Supplementary Material

A Concise Synthetic Method for Constructing 3-Substituted Piperazine-2-Acetic Acid Esters from 1,2-Diamines

Srinivas Chamakuri [1,*], Sunny Ann Tang [1], Kevin A. Tran [1], Shiva Krishna Reddy Guduru [1], Peter K. Bolin [1], Kevin R. MacKenzie [1,2] and Damian W. Young [1,2,3,*]

[1] Center for Drug Discovery, Department of Pathology and Immunology, Baylor College of Medicine, One Baylor Plaza, Houston, TX 77030, USA; sunny.tang@bcm.edu (S.A.T.); kevin.tran@bcm.edu (K.A.T.); shiva.guduru@bcm.edu (S.K.R.G.); peter.bolin@bcm.edu (P.K.B.); kevin.mackenzie@bcm.edu (K.R.M.)
[2] Department of Pharmacology and Chemical Biology, Baylor College of Medicine, One Baylor Plaza,
Houston, TX 77030, USA
[3] Verna and Marrs McLean Department of Biochemistry and Molecular Biology, Baylor College of Medicine, Houston, TX 77030, USA
* Correspondence: srinivas.chamakuri@bcm.edu (S.C.); damian.young@bcm.edu (D.W.Y.)

Supporting Information
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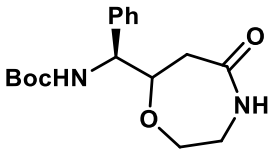
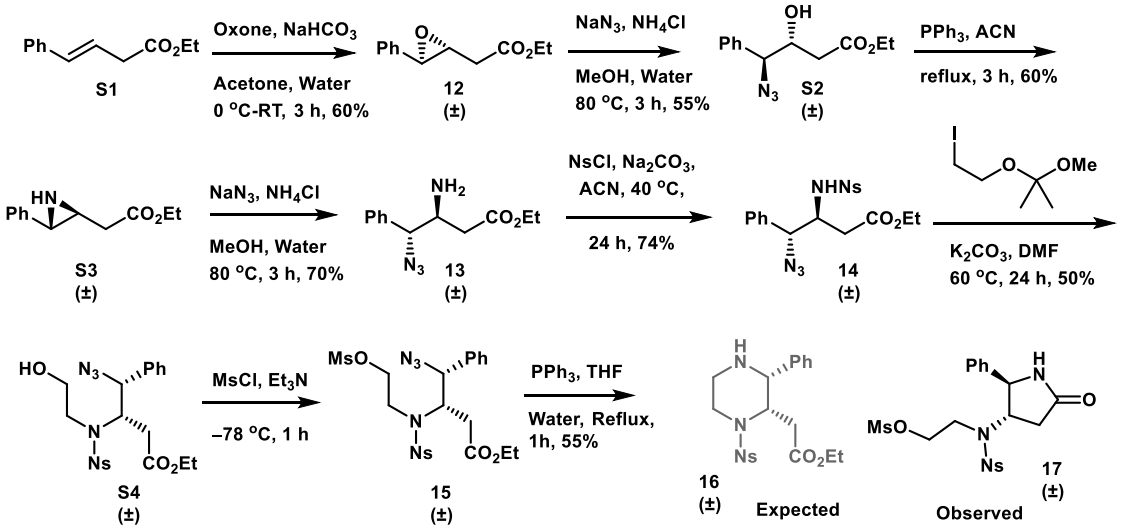


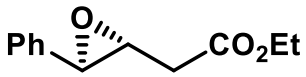
11

Tert-butyl ((1S)-(5-oxo-1,4-oxazepan-7-yl)(phenyl)methyl)carbamate (11): Into a round-bottomed flask equipped with magnetic stir bar and septum, α, β- unsaturated olefin (**1 equiv.**) was dissolved in anhydrous DCM. Next, ethanolamine (**4 equiv.**) was added by syringe. The homogeneous solution was stirred at rt for 48h, after which time the TLC and LCMS showed no starting material remaining. The reaction mixture was evaporated to an oil, redissolved in ethyl acetate and washed with water and brine. The organic phase was collected and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure to give the crude residue. Purification by silica gel chromatography (ethyl acetate/ hexanes) provided the pure product 11. Yield; 40%. Formula: $C_{17}H_{24}N_2O_4$; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.43-7.41 (m, 2H), 7.38 – 7.31 (m, 3H), 7.03 (bs, 1H), 5.84 (bs, 1H), 3.73 – 3.66 (m, 2H), 3.45 (bs, 1H), 3.42 – 3.34 (m, 2H), 3.19 – 3.17 (m, 2H), 1.49-1.47 (m, 9H).



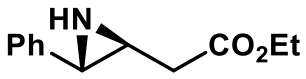
Scheme S1: synthesis of 3-Phenyl substituted piperazines-2-acetic acid ester

The following compounds synthesized by following literature procedure [1 & 2].



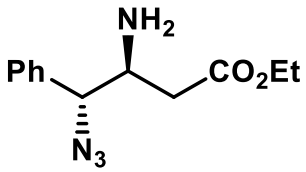
12
(±)

Ethyl 2-(3-phenyloxiran-2-yl)acetate (12); Formula: $C_{12}H_{14}O_3$; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.39 – 7.30 (m, 5H), 4.22 (qd, *J* = 7.1, 1.9 Hz, 2H), 3.74 (d, *J* = 2.1 Hz, 1H), 3.37 (ddd, *J* = 6.2, 5.2, 2.1 Hz, 1H), 2.79 – 2.69 (m, 2H), 1.31 (t, *J* = 7.2 Hz, 3H).



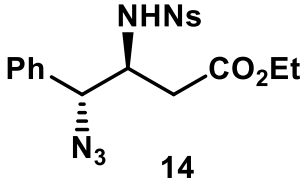
S3
(±)

Ethyl 2-(3-phenylaziridin-2-yl)acetate; Formula: $C_{12}H_{15}NO_2$; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.35 – 7.32 (m, 2H), 7.28 – 7.25 (m, 1H), 7.24 – 7.22 (m, 2H), 4.20 (qd, *J* = 7.1, 1.4 Hz, 2H), 2.81 – 2.79 (m, 1H), 2.69 – 2.62 (m, 1H), 2.57-2.53 (m, 2H), 1.30 (t, *J* = 7.2 Hz, 3H).



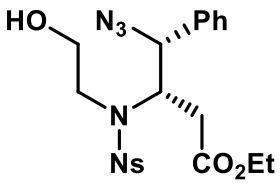
13
(±)

Ethyl (3S,4R)-3-amino-4-azido-4-phenylbutanoate; Formula: $C_{12}H_{16}N_4O_2$; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.46 – 7.44 (m, 2H), 7.41 – 7.39 (m, 3H), 4.81 – 4.74 (m, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 3.60 – 3.57 (m, 1H), 2.68 (dd, *J* = 16.5, 3.8 Hz, 1H), 2.55 – 2.51 (m, 1H), 1.28 (t, *J* = 7.1, 3H).



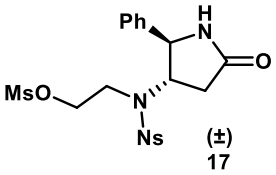
14
(±)

Ethyl (3S,4R)-4-azido-3-((4-nitrophenyl)sulfonamido)-4-phenylbutanoate; Formula: $C_{18}H_{19}N_5O_6S$; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.01 – 7.99 (m, 1H), 7.86 – 7.84 (m, 1H), 7.71 – 7.67 (m, 2H), 7.28 – 7.24 (m, 5H), 6.12 (d, *J* = 8.5 Hz, 1H), 4.93 (d, *J* = 6.3 Hz, 1H), 4.15-4.10 (m, 1H), 4.05 (q, *J* = 7.1 Hz, 2H), 2.72 (dd, *J* = 16.6, 6.3 Hz, 1H), 2.52 (dd, *J* = 16.6, 4.8 Hz, 1H), 1.23 (t, *J* = 7.1, 3H).



S4
(±)

Ethyl (3S,4R)-4-azido-3-((N-(2-hydroxyethyl)-4-nitrophenyl)sulfonamido)-4-phenylbutanoate; Formula: $C_{20}H_{23}N_5O_7S$; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.05 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.72 (td, *J* = 7.6, 1.4 Hz, 1H), 7.68 (td, *J* = 7.7, 1.4 Hz, 1H), 7.60 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.35-7.31 (m, 5H), 5.10 (d, *J* = 5.7 Hz, 1H), 4.43-4.40 (m, 1H), 3.96 (q, *J* = 7.1 Hz, 2H), 3.88 (ddd, *J* = 11.5, 6.9, 4.3 Hz, 1H), 3.83 – 3.79 (m, 1H), 3.72 (ddd, *J* = 15.7, 6.9, 4.3 Hz, 1H), 3.43-3.40 (m, 1H), 2.90 (dd, *J* = 16.4, 8.3 Hz, 1H), 2.75 (dd, *J* = 16.4, 5.0 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H).

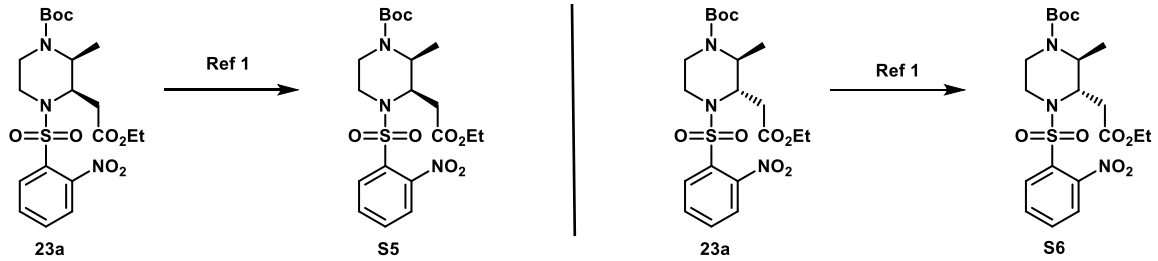


(±)
17

Note: Crude NMR;

2-((4-nitro-N-((2R,3S)-5-oxo-2-phenylpyrrolidin-3-yl)phenyl)sulfonamido)ethyl methanesulfonate; Formula: $C_{19}H_{21}N_3O_8S_2$; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.99 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.76 (ddd, *J* = 8.0, 7.4, 1.4 Hz, 1H), 7.68 – 7.64 (m, 1H), 7.38 – 7.32 (m, 3H), 7.31 – 7.26 (m, 4H), 5.58 (d, *J* = 4.3 Hz, 1H), 4.72 (ddd, *J* = 8.8, 5.2, 4.3 Hz, 1H), 4.46 (t, *J* = 5.6 Hz, 2H), 3.81-3.77 (m, 2H), 3.07 (s, 3H), 3.02 – 2.95 (m, 1H), 2.76 – 2.69 (m, 1H).

23a and 24a are further subjected to Boc-protection on N_1 and compared with literature compounds [1].



Analyzing NMR data for the 23b and 24b piperazine products

Assignments for a single set of resonances were unambiguously inferred for **23b** and **24b** in deuterated chloroform from 2D HSQC and HMBC spectra (Table 1).

The expected ^{1}H-^{1}H J couplings for six membered rings in a canonical chair conformation are: ^{2}J ~12-15 Hz; $^{3}J_{ax\text{-}ax}$ 12-13 Hz, $^{3}J_{ax\text{-}eq}$ ~3.5-4.0 Hz, $^{3}J_{eq\text{-}eq}$ < 2 Hz.

^{3}J analysis for 23b. H2 (4.59 ppm) is a multiplet due to the ester CH_2, but splitting of H3 (3.95 ppm, 3.2 Hz doublet) matches $^{3}J_{ax\text{-}eq}$. H6 (3.74 ppm, 13.5 Hz doublet, <2Hz multiplet) and H6′ (3.16 ppm, 13.5 Hz, 3.0 Hz, doublet of doublets) share the 13.5 Hz ^{2}J and H6′ adds 13.5 Hz $^{3}J_{ax\text{-}ax}$; the H6′ 3.0 Hz coupling matches $^{3}J_{ax\text{-}eq}$, and the small unresolved H6 coupling matches $^{3}J_{eq\text{-}eq}$. H5 (3.06 ppm, 11.6 Hz doublet, unresolved) and H5′ (2.89 ppm, 12.0 Hz doublet of doublets, 3.5 Hz further doublet) share the 11.6 Hz ^{2}J to which H5′ adds a large splitting that matches $^{3}J_{ax\text{-}ax}$; the H5′ 3.5 Hz coupling matches $^{3}J_{ax\text{-}eq}$, and the unresolved H5 coupling matches $^{3}J_{eq\text{-}eq}$. The ^{3}J couplings within the ring indicate that 23b adopts a single chair, and the $^{3}J_{ax\text{-}eq}$ between H2 and H3 show that this is the **cis product**. The ester methylene protons have nearly identical chemical shifts, indicating that the C2-E1 bond rotates freely with almost no rotamer bias. The ethyl group protons show no evidence of exchange.

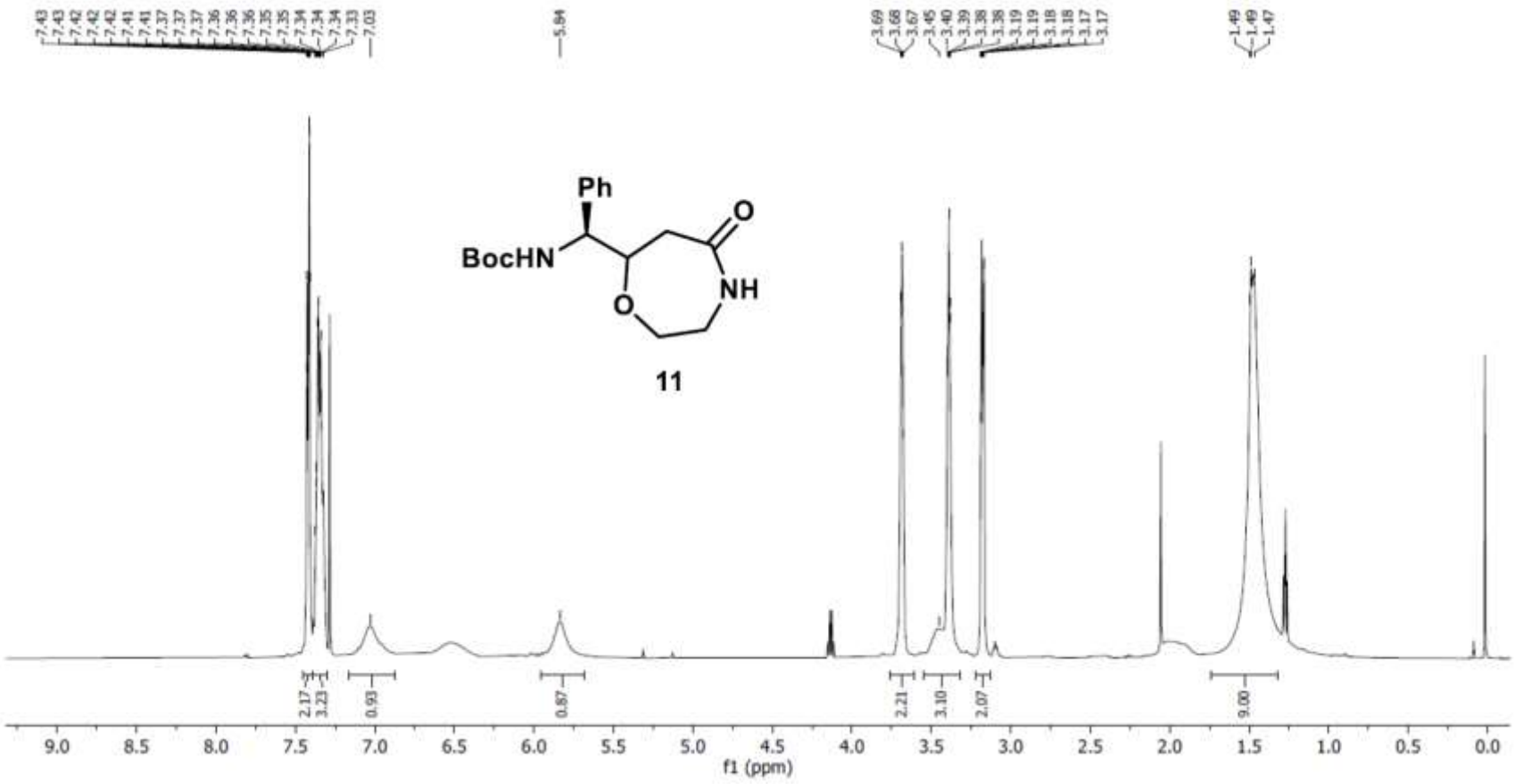
Table 1. Chemical shift assignments from 600 MHz

	23b		**24b**		
	^{1}H	^{13}C	^{1}H	^{13}C	Δδ
2	4.59	55.93	4.67	54.55	-1.4
3	3.95	62.94	3.96	57.14	-5.8
5	3.06	46.61	2.96	39.99	-6.6
5′	2.89		2.80		
6	3.74	41.05	3.51	41.58	+0.5
6′	3.16		3.25		
E1	2.37	31.00	3.18	36.03	+5.0
E1′			2.54		
E2		170.52		170.94	+0.4
Et1	3.62	60.49	4.07*	61.11	+0.6
Et2	0.96	13.95	1.19	14.20	+0.2

^{3}J analysis for 24b. H2 (4.67 ppm, 9.4 Hz, ~3Hz, doublet of doublets) shows a 9.4 Hz coupling to E1; with a pair of doublets of ~3 Hz, $^{3}J_{H2\text{-}H3}$ is a poor match to $^{3}J_{eq\text{-}eq}$ but it could be $^{3}J_{ax\text{-}eq}$ or result from averaging of $^{3}J_{eq\text{-}eq}$ with a little $^{3}J_{ax\text{-}ax}$. The broad H3 peak (3.95 ppm, 6 Hz FWHM, unresolved) could mask any doublet of about 3 Hz or smaller, and so is of little help. E1 (3.18 ppm, 16.3 Hz, 9.4 Hz, doublet of doublets) and E1′ (2.54 ppm, 16.3 Hz, 3.4 Hz, doublet of doublets) share a 16.3 Hz ^{2}J; the E1 9.4 Hz doublet is due to H2, as is the E1′ 3.4 Hz doublet. The different values for $^{3}J_{H2\text{-}E1}$ and $^{3}J_{H2\text{-}E1'}$ mean that any averaging about the C2-E1 bond must be highly rotamer biased, consistent with the E1/E1′ chemical shift differences. H5 (2.96 ppm, 11.9 Hz, 3.9 Hz, doublet of doublets) and H5′ (2.80 ppm, 12.2 Hz, 3.0 Hz, doublet of doublets) share the 12.2 Hz ^{2}J and H5 adds one coupling that is somewhat weaker than $^{3}J_{ax\text{-}ax}$ and another that is at the upper limit of or stronger than $^{3}J_{ax\text{-}eq}$. The two H5′ ~3.0 Hz couplings are too strong and too similar to correspond to both $^{3}J_{eq\text{-}eq}$ and $^{3}J_{ax\text{-}eq}$. H6 (3.51 ppm, 12.9 Hz, 3.0 Hz, doublet of doublets) and H6′ (3.25 ppm, 12.1, 3.5 Hz, doublet of doublets) share the 12.9 Hz ^{2}J and H6′ adds a strong coupling that seems weaker than $^{3}J_{ax\text{-}ax}$; the H6′ 3.5 Hz coupling could however be $^{3}J_{ax\text{-}eq}$. The lack of a small $^{3}J_{eq\text{-}eq}$ (< 2 Hz) between the H5s/H6s or between H2 and H3 indicates that 24b does not adopt a single chair; we infer that it averages between the diaxial (predominant) and diequatorial species. The multiplet pattern for the ethyl CH_2 is consistent with some kind of exchange.

