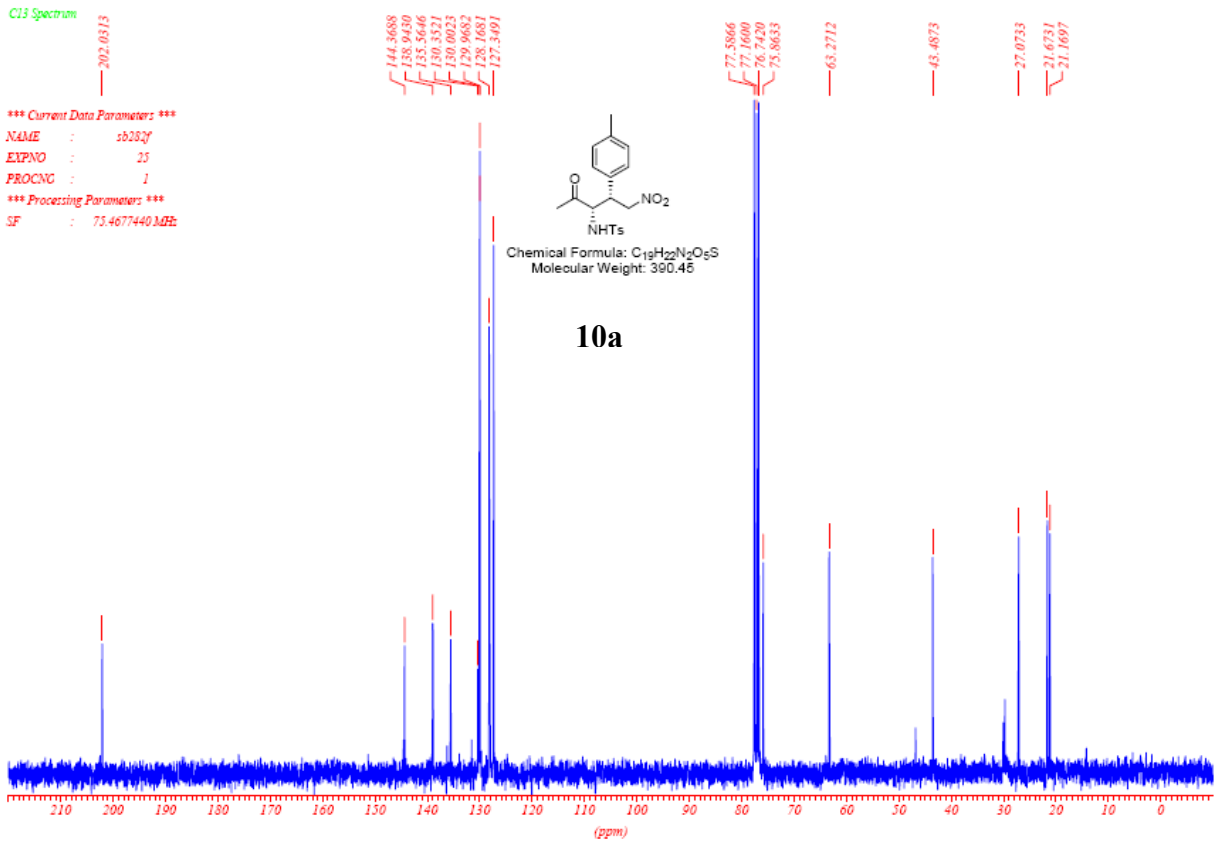
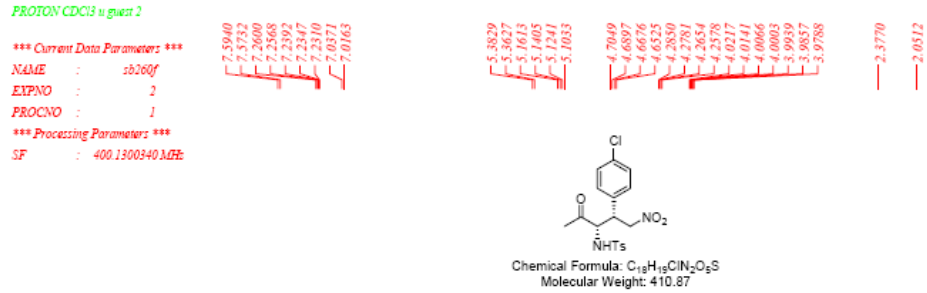
Molecular Weight: 390.45

10a

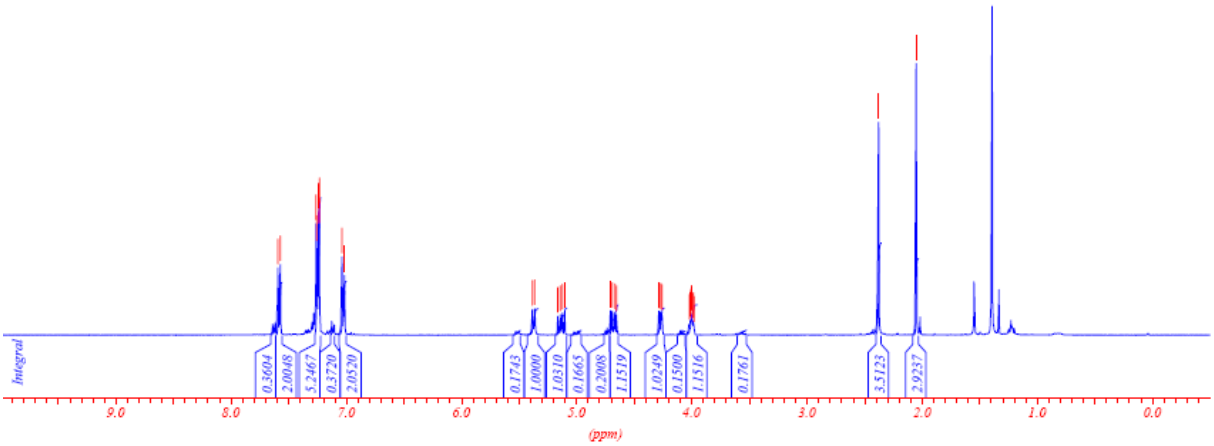

Integral
0.4361
2.0680
3.4504
0.5163
2.3421
2.0853
0.1880
1.0000
1.0333
0.2040
1.1775
1.0252
1.1708
0.2145
3.6987
3.7288
3.0228
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

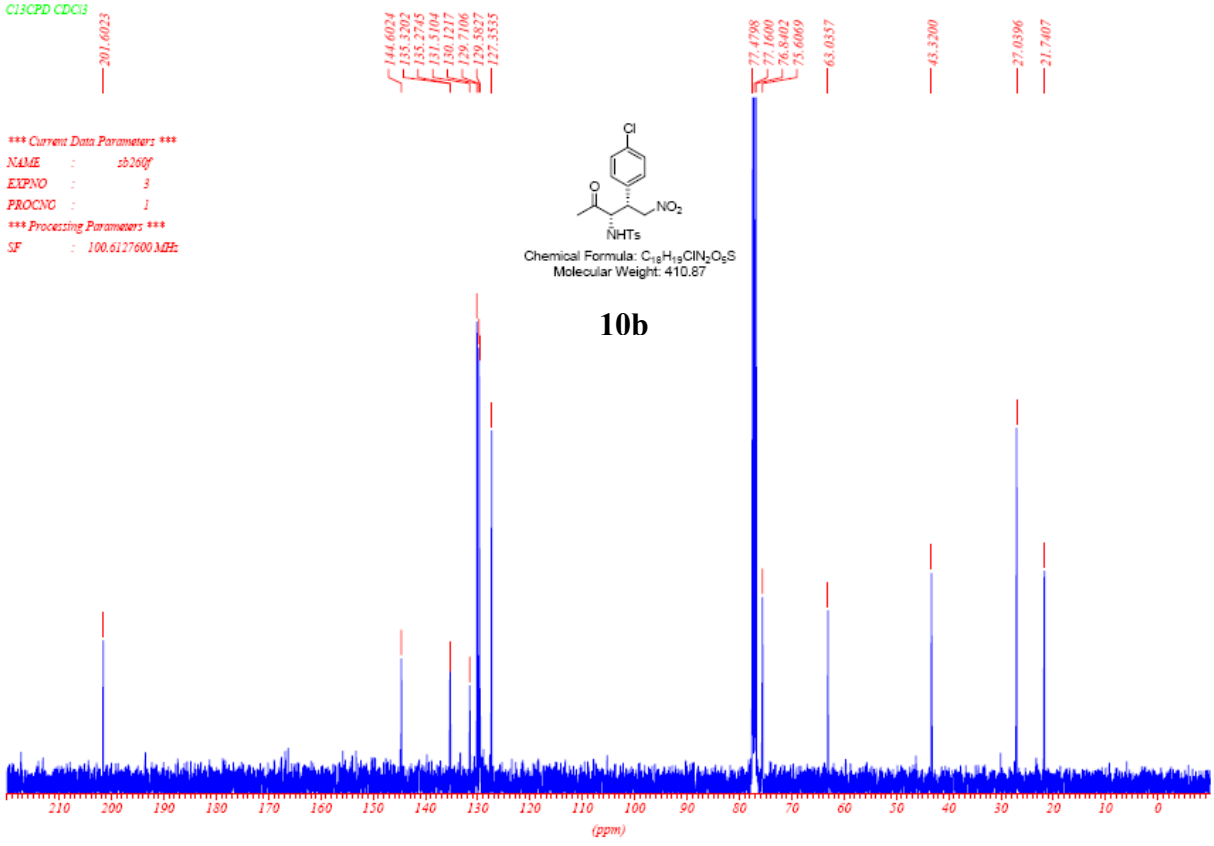

C13 Spectrum
202.0313
144.3688
138.9430
135.5646
130.3521
130.0023
129.9682
128.1681
127.3491
77.5866
77.1600
76.7420
75.8633
63.2712
43.4873
27.0733
21.6731
21.1697
*** Current Data Parameters ***
NAME : sb282f
EXPNO : 25
PROCNO : 1
*** Processing Parameters ***
SF : 75.4677440 MHz
O
NO2
NHTs
Chemical Formula: C19H22N2O5S
Molecular Weight: 390.45
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

10a

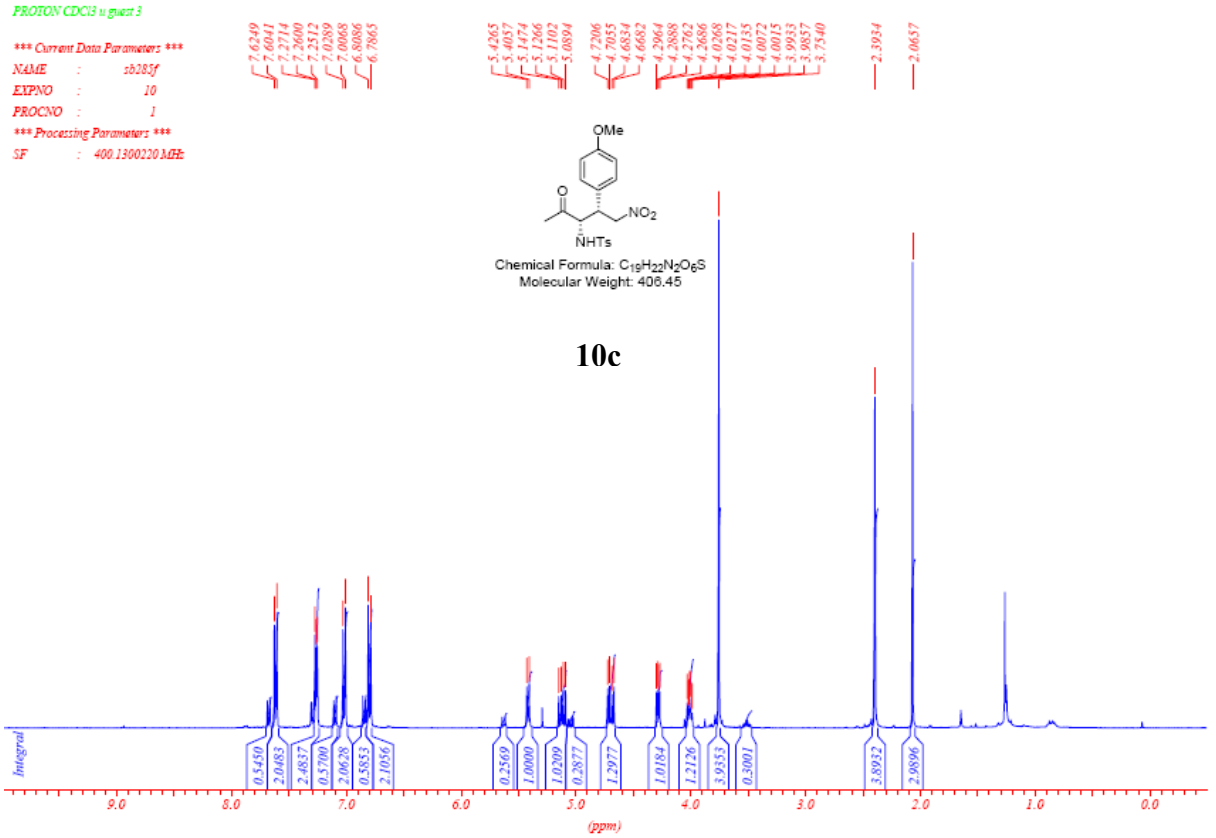

PROTON CDCl3 u guest 2
*** Current Data Parameters ***
NAME : zb260f
EXPNO : 2
PROCNO : 1
*** Processing Parameters ***
SF : 400.1300340 MHz
Cl
O
NO2
NHTs
Chemical Formula: C18H19ClN2O5S
Molecular Weight: 410.87

