Thioimidazoline based compounds reverse glucocorticoid resistance in human acute lymphoblastic leukemia xenografts

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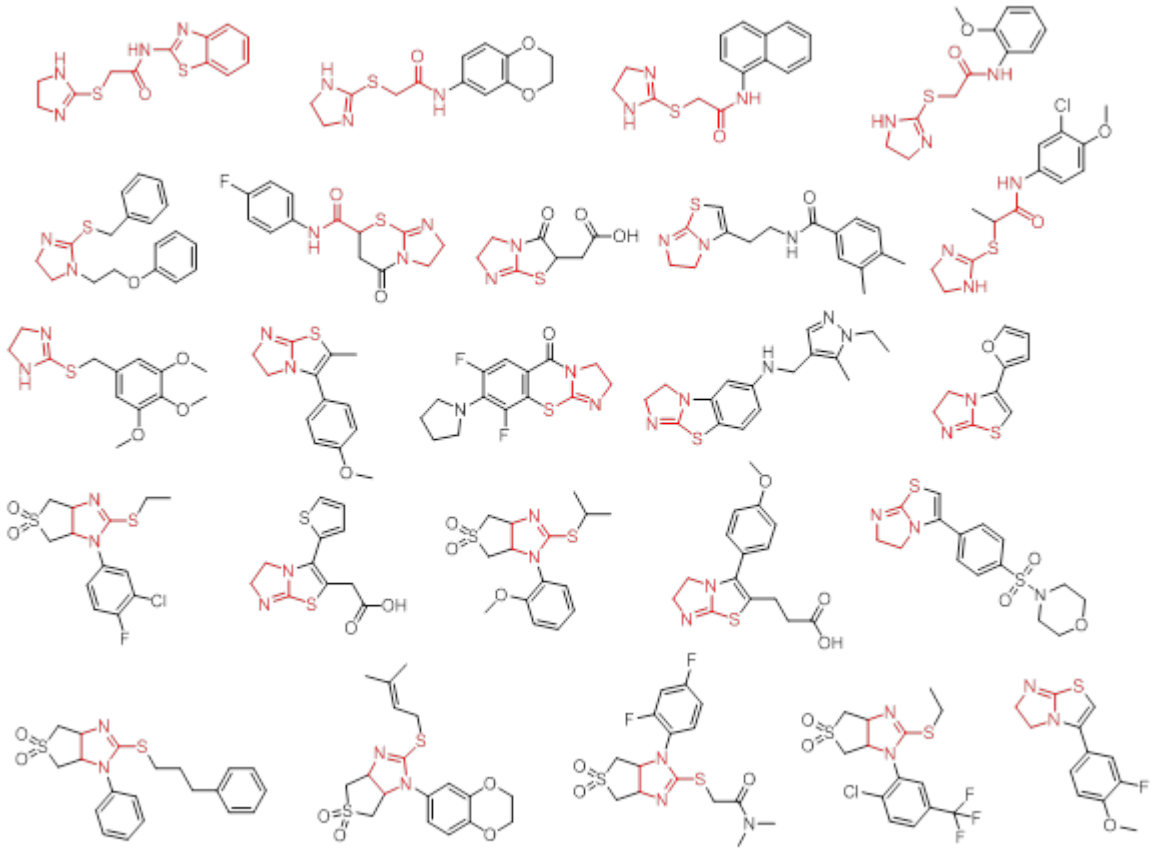
BIOLOGICAL SUPPLEMENTARY MATERIAL

Biological Data

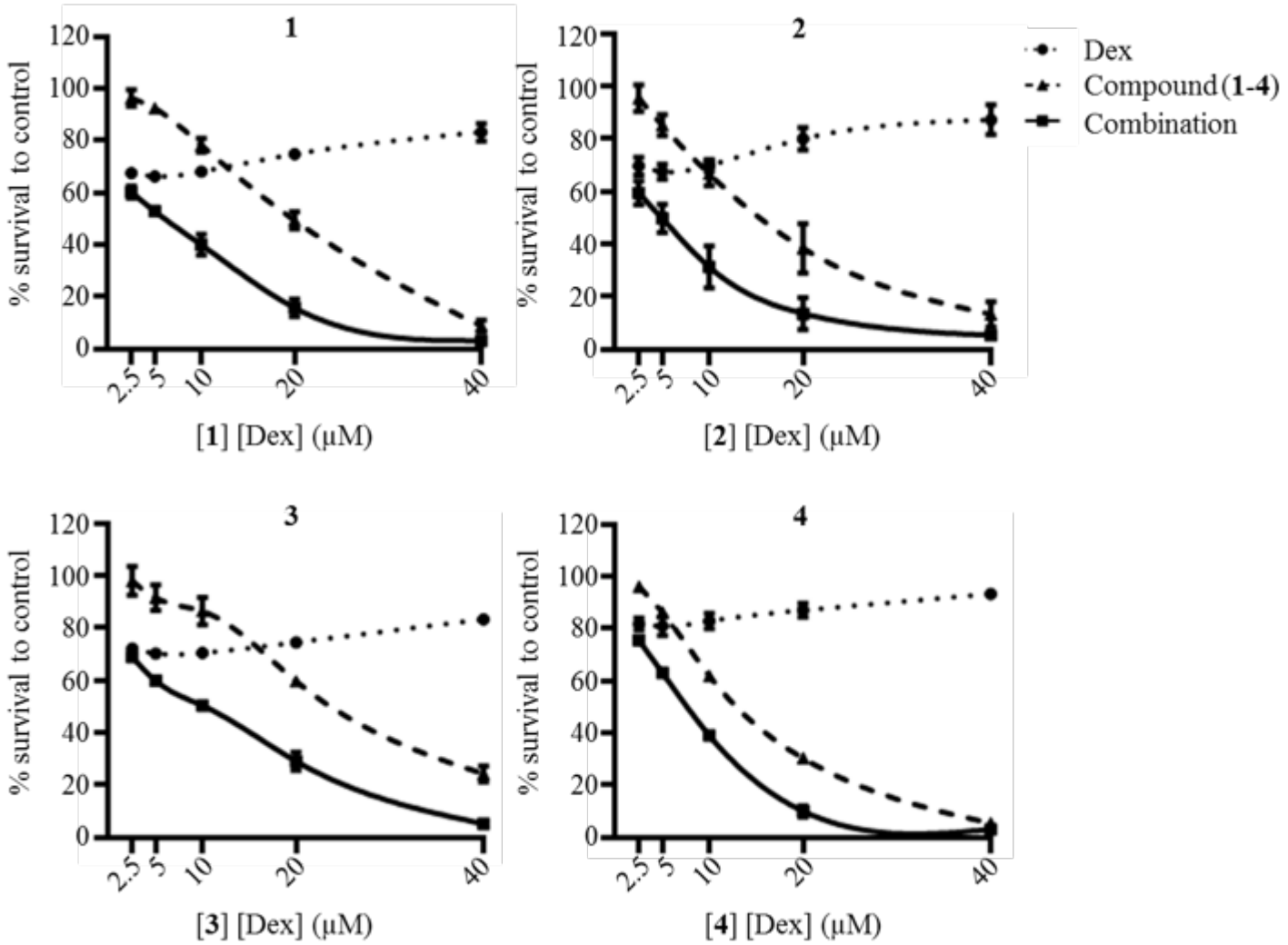
Supple. Figure 1

Structures of the 24 thioimidazoline containing compounds identified in the HTS assay that are not dexamethasone sensitizers. Highlighted in red is the thioimidazoline substructure common to the four lead dexamethasone sensitizers (compounds **1**-**4**). Also highlighted in red are substructures common to at least one of the four lead dexamethasone sensitizers (compounds **1**-**4**).



Supple. Figure 2

Ex vivo efficacy of compounds (**1**-**4**) in combination with dexamethasone against ALL xenograft cells. ALL-19 cells were exposed to compound (**1**-**4**), dexamethasone (Dex), or both in combination at a 1:1 fixed-ratio of concentrations for 48 h. Cell viability was assessed by Alamar Blue assay. Each data point represents the mean ± SEM of three independent experiments.



Supple. Table 1

ALL-19 xenograft cells were exposed to compound (**1**-**4**), dexamethasone, or both in combination at a 1:1 fixed-ratio of concentrations for 48 h. Cell sensitivity was then assessed by Alamar Blue assay. Deviation from Bliss-additivity was calculated at each tested dose, where synery is defined as a positive deviation, additive effect as no deviation, and antagonism as a negative deviation.

		Deviation from Bliss-Additivity at each tested concentration;						
Cmpd	Glucocorticoid	2.5 µM	5 µM	10 µM	20 µM	40 µM	Median	Comb. Effect
1	dexamethasone	0.05	0.08	0.13	**0.21**	0.04	0.08	synergy
2	dexamethasone	0.07	0.08	0.15	0.17	0.06	0.08	synergy
3	dexamethasone	0.02	0.04	0.11	0.15	0.15	0.11	synergy
4	dexamethasone	0.03	0.07	0.12	0.16	0.02	0.07	synergy

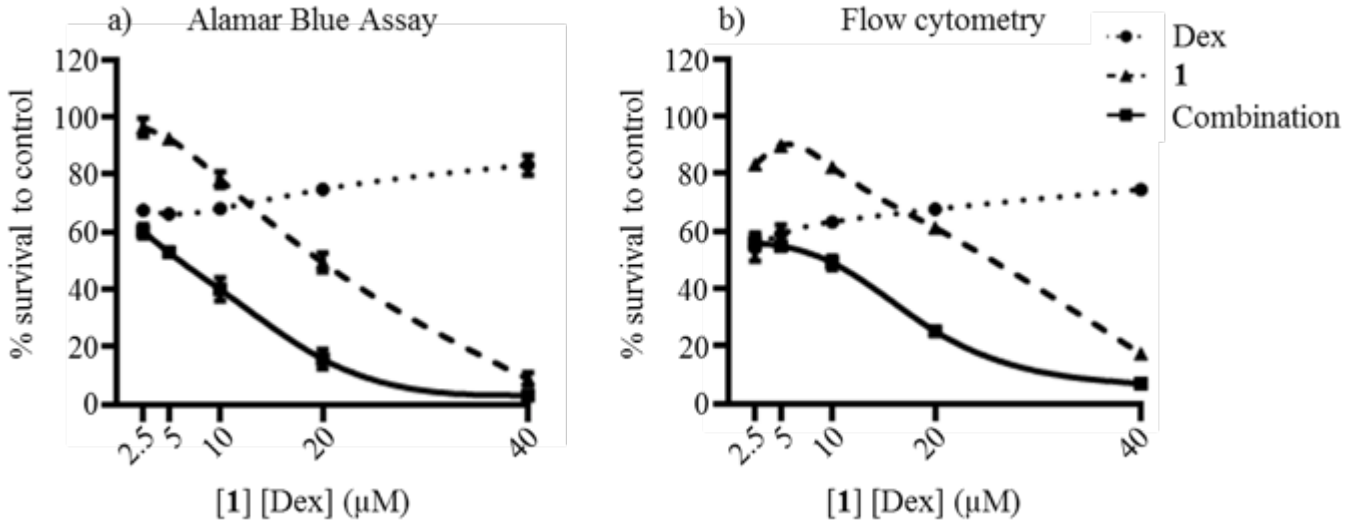
Supple. Table 2

Combination effects of **1** and glucocorticoids *ex vivo* against ALL-19 xenograft cells. ALL-19 xenograft cells were exposed to **1**, glucocorticoid, or both in combination at a fixed-ratio of concentrations for 48 h. Cell viability was assessed by Alamar Blue assay. Deviation from Bliss-additivity was calculated at each tested dose, where synery is defined as a positive deviation, additive effect as no deviation, and antagonism as a negative deviation.

		Deviation from Bliss-Additivity at each tested concentration;						
Cmpd	Glucocorticoid	2.5 µM	5 µM	10 µM	20 µM	40 µM	Median	Comb. Effect
1	dexamethasone	0.05	0.08	0.13	0.21	0.04	0.08	synergy
1	prednisolone	0.06	0.11	0.15	0.29	0.20	0.15	synergy

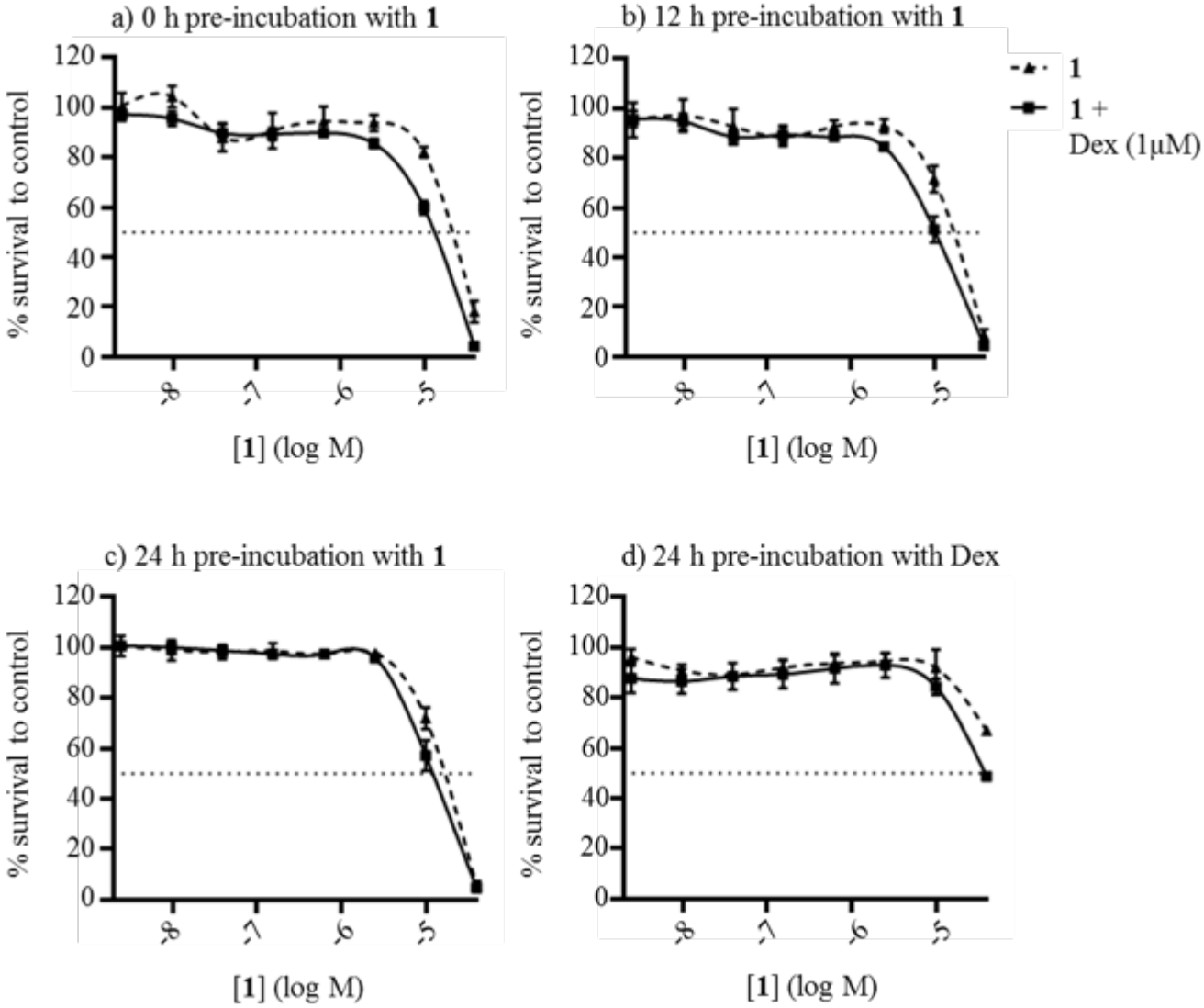
Supple. Figure 3

Synergistic antileukemic effects of **1** and dexamethasone *ex vivo* against ALL-19 xenograft cells. **(a)** ALL-19 cells were exposed to **1**, dexamethasone (Dex), or both in combination at a fixed-ratio of concentrations for 48 h. Cell viability was assessed by Alamar Blue assay. **(b)** ALL-19 cells were exposed to **1**, Dex, or both in combination at a fixed-ratio of concentrations for 48 h. Cell viability was assessed by flow cytometry, a direct measure of cell viability. Each data point represents the mean ± SEM of three independent experiments.



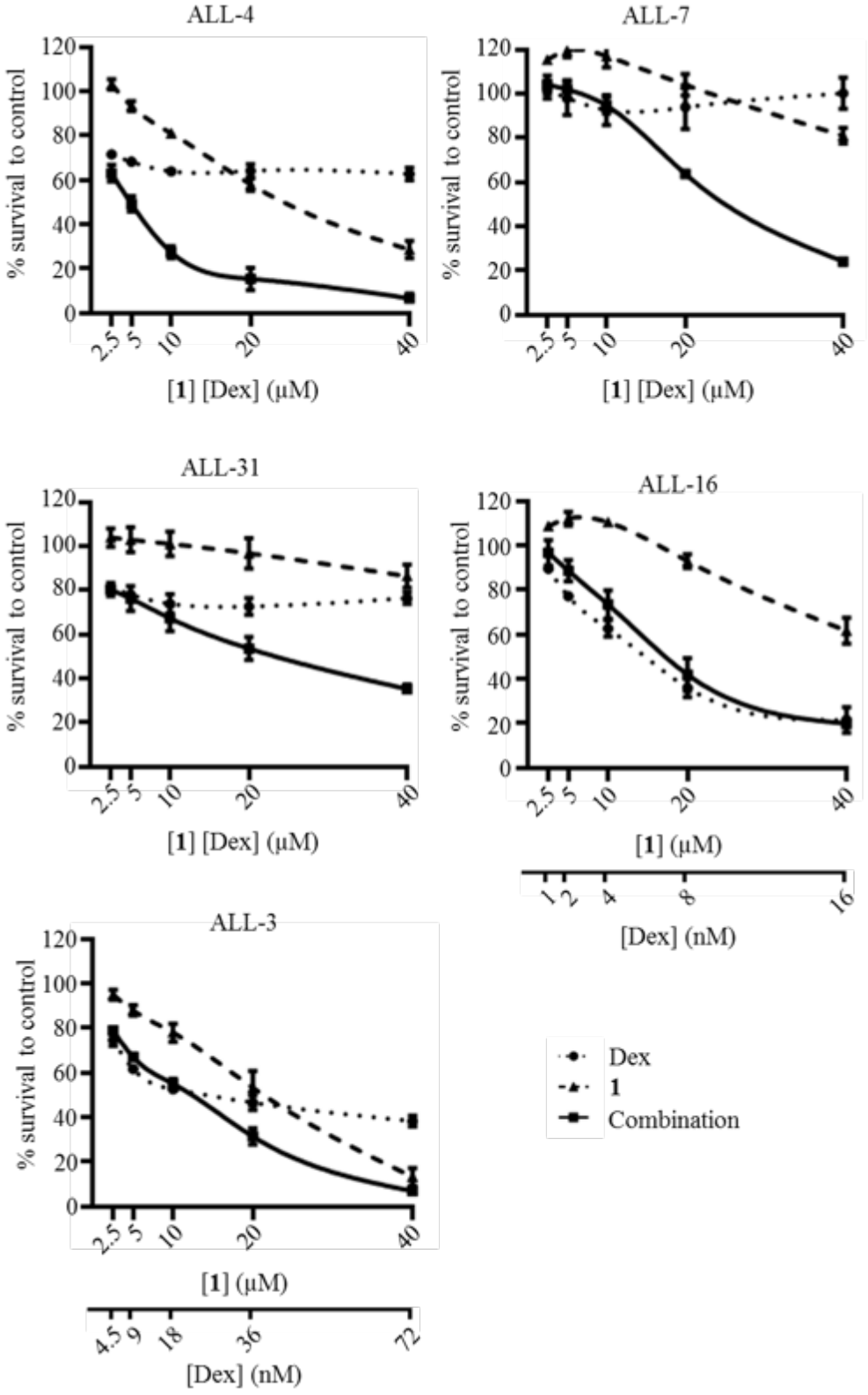
Supple. Figure 4

Order of addition experiments with **1** and dexamethasone on ALL-19. **(a)** ALL-19 cells were treated simultaneously with a dose-response of **1** ± 1 µM dexamethasone for 48 h. **(b)** ALL-19 cells were pre-treated for 12 h with **1** before the addition of ± 1 µM dexamethasone for 48 h. **(c)** ALL-19 cells were pre-treated for 24 h with **1** before the addition of ± 1 µM dexamethasone for 48 h. **(d)** ALL-19 cells were pre-treated for 24 h with ± 1 µM dexamethasone before the addition of **1** for 24 h. For each treatment cells were exposed to dexamethasone for 48 h. The cell viability was calculated relative to dexamethasone treated controls (combination) or DMSO treated controls (**1** alone). Each data point represents the mean ± SEM of 3 independent experiments.



Supple. Figure 5

Ex vivo efficacy of **1** in combination with dexamethasone against ALL xenograft cells (ALL-4, ALL-7, ALL-31, ALL-16 and ALL-3). Xenograft cells were exposed to **1**, dexamethasone (Dex), or both in combination at a fixed-ratio of concentrations for 48 h. Cell viability was assessed by Alamar Blue assay. Each data point represents the mean ± SEM of three independent experiments.



Supple. Table 3

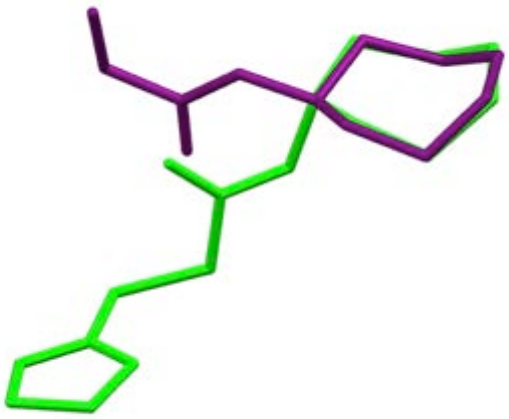
Xenograft cells were exposed to **1**, dexamethasone, or both in combination at a fixed-ratio of concentrations for 48 h. Cell sensitivity was then assessed by Alamar Blue assay. The ratio of **1** to dexamaethasone (**1**/Dex ratio) is determined from single agent assays. Deviation from Bliss-additivity was calculated at each tested dose, where synery is defined as a positive deviation, additive effect as no deviation, and antagonism as a negative deviation.

Where, BCP-ALL, B-cell precursor ALL; Ph^+, Philadelphia chromosome-positive; Bi, Biphenotypic; MLL, Mixed Lineage Leukemia

Xenograft	Subtype	Dex stratification	**1**/Dex ratio	Deviation from Bliss-Additivity at each tested concentration of **1**; 2.5 µM	5 µM	10 µM	20 µM	40 µM	Median	Comb. Effect
ALL-4	BCP-ALL, Ph^+	resistant	1:1	0.09	0.14	0.24	0.22	0.11	0.14	synergy
ALL-7	BCP-ALL, Bi	resistant	1:1	-	-0.02	-0.02	0.30	0.52	0.14	synergy
ALL-31	T-cell ALL	resistant	1:1	0.01	0.02	0.07	0.17	0.31	0.07	synergy
ALL-16	T-cell ALL	sensitive	2500:1	-0.07	-0.12	-0.11	-0.08	-0.07	-0.08	antagonism
ALL-3	MLL	sensitive	5000:9	-0.08	-0.13	-0.14	-0.07	-0.02	-0.08	antagonism

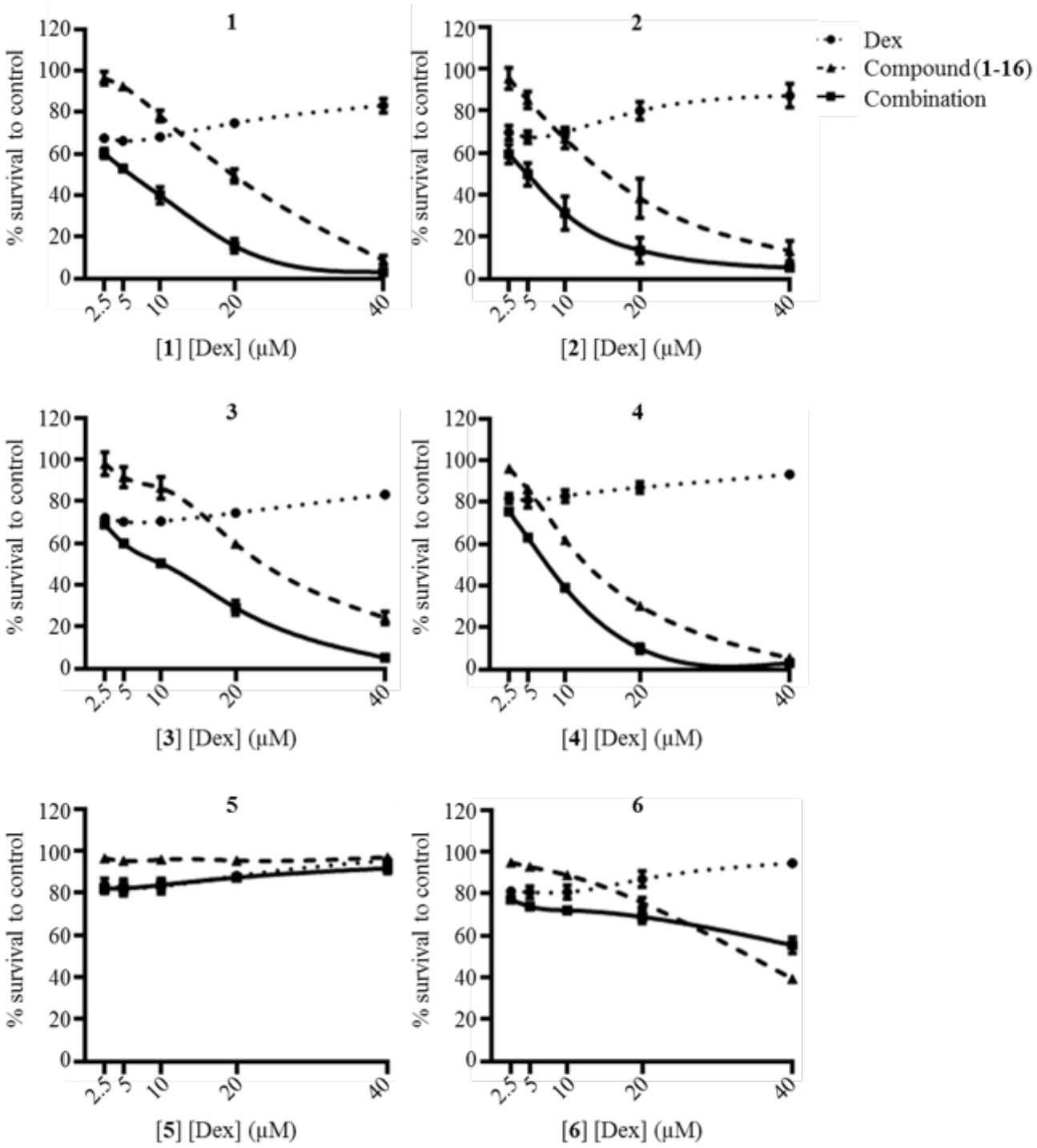
Supple. Figure 6

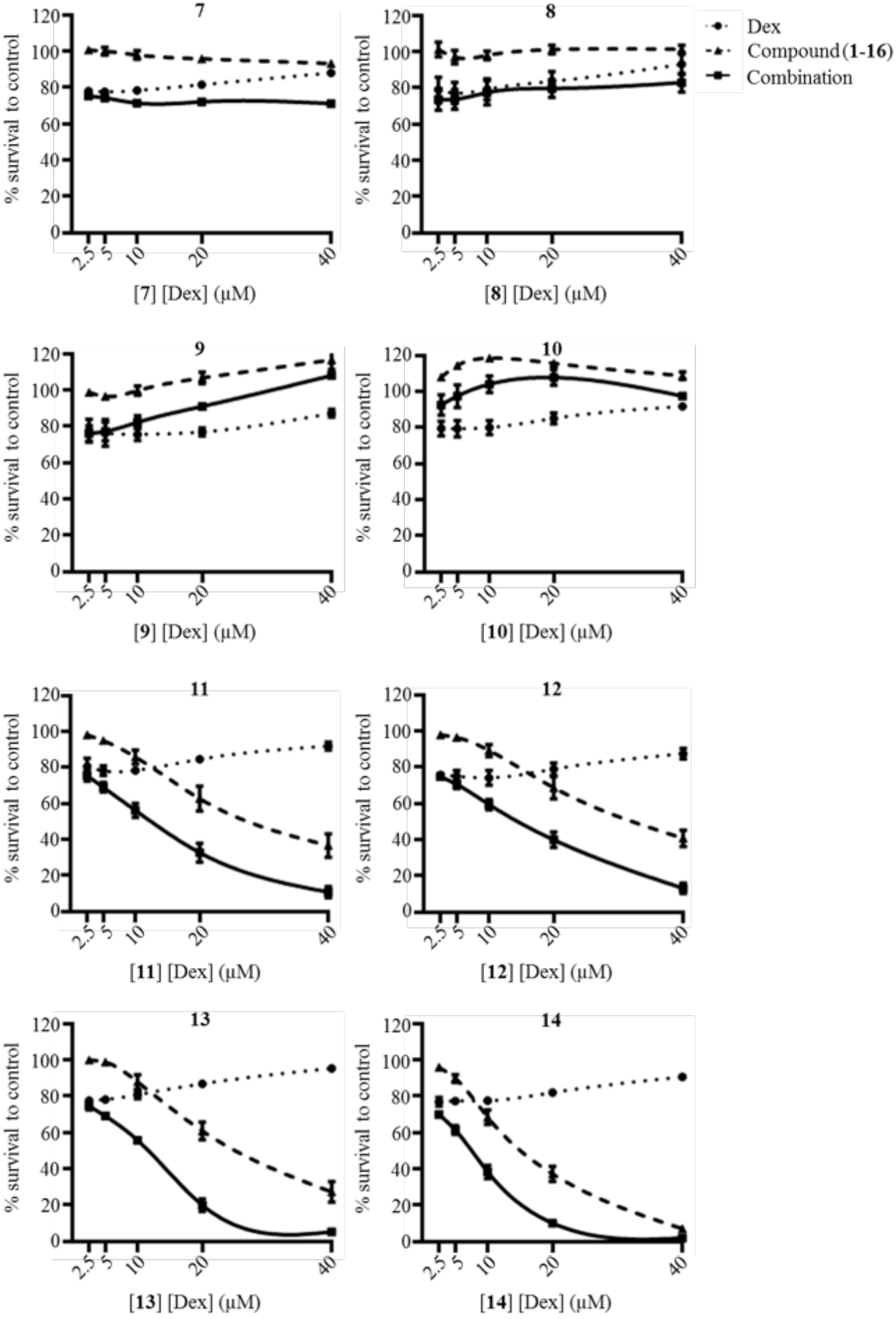
Crystal structures of compound **1** (green) and compound **6** (purple) with cycloheptane rings overlaid.

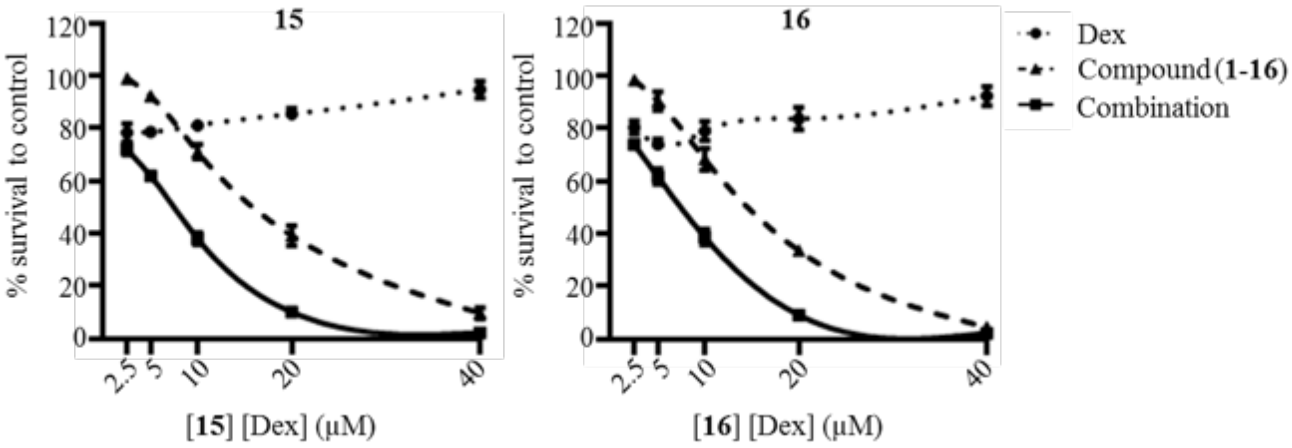


Supple. Figure 7

Ex vivo efficacy of compounds (**1-16**) in combination with dexamethasone against ALL xenograft cells. ALL-19 cells were exposed to compound (**1-16**), dexamethasone (Dex), or both in combination at a 1:1 fixed-ratio of concentrations for 48 h. Cell viability was assessed by Alamar Blue assay. Each data point represents the mean ± SEM of three independent experiments.




7
8
9
10
11
12
13
14
% survival to control
[7] [Dex] (μM)
[8] [Dex] (μM)
[9] [Dex] (μM)
[10] [Dex] (μM)
[11] [Dex] (μM)
[12] [Dex] (μM)
[13] [Dex] (μM)
[14] [Dex] (μM)
Dex
Compound (1-16)
Combination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16
% survival to control
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100
80
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0
2.5
5
10
20
40
[15] [Dex] (μM)
[16] [Dex] (μM)
Dex
Compound (1-16)
Combination

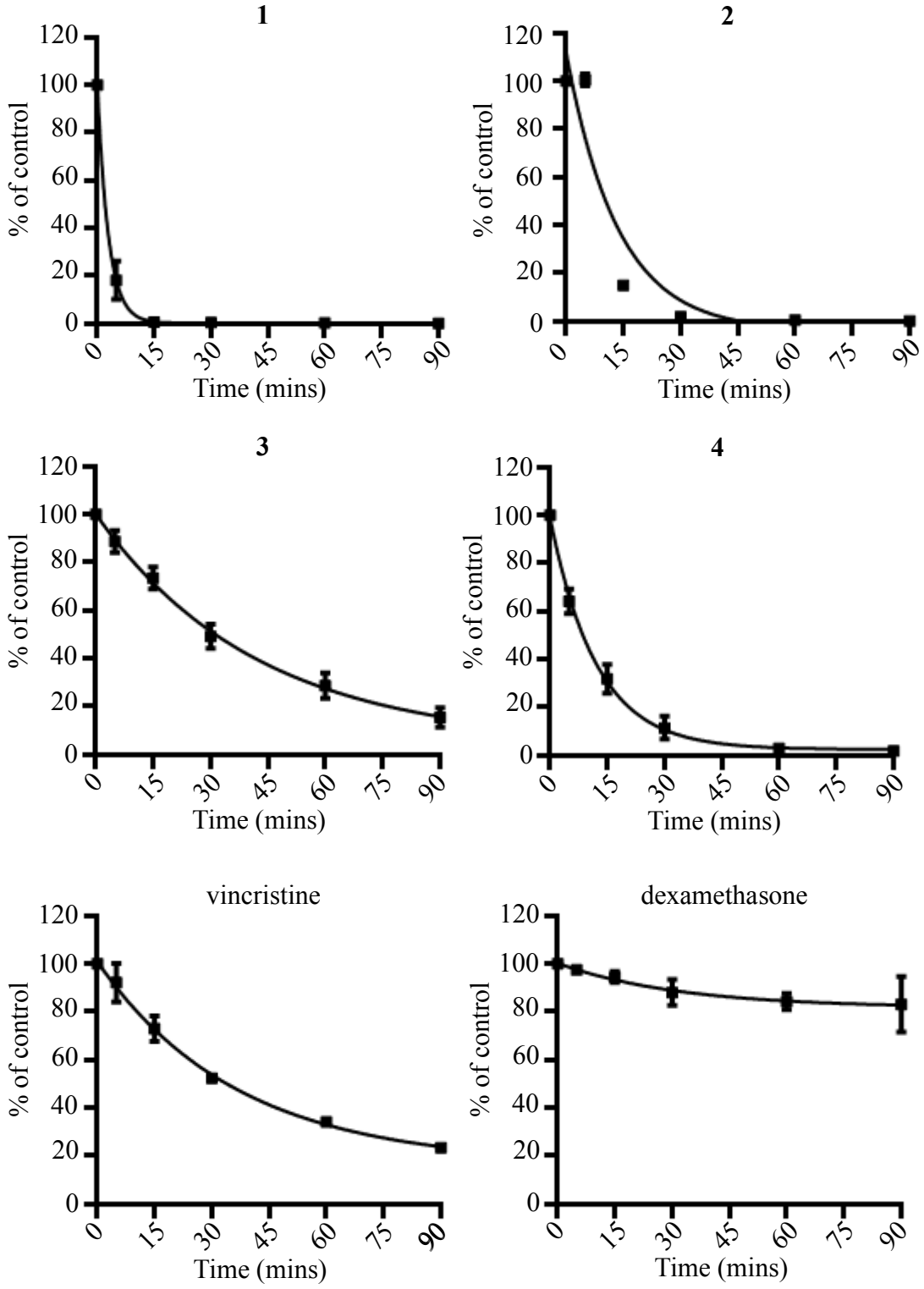
Supple. Table 4

ALL-19 xenograft cells were exposed to compound (**1-16**), dexamethasone, or both in combination at a 1:1 fixed-ratio of concentrations for 48 h. Cell sensitivity was then assessed by Alamar Blue assay. Deviation from Bliss-additivity was calculated at each tested dose, where synery is defined as a positive deviation, additive effect as no deviation, and antagonism as a negative deviation.

		Deviation from Bliss-Additivity at each tested concentration;						
Cmpd	Glucocorticoid	2.5 μM	5 μM	10 μM	20 μM	40 μM	Median	Comb. Effect
1	dexamethasone	0.05	0.08	0.13	0.21	0.04	0.08	synergy
2	dexamethasone	0.07	0.08	0.15	0.17	0.06	0.08	synergy
3	dexamethasone	0.02	0.04	0.11	0.15	0.15	0.11	synergy
4	dexamethasone	0.03	0.07	0.12	0.16	0.02	0.07	synergy
5	dexamethasone	-0.01	-0.04	-0.04	-0.03	0.01	-0.03	antagonism
6	dexamethasone	0.00	0.01	0.00	-0.03	-0.18	0.00	additive
7	dexamethasone	0.03	0.03	0.05	0.06	0.11	0.05	synergy
8	dexamethasone	0.05	0.01	0.00	0.04	0.10	0.04	synergy
9	dexamethasone	0.01	-0.04	-0.07	-0.14	-0.13	-0.07	antagonism
10	dexamethasone	-0.13	-0.18	-0.20	-0.15	-0.06	-0.15	antagonism
11	dexamethasone	0.04	0.05	0.11	0.20	0.23	0.11	synergy
12	dexamethasone	-0.01	0.02	0.07	0.14	0.23	0.07	synergy
13	dexamethasone	0.03	0.08	0.15	0.33	0.21	0.15	synergy
14	dexamethasone	0.04	0.08	0.15	0.20	0.05	0.08	synergy
15	dexamethasone	0.06	0.11	0.20	0.23	0.07	0.11	synergy
16	dexamethasone	0.05	0.05	0.15	0.19	0.02	0.05	synergy

Supple. Figure 8

Ex vivo stability of compounds in liver microsomes. Compounds were exposed to mouse liver microsomes for up to 90 mins. Each data point represents the mean ± SEM of three independent experiments.



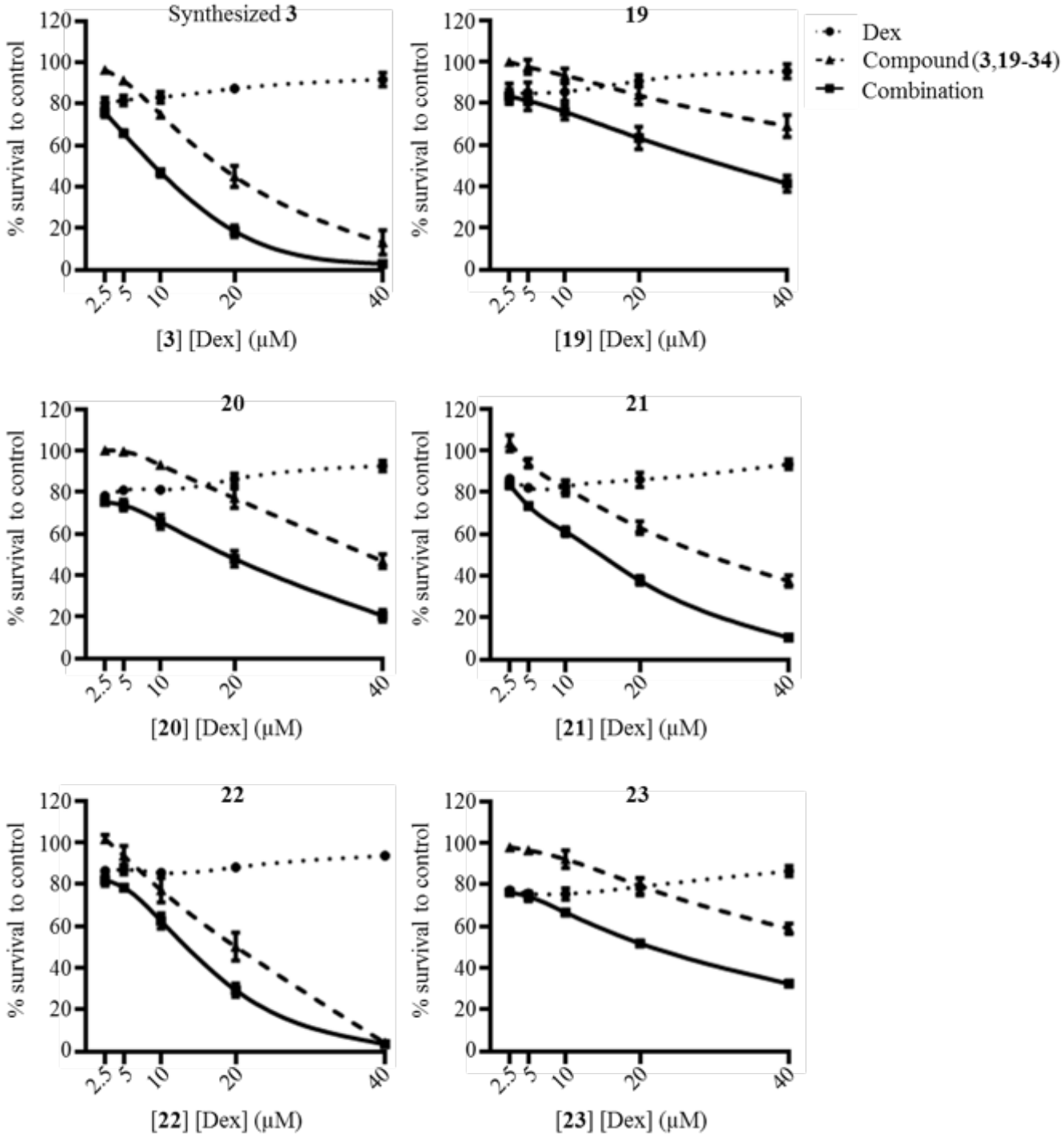
Supple. Table 5

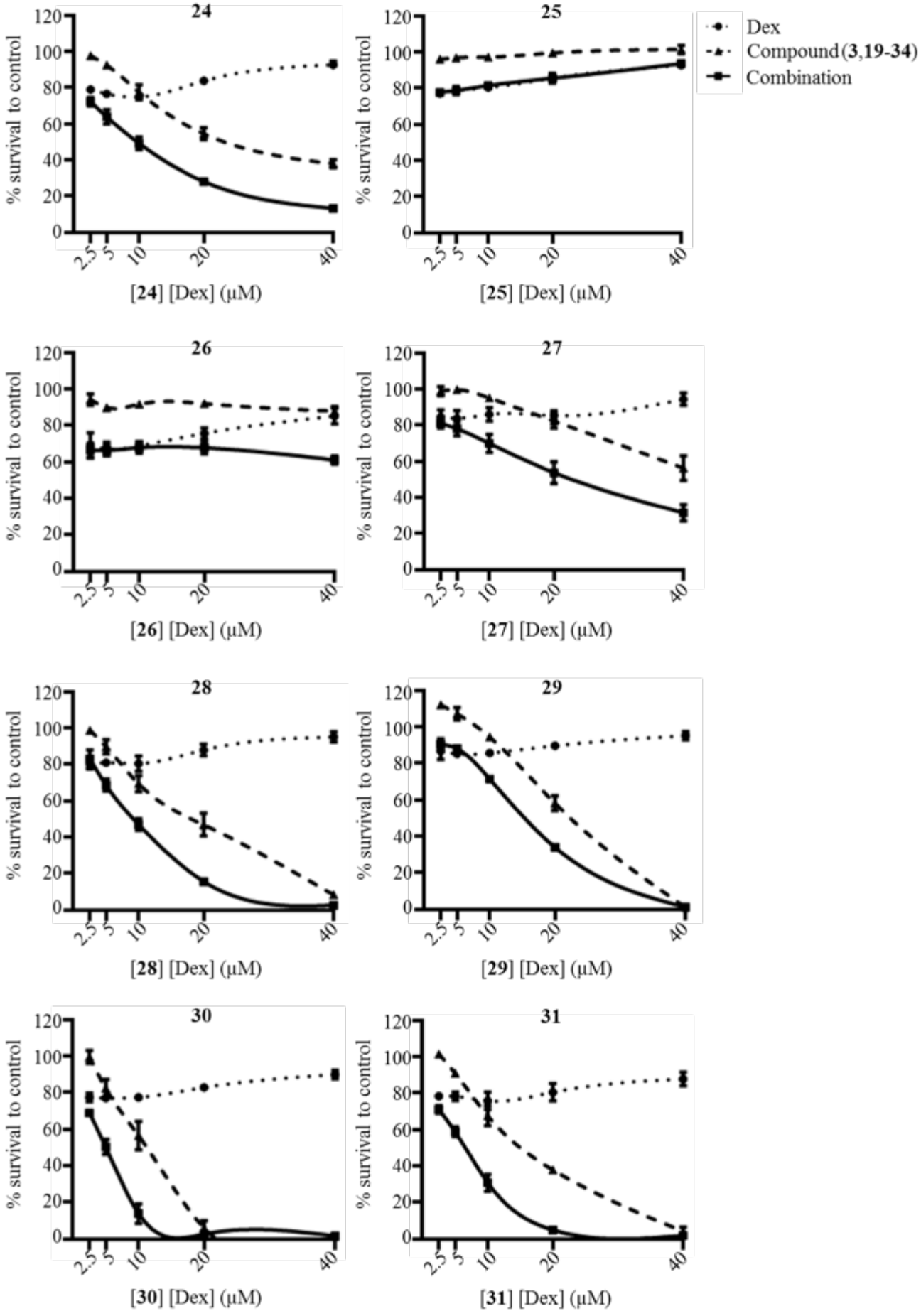
Ex vivo stability of compounds in liver microsomes. Compounds were exposed to mouse liver microsomes for up to 90 mins. Half-life values were calculated from one phase exponential decay curves.

	1	**2**	**3**	**4**	Dex	Vincristine
Half-life (mins)	2.0	9.4	29.2	8.4	> 90	25.4

Supple. Figure 9

Ex vivo efficacy of compounds (synthesized **3**, **19-34**) in combination with dexamethasone against ALL xenograft cells. ALL-19 cells were exposed to compound (synthesized **3**, **19-34**), dexamethasone (Dex), or both in combination at a 1:1 fixed-ratio of concentrations for 48 h. Cell viability was assessed by Alamar Blue assay. Each data point represents the mean ± SEM of three independent experiments.

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29
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80
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40
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0
2.5
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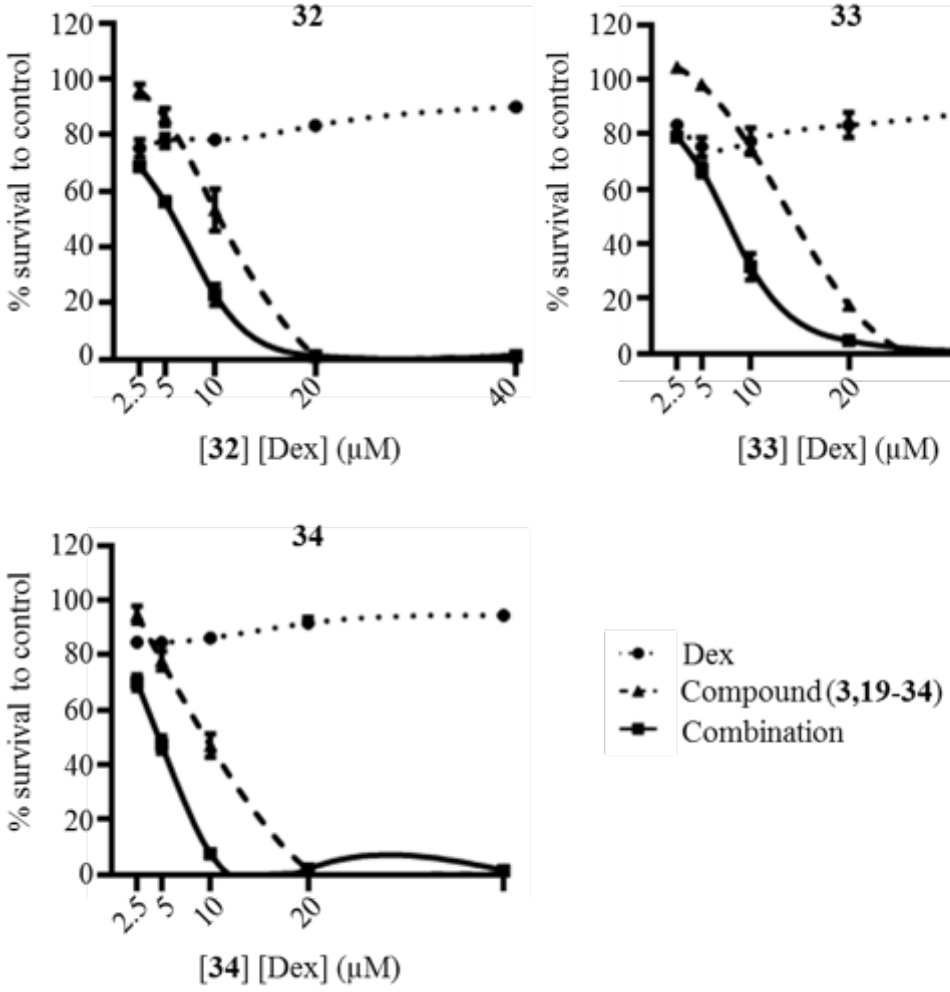
10
20
40
[24] [Dex] (μM)
[25] [Dex] (μM)
[26] [Dex] (μM)
[27] [Dex] (μM)
[28] [Dex] (μM)
[29] [Dex] (μM)
[30] [Dex] (μM)
[31] [Dex] (μM)
Dex
Compound (3,19-34)
Combination


32
33
34
% survival to control
120
100
80
60
40
20
0
2.5
5
10
20
40
[32] [Dex] (μM)
[33] [Dex] (μM)
[34] [Dex] (μM)
Dex
Compound (3,19-34)
Combination

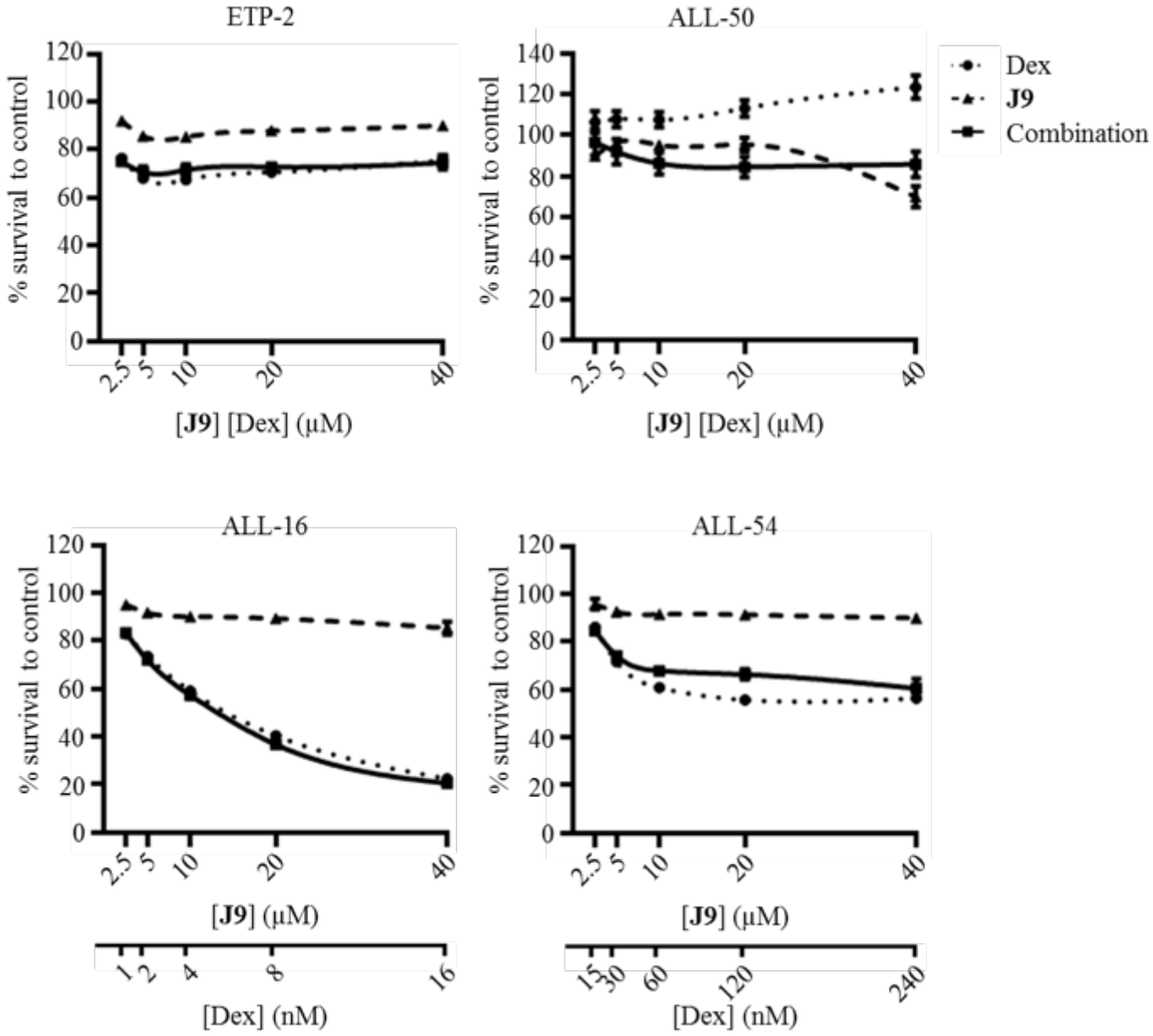
Supple. Table 6

ALL-19 xenograft cells were exposed to compound (synthesized **3**, **19**-**34**), dexamethasone, or both in combination at a 1:1 fixed-ratio of concentrations for 48 h. Cell sensitivity was then assessed by Alamar Blue assay. Deviation from Bliss-additivity was calculated at each tested dose, where synery is defined as a positive deviation, additive effect as no deviation, and antagonism as a negative deviation.

		Deviation from Bliss-Additivity at each tested concentration;						
Cmpd	Glucocorticoid	2.5 μM	5 μM	10 μM	20 μM	40 μM	Median	Comb. Effect
3	dexamethasone	0.02	0.09	0.15	0.21	0.09	0.09	synergy
19	dexamethasone	0.01	0.01	0.04	0.13	0.25	0.04	synergy
20	dexamethasone	0.03	0.07	0.10	0.19	0.23	0.10	synergy
21	dexamethasone	0.03	0.04	0.07	0.17	0.25	0.07	synergy
22	dexamethasone	0.05	0.03	0.04	0.15	0.00	0.04	synergy
23	dexamethasone	-0.01	-0.01	0.03	0.11	0.18	0.03	synergy
24	dexamethasone	0.05	0.07	0.09	0.18	0.22	0.09	synergy
25	dexamethasone	-0.04	-0.03	-0.03	0.00	-0.01	-0.02	antagonism
26	dexamethasone	-0.01	-0.06	-0.05	0.02	0.14	-0.01	antagonism
27	dexamethasone	-0.01	-0.01	0.03	0.11	0.18	0.03	synergy
28	dexamethasone	-0.04	0.04	0.09	0.26	0.05	0.05	synergy
29	dexamethasone	-0.04	-0.03	0.09	0.18	0.00	0.00	additive
30	dexamethasone	0.08	0.13	0.30	0.02	0.00	0.08	synergy
31	dexamethasone	0.07	0.12	0.20	0.26	0.02	0.12	synergy
32	dexamethasone	0.10	0.19	0.33	0.00	0.00	0.10	synergy
33	dexamethasone	0.04	0.07	0.26	0.10	0.00	0.07	synergy
34	dexamethasone	0.03	0.11	0.19	0.00	0.00	0.03	synergy

Supple. Figure 10

Ex vivo efficacy of **J9** in combination with dexamethasone against ALL xenograft cells (ALL-50, ETP-2, ALL-16 and ALL-54). Xenograft cells were exposed to **J9**, dexamethasone (Dex), or both in combination at a fixed-ratio of concentrations for 48 h. Cell viability was assessed by Alamar Blue assay. Each data point represents the mean ± SEM of three independent experiments.

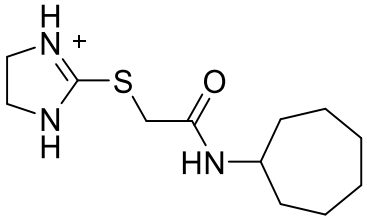


Supple. Table 7

Xenograft cells were exposed to **J9**, dexamethasone, or both in combination at a fixed-ratio of concentrations for 48 h. Cell sensitivity was then assessed by Alamar Blue assay. The ratio of **J9** to dexamaethasone (**J9**/Dex ratio) is determined from single agent assays. Deviation from Bliss-additivity was calculated at each tested dose, where synery is defined as a positive deviation, additive effect as no deviation, and antagonism as a negative deviation.