References:

1. Reddy Guduru, S.K.; Chamakuri, S.; Raji, I.O.; MacKenzie, K.R.; Santini, C.; Young, D.W. Synthesis of Enantiomerically Pure 3-Substituted Piperazine-2-acetic Acid Esters as Intermediates for Library Production. *J. Org. Chem.* **2018**, *83*, 11777–11793.
2. Bisol, T.B.; Bortoluzzi, A.J.; Sa, M.M. Nucleophilic ring-opening of epoxide and aziridine acetates for the stereodivergent synthesis of β-hydroxy and β-amino γ-lactams. *J. Org. Chem.* **2011**, *76*, 948–962.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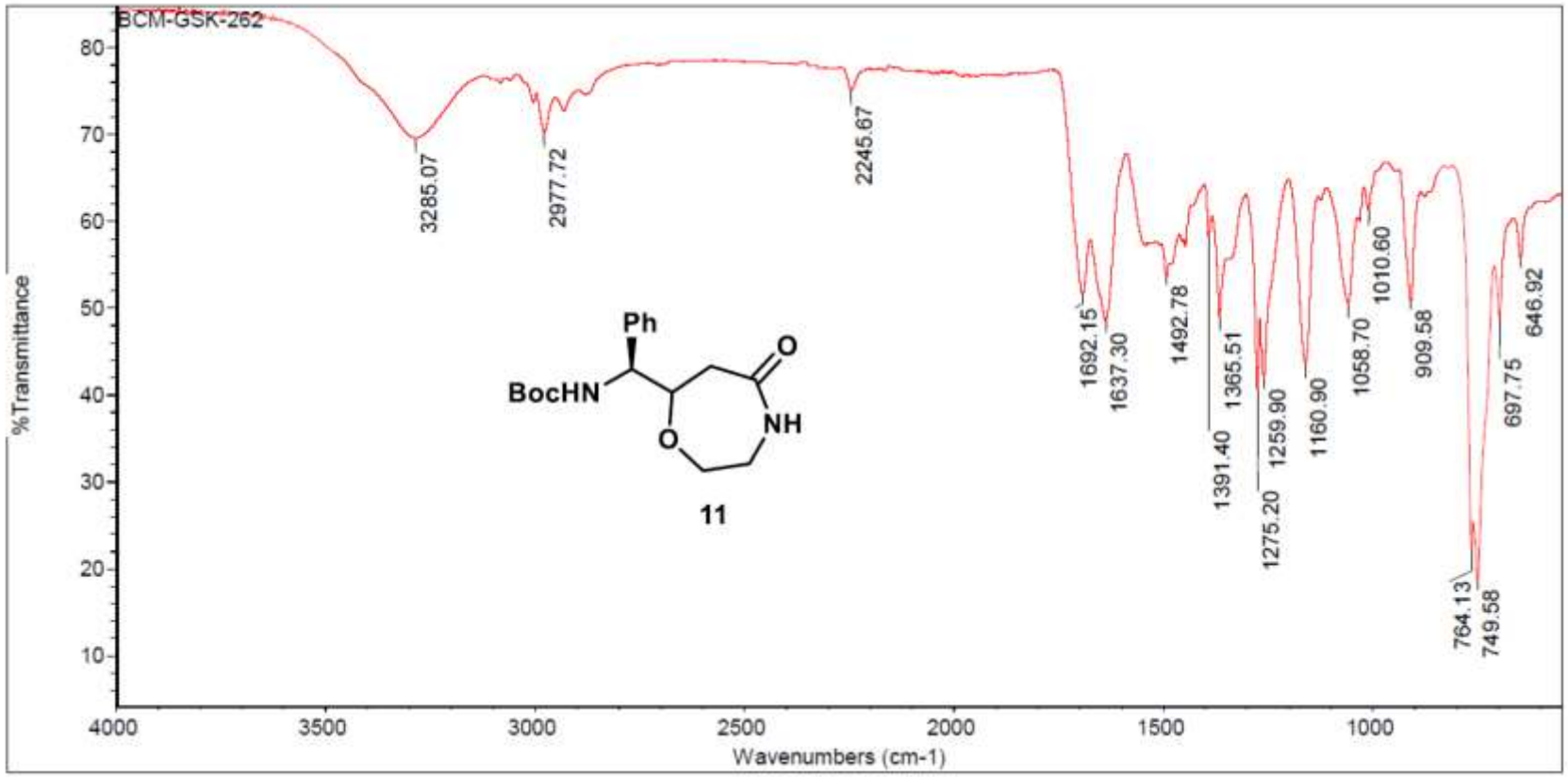
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20
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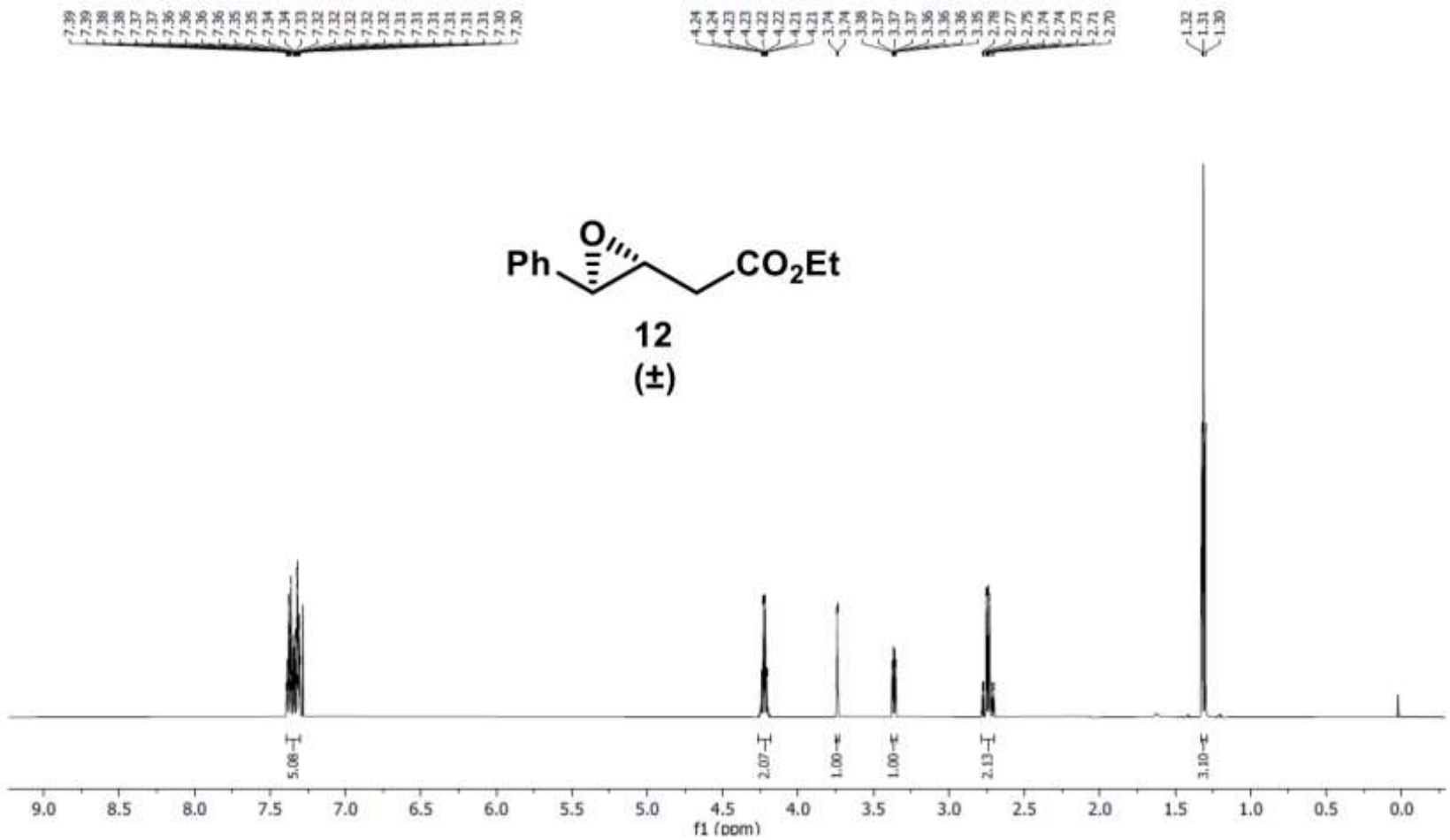