10b

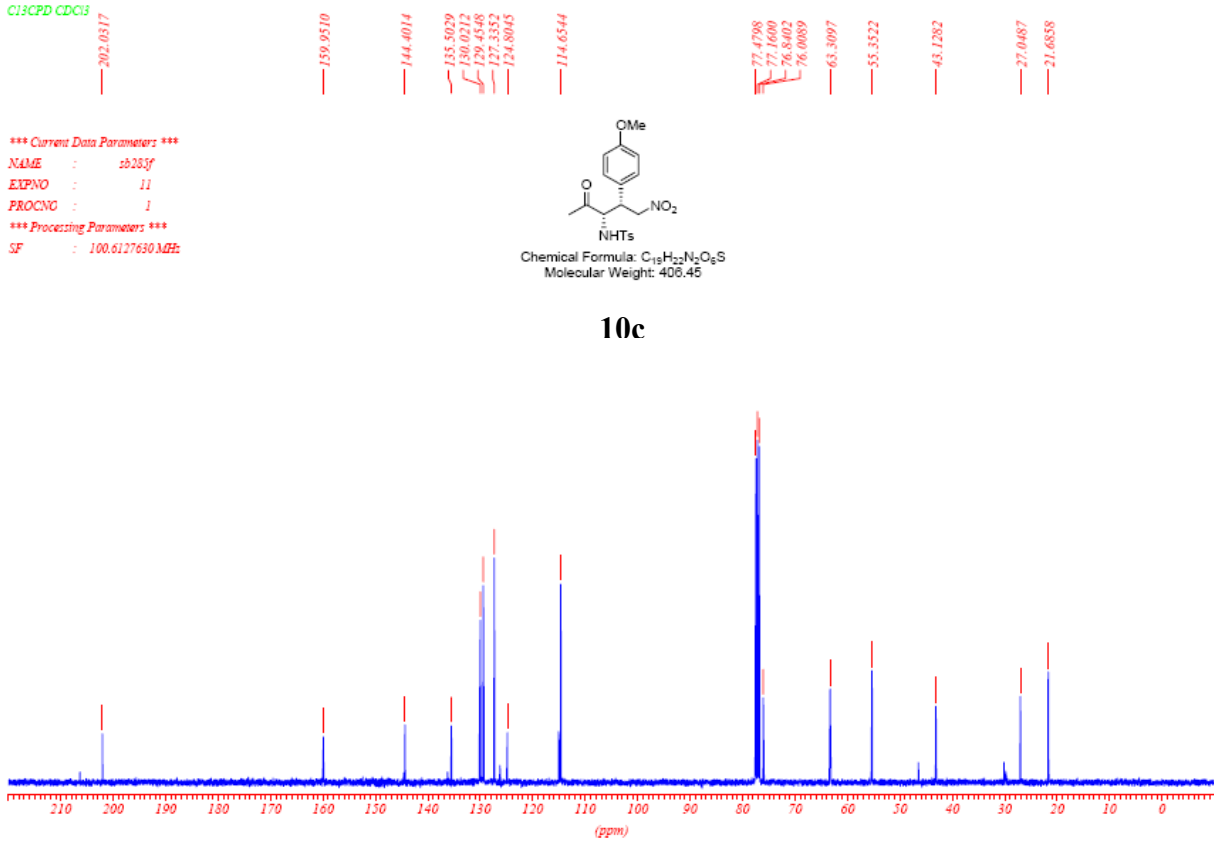

Integral
(ppm)


C13CPD CDCl3
*** Current Data Parameters ***
NAME : zb260f
EXPNO : 3
PROCNO : 1
*** Processing Parameters ***
SF : 100.6127600 MHz
Cl
O
NO2
NHTs
Chemical Formula: C18H19ClN2O5S
Molecular Weight: 410.87
(ppm)

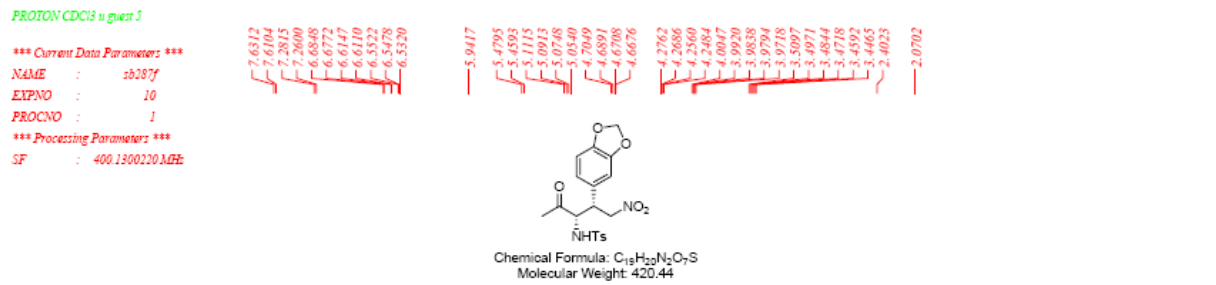
10b


PROTON CDCl3 u guest 3
*** Current Data Parameters ***
NAME : sb285f
EXPNO : 10
PROCNO : 1
*** Processing Parameters ***
SF : 400.1300220 MHz
7.6249
7.6041
7.2714
7.2600
7.2512
7.0289
7.0068
6.8086
6.7965
5.4265
5.4057
5.1474
5.1266
5.1102
5.0894
4.7206
4.7055
4.6834
4.6682
4.2964
4.2888
4.2762
4.2686
4.0268
4.0217
4.0135
4.0072
4.0015
3.9933
3.9857
3.7540
2.3934
2.0657
OMe
O
NO2
NHTs
Chemical Formula: C19H22N2O6S
Molecular Weight: 406.45
Integral
0.5450
2.0483
2.4837
0.5700
2.0628
0.5853
2.1056
0.2569
1.0000
1.0209
0.2877
1.2977
1.0184
1.2126
3.9353
0.3001
3.8932
2.9896
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

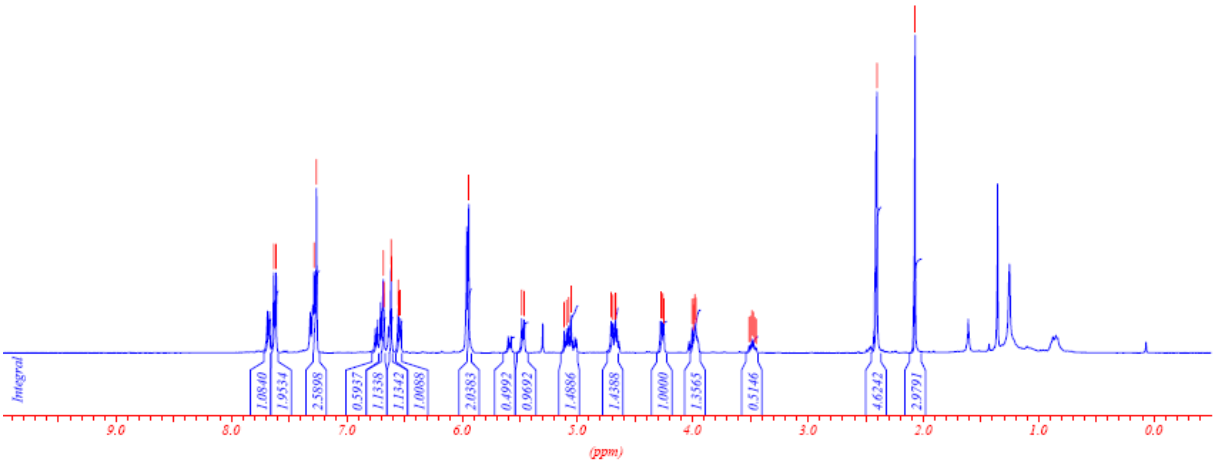
10c

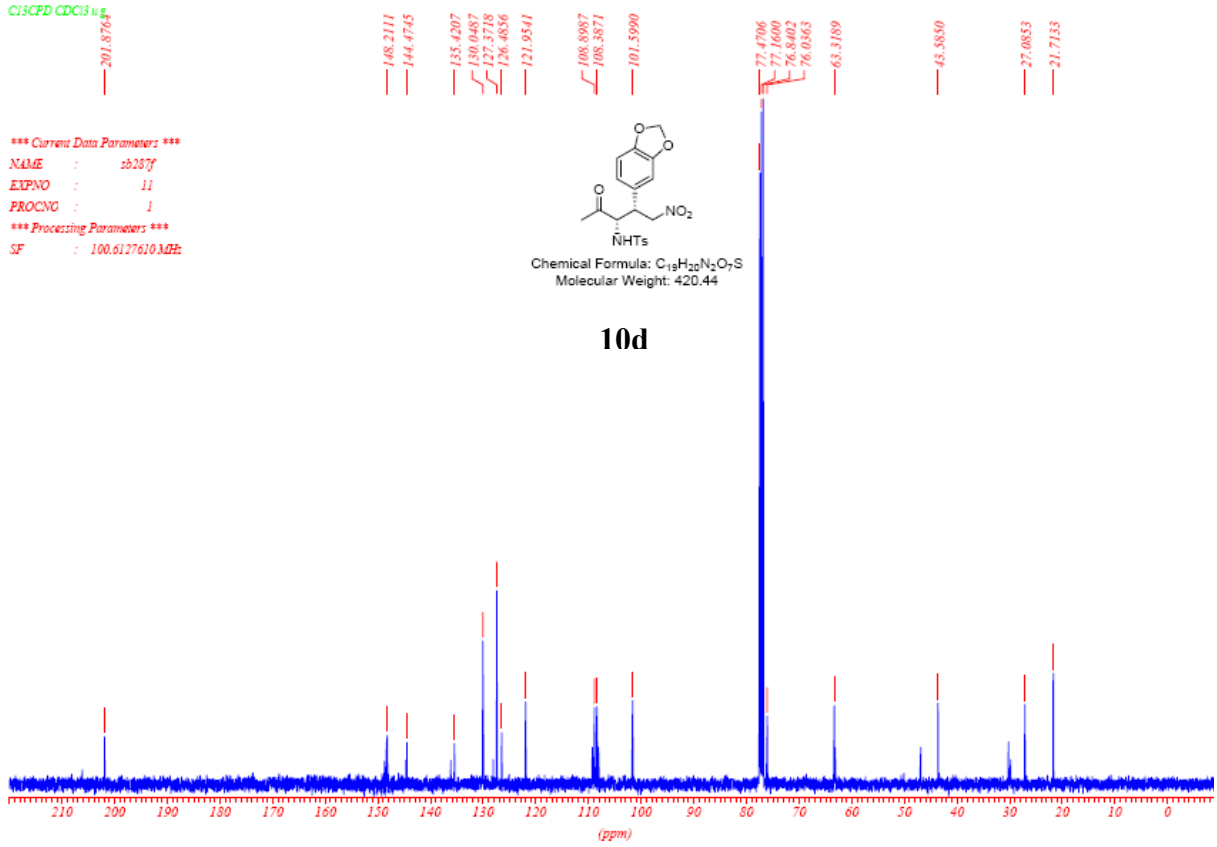

C13CPD CDCl3
202.0317
159.9510
144.4014
135.5029
130.0212
129.4548
127.3352
124.8045
114.6544
77.4798
77.1600
76.8402
76.0089
63.3097
55.3522
43.1282
27.0487
21.6858
*** Current Data Parameters ***
NAME : sb285f
EXPNO : 11
PROCNO : 1
*** Processing Parameters ***
SF : 100.6127630 MHz
OMe
O
NO2
NHTs
Chemical Formula: C19H22N2O6S
Molecular Weight: 406.45
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