Xenograft	Subtype	Dex stratification	**J9**/Dex ratio	Deviation from Bliss-Additivity at each tested concentration of **J9**; 2.5 µM	5 µM	10 µM	20 µM	40 µM	Median	Comb. Effect
ALL-19	BCP-ALL	resistant	1:1	-0.10	-0.11	-0.15	-0.13	-0.10	-0.11	antagonism
ALL-50	BCP-ALL	resistant	1:1	-	-	-	-	-	-	-
ALL-54	BCP-ALL	sensitive	500:3	-0.02	-0.08	-0.12	-0.15	-0.10	-0.10	antagonism
ALL-31	T-cell ALL	resistant	1:1	0.02	-0.02	-0.06	-0.08	-0.11	-0.06	antagonism
ETP-2	T-cell ALL	resistant	1:1	-0.05	-0.12	-0.14	-0.11	-0.06	-0.11	antagonism
ALL-16	T-cell ALL	sensitive	2500:1	-0.05	-0.04	-0.04	-0.01	-0.01	-0.04	antagonism

CHEMICAL SUPPLEMENTARY MATERIAL

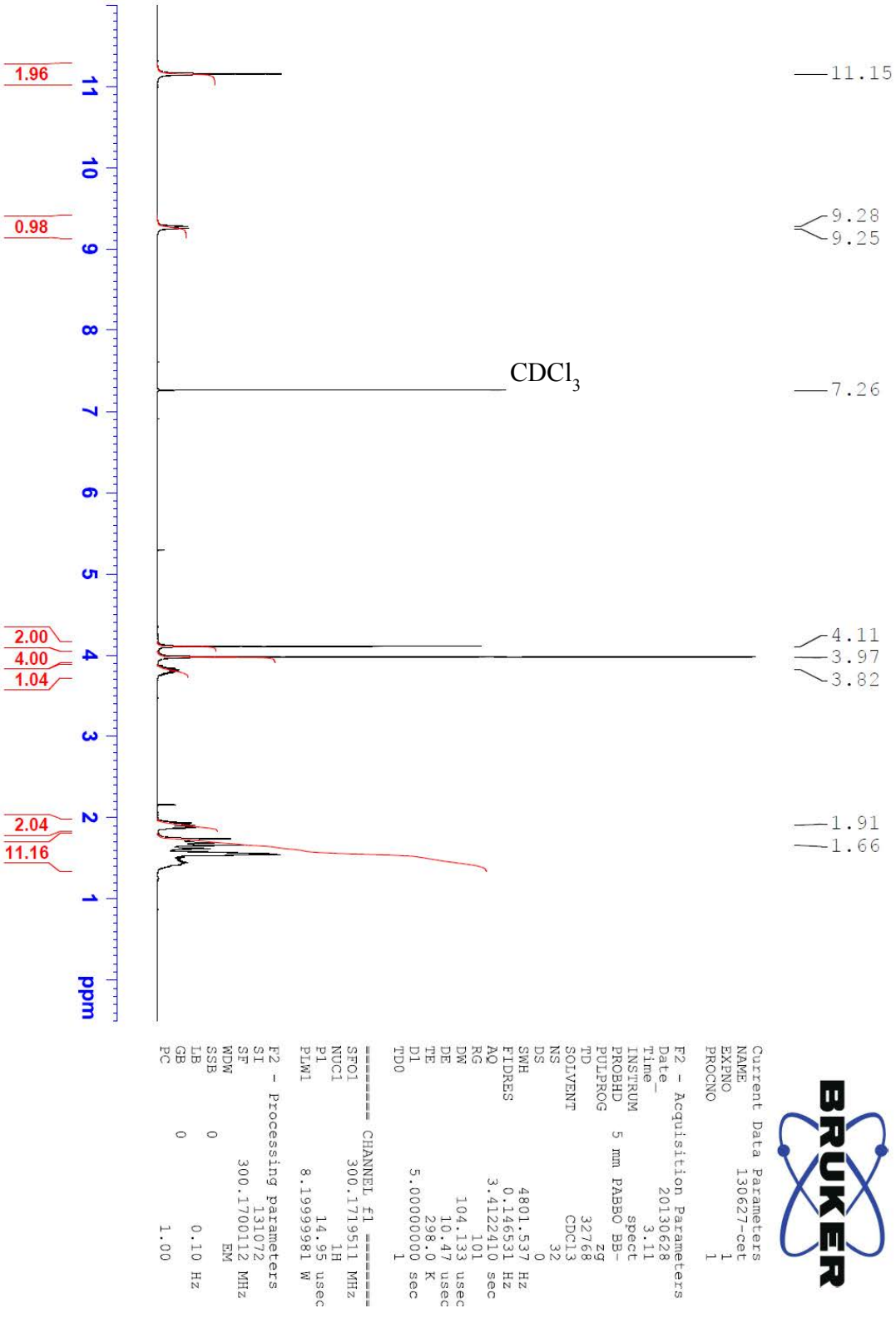


Spectra for Compound 1

^{1}H NMR Compound 1

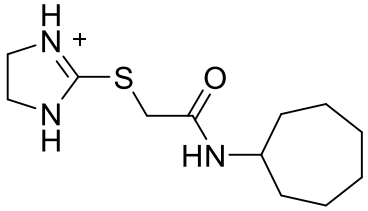
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cycloheptylacetamide

^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **1**

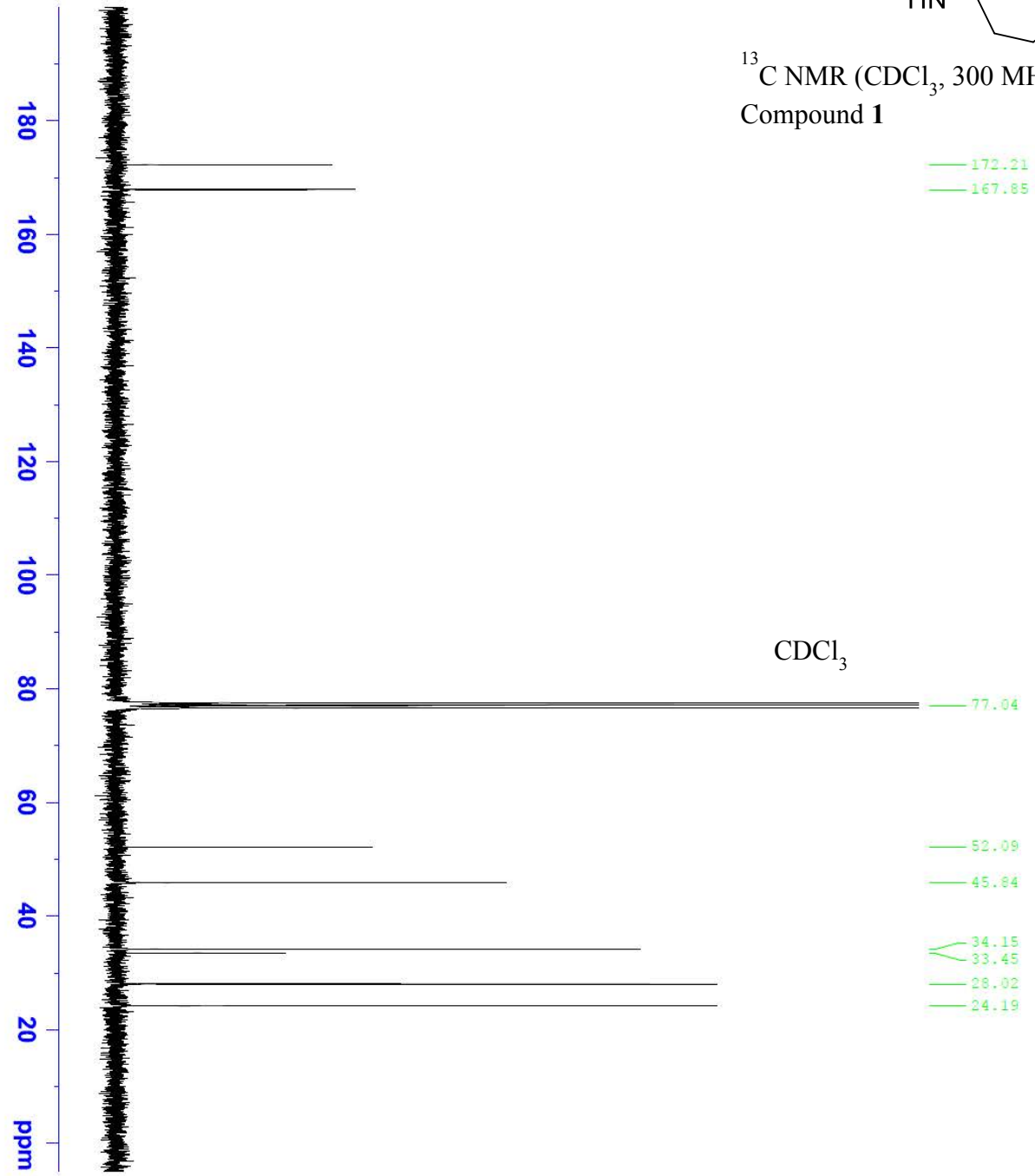


^{13}C NMR Compound 1

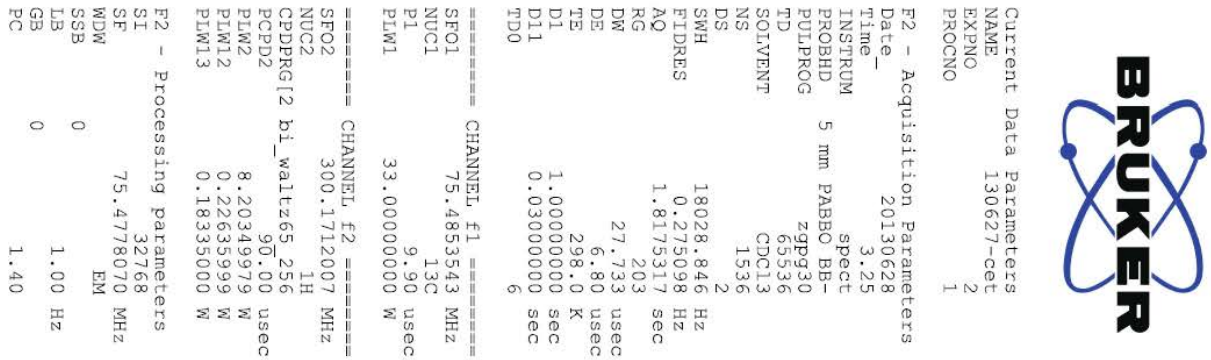
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cycloheptylacetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **1**



$CDCl_3$



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^{1}H-^{1}H COSY Compound 1

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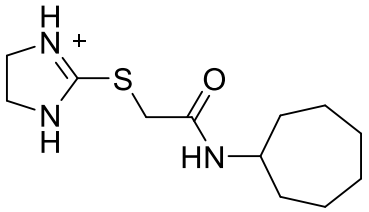

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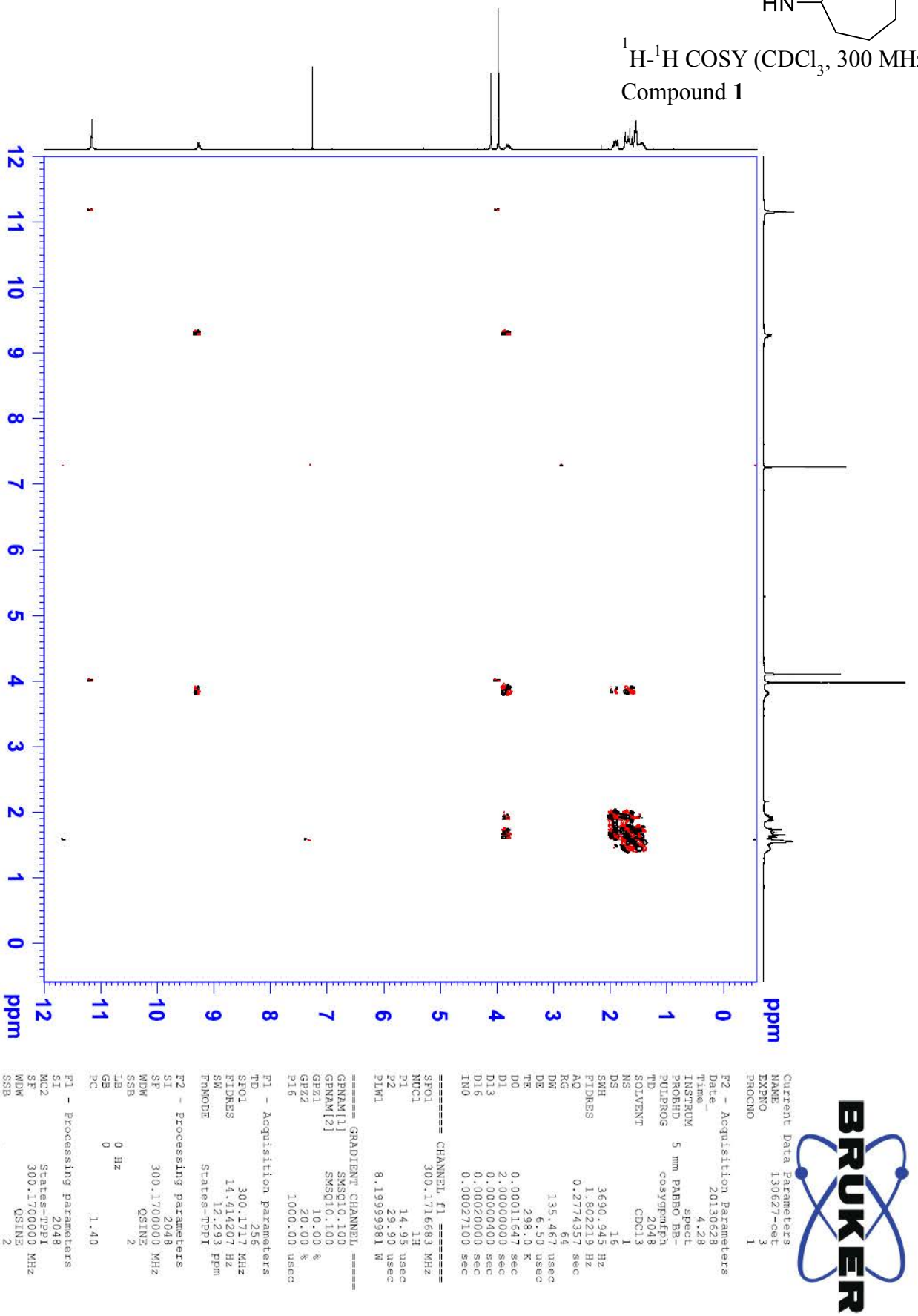
Compound **1**



^{1}H-^{13}C HSQC Compound 1

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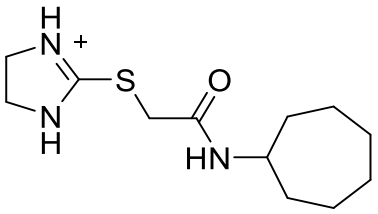
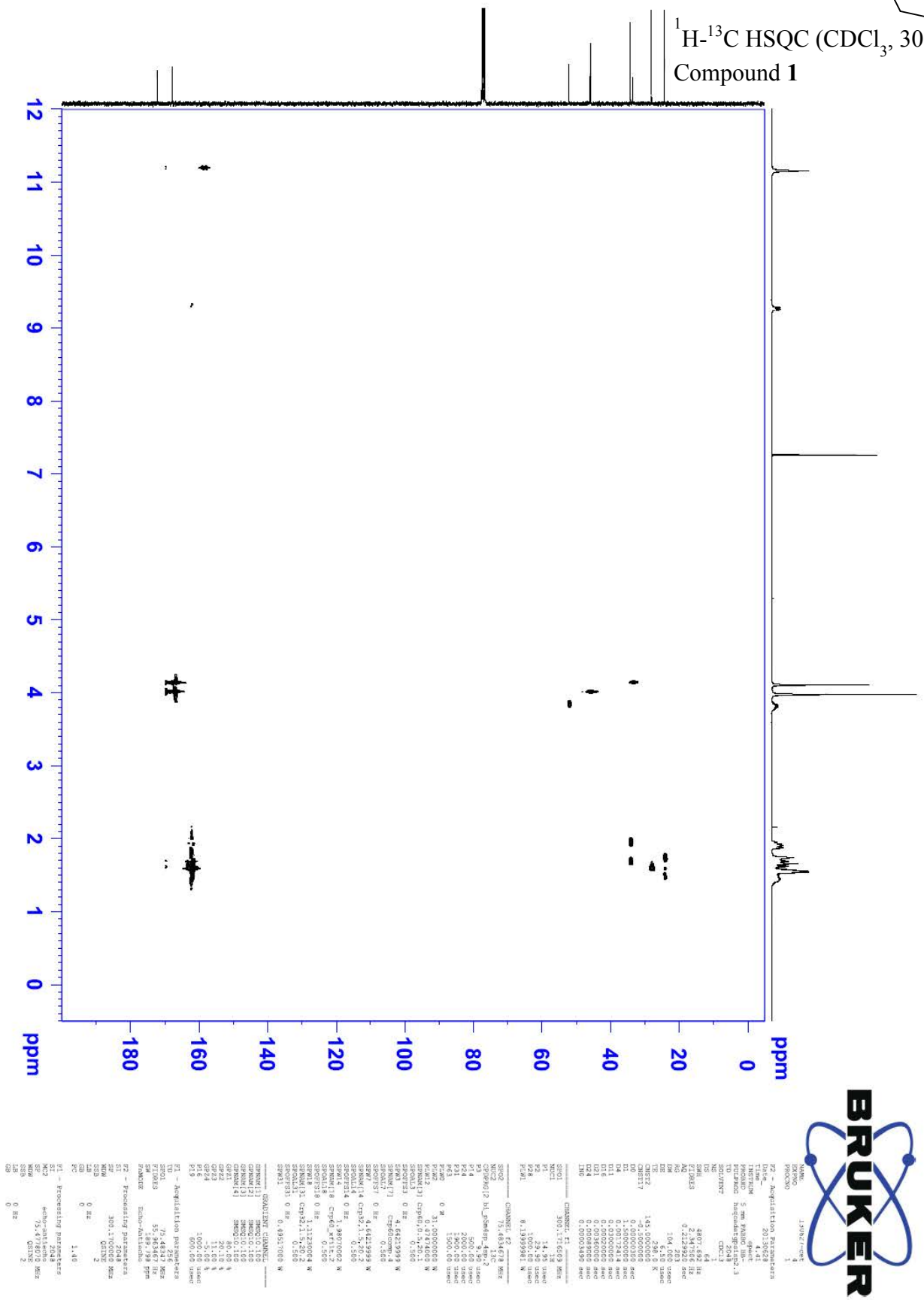
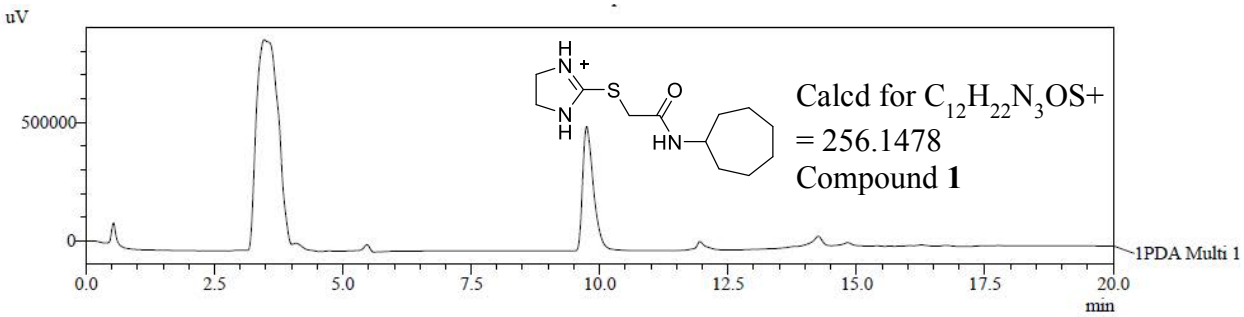
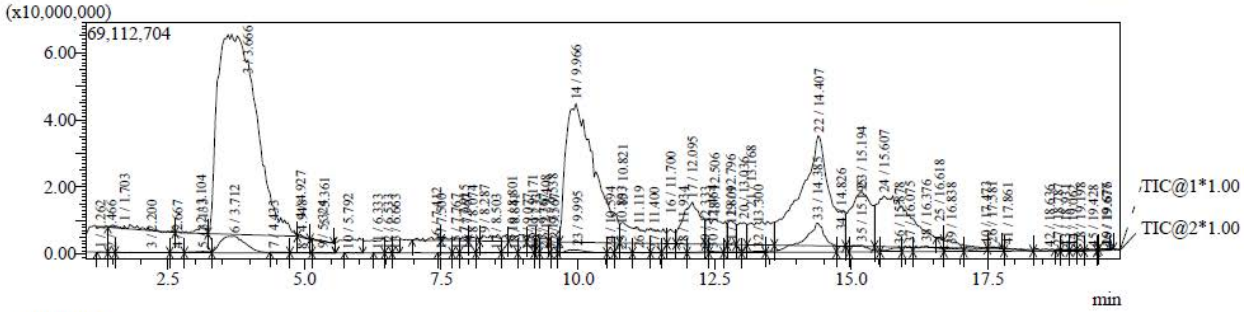


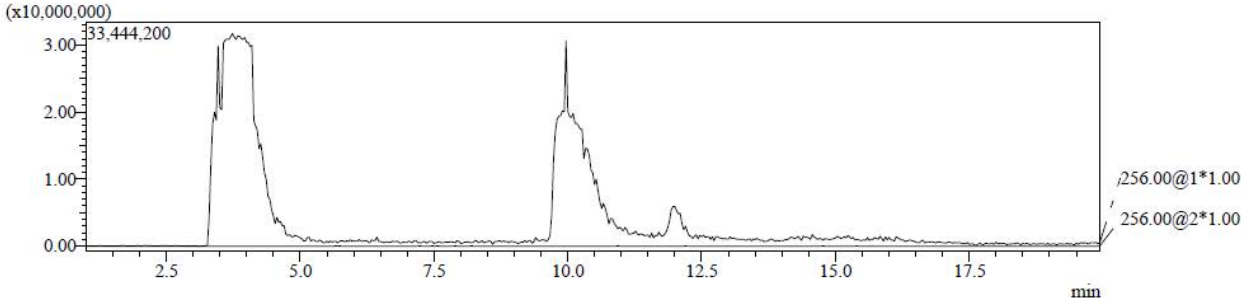
^{1}H-^{13}C HSQC ($CDCl_3$, 300 MHz)
Compound **1**



LCMS Compound 1

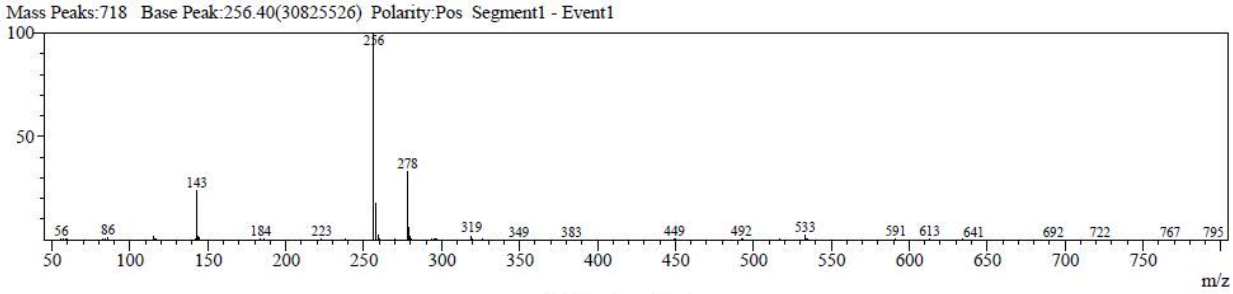






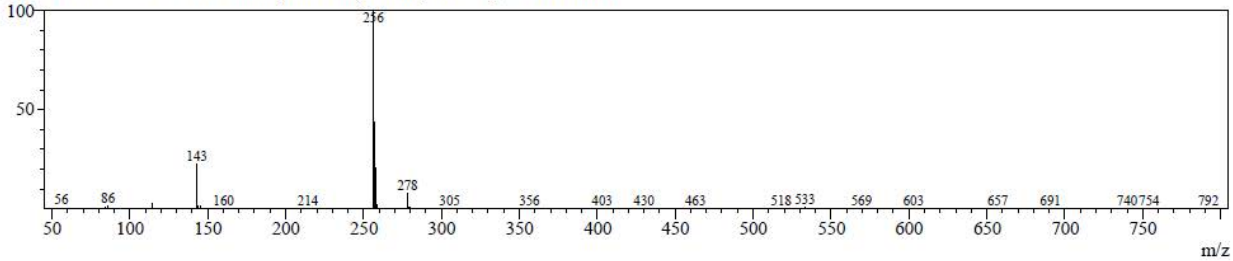
MS Spectrum Graph

Ret.Time:3.667(Scan#:161)
BG Mode:?
Mass Peaks:718 Base Peak:256.40(30825526) Polarity:Pos Segment1 - Event1

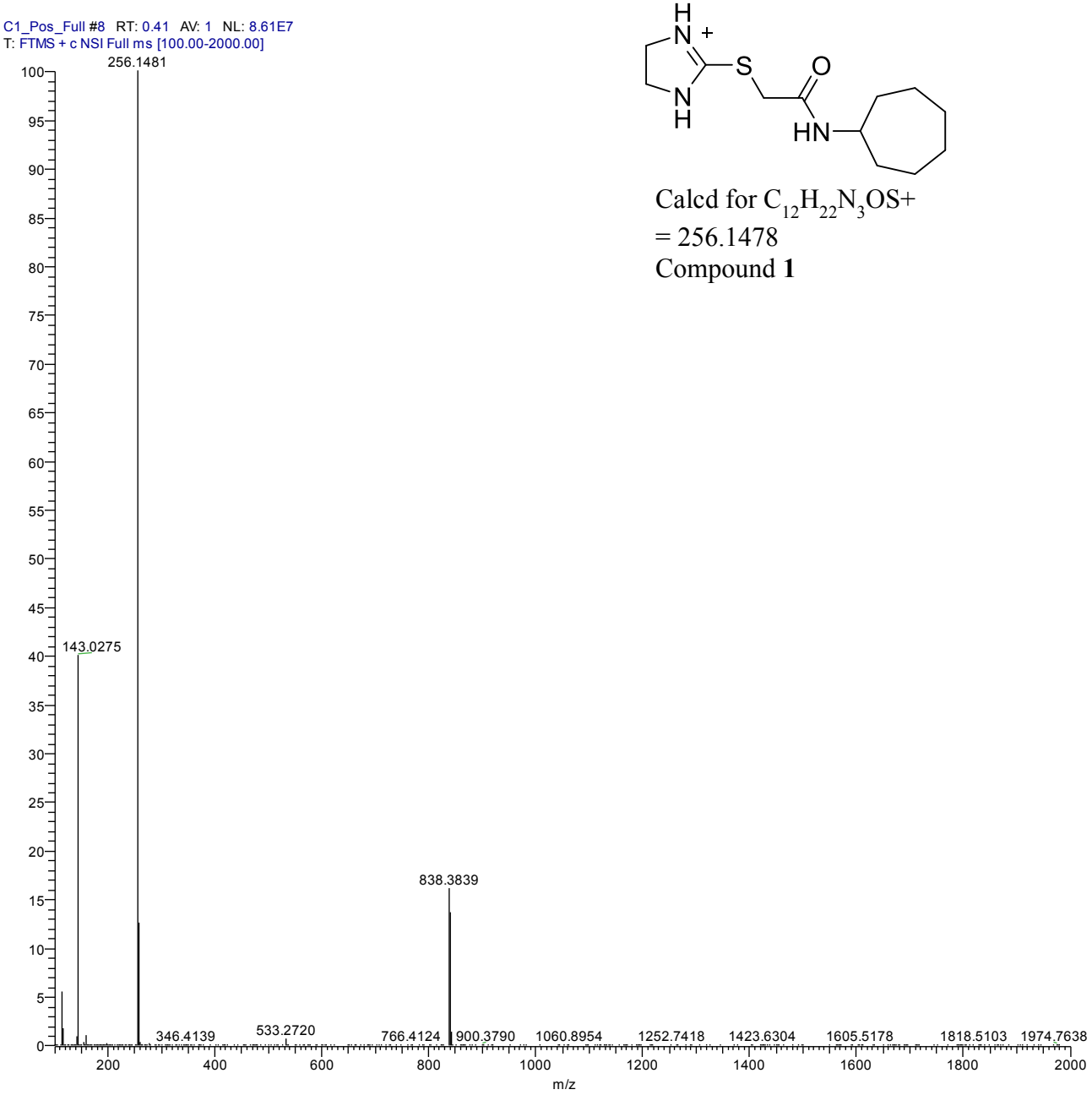


MS Spectrum Graph

Ret.Time:9.933(Scan#:537)
BG Mode:None
Mass Peaks:733 Base Peak:256.10(20052410) Polarity:Pos Segment1 - Event1



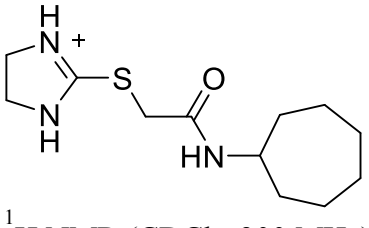
HRMS(ESI) Compound 1



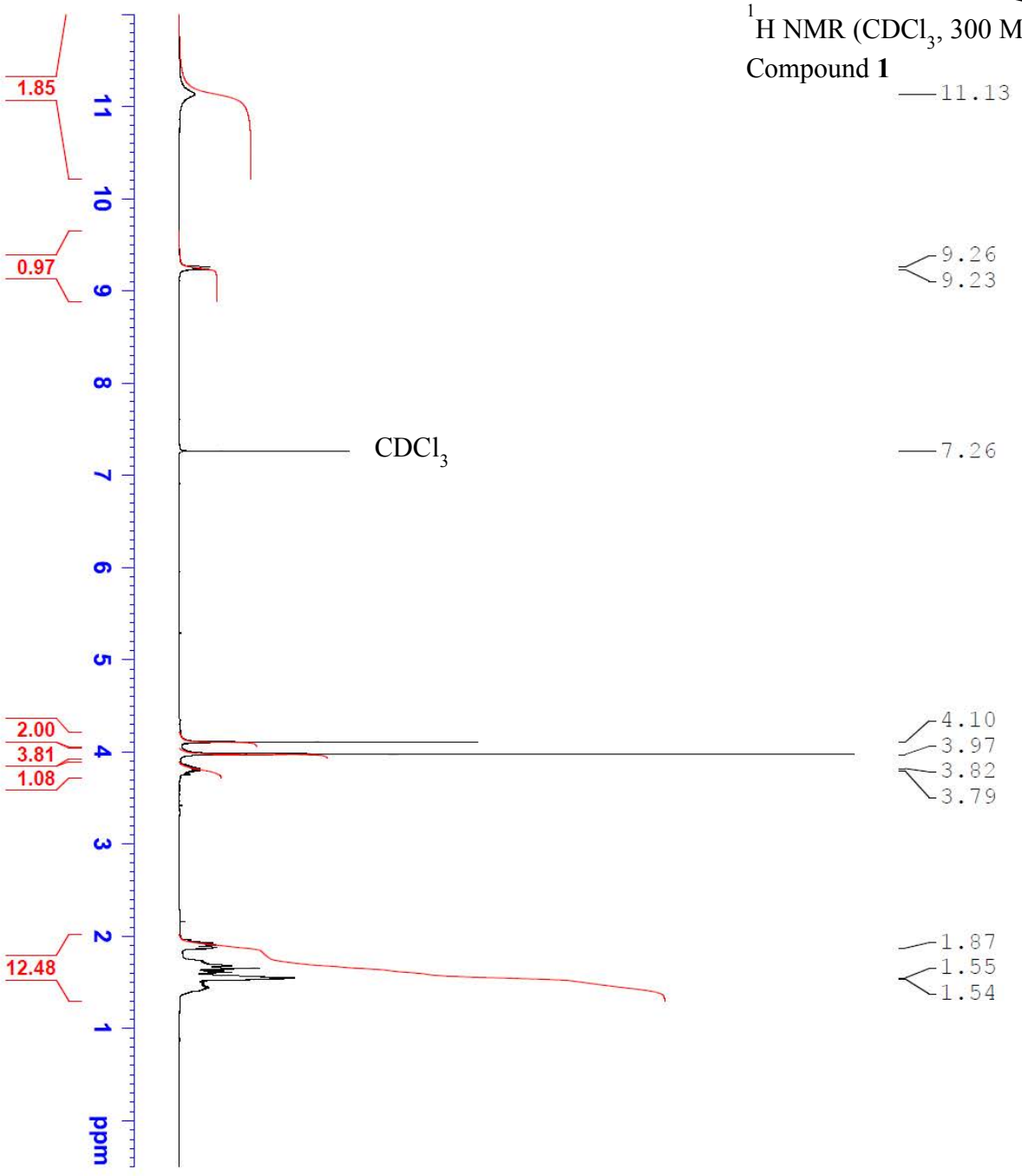
Calcd for $C_{12}H_{22}N_3OS^+$
= 256.1478
Compound **1**

^{1}H NMR Compound 1, purchased

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cycloheptylacetamide



^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **1**



Current Data Parameters
NAME 130812-cet
EXPNO 1
PROCNO 1

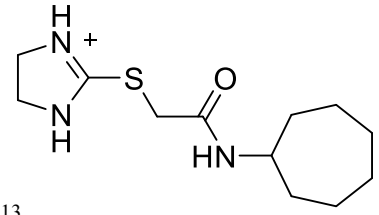
F2 - Acquisition Parameters
Date_ 20130812
Time_ 21.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4801.537 Hz
FIDRES 0.146531 Hz
AQ 3.4122410 sec
RG 90.5
DW 104.133 usec
DE 10.47 usec
TE 298.0 K
D1 5.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 300.1719511 MHz
NUC1 1H
P1 14.95 usec
PLW1 8.19999981 W

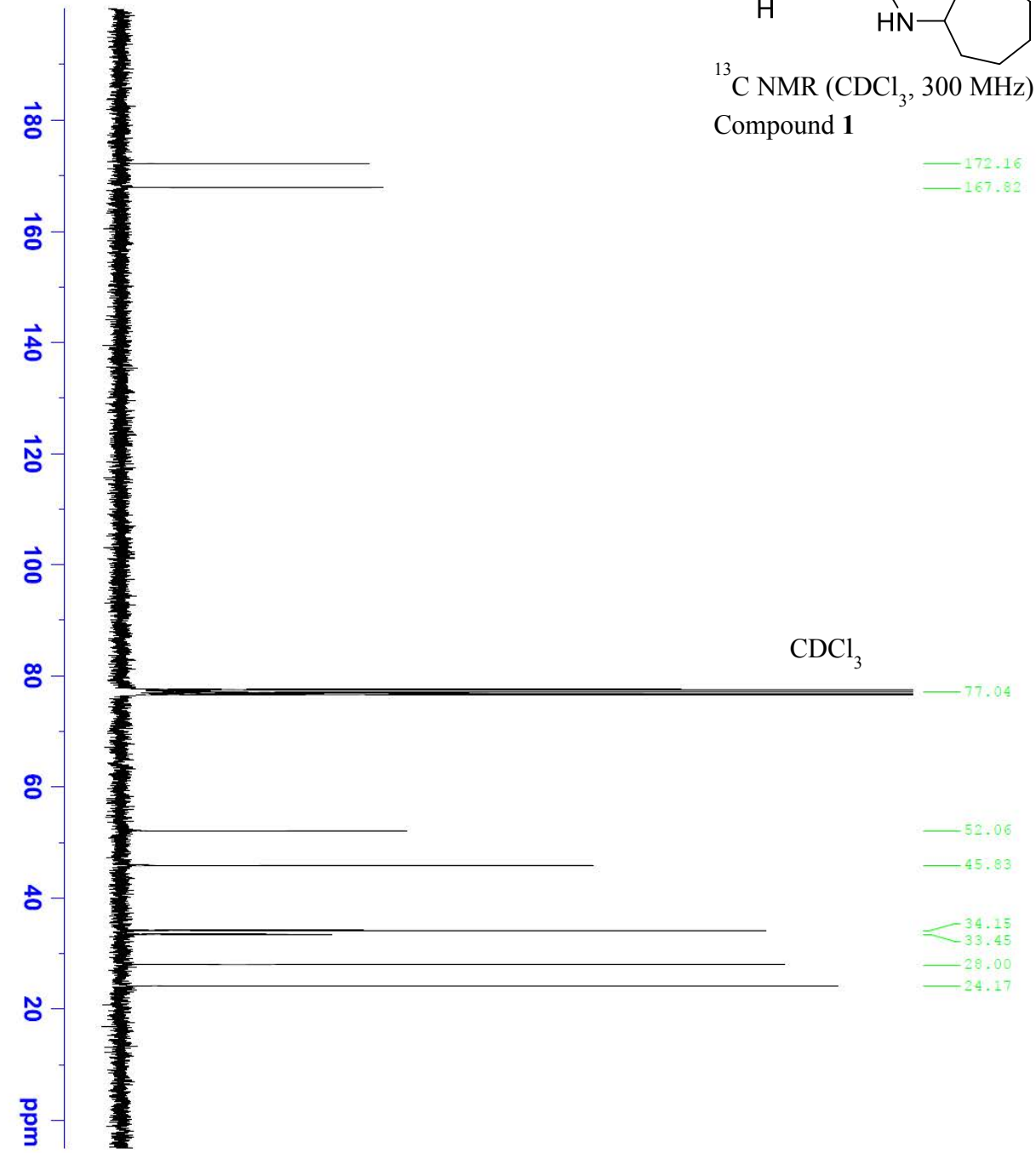
F2 - Processing parameters
SI 131072
SF 300.1700074 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

^{13}C NMR Compound 1, purchased

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cycloheptylacetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **1**

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Current Data Parameters
NAME                  130812-cet
EXPNO                          2
PROCNO                         1

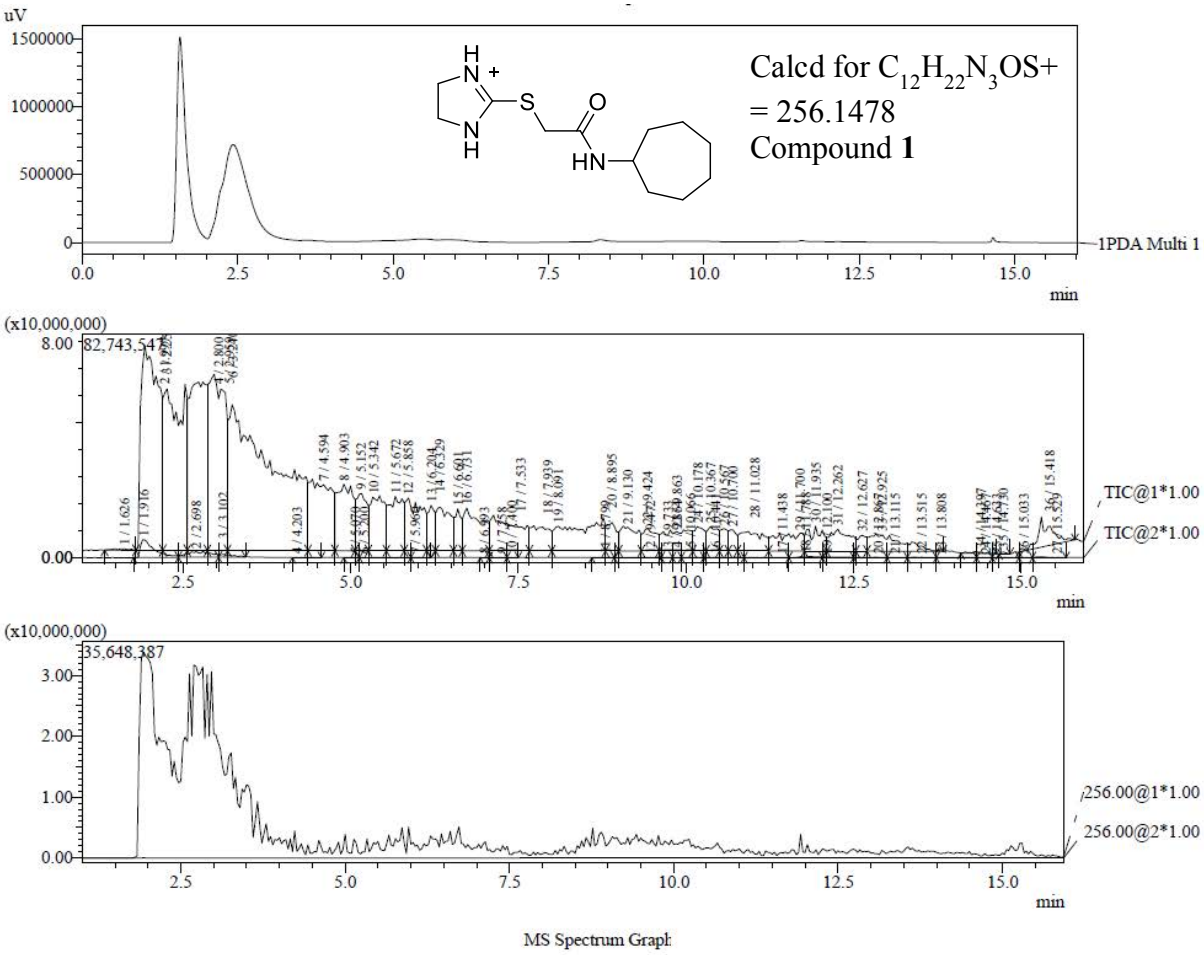
F2 - Acquisition Parameters
Date_                 20130812
Time_                     22.10
INSTRUM                    spect
PROBHD     5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1536
DS                          2
SWH                 18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                        203
DW                     27.733 usec
DE                      6.80 usec
TE                     298.0 K
D1                     1.00000000 sec
D11                    0.03000000 sec
TD0                           6

======== CHANNEL f1 ========
SFO1                75.4853543 MHz
NUC1                       13C
P1                        9.90 usec
PLW1                33.00000000 W

======== CHANNEL f2 ========
SFO2                300.1712007 MHz
NUC2                       1H
CPDPRG[2 bi_waltz65_256
PCPD2                  90.00 usec
PLW2                8.20349979 W
PLW12               0.22635999 W
PLW13               0.18335000 W

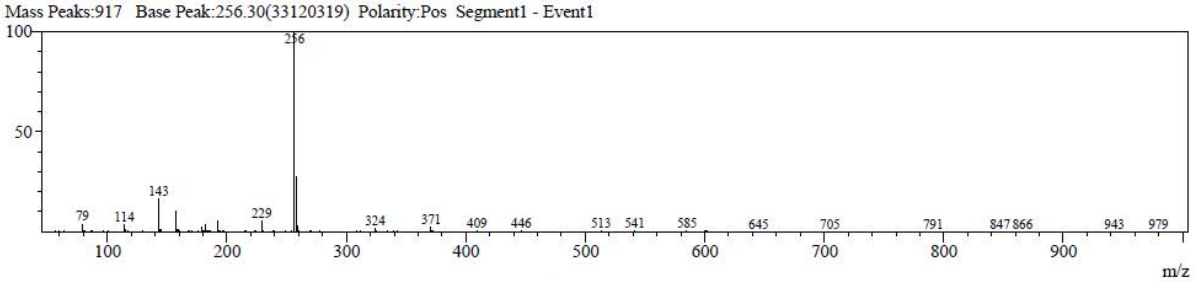
F2 - Processing parameters
SI                        32768
SF                  75.4778070 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

LCMS Compound 1, purchased



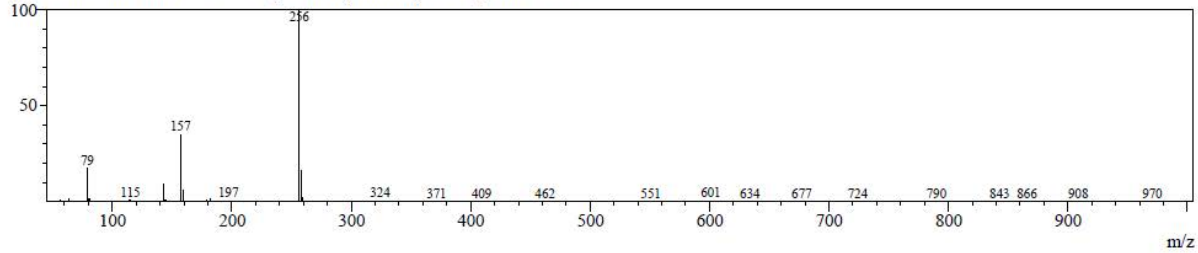
MS Spectrum Graph

Ret.Time:1.967(Scan#:59)
BG Mode:None
Mass Peaks:917 Base Peak:256.30(33120319) Polarity:Pos Segment1 - Event1



MS Spectrum Graph

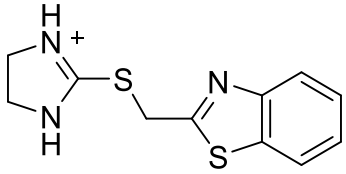
Ret.Time:2.767(Scan#:107)
BG Mode:?
Mass Peaks:917 Base Peak:256.30(30016815) Polarity:Pos Segment1 - Event1



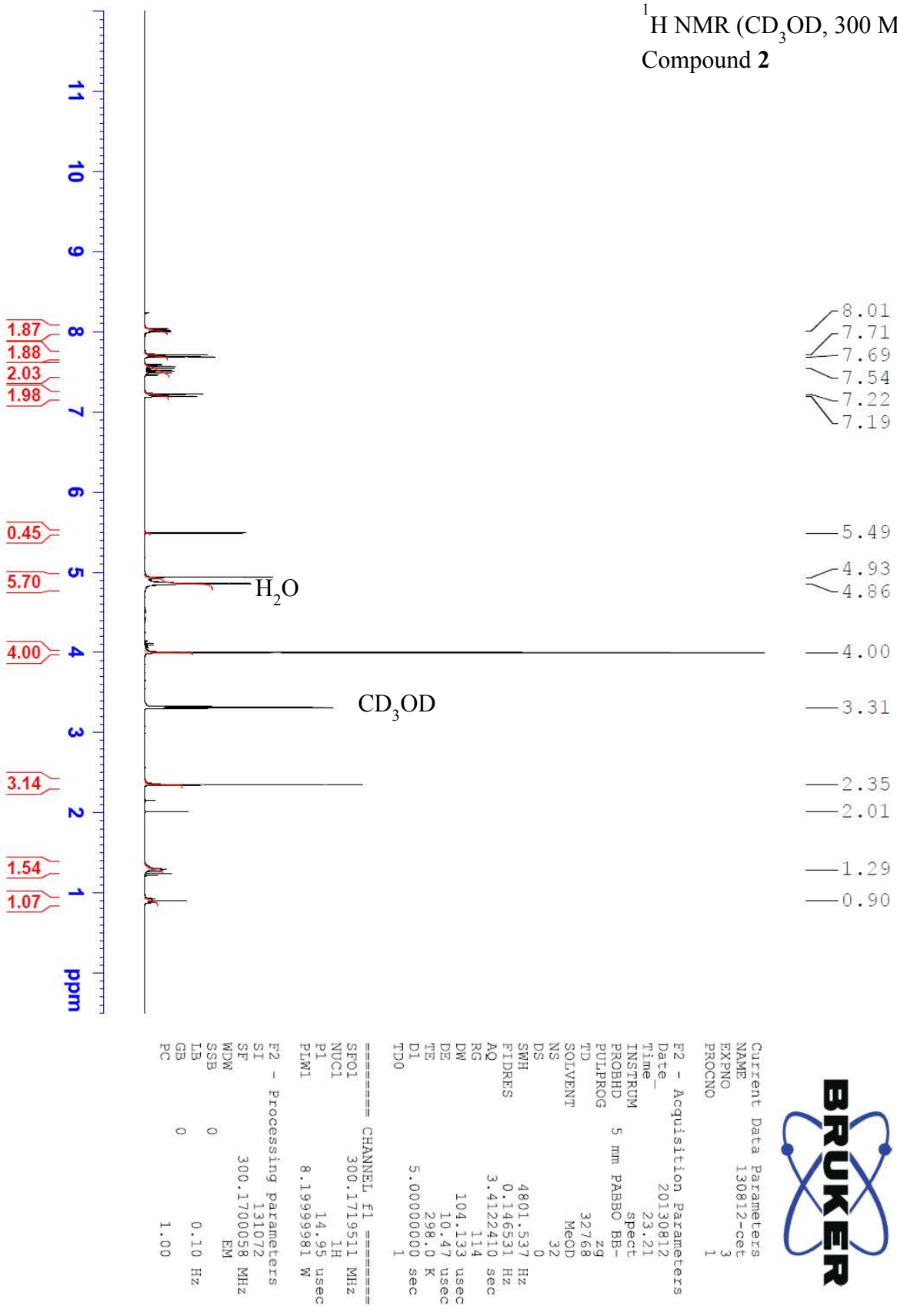
Spectra for Compound 2

^{1}H NMR Compound 2

2-(((4,5-dihydro-1H-imidazol-2-yl)thio)methyl)benzo[d]thiazole

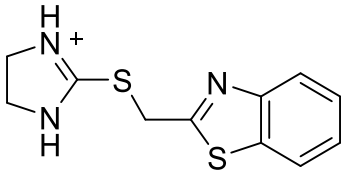


^{1}H NMR (CD_3OD, 300 MHz)
Compound **2**

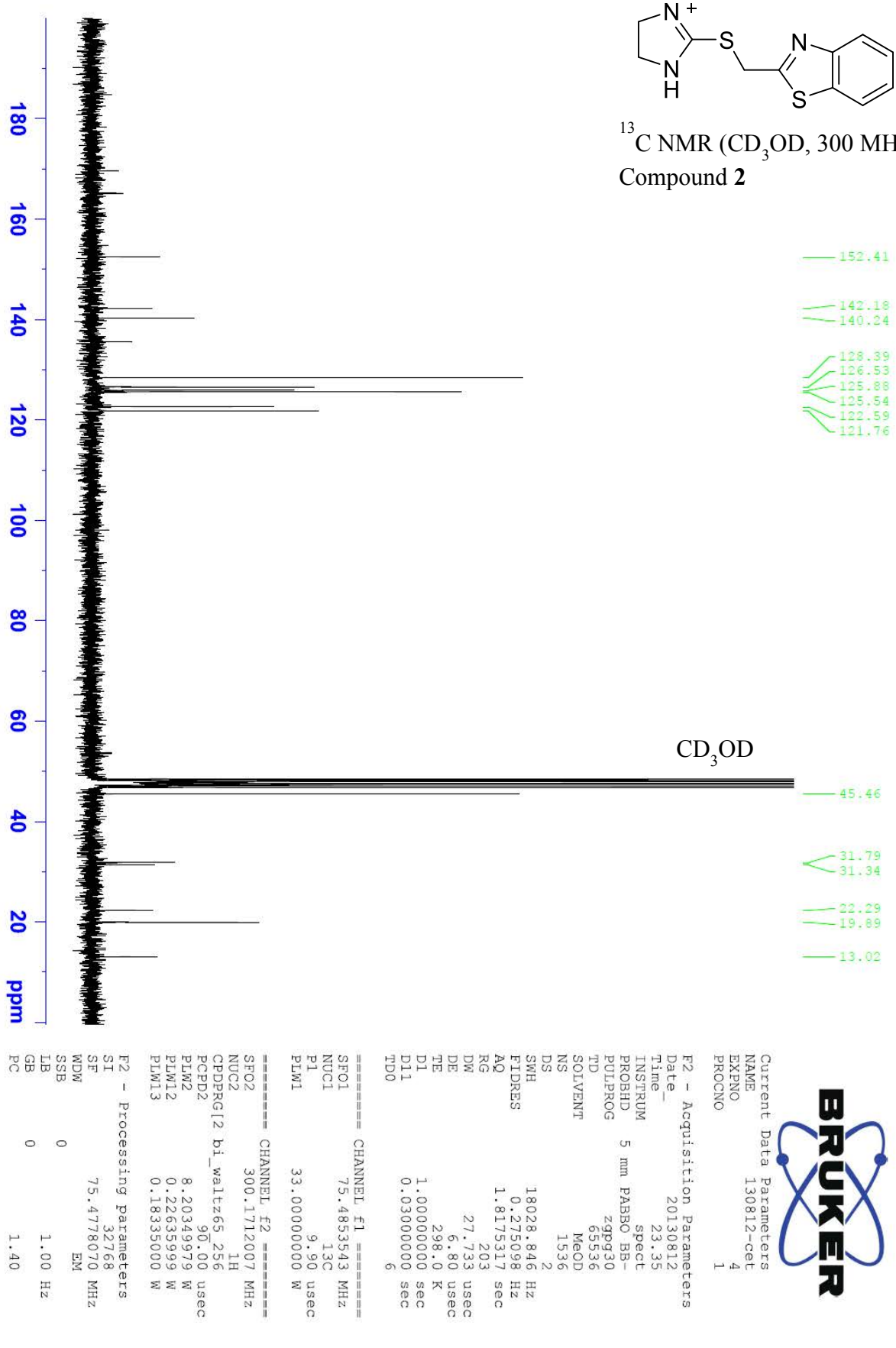


^{13}C NMR Compound 2

2-(((4,5-dihydro-1H-imidazol-2-yl)thio)methyl)benzo[d]thiazole



^{13}C NMR (CD_3OD, 300 MHz)
Compound **2**



Current Data Parameters
NAME 130812-cet
EXPNO 4
PROCNO 1

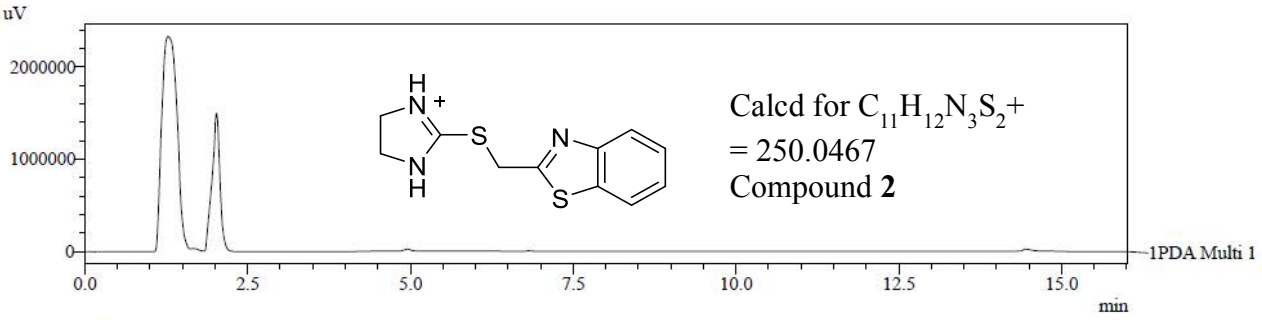
F2 - Acquisition Parameters
Date_ 20130812
Time_ 23.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1536
DS 2
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 203
DW 27.733 usec
DE 6.80 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 6

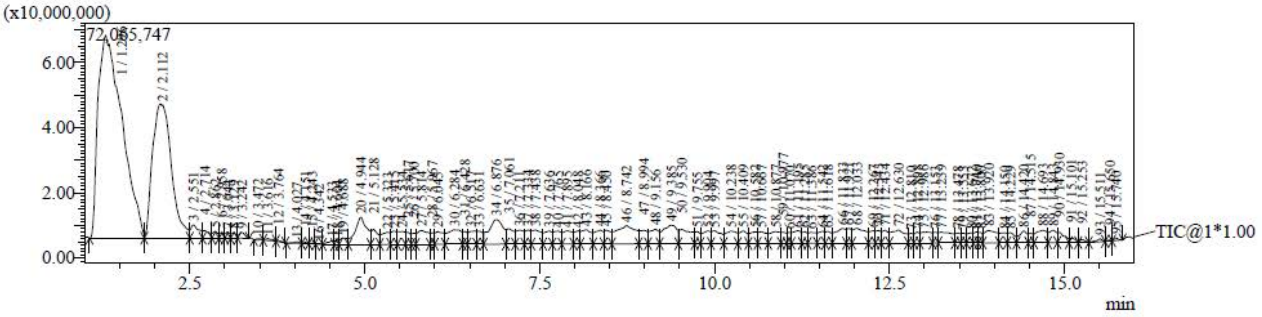
========== CHANNEL f1 ==========
SFO1 75.4853543 MHz
NUC1 13C
P1 9.90 usec
PLW1 33.00000000 W

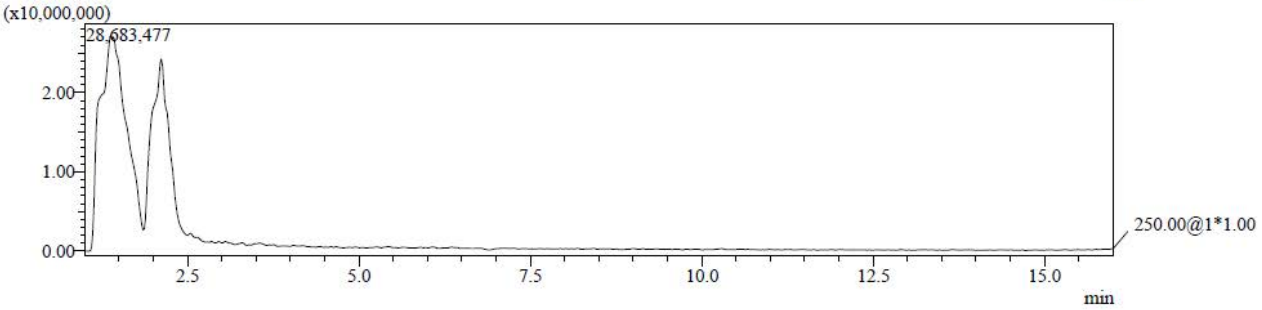
========== CHANNEL f2 ==========
SFO2 300.1712007 MHz
NUC2 1H
CPDPRG[2 bi_waltz65_256
PCPD2 90.00 usec
PLW2 8.20349979 W
PLW12 0.22635999 W
PLW13 0.18335000 W

F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

LCMS Compound 2

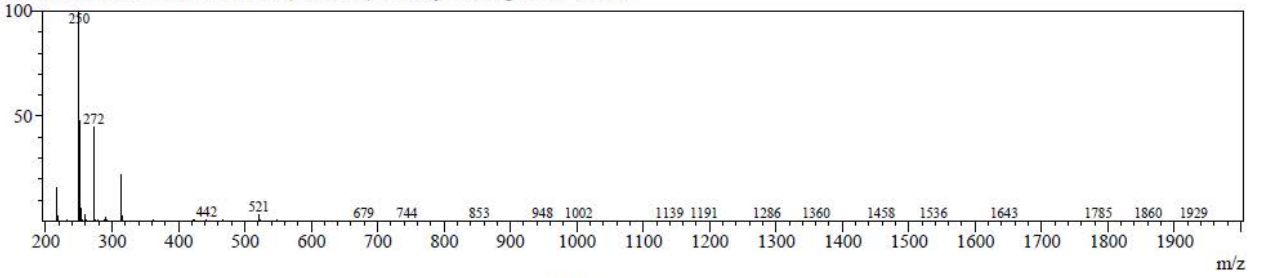






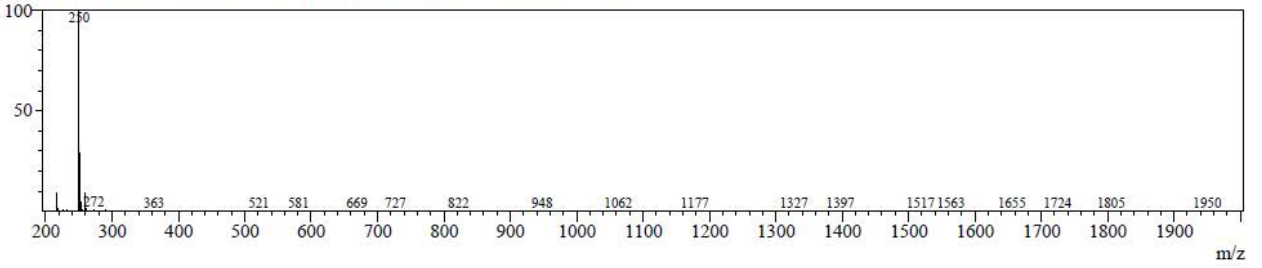
MS Spectrum Graph

Ret.Time:1.300(Scan#:19)
BG Mode:None
Mass Peaks:1726 Base Peak:249.90(19951309) Polarity:Pos Segment1 - Event1



MS Spectrum Graph

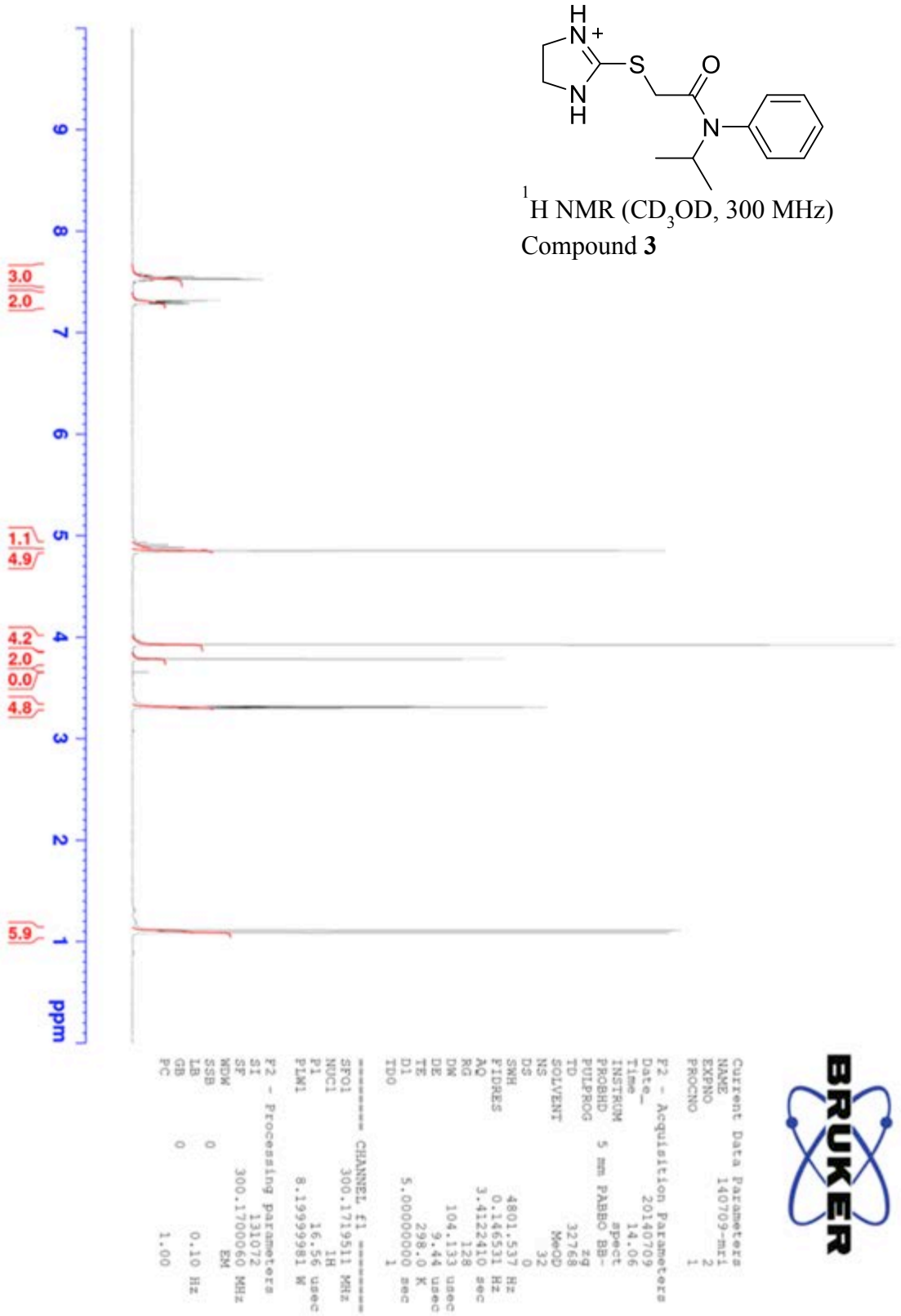
Ret.Time:2.100(Scan#:67)
BG Mode:None
Mass Peaks:1733 Base Peak:250.20(24576840) Polarity:Pos Segment1 - Event1



Spectra for Compound 3

^{1}H NMR Compound 3

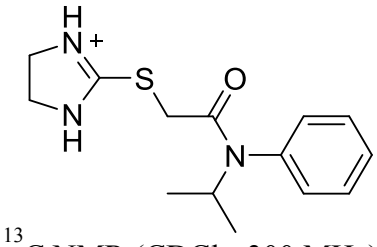
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-phenylacetamide