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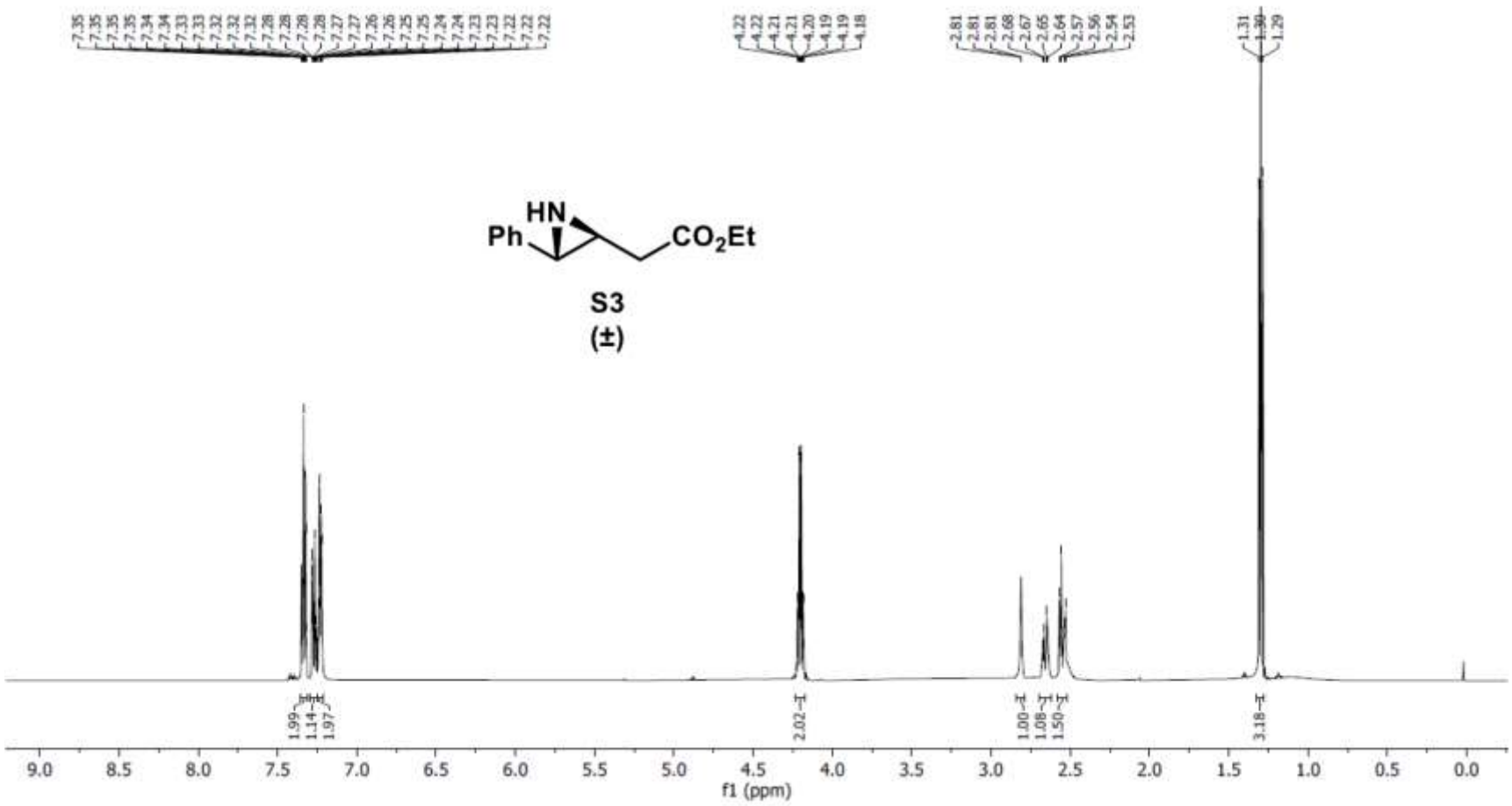
f1 (ppm)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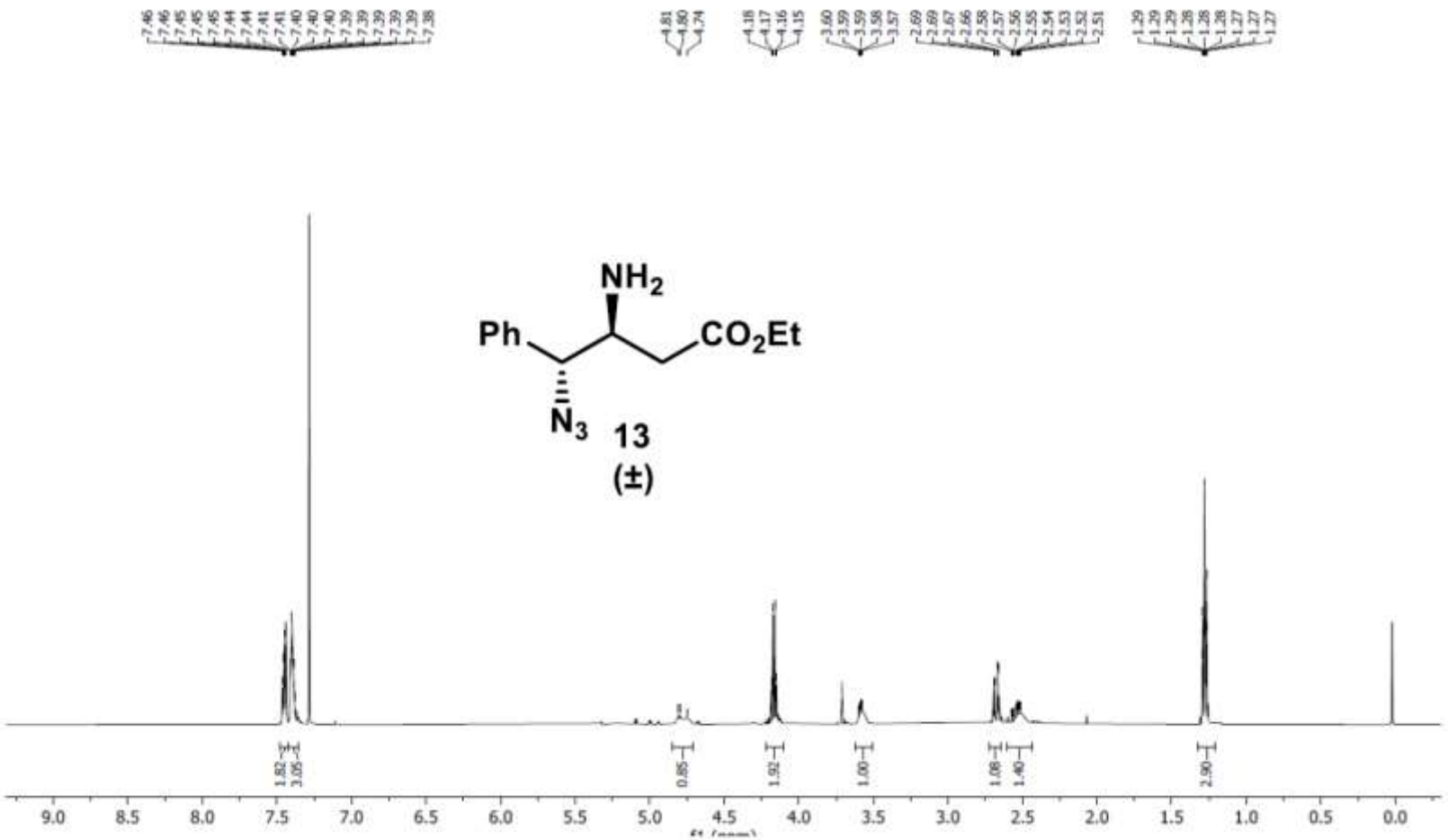
S3
(±)
f1 (ppm)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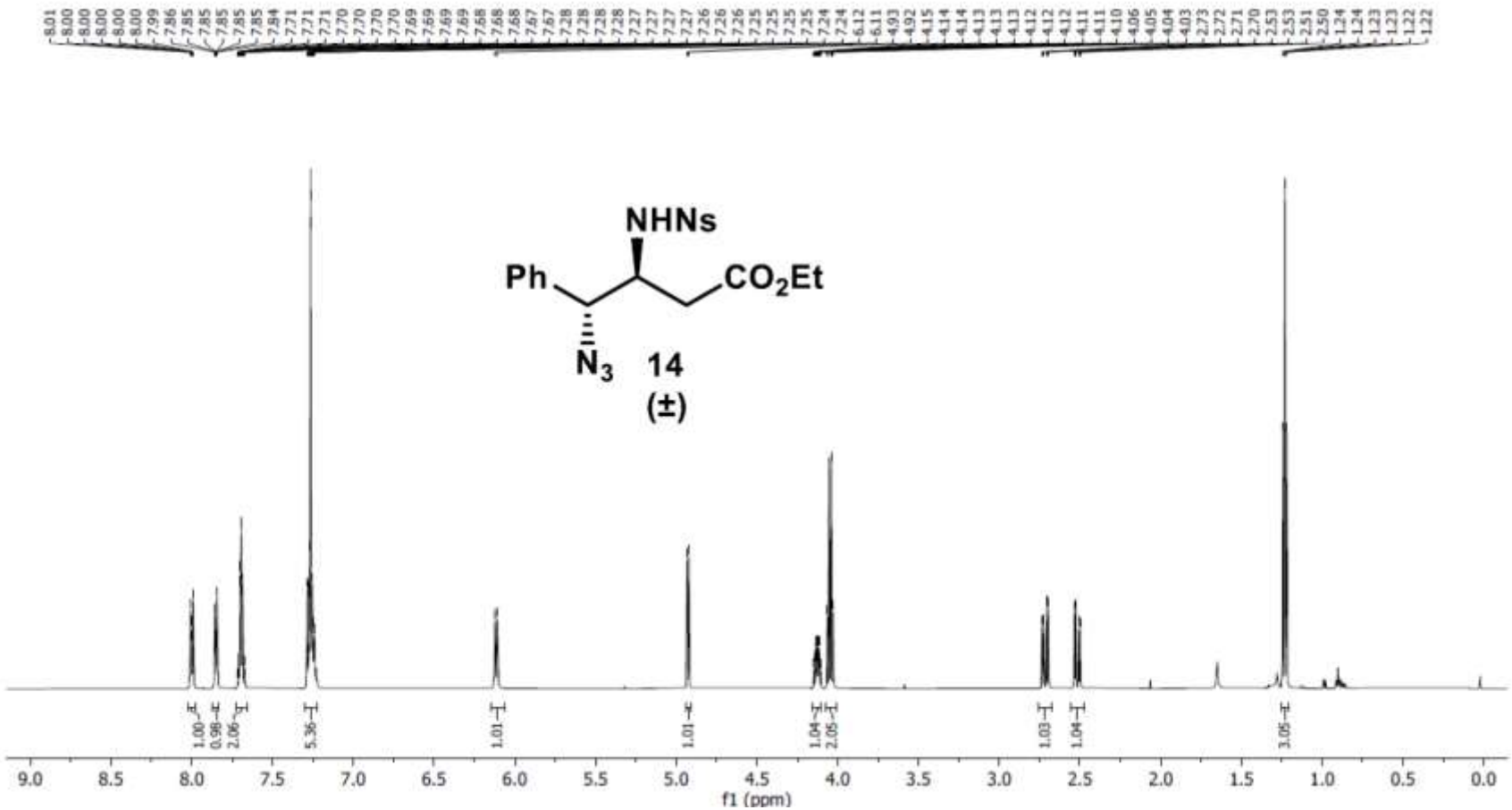
(±)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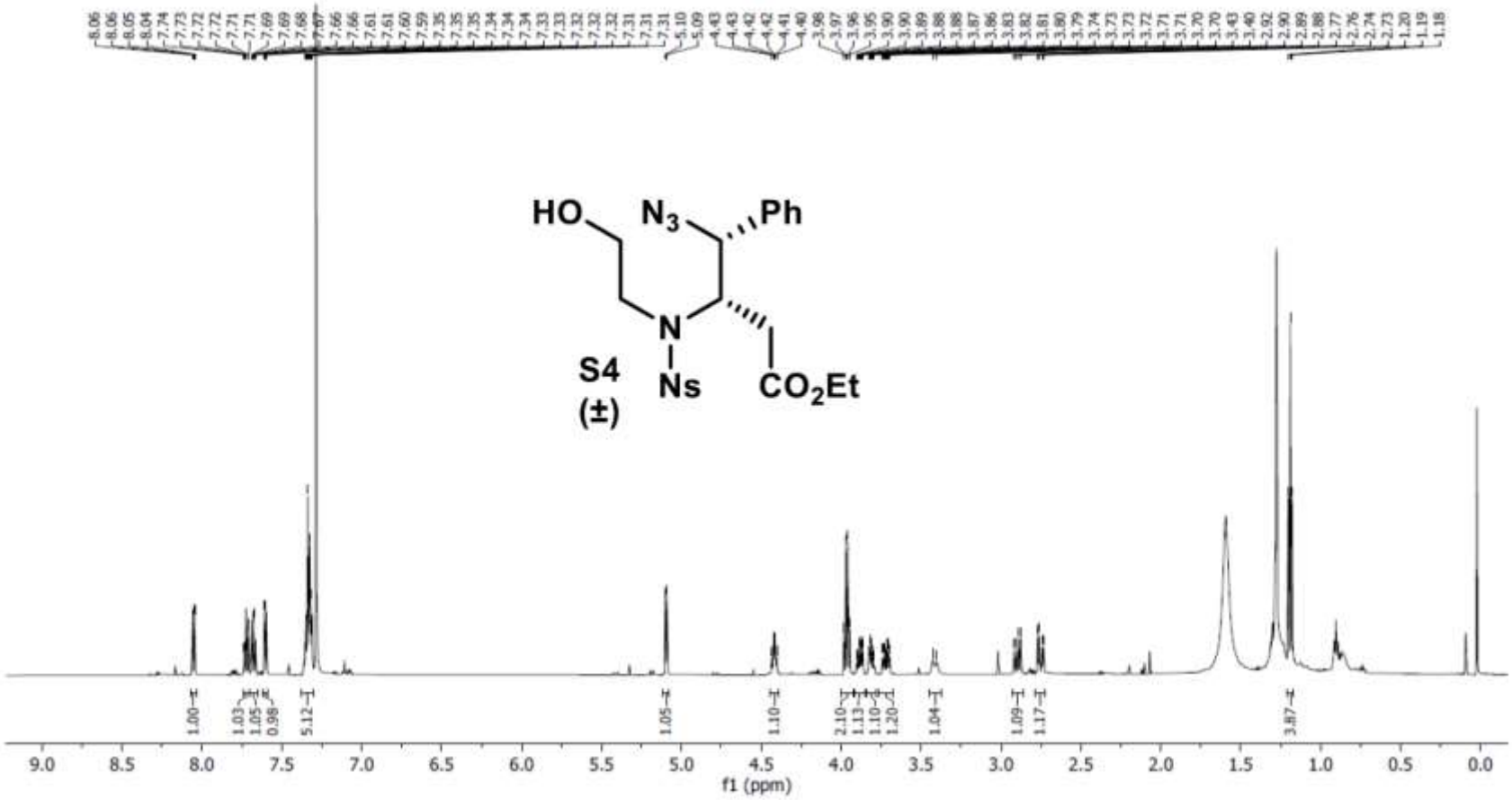
(±)
f1 (ppm)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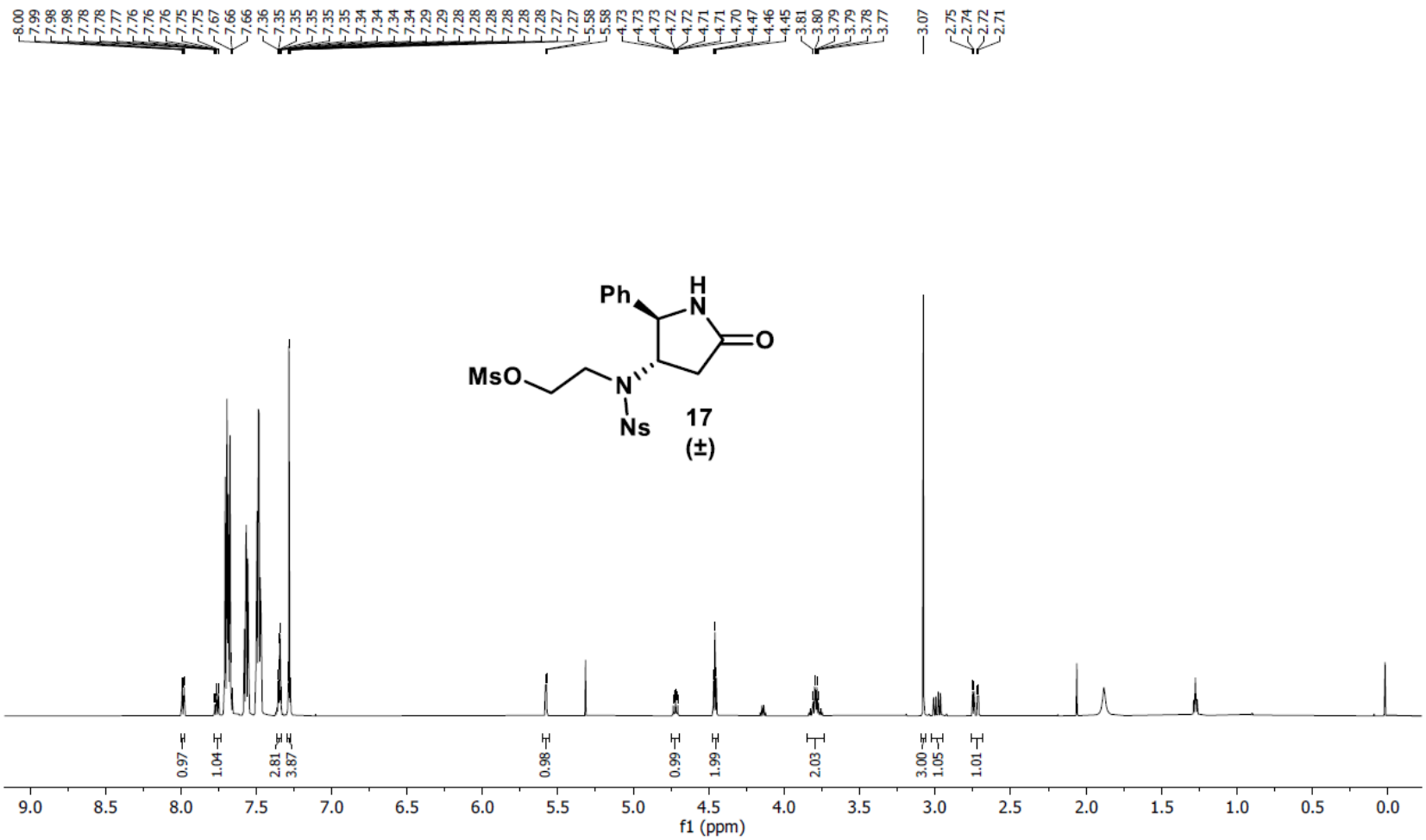
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(±)
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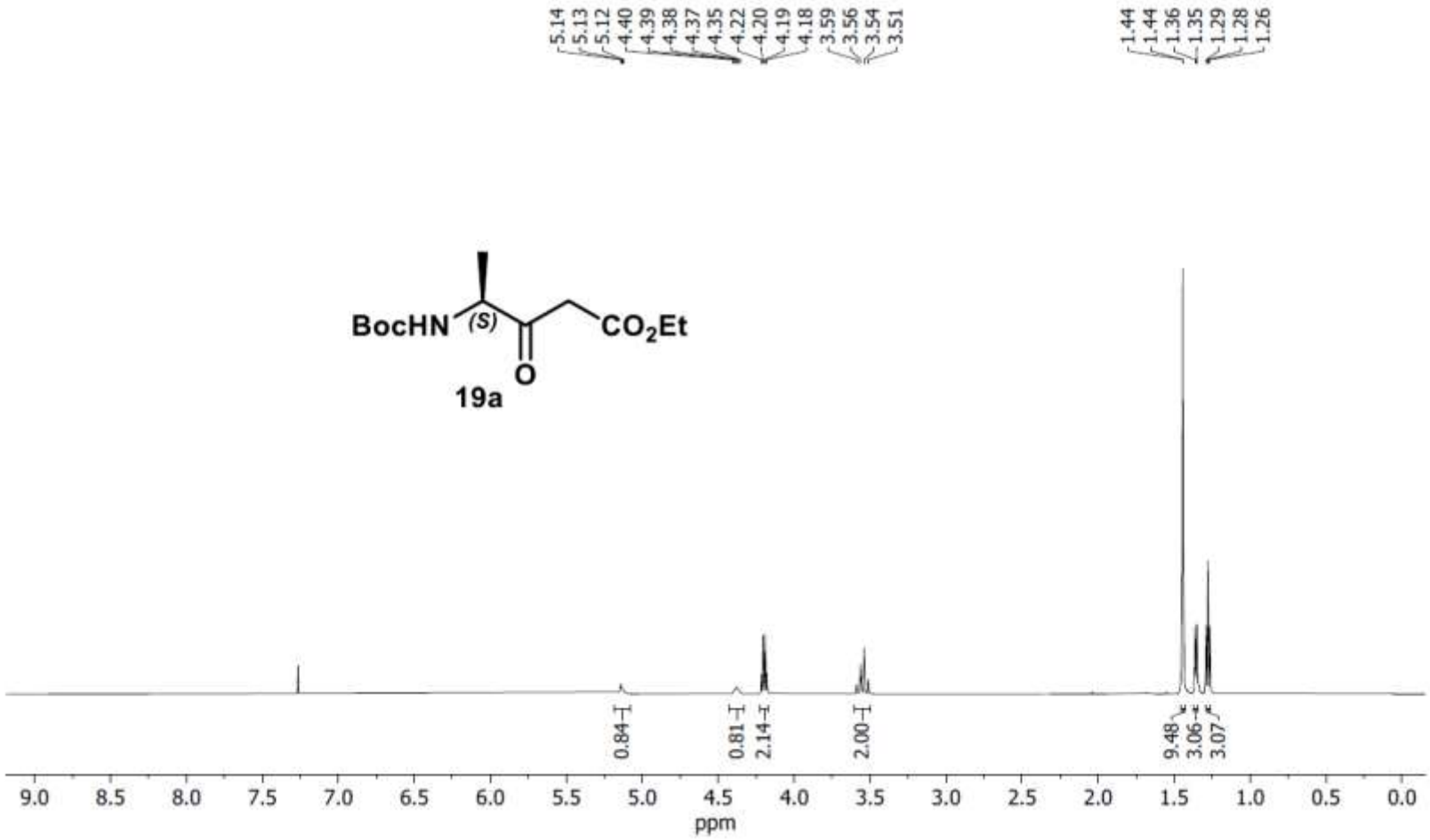