10c

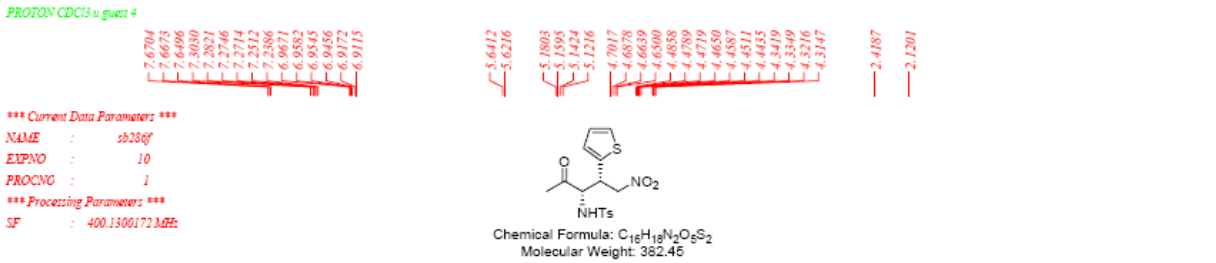

PROTON CDCl3 u guest 5
*** Current Data Parameters ***
NAME : sb287f
EXPNO : 10
PROCNO : 1
*** Processing Parameters ***
SF : 400.1300220 MHz
7.6312
7.6104
7.2815
7.2600
6.6848
6.6772
6.6147
6.6110
6.5522
6.5478
6.5320
5.9417
5.4795
5.4593
5.1115
5.0913
5.0748
5.0540
4.7049
4.6891
4.6708
4.6676
4.2762
4.2686
4.2560
4.2484
4.0047
3.9920
3.9838
3.9794
3.9718
3.5097
3.4971
3.4844
3.4718
3.4592
3.4465
2.4023
2.0702
NO2
NHTs
Chemical Formula: C19H20N2O7S
Molecular Weight: 420.44

10d

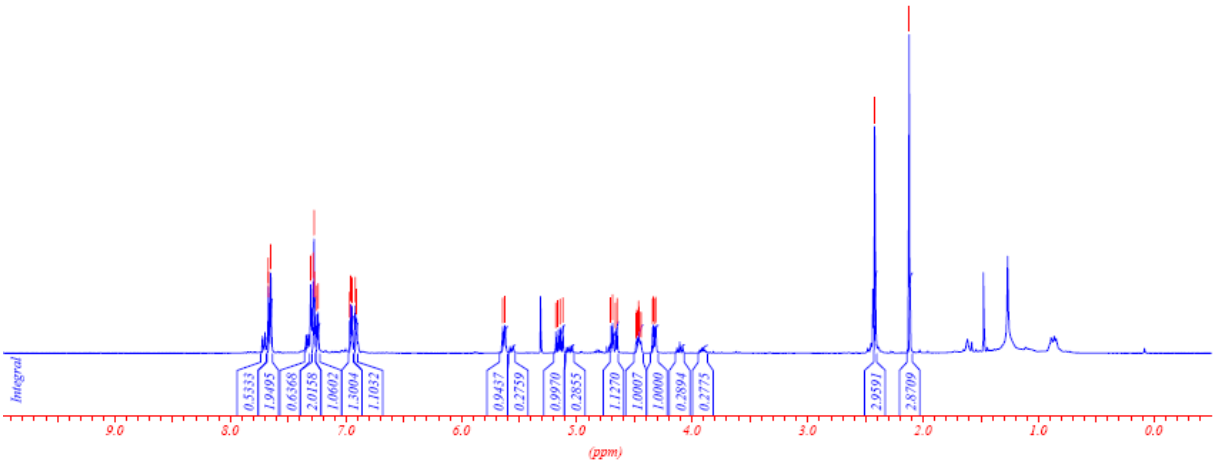

Integral
1.0840
1.9534
2.5898
0.5937
1.1358
1.1342
1.0088
2.0383
0.4992
0.9692
1.4886
1.4388
1.0000
1.3565
0.5146
4.6242
2.9791
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

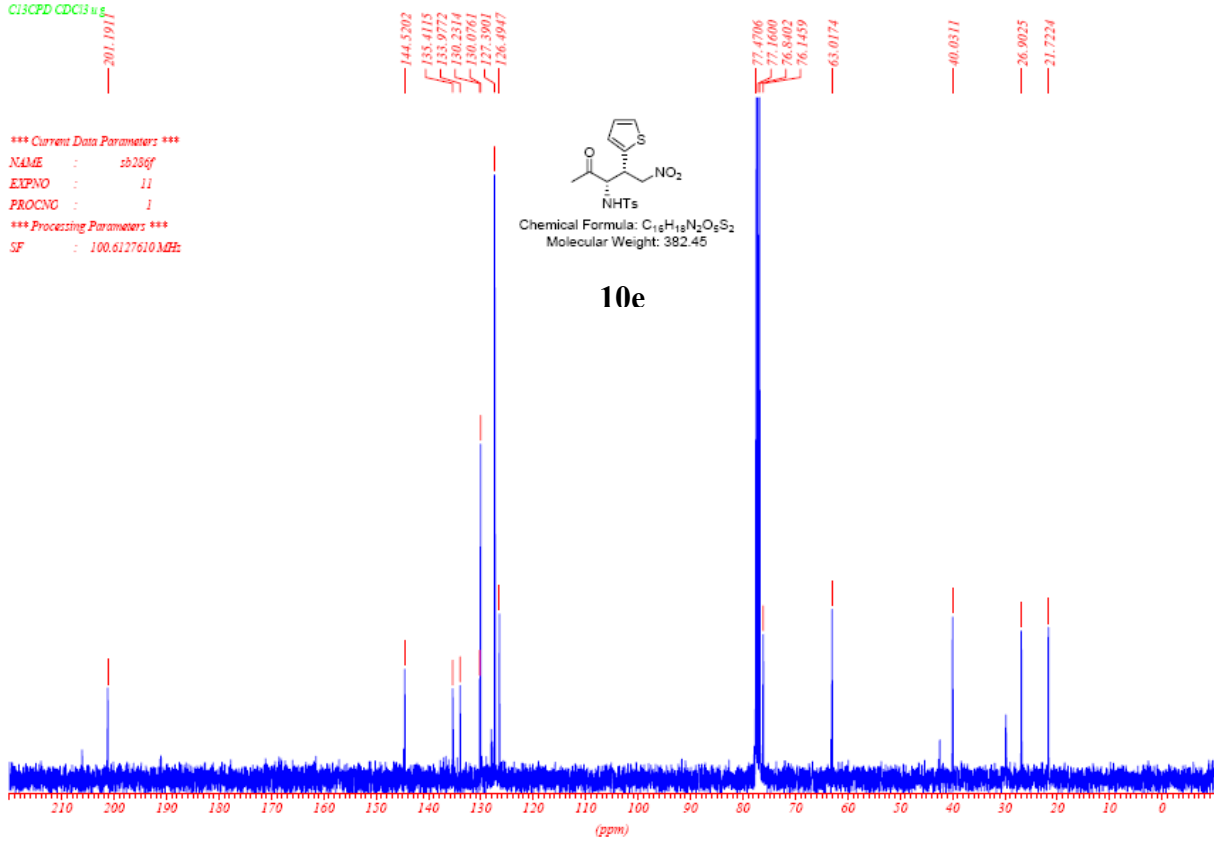

C13CPD CDCl3 u g
201.8764
148.2111
144.4745
135.4207
130.0487
127.3718
126.4856
121.9541
108.8987
108.3871
101.5990
77.4706
77.1600
76.8402
76.0363
63.3189
43.5850
27.0853
21.7133
*** Current Data Parameters ***
NAME : sb287f
EXPNO : 11
PROCNO : 1
*** Processing Parameters ***
SF : 100.6127610 MHz
NO2
NHTs
Chemical Formula: C19H20N2O7S
Molecular Weight: 420.44
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

10d

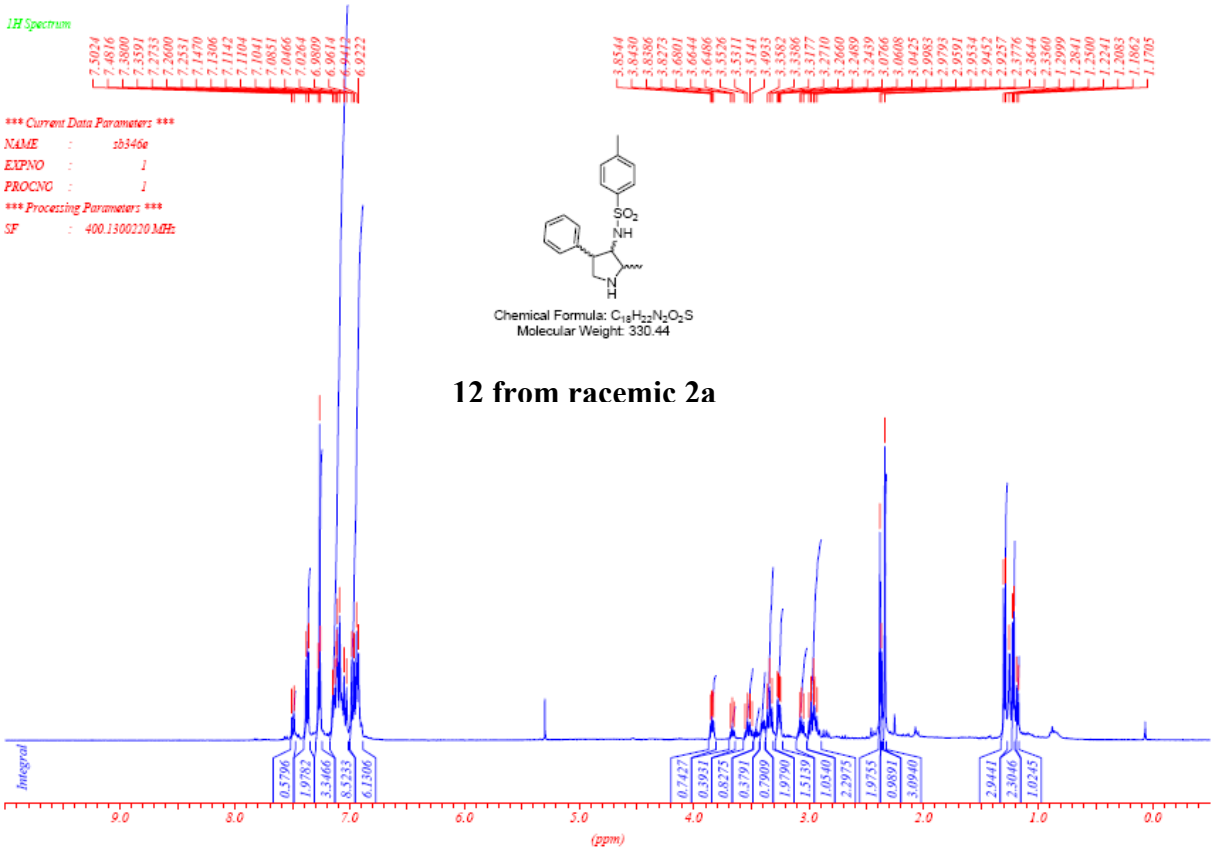

PROTON CDCl3 u guest 4
7.6704
7.6673
7.6496
7.3050
7.2821
7.2746
7.2714
7.2512
7.2386
6.9671
6.9582
6.9345
6.9456
6.9172
6.9115
5.6412
5.6216
5.1803
5.1595
5.1424
5.1216
4.7017
4.6878
4.6639
4.6300
4.4858
4.4789
4.4719
4.4650
4.4587
4.4511
4.4435
4.3419
4.3349
4.3216
4.3147
2.4187
2.1201
*** Current Data Parameters ***
NAME : sb286f
EXPNO : 10
PROCNO : 1
*** Processing Parameters ***
SF : 400.1300172 MHz
S
O
NO2
NHTs
Chemical Formula: C16H18N2O5S2
Molecular Weight: 382.45

10e


Integral
0.5333
1.9495
0.6366
2.0158
1.0602
1.3004
1.1032
0.9437
0.2759
0.9970
0.2855
1.1270
1.0007
1.0000
0.2894
0.2775
2.9591
2.8709
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