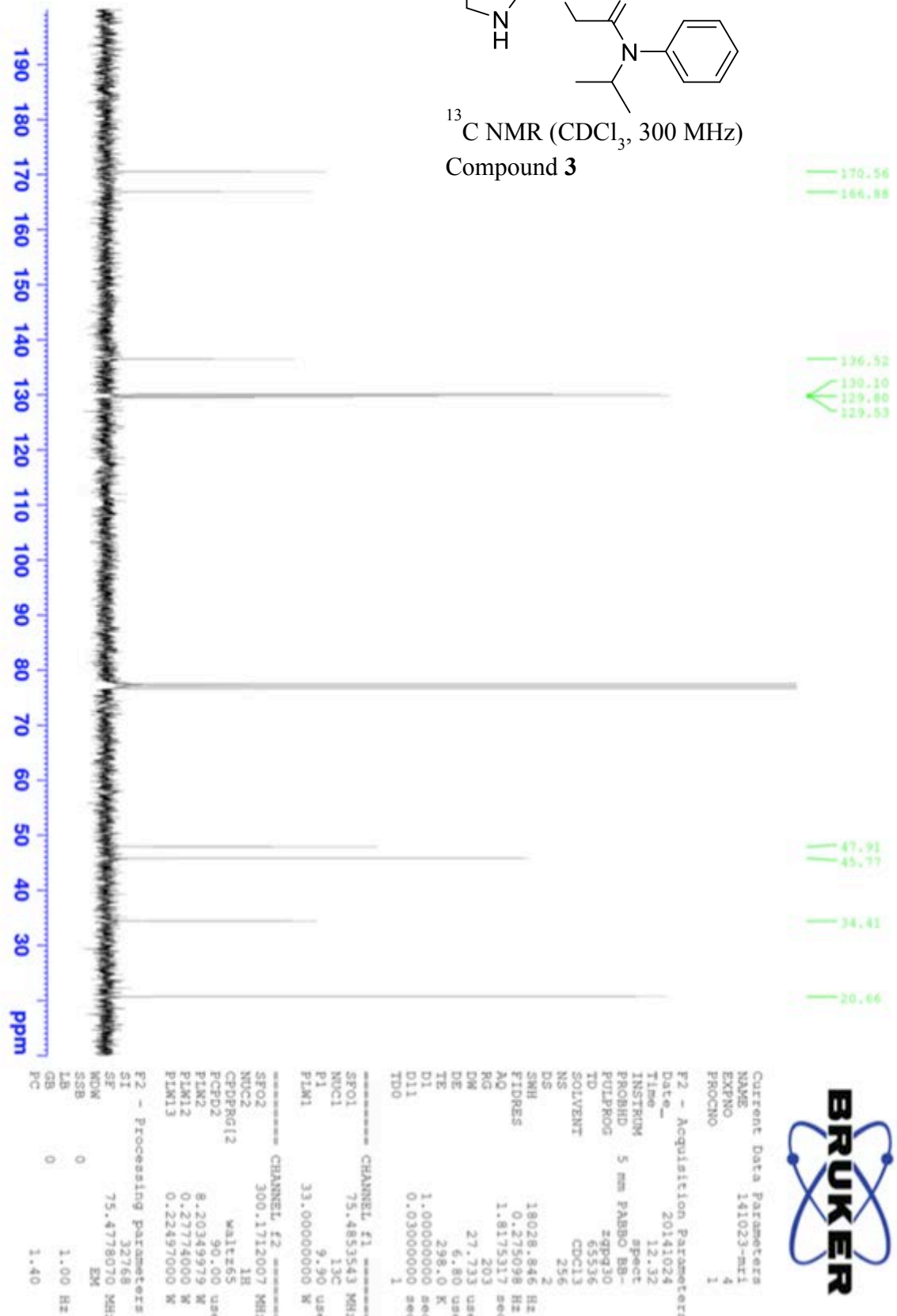
^{1}H NMR (CD_3OD, 300 MHz)
Compound **3**

^{13}C NMR Compound 3

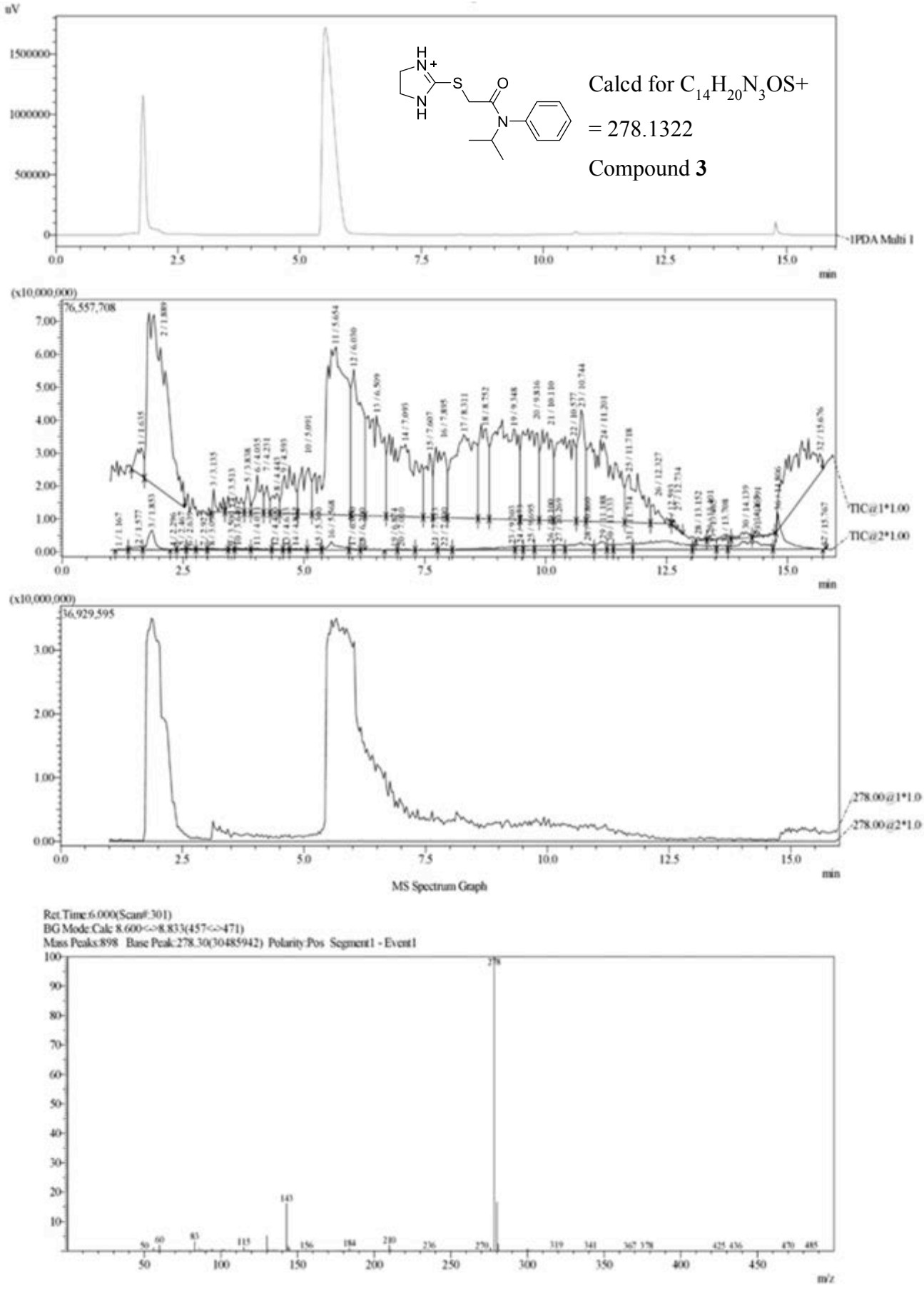
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-phenylacetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **3**

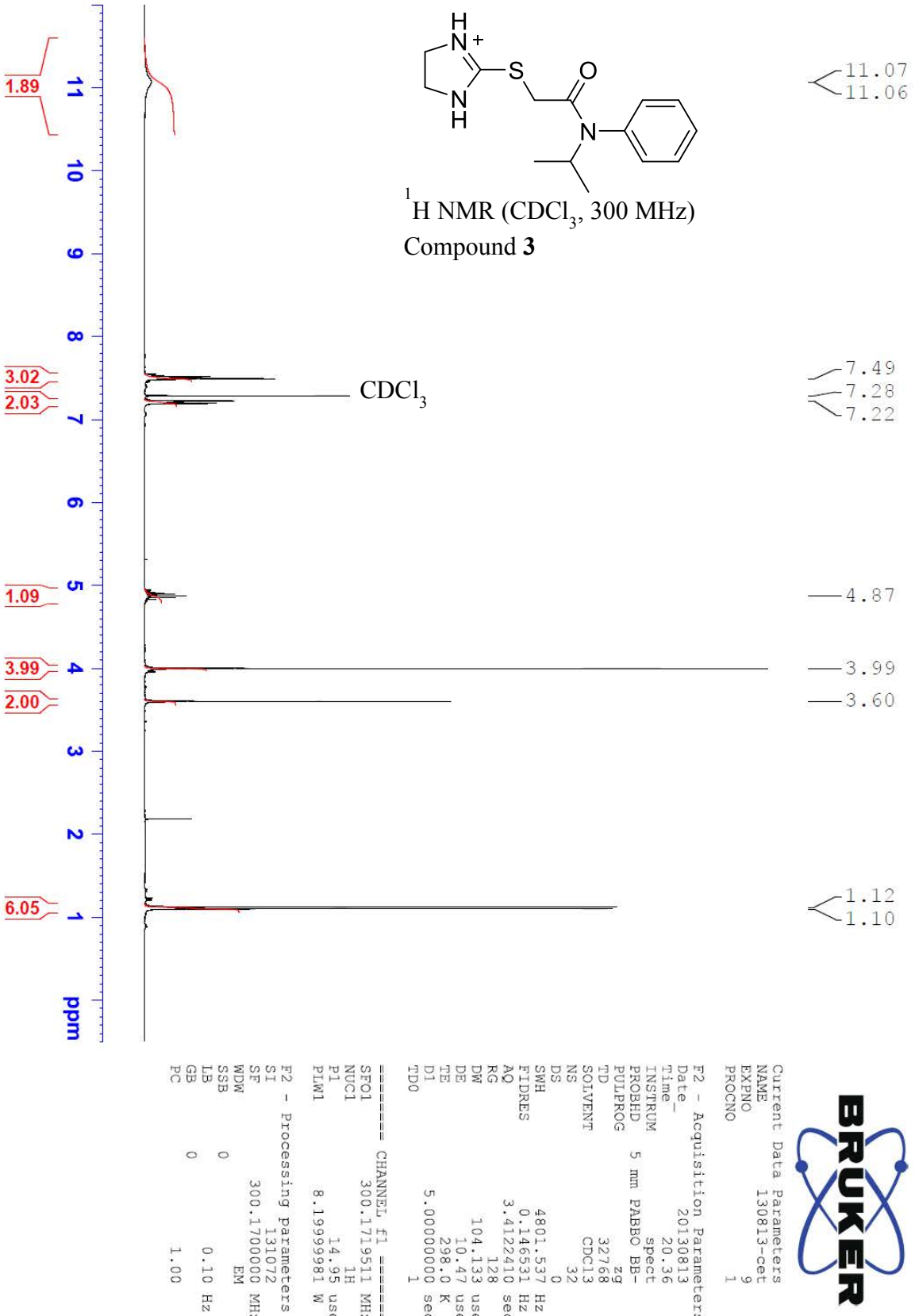


LCMS Compound 3



^{1}H NMR Compound 3, purchased

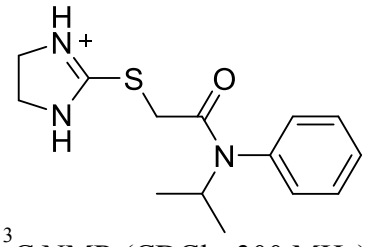
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-phenylacetamide



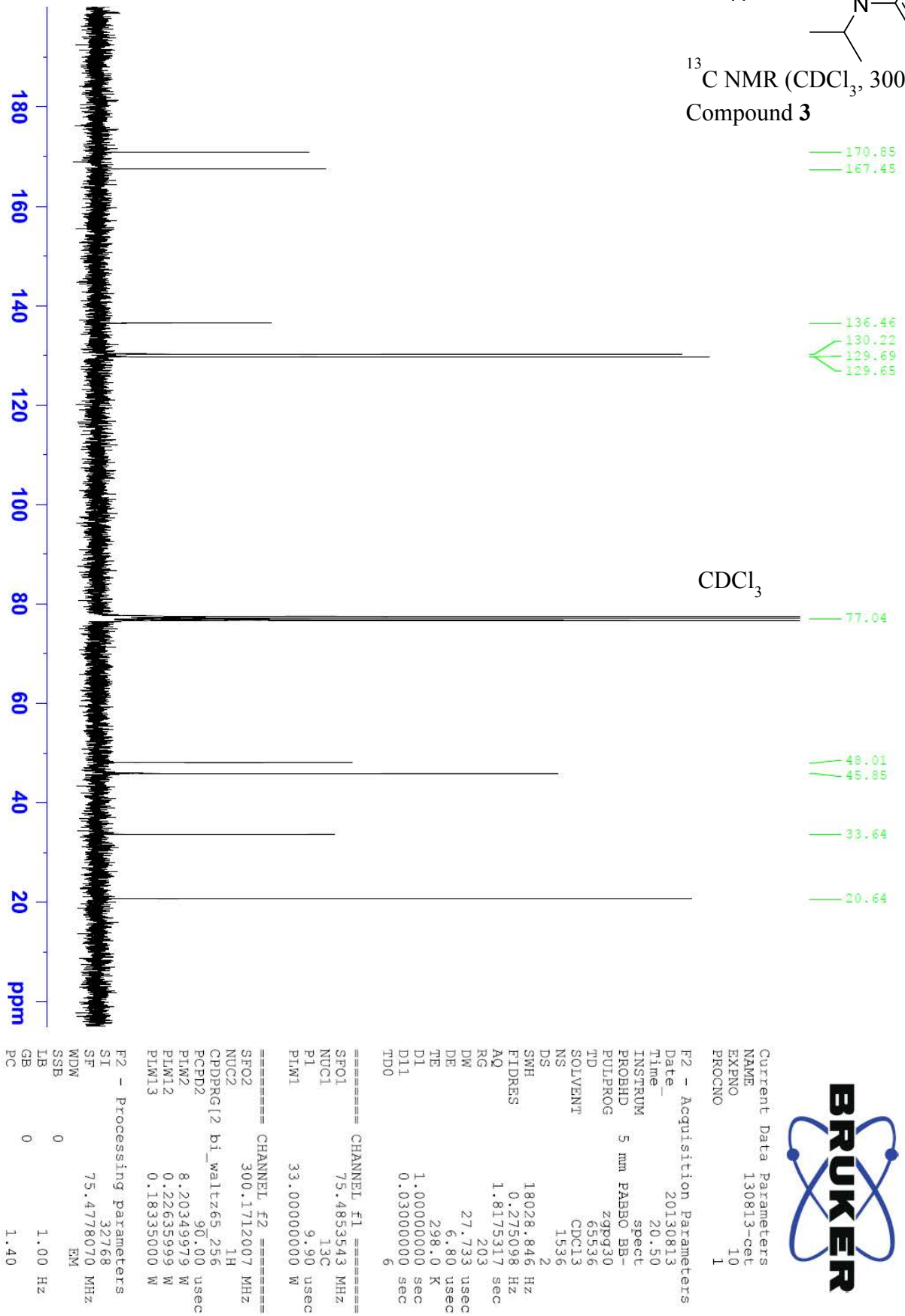
^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **3**

^{13}C NMR Compound 3, purchased

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-phenylacetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **3**

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Current Data Parameters
NAME                  130813-cet
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20130813
Time_                      20.50
INSTRUM                     spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1536
DS                           2
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                         203
DW                      27.733 usec
DE                       6.80 usec
TE                      298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            6

======== CHANNEL f1 ========
SFO1                 75.4853543 MHz
NUC1                        13C
P1                        9.90 usec
PLW1                33.00000000 W

======== CHANNEL f2 ========
SFO2                 300.1712007 MHz
NUC2                        1H
CPDPRG[2 bi_waltz65_256
PCPD2                     90.00 usec
PLW2                 8.20349979 W
PLW12                0.22635999 W
PLW13                0.18335000 W

F2 - Processing parameters
SI                         32768
SF                  75.4778070 MHz
WDW                            EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
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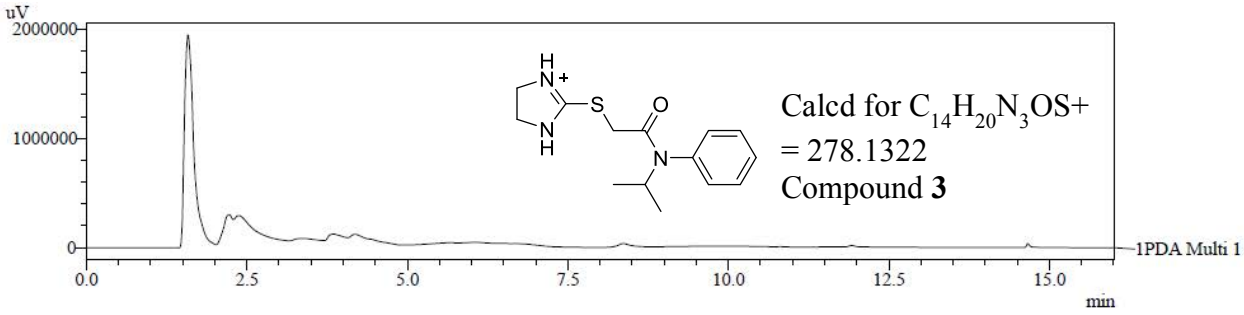
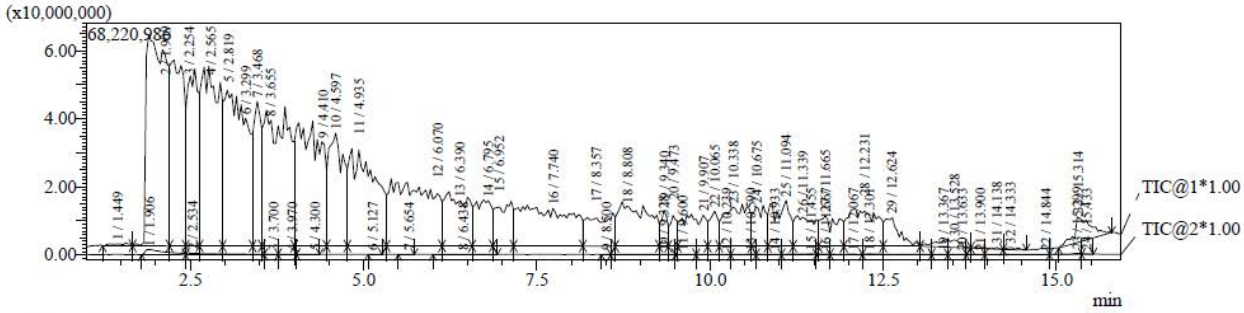
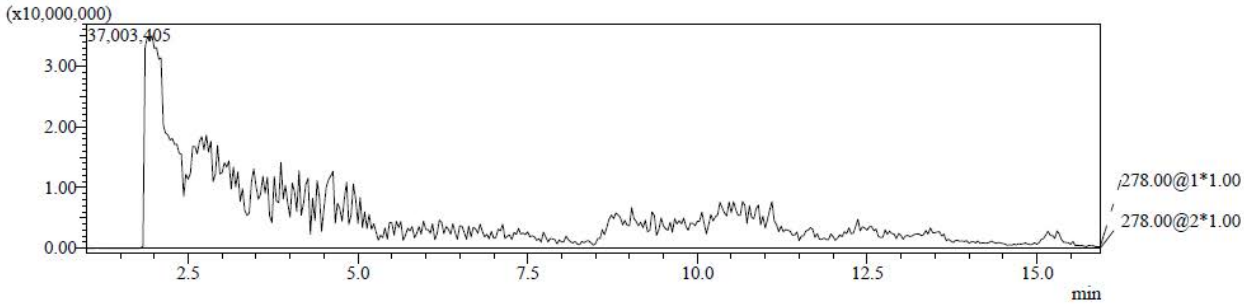
LCMS Compound 3, purchased

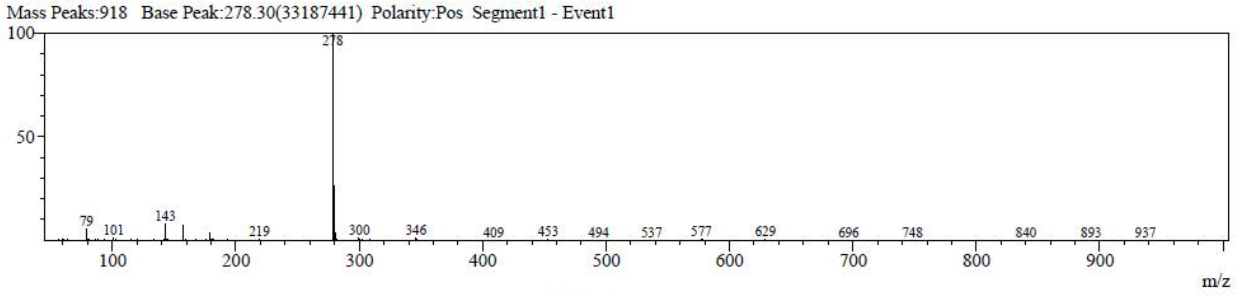






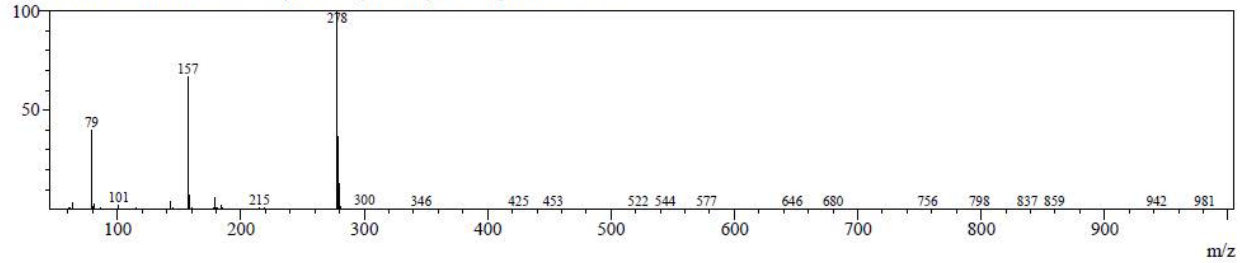
MS Spectrum Grapl:

Ret.Time:1.867(Scan#:53)
BG Mode:None
Mass Peaks:918 Base Peak:278.30(33187441) Polarity:Pos Segment1 - Event1



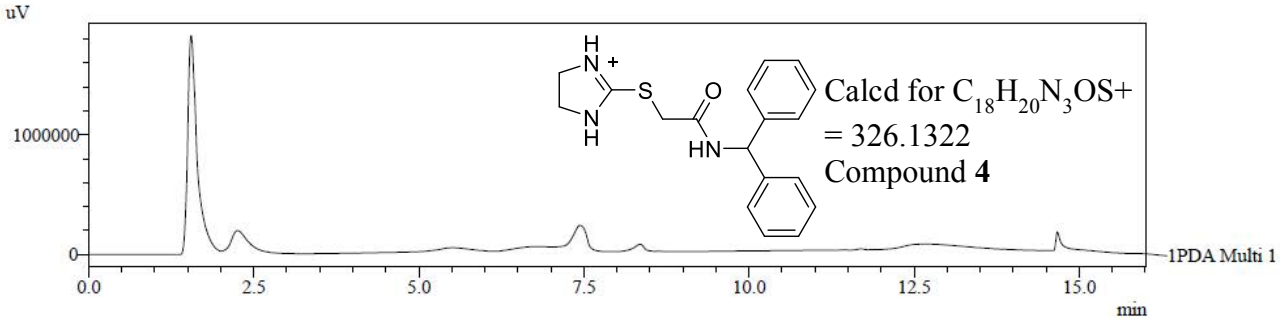
MS Spectrum Grapl:

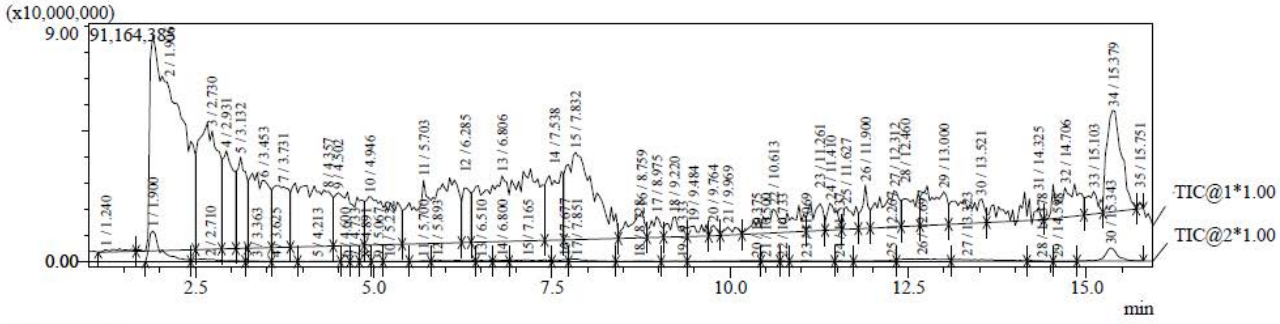
Ret.Time:2.300(Scan#:79)
BG Mode:None
Mass Peaks:929 Base Peak:277.90(17125472) Polarity:Pos Segment1 - Event1

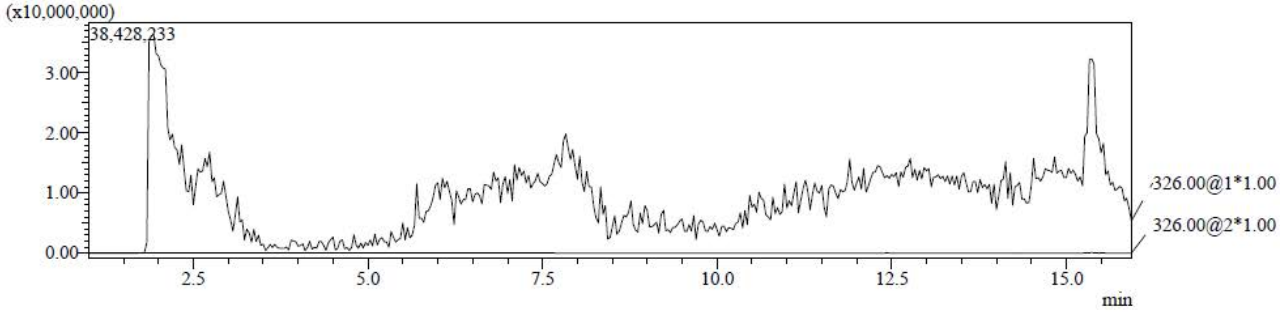


Spectra for Compound 4

LCMS Compound 4

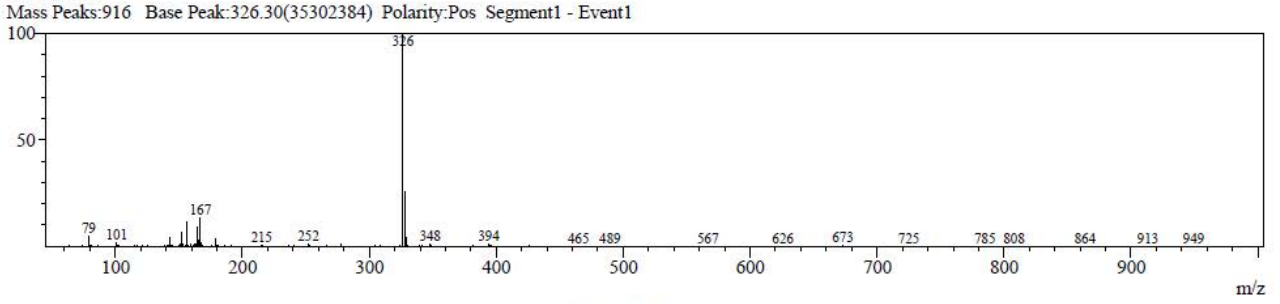






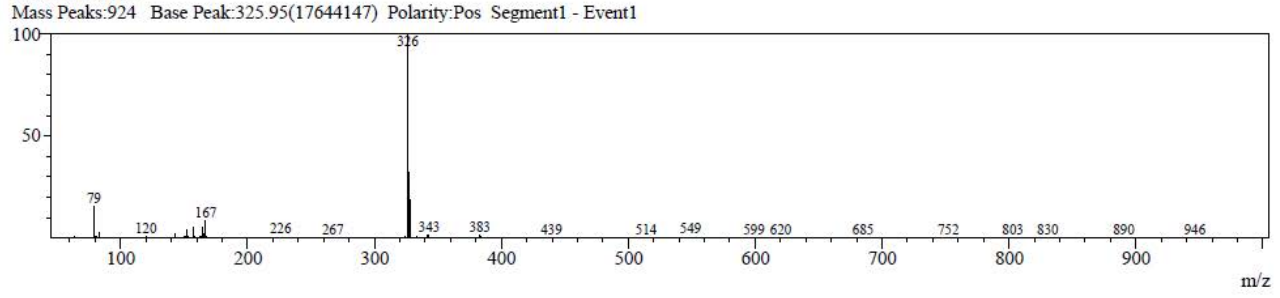
MS Spectrum Grapl:

Ret.Time:1.867(Scan#:53)
BG Mode:?
Mass Peaks:916 Base Peak:326.30(35302384) Polarity:Pos Segment1 - Event1



MS Spectrum Grapl:

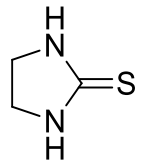
Ret.Time:7.867(Scan#:413)
BG Mode:None
Mass Peaks:924 Base Peak:325.95(17644147) Polarity:Pos Segment1 - Event1



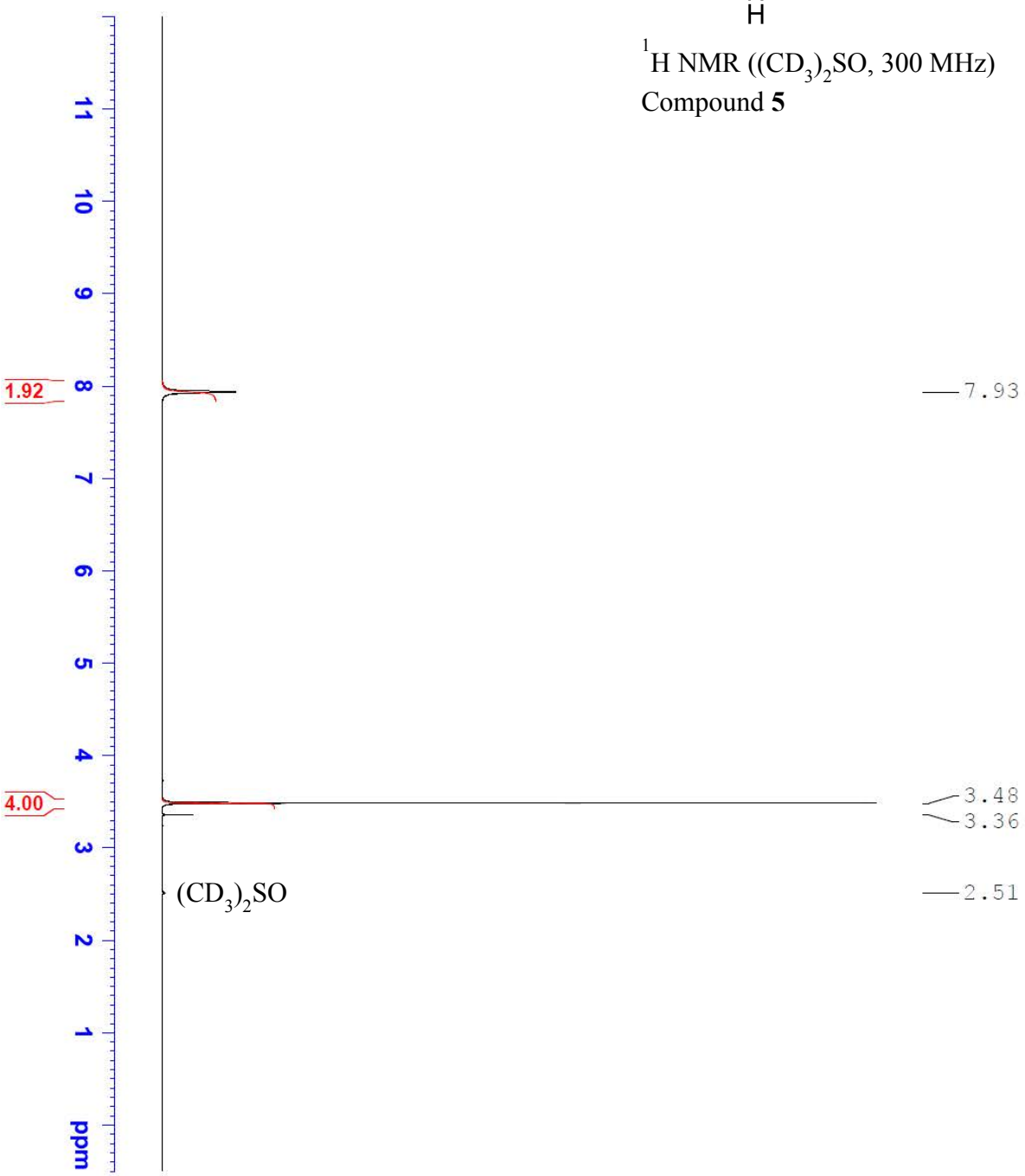
Spectra for Compound 5

^{1}H NMR Compound 5

imidazolidine-2-thione



^{1}H NMR ($(CD_3)_2SO$, 300 MHz)
Compound **5**

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Current Data Parameters
NAME                  131018-cet
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
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Time_                      15.38
INSTRUM                     spect
PROBHD     5 mm PABBO BB-
PULPROG                      zg
TD                        32768
SOLVENT                    DMSO
NS                           32
DS                            0
SWH                    4801.537 Hz
FIDRES                 0.146531 Hz
AQ                     3.4122410 sec
RG                          22.6
DW                       104.133 usec
DE                        10.47 usec
TE                        298.0 K
D1                     5.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                 300.1719511 MHz
NUC1                           1H
P1                         14.95 usec
PLW1                   8.19999981 W

F2 - Processing parameters
SI                           131072
SF                   300.1700000 MHz
WDW                            EM
SSB                             0
LB                           0.10 Hz
GB                              0
PC                           1.00
```
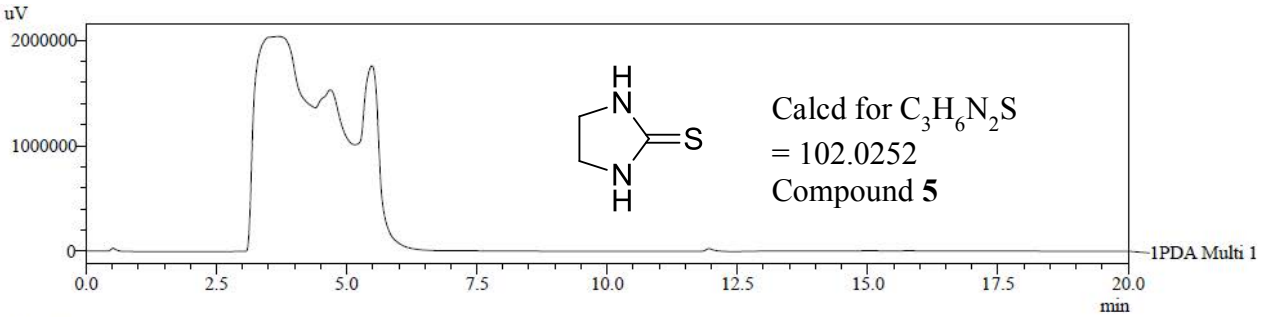
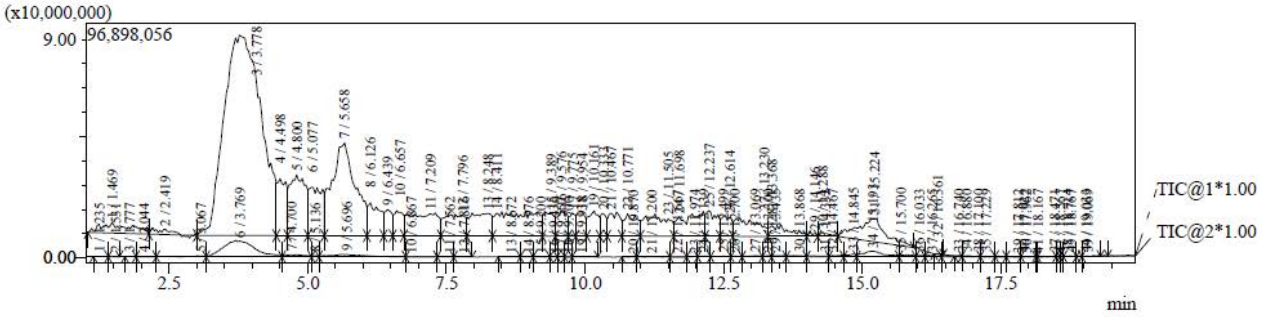
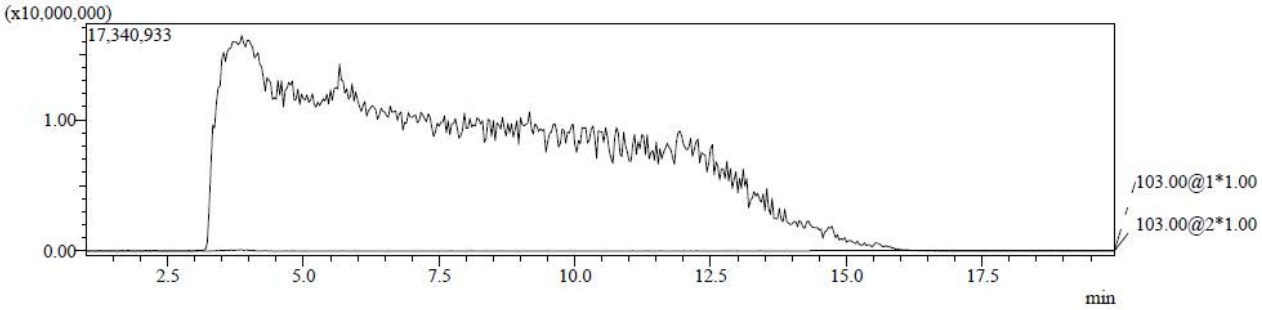

LCMS Compound 5

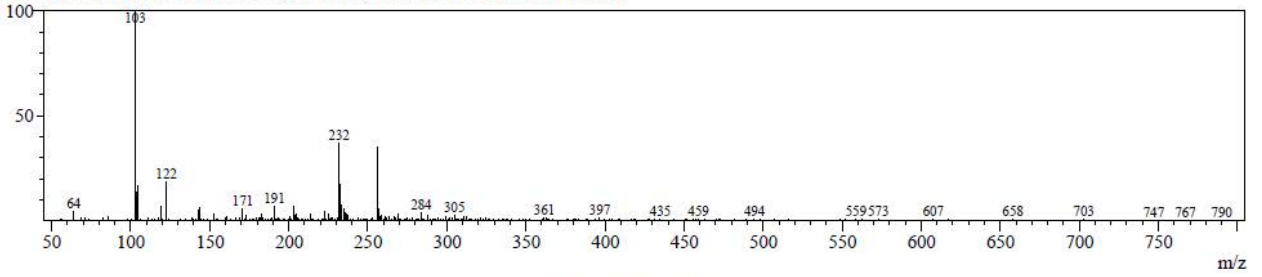






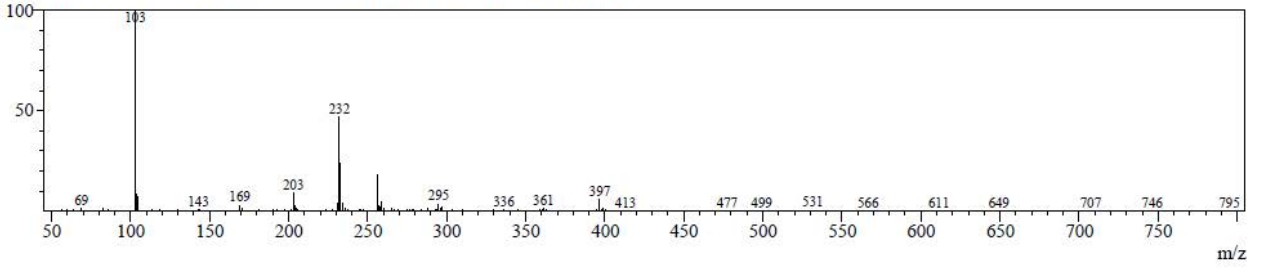
MS Spectrum Graph

Ret.Time:3.800(Scan#:169)
BG Mode:None
Mass Peaks:701 Base Peak:102.90(15745096) Polarity:Pos Segment1 - Event1



MS Spectrum Graph

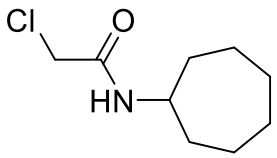
Ret.Time:5.733(Scan#:285)
BG Mode:None
Mass Peaks:709 Base Peak:102.85(12955274) Polarity:Pos Segment1 - Event1



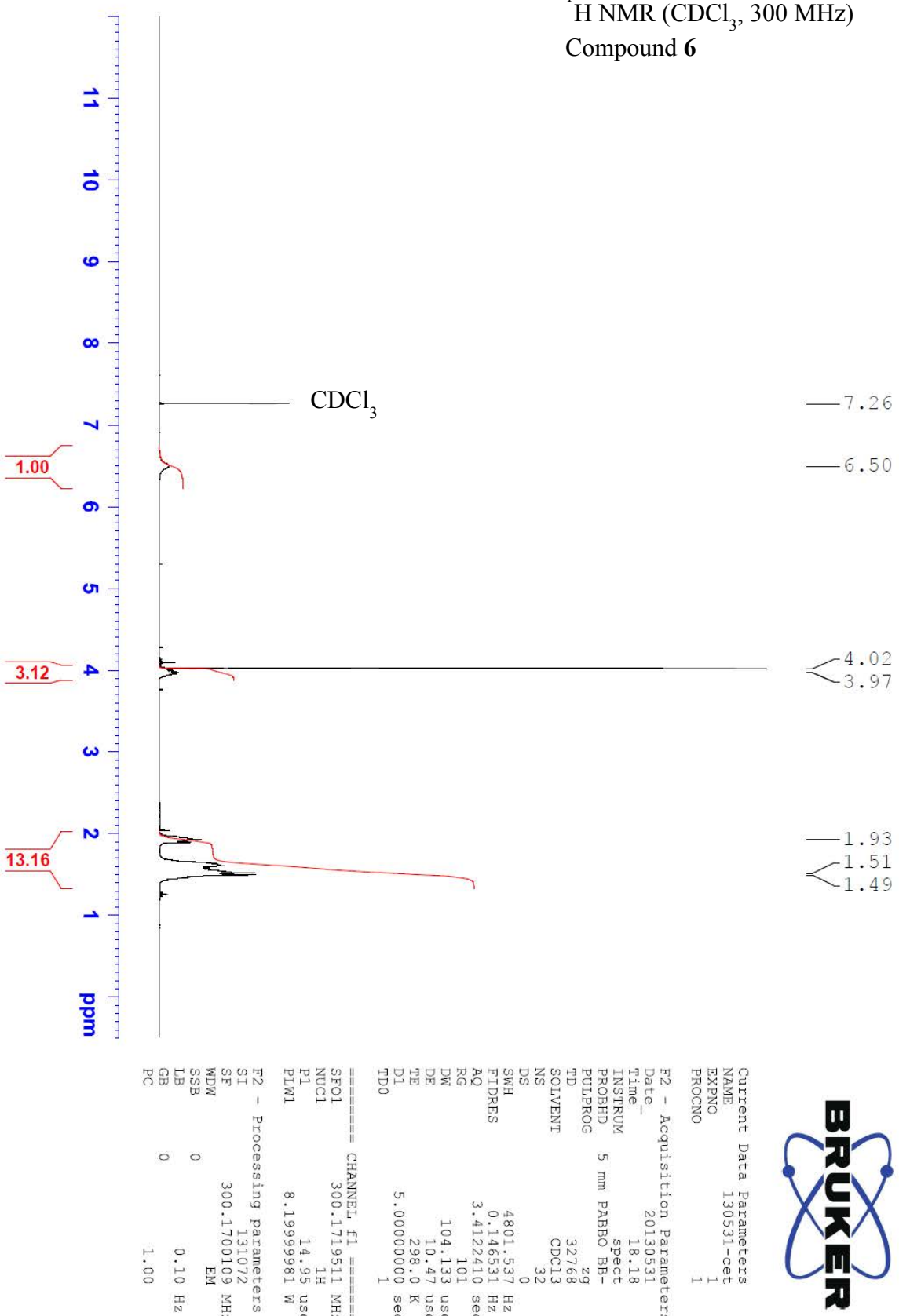
Spectra for Compound 6

^{1}H NMR Compound 6

2-chloro-N-cycloheptylacetamide

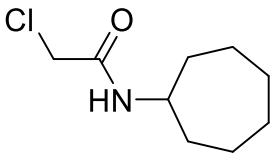


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **6**

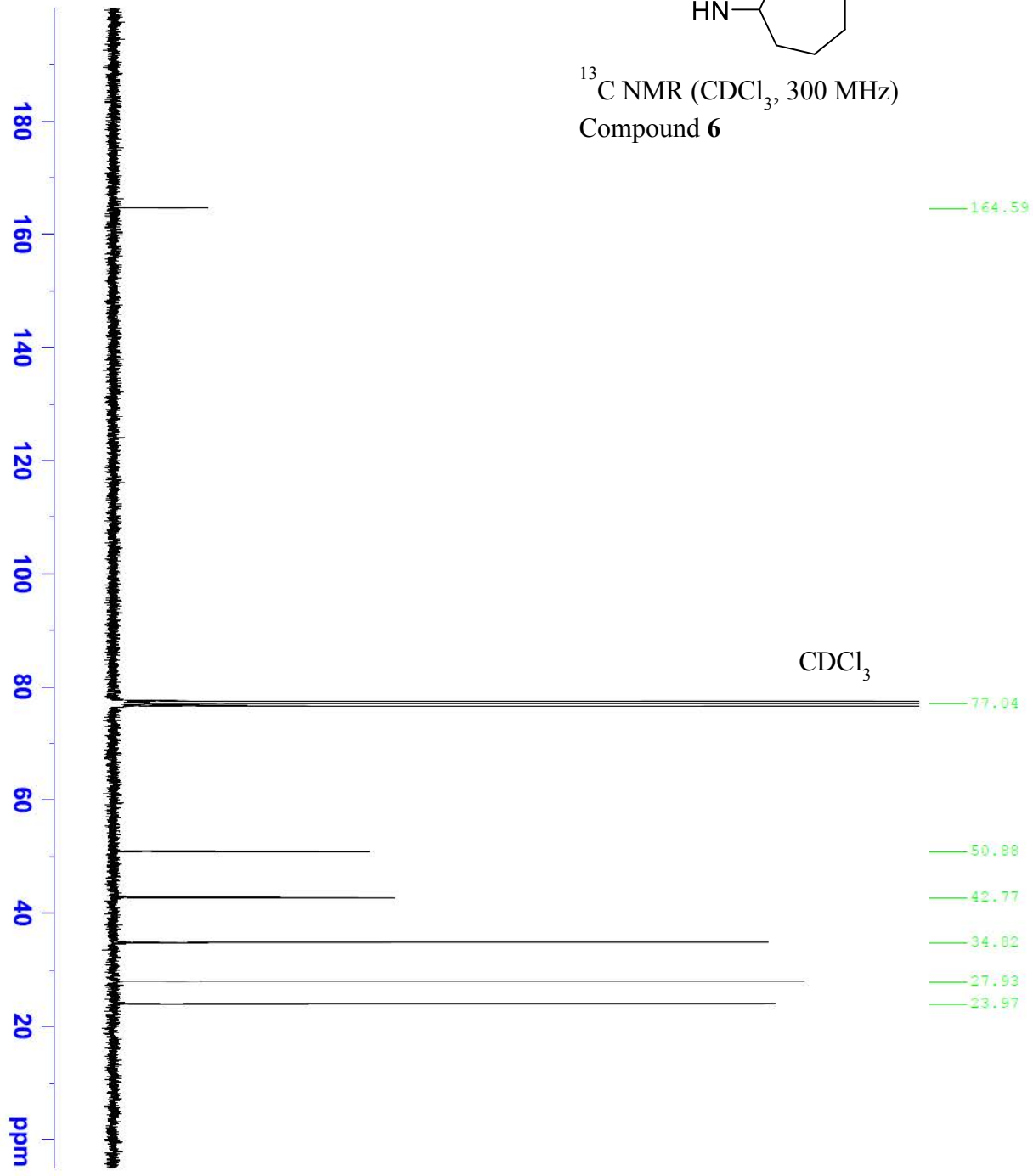


^{13}C NMR Compound 6

2-chloro-N-cycloheptylacetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **6**



Current Data Parameters
NAME 130531-cet
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130531
Time_ 18.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1536
DS 2
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 203
DW 27.733 usec
DE 6.80 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 6

======== CHANNEL f1 ========
SFO1 75.4853543 MHz
NUC1 13C
P1 9.90 usec
PLW1 33.00000000 W

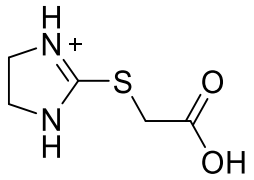
======== CHANNEL f2 ========
SFO2 300.1712007 MHz
NUC2 1H
CPDPRG[2 bi_waltz65_256
PCPD2 90.00 usec
PLW2 8.20349979 W
PLW12 0.22635999 W
PLW13 0.18335000 W

F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

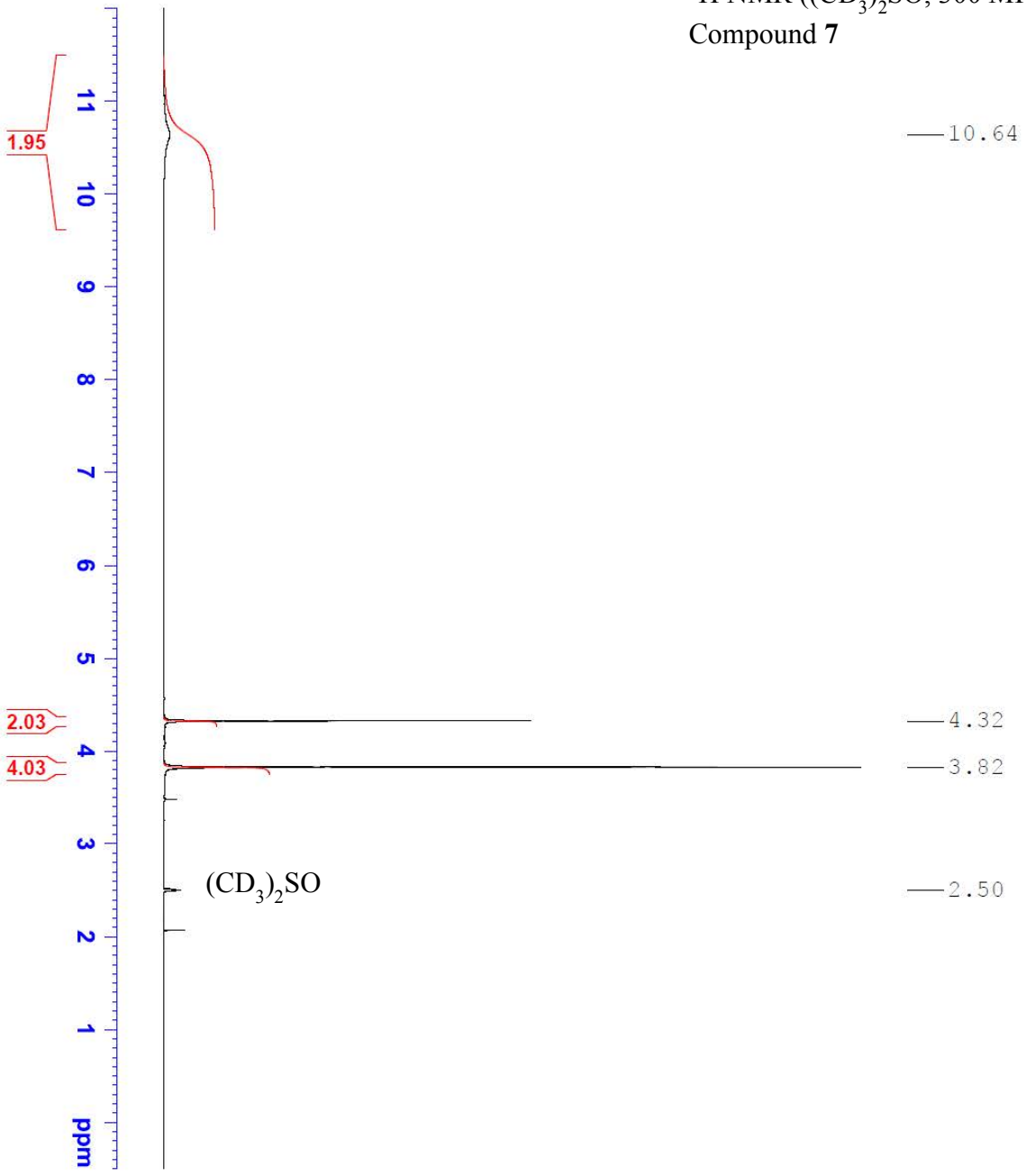
Spectra for Compound 7

^{1}H NMR Compound 7

2-((4,5-dihydro-1H-imidazol-2-yl)thio)acetic acid



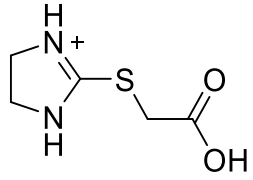
^{1}H NMR ($(CD_3)_2SO$, 300 MHz)
Compound **7**



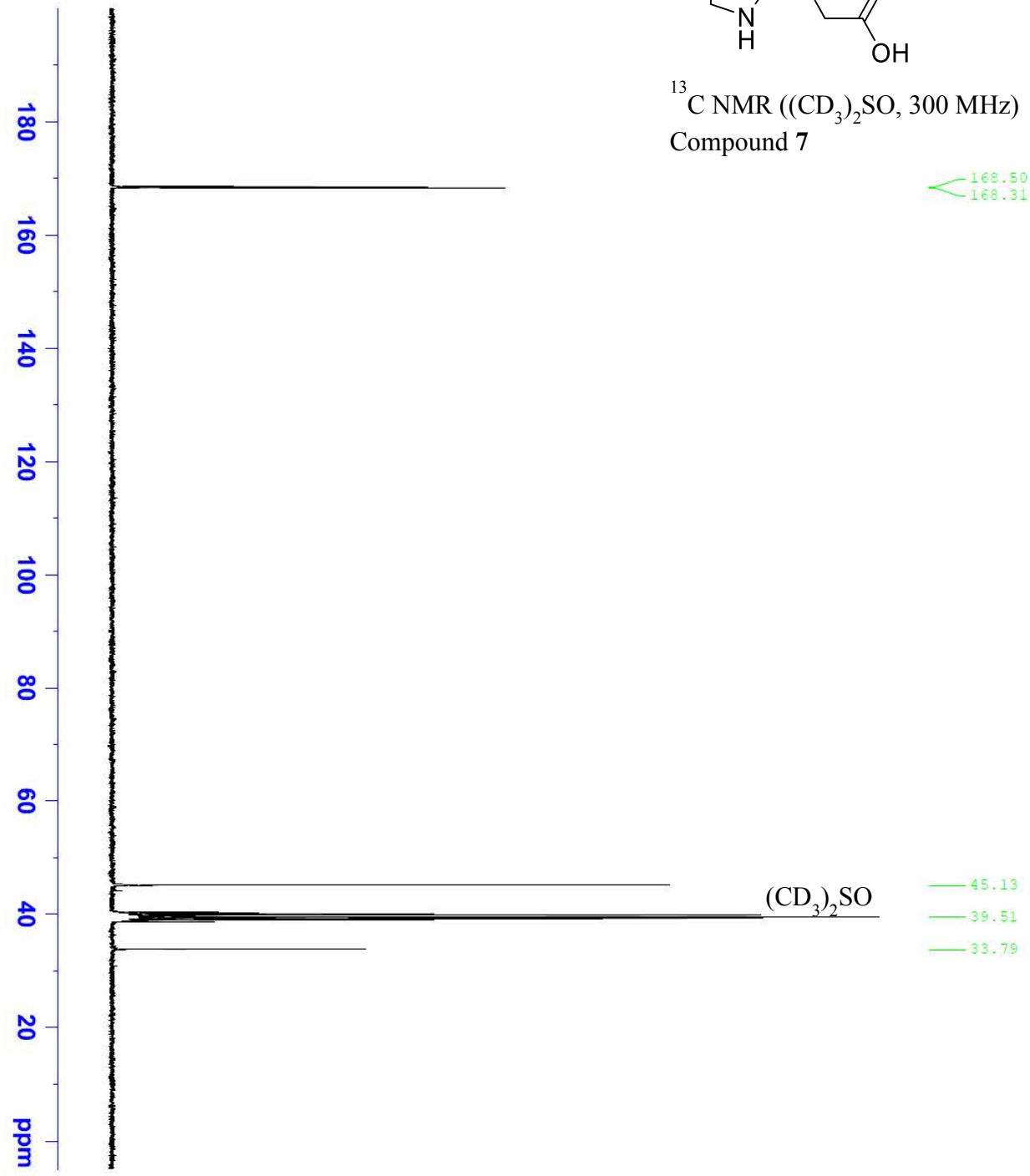

```
Current Data Parameters
NAME                  130206-cet
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                 20130206
Time                     12.42
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                     zg
TD                       32768
SOLVENT                   DMSO
NS                            8
DS                            0
SWH                  4801.537 Hz
FIDRES               0.146531 Hz
AQ                   3.4122410 sec
RG                         45.2
DW                    104.133 usec
DE                     10.47 usec
TE                     298.0 K
D1                    5.00000000 sec
TD0                           1

========== CHANNEL f1 ==========
SFO1                 300.1719511 MHz
NUC1                          1H
P1                         14.95 usec
PLW1                 8.19999981 W

F2 - Processing parameters
SI                          131072
SF                   300.1700078 MHz
WDW                           EM
SSB                            0
LB                          0.10 Hz
GB                             0
PC                          1.00
```

^{13}C NMR Compound 7

2-((4,5-dihydro-1H-imidazol-2-yl)thio)acetic acid



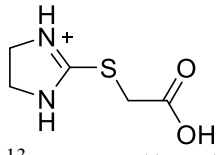
^{13}C NMR ($(CD_3)_2SO$, 300 MHz)
Compound **7**



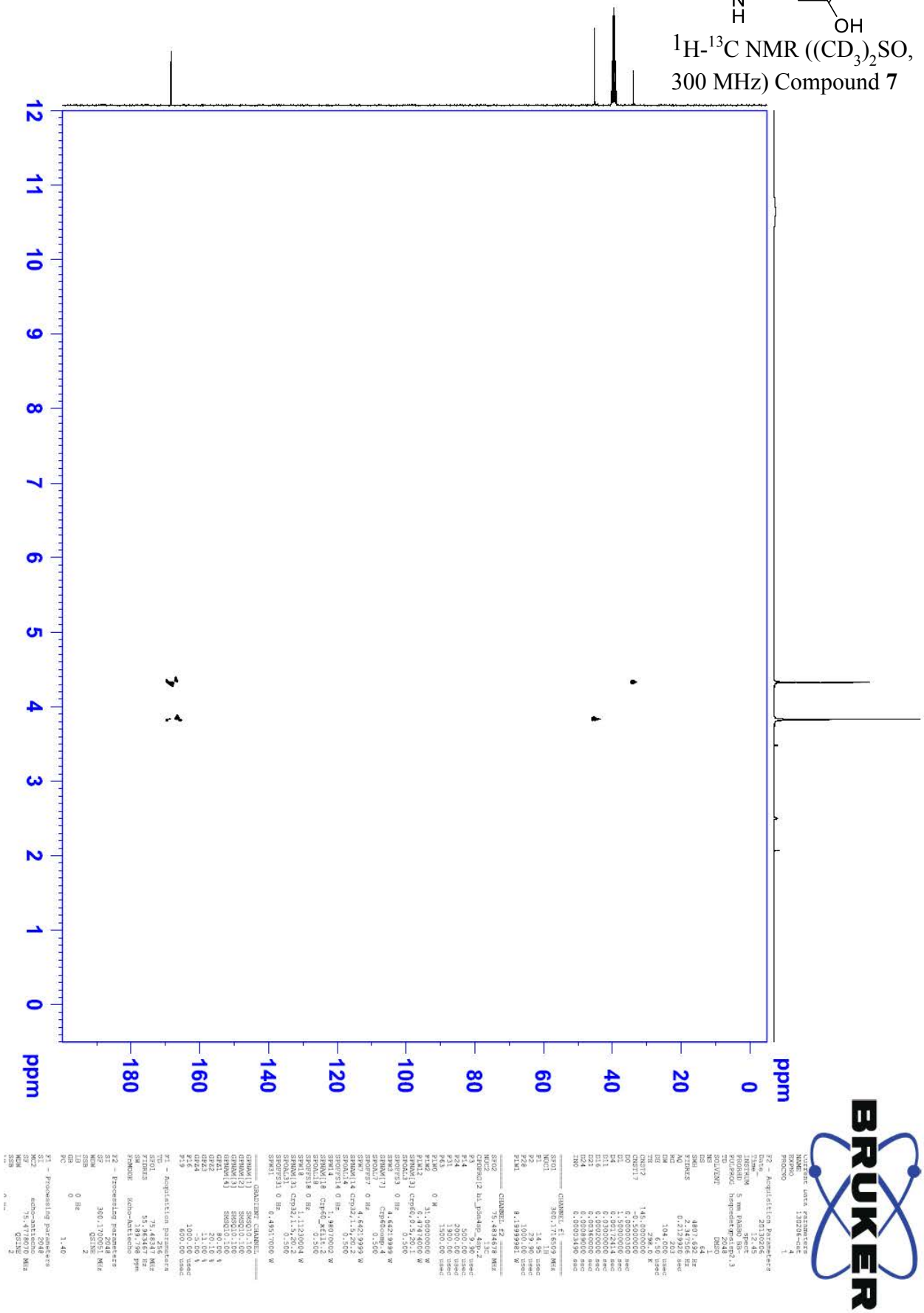

```
Current Data Parameters
NAME                  130203-cet
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20130204
Time_                       7.30
INSTRUM                     spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                   DMSO
NS                        1536
DS                           2
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                          203
DW                       27.733 usec
DE                        6.80 usec
TE                       298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                           6

======== CHANNEL f1 ========
SFO1                 75.4853543 MHz
NUC1                         13C
P1                         9.90 usec
PLW1                33.00000000 W

======== CHANNEL f2 ========
SFO2                 300.1712007 MHz
NUC2                         1H
CPDPRG[2 bi_waltz65_256
PCPD2                     90.00 usec
PLW2                 8.20349979 W
PLW12                0.22635999 W
PLW13                0.18335000 W

F2 - Processing parameters
SI                        32768
SF                  75.4778380 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