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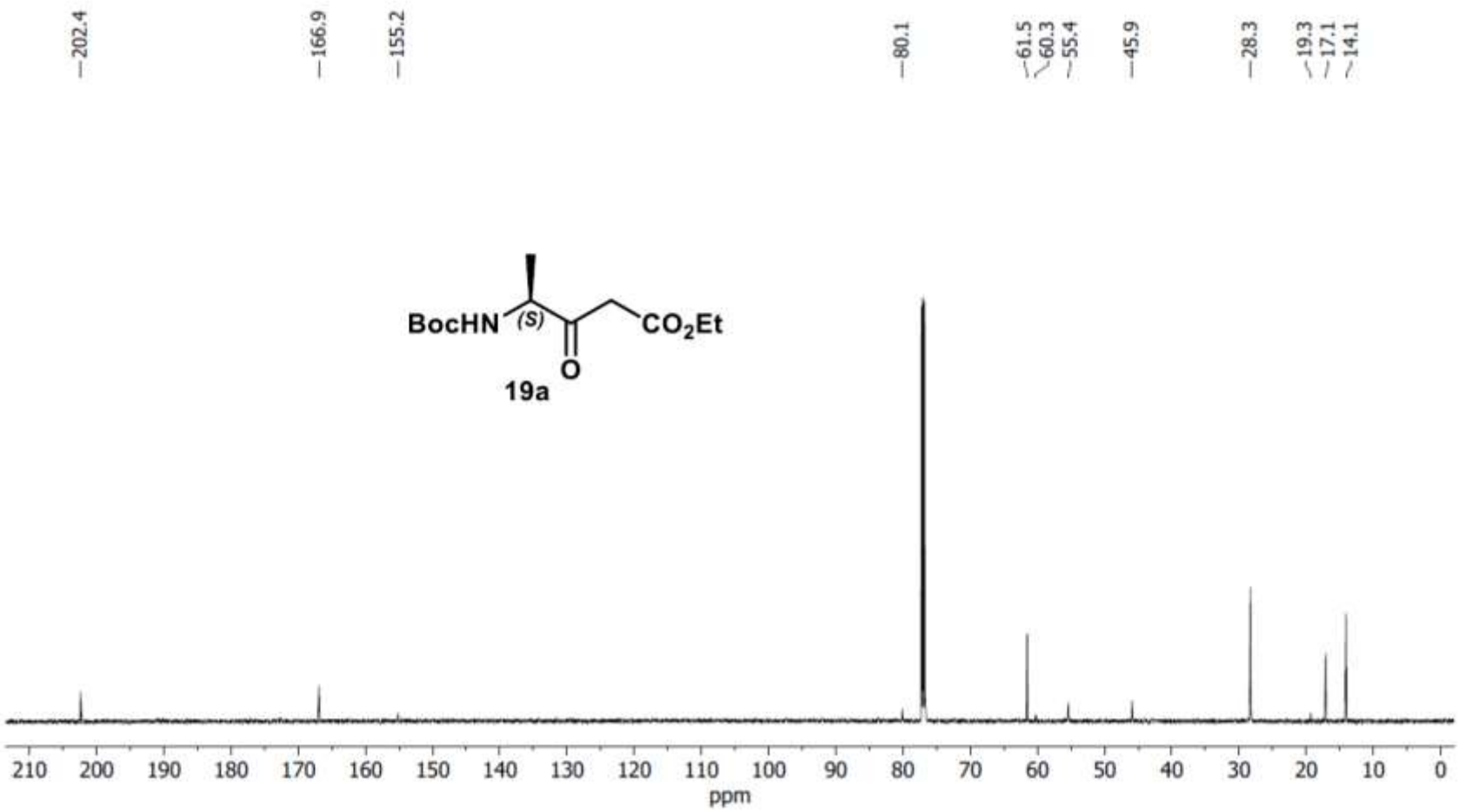
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CO2Et
O
19a
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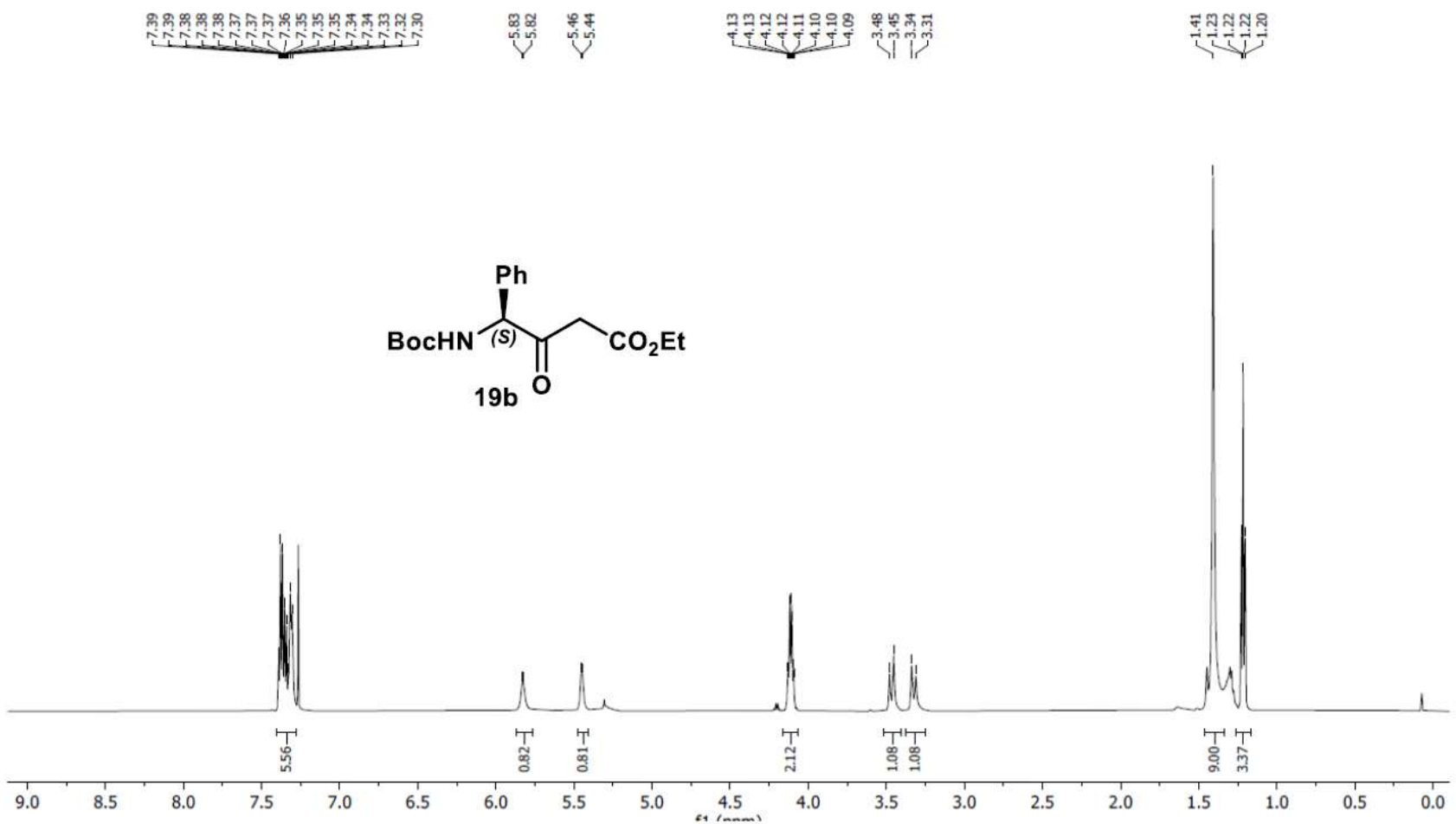
ppm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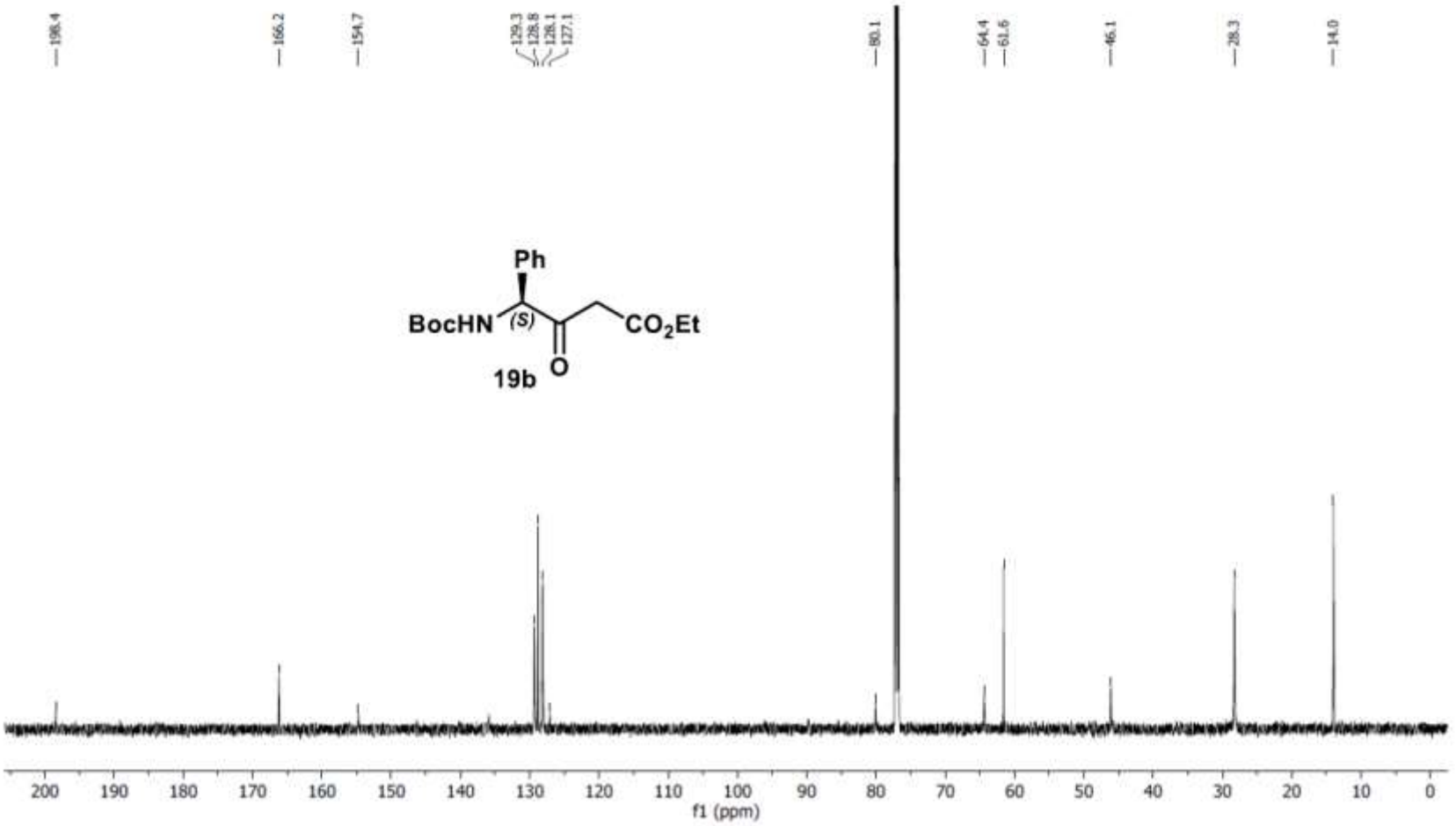
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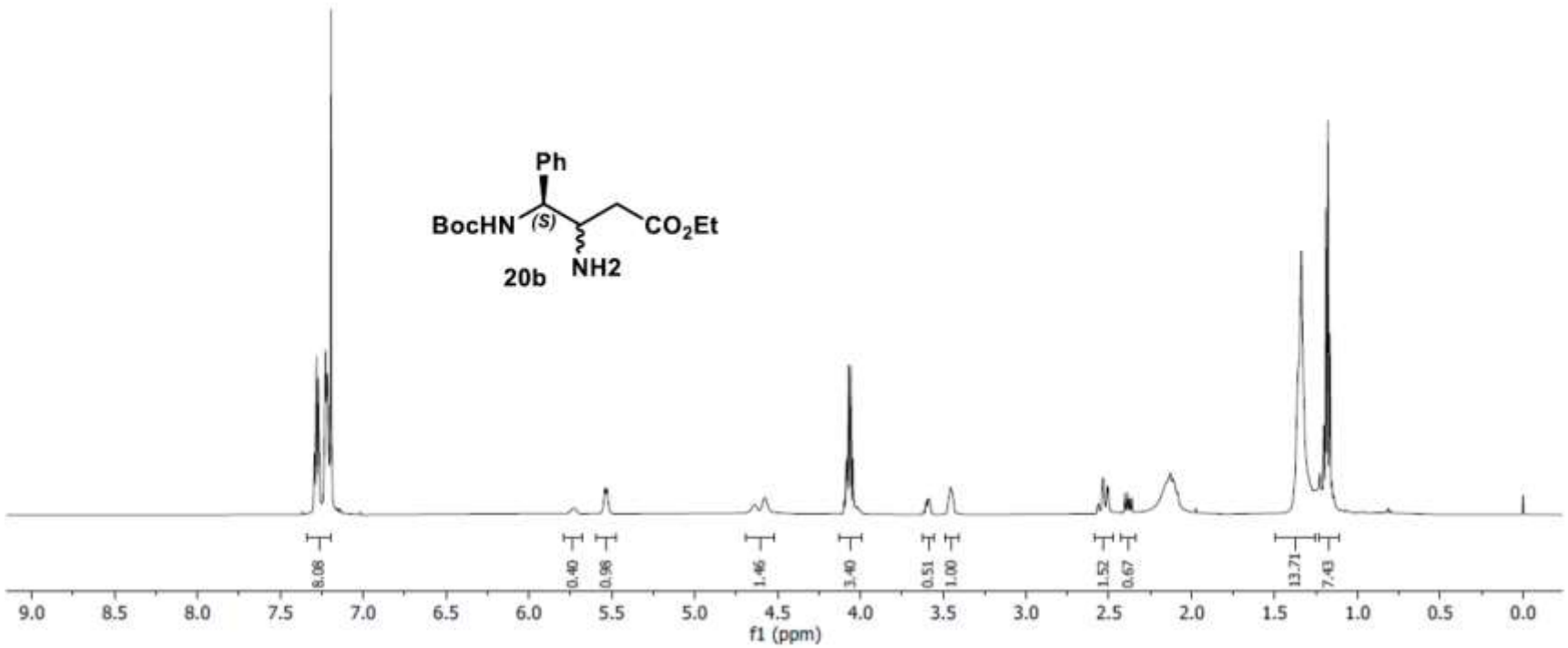
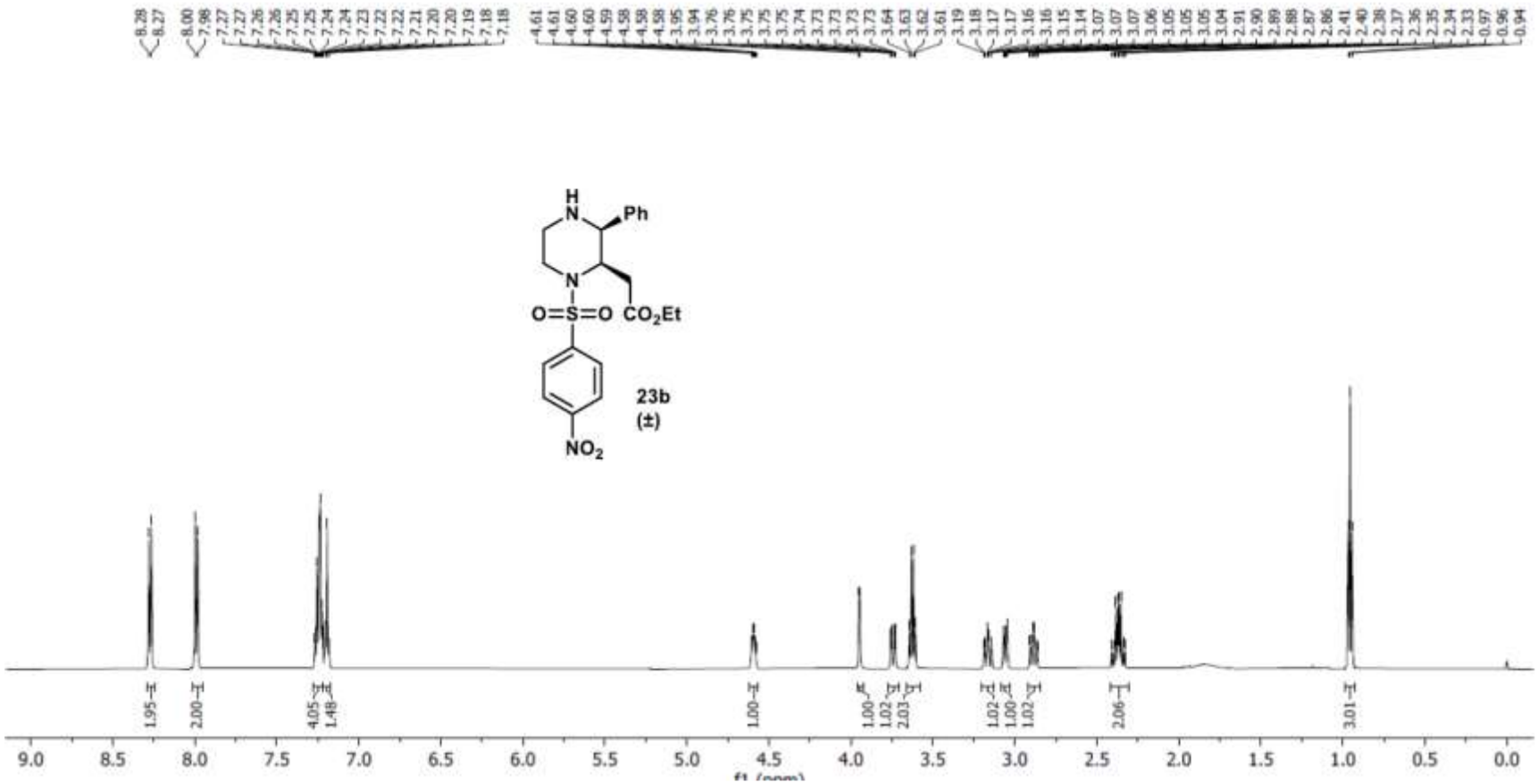
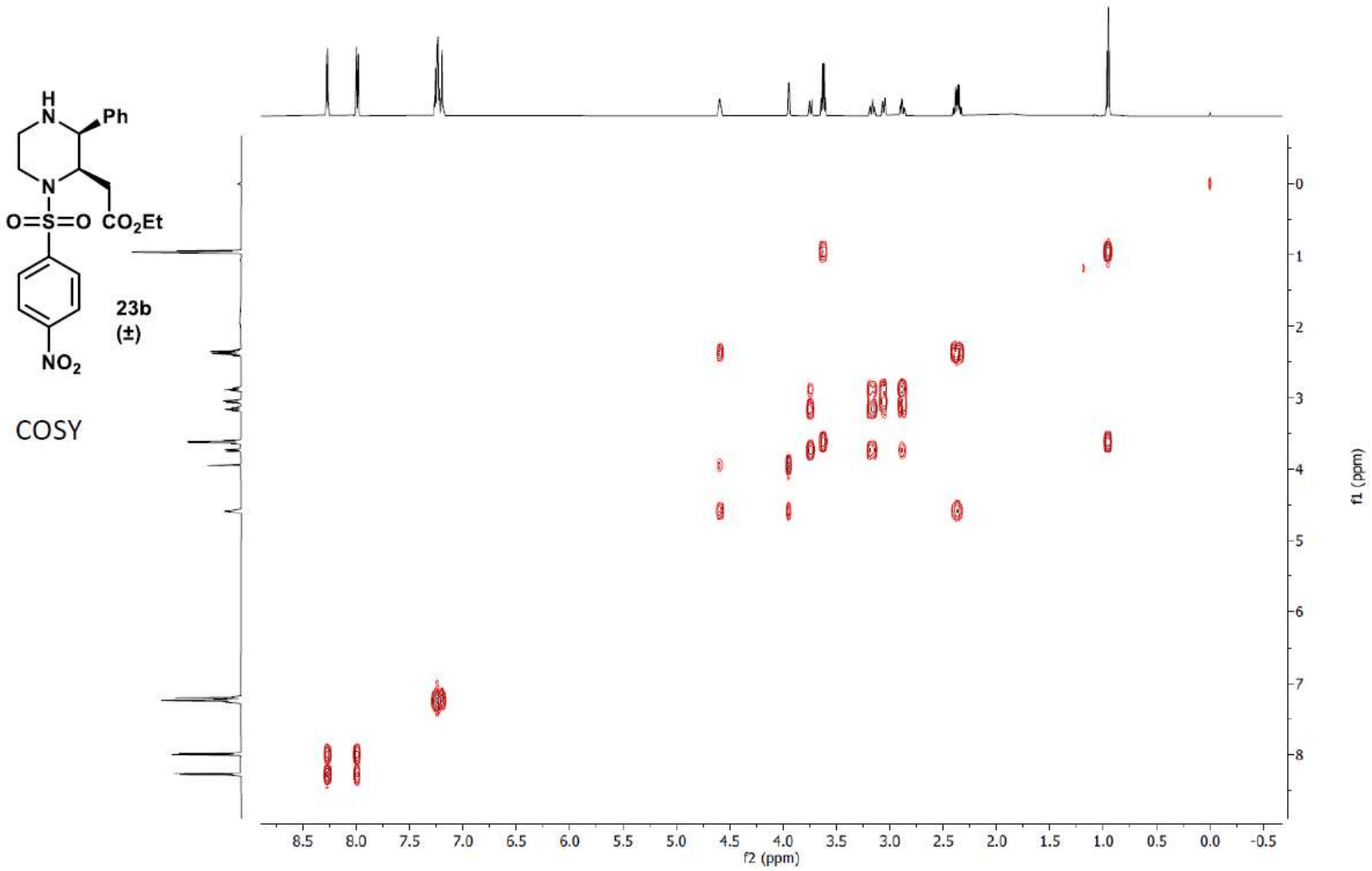
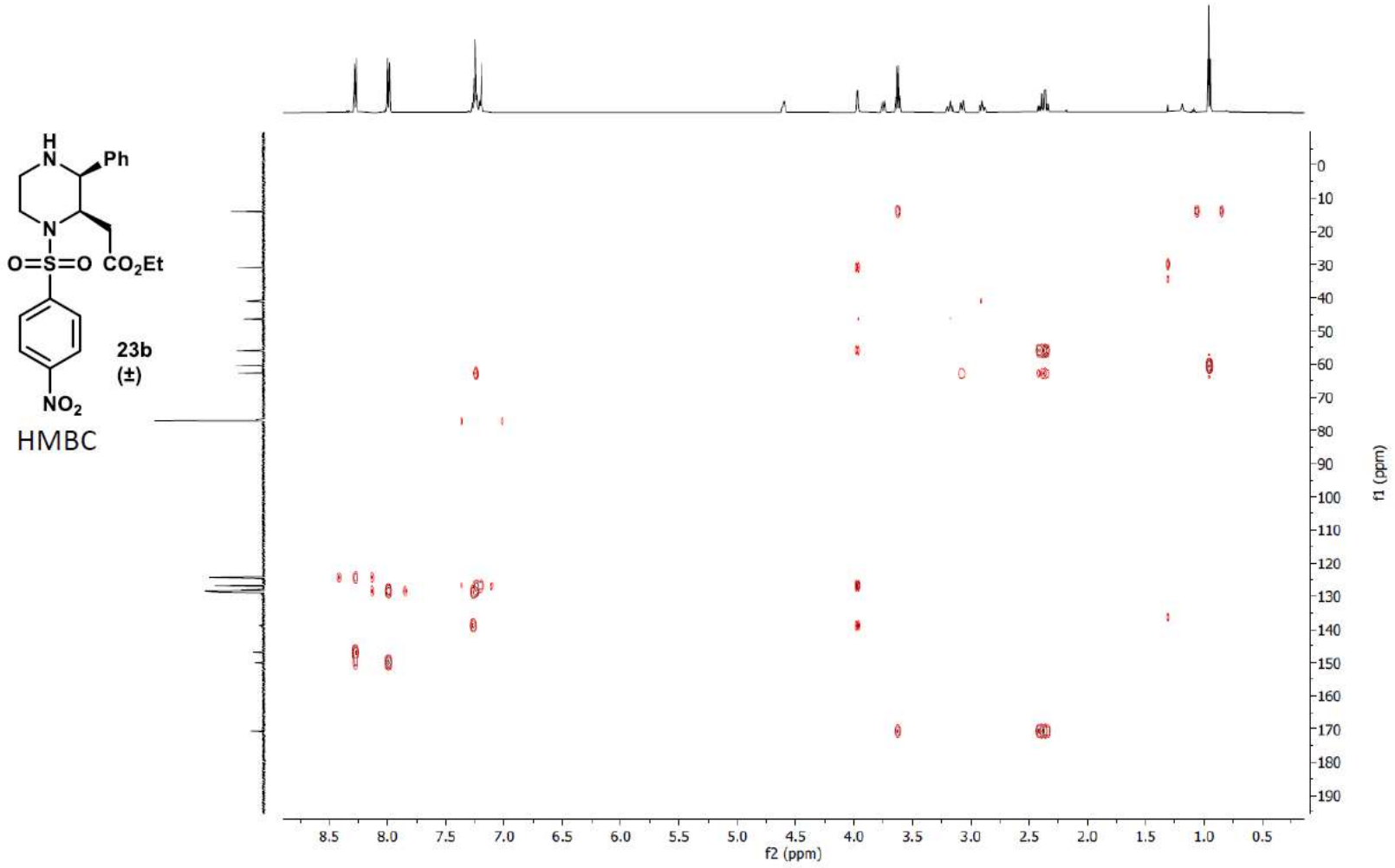
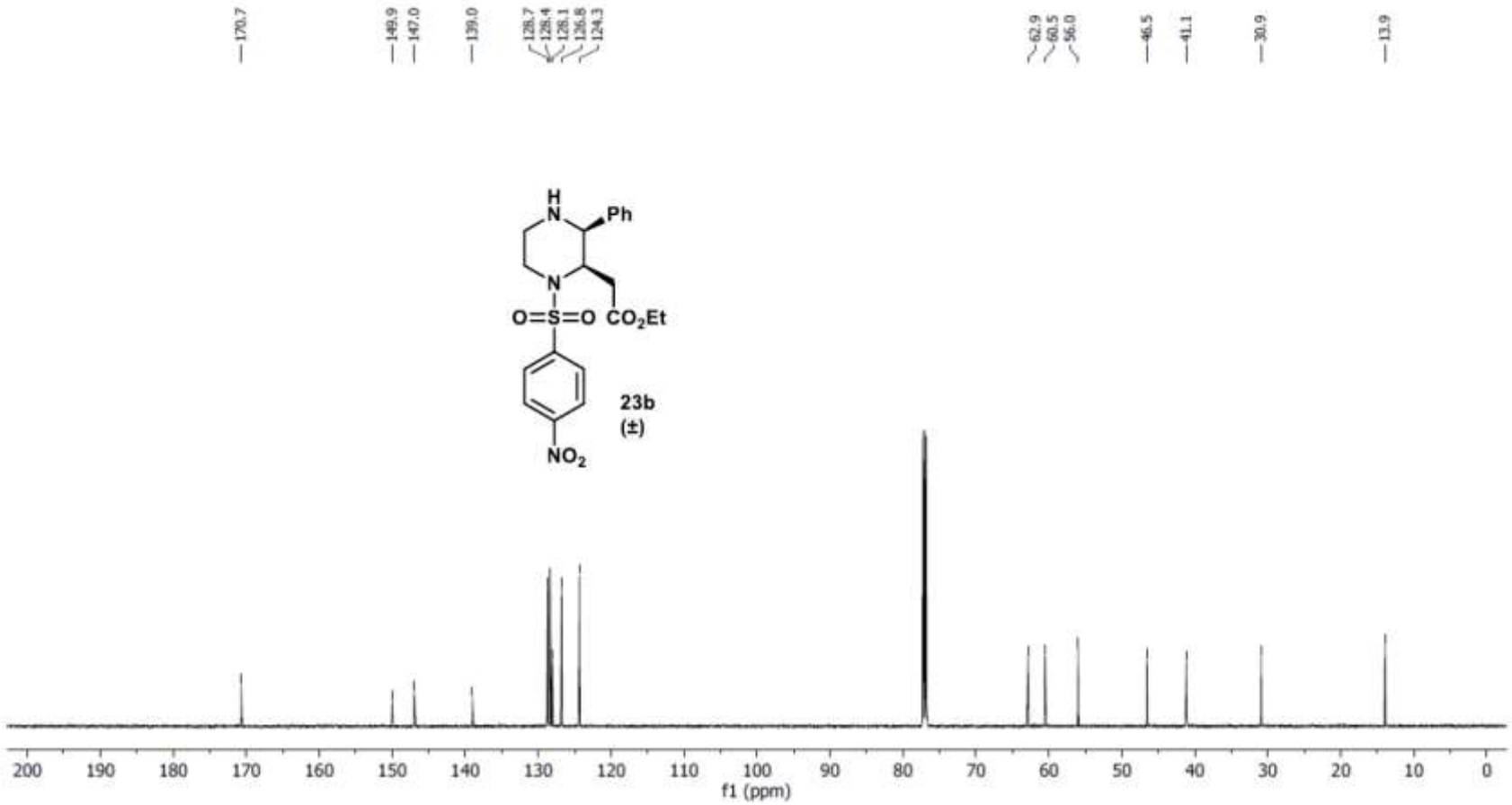
O
19b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