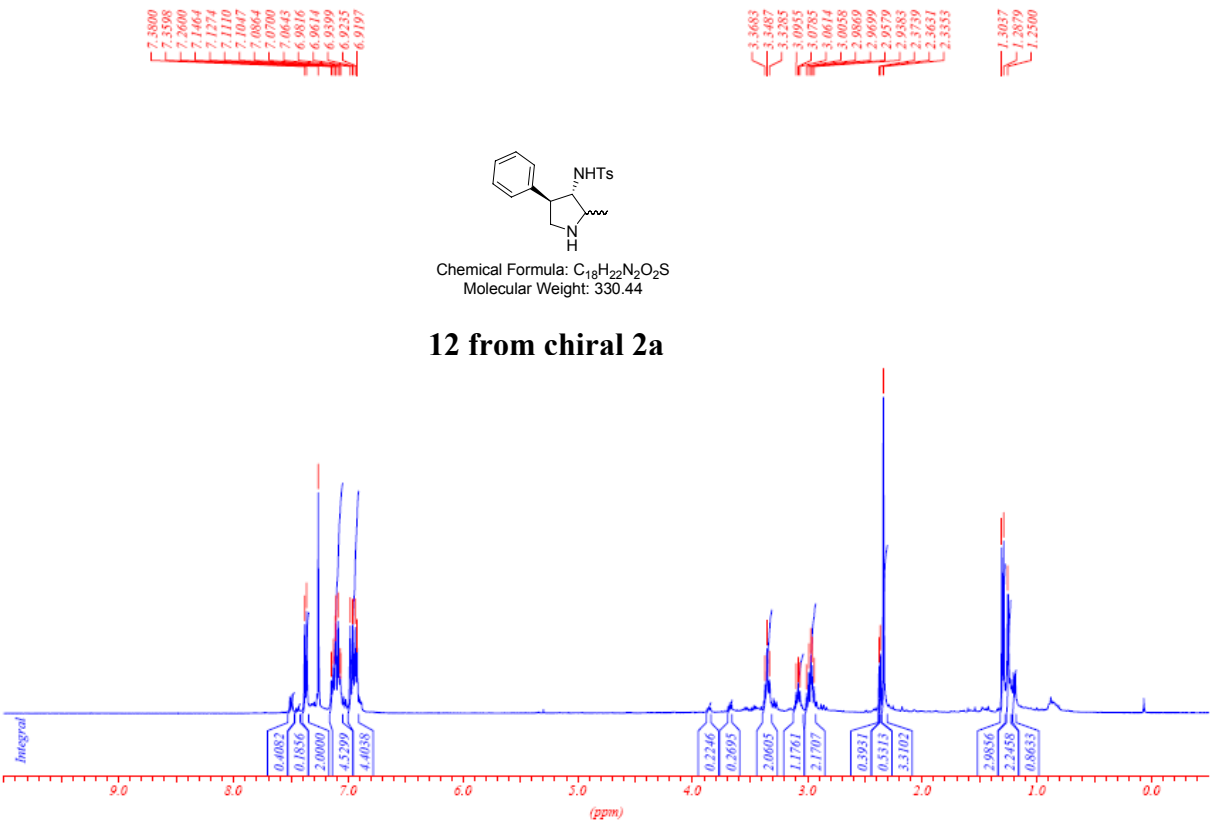

C13CPD CDCl3 u g
201.1911
144.5202
135.4115
133.9772
130.2314
130.0761
127.3901
126.4947
77.4706
77.1600
76.8402
76.1459
63.0174
40.0311
26.9025
21.7224
*** Current Data Parameters ***
NAME : sb286f
EXPNO : 11
PROCNO : 1
*** Processing Parameters ***
SF : 100.6127610 MHz
S
O
NO2
NHTs
Chemical Formula: C16H18N2O5S2
Molecular Weight: 382.45
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

10e



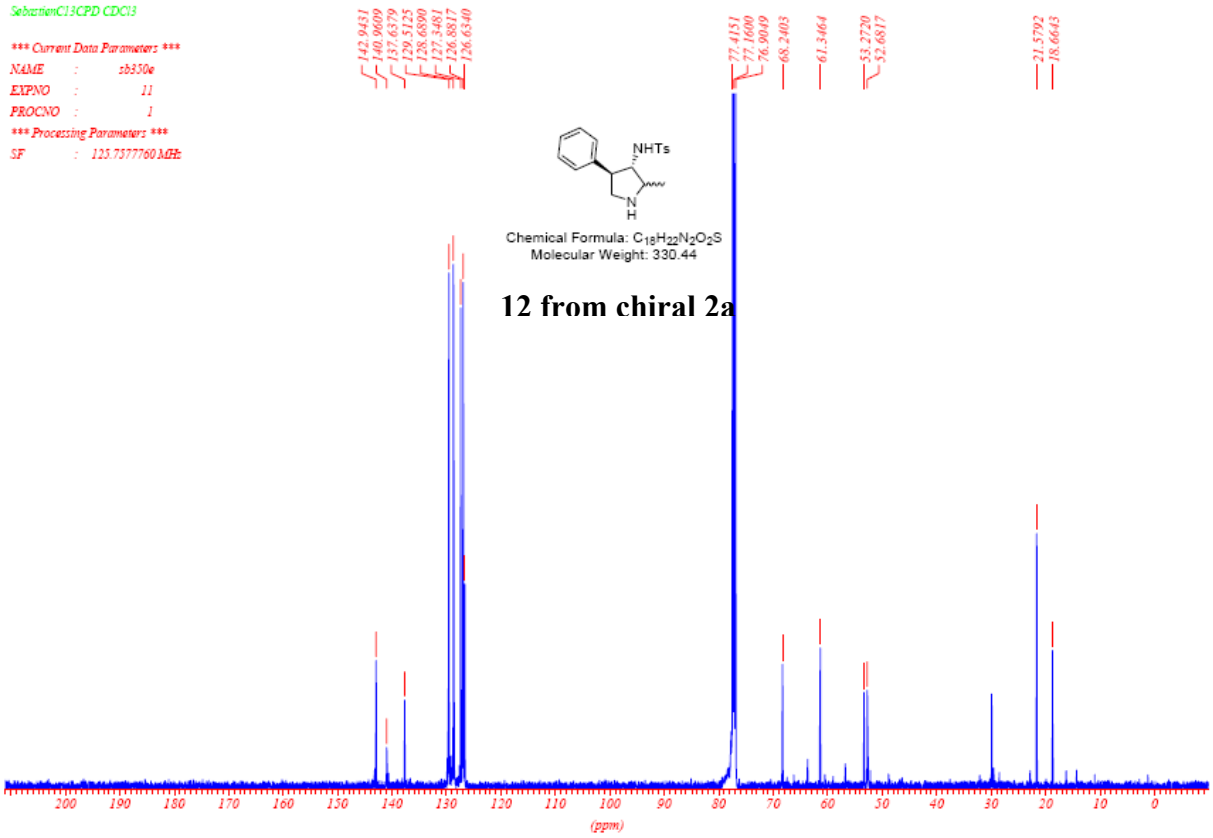
Chemical Formula: $C_{18}H_{22}N_2O_2S$
Molecular Weight: 330.44