^{1}H-^{13}C HSQC Compound 7

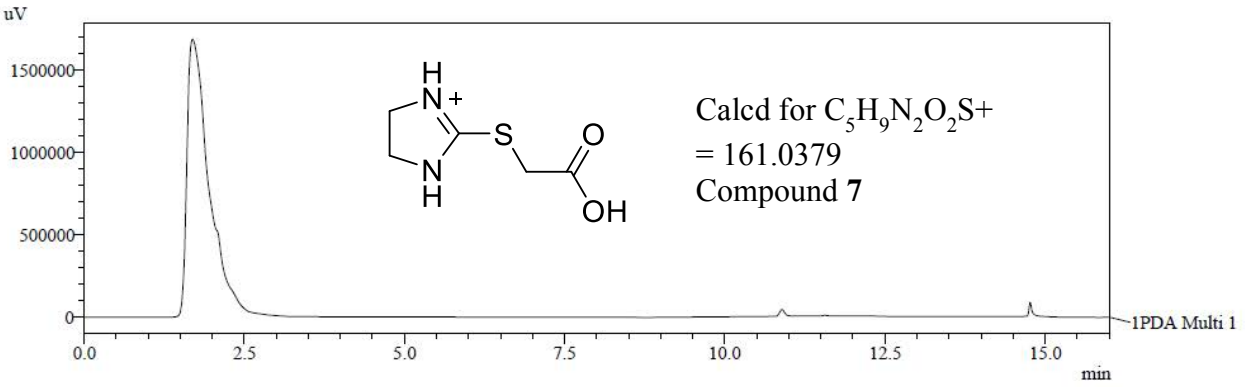
2-((4,5-dihydro-1H-imidazol-2-yl)thio)acetic acid

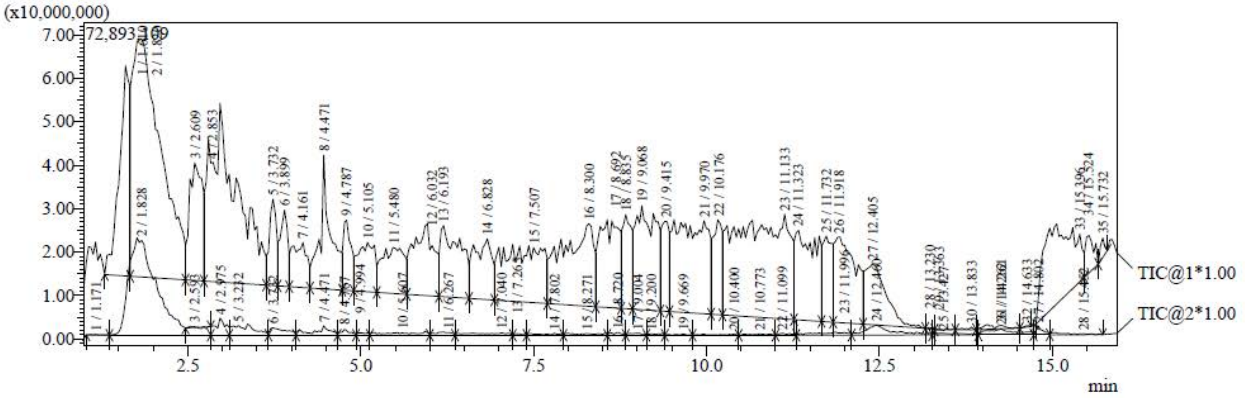


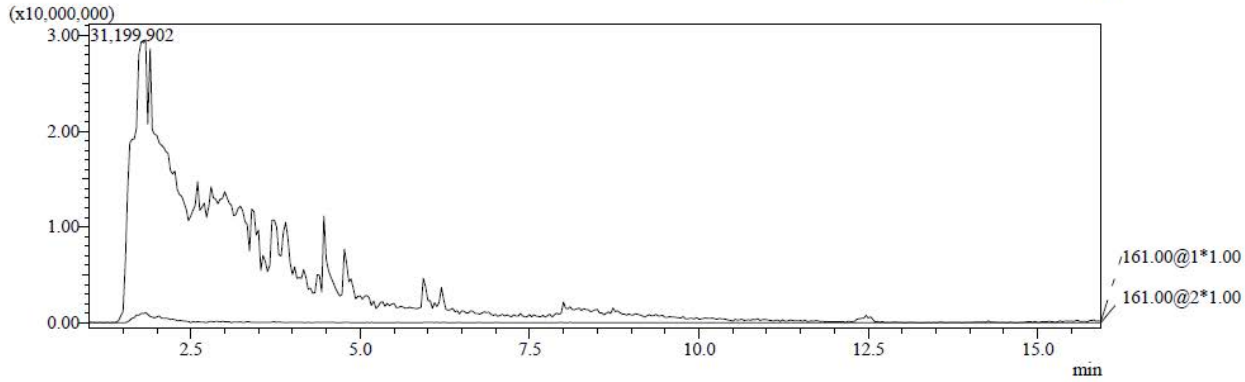
^{1}H-^{13}C NMR ($(CD_3)_2SO$, 300 MHz) Compound **7**



LCMS Compound 7

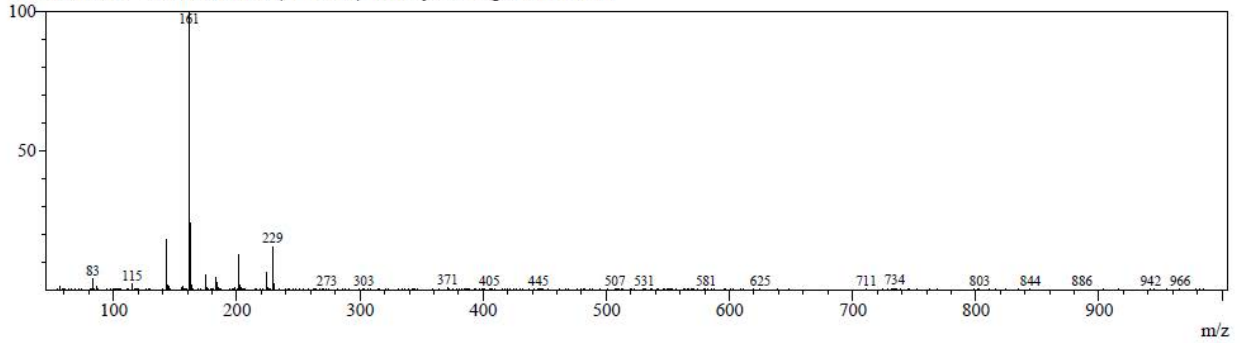




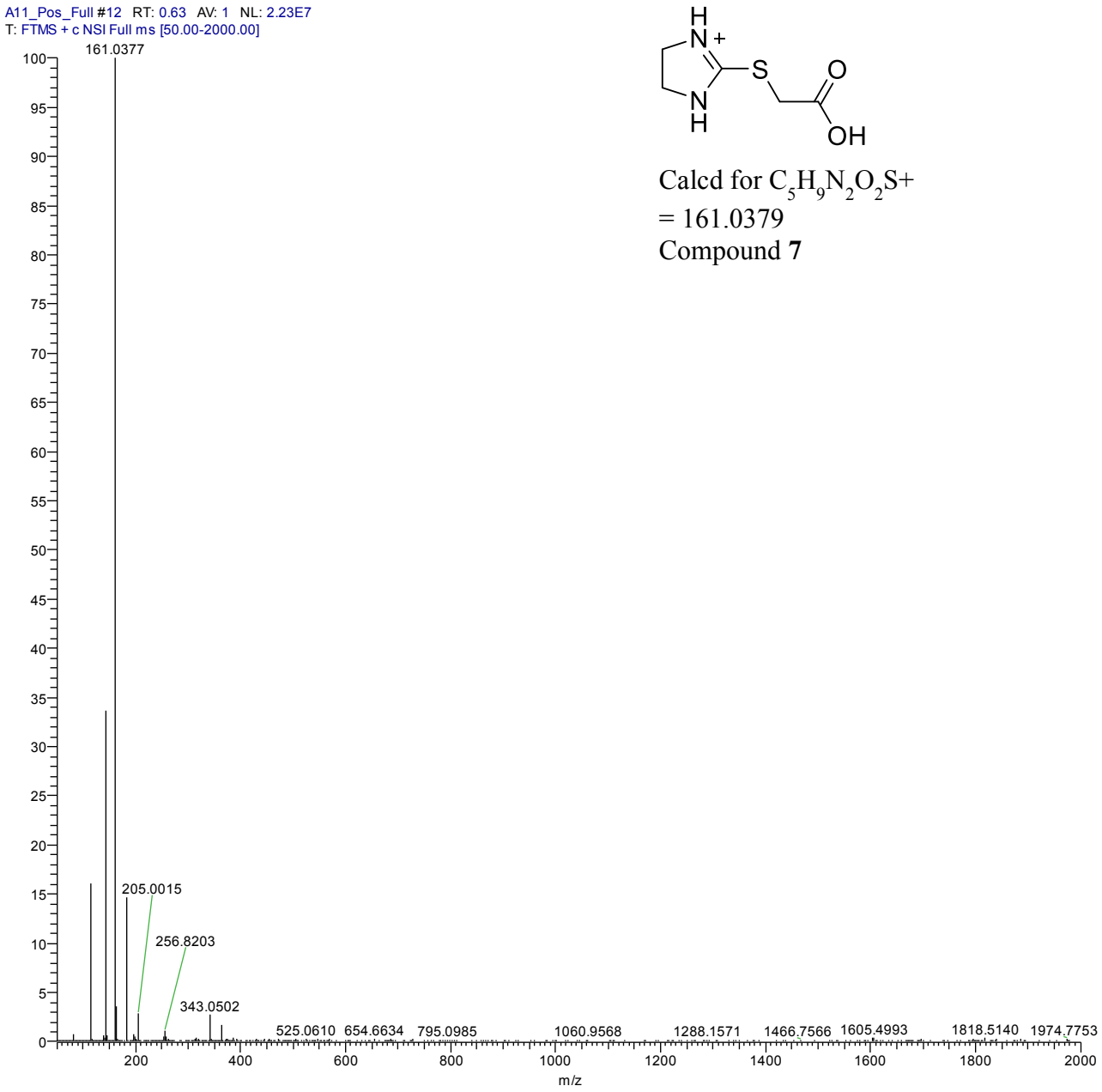


MS Spectrum Graph

Ret.Time:1.800(Scan#:49)
BG Mode:?
Mass Peaks:910 Base Peak:161.20(29340087) Polarity:Pos Segment1 - Event1



HRMS(ESI) Compound 7

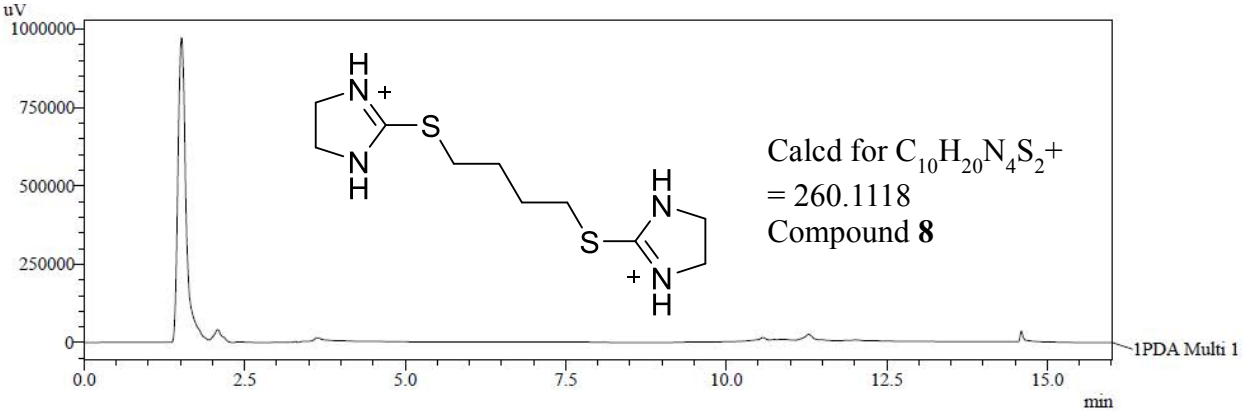


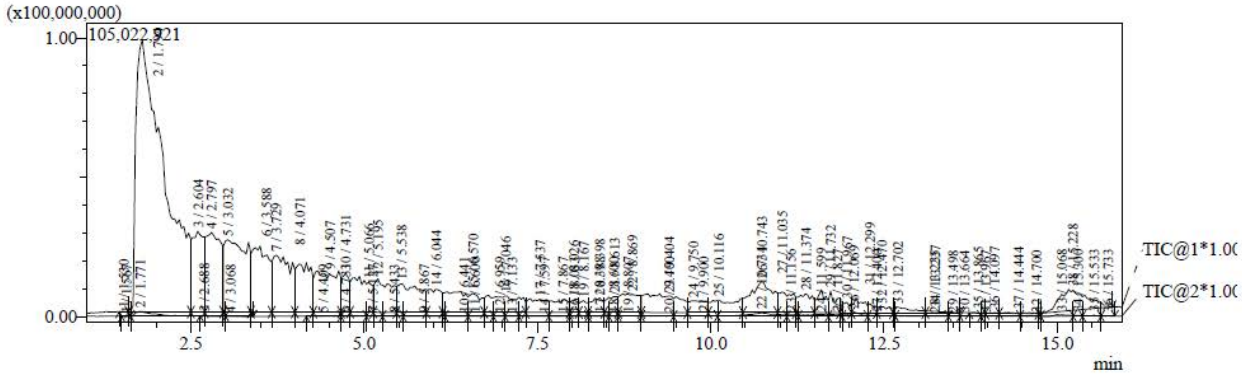
Calcd for $C_5H_9N_2O_2S^+$ = 161.0379

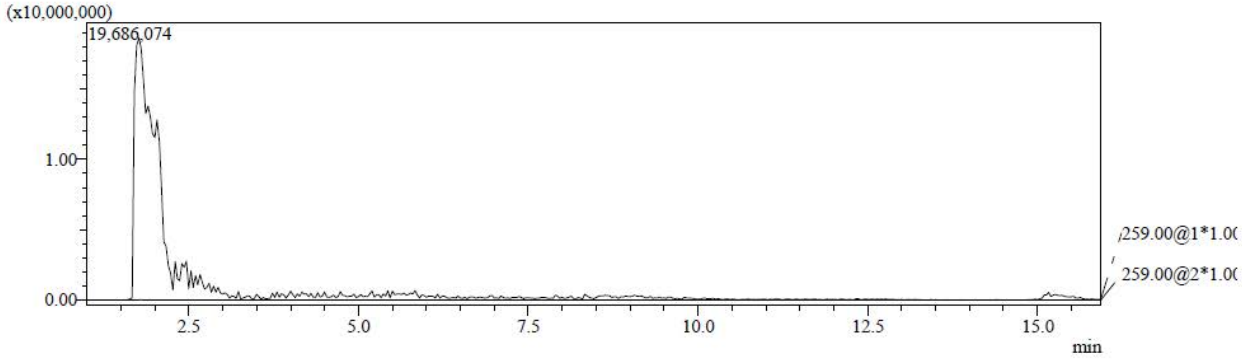
Compound **7**

Spectra for Compound 8

LCMS Compound 8

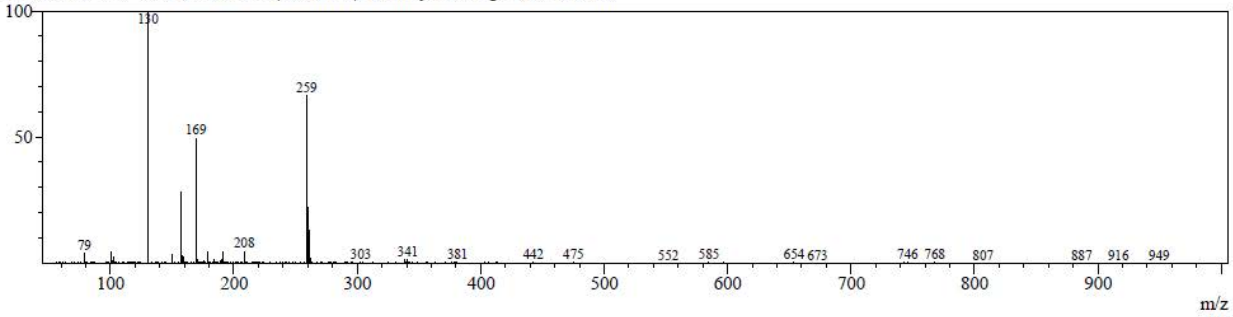




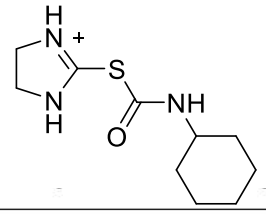


MS Spectrum Graph

Ret.Time:1.767(Scan#:47)
BG Mode:?
Mass Peaks:921 Base Peak:130.30(28046191) Polarity:Pos Segment1 - Event1

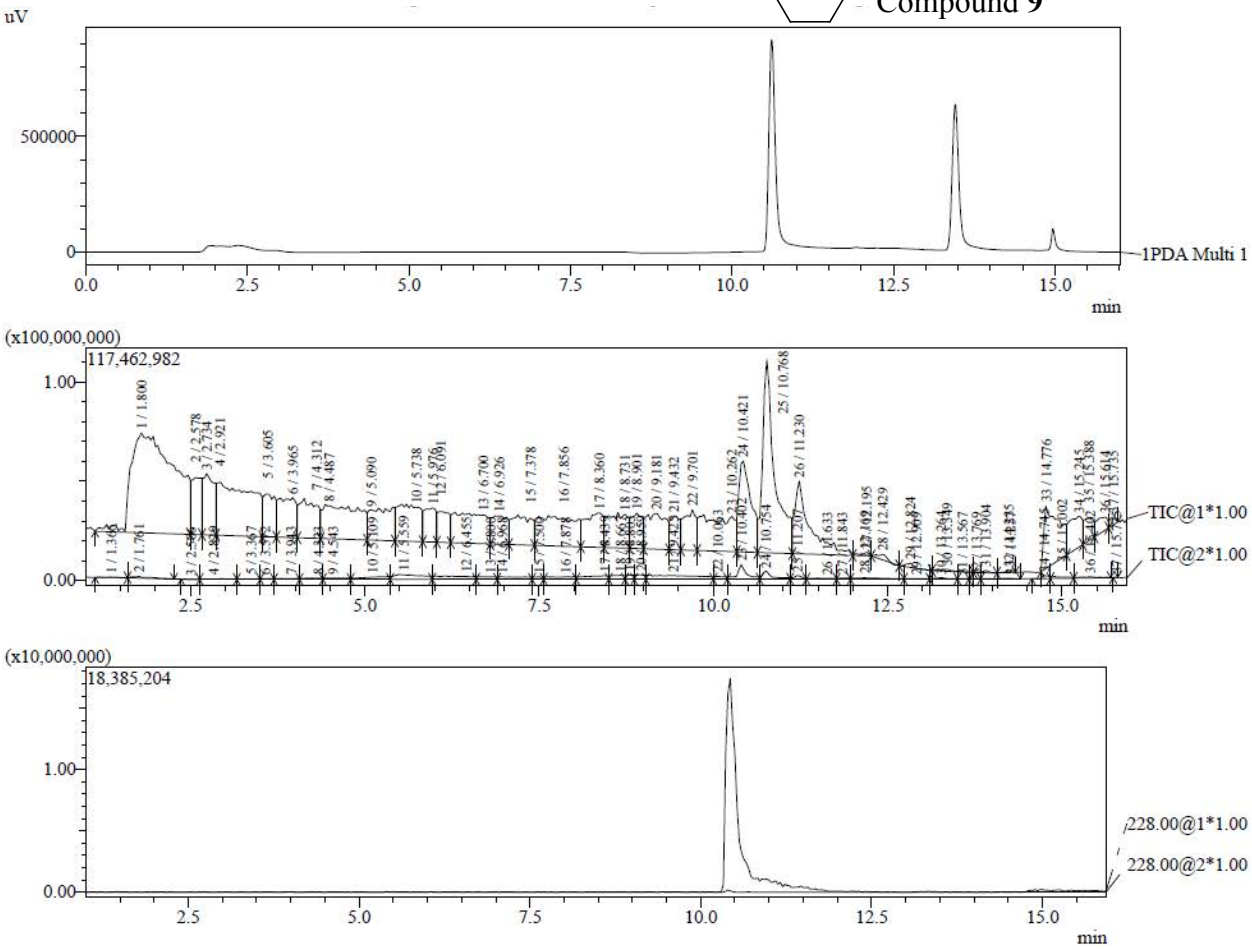


Spectra for Compound 9



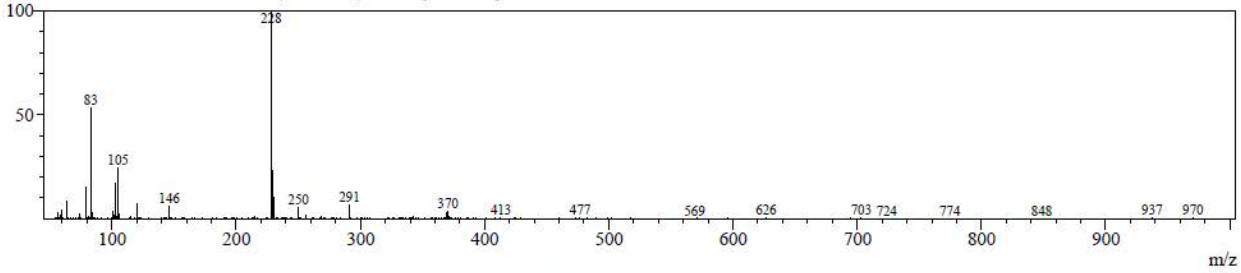
Calcd for $C_{10}H_{18}N_3OS^+$ = 228.1165
Compound **9**

LCMS Compound 9



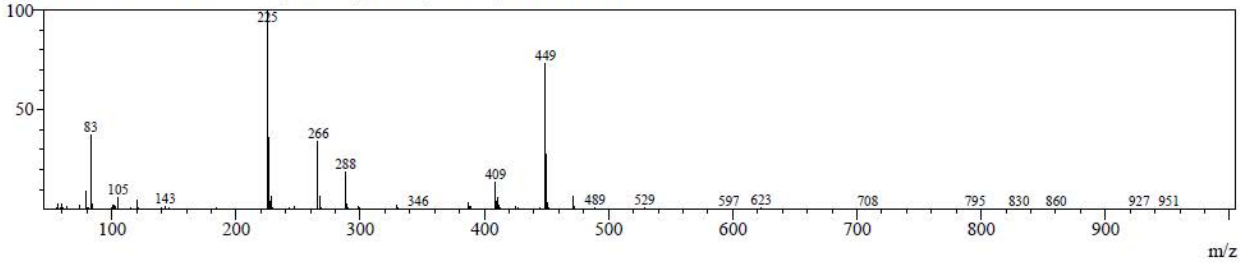
MS Spectrum Graph

Ret.Time:10.400(Scan#:565)
BG Mode:?
Mass Peaks:909 Base Peak:227.90(16152514) Polarity:Pos Segment1 - Event1



MS Spectrum Graph

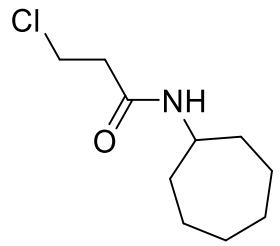
Ret.Time:10.733(Scan#:585)
BG Mode:?
Mass Peaks:919 Base Peak:225.10(20105695) Polarity:Pos Segment1 - Event1



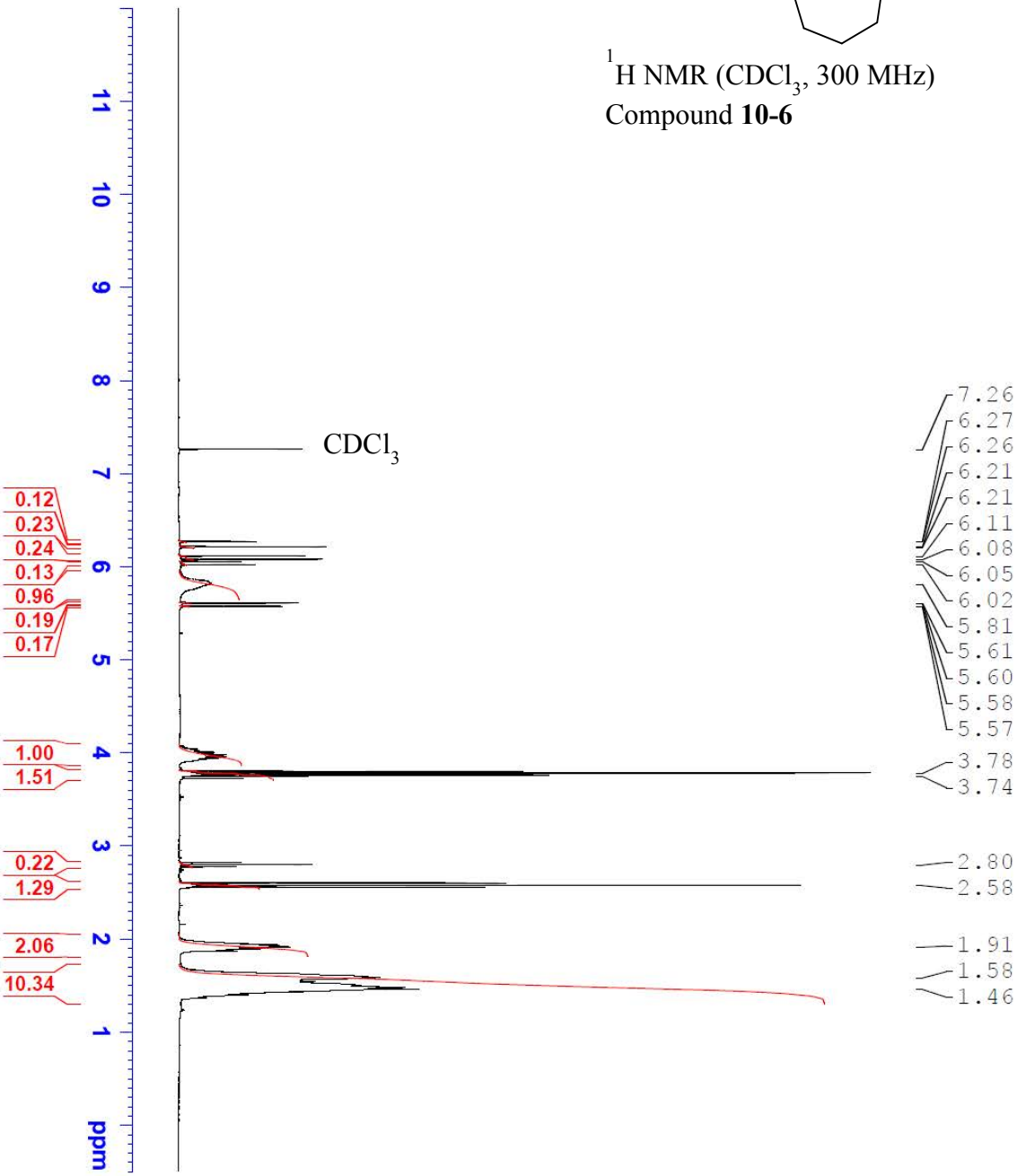
Spectra for Compound 10

^{1}H NMR Compound 10-6

3-chloro-N-cycloheptylpropanamide



^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **10-6**



Current Data Parameters
NAME 130830-cet
EXPNO 5
PROCNO 1

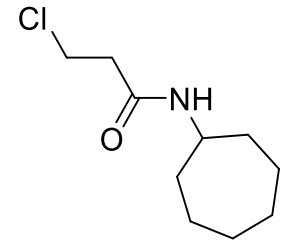
F2 - Acquisition Parameters
Date_ 20130830
Time_ 20.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4801.537 Hz
FIDRES 0.146531 Hz
AQ 3.4122410 sec
RG 36
DW 104.133 usec
DE 10.47 usec
TE 298.0 K
D1 5.00000000 sec
TD0 1

========== CHANNEL f1 ==========
SFO1 300.1719511 MHz
NUC1 1H
P1 14.95 usec
PLW1 8.19999981 W

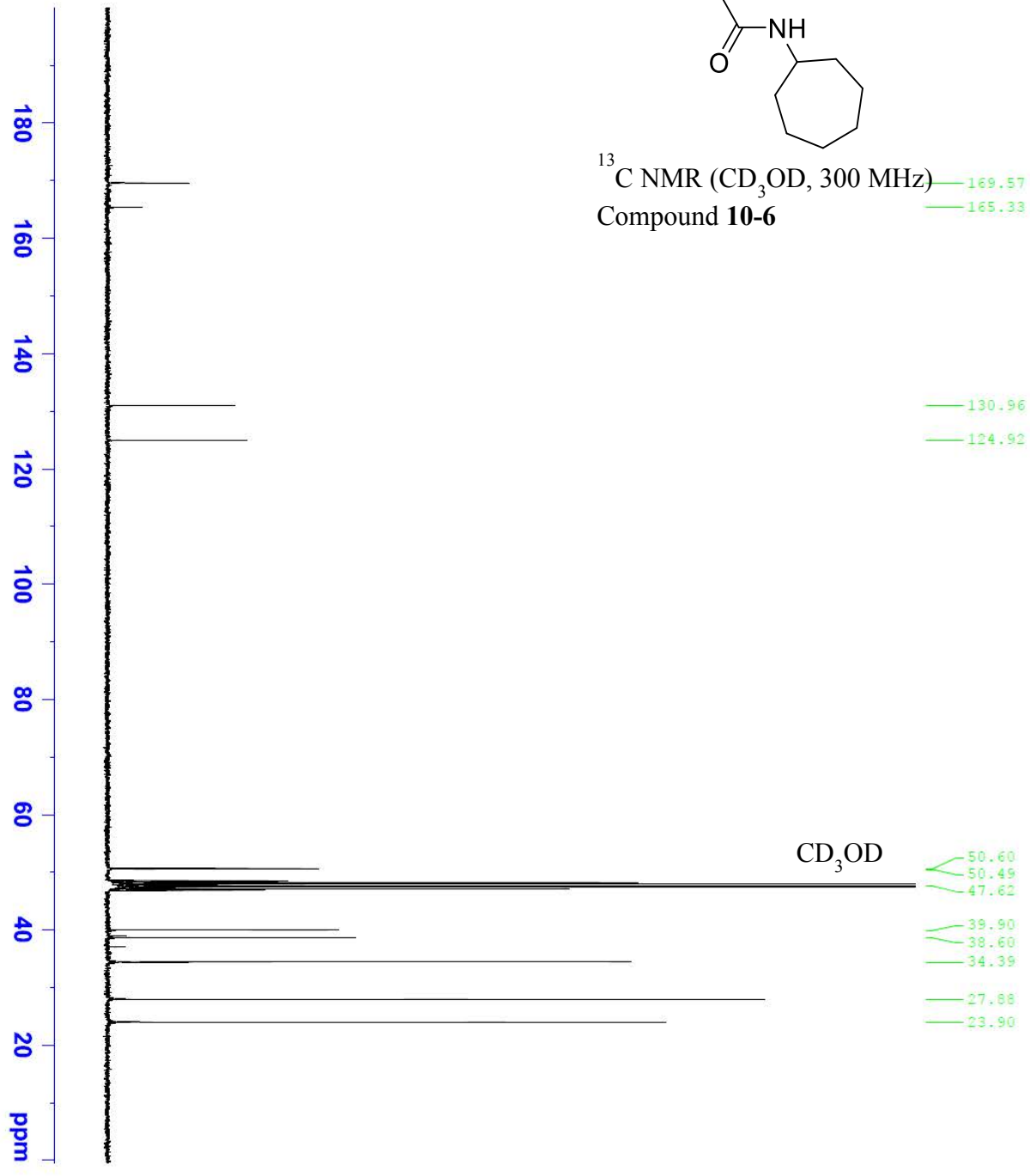
F2 - Processing parameters
SI 131072
SF 300.1700072 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

^{13}C NMR Compound 10-6

3-chloro-N-cycloheptylpropanamide

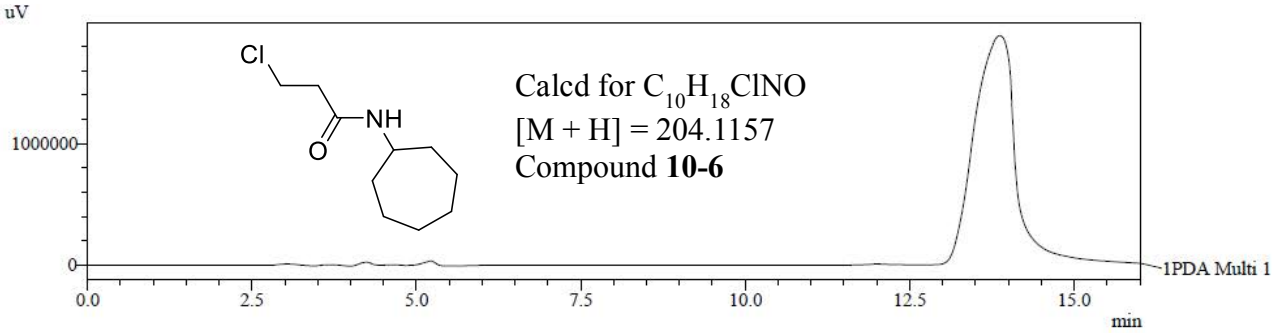


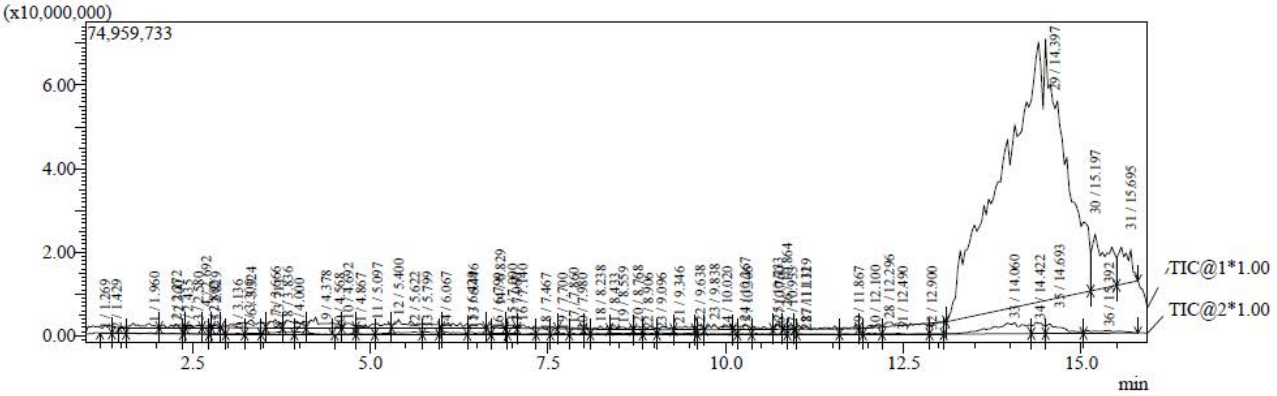
^{13}C NMR (CD_3OD, 300 MHz)
Compound **10-6**

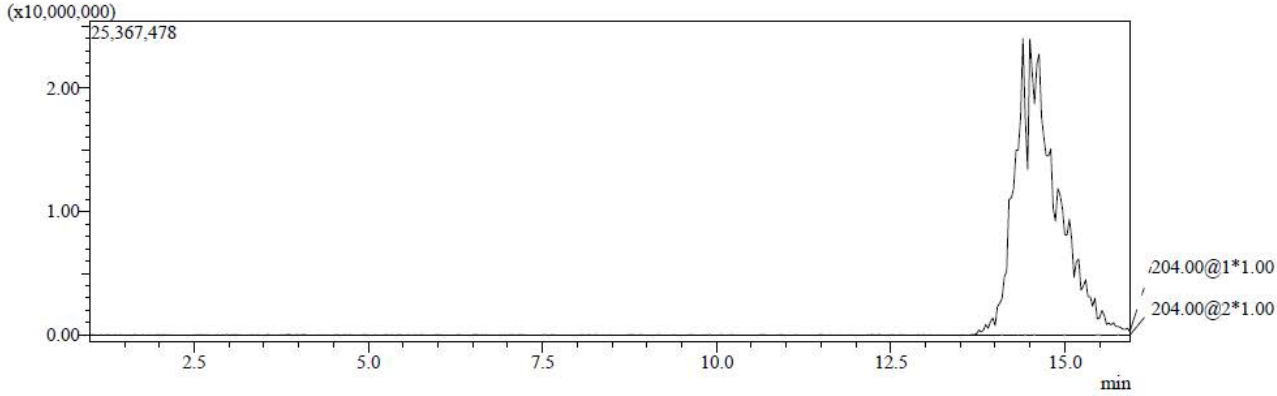



```
Current Data Parameters
NAME                 141130-cet
EXPNO                        12
PROCNO                        1

F2 - Acquisition Parameters
Date_               20141201
Time_                   3.05
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT               MeOD
NS                     1536
DS                        2
SWH               18028.846 Hz
FIDRES                0.275098 Hz
AQ                1.8175317 sec
RG                      203
DW                   27.733 usec
DE                    6.80 usec
TE                   298.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                       6

========== CHANNEL f1 ========
SFO1             75.4853543 MHz
NUC1                      13C
P1                   9.90 usec
PLW1            33.00000000 W

========== CHANNEL f2 ========
SFO2            300.1712007 MHz
NUC2                       1H
CPDPRG[2 bi_waltz65_256
PCPD2                90.00 usec
PLW2            8.20349979 W
PLW12           0.27774000 W
PLW13           0.22497000 W

F2 - Processing parameters
SI                       32768
SF              75.4778070 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

LCMS Compound 10-6

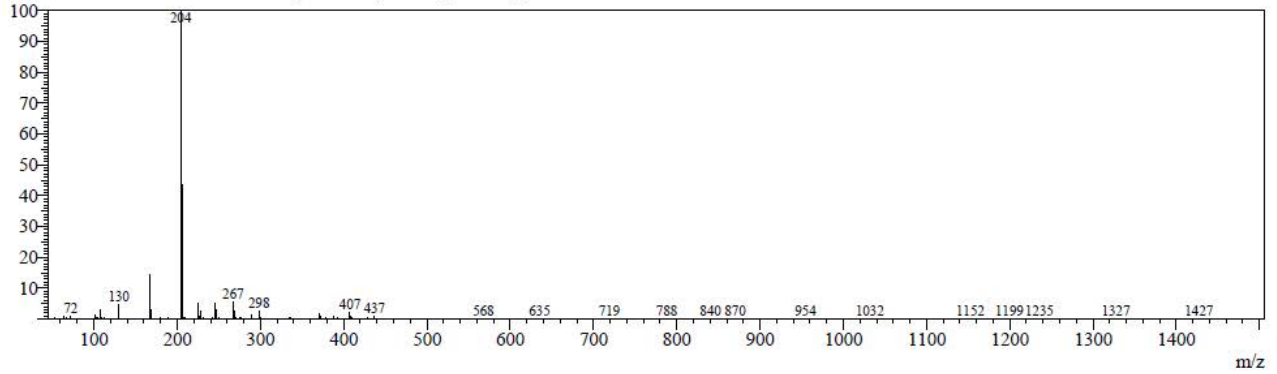




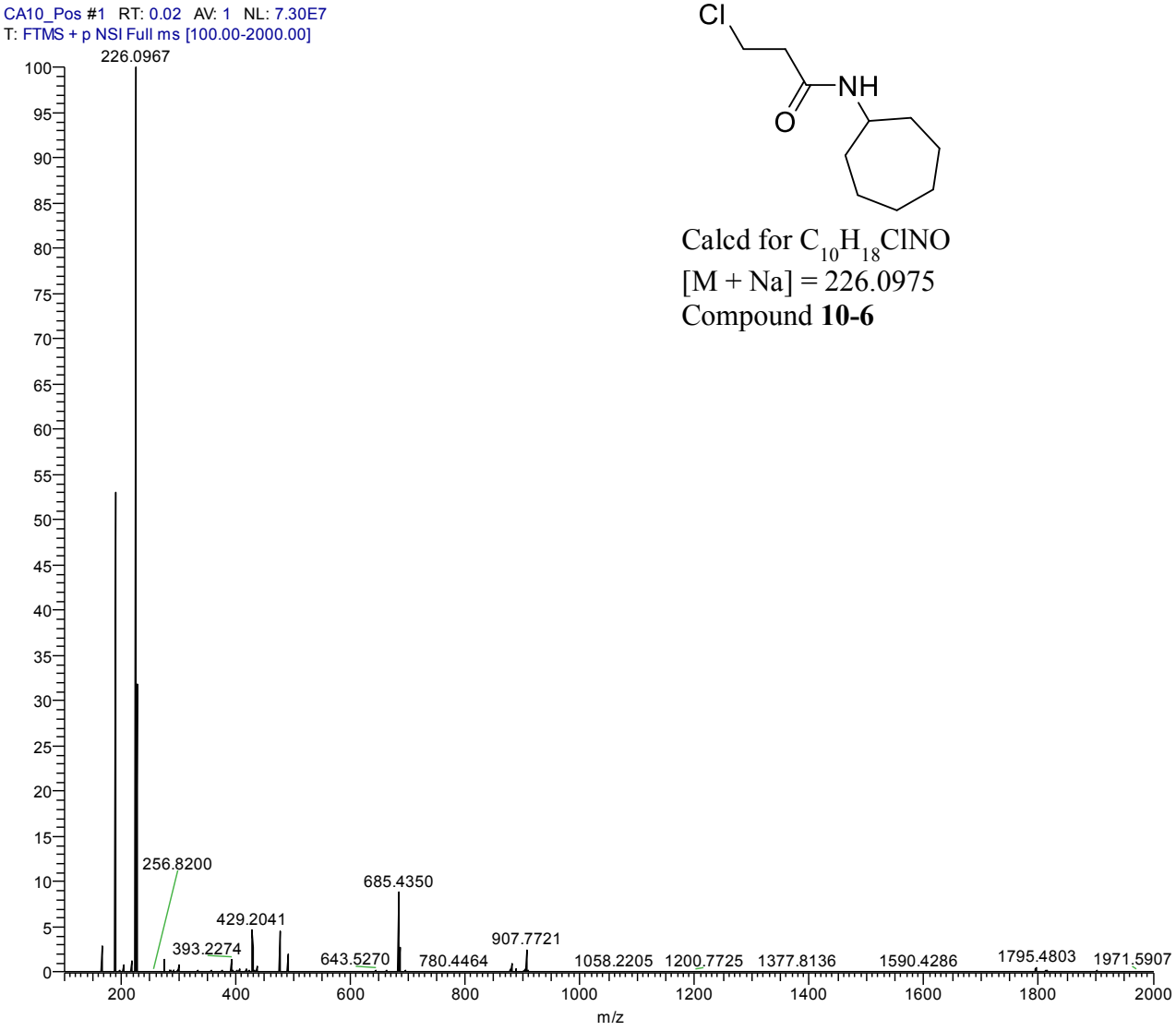


MS Spectrum Grapl

Ret.Time:14.633(Scan#:819)
BG Mode:None
Mass Peaks:1251 Base Peak:204.05(22754668) Polarity:Pos Segment1 - Event1



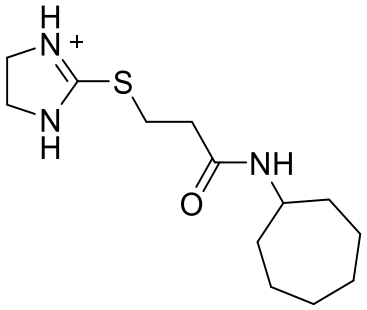
HRMS(ESI) Compound 10-6



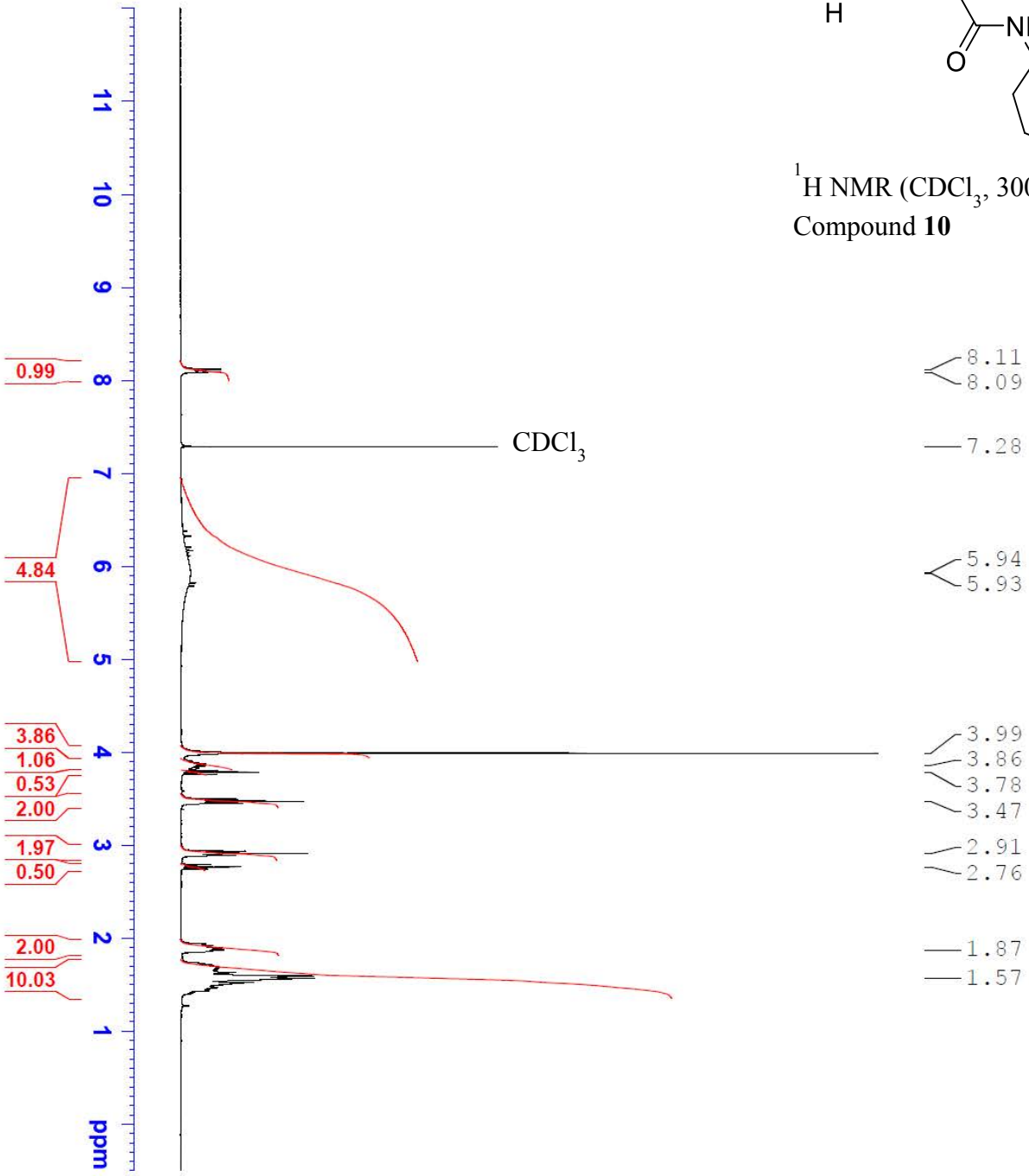
Calcd for $C_{10}H_{18}ClNO$
[M + Na] = 226.0975
Compound **10-6**

^{1}H NMR Compound 10

3-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cycloheptylpropanamide



^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **10**



Current Data Parameters
NAME 141207-cet
EXPNO 17
PROCNO 1

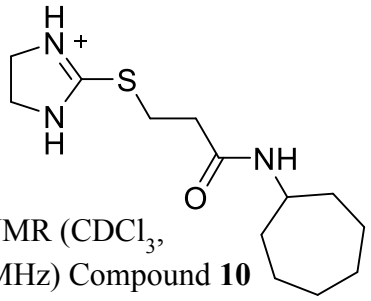
F2 - Acquisition Parameters
Date_ 20141207
Time_ 22.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 4801.537 Hz
FIDRES 0.146531 Hz
AQ 3.4122410 sec
RG 114
DW 104.133 usec
DE 9.44 usec
TE 298.0 K
D1 5.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 300.1719511 MHz
NUC1 1H
P1 16.56 usec
PLW1 8.19999981 W

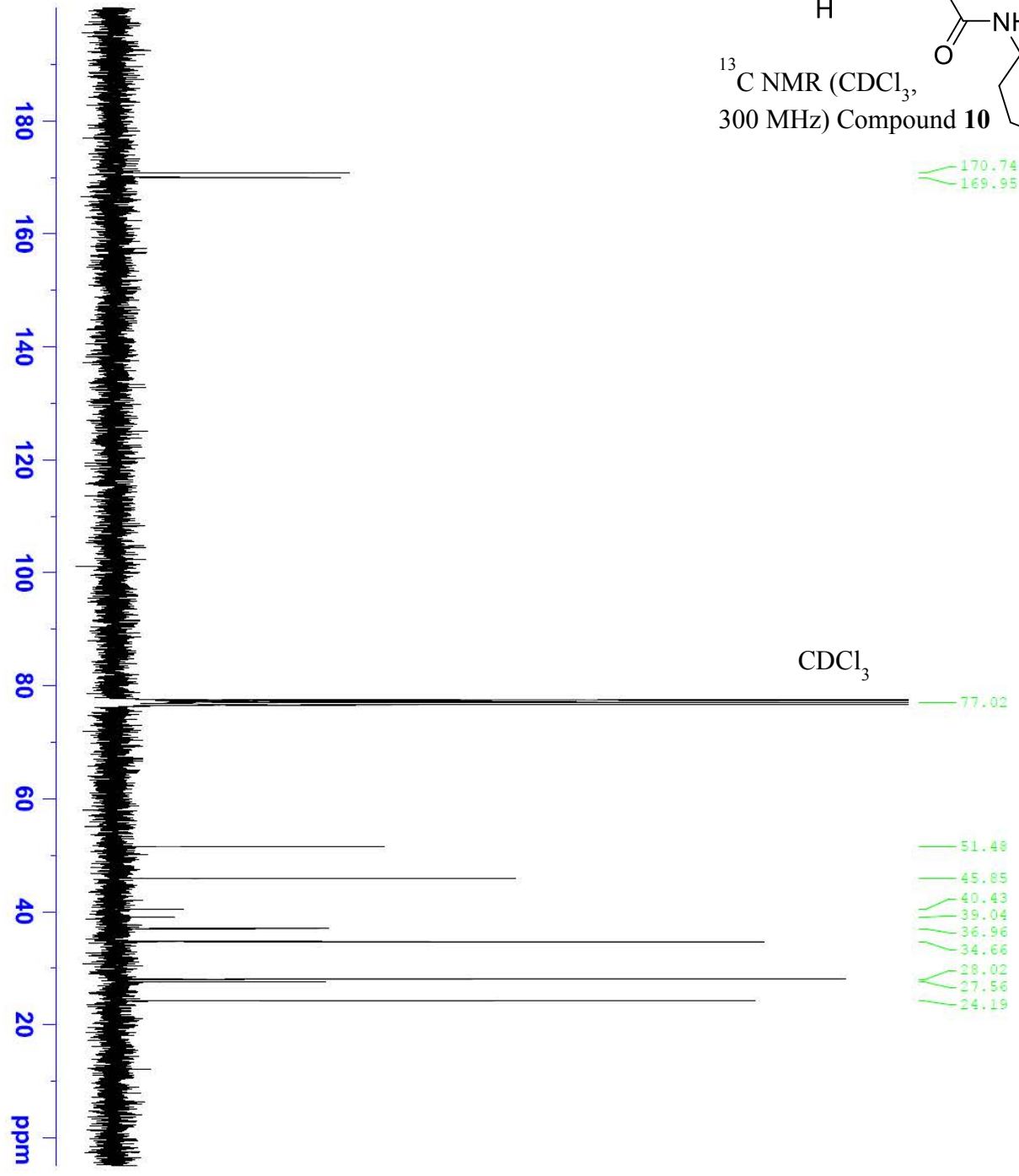
F2 - Processing parameters
SI 131072
SF 300.1700000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

^{13}C NMR Compound 10

3-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cycloheptylpropanamide



^{13}C NMR ($CDCl_3$, 300 MHz) Compound **10**



Current Data Parameters
NAME 141207-cet
EXPNO 18
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141207
Time_ 22.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1536
DS 2
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 203
DW 27.733 usec
DE 6.80 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 6

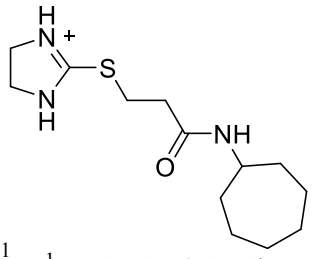
======== CHANNEL f1 ========
SFO1 75.4853543 MHz
NUC1 13C
P1 9.90 usec
PLW1 33.00000000 W

======== CHANNEL f2 ========
SFO2 300.1712007 MHz
NUC2 1H
CPDPRG[2 bi_waltz65_256
PCPD2 90.00 usec
PLW2 8.20349979 W
PLW12 0.27774000 W
PLW13 0.22497000 W

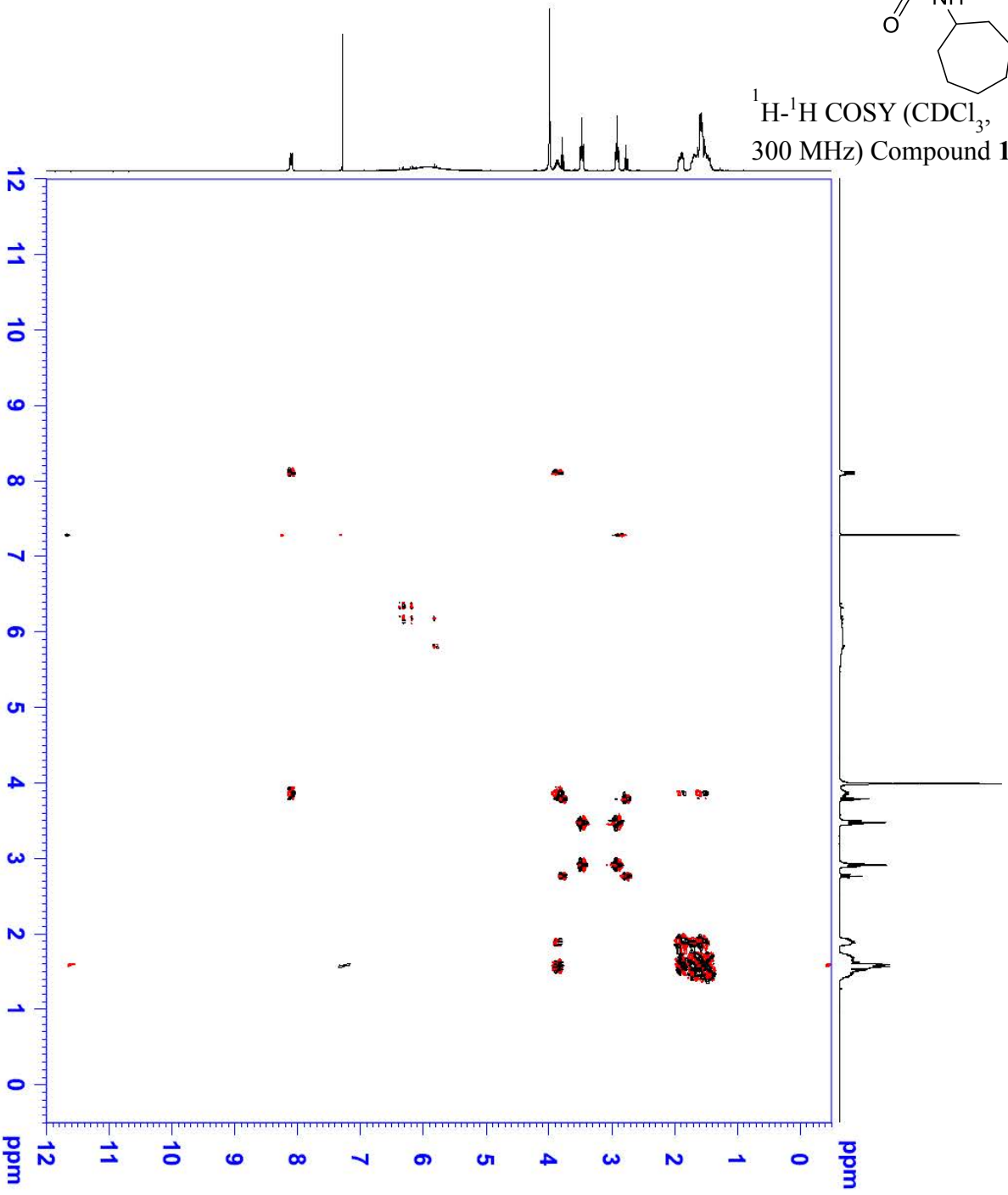
F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{1}H-^{1}H COSY Compound 10

3-((4,5-dihydro-1H-imidazol-2-yl)thio)-N cycloheptylpropanamide



^{1}H-^{1}H COSY ($CDCl_3$, 300 MHz) Compound **10**

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Current Data Parameters
NAME                  141207-cet
EXPNO                         19
PROCNO                         1

F2 - Acquisition Parameters
Date_              20141208
Time_                  0.01
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             cosygpmfph
TD                       2048
SOLVENT                 CDCl3
NS                          1
DS                         16
SWH                3690.945 Hz
FIDRES             1.802219 Hz
AQ                 0.2774357 sec
RG                        114
DW                    135.467 usec
DE                      6.50 usec
TE                     298.0 K
D0                  0.00011442 sec
D1                  2.00000000 sec
D13                 0.00000400 sec
D16                 0.00020000 sec
IN0                 0.00027100 sec

======== CHANNEL f1 ========
SFO1          300.1716683 MHz
NUC1                      1H
P1                     16.56 usec
P2                     33.12 usec
PLW1            8.19999981 W

====== GRADIENT CHANNEL =====
GPNAM[1]          SMSQ10.100
GPNAM[2]          SMSQ10.100
GPZ1                   10.00 %
GPZ2                   20.00 %
P16                  1000.00 usec

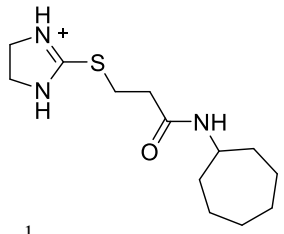
F1 - Acquisition parameters
TD                        256
SFO1          300.1717 MHz
FIDRES             14.414207 Hz
SW                 12.293 ppm
FnMODE          States-TPPI

F2 - Processing parameters
SI                       2048
SF            300.1700000 MHz
WDW                     QSINE
SSB                         2
LB                     0 Hz
GB                          0
PC                       1.40

F1 - Processing parameters
SI                       2048
MC2             States-TPPI
SF            300.1700000 MHz
WDW                     QSINE
SSB                         2
LB                     0 Hz
GB                          0
```

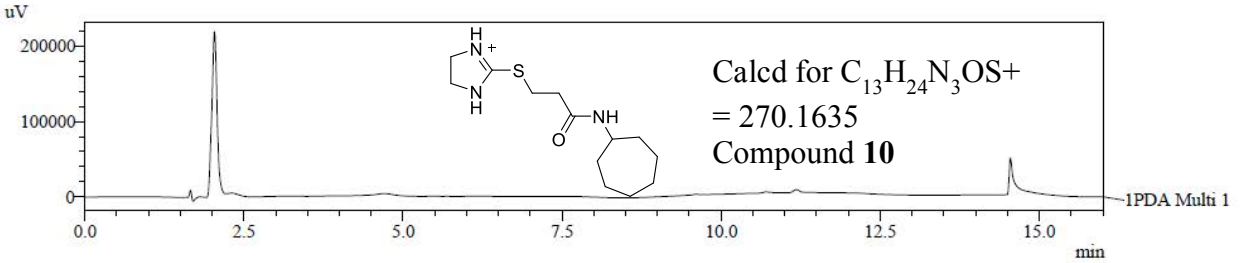
^{1}H-^{13}C HSQC Compound 10

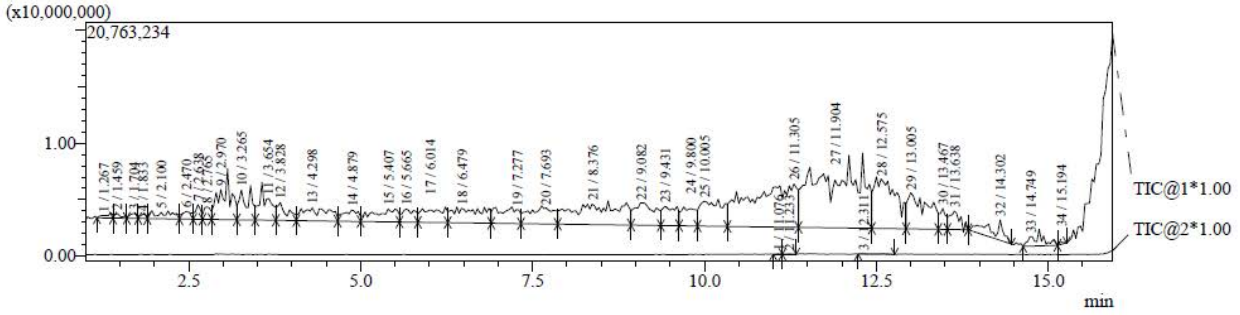
3-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cycloheptylpropanamide