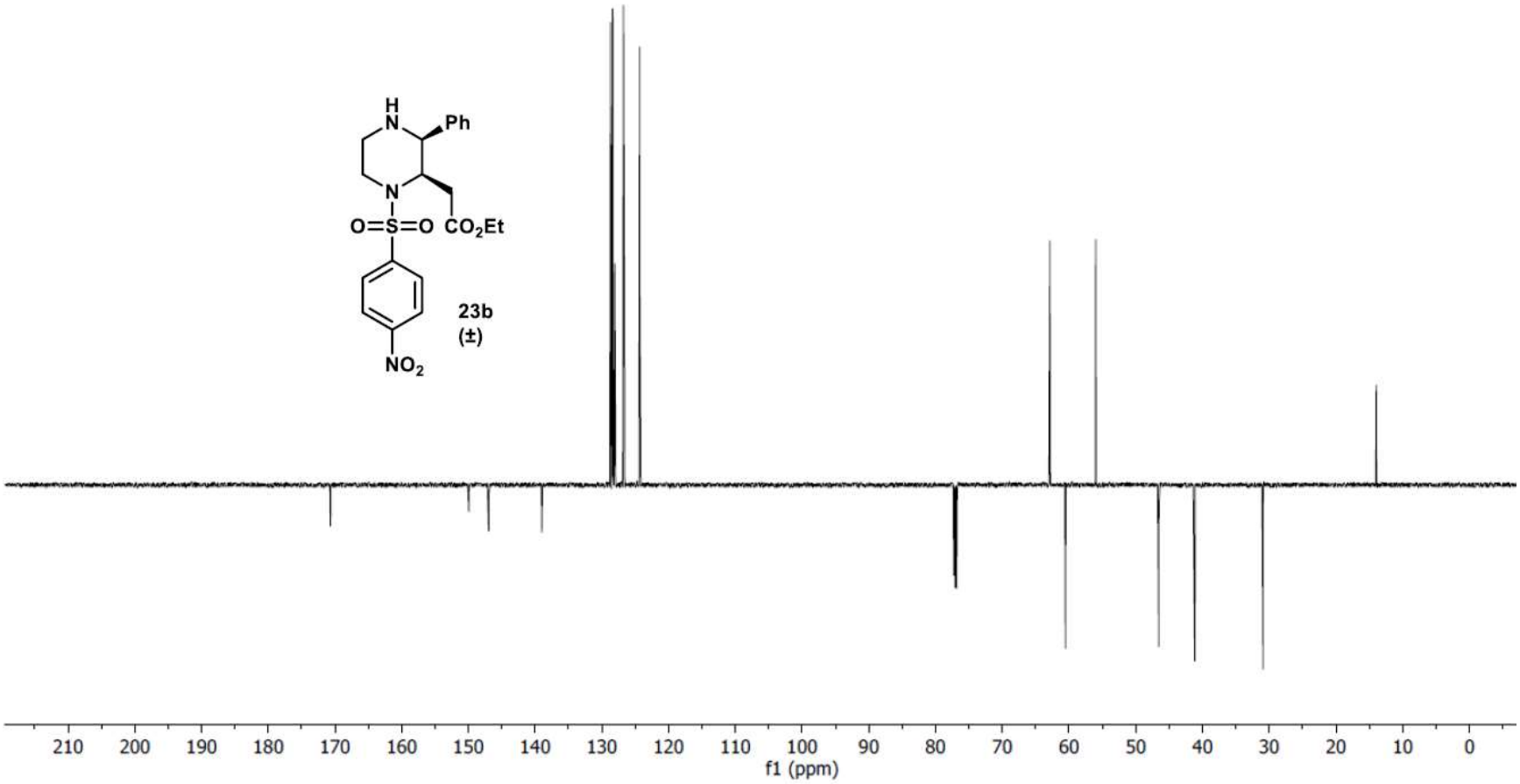

Ph
BocHN
(S)
CO2Et
NH2
20b
8.08
0.40
0.98
1.46
3.40
0.51
1.00
1.52
0.67
13.71
7.43
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

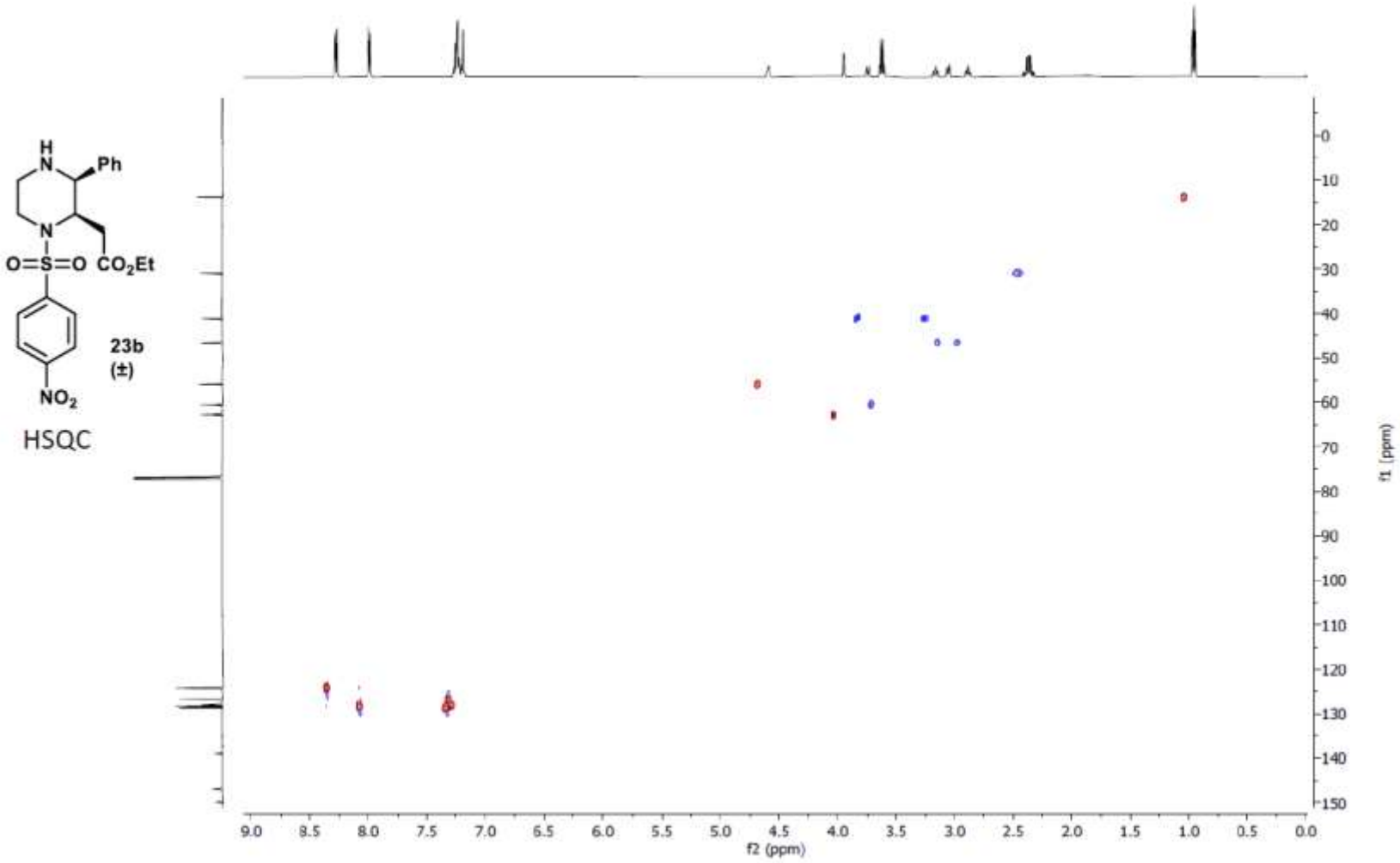

8.28
8.27
8.00
7.98
7.27
7.27
7.27
7.26
7.26
7.25
7.25
7.24
7.24
7.23
7.22
7.22
7.21
7.20
7.20
7.19
7.18
7.18
4.61
4.61
4.60
4.60
4.59
4.58
4.58
4.58
3.95
3.94
3.76
3.76
3.75
3.75
3.75
3.74
3.73
3.73
3.73
3.73
3.64
3.63
3.62
3.61
3.19
3.18
3.17
3.17
3.16
3.16
3.15
3.14
3.07
3.07
3.07
3.06
3.05
3.05
3.05
3.04
2.91
2.90
2.89
2.88
2.87
2.86
2.41
2.40
2.38
2.37
2.36
2.35
2.34
2.33
0.97
0.96
0.94
H
N
Ph
N
O=S=O
CO2Et
23b
(±)
NO2
1.95
2.00
4.05
1.48
1.00
1.00
1.02
2.03
1.02
1.00
1.02
2.06
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