12 from racemic 2a

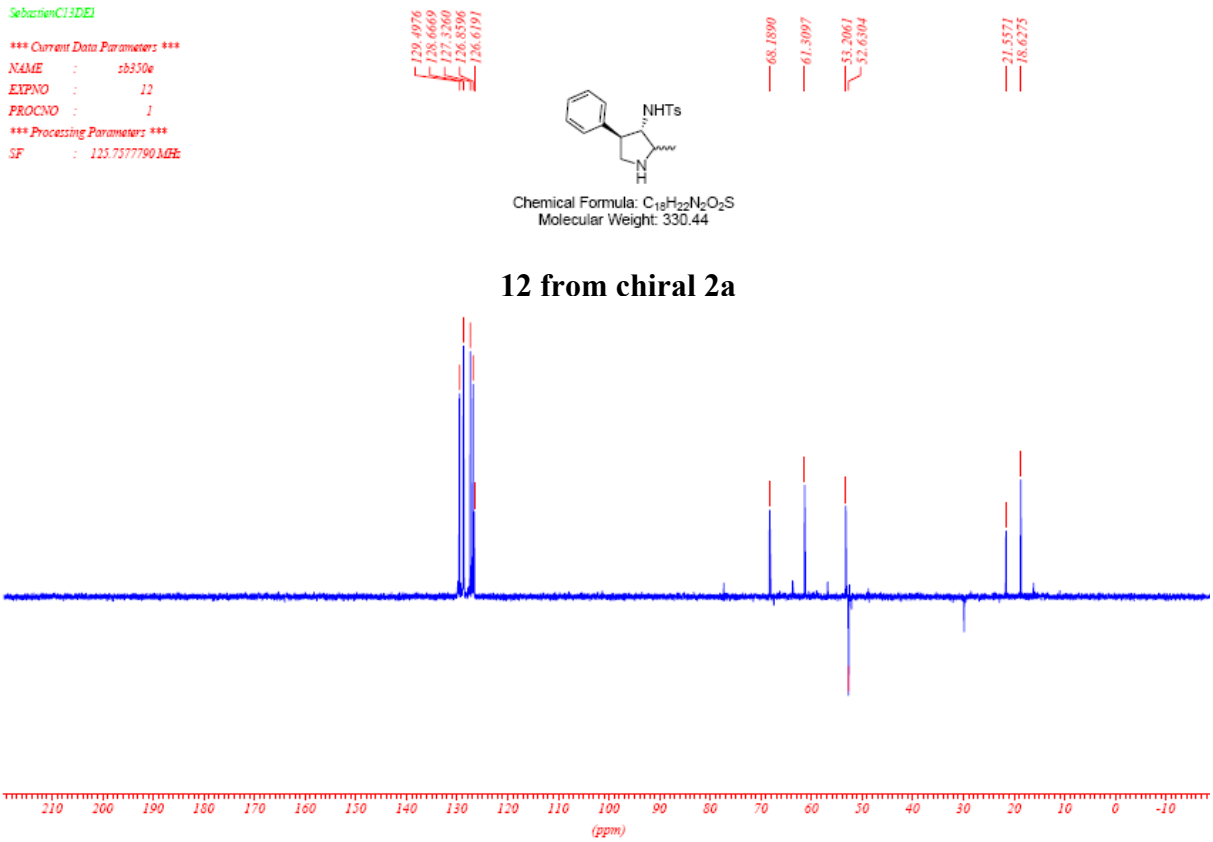


Chemical Formula: $C_{18}H_{22}N_2O_2S$
Molecular Weight: 330.44

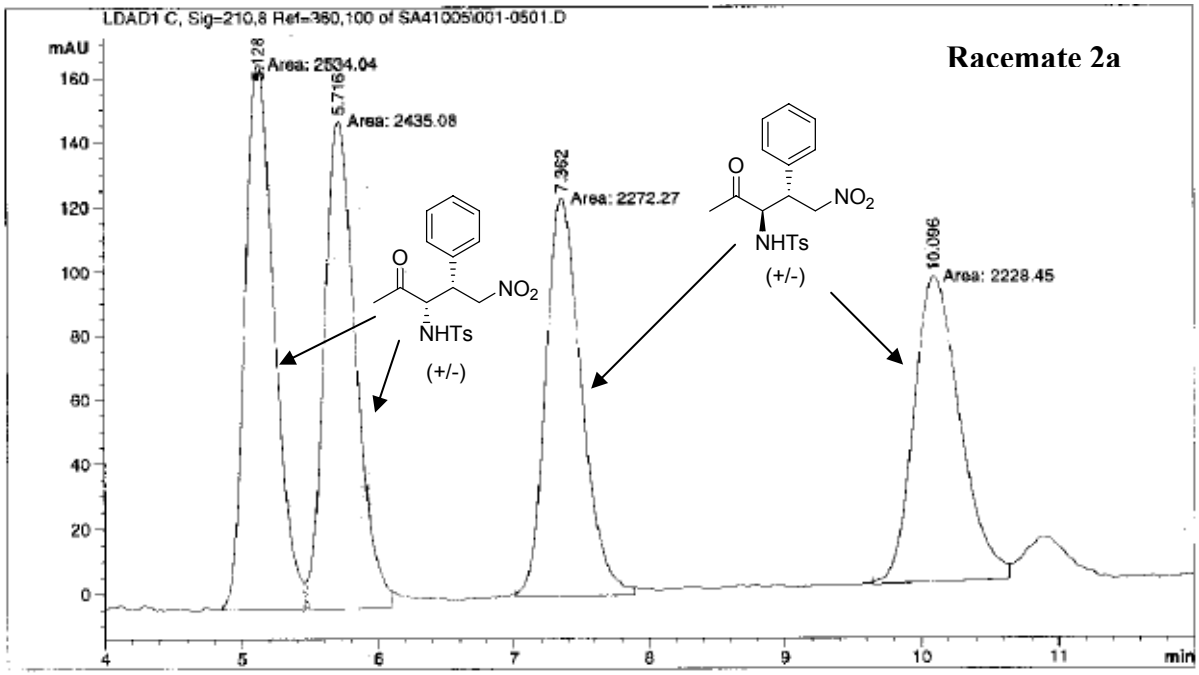
12 from chiral 2a



12 from chiral 2a

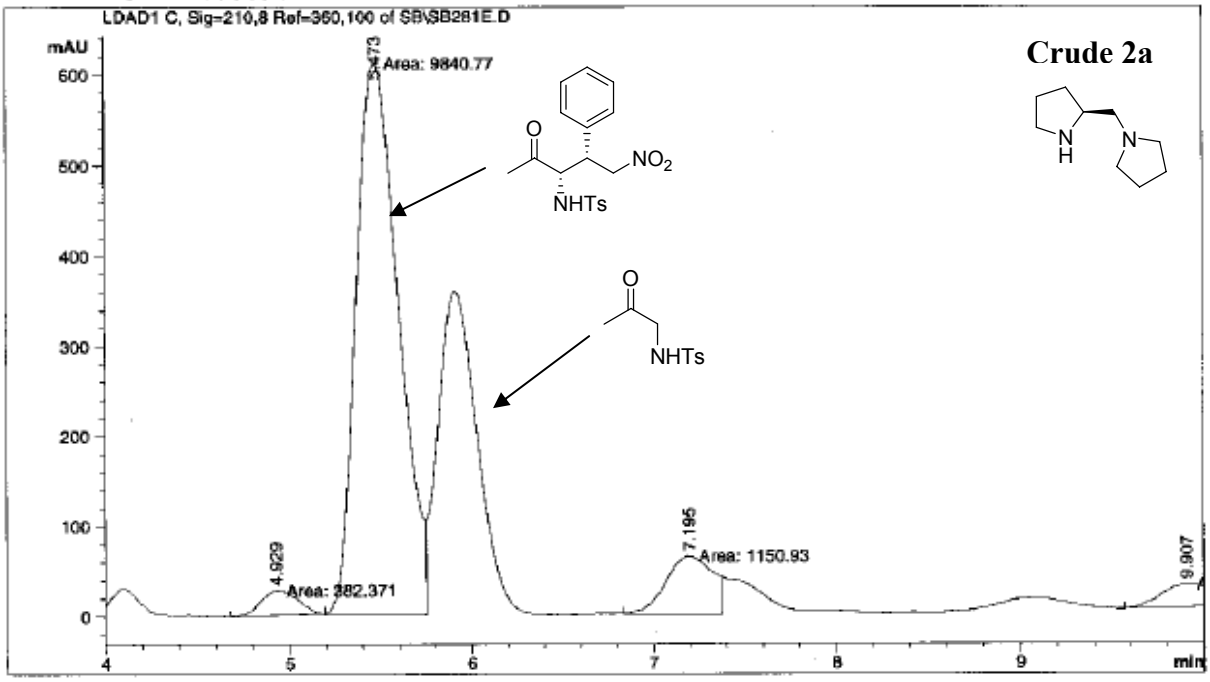


12 from chiral 2a



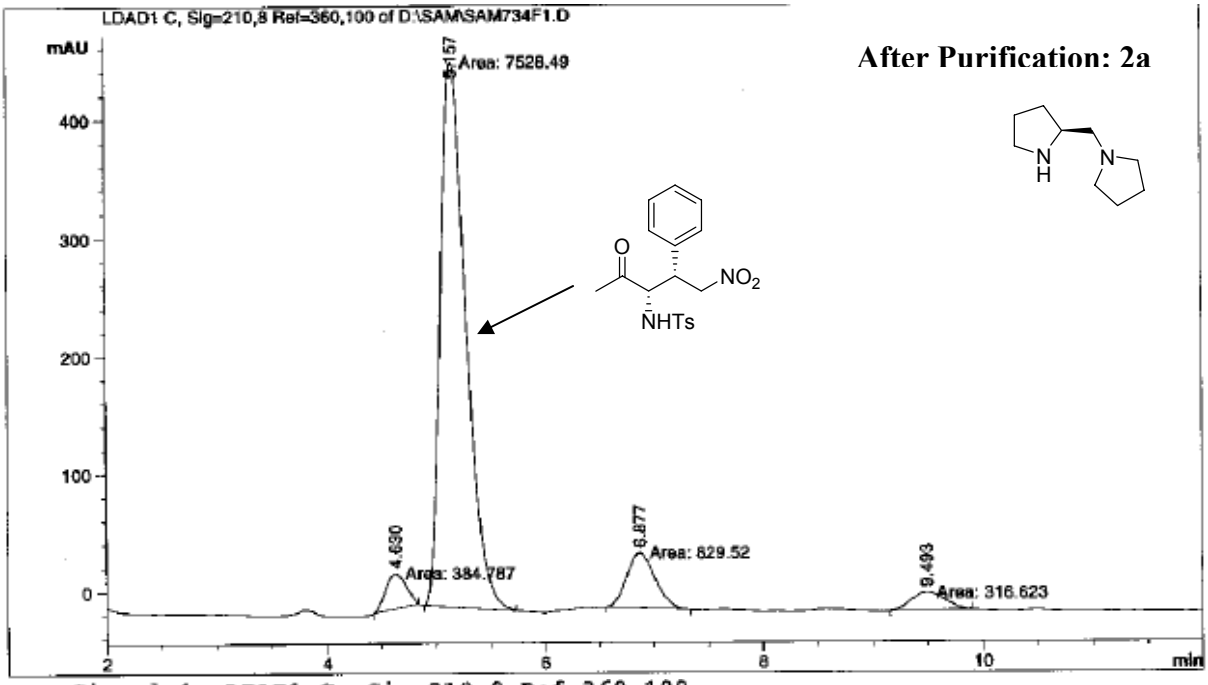
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.128	MM	0.250	2534.04224	169.25511	26.7591
2	5.716	MM	0.268	2435.07788	151.37775	25.7140
3	7.362	MM	0.306	2272.27075	123.60941	23.9948
4	10.096	MM	0.390	2228.44897	95.11417	23.5321



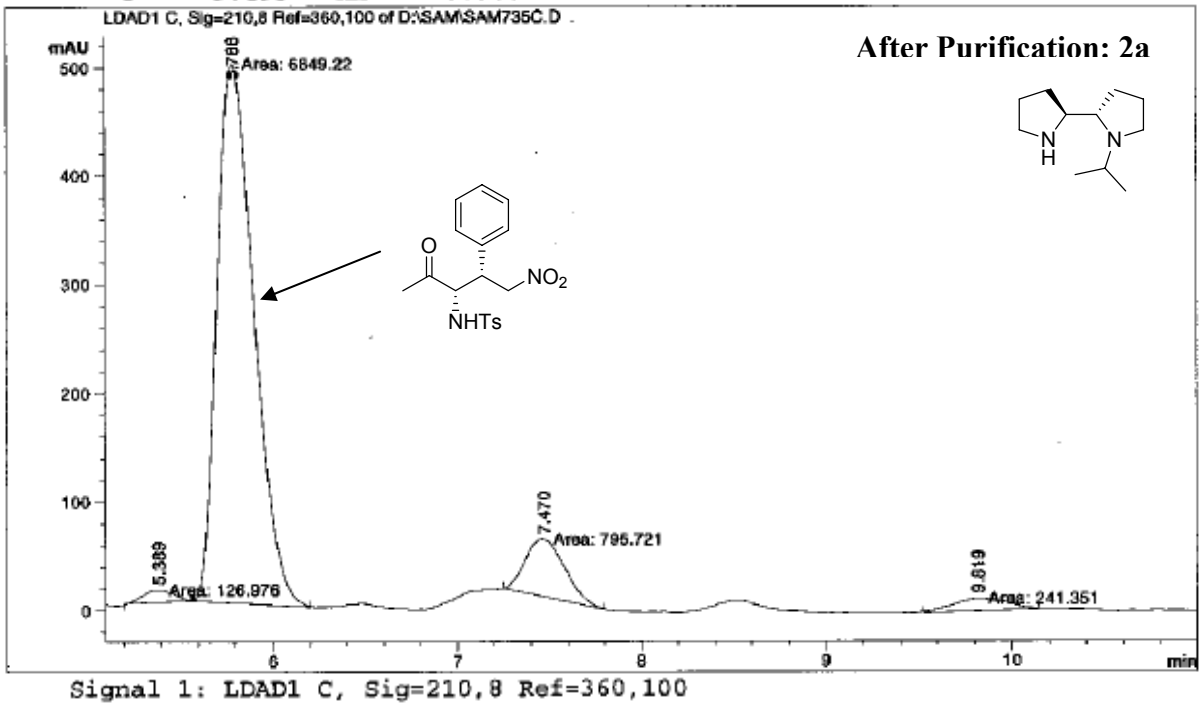
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	4.929	MM	0.233	382.37085	27.32365	3.2053
2	5.473	MM	0.269	9840.77344	610.59174	82.4922
3	7.195	MM	0.300	1150.92578	63.90030	9.6479
4	9.907	MM	0.371	555.26501	24.93450	4.6546



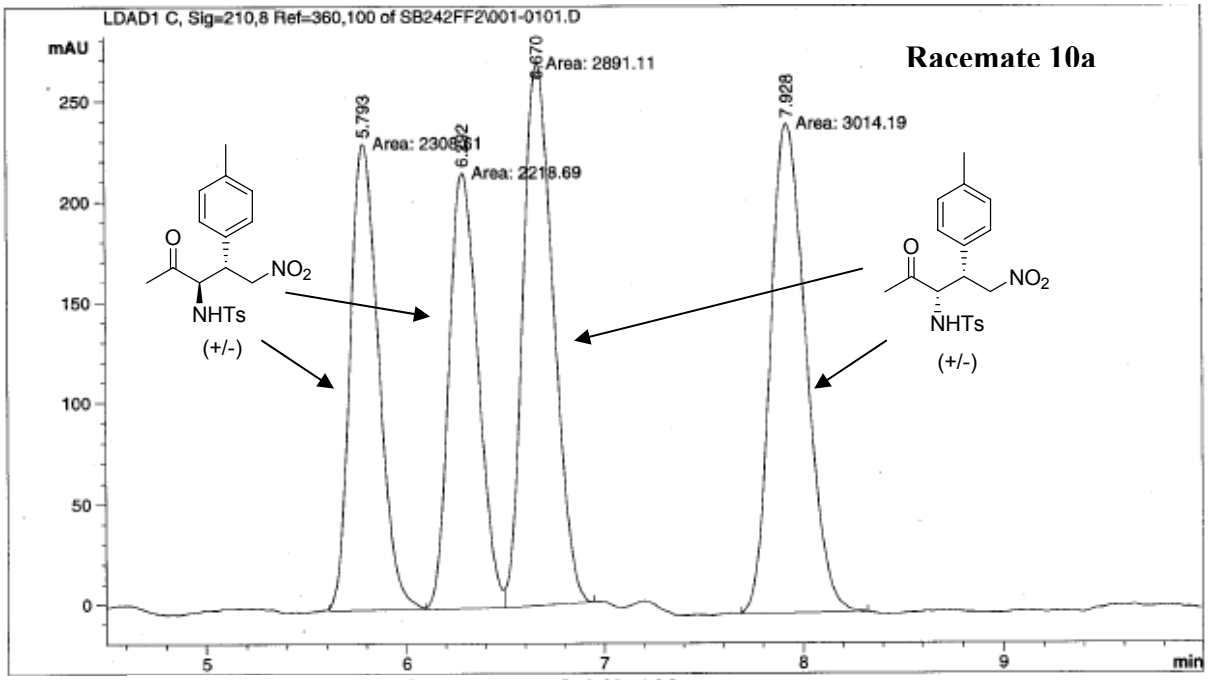
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	4.630	MM	0.217	384.78690	29.59315	4.2474
2	5.157	MM	0.272	7528.49023	460.83853	83.1012
3	6.877	MM	0.298	829.52045	46.36420	9.1564
4	9.493	MM	0.360	316.62259	14.66685	3.4950



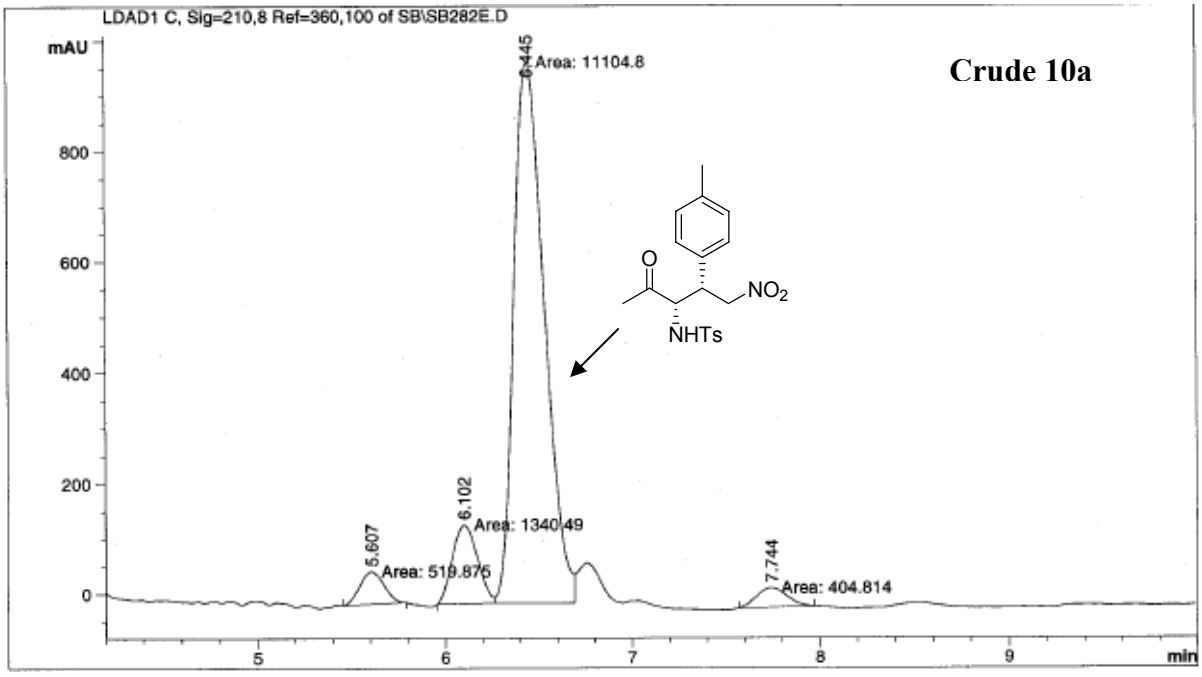
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.389	MM	0.186	126.97584	11.37109	1.5846
2	5.788	MM	0.230	6849.21533	497.01230	85.4735
3	7.470	MM	0.250	795.72144	53.15287	9.9301
4	9.819	MM	0.345	241.35135	11.64454	3.0119