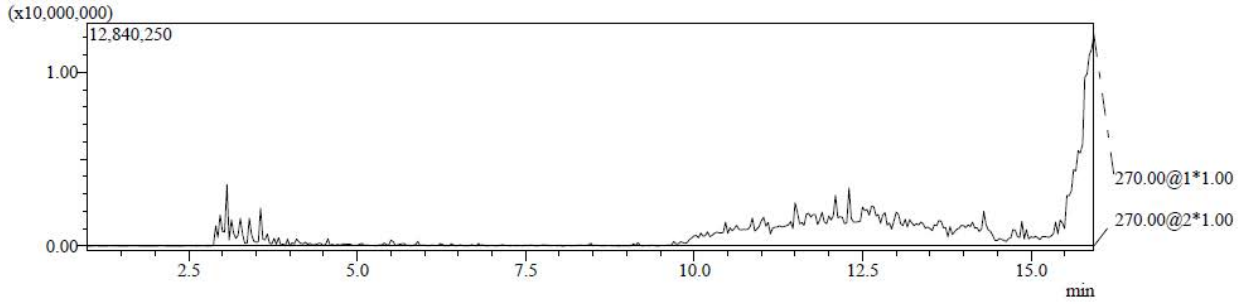


^{1}H-^{13}C HSQC ($CDCl_3$, 300MHz) Compound **10**

LCMS Compound 10

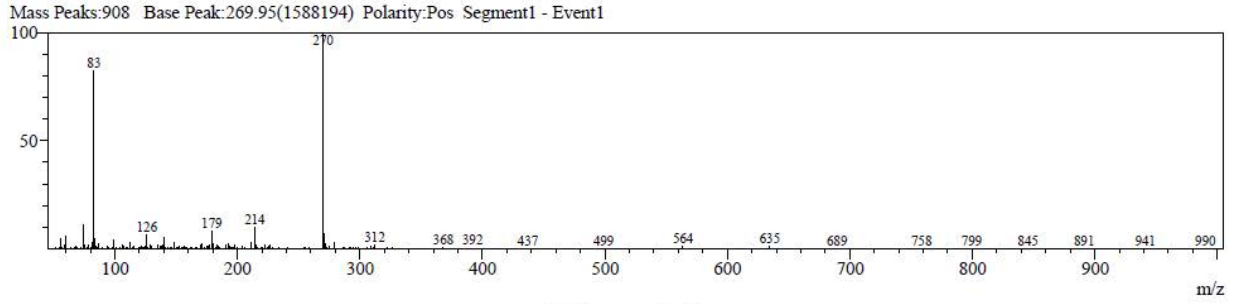






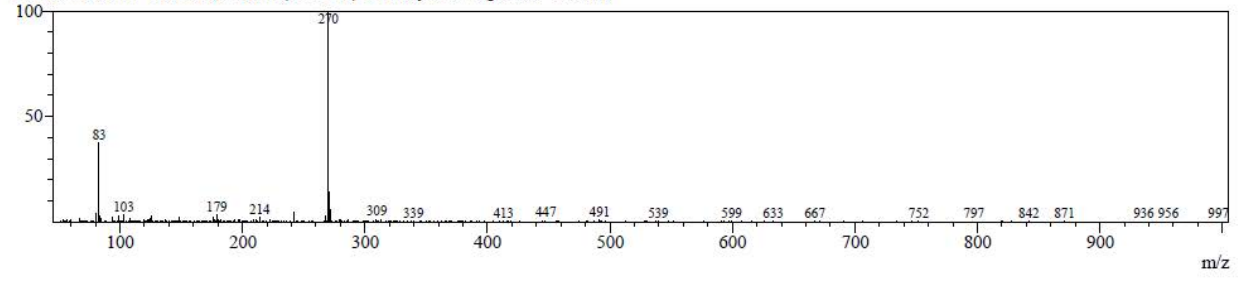
MS Spectrum Graph

Ret.Time:3.267(Scan#:137)
BG Mode:?
Mass Peaks:908 Base Peak:269.95(1588194) Polarity:Pos Segment1 - Event1

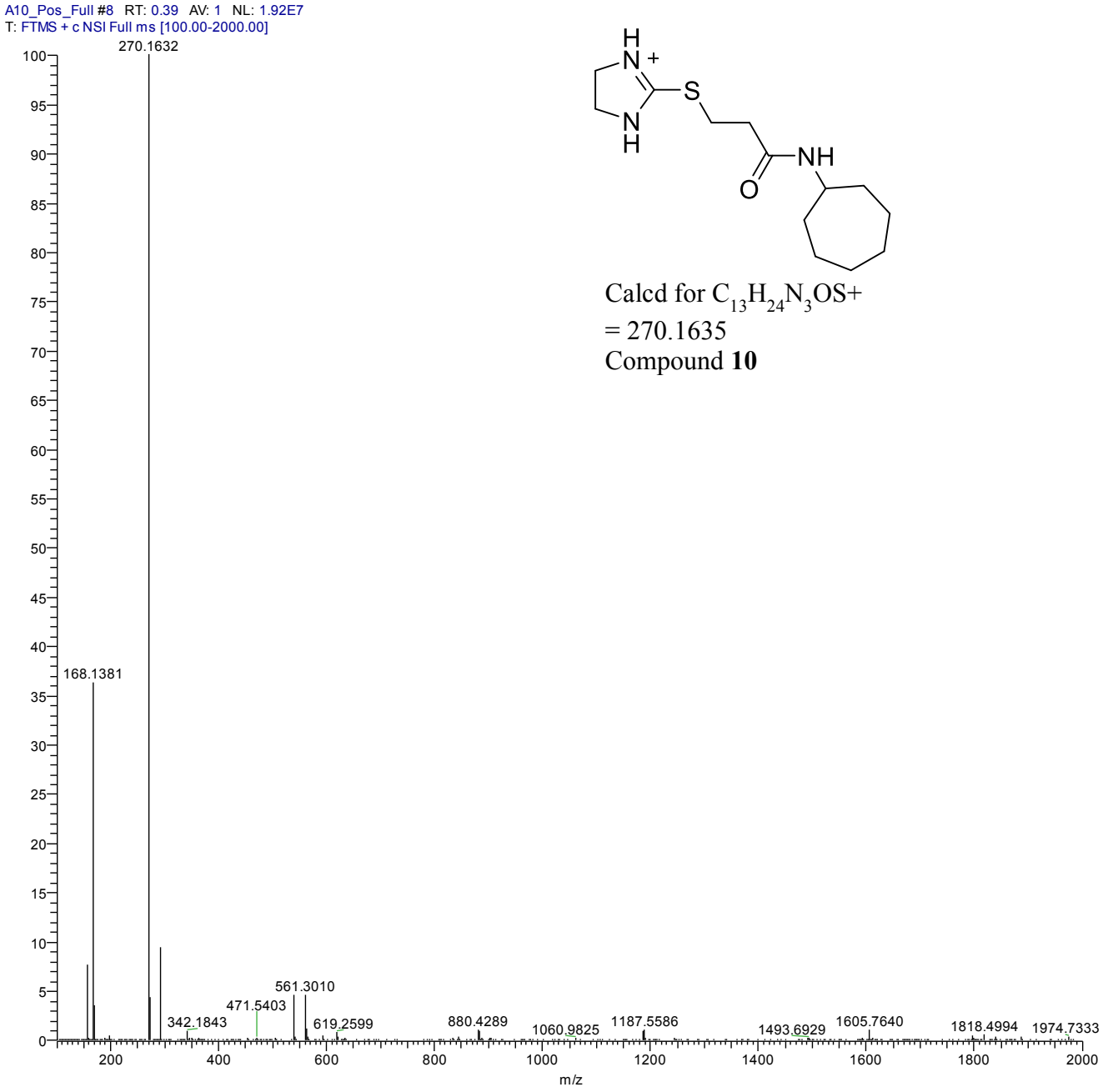


MS Spectrum Graph

Ret.Time:12.300(Scan#:679)
BG Mode:?
Mass Peaks:903 Base Peak:269.95(3355242) Polarity:Pos Segment1 - Event1



HRMS(ESI) Compound 10



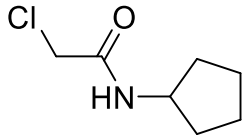
Calcd for $C_{13}H_{24}N_3OS^+$ = 270.1635