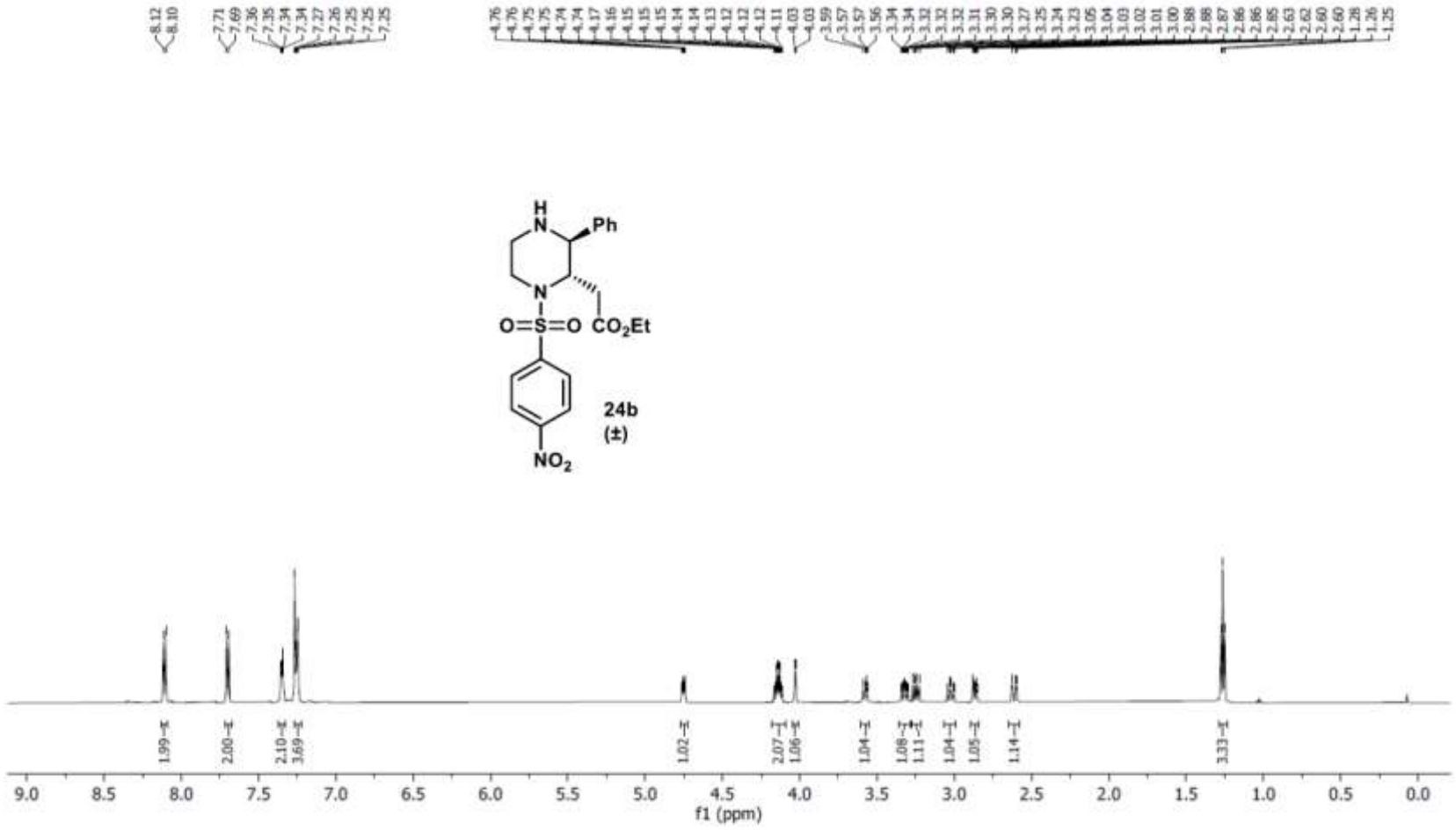

H
N
Ph
N
O=S=O
CO2Et
NO2
23b
(±)
COSY
f2 (ppm)
f1 (ppm)


Ph
O=S=O
CO2Et
NO2
23b
(±)
HMBC
f2 (ppm)
f1 (ppm)


170.7
149.9
147.0
139.0
128.7
128.4
128.1
126.8
124.3
62.9
60.5
56.0
46.5
41.1
30.9
13.9
H
N
Ph
N
O=S=O
CO2Et
NO2
23b
(±)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


H
N
Ph
N
O=S=O
CO2Et
NO2
23b
(±)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Ph
O=S=O
CO2Et
NO2
23b
(±)
HSQC
f2 (ppm)
f1 (ppm)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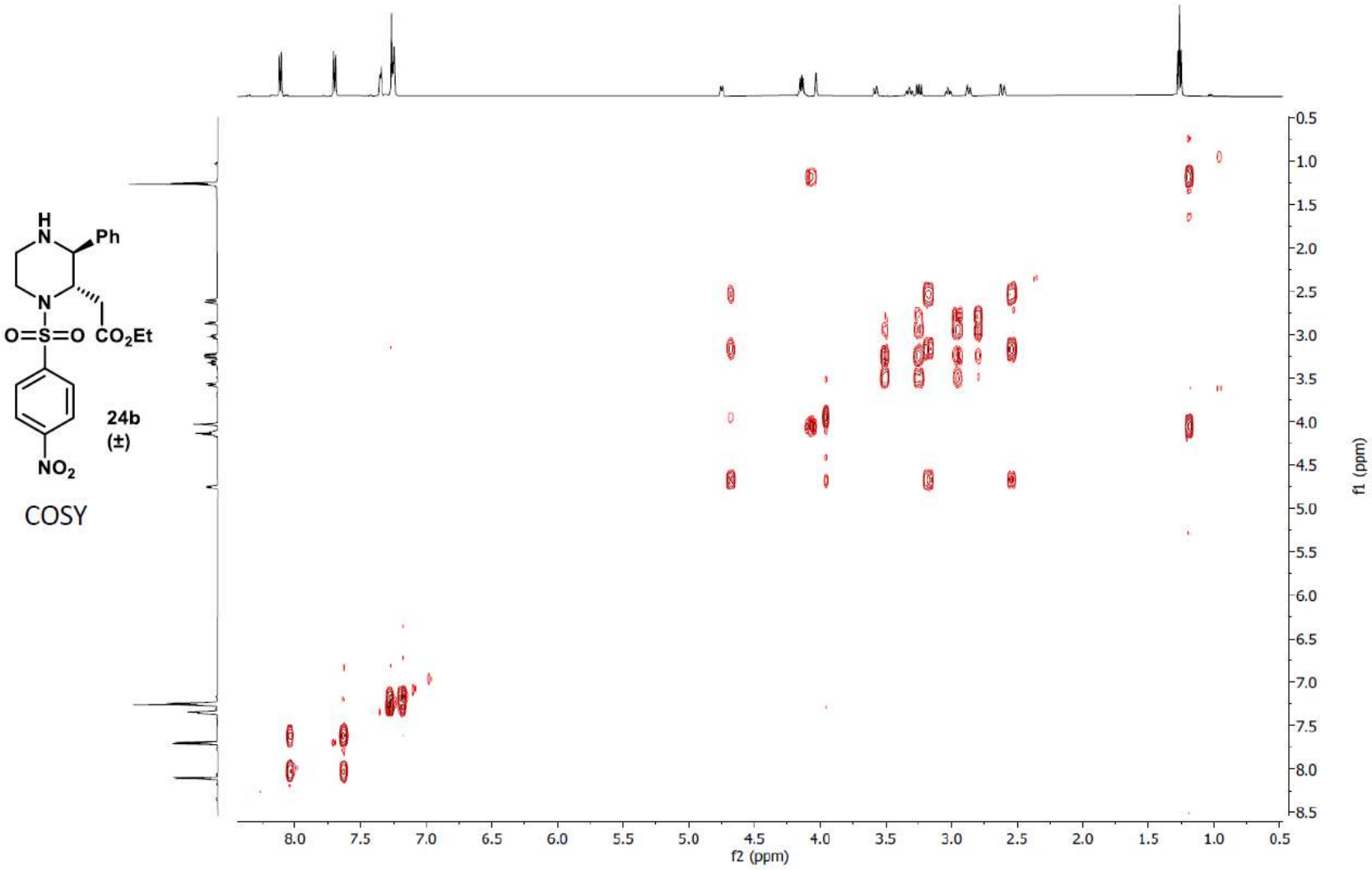
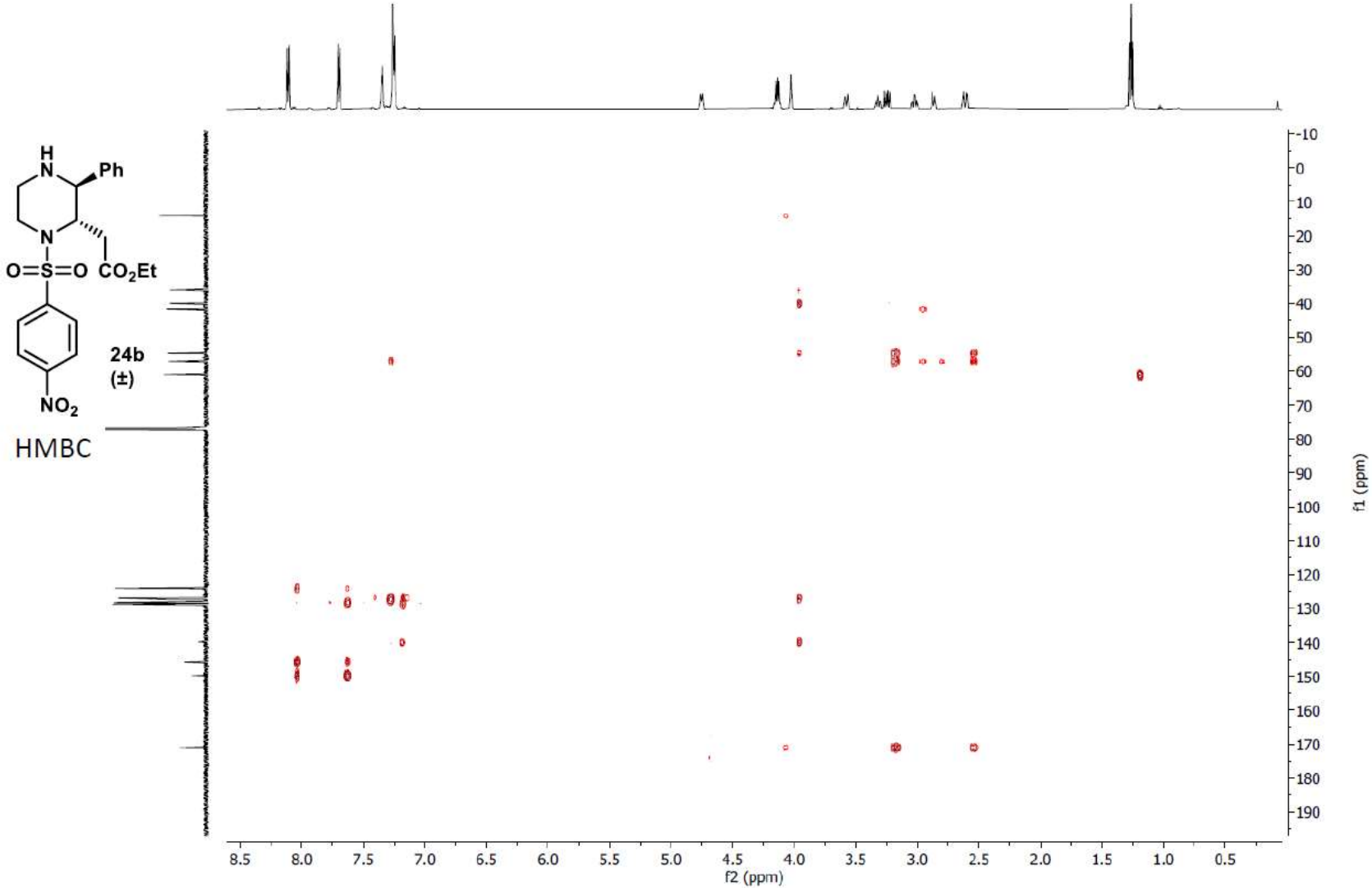
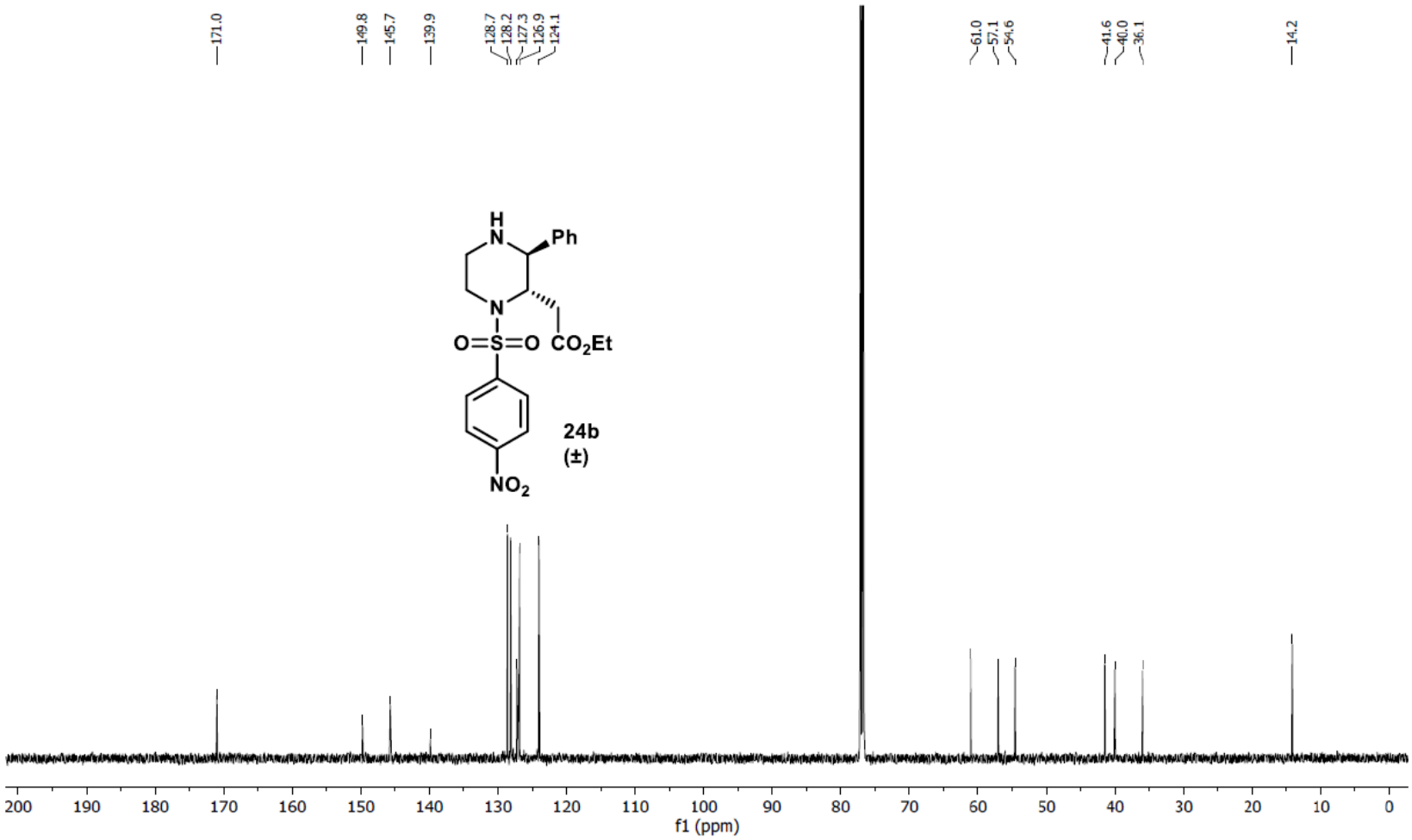
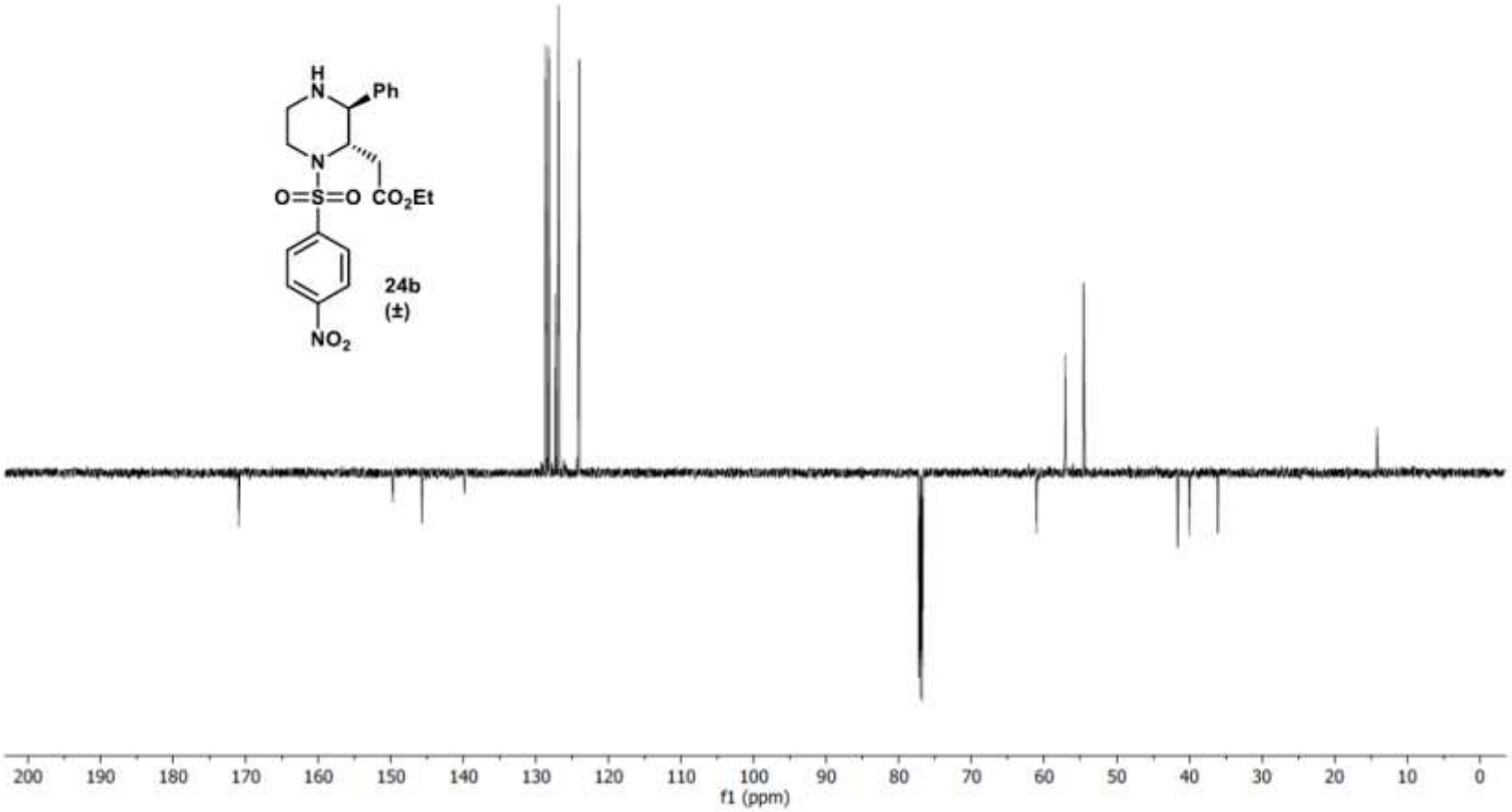
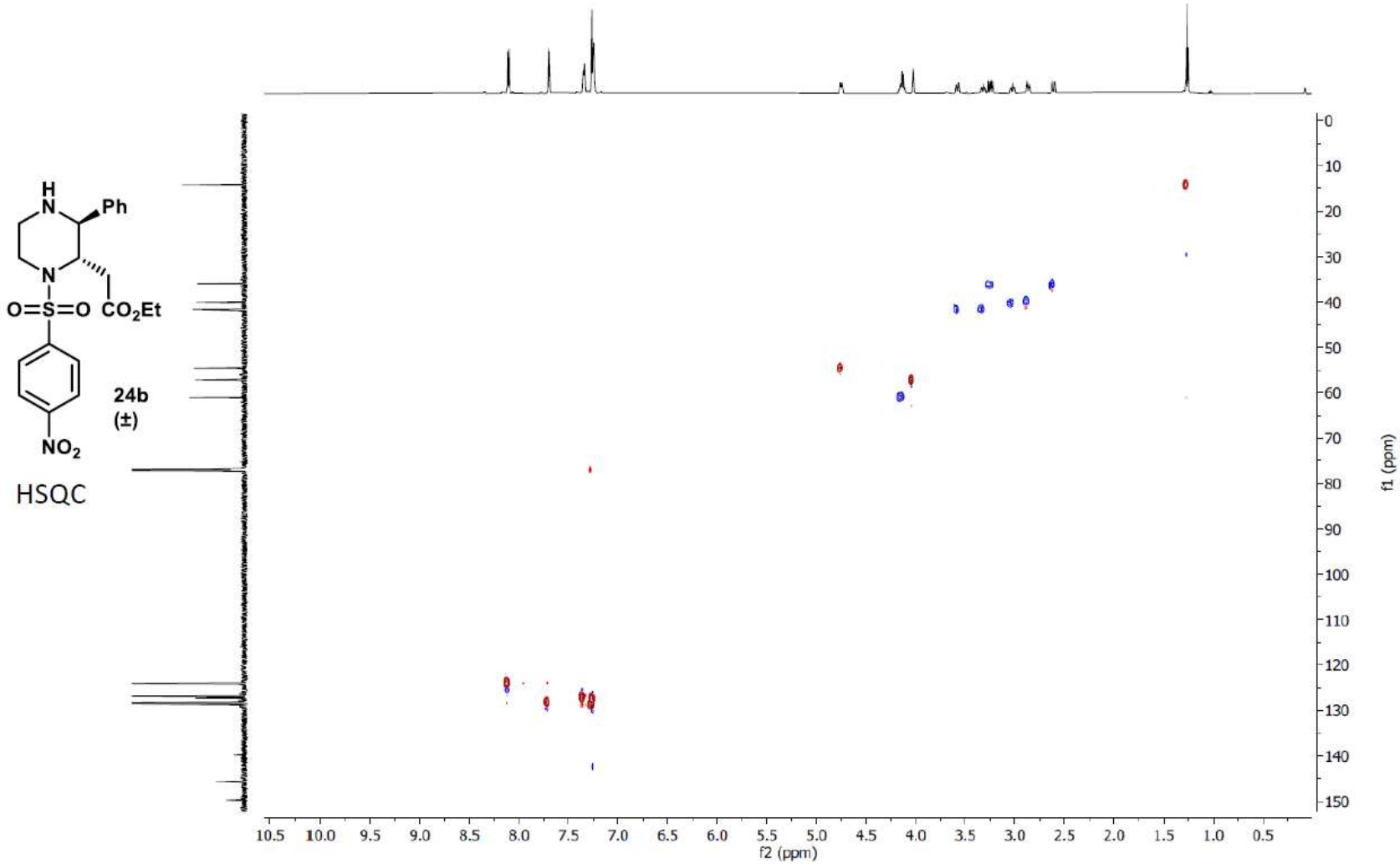
7.34
7.27
7.26
7.25
7.25
7.25
4.76
4.76
4.75
4.75
4.74
4.74
4.17
4.16
4.15
4.15
4.14
4.14
4.13
4.12
4.12
4.12
4.11
4.03
4.03
3.59
3.57
3.57
3.56
3.34
3.34
3.32
3.32
3.32
3.31
3.30
3.30
3.27
3.25
3.24
3.23
3.05
3.04
3.03
3.02
3.01
3.00
2.88
2.88
2.87
2.86
2.86
2.85
2.63
2.62
2.60
2.60
1.28
1.26
1.25
H
N
Ph
N
O=S=O
CO2Et
24b
(±)
NO2
1.99
2.00
2.10
3.69
1.02
2.07
1.06
1.04
1.08
1.11
1.04
1.05
1.14
3.33
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