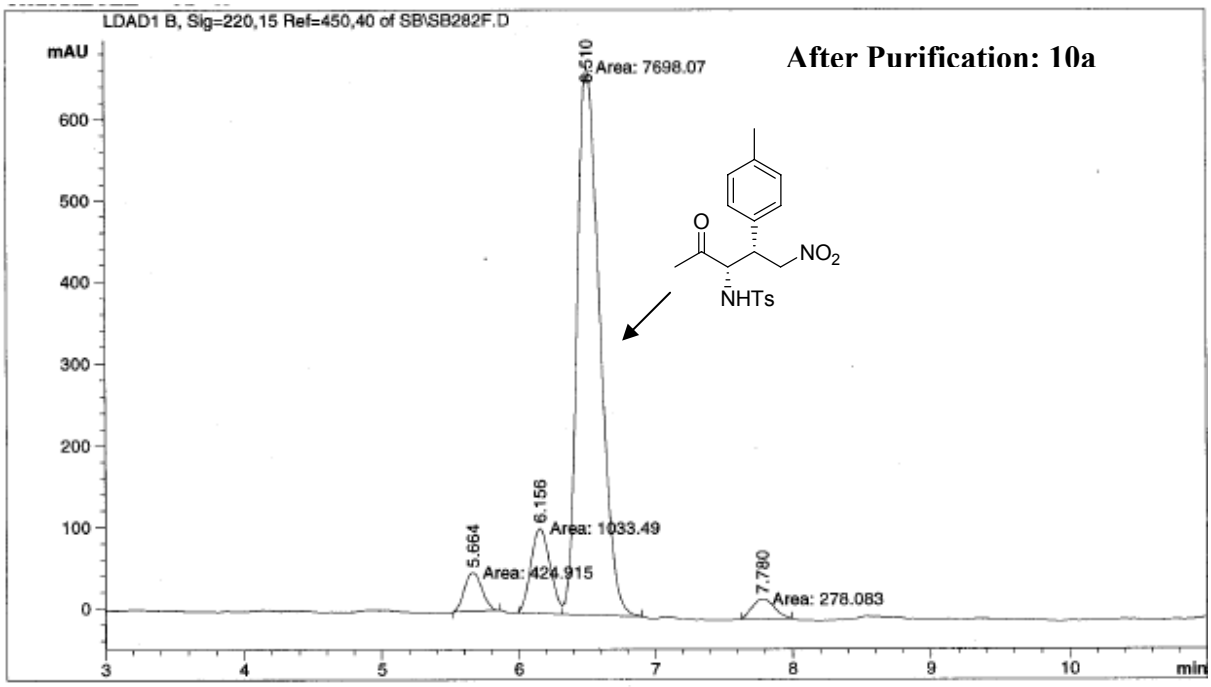
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.793	MM	0.166	2308.61206	231.89815	22.1288
2	6.292	MM	0.171	2218.68872	216.70999	21.2669
3	6.670	MM	0.179	2891.11401	269.02545	27.7123
4	7.928	MM	0.206	3014.19434	243.36299	28.8920



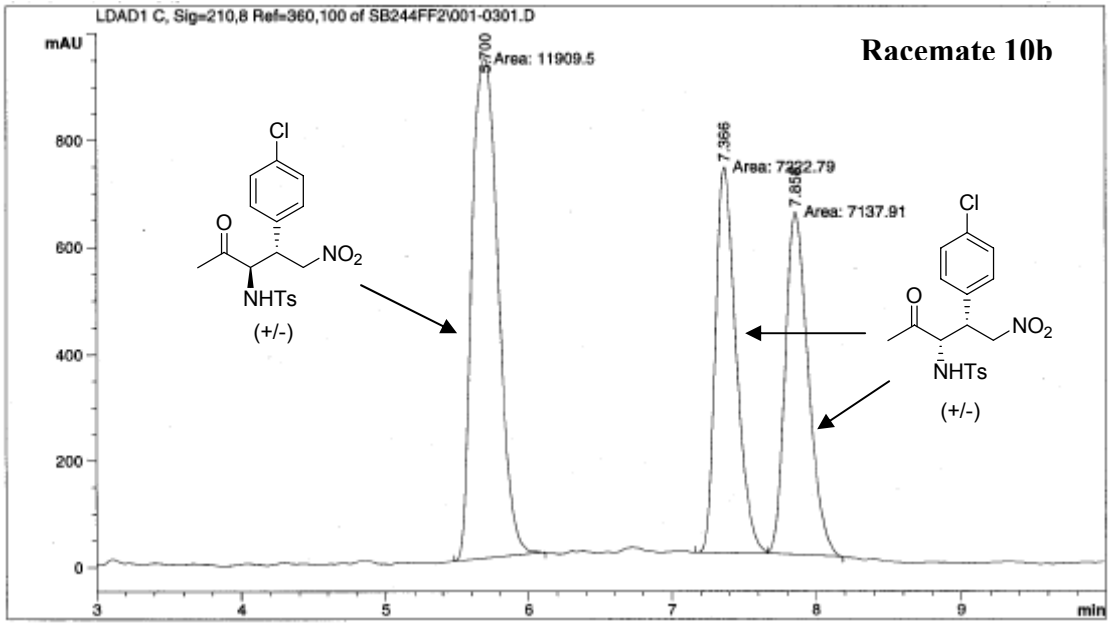
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.607	MM	0.148	519.87463	58.50688	3.8884
2	6.102	MM	0.156	1340.49121	142.99600	10.0261
3	6.445	MM	0.189	11104.84668	977.25598	83.0578
4	7.744	MM	0.190	404.81436	35.53560	3.0278



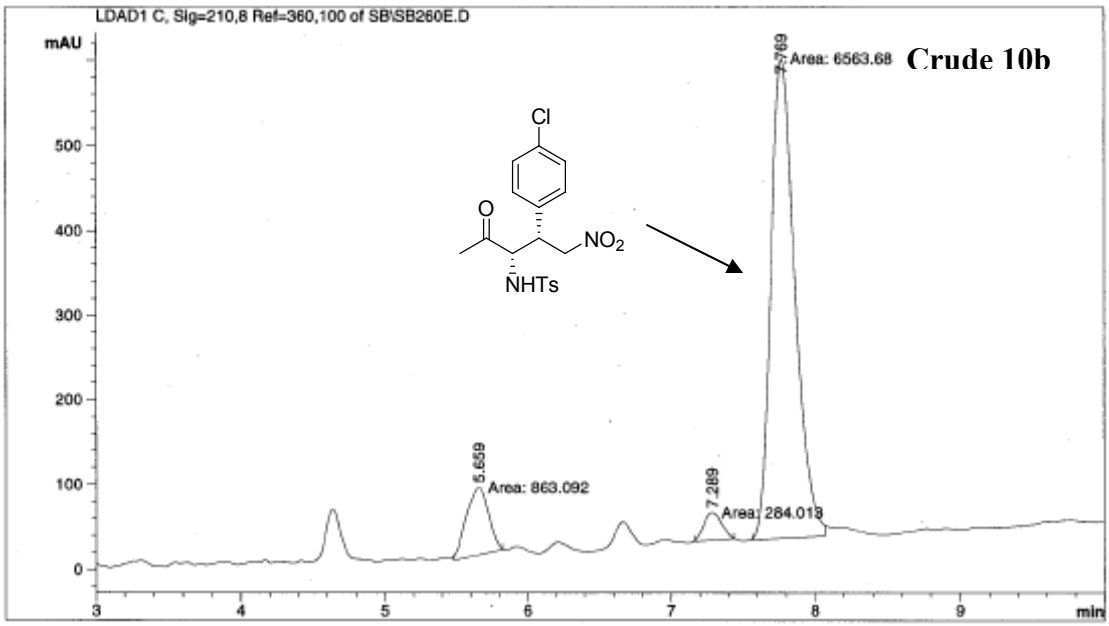
Signal 1: LDAD1 B, Sig=220,15 Ref=450,40

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.664	MM	0.149	424.91486	47.56963	4.5038
2	6.156	MM	0.164	1033.48560	104.78843	10.9543
3	6.510	MM	0.191	7698.06982	671.62079	81.5944
4	7.780	MM	0.189	278.08273	24.50866	2.9475



Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.700	MM	0.212	11909.52148	936.33539	45.3347
2	7.366	MM	0.166	7222.79102	723.69702	27.4942
3	7.858	MM	0.185	7137.90771	642.76190	27.1711

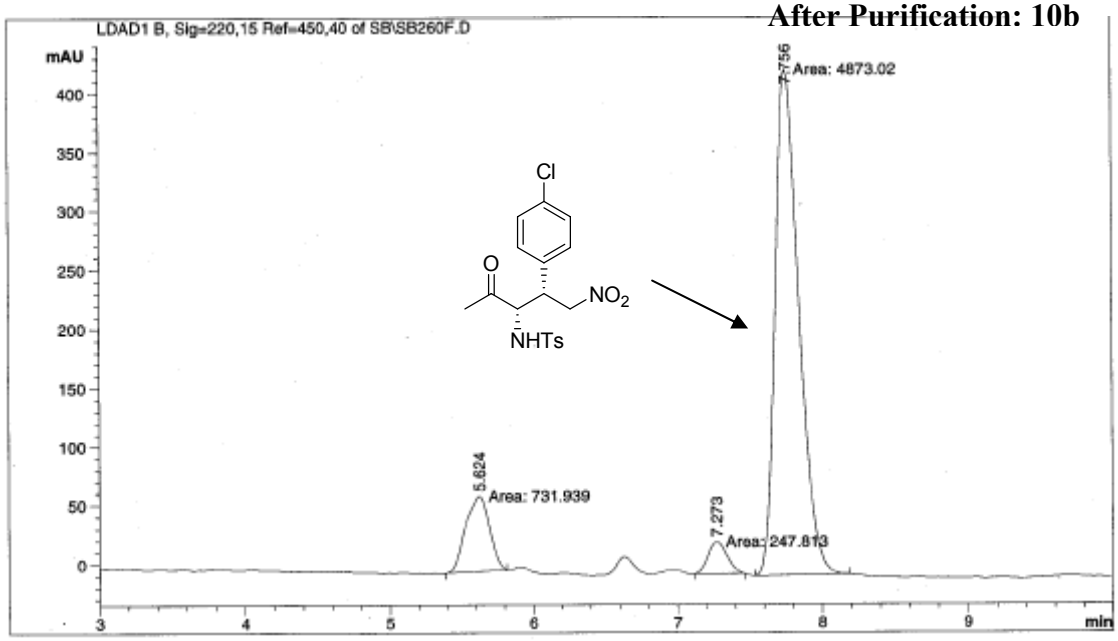


Crude 10b

Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

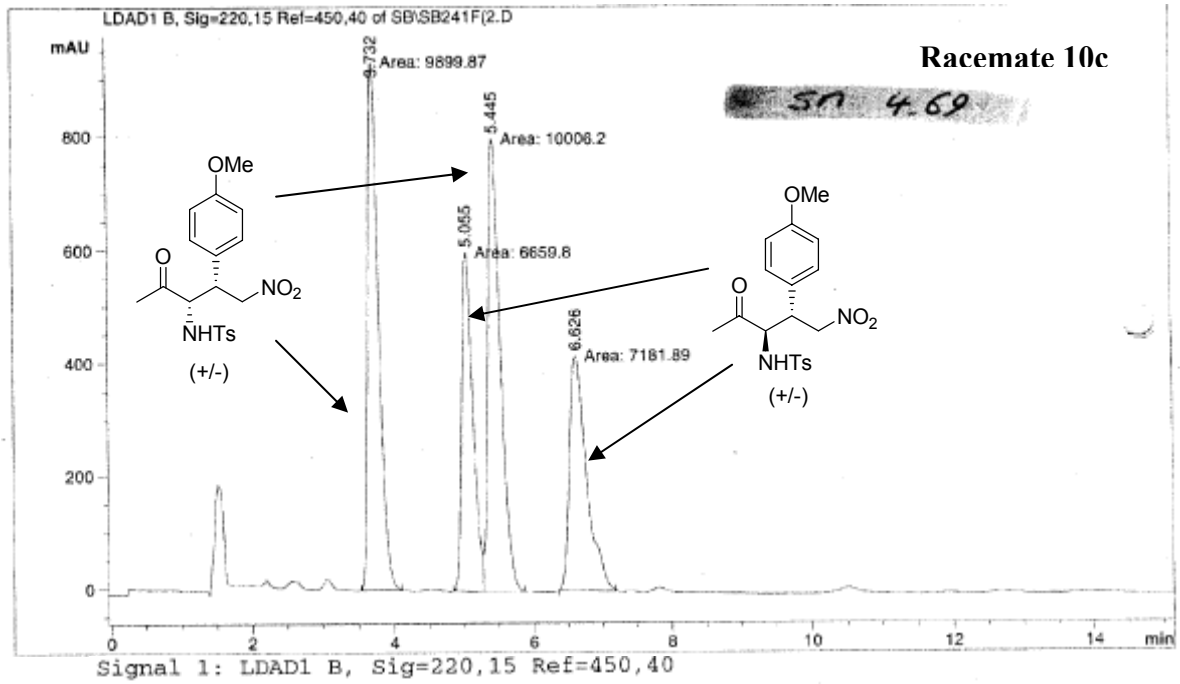
Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.659	MM	0.181	863.09235	79.31374	11.1933
2	7.289	MM	0.146	284.01294	32.47560	3.6833
3	7.769	MM	0.193	6563.68457	565.88776	85.1234