Compound **10**

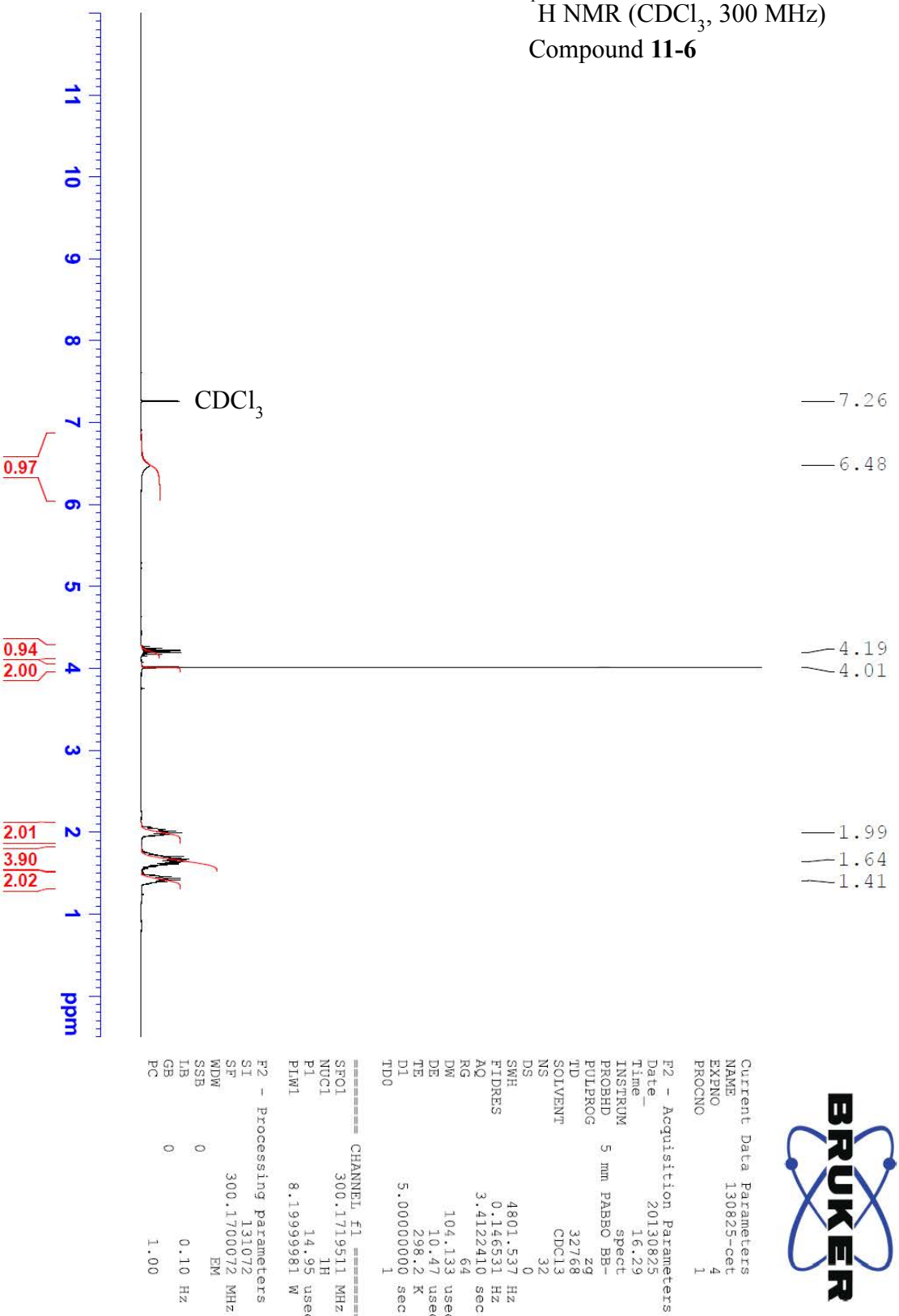
Spectra for Compound 11

^{1}H NMR Compound 11-6

2-chloro-N-cyclopentylacetamide

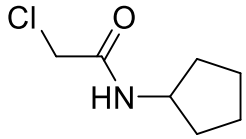


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **11-6**

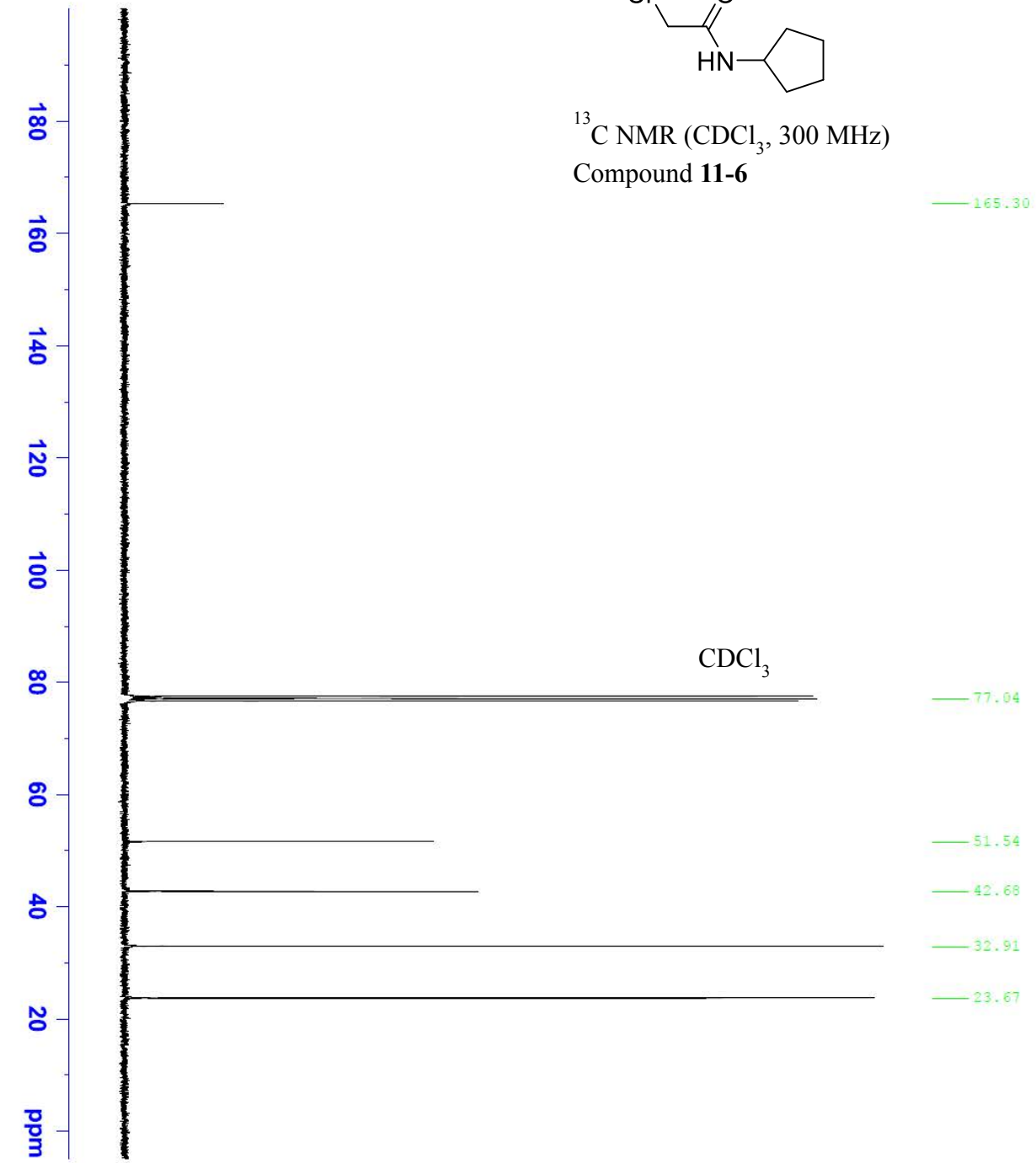


^{13}C NMR Compound 11-6

2-chloro-N-cyclopentylacetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **11-6**




```
Current Data Parameters
NAME                  130825-cet
EXPNO                          5
PROCNO                         1

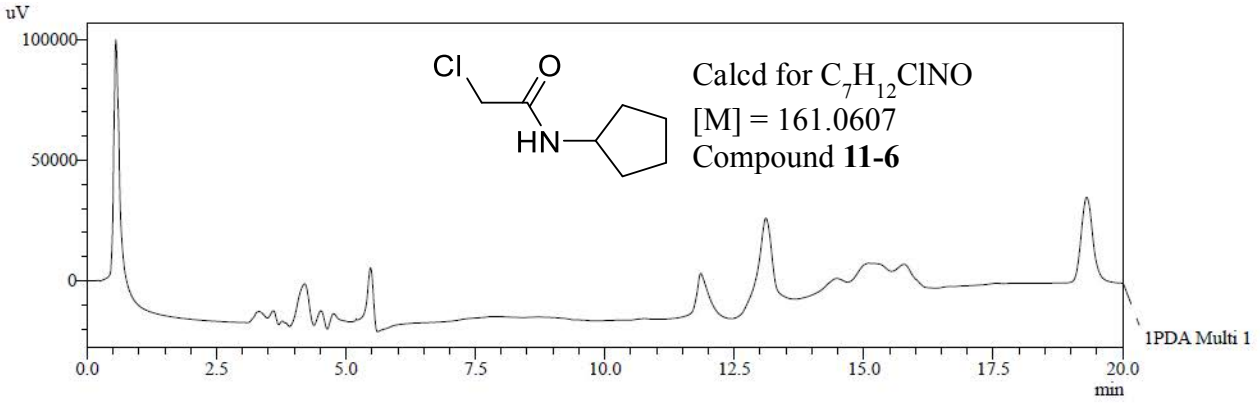
F2 - Acquisition Parameters
Date_                   20130825
Time_                      16.43
INSTRUM                    spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1536
DS                           2
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                         203
DW                      27.733 usec
DE                       6.80 usec
TE                      298.5 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            6

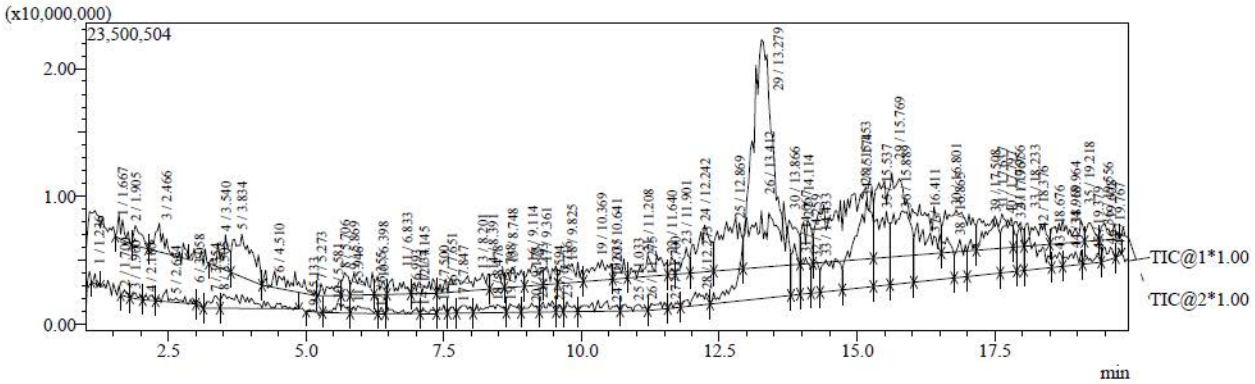
======== CHANNEL f1 ========
SFO1          75.4853543 MHz
NUC1                    13C
P1                     9.90 usec
PLW1          33.00000000 W

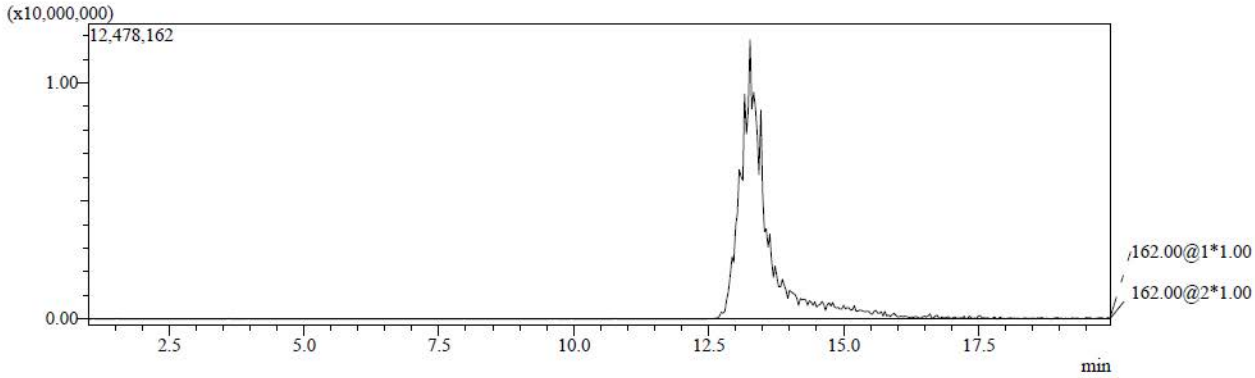
======== CHANNEL f2 ========
SFO2          300.1712007 MHz
NUC2                    1H
CPDPRG[2 bi_waltz65_256
PCPD2                  90.00 usec
PLW2           8.20349979 W
PLW12          0.22635999 W
PLW13          0.18335000 W

F2 - Processing parameters
SI                      32768
SF                75.4778070 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

LCMS Compound 11-6

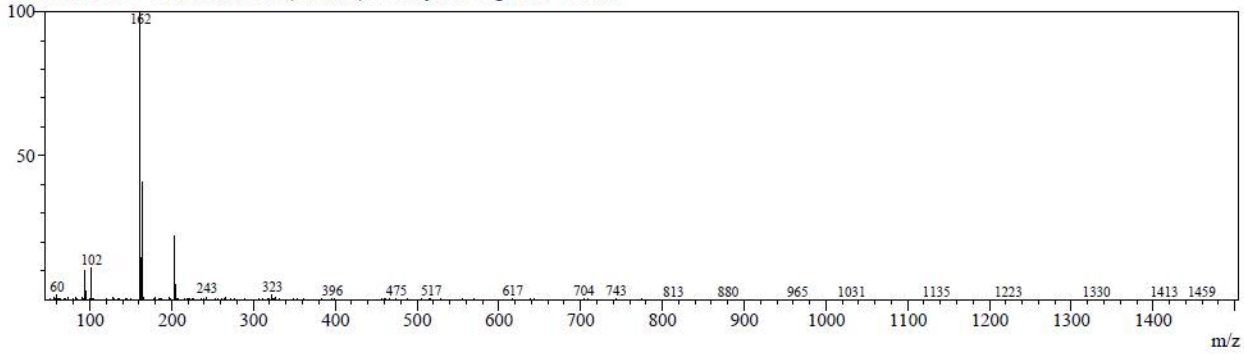




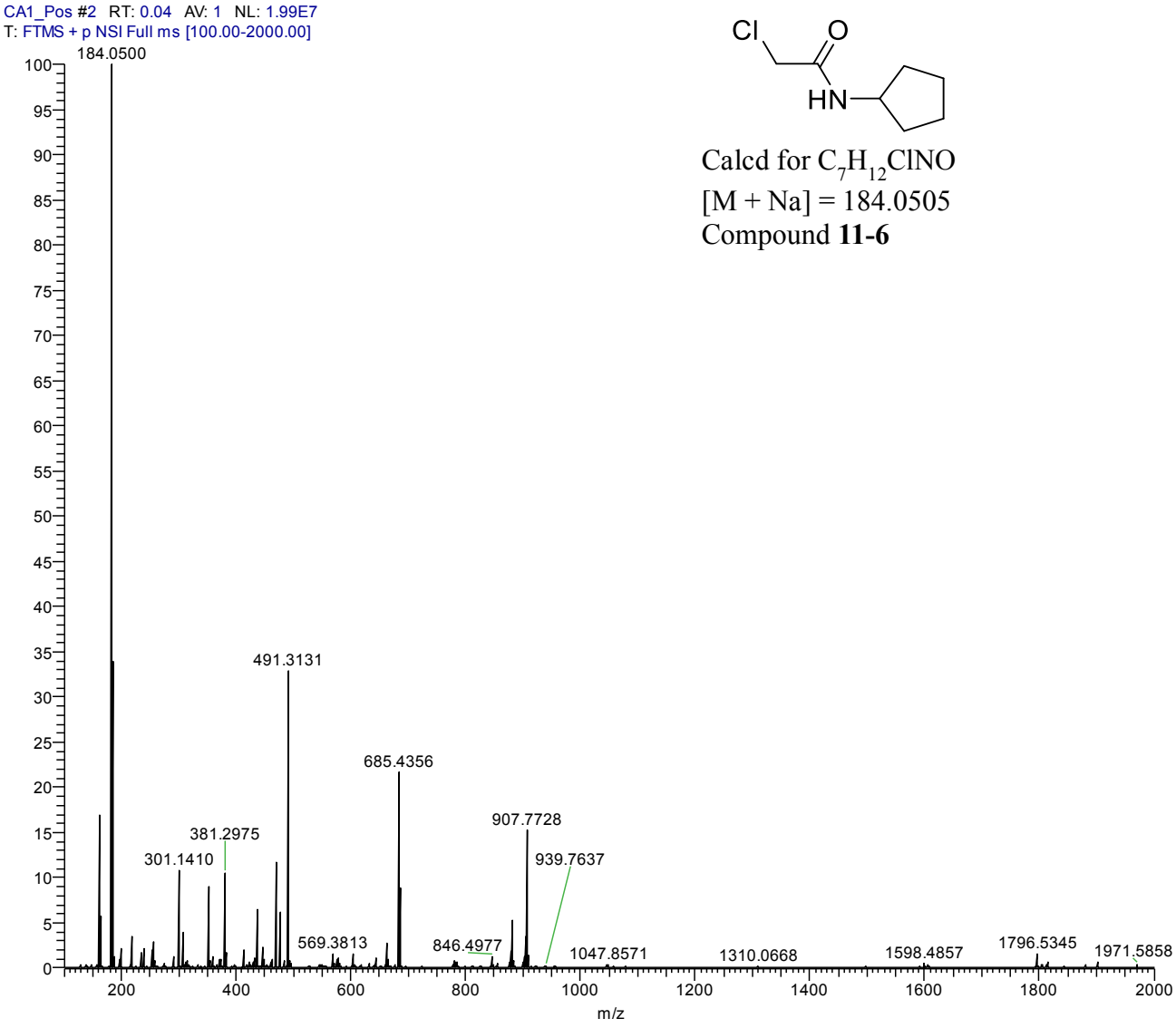


MS Spectrum Graph

Ret.Time:13.300(Scan#:739)
BG Mode:None
Mass Peaks:1132 Base Peak:161.80(8894732) Polarity:Pos Segment1 - Event1



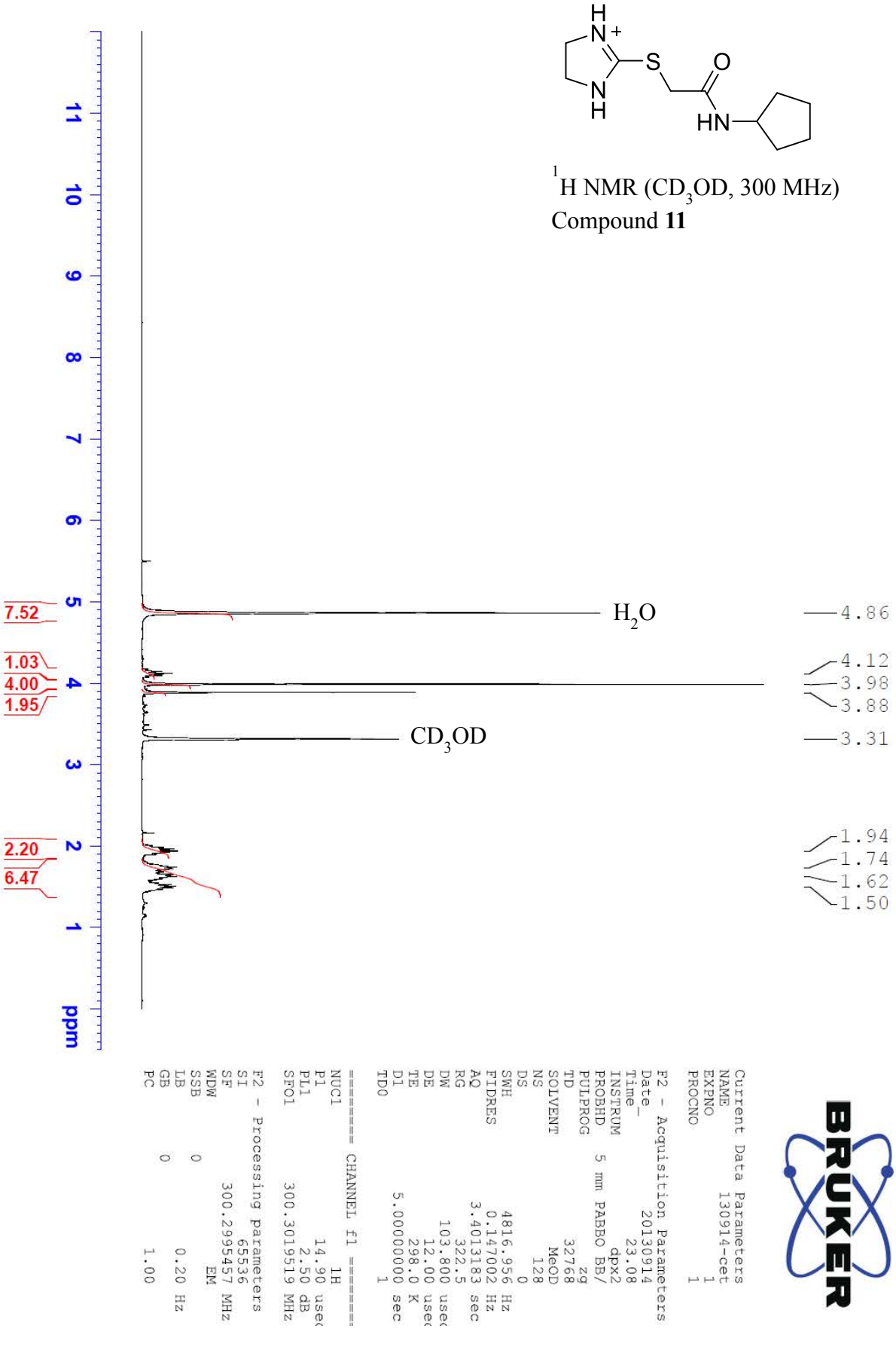
HRMS(ESI) Compound 11-6

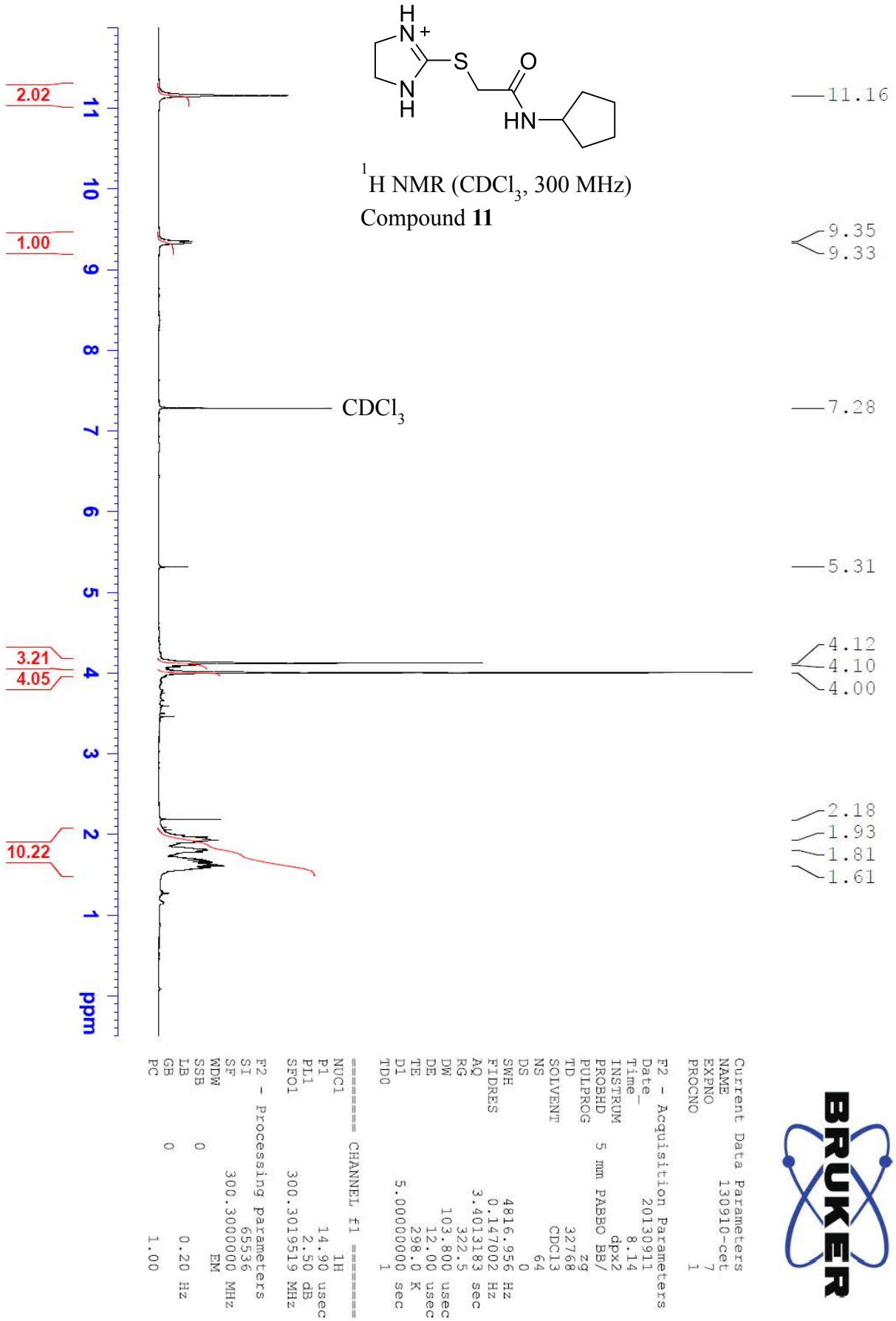


Calcd for $C_7H_{12}ClNO$
[M + Na] = 184.0505
Compound **11-6**

^{1}H NMR Compound 11

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cyclopentylacetamide

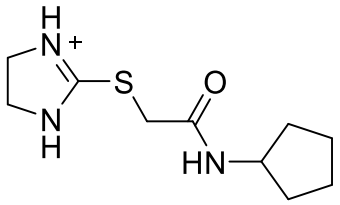




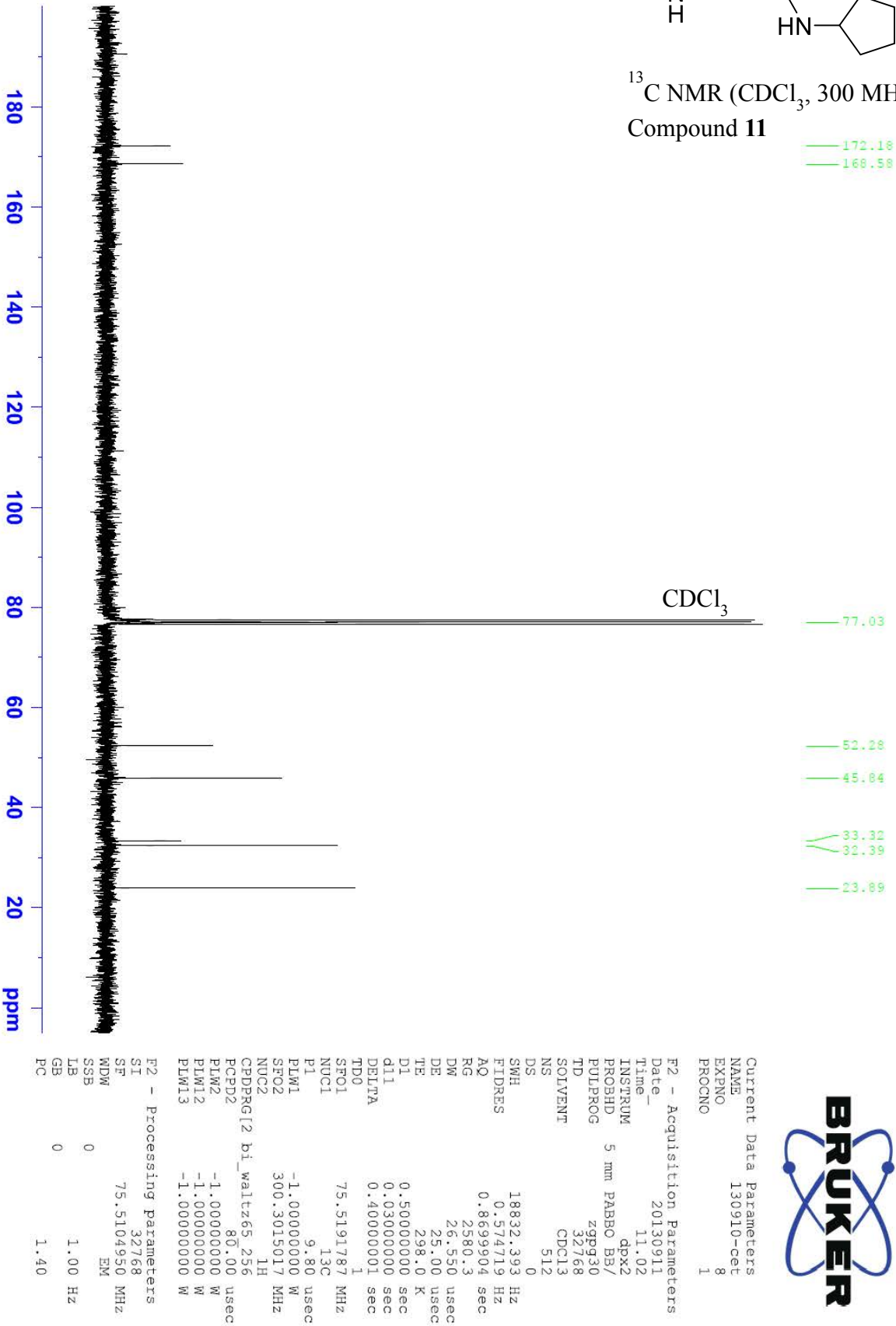
^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **11**

^{13}C NMR Compound 11

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cyclopentylacetamide

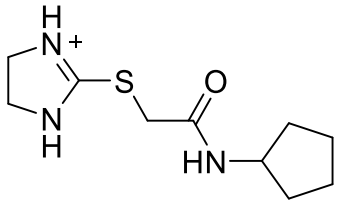


^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **11**

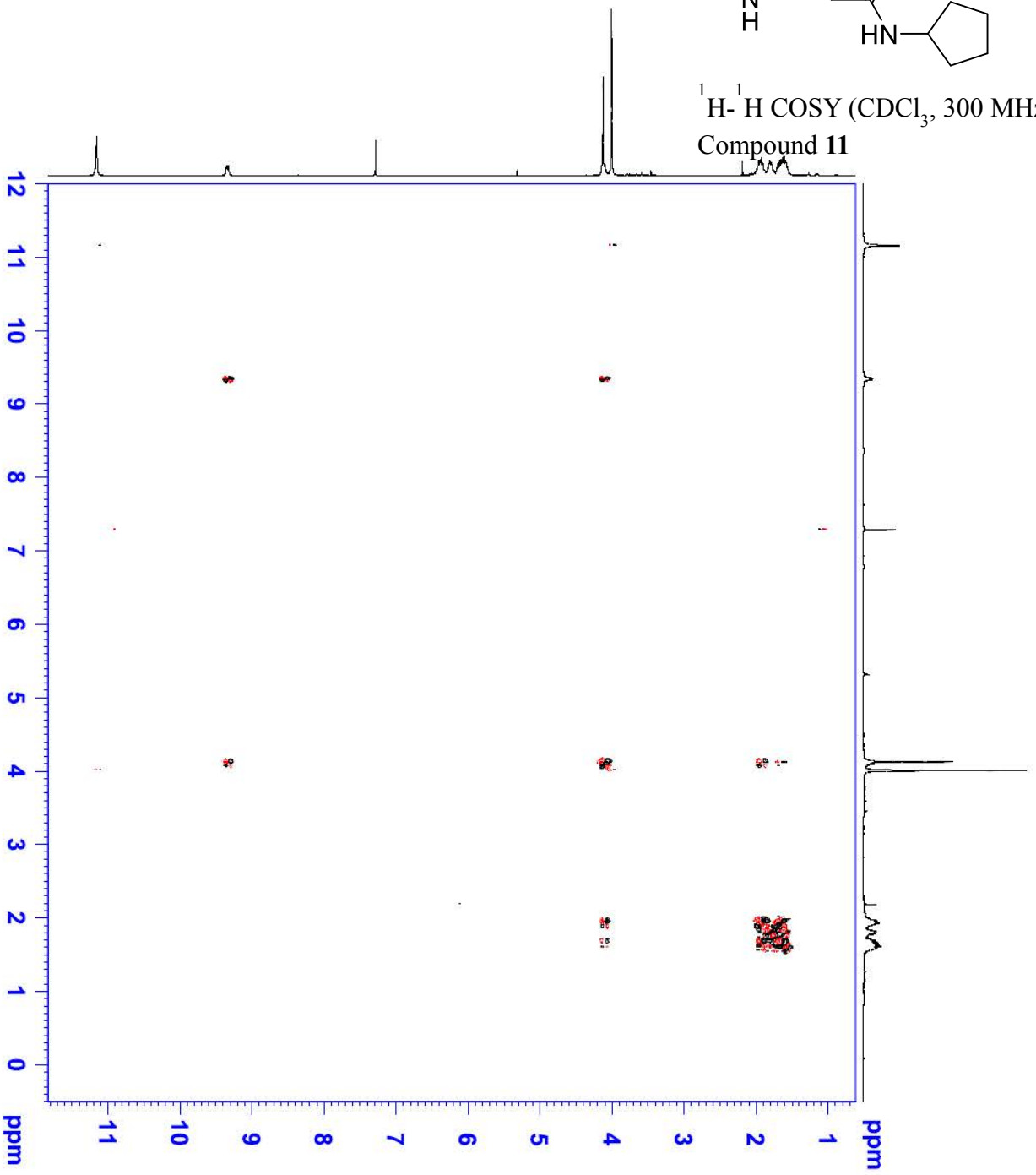


^{1}H-^{1}H COSY Compound 11

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cyclopentylacetamide



^{1}H-^{1}H COSY ($CDCl_3$, 300 MHz)
Compound **11**



Current Data Parameters
NAME 130910-cet
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130911
Time 11.10
INSTRUM dpx2
PROBHD 5 mm PABBO BB/
PULPROG cosygpmfph
TD 2048
SOLVENT CDCl3
NS 1
DS 16
SWH 4496.403 Hz
FIDRES 2.195509 Hz
AQ 0.2277376 sec
RG 256
DW 111.200 usec
DE 6.00 usec
TE 298.0 K
d0 0.00009223 sec
D1 2.00000000 sec
d13 0.00000400 sec
D16 0.00020000 sec
d20 0.00120400 sec
IN0 0.00022240 sec
STLCNT 0

====== CHANNEL f1 ======
NUC1 1H
P1 14.90 usec
p2 29.80 usec
PL1 2.50 dB
SFO1 300.3018018 MHz

====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPZ1 10.00 %
GPZ2 20.00 %
P16 1000.00 usec

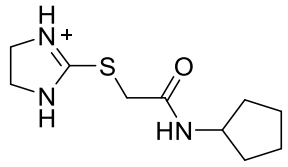
F1 - Acquisition parameters
TD 256
SFO1 300.3018 MHz
FIDRES 17.564074 Hz
SW 14.973 ppm
FnMODE States-TPPI

F2 - Processing parameters
SI 1024
SF 300.3000000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

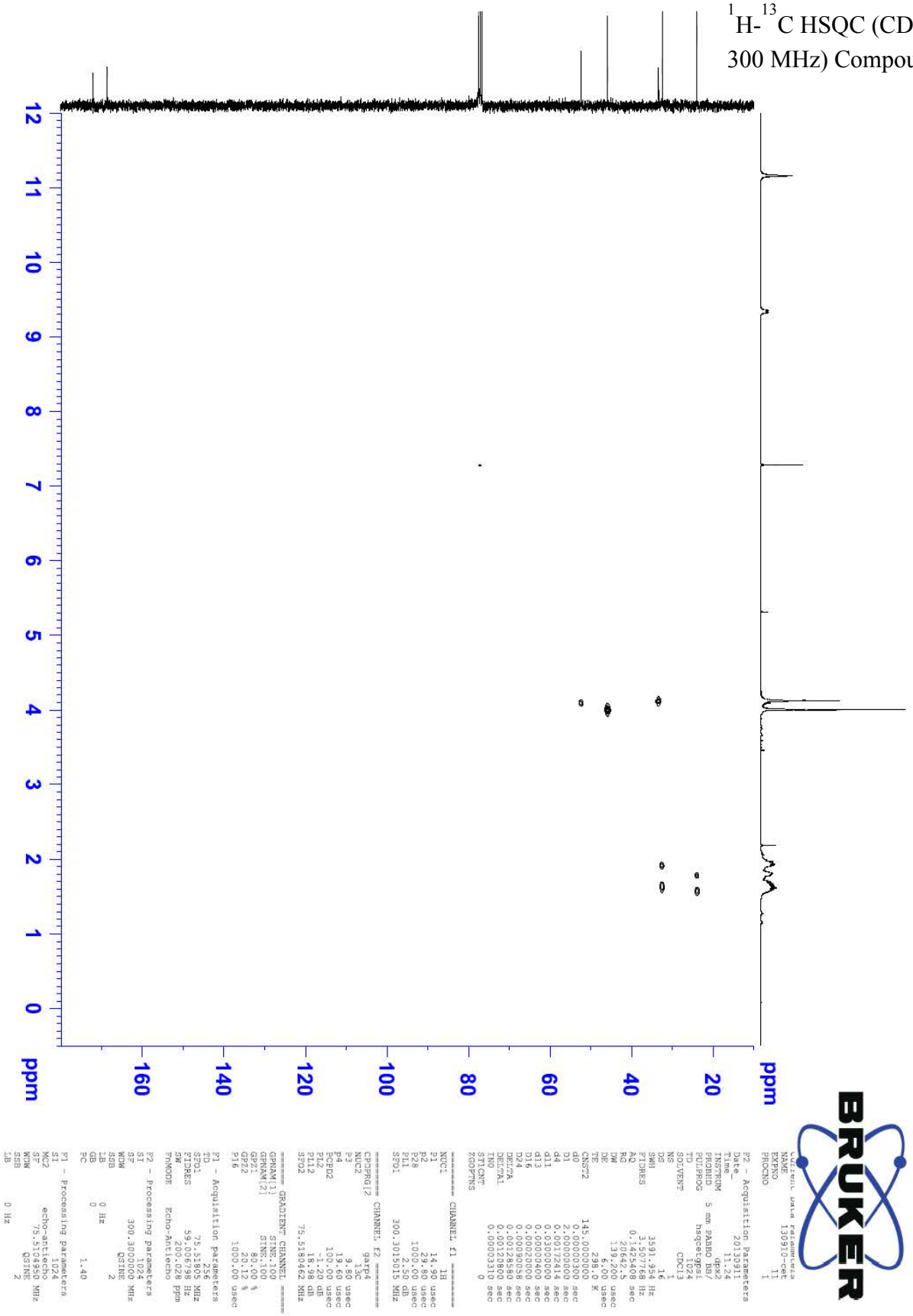
F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 300.3000000 MHz
WDW QSINE
SSB 2
LB 0 Hz

^{1}H-^{13}C HSQC Compound 11

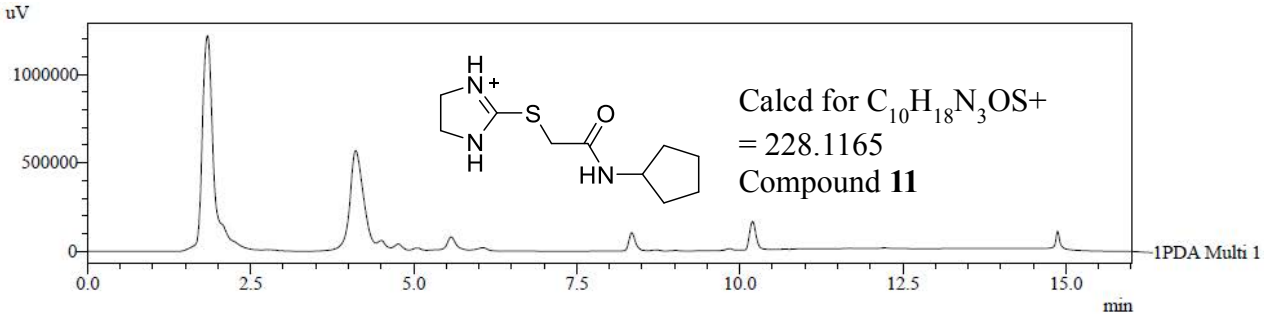
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cyclopentylacetamide

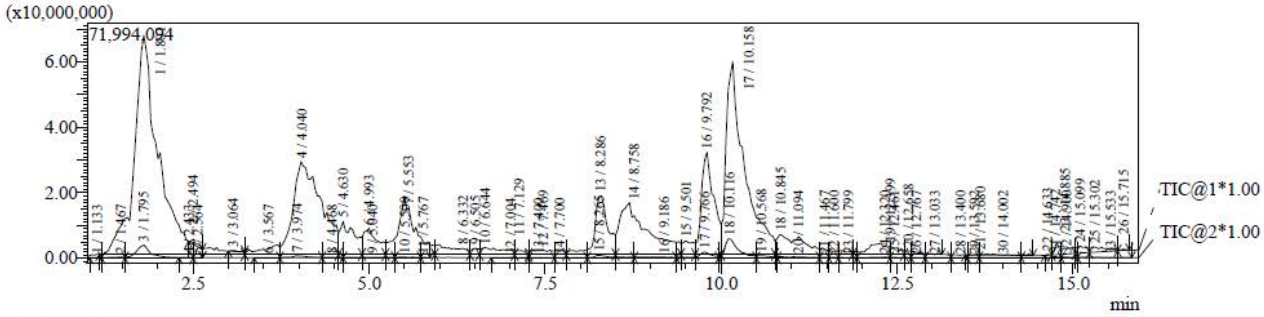


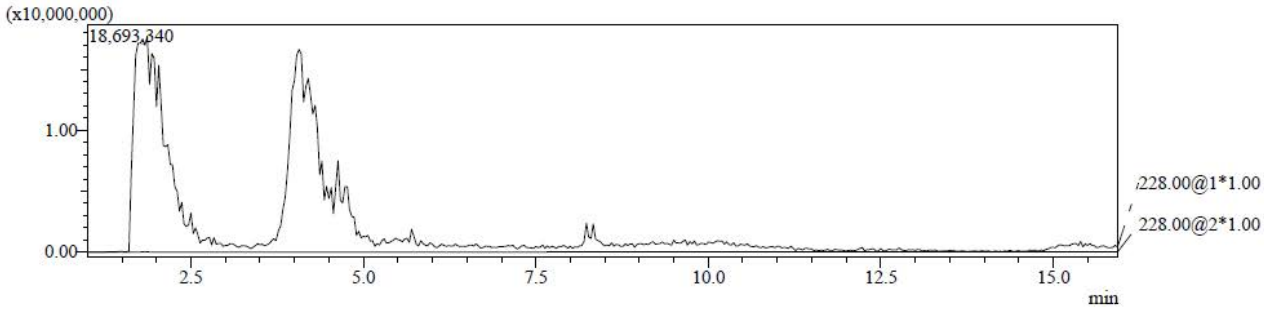
^{1}H-^{13}C HSQC ($CDCl_3$, 300 MHz) Compound **11**



LCMS Compound 11

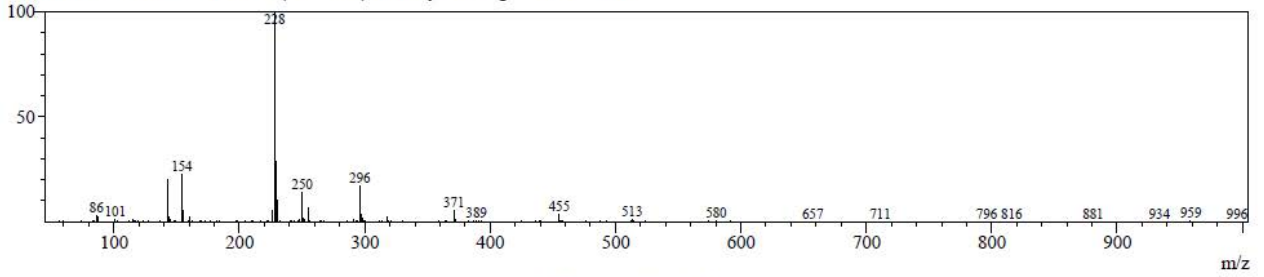






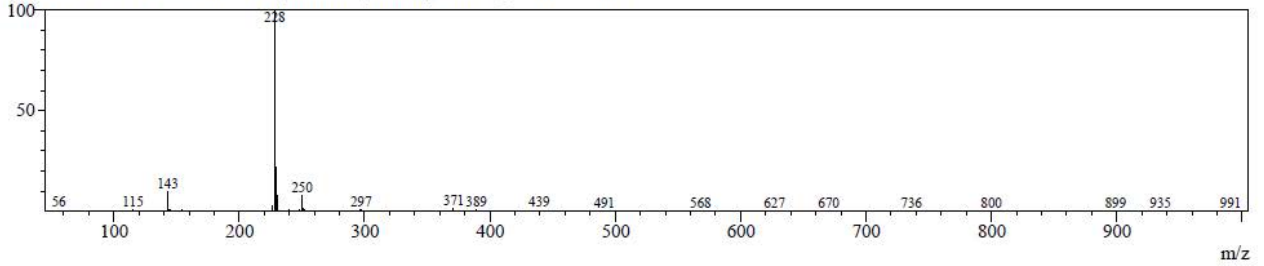
MS Spectrum Graph

Ret.Time:1.900(Scan#:55)
BG Mode:None
Mass Peaks:897 Base Peak:227.90(13775498) Polarity:Pos Segment1 - Event1

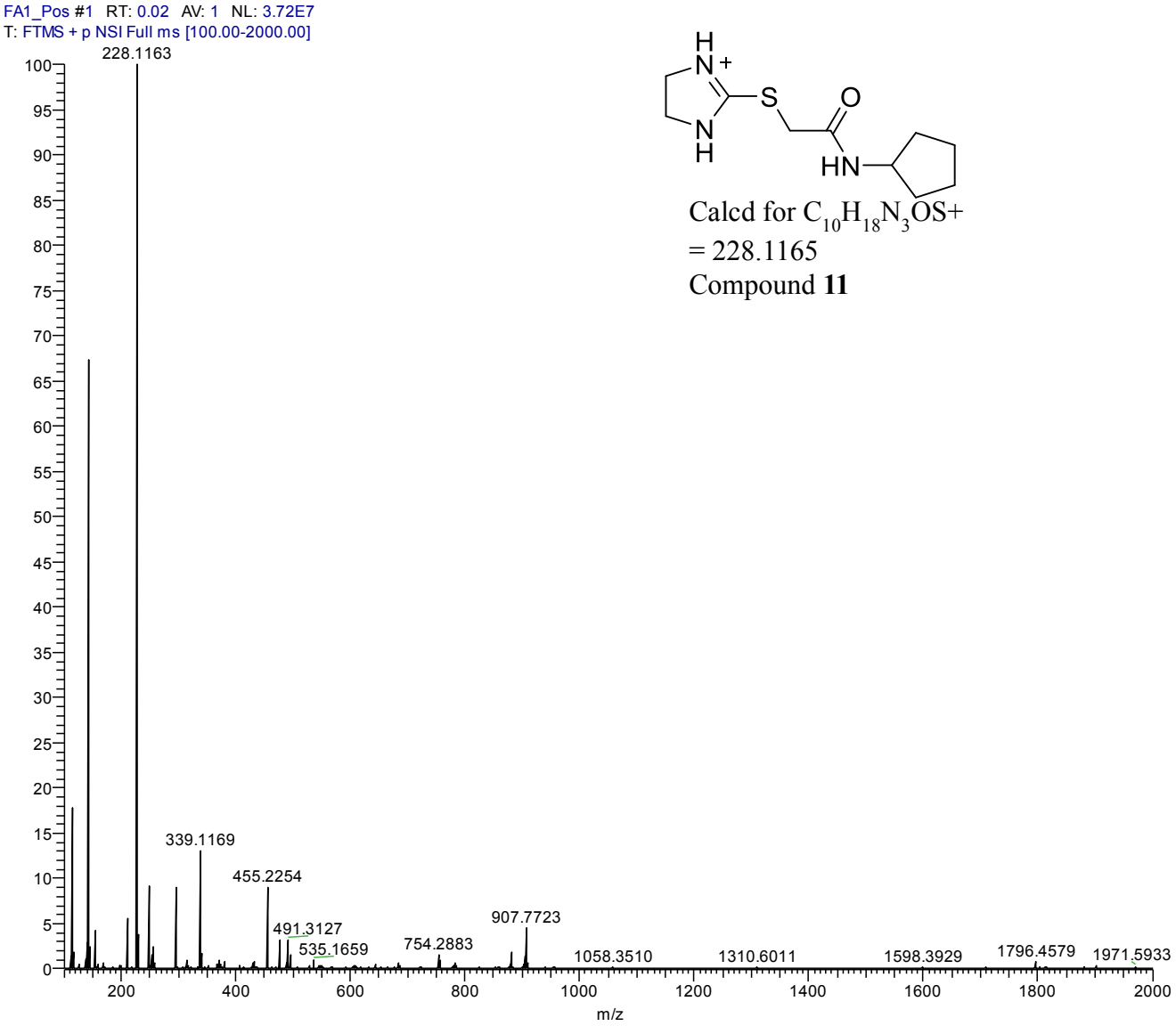


MS Spectrum Graph

Ret.Time:4.067(Scan#:185)
BG Mode:?
Mass Peaks:921 Base Peak:228.00(16629562) Polarity:Pos Segment1 - Event1



HRMS(ESI) Compound 11



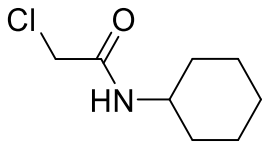
Calcd for $C_{10}H_{18}N_3OS^+$ = 228.1165

Compound **11**

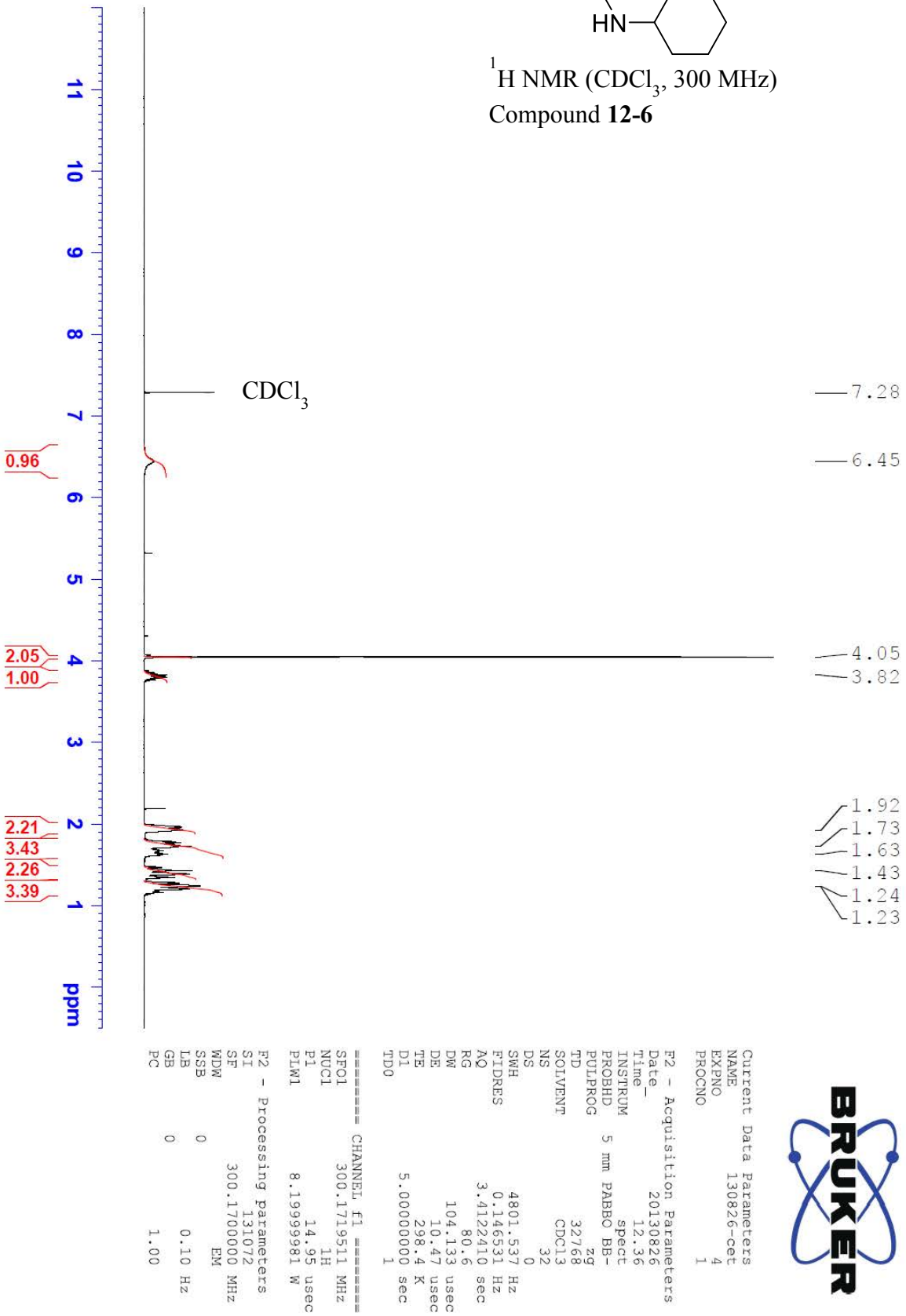
Spectra for Compound 12

^{1}H NMR Compound 12-6

2-chloro-N-cyclohexylacetamide

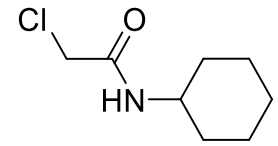


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **12-6**

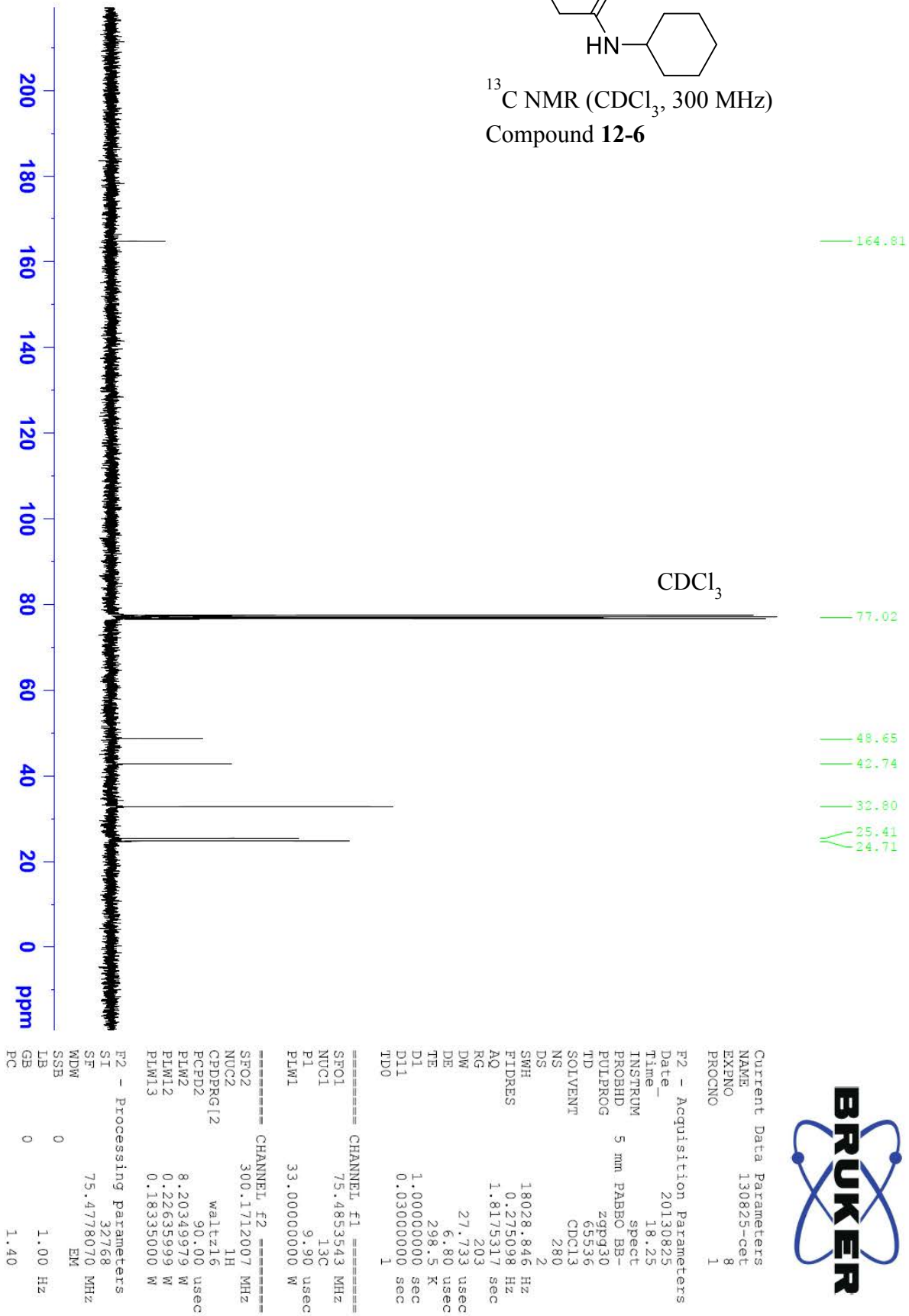


^{13}C NMR Compound 12-6

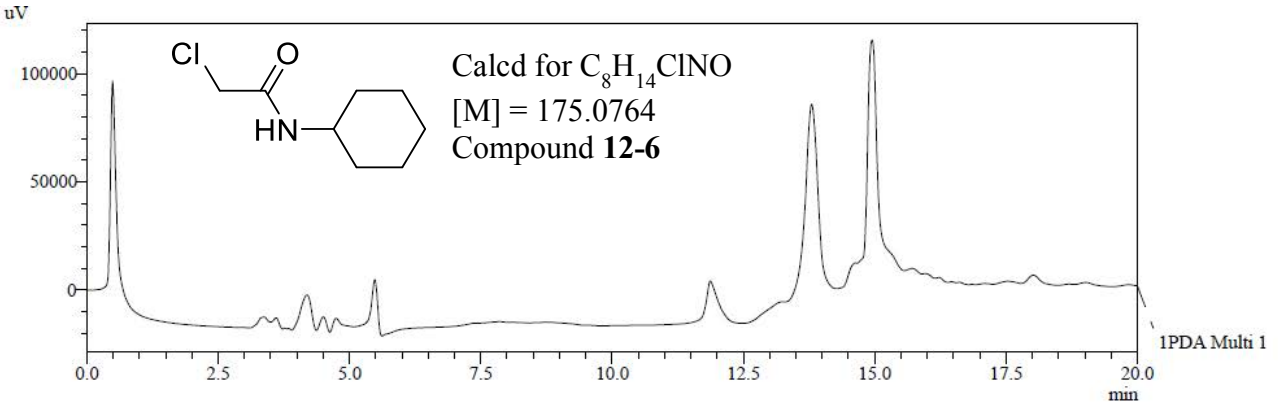
2-chloro-N-cyclohexylacetamide

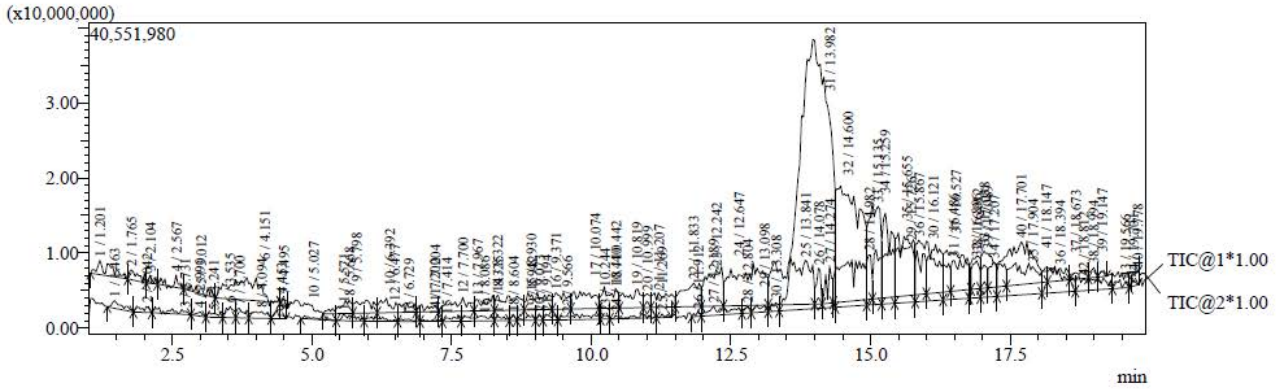


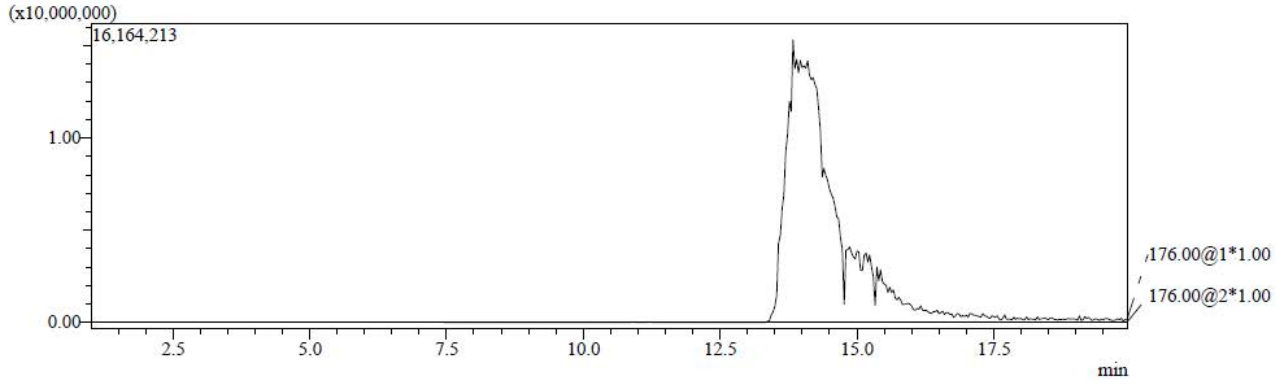
^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **12-6**



LCMS Compound 12-6

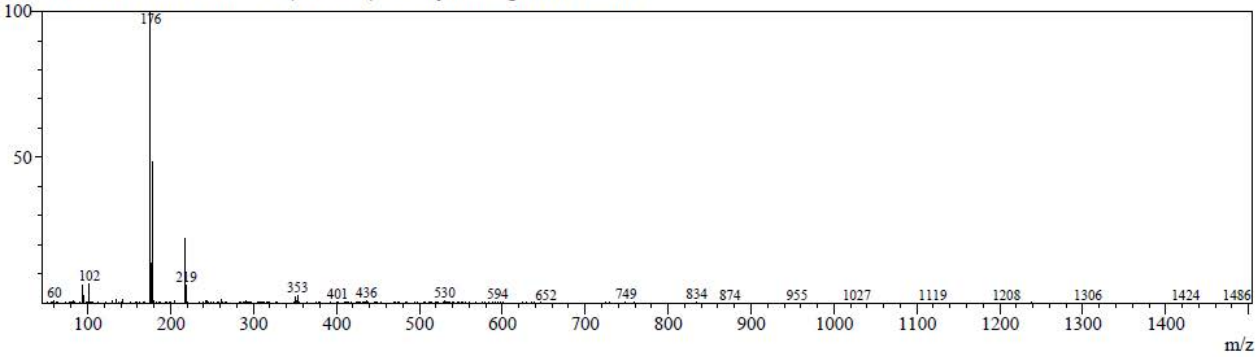




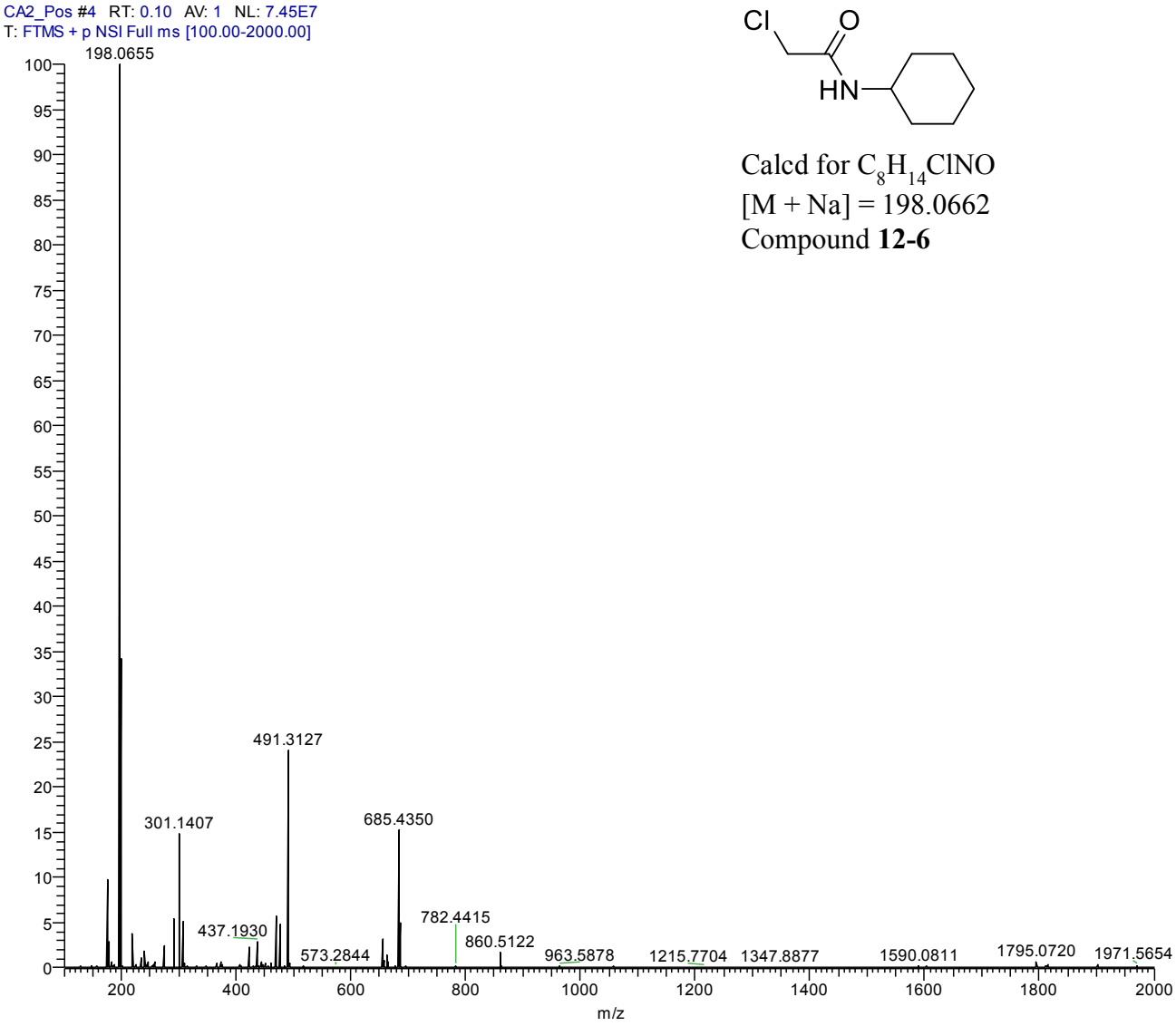


MS Spectrum Grapl

Ret.Time:13.933(Scan#:777)
BG Mode:?
Mass Peaks:1197 Base Peak:175.80(13521934) Polarity:Pos Segment1 - Event1



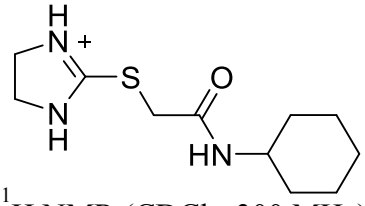
HRMS(ESI) Compound 12-6



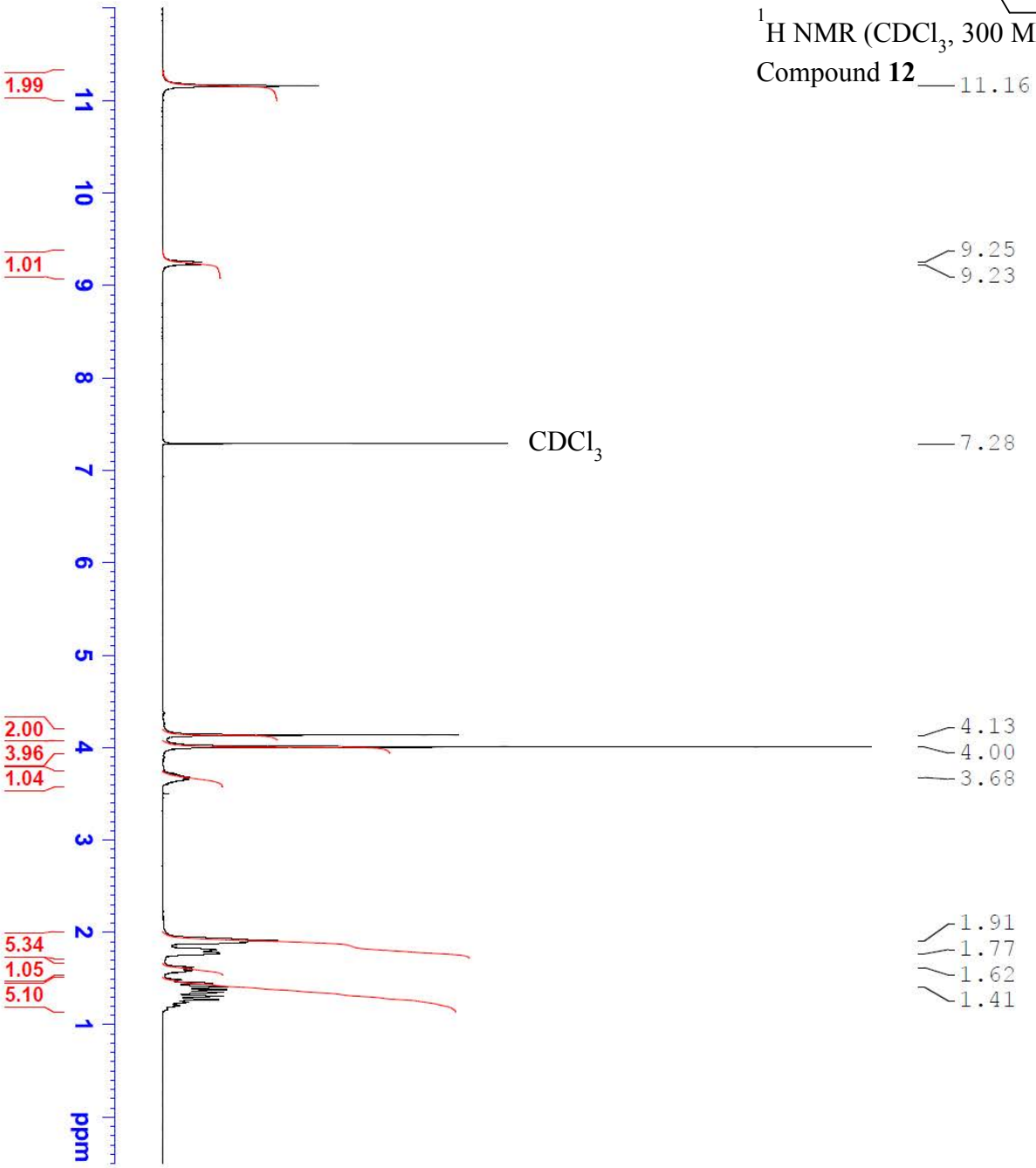
Calcd for $C_8H_{14}ClNO$
[M + Na] = 198.0662
Compound **12-6**

^{1}H NMR Compound 12

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cyclohexylacetamide



^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **12**



Current Data Parameters
NAME 141207-cet
EXPNO 2
PROCNO 1

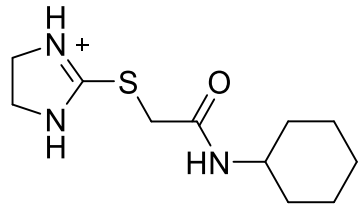
F2 - Acquisition Parameters
Date_ 20141207
Time_ 13.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4801.537 Hz
FIDRES 0.146531 Hz
AQ 3.4122410 sec
RG 114
DW 104.133 usec
DE 9.44 usec
TE 298.0 K
D1 5.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 300.1719511 MHz
NUC1 1H
P1 16.56 usec
PLW1 8.19999981 W

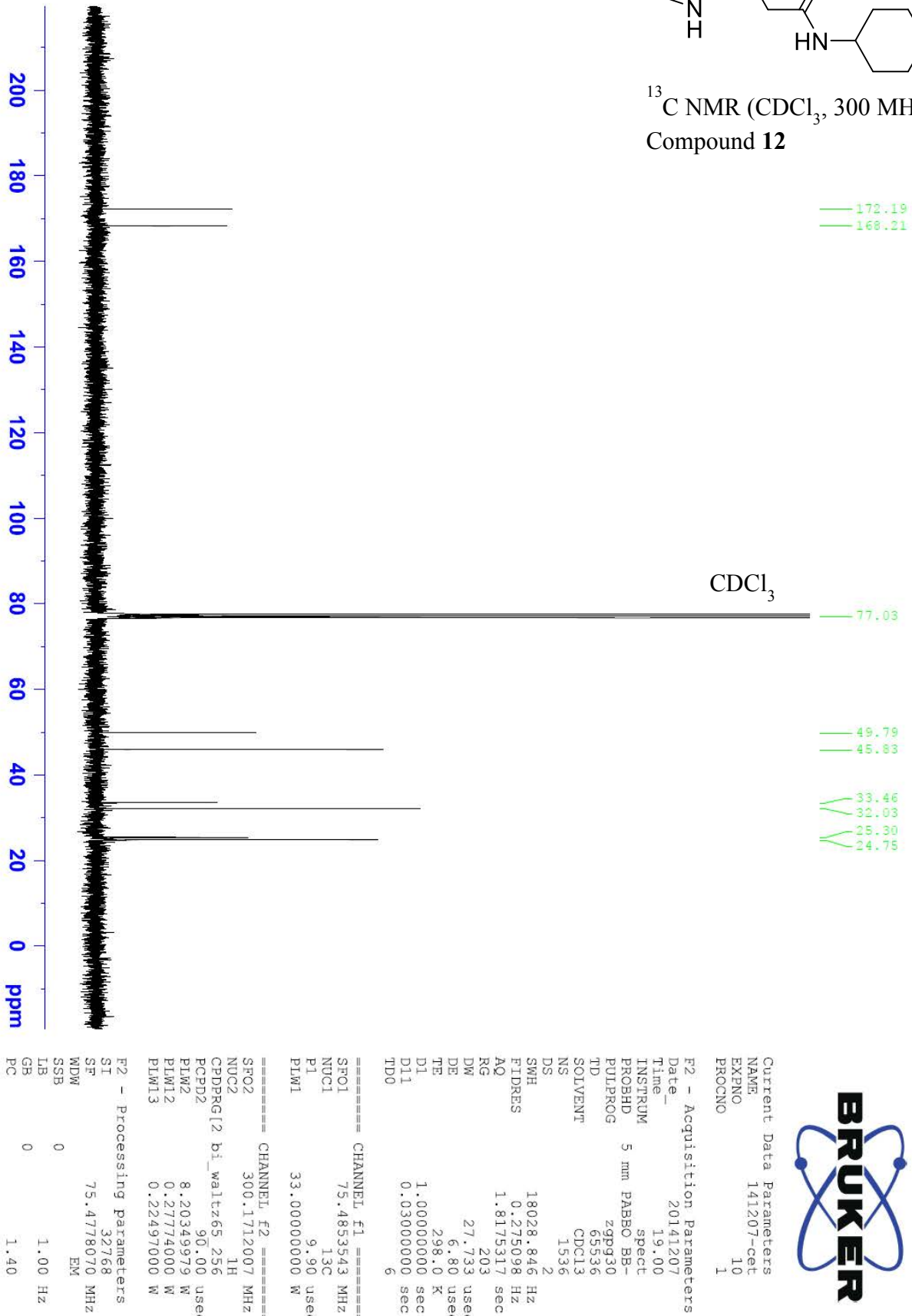
F2 - Processing parameters
SI 131072
SF 300.1700000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

^{13}C NMR Compound 12

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cyclohexylacetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **12**

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Current Data Parameters
NAME                  141207-cet
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
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Time_                19.00
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      1536
DS                         2
SWH               18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                       203
DW                    27.733 usec
DE                     6.80 usec
TE                     298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                           6

======== CHANNEL f1 ========
SFO1            75.4853543 MHz
NUC1                      13C
P1                   9.90 usec
PLW1            33.00000000 W

======== CHANNEL f2 ========
SFO2           300.1712007 MHz
NUC2                       1H
CPDPRG[2 bi_waltz65_256
PCPD2                 90.00 usec
PLW2             8.20349979 W
PLW12            0.27774000 W
PLW13            0.22497000 W

F2 - Processing parameters
SI                        32768
SF              75.4778070 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```
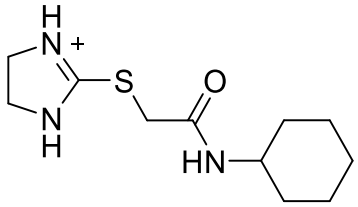
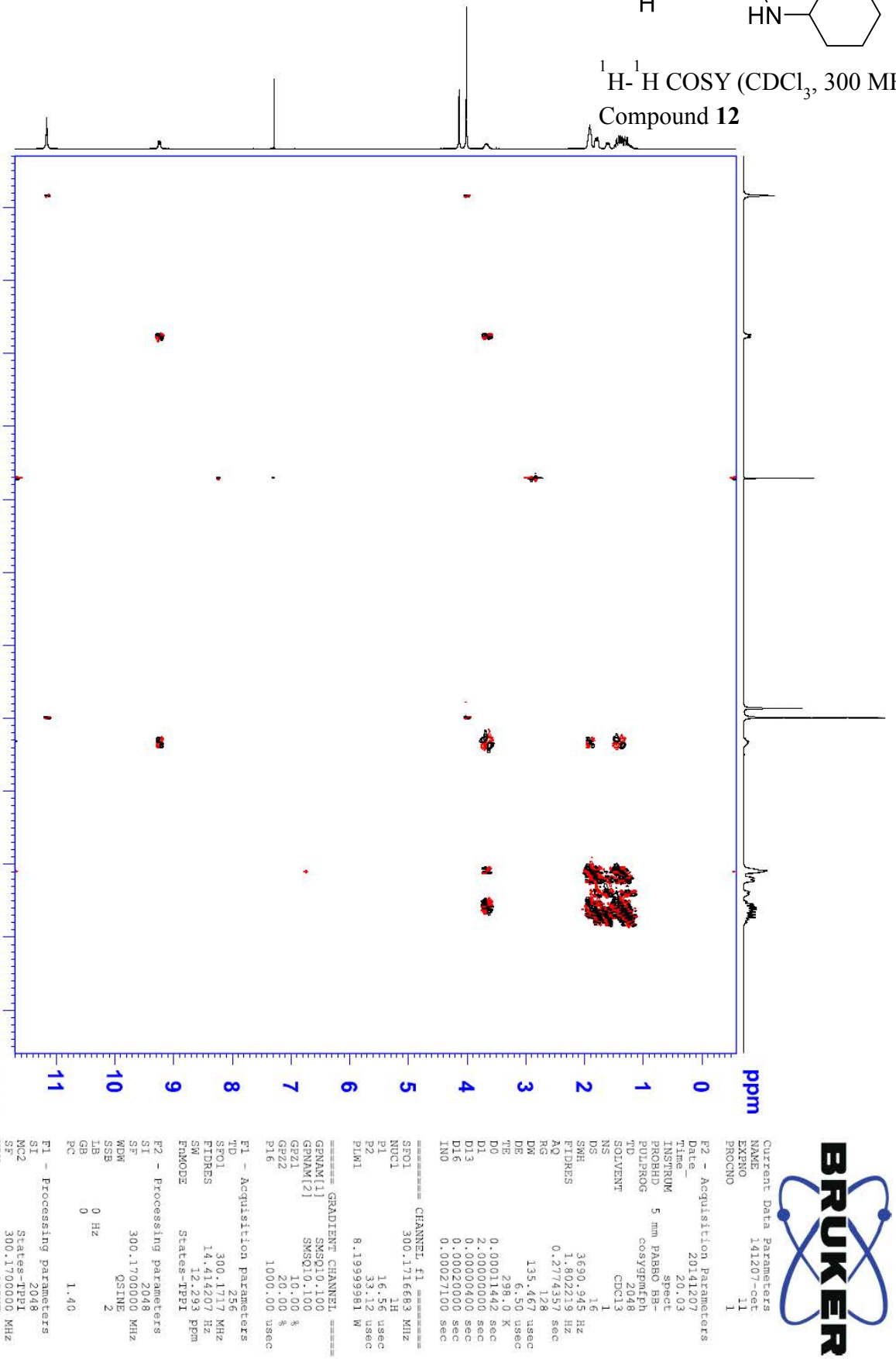

^{1}H-^{1}H COSY Compound 12

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cyclohexylacetamide

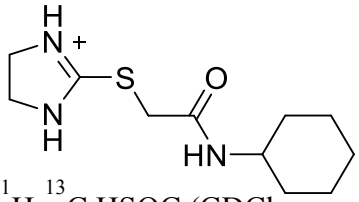


^{1}H- ^{1}H COSY ($CDCl_3$, 300 MHz)
Compound **12**



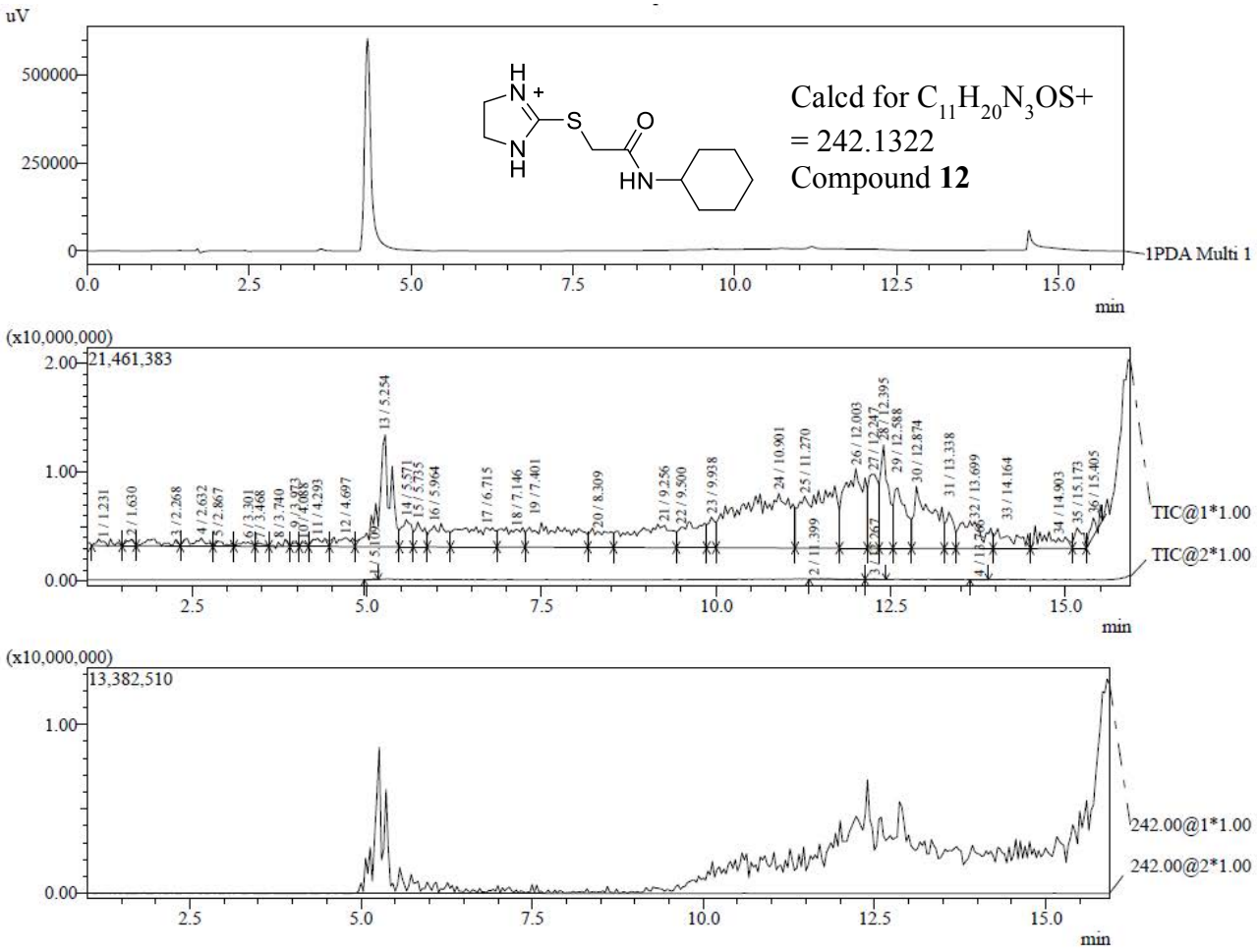
^{1}H-^{13}C HSQC Compound 12

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-cyclohexylacetamide



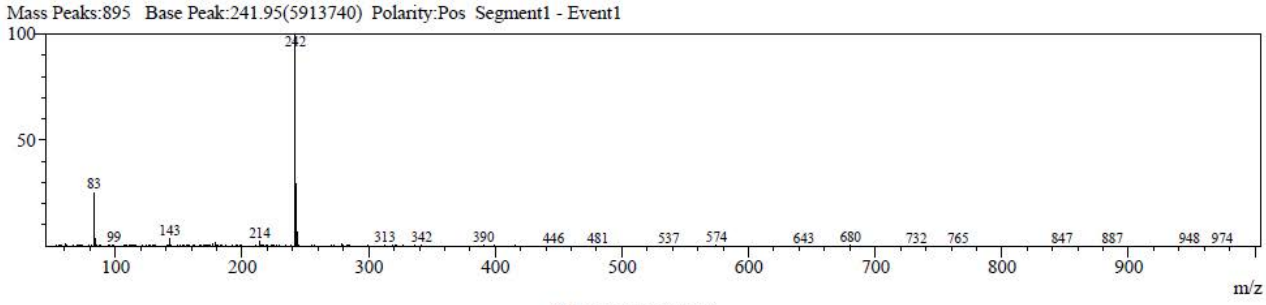
^{1}H-^{13}C HSQC ($CDCl_3$, 300MHz) Compound **12**

LCMS Compound 12



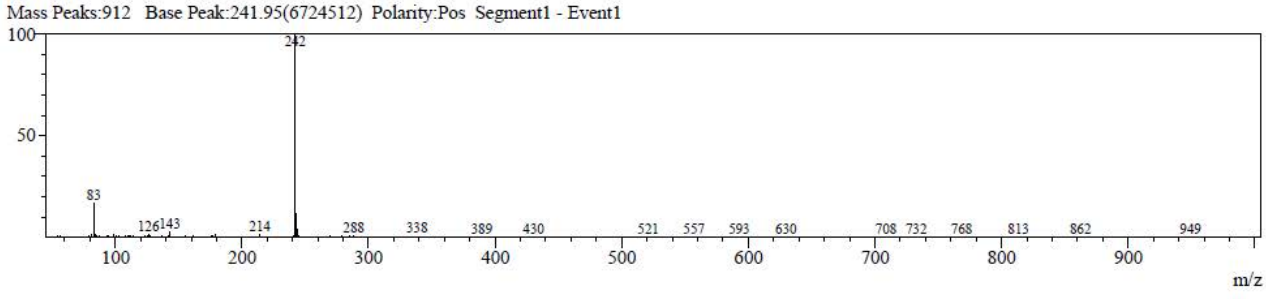
MS Spectrum Grapl:

Ret.Time:5.233(Scan#:255)
BG Mode:?
Mass Peaks:895 Base Peak:241.95(5913740) Polarity:Pos Segment1 - Event1

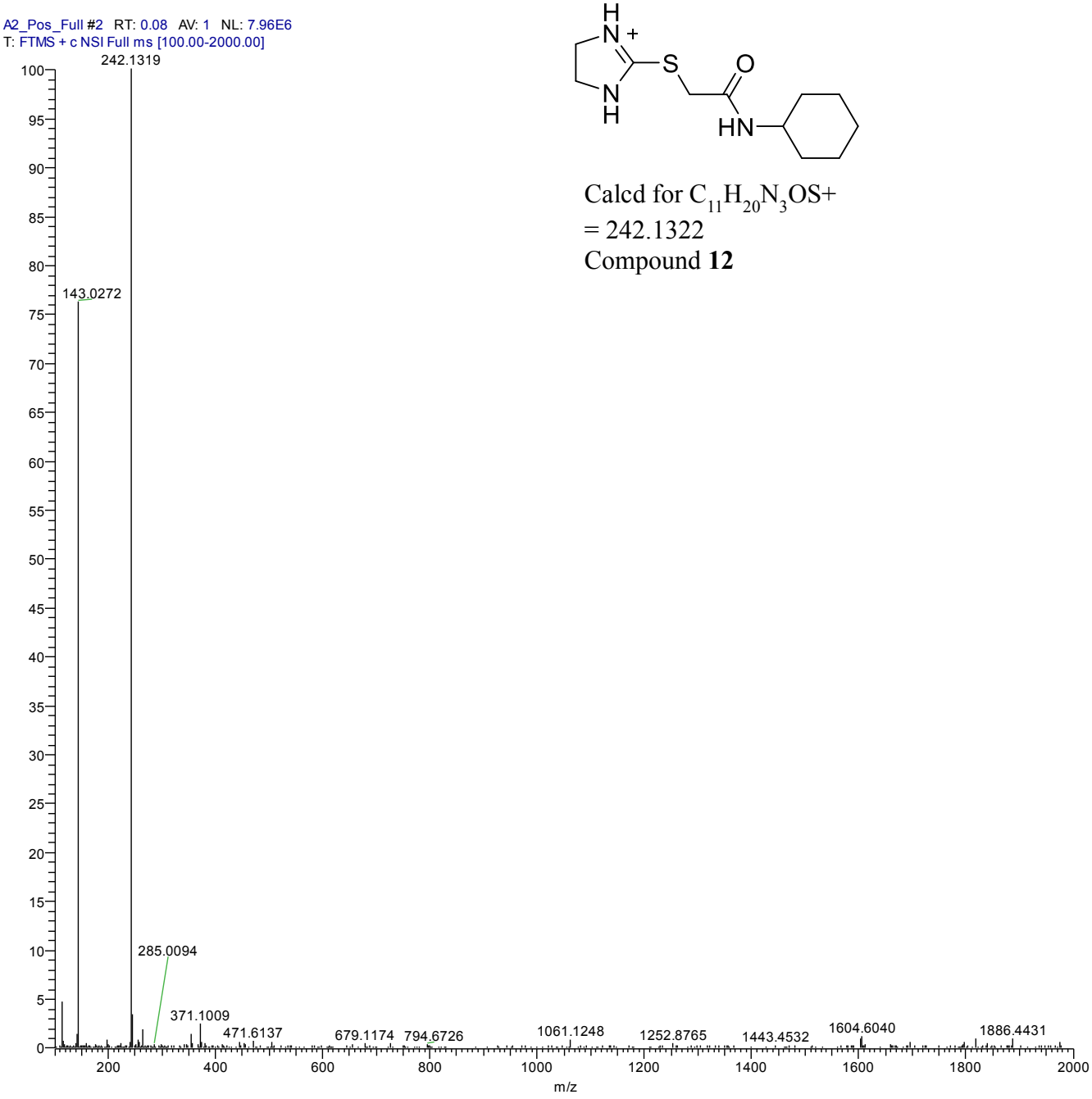


MS Spectrum Grapl:

Ret.Time:12.400(Scan#:685)
BG Mode:?
Mass Peaks:912 Base Peak:241.95(6724512) Polarity:Pos Segment1 - Event1



HRMS(ESI) Compound 12

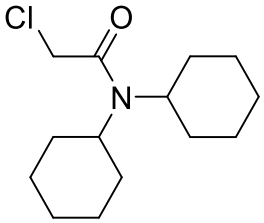


Calcd for $C_{11}H_{20}N_3OS^+$
= 242.1322
Compound **12**

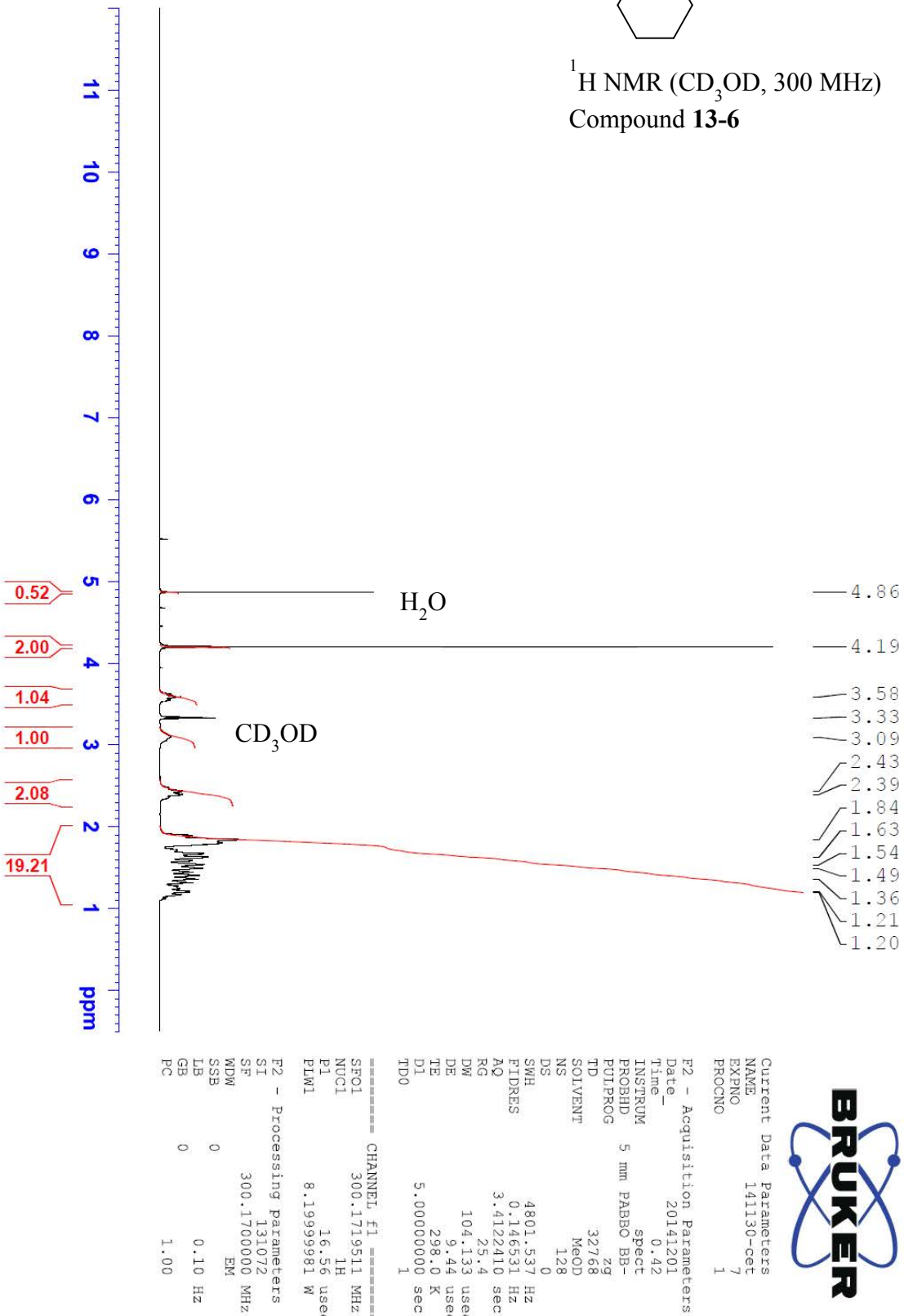
Spectra for Compound 13