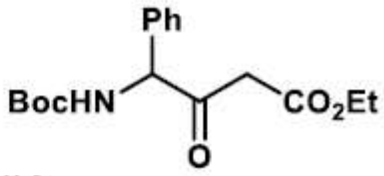

24b
(±)
COSY
f2 (ppm)
f1 (ppm)


Ph
O=S=O
CO2Et
NO2
24b
(±)
HMBC
f2 (ppm)
f1 (ppm)


171.0
149.8
145.7
139.9
128.7
128.2
127.3
126.9
124.1
61.0
57.1
54.6
41.6
40.0
36.1
14.2
H
N
Ph
N
O=S=O
CO2Et
NO2
24b
(±)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

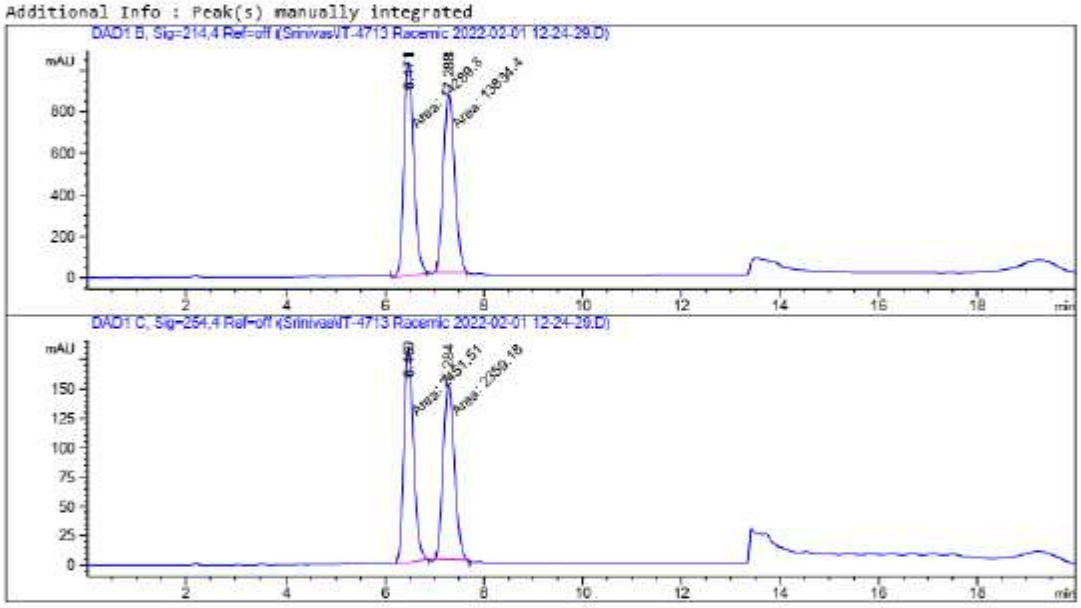

H
N
Ph
N
O=S=O
CO2Et
NO2
24b
(±)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


H
N
Ph
N
O=S=O
CO2Et
NO2
24b
(±)
HSQC
f2 (ppm)
f1 (ppm)

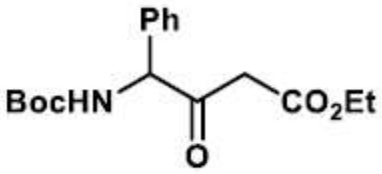


Data File C:\Chem32\1\Data\Srinivas\IT-4713 Racemic 2022-02-01 12-24-29.D
Sample Name: IT-4713 Racemic

```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : CHIRAL                        Location :   2
Injection Date  : 2/1/2022 12:31:15 PM
                                                Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\METHODS\IA_nHep_Ethanol_Custom.M
Last changed    : 2/1/2022 12:30:24 PM by SYSTEM
Analysis Method : C:\Chem32\1\Methods\Method_develop_Chiral_IF_nHep_Ethanol.M
Last changed    : 3/18/2021 4:41:28 PM by SYSTEM
Method Info     : method development for normal phase chiral separations ChiralPak IF with n-
                  Heptane and Ethanol

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




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

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


Signal 1: DAD1 B, Sig=214,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.471 MM    0.2324 1.42898e4  1024.70313  50.8096
   2   7.288 MM    0.2676 1.38344e4   861.54517  49.1904
```



Data File C:\Chem32\1\Data\Srinivas\IT-4713 Racemic 2022-02-01 12-24-29.D
Sample Name: IT-4713 Racemic

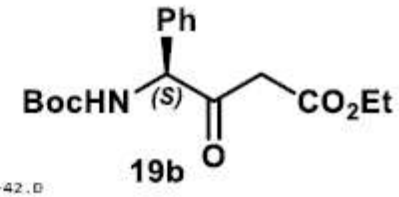
Totals : 2.81242e4 1886.24829

Signal 2: DAD1 C, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.469	MM	0.2270	2451.50684	180.02174	50.9596
2	7.284	MM	0.2650	2359.18286	148.37228	49.0404

Totals : 4810.68970 328.39403

*** End of Report ***

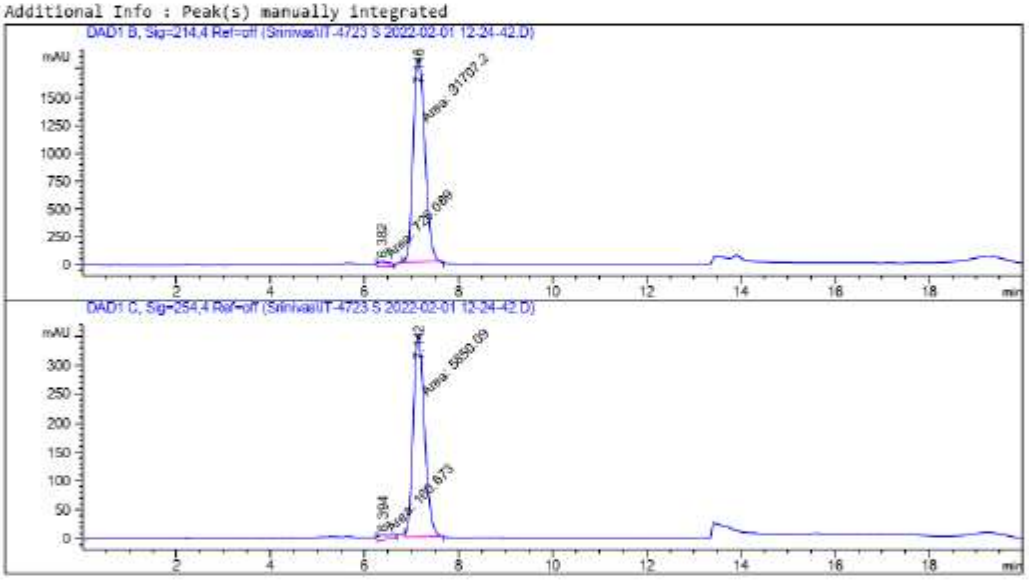


19b

Data File C:\Chem32\1\Data\Srinivas\IT-4723 S 2022-02-01 12-24-42.D
Sample Name: IT-4723 S

Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : CHIRAL Location : 3
Injection Date : 2/1/2022 1:13:39 PM
Inj Volume : 5.000 µl
Acq. Method : C:\CHEM32\1\METHODS\IA_nHep_Ethanol_Custom.M
Last changed : 2/1/2022 12:30:24 PM by SYSTEM
Analysis Method : C:\Chem32\1\Methods\Method_develop_Chiral_IF_nHep_Ethanol.M
Last changed : 3/18/2021 4:41:28 PM by SYSTEM
Method Info : method development for normal phase chiral separations ChiralPak IF with n-Heptane and Ethanol

Additional Info : Peak(s) manually integrated

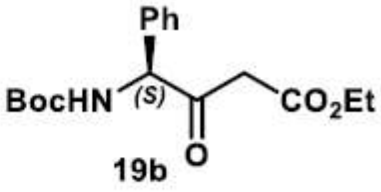


Area Percent Report

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

Signal 1: DAD1 B, Sig=214,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.382	MM	0.2856	726.08911	42.37216	2.2387
2	7.146	MM	0.2922	3.17072e4	1808.41626	97.7613



19b

Data File C:\Chem32\1\Data\Srinivas\IT-4723 S 2022-02-01 12-24-42.D
Sample Name: IT-4723 S

Totals : 3.24333e4 1850.78842

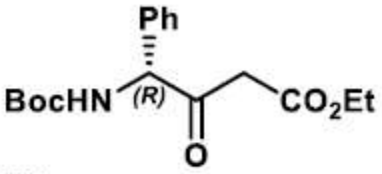
Signal 2: DAD1 C, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.394	MM	0.3181	160.67343	8.41785	2.6731
2	7.142	MM	0.2792	5850.09180	349.22180	97.3269