After Purification: 10b



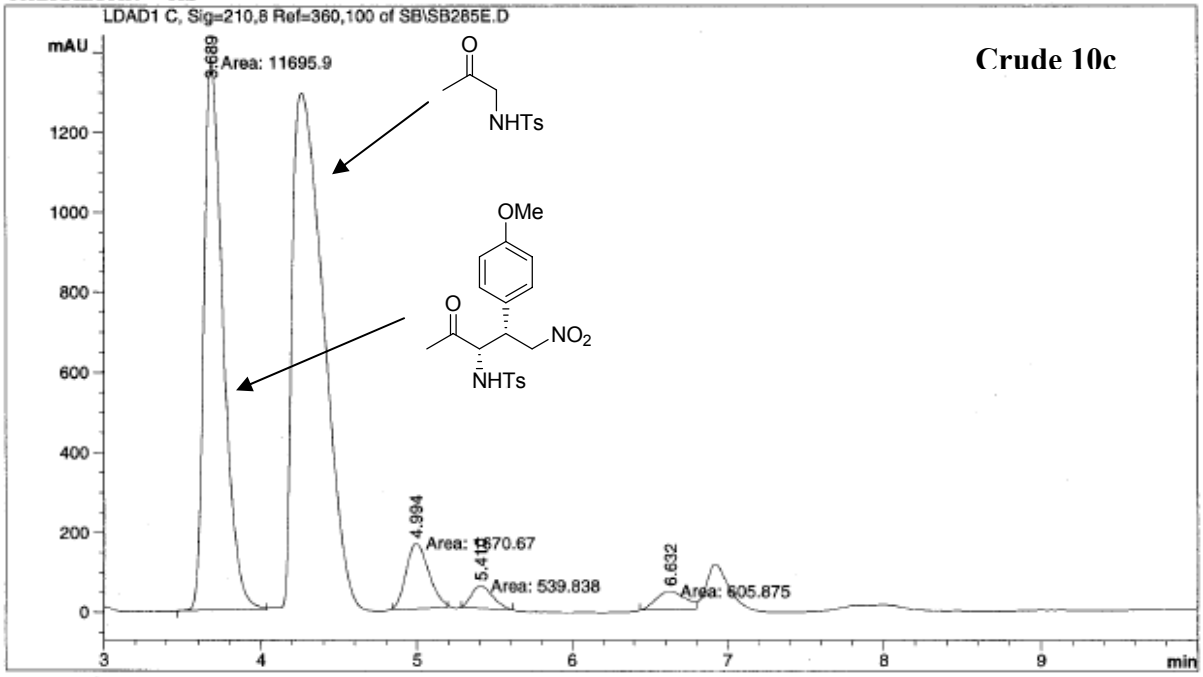
Signal 1: LDAD1 B, Sig=220,15 Ref=450,40

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.624	MM	0.193	731.93878	63.25952	12.5058
2	7.273	MM	0.153	247.81277	27.01921	4.2341
3	7.756	MM	0.189	4873.02295	429.58081	83.2601



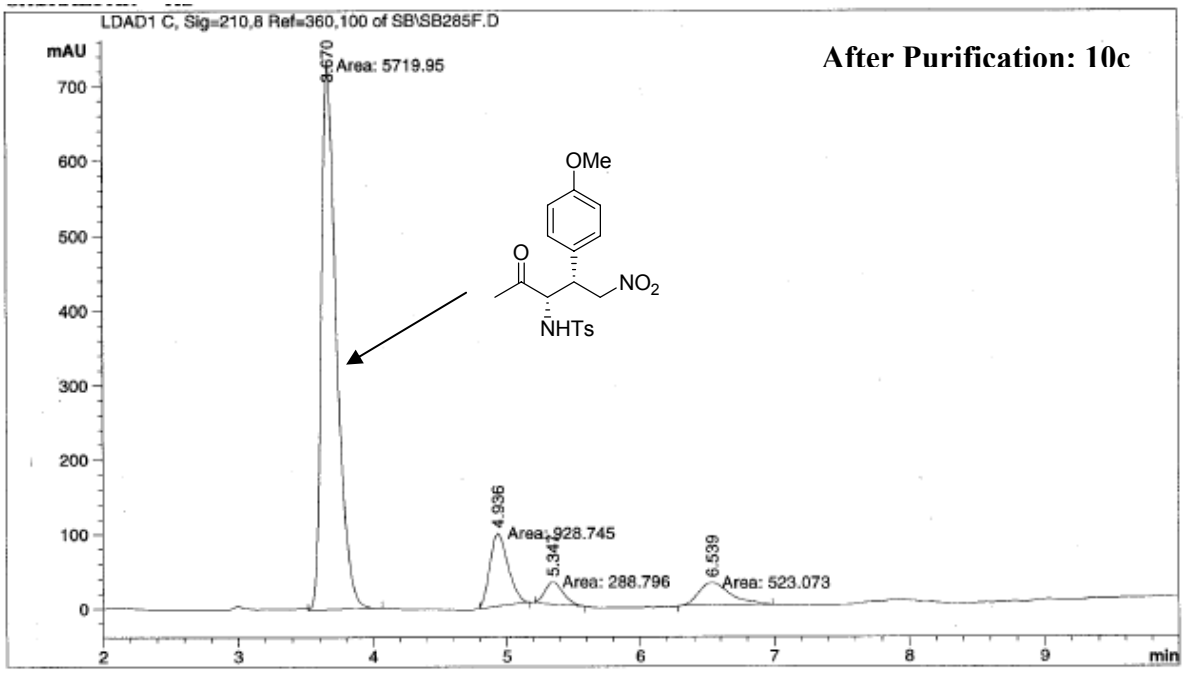
Signal 1: LDAD1 B, Sig=220,15 Ref=450,40

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	3.732	MM	0.177	9899.86719	933.40405	29.3349
2	5.055	MM	0.185	6659.79834	599.60590	19.7340
3	5.445	MM	0.208	10006.24219	800.41681	29.6501
4	6.626	MM	0.288	7181.89111	415.73453	21.2811



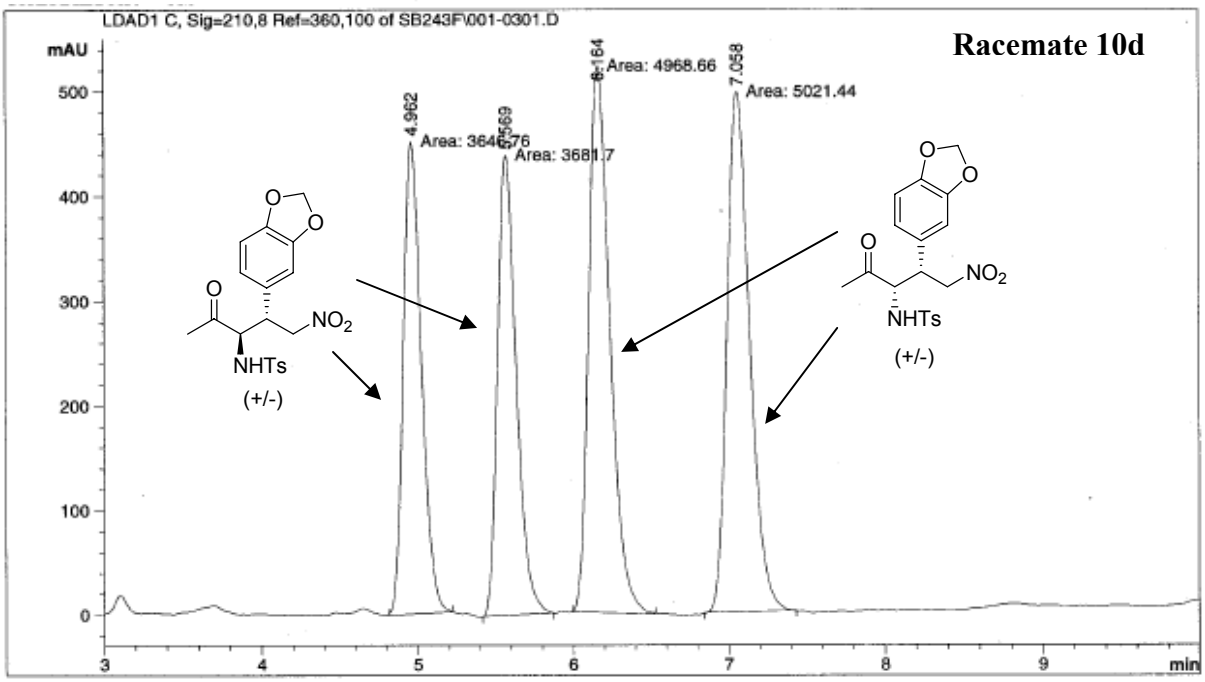
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	3.689	MM	0.142	11695.92578	1369.94421	80.5931
2	4.994	MM	0.169	1670.66931	165.06818	11.5121
3	5.410	MM	0.160	539.83789	56.11963	3.7199
4	6.632	MM	0.223	605.87549	45.37632	4.1749



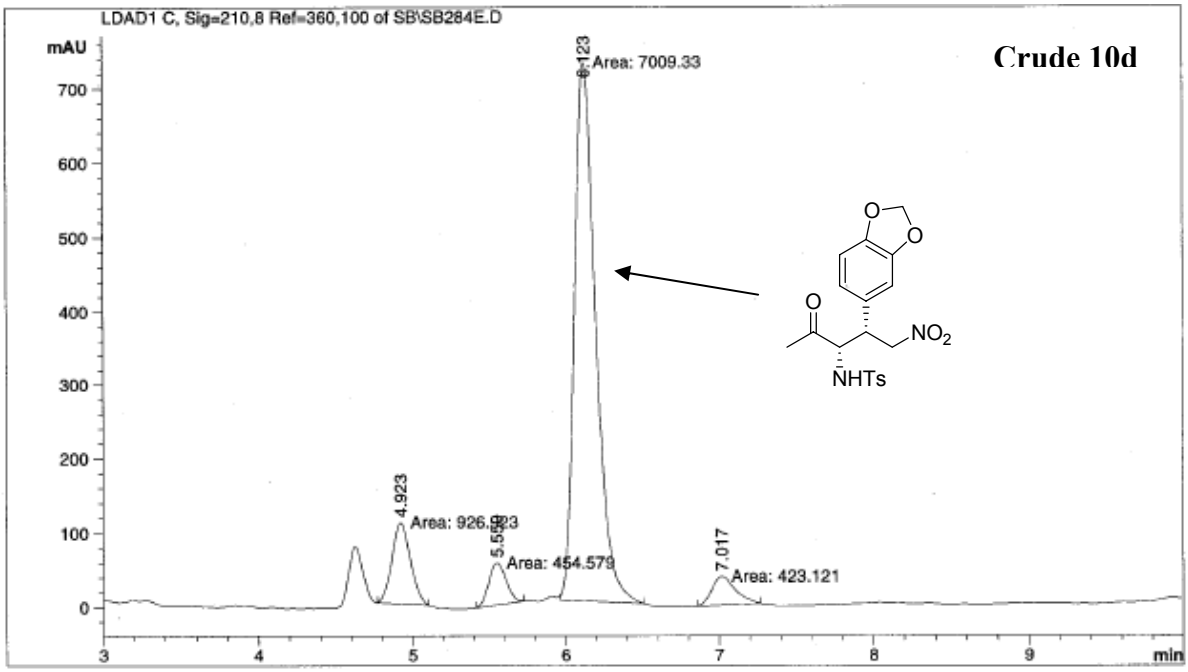
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	3.670	MM	0.131	5719.95410	729.31555	76.6691
2	4.936	MM	0.158	928.74506	98.00354	12.4487
3	5.347	MM	0.159	288.79590	30.26731	3.8710
4	6.539	MM	0.280	523.07343	31.10322	7.0112