^{1}H NMR Compound 13-6

2-chloro-N,N-dicyclohexylacetamide

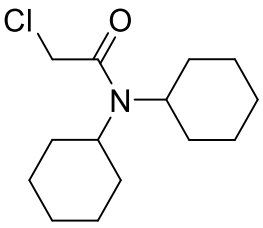


^{1}H NMR (CD_3OD, 300 MHz)
Compound **13-6**

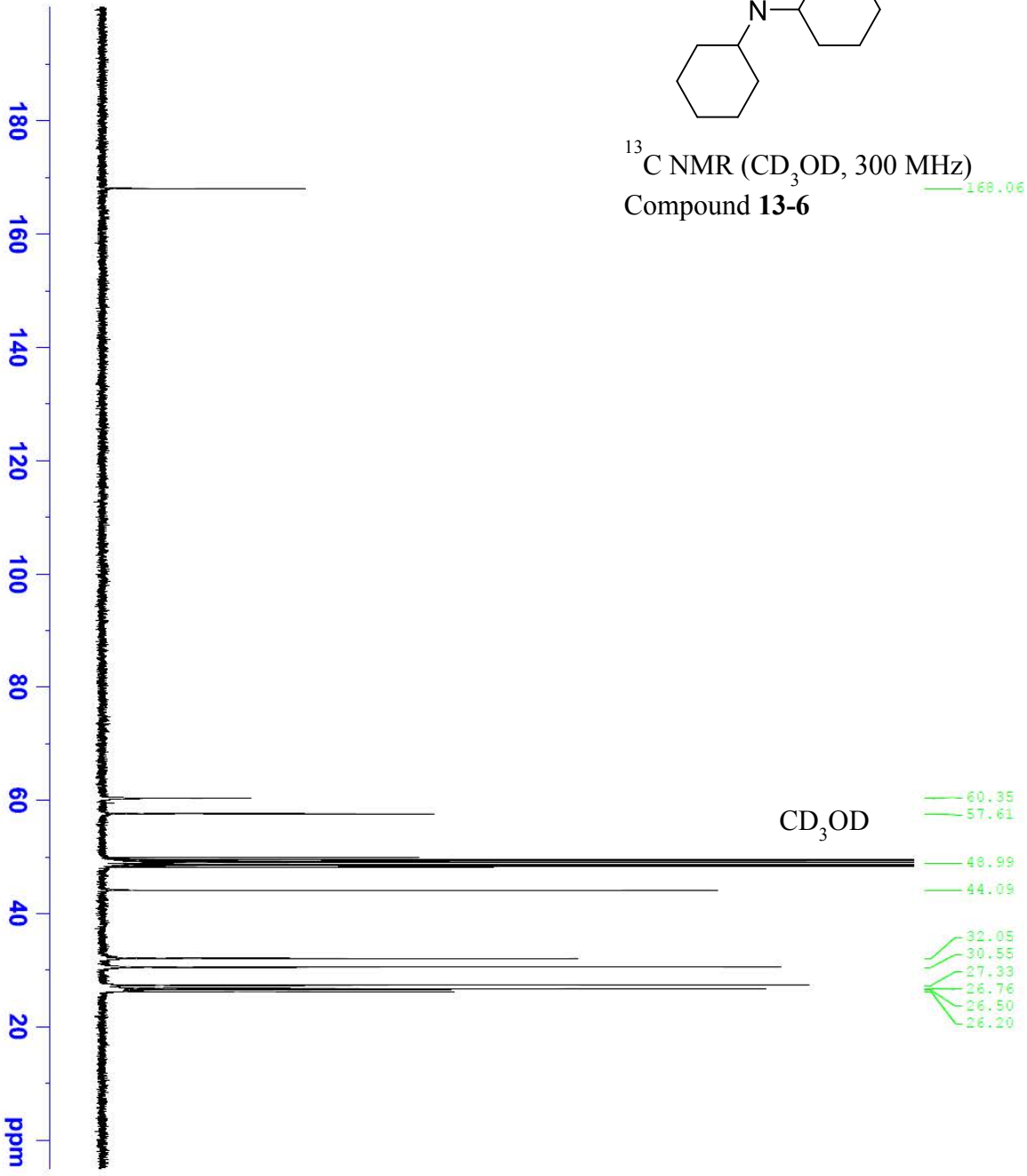


^{13}C NMR Compound 13-6

2-chloro-N,N-dicyclohexylacetamide



^{13}C NMR (CD_3OD, 300 MHz)
Compound **13-6**



Current Data Parameters
NAME 141130-cet
EXPNO 8
PROCNO 1

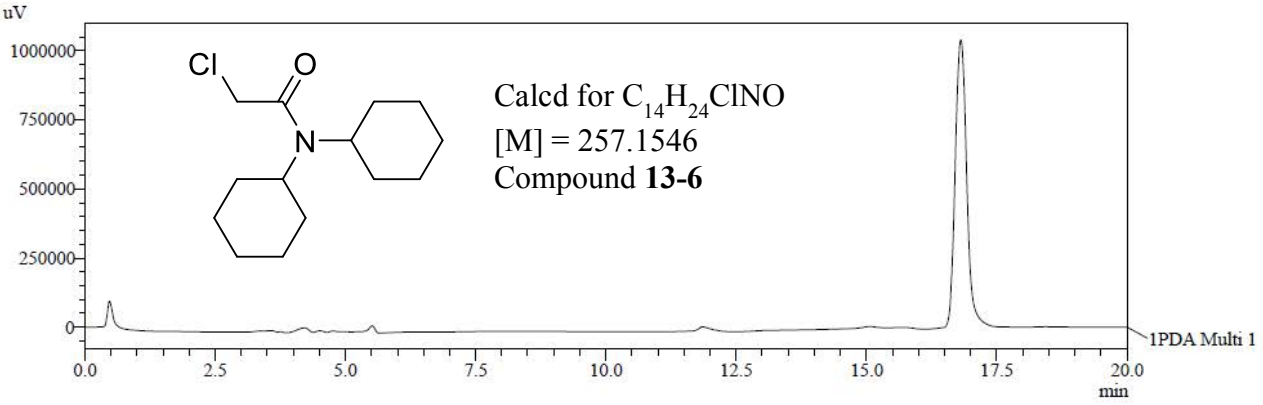
F2 - Acquisition Parameters
Date_ 20141201
Time_ 0.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1536
DS 2
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 203
DW 27.733 usec
DE 6.80 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 6

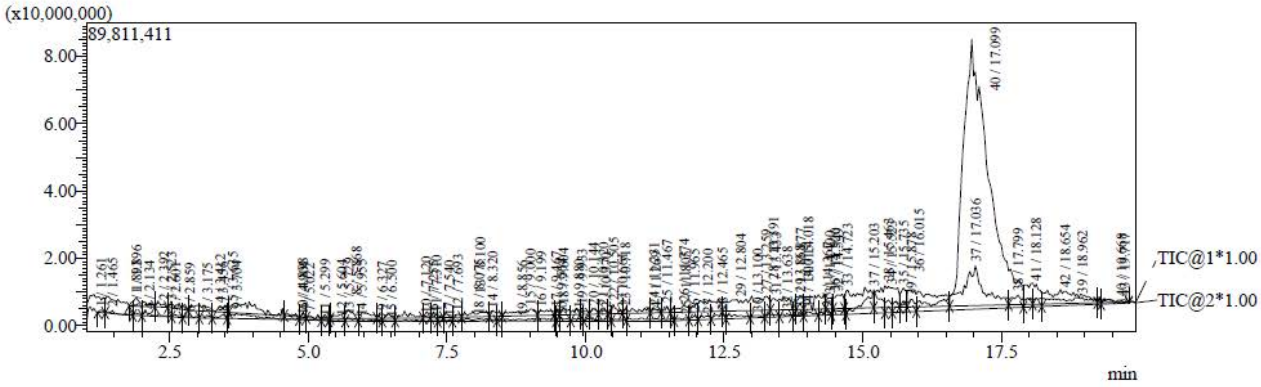
======== CHANNEL f1 ========
SFO1 75.4853543 MHz
NUC1 13C
P1 9.90 usec
PLW1 33.00000000 W

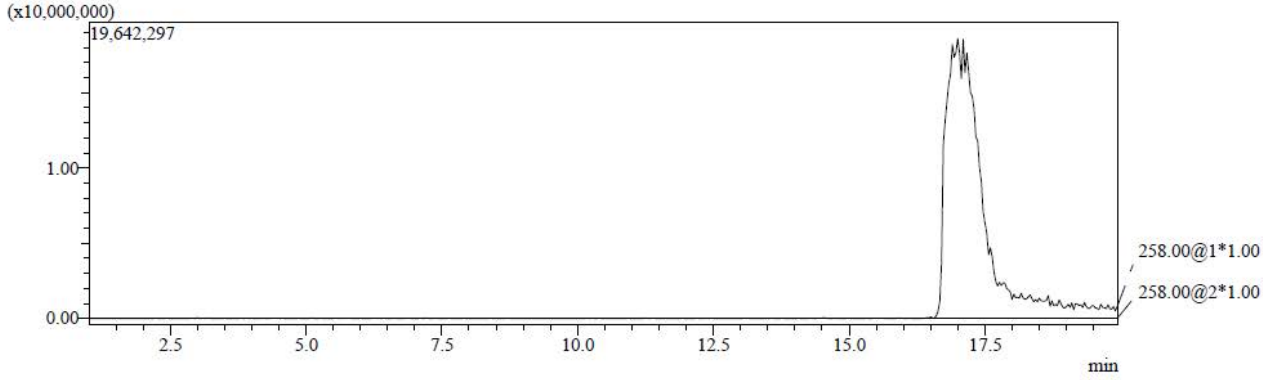
======== CHANNEL f2 ========
SFO2 300.1712007 MHz
NUC2 1H
CPDPRG[2 bi_waltz65_256
PCPD2 90.00 usec
PLW2 8.20349979 W
PLW12 0.27774000 W
PLW13 0.22497000 W

F2 - Processing parameters
SI 32768
SF 75.4777039 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

LCMS Compound 13-6

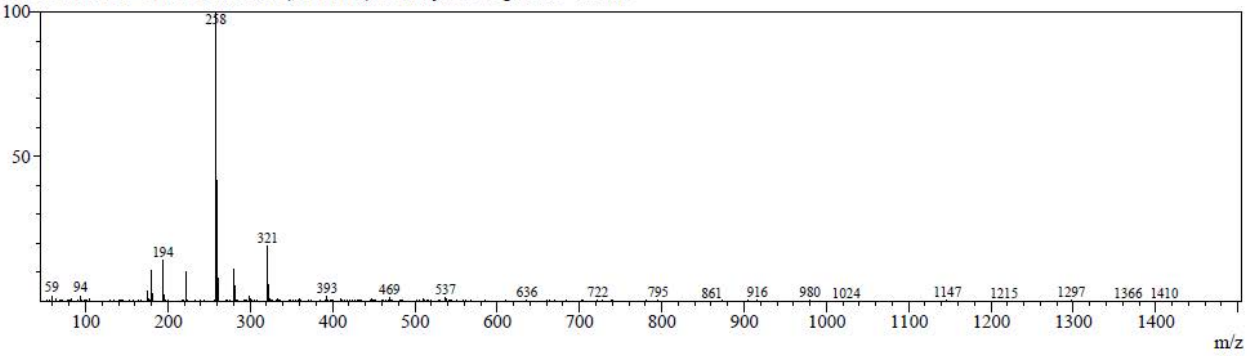




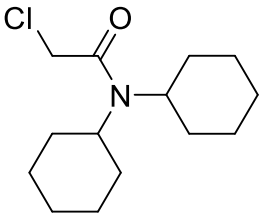


MS Spectrum Graph

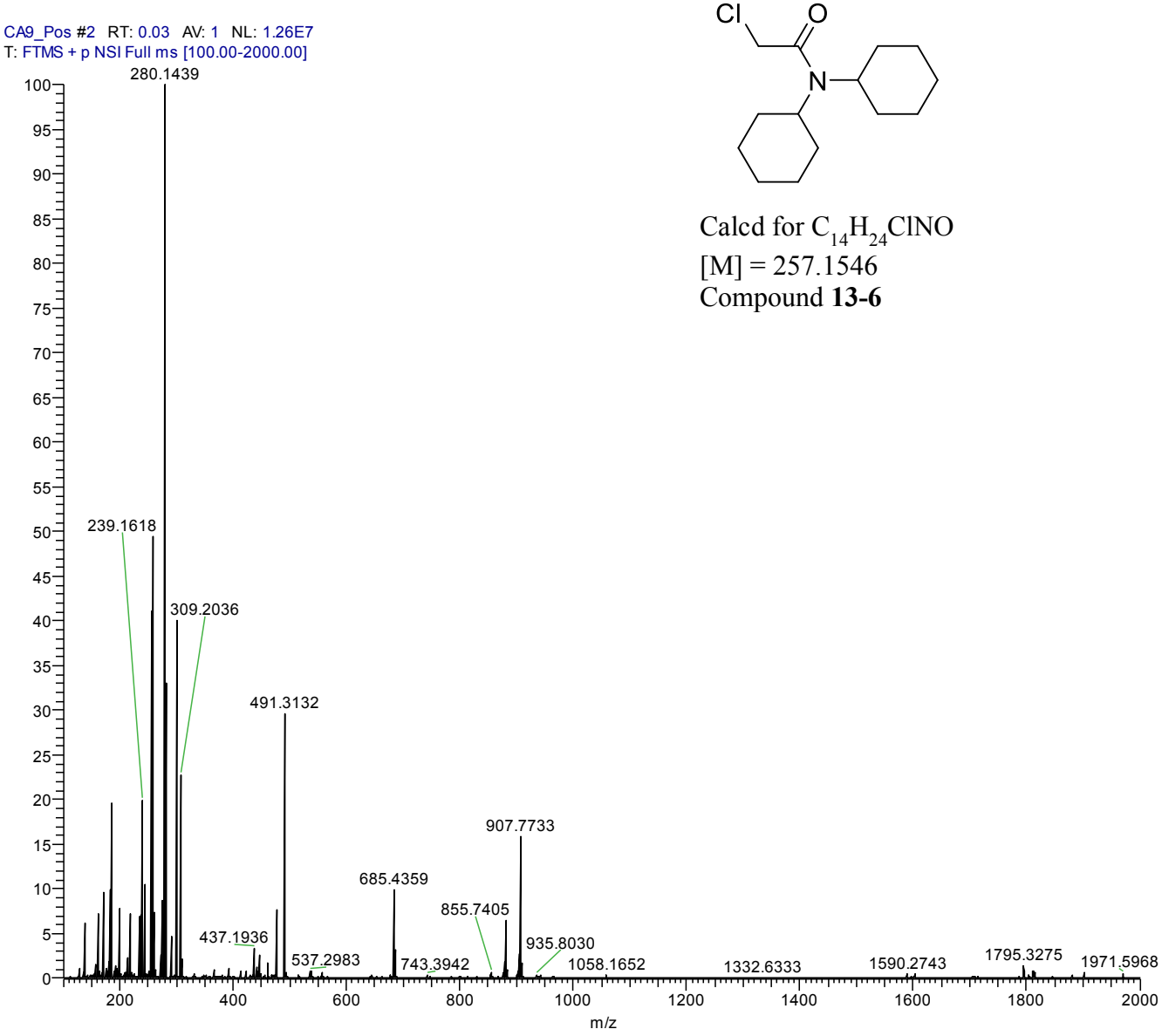
Ret.Time:17.233(Scan#:975)
BG Mode:None
Mass Peaks:1127 Base Peak:257.90(14994152) Polarity:Pos Segment1 - Event1



HRMS(ESI) Compound 13-6

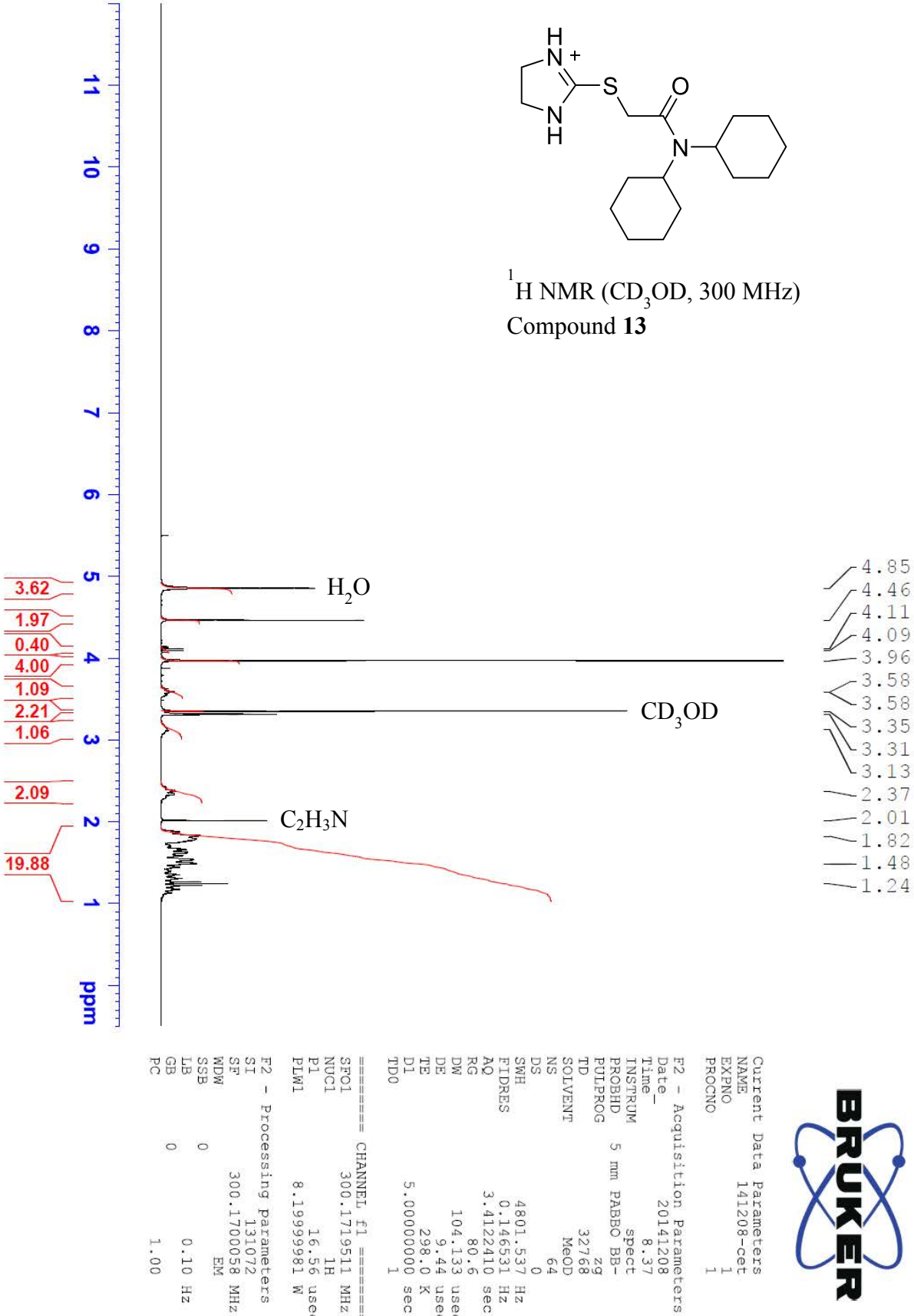


Calcd for $C_{14}H_{24}ClNO$
[M] = 257.1546
Compound **13-6**



^{1}H NMR Compound 13

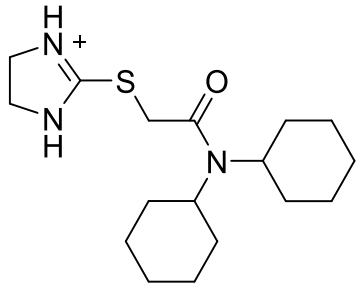
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N,N-dicyclohexylacetamide



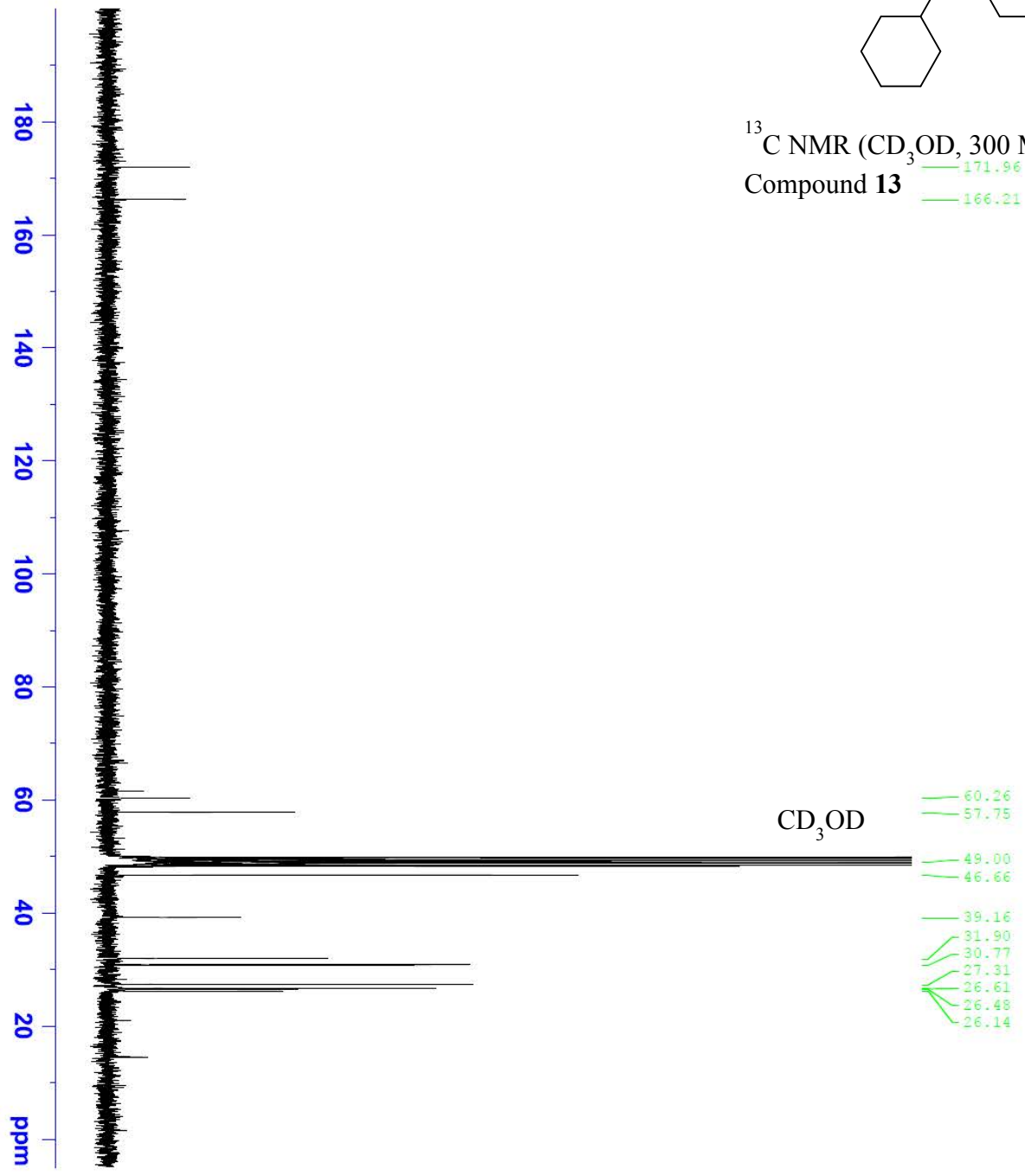
^{1}H NMR (CD_3OD, 300 MHz)
Compound **13**

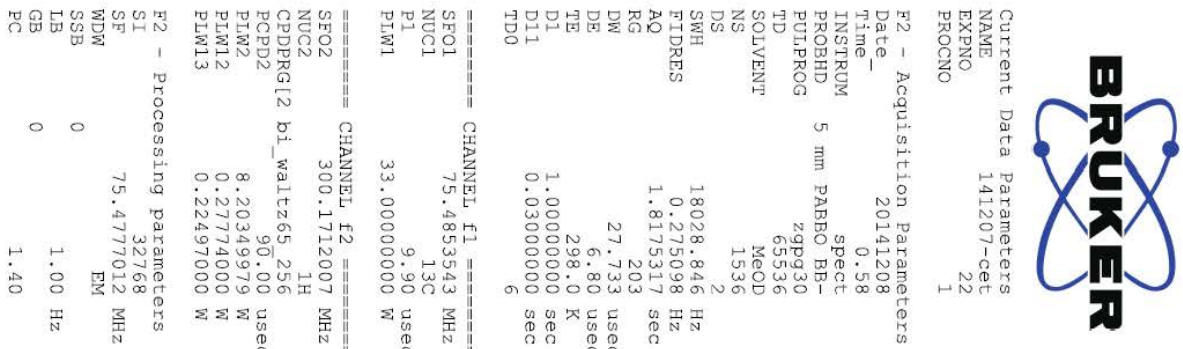
^{13}C NMR Compound 13

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N,N-dicyclohexylacetamide



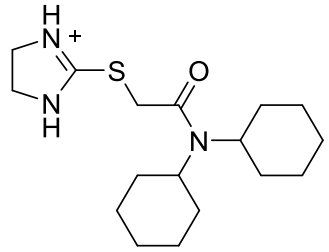
^{13}C NMR (CD_3OD, 300 MHz)
Compound **13**



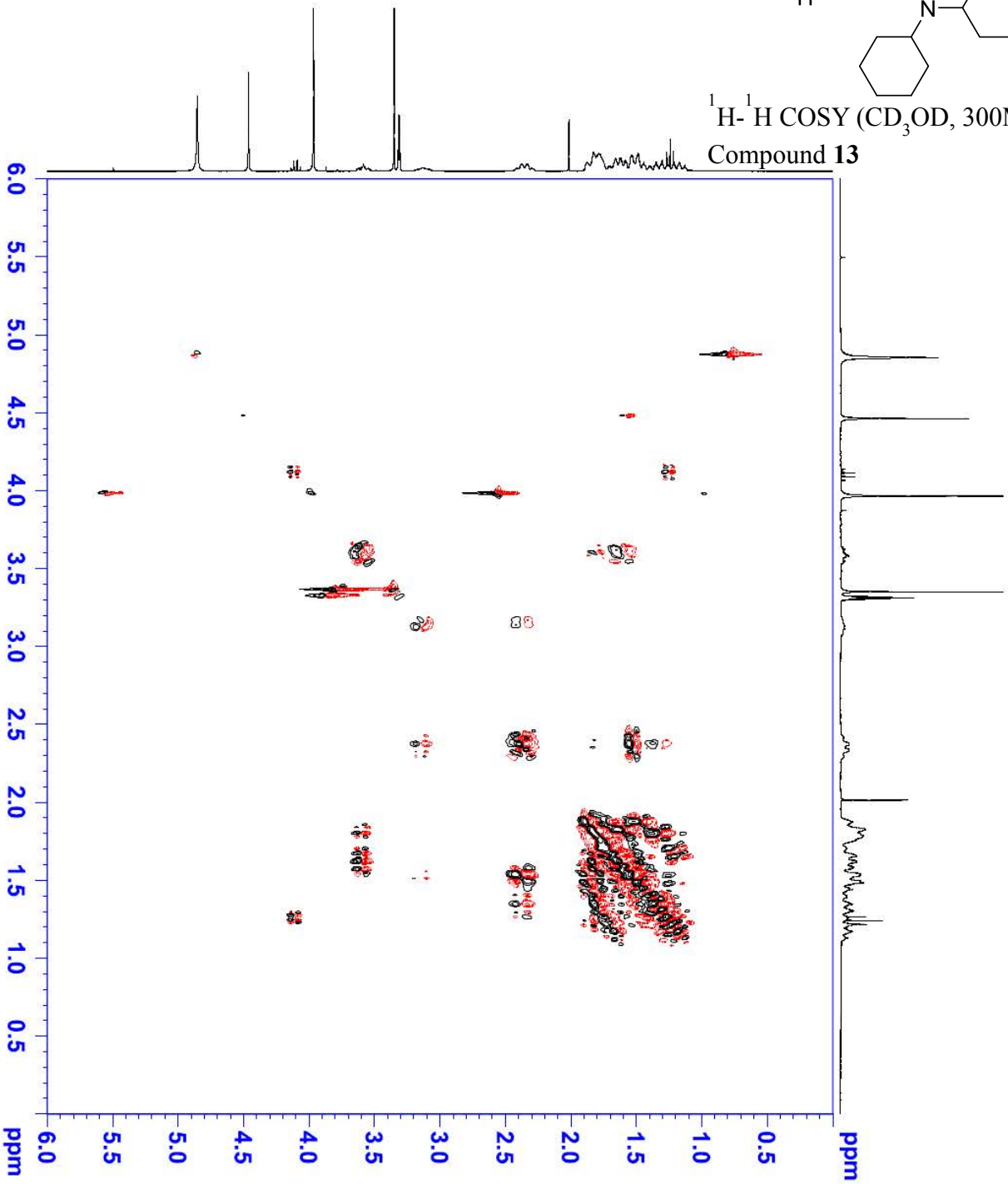


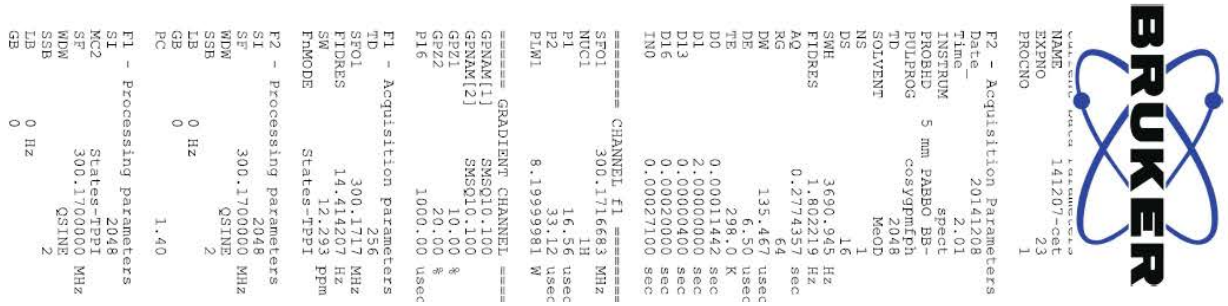
^{1}H-^{1}H COSY Compound 13

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N,N-dicyclohexylacetamide



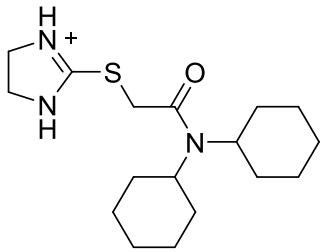
^{1}H-^{1}H COSY (CD_3OD, 300MHz)
Compound **13**

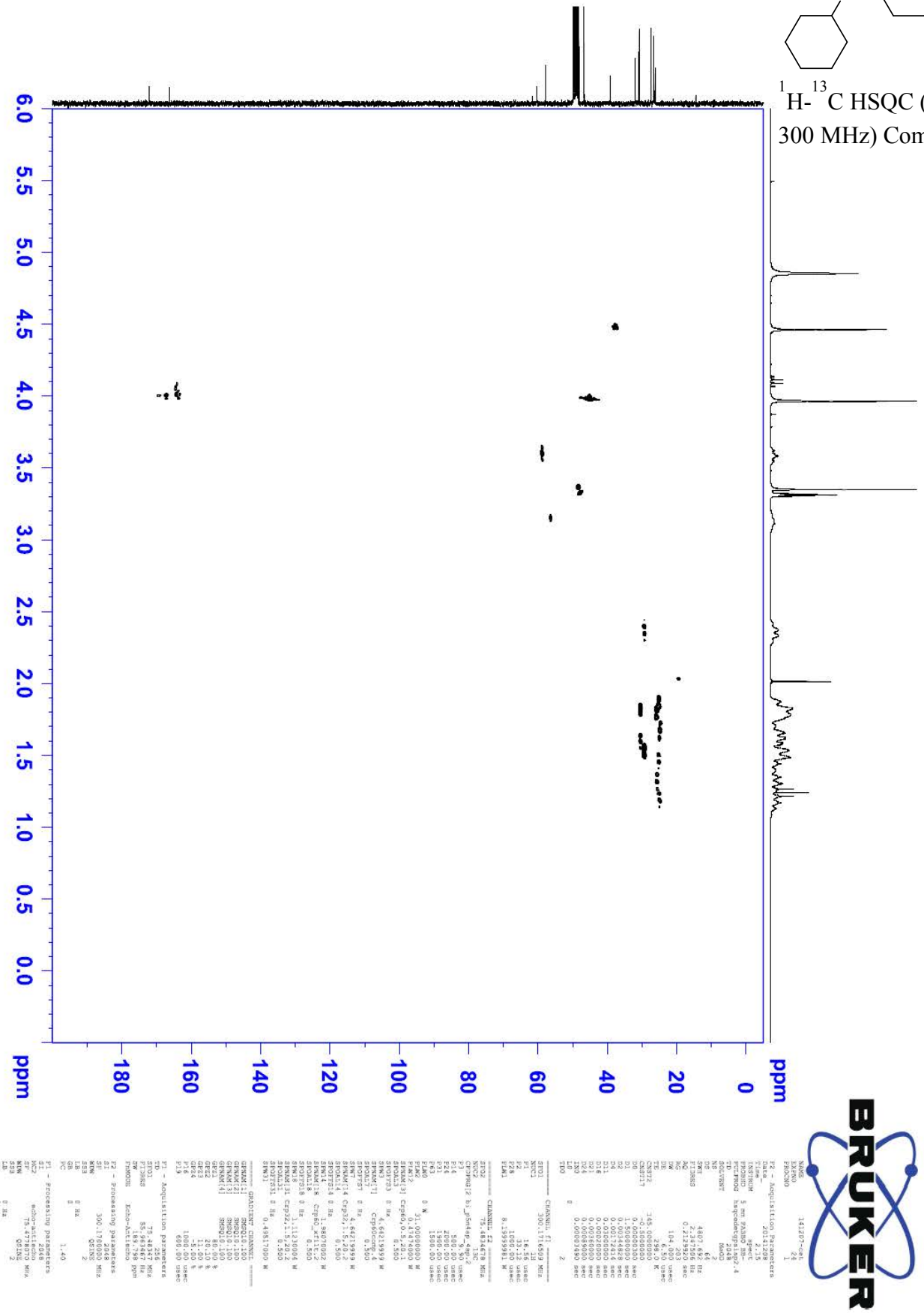




^{1}H-^{13}C HSQC Compound 13

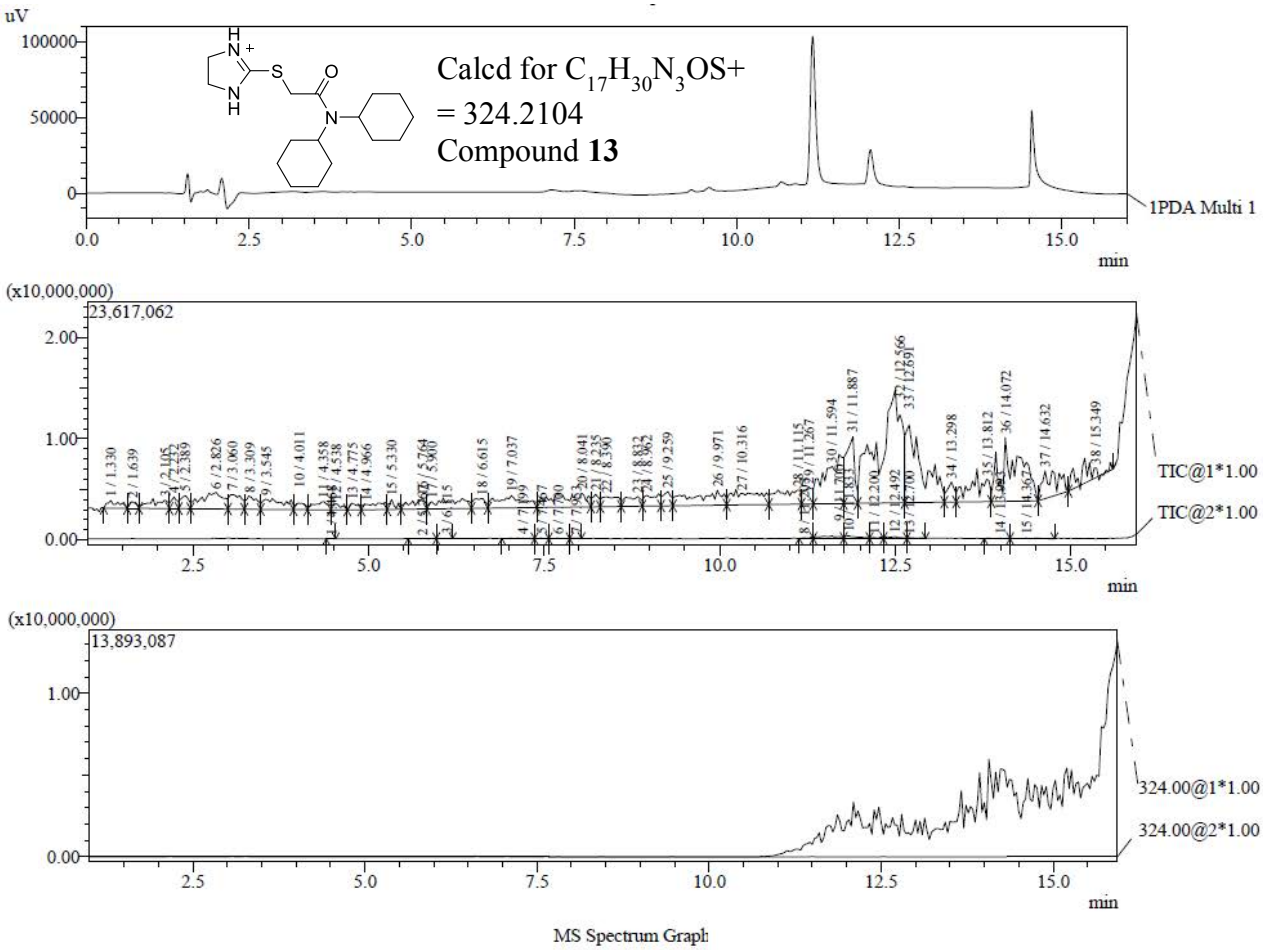
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N,N-dicyclohexylacetamide





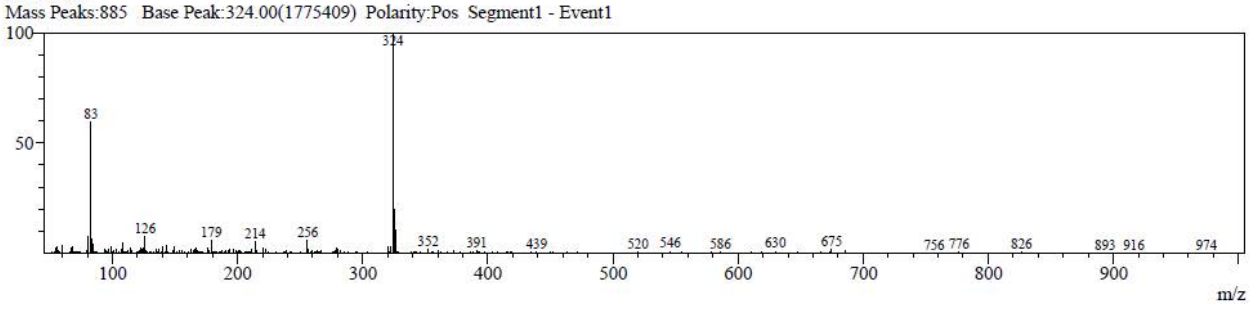
^{1}H-^{13}C HSQC (CD_3OD, 300 MHz) Compound **13**

LCMS Compound 13



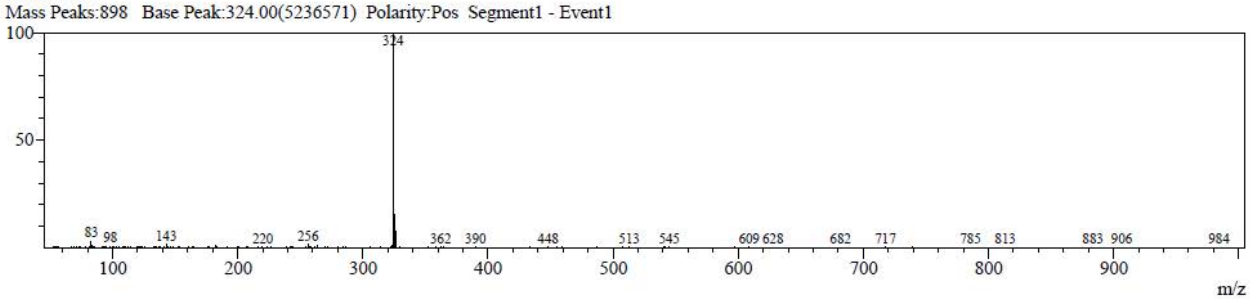
MS Spectrum Graph

Ret.Time:12.300(Scan#:679)
BG Mode:?
Mass Peaks:885 Base Peak:324.00(1775409) Polarity:Pos Segment1 - Event1

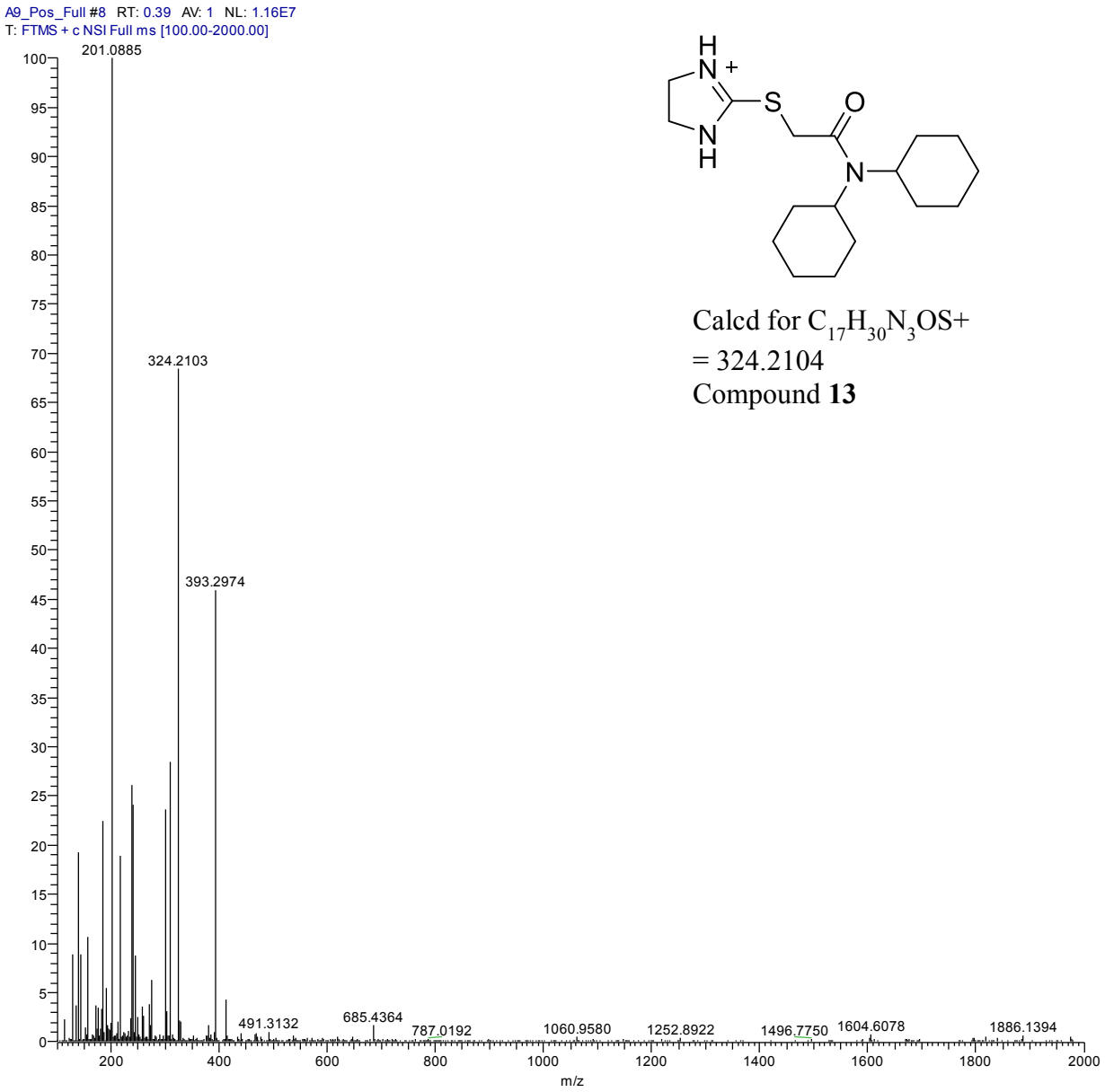


MS Spectrum Graph

Ret.Time:14.300(Scan#:799)
BG Mode:?
Mass Peaks:898 Base Peak:324.00(5236571) Polarity:Pos Segment1 - Event1

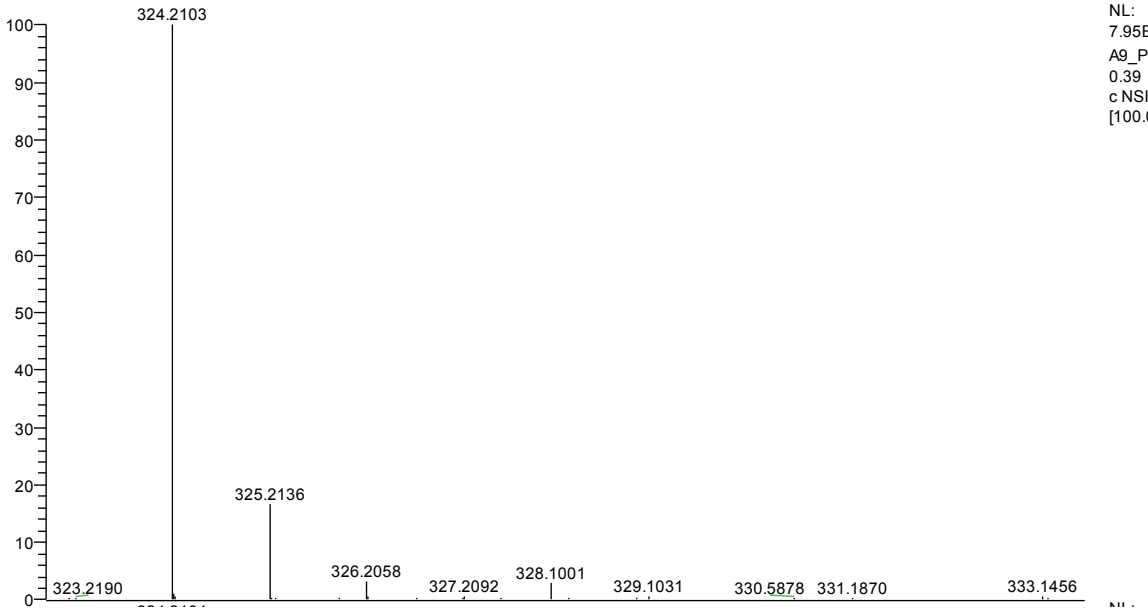


HRMS(ESI) Compound 13



Calcd for $C_{17}H_{30}N_3OS^+$ = 324.2104

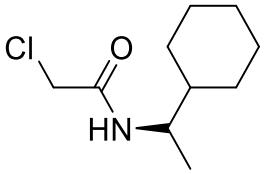
Compound **13**



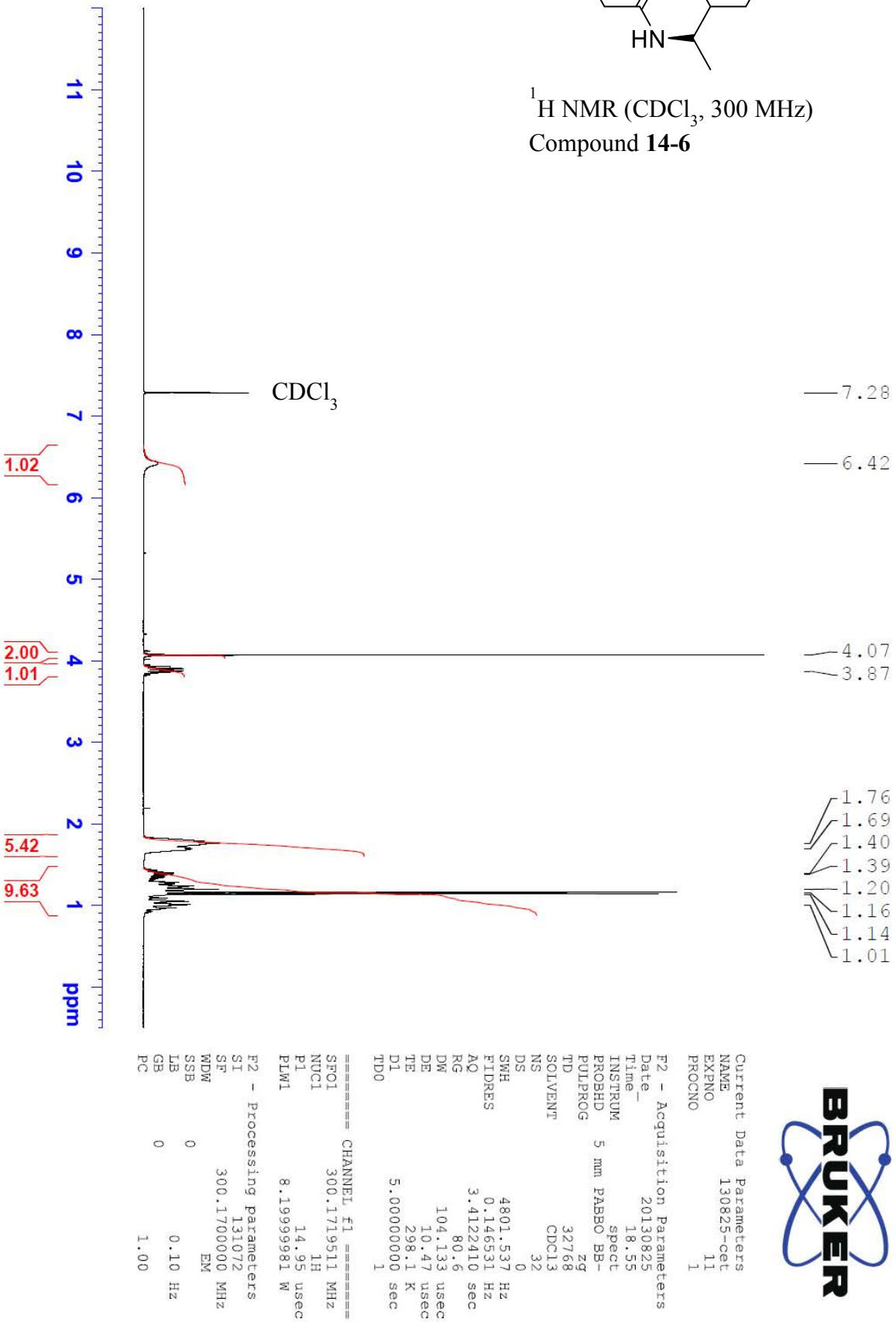
Spectra for Compound 14

^{1}H NMR Compound 14-6

(R)-2-chloro-N-(1-cyclohexylethyl)acetamide

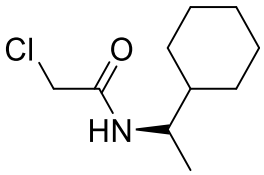


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **14-6**

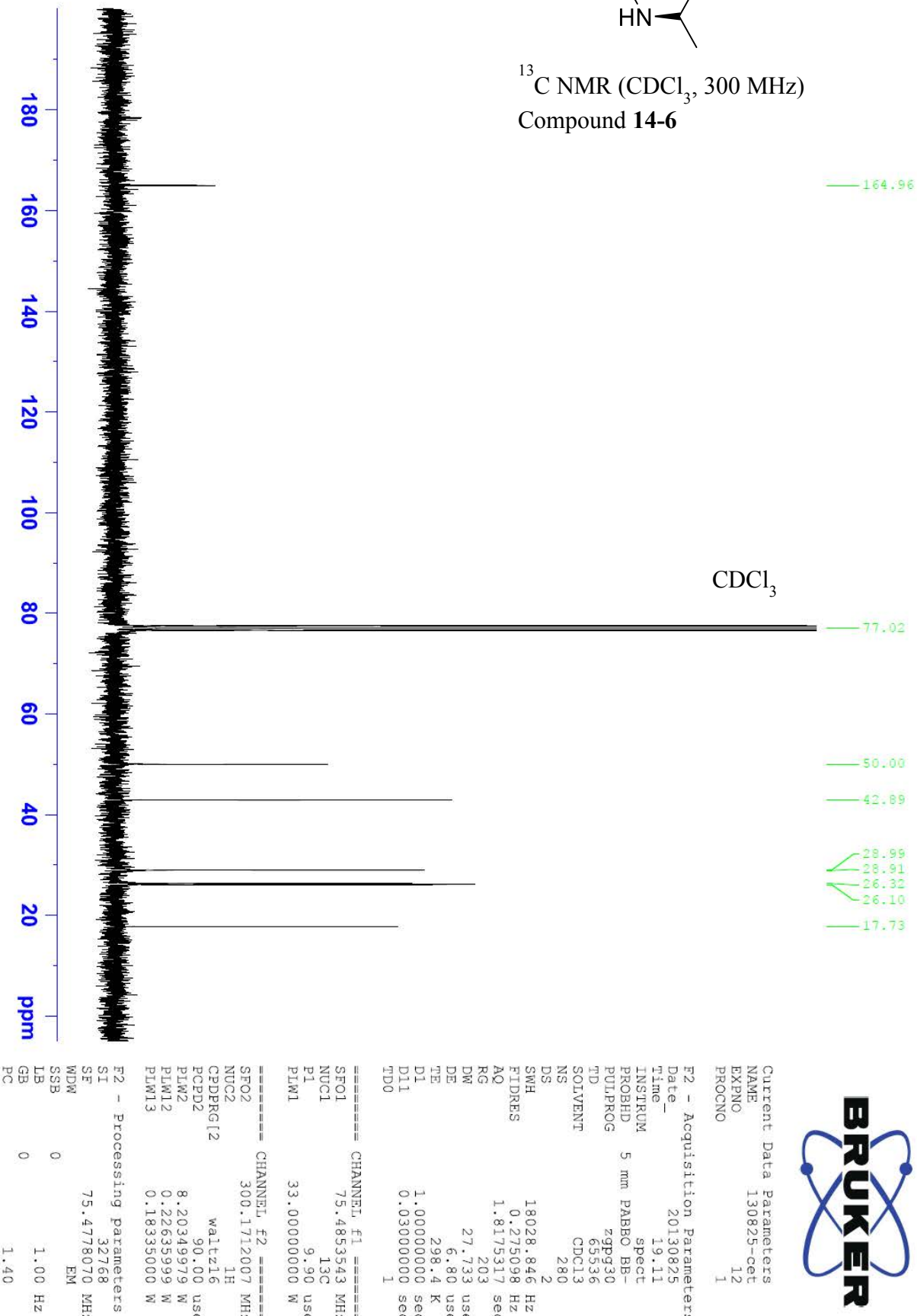


^{13}C NMR Compound 14-6

(R)-2-chloro-N-(1-cyclohexylethyl)acetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **14-6**

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Current Data Parameters
NAME                  130825-cet
EXPNO                         12
PROCNO                         1

F2 - Acquisition Parameters
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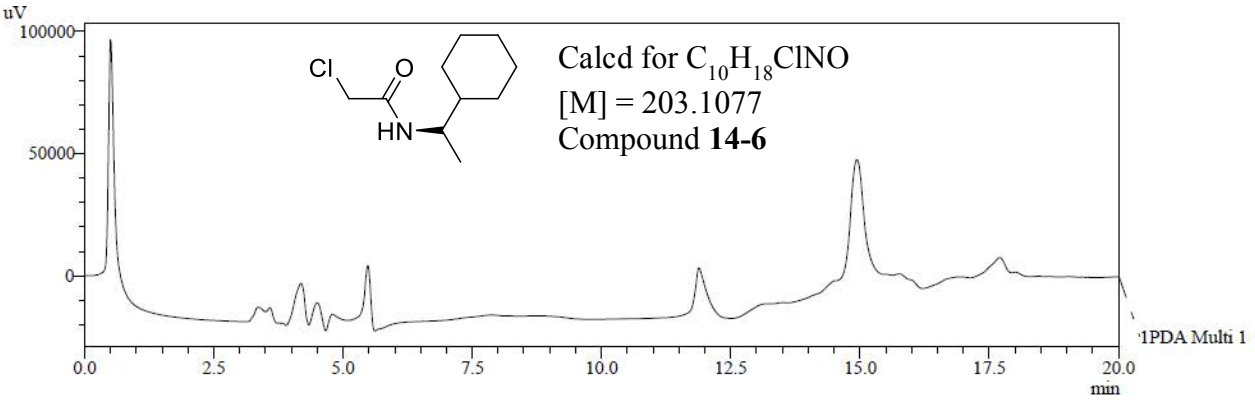
Time_                   19.11
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     280
DS                       2
SWH              18028.846 Hz
FIDRES               0.275098 Hz
AQ                1.8175317 sec
RG                     203
DW                27.733 usec
DE                 6.80 usec
TE                 298.4 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                       1

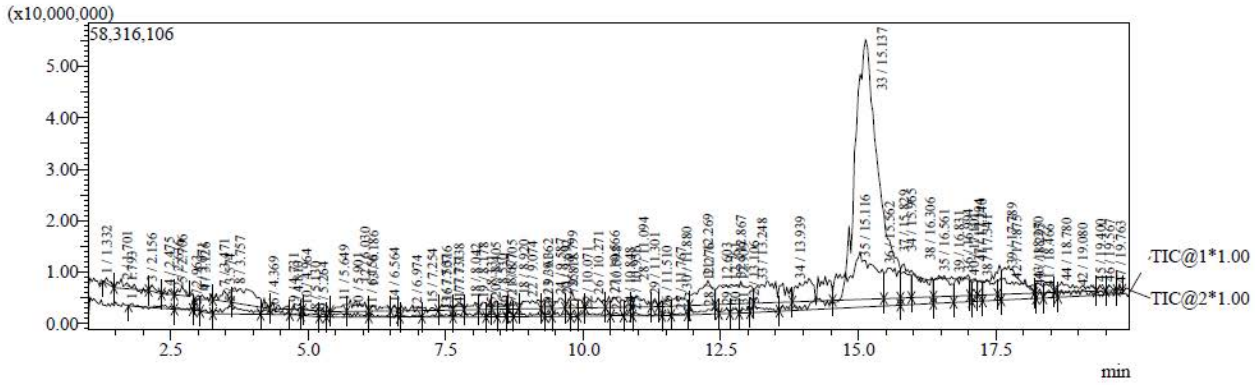
======== CHANNEL f1 ========
SFO1            75.4853543 MHz
NUC1                     13C
P1                      9.90 usec
PLW1           33.00000000 W

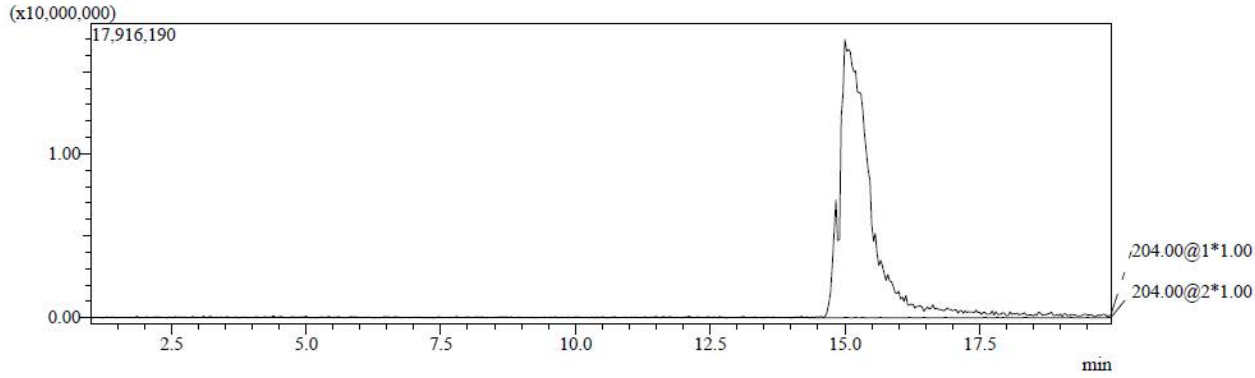
======== CHANNEL f2 ========
SFO2           300.1712007 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2           8.20349979 W
PLW12          0.22635999 W
PLW13          0.18335000 W

F2 - Processing parameters
SI                       32768
SF              75.4778070 MHz
WDW                         EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

LCMS Compound 14-6

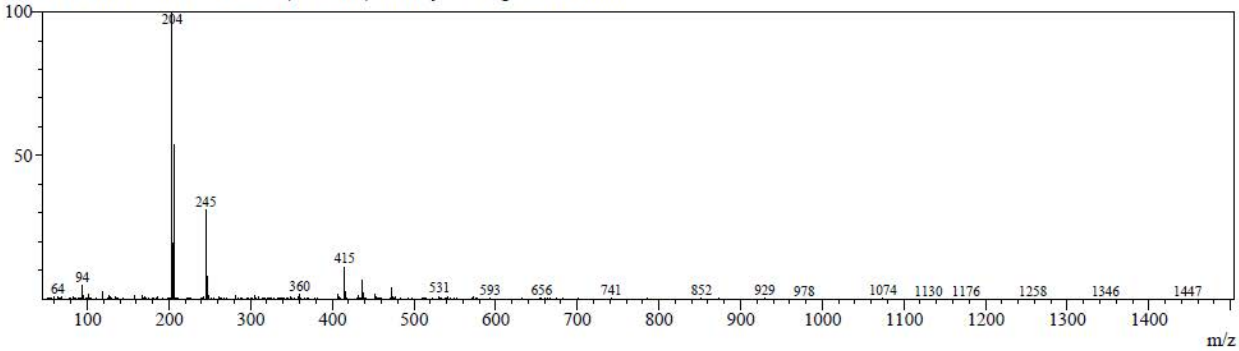




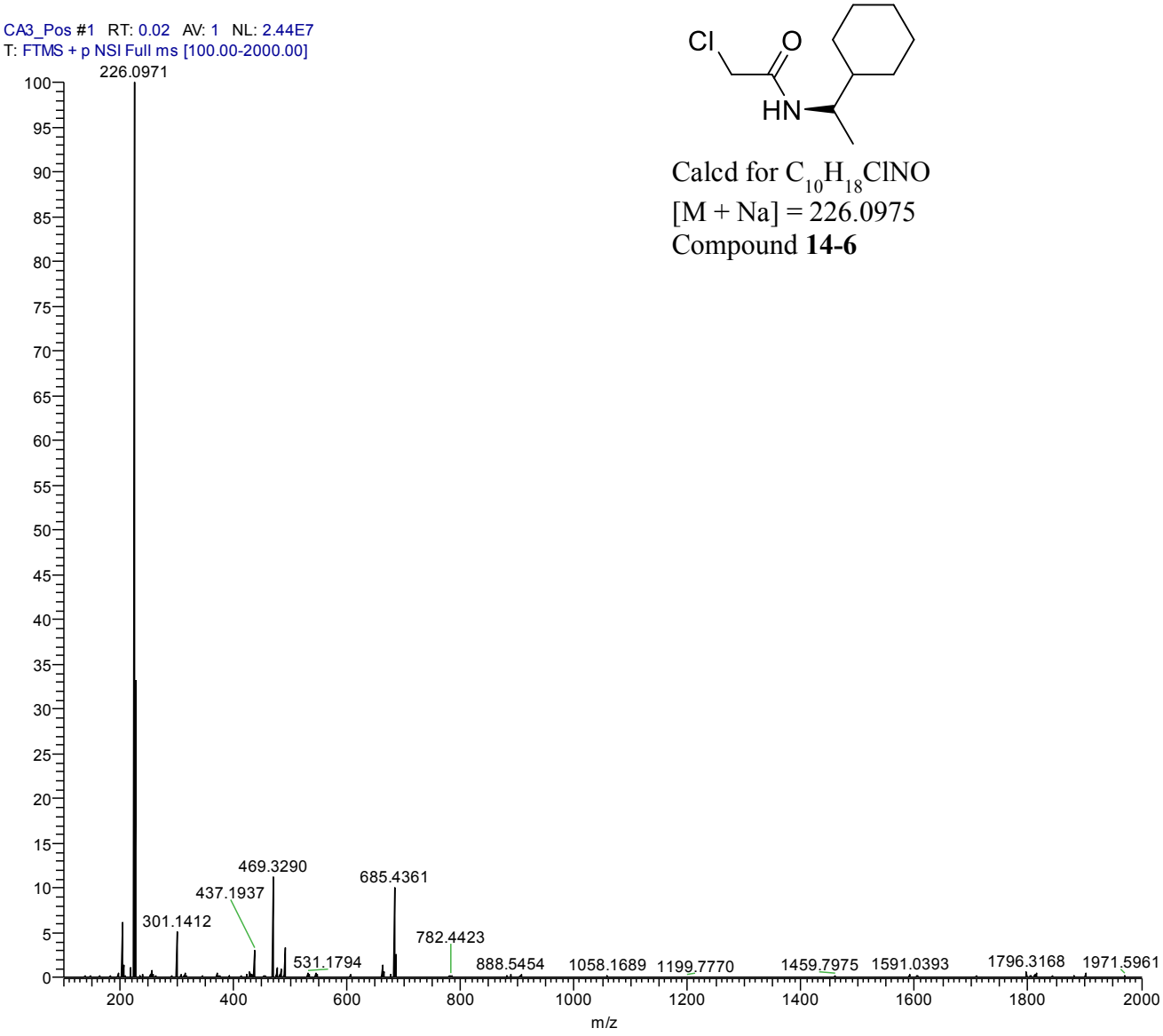


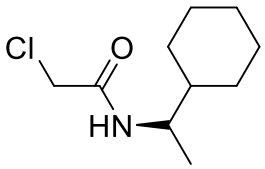
MS Spectrum Graph

Ret.Time:15.100(Scan#:847)
BG Mode:None
Mass Peaks:1194 Base Peak:203.80(16233198) Polarity:Pos Segment1 - Event1



HRMS(ESI) Compound 14-6

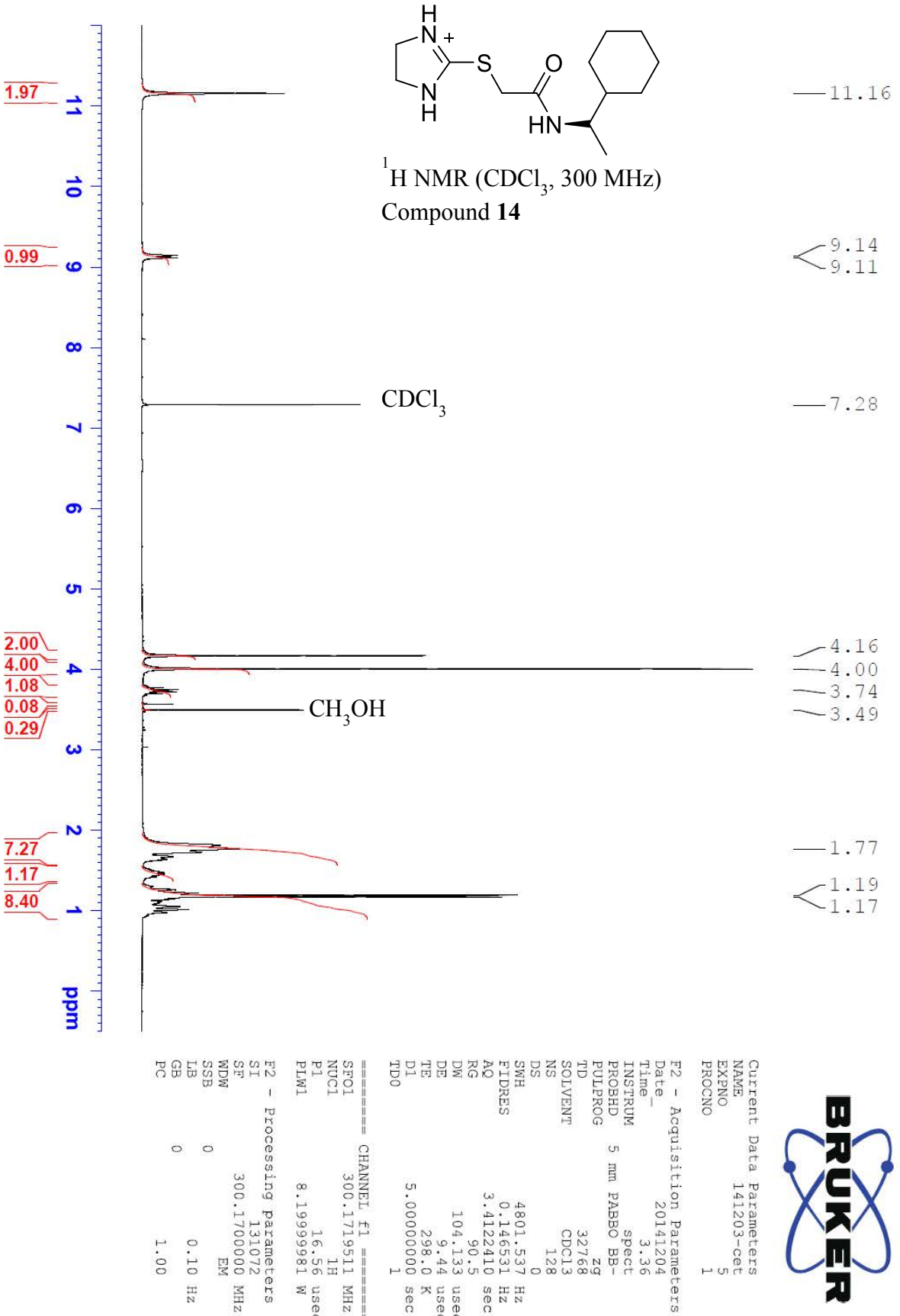




Calcd for $C_{10}H_{18}ClNO$
[M + Na] = 226.0975
Compound **14-6**

^{1}H NMR Compound 14

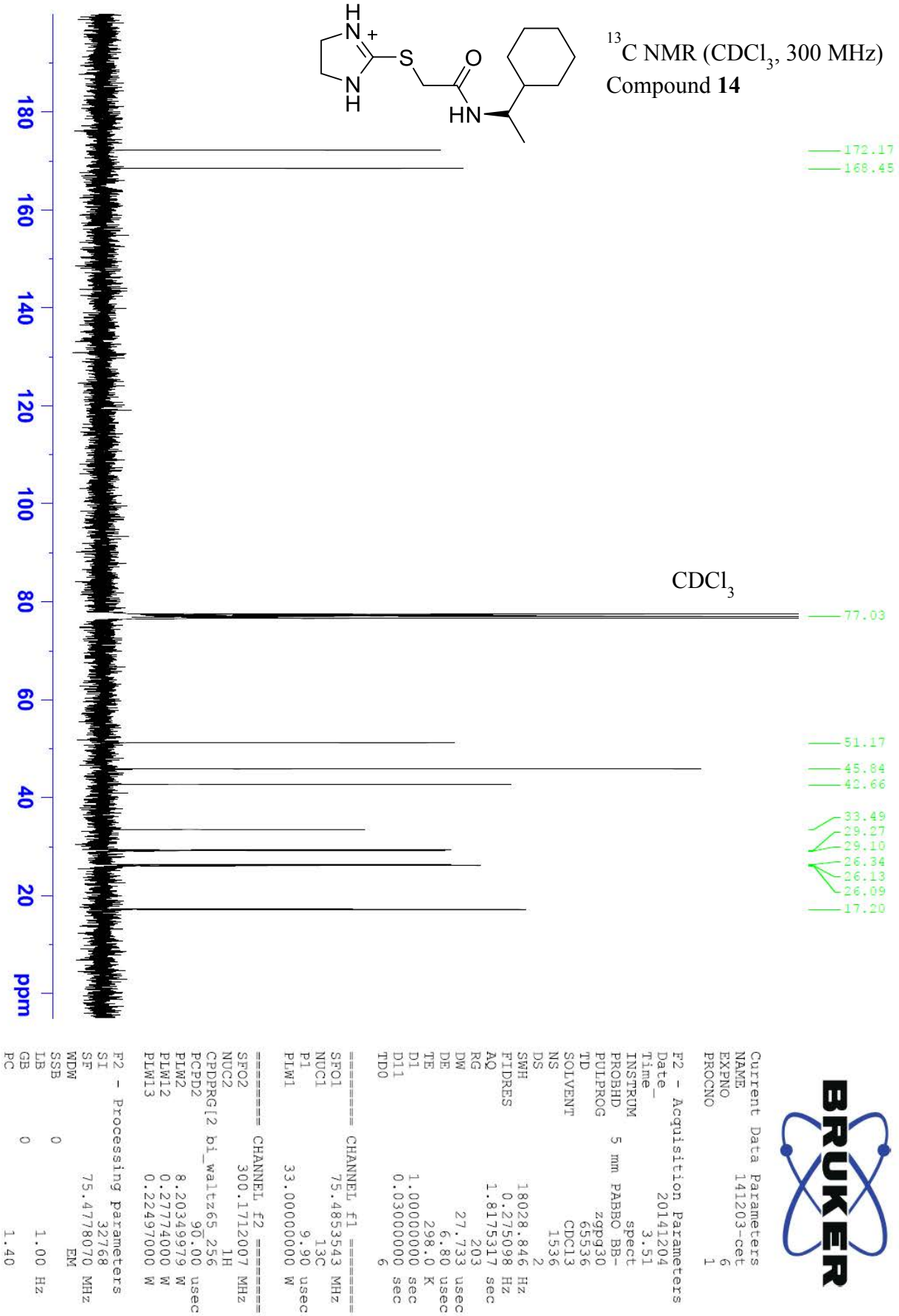
(R)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(1-cyclohexylethyl)acetamide



^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **14**

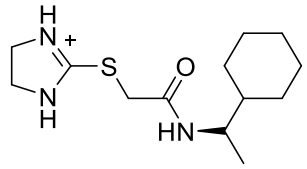
^{13}C NMR Compound 14

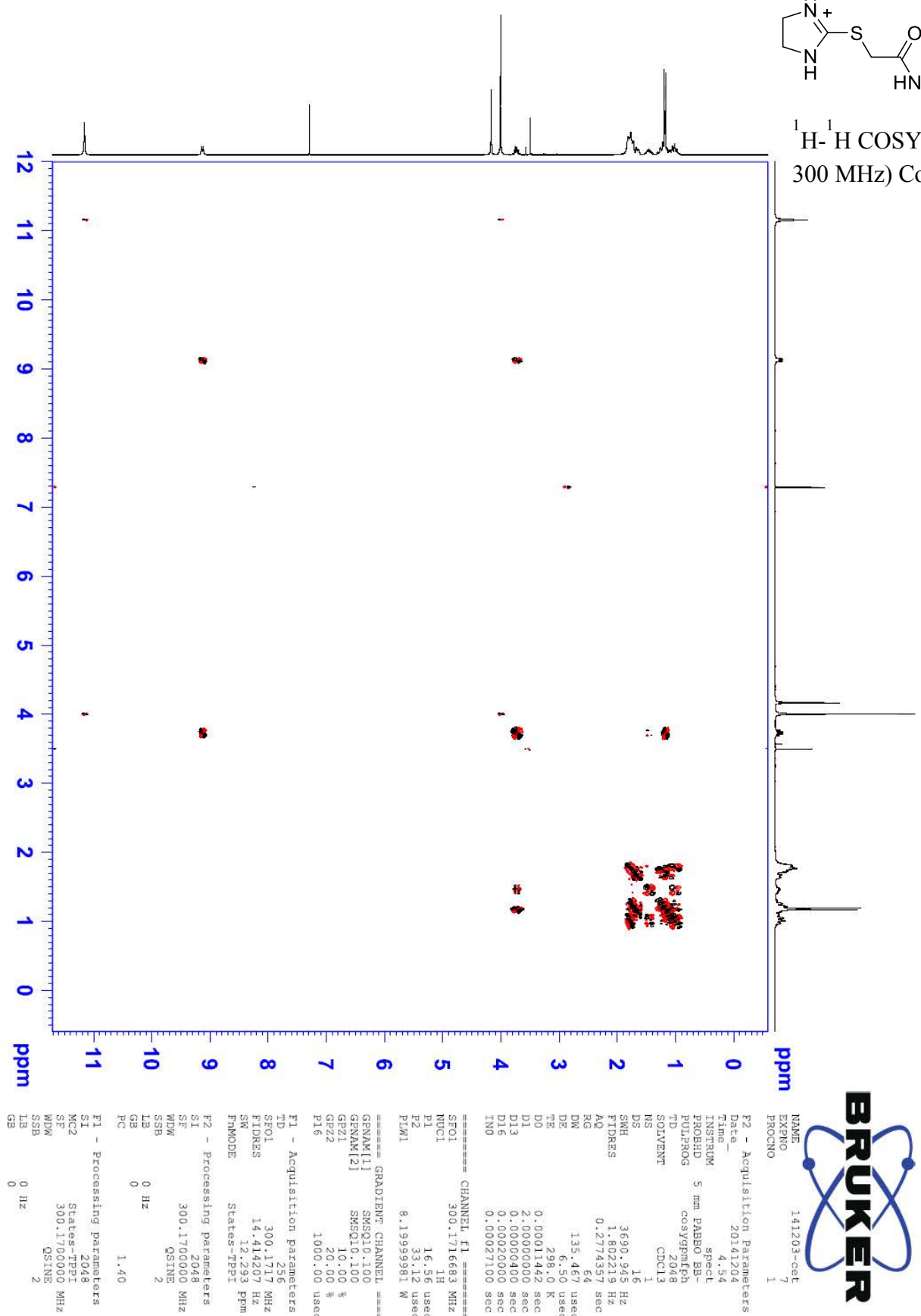
(R)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(1-cyclohexylethyl)acetamide



^{1}H-^{1}H COSY Compound 14

(R)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(1-cyclohexylethyl)acetamide

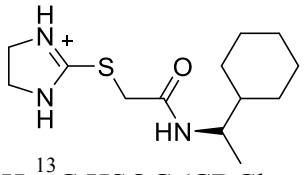




^{1}H-^{1}H COSY ($CDCl_3$, 300 MHz) Compound **14**

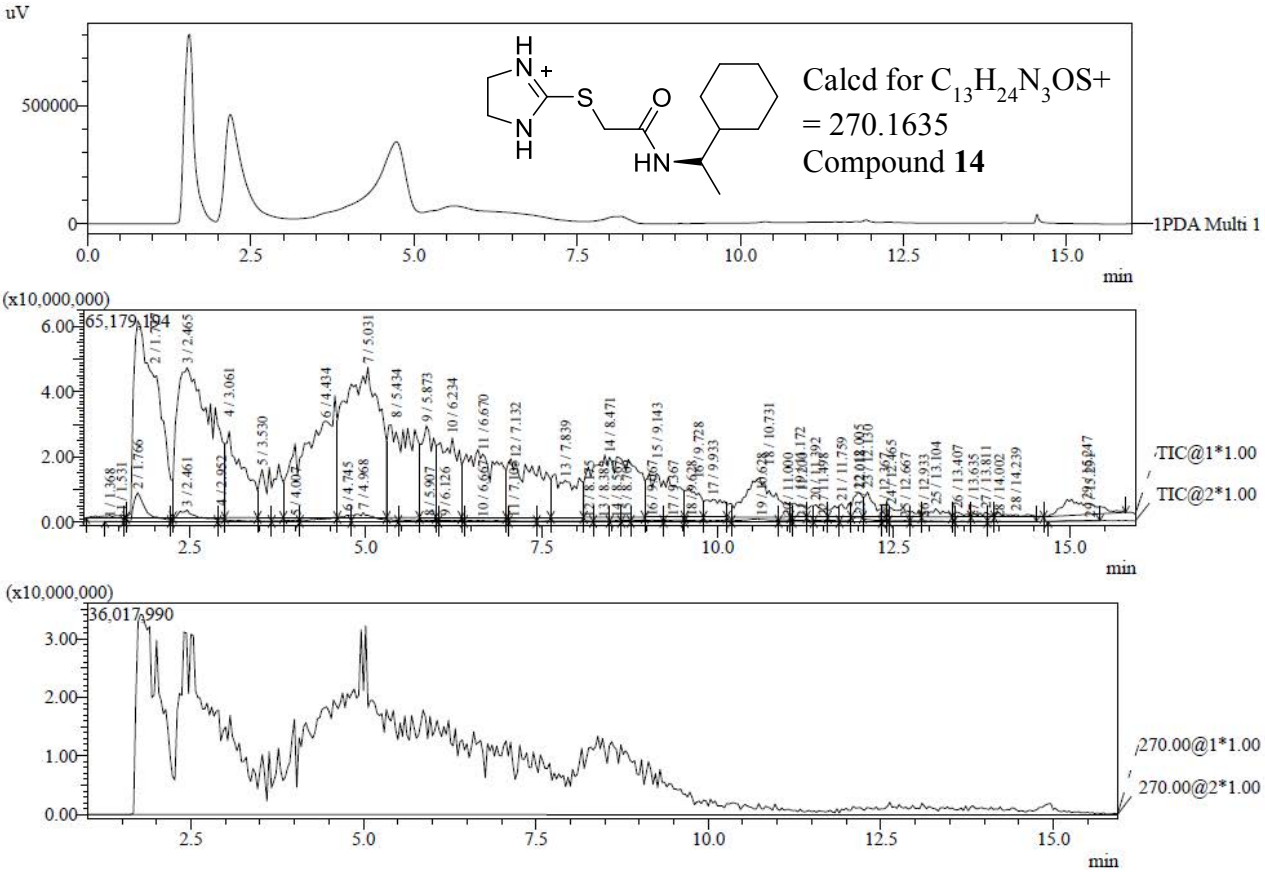
^{1}H-^{13}C HSQC Compound 14

(R)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(1-cyclohexylethyl)acetamide



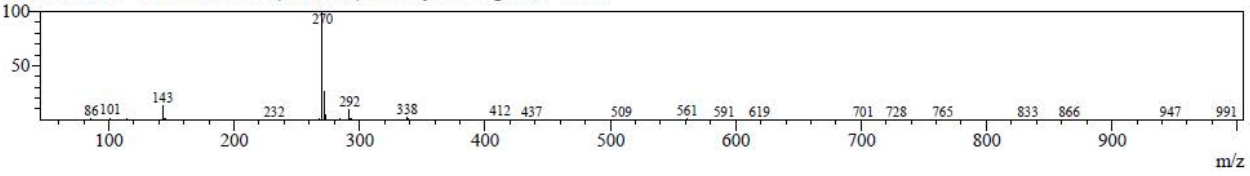
^{1}H-^{13}C HSQC ($CDCl_3$, 300MHz) Compound **14**

LCMS Compound 14



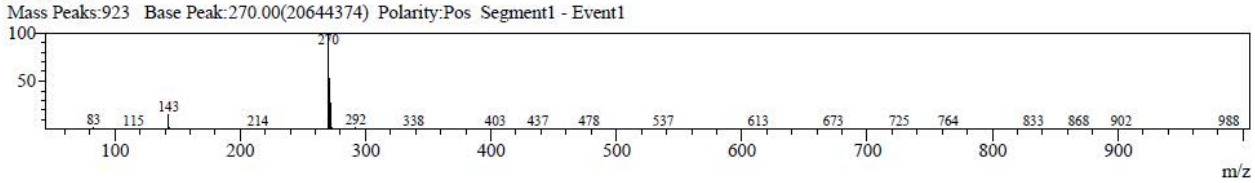
MS Spectrum Graph

Ret.Time:1.767(Scan#:47)
BG Mode:None
Mass Peaks:919 Base Peak:270.35(34122307) Polarity:Pos Segment1 - Event



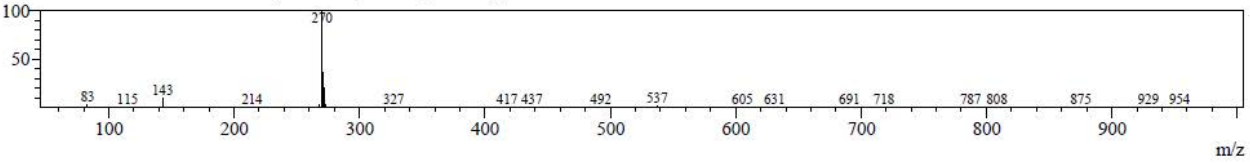
MS Spectrum Graph

Ret.Time:2.467(Scan#:89)
BG Mode:?
Mass Peaks:923 Base Peak:270.00(20644374) Polarity:Pos Segment1 - Event1

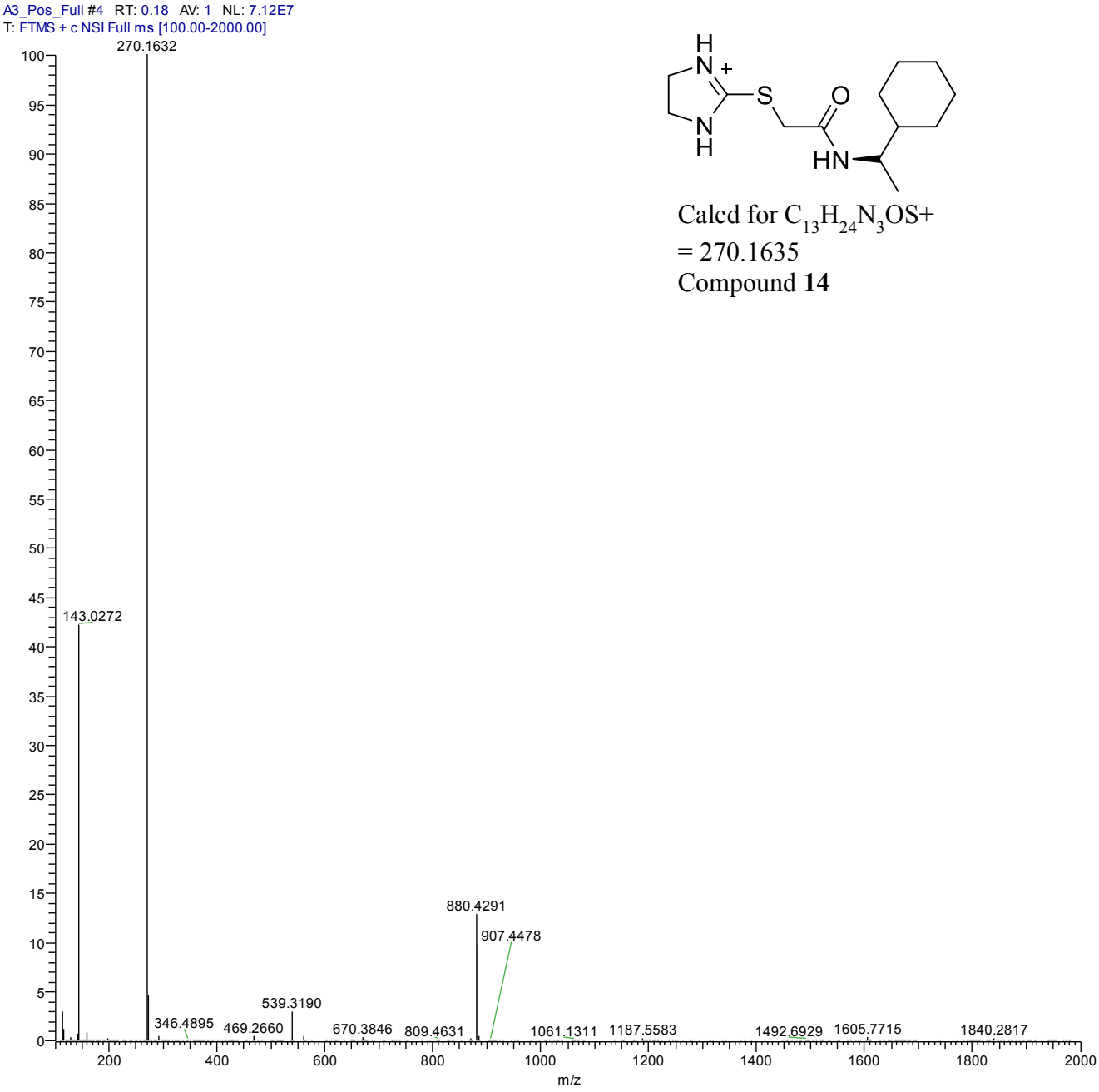


MS Spectrum Graph

Ret.Time:5.000(Scan#:241)
BG Mode:None
Mass Peaks:923 Base Peak:269.95(21124823) Polarity:Pos Segment1 - Event



HRMS(ESI) Compound 14



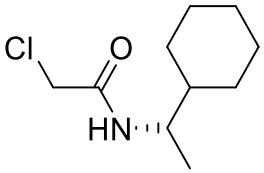
Calcd for $C_{13}H_{24}N_3OS^+$ = 270.1635

Compound **14**

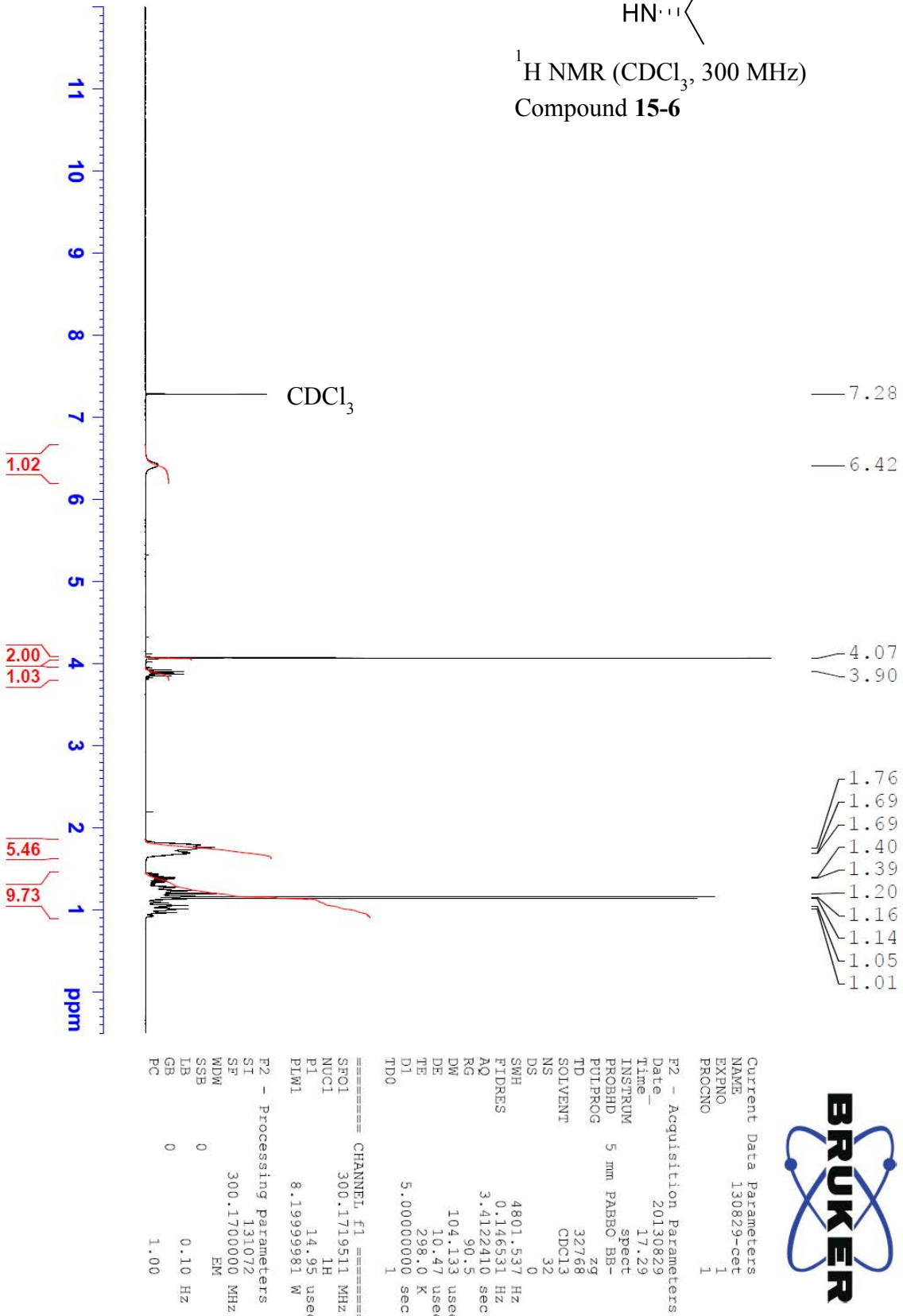
Spectra for Compound 15

^{1}H NMR Compound 15-6

(S)-2-chloro-N-(1-cyclohexylethyl)acetamide

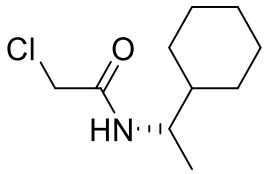


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **15-6**

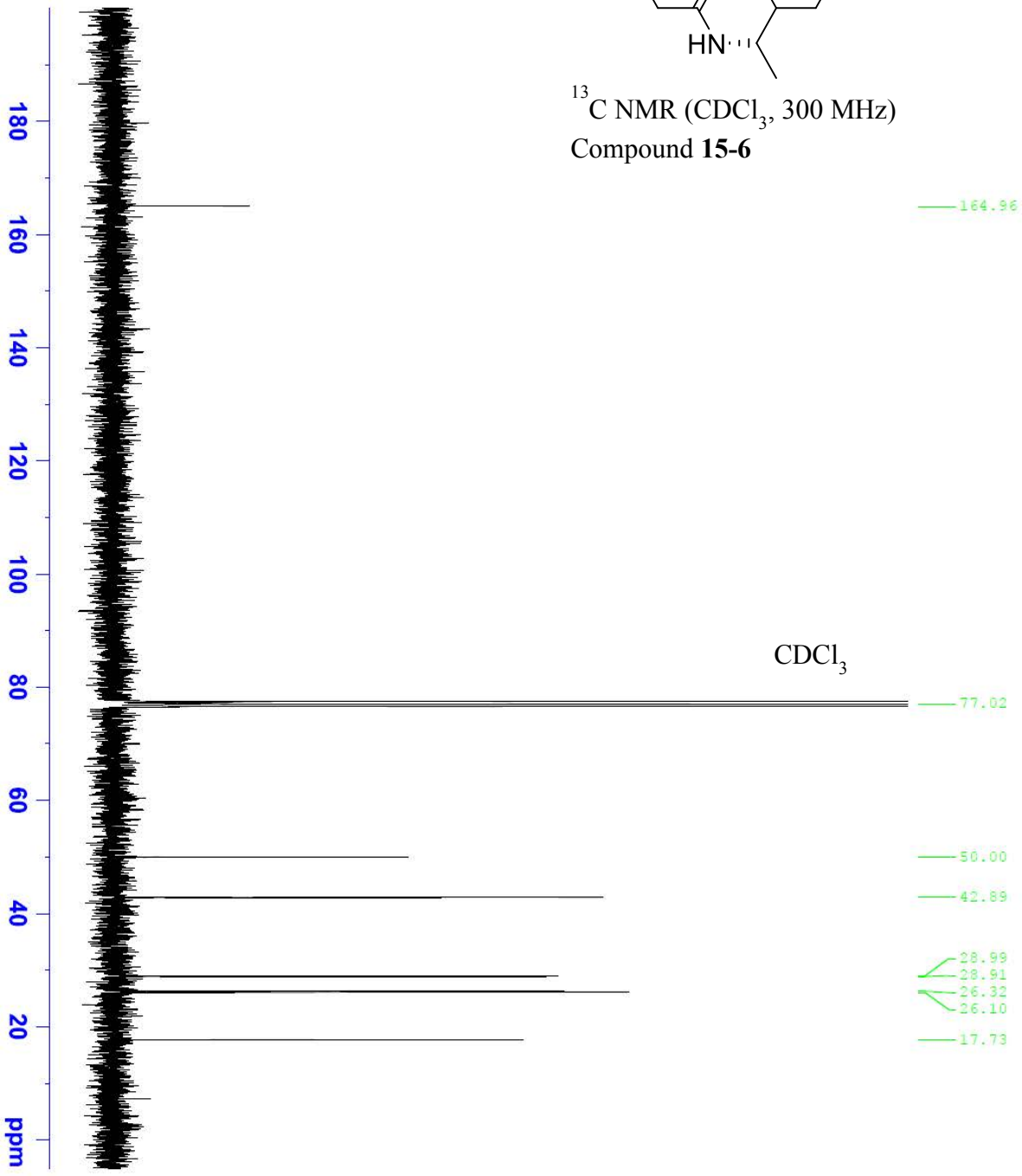


^{13}C NMR Compound 15-6

(S)-2-chloro-N-(1-cyclohexylethyl)acetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **15-6**

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Current Data Parameters
NAME                  130829-cet
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20130829
Time_                      17.44
INSTRUM                     spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         280
DS                           2
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                         203
DW                      27.733 usec
DE                       6.80 usec
TE                     298.0 K
D1                     1.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                 75.4853543 MHz
NUC1                        13C
P1                         9.90 usec
PLW1                33.00000000 W

======== CHANNEL f2 ========
SFO2                 300.1712007 MHz
NUC2                        1H
CPDPRG[2                 waltz16
PCPD2                     90.00 usec
PLW2                 8.20349979 W
PLW12                0.22635999 W
PLW13                0.18335000 W

F2 - Processing parameters
SI                         32768
SF                   75.4778070 MHz
WDW                           EM
SSB                            0
LB                      1.00 Hz
GB                             0
PC                          1.40
```
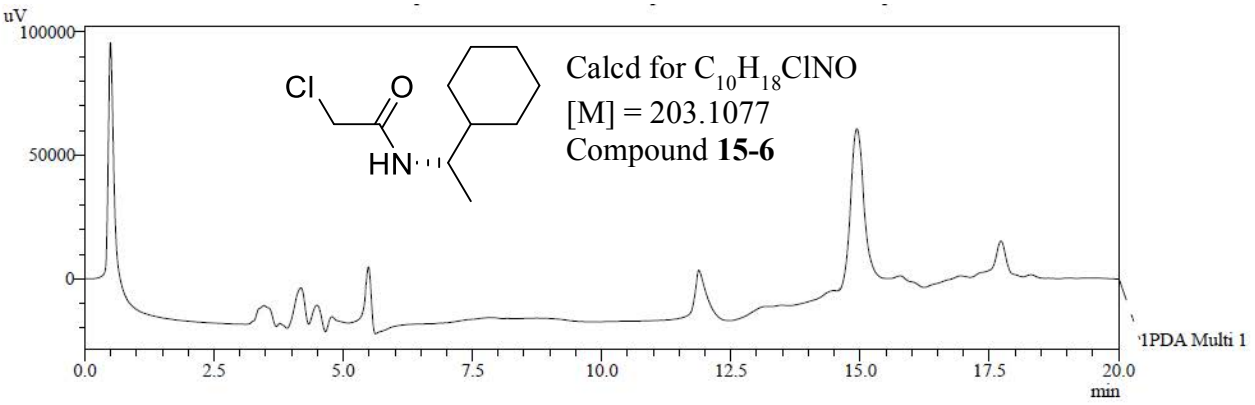
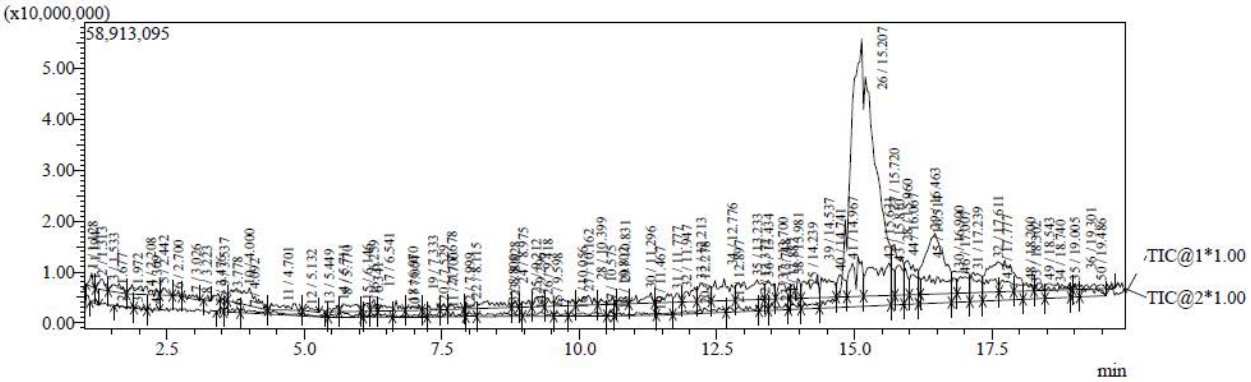
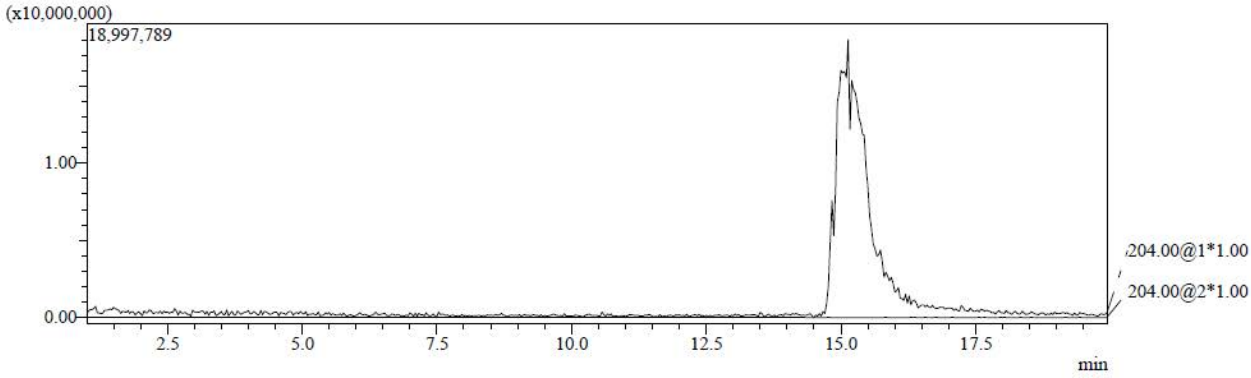