Totals : 6010.76523 357.63965

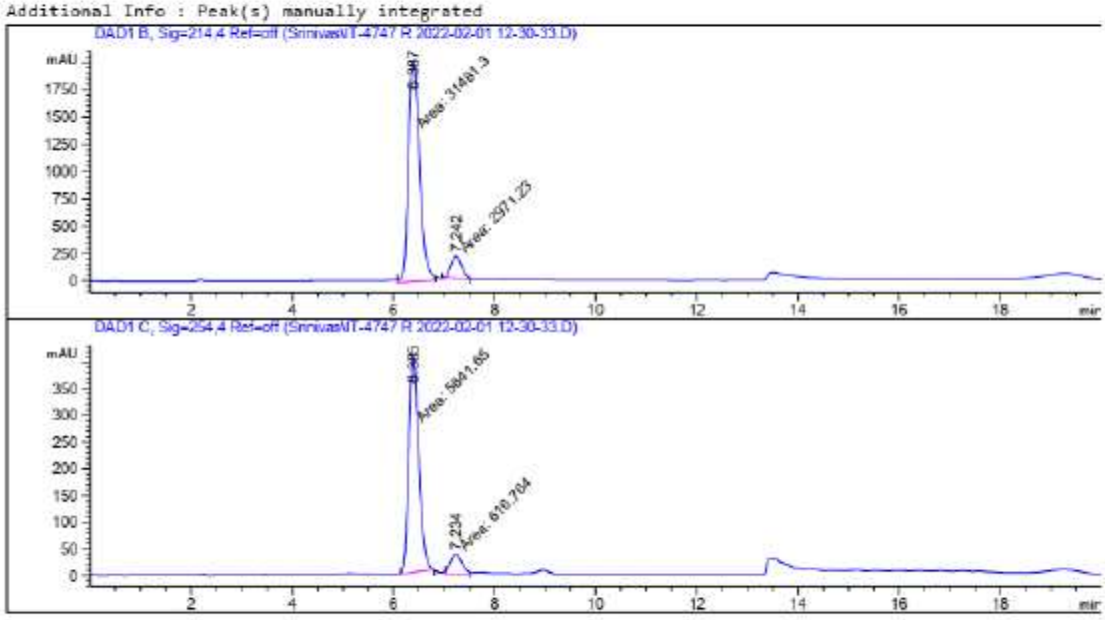
*** End of Report ***

Data File C:\Chem32\1\Data\Srinivas\IT-4747 R 2022-02-01 12-30-33.D
Sample Name: IT-4747 R



Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : CHIRAL Location : 4
Injection Date : 2/1/2022 1:56:02 PM
Inj Volume : 5.000 µl
Acq. Method : C:\CHEM32\1\METHODS\IA_nHep_Ethanol_Custom.M
Last changed : 2/1/2022 12:30:24 PM by SYSTEM
Analysis Method : C:\Chem32\1\Methods\Method_develop_Chiral_IF_nHep_Ethanol.M
Last changed : 3/18/2021 4:41:28 PM by SYSTEM
Method Info : method development for normal phase chiral separations ChiralPak IF with n-Heptane and Ethanol

Additional Info : Peak(s) manually integrated

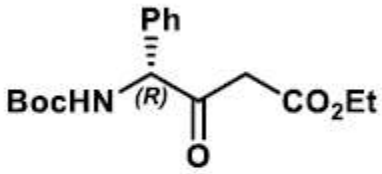


Area Percent Report

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

Signal 1: DAD1 B, Sig=214,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.387	MM	0.2606	3.14813e4	2013.11328	91.3759
2	7.242	MM	0.2471	2971.23193	200.36804	8.6241



Data File C:\Chem32\1\Data\Srinivas\IT-4747 R 2022-02-01 12-30-33.D
Sample Name: IT-4747 R

Totals : 3.44526e4 2213.48132

Signal 2: DAD1 C, Sig=254,4 Ref=off

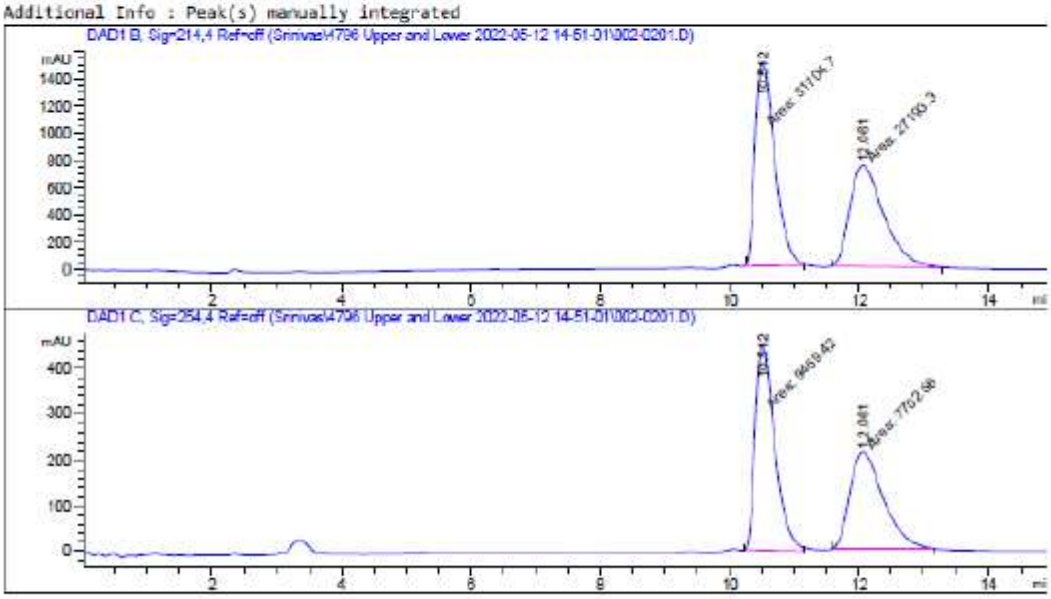
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.385	MM	0.2315	5641.65381	406.22049	90.1451
2	7.234	MM	0.2674	616.76367	38.43676	9.8549

Totals : 6258.41748 444.65725

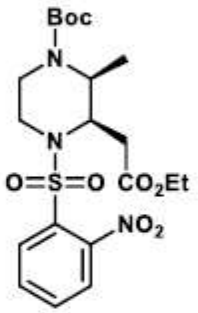
*** End of Report ***

Data File C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\002-0201.D
Sample Name: Upper Racemic

```
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : CHIRAL                           Location :   2
Injection Date  : 5/12/2022 3:09:11 PM                 Inj :   1
                                                 Inj Volume : 10.000 µl
Method          : C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\FAST_
                  Chiral_Scout_IF_nHep_Ethanol.M (Sequence Method)
Last changed    : 5/12/2022 2:51:01 PM by SYSTEM
Method Info     : Scouting method for normal phase chiral separations ChiralPak IF with n-
                  Heptane and Ethanol

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




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.512 MM    0.3518 3.17047e4  1501.98840  53.8298
   2  12.061 MM    0.6105 2.71933e4   742.32043  46.1702

Totals :                  5.88980e4  2244.30884
```



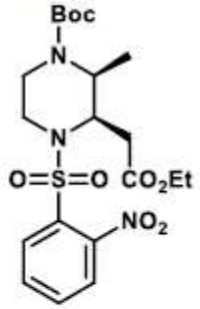
(±)

Data File C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\002-0201.D
Sample Name: Upper Racemic

Signal 2: DAD1 C, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.512	MM	0.3495	9469.42383	451.55734	55.1446
2	12.061	MM	0.6012	7702.55957	213.53856	44.8554
Totals :				1.71728e4	665.09590	

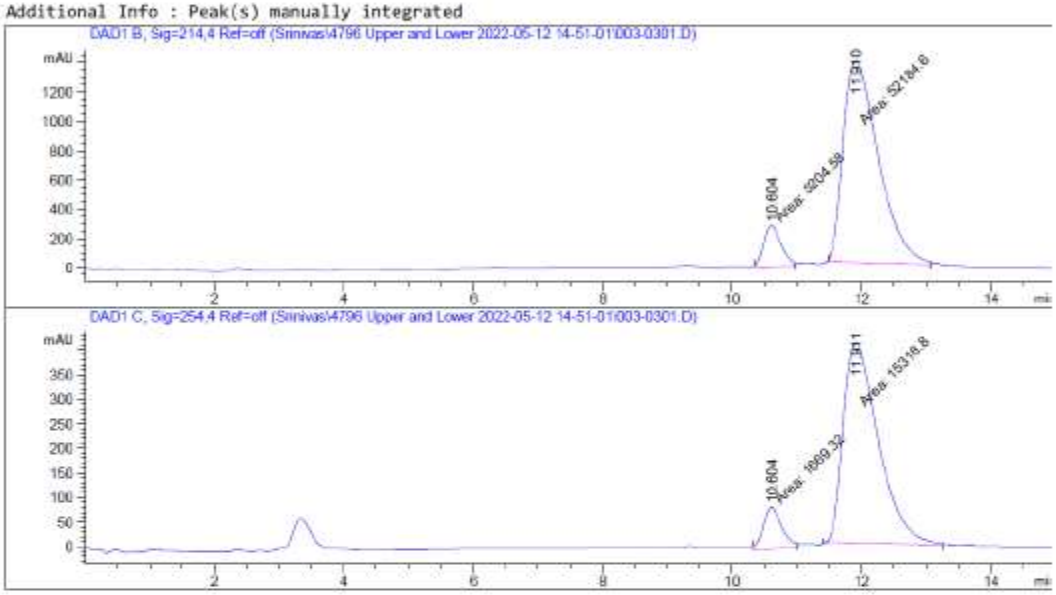
*** End of Report ***



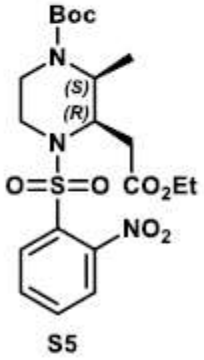
(±)

Data File C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\003-0301.D
Sample Name: 4796 Upper

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   3
Acq. Instrument : CHIRAL                           Location :   3
Injection Date  : 5/12/2022 3:25:22 PM                 Inj :   1
                                                 Inj Volume : 10.000 µl
Method          : C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\FAST_
                  Chiral_Scout_IF_nHep_Ethanol.M (Sequence Method)
Last changed    : 5/12/2022 2:51:01 PM by SYSTEM
Method Info     : Scouting method for normal phase chiral separations ChiralPak IF with n-
                  Heptane and Ethanol

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




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

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.604 MM    0.3087 5204.57568  280.98712   9.0689
   2  11.910 MM    0.6312 5.21846e4  1377.94214  90.9311

Totals :                  5.73891e4  1658.92926
```



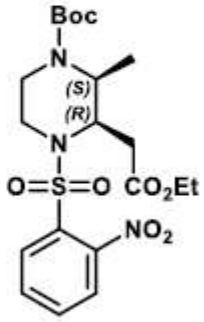
S5

Data File C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\003-0301.D
Sample Name: 4796 Upper

Signal 2: DAD1 C, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.604	MM	0.3263	1669.32361	85.27359	9.8264
2	11.911	MM	0.6264	1.5318804	407.59848	90.1736
Totals :				1.69881e4	492.87207	

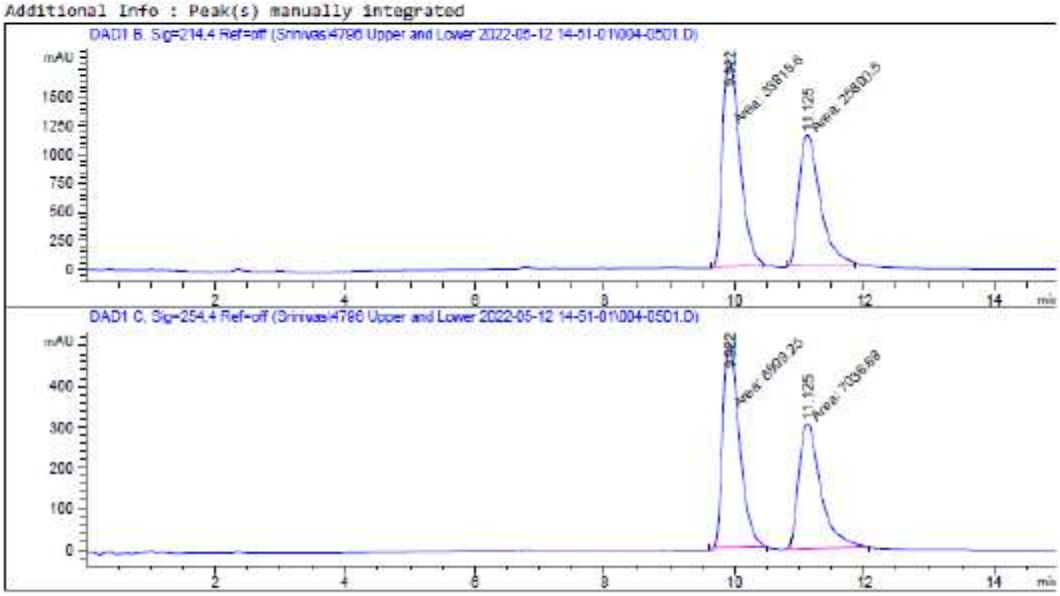
*** End of Report ***



S5

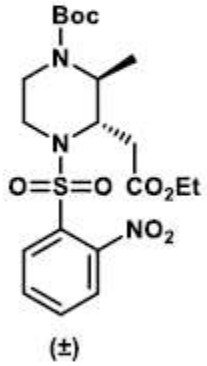
Data File C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\004-0501.D
Sample Name: Lower Racemic

```
Acq. Operator   : SYSTEM                          Seq. Line :   5
Acq. Instrument : CHIRAL                          Location :   4
Injection Date  : 5/12/2022 3:57:49 PM               Inj :   1
                                                  Inj Volume : 10.000 µl
Method          : C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\FAST_
                  Chiral_Scout_IF_nHep_Ethanol.M (Sequence Method)
Last changed    : 5/12/2022 2:51:01 PM by SYSTEM
Method Info     : Scouting method for normal phase chiral separations ChiralPak IF with n-
                  Heptane and Ethanol

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



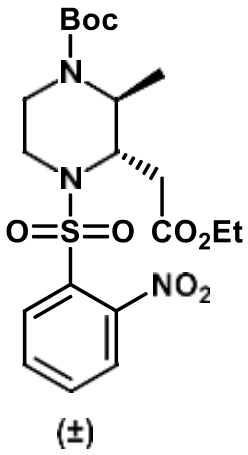
Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.922 MM    0.3119 3.38156e4  1806.72754  56.7222
   2  11.125 MM    0.3794 2.58005e4  1133.49927  43.2778

Totals :                 5.96161e4  2940.22681
```



(±)

Data File C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\004-0501.D
Sample Name: Lower Racemic



(±)

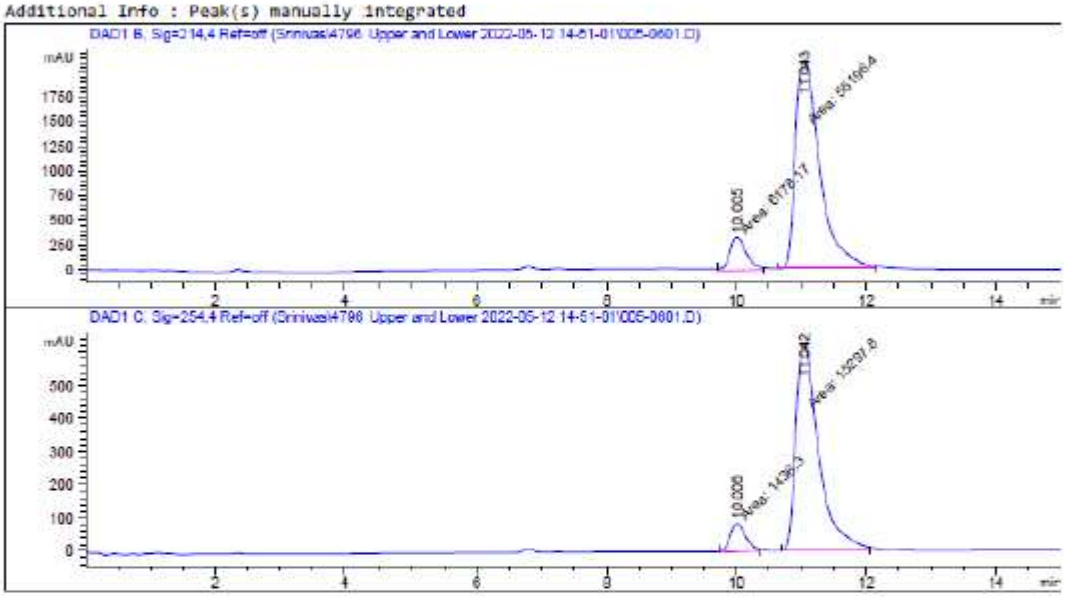
Signal 2: DAD1 C, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.922	MM	0.3000	8999.25293	499.96991	56.1193
2	11.125	MM	0.3837	7036.67822	305.66632	43.8807
Totals :				1.60359e4	805.63623	

*** End of Report ***

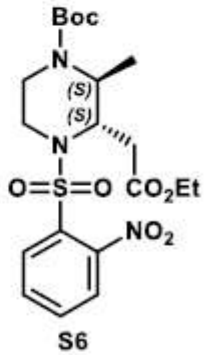
Data File C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\005-0601.D
Sample Name: 4796 Lower

```
Acq. Operator   : SYSTEM                          Seq. Line :   6
Acq. Instrument : CHIRAL                           Location :   5
Injection Date  : 5/12/2022 4:14:00 PM                 Inj :   1
                                                 Inj Volume : 10.000 µl
Method          : C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\FAST_
                  Chiral_Scout_IF_nHep_Ethanol.M (Sequence Method)
Last changed    : 5/12/2022 2:51:01 PM by SYSTEM
Method Info     : Scouting method for normal phase chiral separations ChiralPak IF with n-
                  Heptane and Ethanol

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



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.005 MM    0.3009 6178.16748  342.18903  10.0663
   2  11.043 MM    0.4382 5.51964e4  2099.14819  89.9337

Totals :                  6.13746e4  2441.33722
```



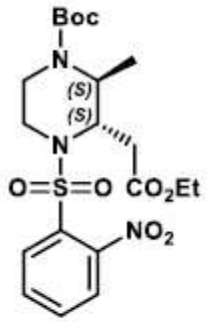
S6

Data File C:\Chem32\1\Data\Srinivas\4796 Upper and Lower 2022-05-12 14-51-01\005-0601.D
Sample Name: 4796 Lower

Signal 2: DAD1 C, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.006	MM	0.2793	1436.29565	85.71114	8.5830
2	11.042	MM	0.4102	1.52978e4	621.56360	91.4170
Totals :				1.67341e4	707.27473	

*** End of Report ***



S6