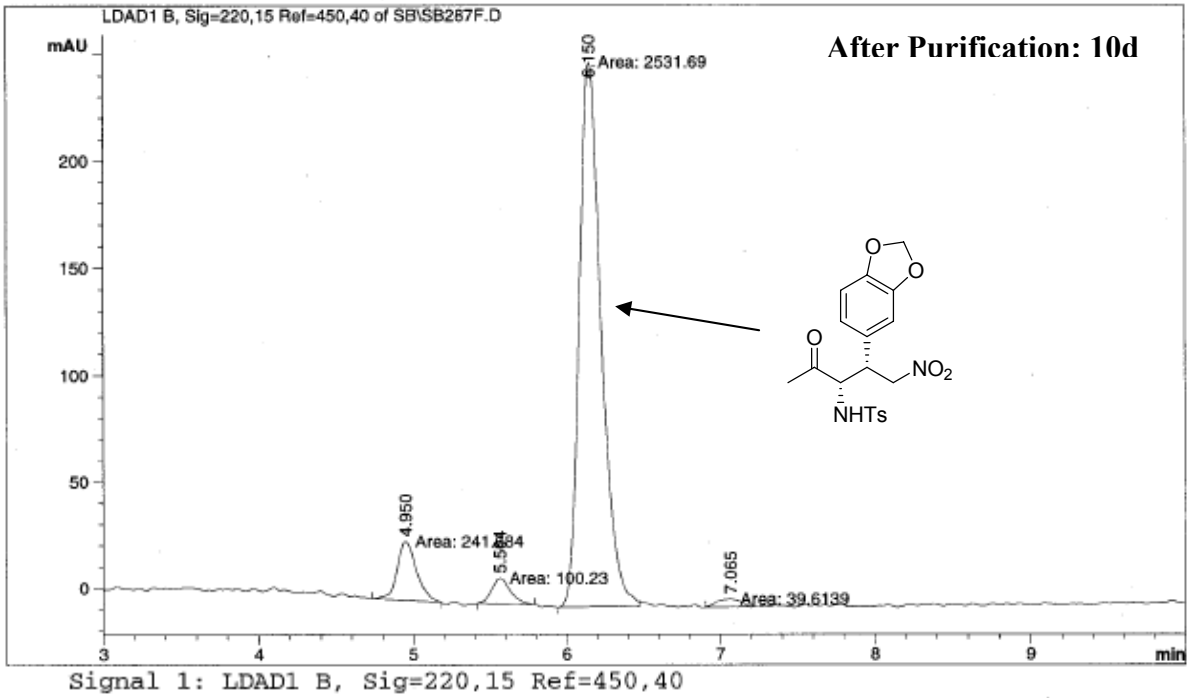
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	4.962	MM	0.135	3646.76465	451.88007	21.0570
2	5.569	MM	0.140	3681.69946	439.81070	21.2587
3	6.164	MM	0.158	4968.65723	522.74951	28.6898
4	7.058	MM	0.168	5021.44238	497.32181	28.9946



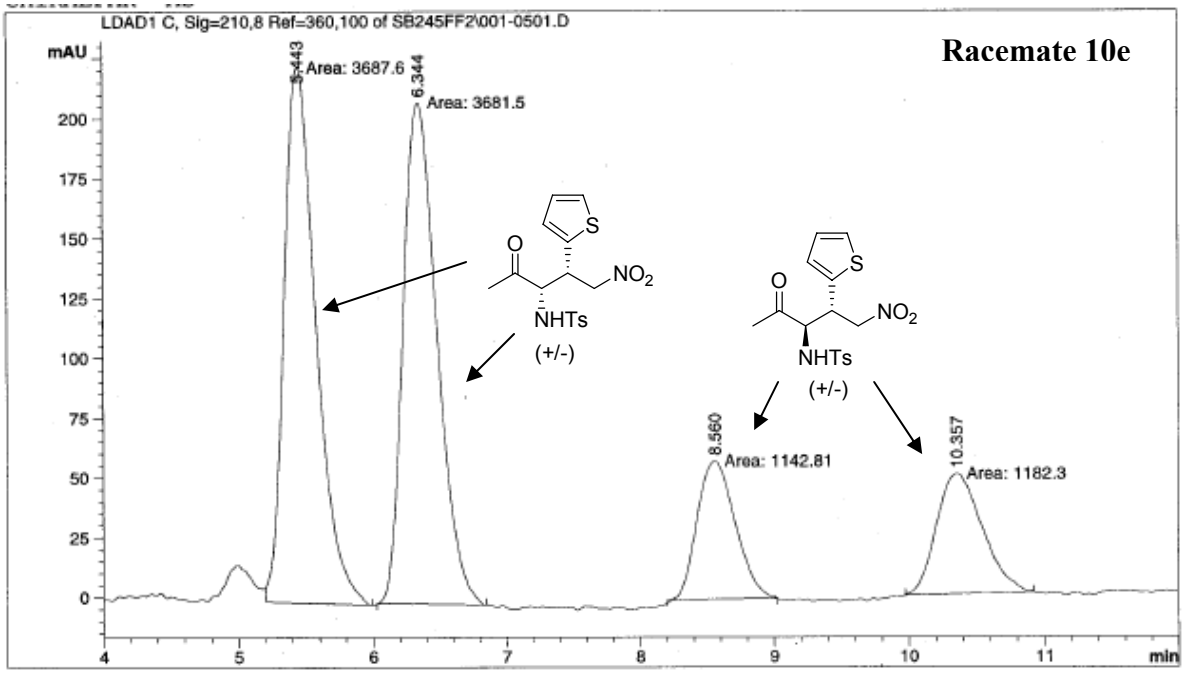
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	4.923	MM	0.140	926.92279	110.43447	10.5165
2	5.550	MM	0.132	454.57877	57.48417	5.1575
3	6.123	MM	0.161	7009.33398	727.43286	79.5254
4	7.017	MM	0.178	423.12091	39.69188	4.8006



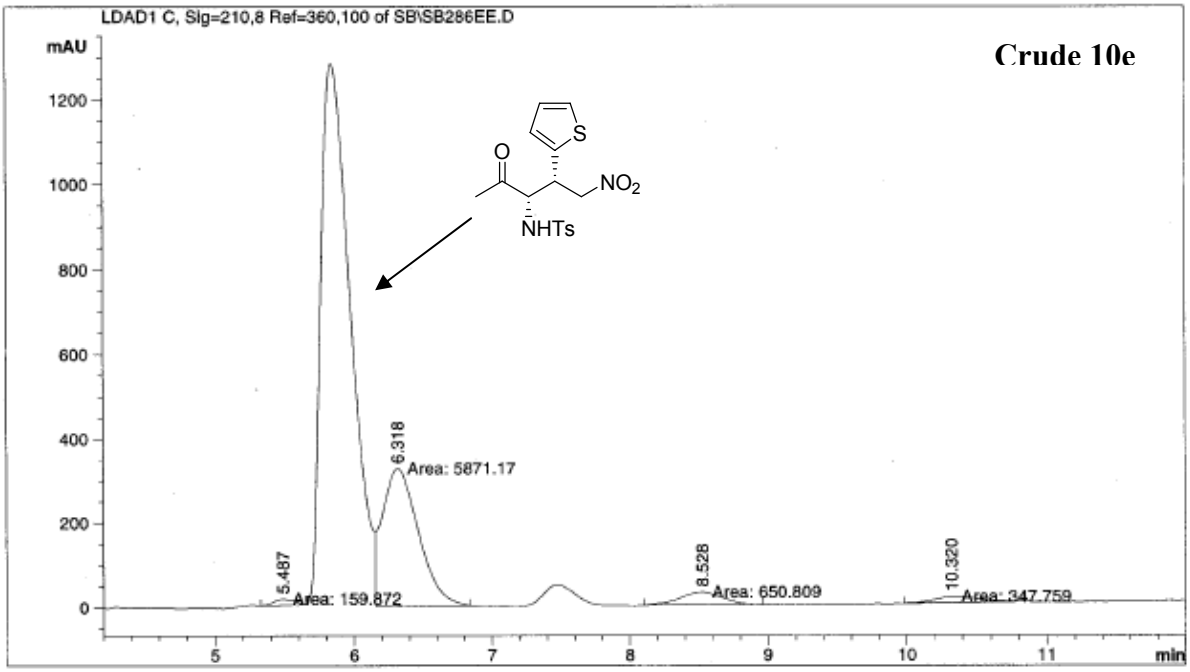
Signal 1: LDAD1 B, Sig=220,15 Ref=450,40

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	4.950	MM	0.146	241.68372	27.66122	8.2961
2	5.564	MM	0.141	100.23045	11.82845	3.4405
3	6.150	MM	0.165	2531.68799	254.98694	86.9035
4	7.065	MM	0.180	39.61385	3.65910	1.3598



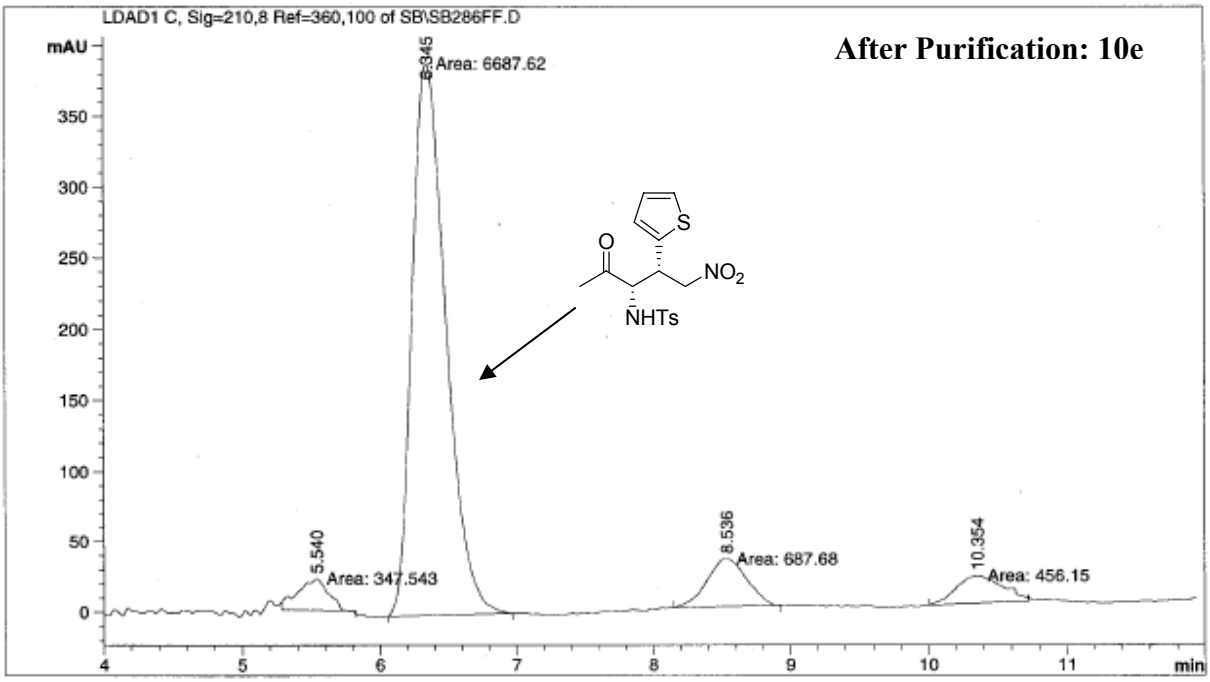
Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.443	MM	0.275	3687.59839	223.59784	38.0392
2	6.344	MM	0.293	3681.49585	209.53828	37.9763
3	8.560	MM	0.329	1142.80859	57.84394	11.7886
4	10.357	MM	0.392	1182.30273	50.30948	12.1960



Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.487	MM	0.178	159.87218	14.97244	2.2743
2	6.318	MM	0.301	5871.17334	324.89310	83.5206
3	8.528	MM	0.361	650.80859	30.03817	9.2581
4	10.320	MM	0.381	347.75888	15.21437	4.9471



Signal 1: LDAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.540	MM	0.257	347.54312	22.52427	4.2492
2	6.345	MM	0.286	6687.62207	389.43121	81.7658
3	8.536	MM	0.339	687.68036	33.82632	8.4079
4	10.354	MM	0.388	456.14972	19.58657	5.5771