LCMS Compound 15-6

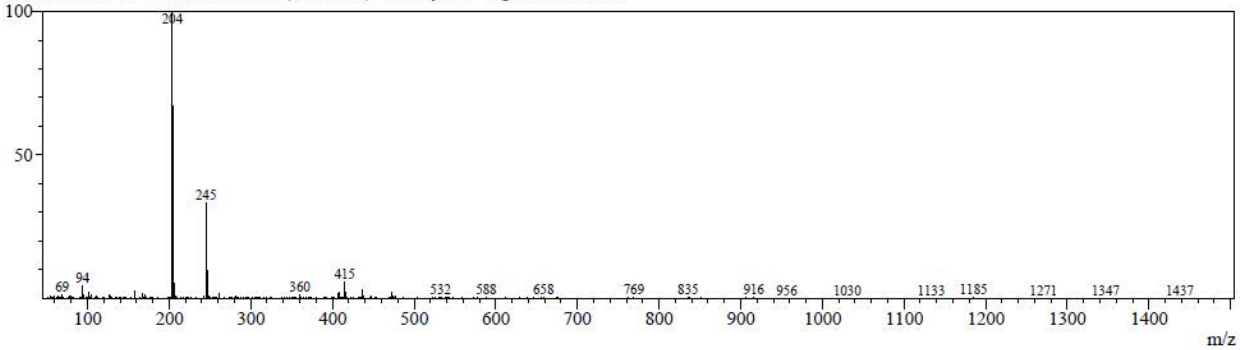




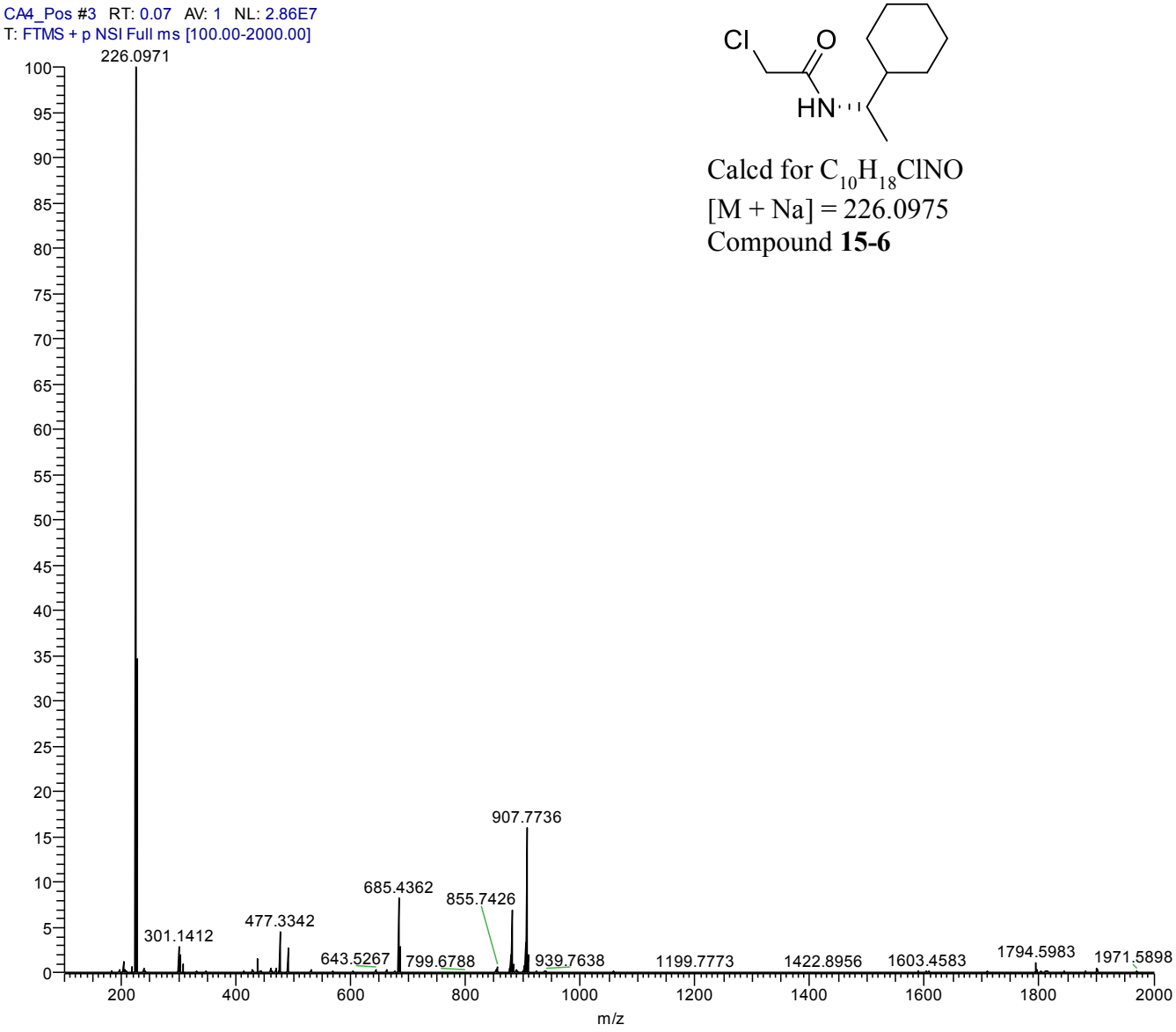


MS Spectrum Graph

Ret.Time:15.033(Scan#:843)
BG Mode:?
Mass Peaks:1212 Base Peak:203.80(15841496) Polarity:Pos Segment1 - Event1



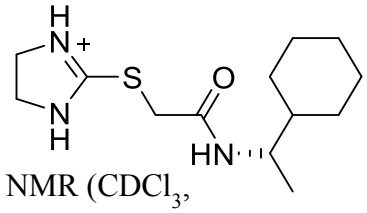
HRMS(ESI) Compound 15-6



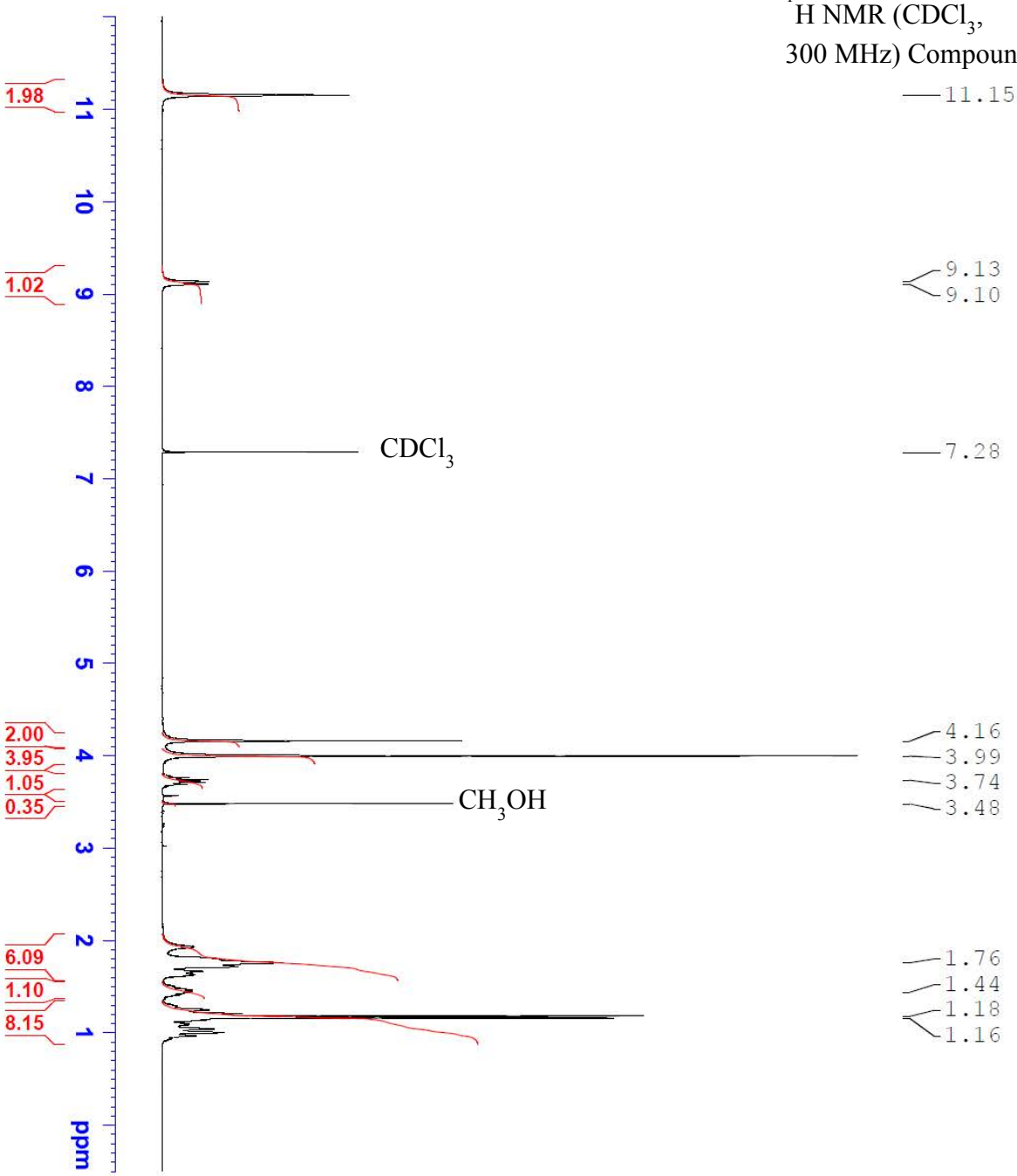
Calcd for $C_{10}H_{18}ClNO$
[M + Na] = 226.0975
Compound **15-6**

^{1}H NMR Compound 15

(S)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(1-cyclohexylethyl)acetamide



^{1}H NMR ($CDCl_3$, 300 MHz) Compound **15**



Current Data Parameters
NAME 141207-cet
EXPNO 5
PROCNO 1

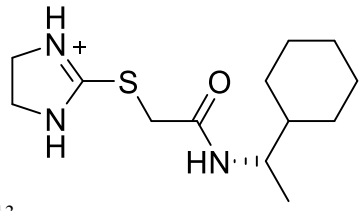
F2 - Acquisition Parameters
Date_ 20141207
Time 16.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 4801.537 Hz
FIDRES 0.146531 Hz
AQ 3.4122410 sec
RG 71.8
DW 104.133 usec
DE 9.44 usec
TE 298.0 K
D1 5.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 300.1719511 MHz
NUC1 1H
P1 16.56 usec
PLW1 8.19999981 W

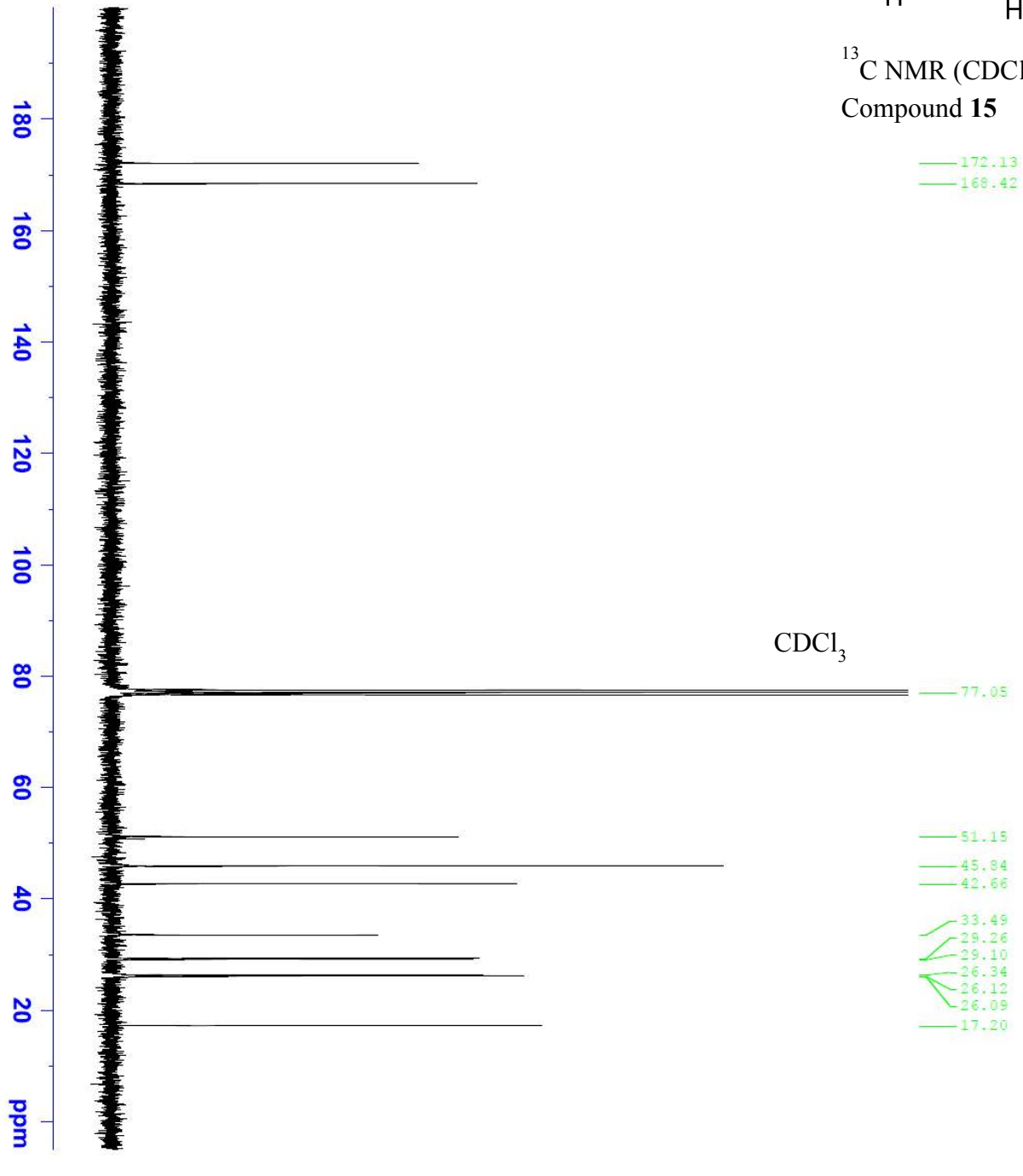
F2 - Processing parameters
SI 131072
SF 300.1700000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

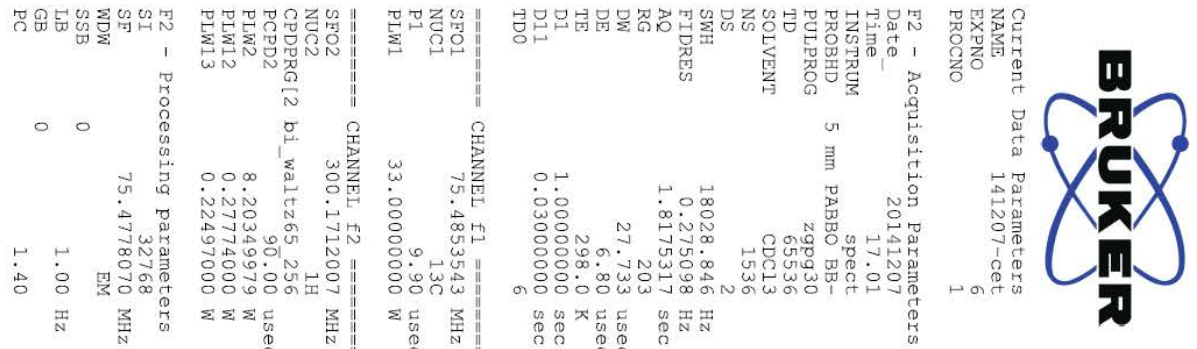
^{13}C NMR Compound 15

(S)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(1-cyclohexylethyl)acetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **15**





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Current Data Parameters
NAME                 141207-cet
EXPNO                         6
PROCNO                        1

F2 - Acquisition Parameters
Date_               20141207
Time_                   17.01
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1536
DS                       2
SWH                180028.846 Hz
FIDRES              0.275098 Hz
AQ                  1.8175317 sec
RG                        203
DW                   27.733 usec
DE                    6.80 usec
TE                   298.0 K
D1                  1.00000000 sec
D11                 0.03000000 sec
TD0                          6

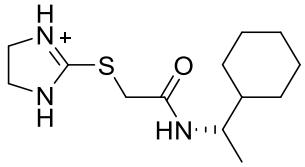
========== CHANNEL f1 ========
SFO1            75.4853543 MHz
NUC1                      13C
P1                   9.90 usec
PLW1           33.00000000 W

========== CHANNEL f2 ========
SFO2           300.1712007 MHz
NUC2                       1H
CPDPRG[2 bi_waltz65_256
PCPD2                90.00 usec
PLW2           8.20349979 W
PLW12          0.27774000 W
PLW13          0.22497000 W

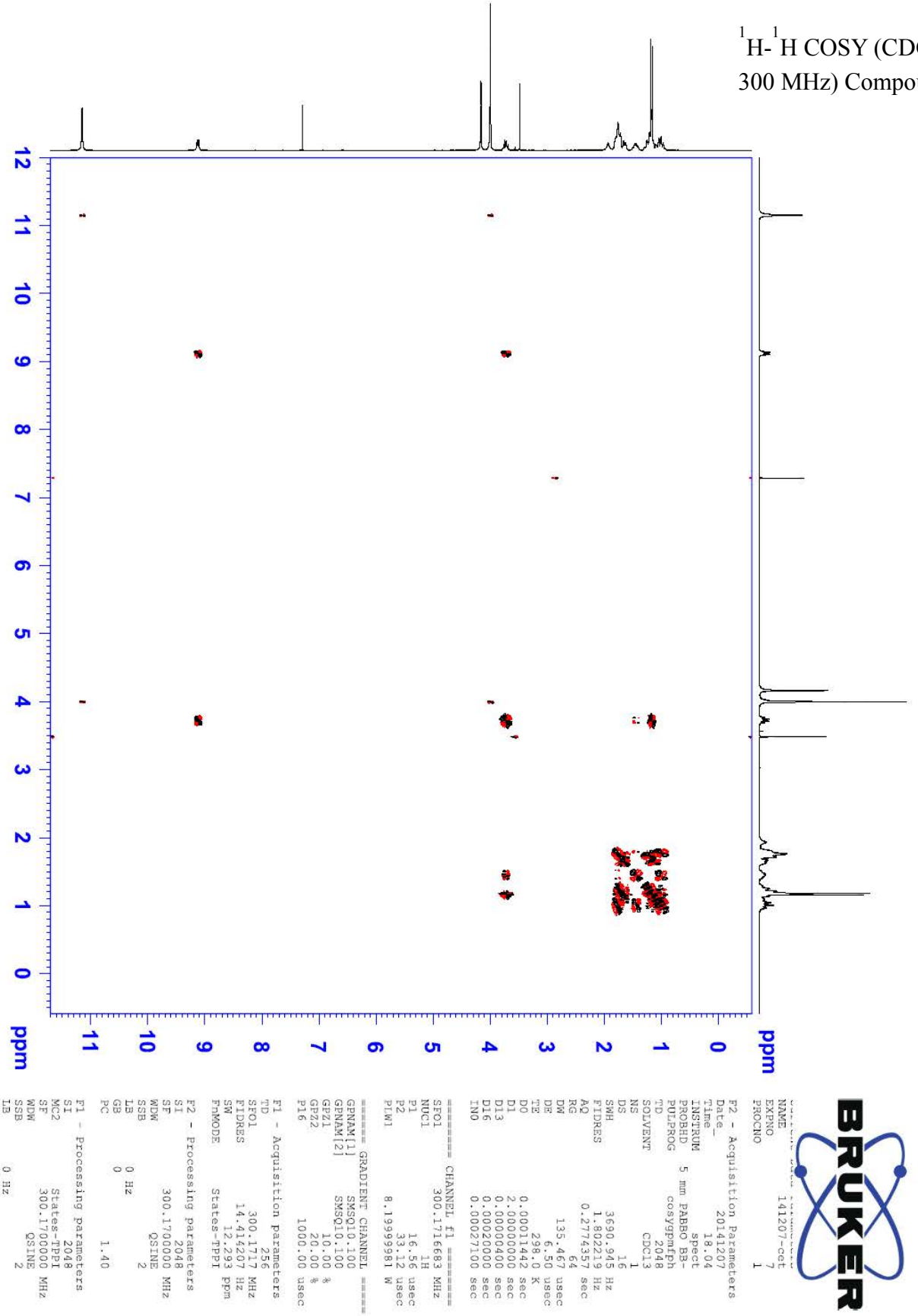
F2 - Processing parameters
SI                       32768
SF              75.4778070 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                    1.40
```

^{1}H-^{1}H COSY Compound 15

(S)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(1-cyclohexylethyl)acetamide



^{1}H-^{1}H COSY ($CDCl_3$, 300 MHz) Compound **15**

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F2 - Acquisition Parameters
Date_              20141207
Time_                18.04
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG          cosygpmfph
TD                     2048
SOLVENT               CDCl3
NS                        1
DS                       16
SWH              3690.945 Hz
FIDRES           1.802219 Hz
AQ               0.2774357 sec
RG                       64
DW                135.467 usec
DE                  6.50 usec
TE                298.0 K
D0               0.00011442 sec
D1               2.00000000 sec
D13              0.00000400 sec
D16              0.00020000 sec
IN0              0.00027100 sec

======== CHANNEL f1 ========
SFO1            300.1716683 MHz
NUC1                        1H
P1                       16.56 usec
P2                       33.12 usec
PLW1             8.19999981 W

======= GRADIENT CHANNEL =====
GPNAM[1]         SMSQ10.100
GPNAM[2]         SMSQ10.100
GPZ1                  10.00 %
GPZ2                  20.00 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                       256
SFO1            300.1717 MHz
FIDRES           14.414207 Hz
SW               12.293 ppm
FnMODE           States-TPPI

F2 - Processing parameters
SI                      2048
SF              300.1700000 MHz
WDW                    QSINE
SSB                         2
LB                      0 Hz
GB                         0
PC                      1.40

F1 - Processing parameters
SI                      2048
MC2              States-TPPI
SF              300.1700000 MHz
WDW                    QSINE
SSB                         2
LB                      0 Hz
GB                         0
```
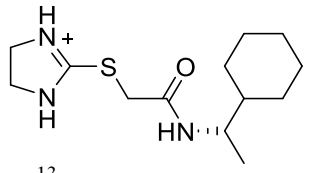
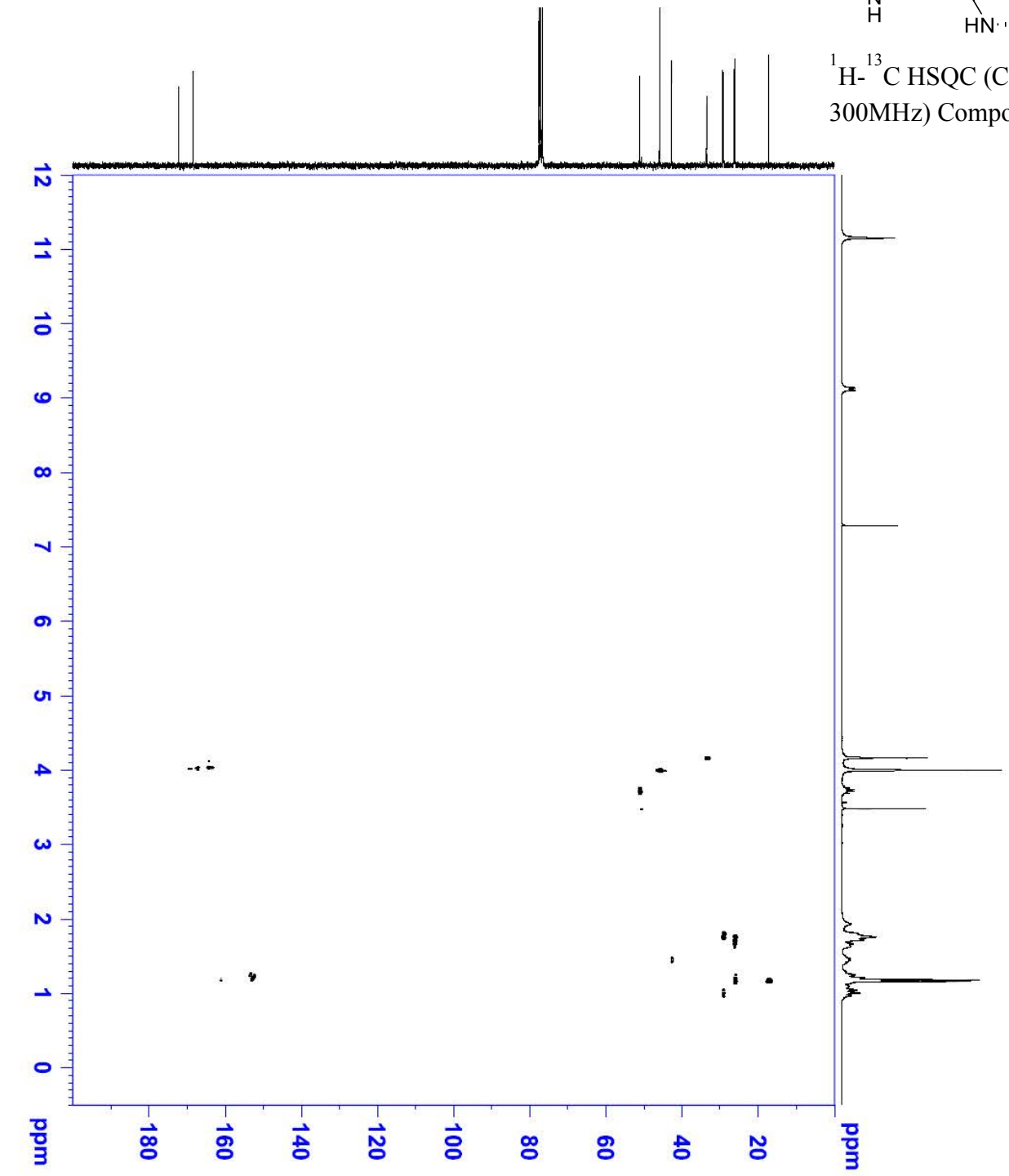
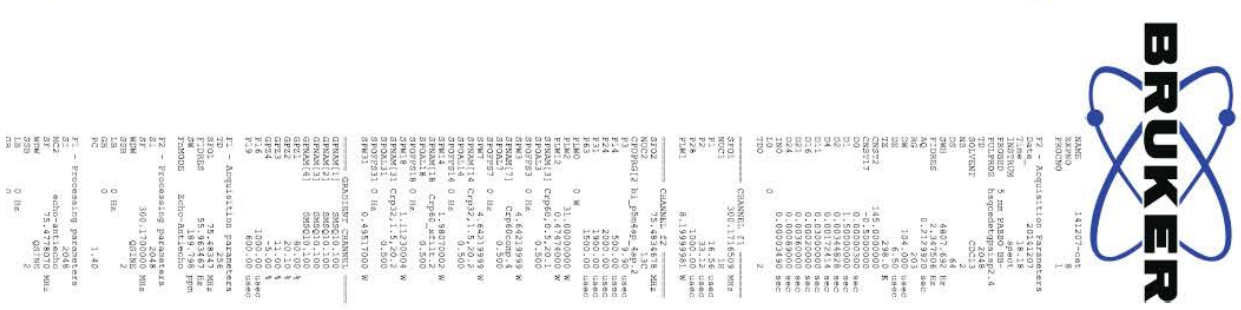

^{1}H-^{13}C HSQC Compound 15

(S)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(1-cyclohexylethyl)acetamide

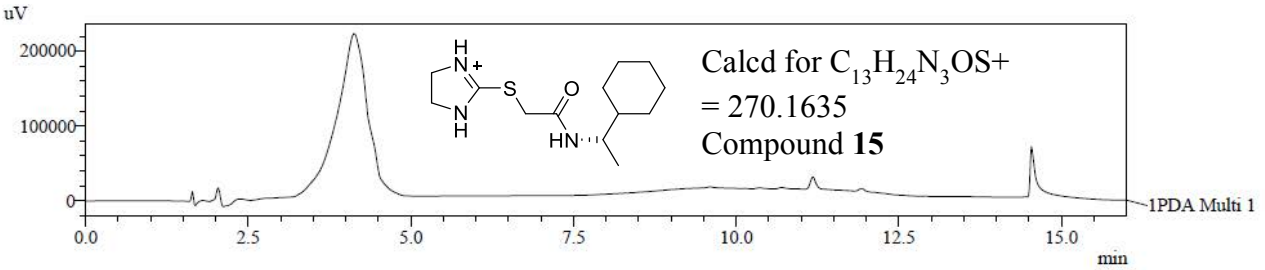


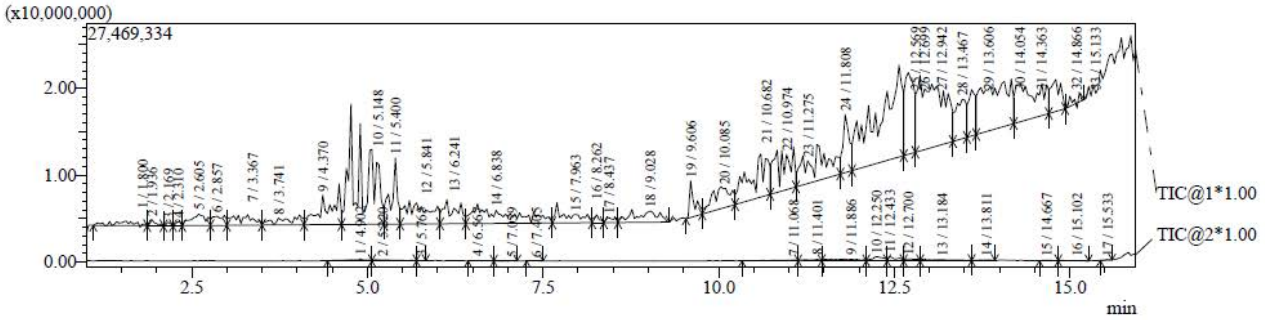
^{1}H-^{13}C HSQC ($CDCl_3$, 300MHz) Compound **15**

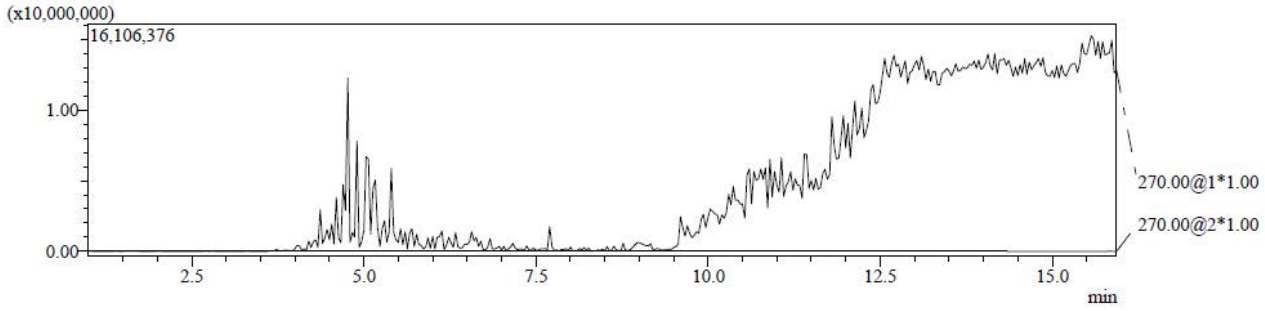




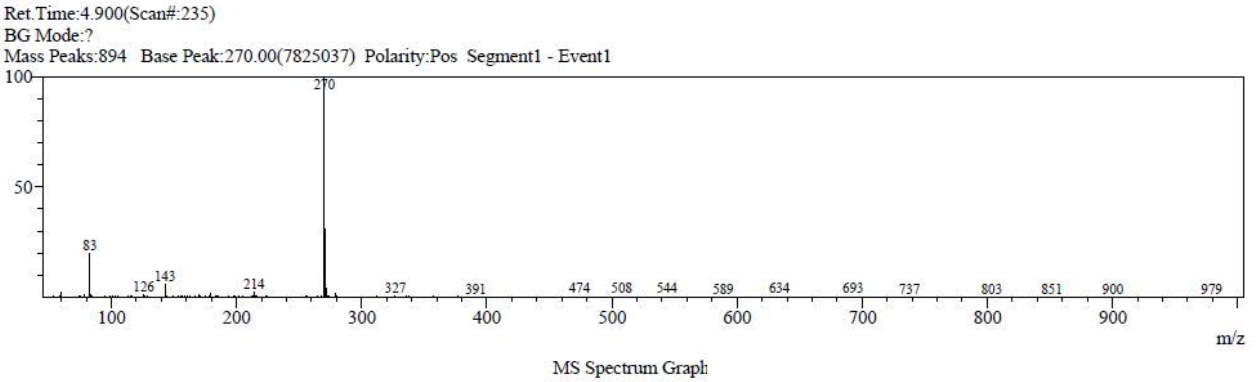
LCMS Compound 15



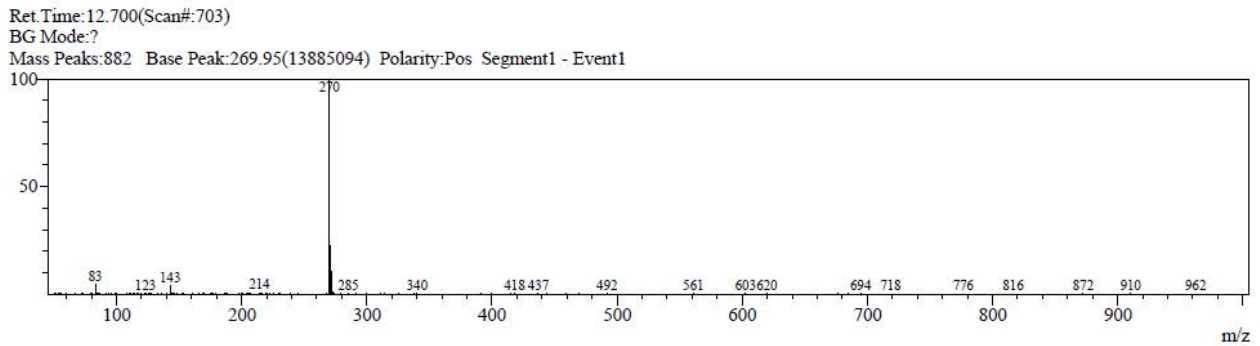




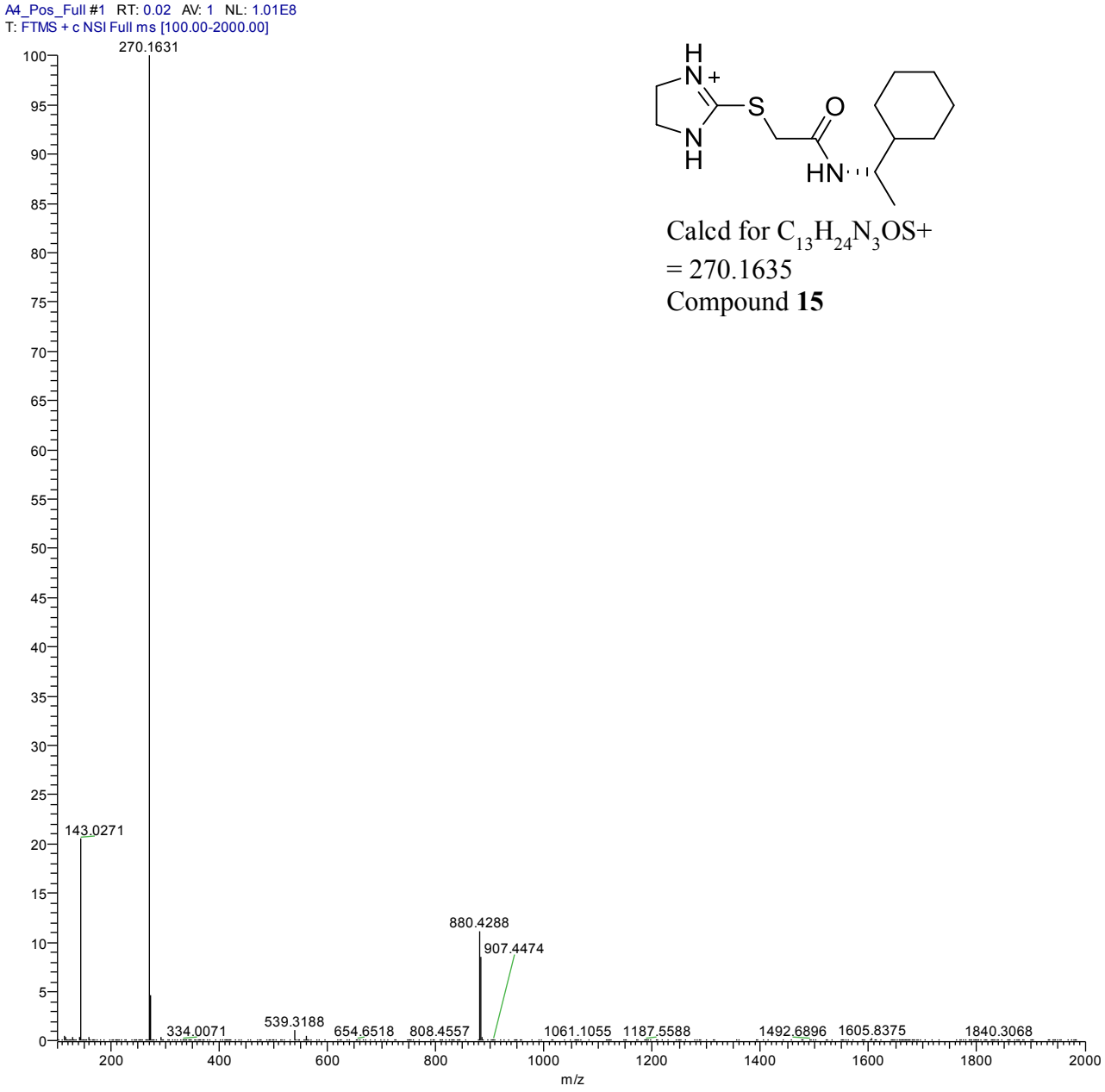
MS Spectrum Graph



MS Spectrum Graph



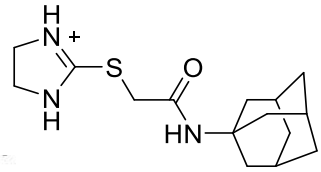
HRMS(ESI) Compound 15



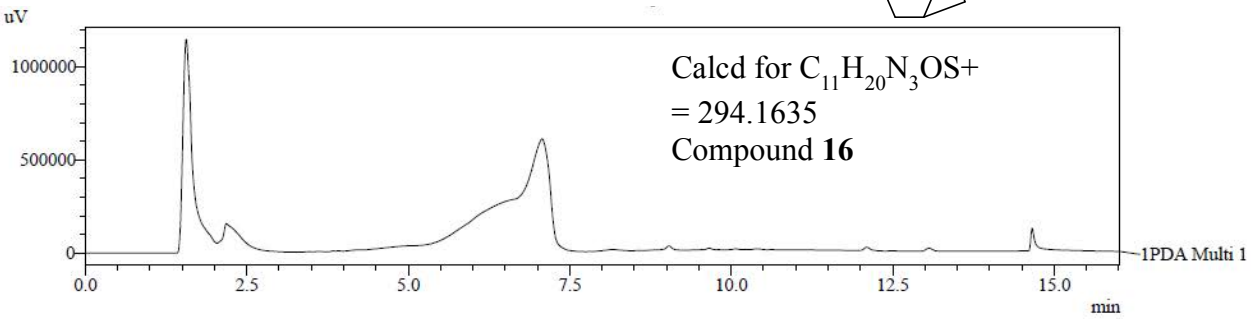
Calcd for $C_{13}H_{24}N_3OS^+$ = 270.1635

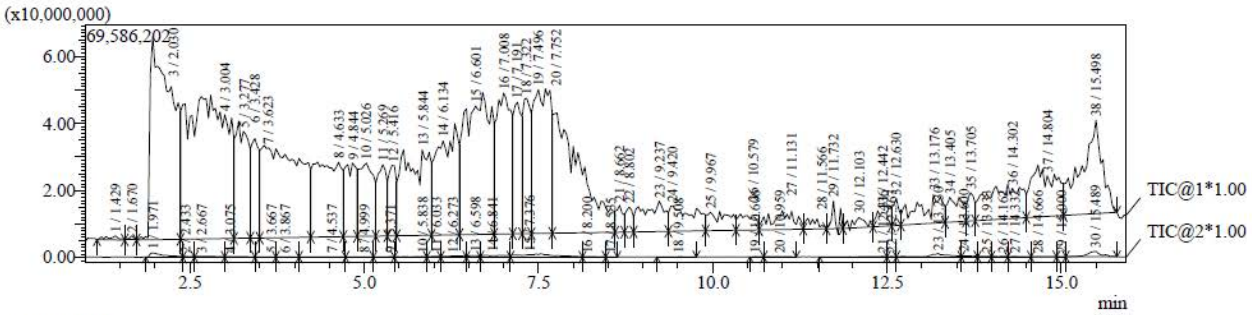
Compound **15**

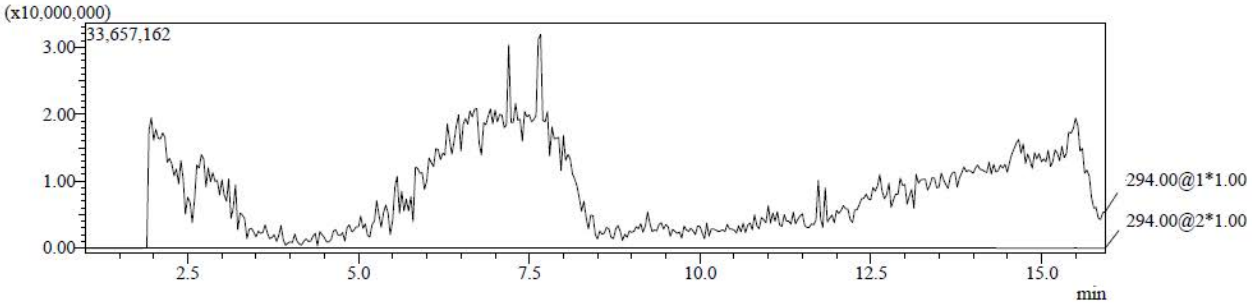
Spectra for Compound 16



LCMS Compound 16

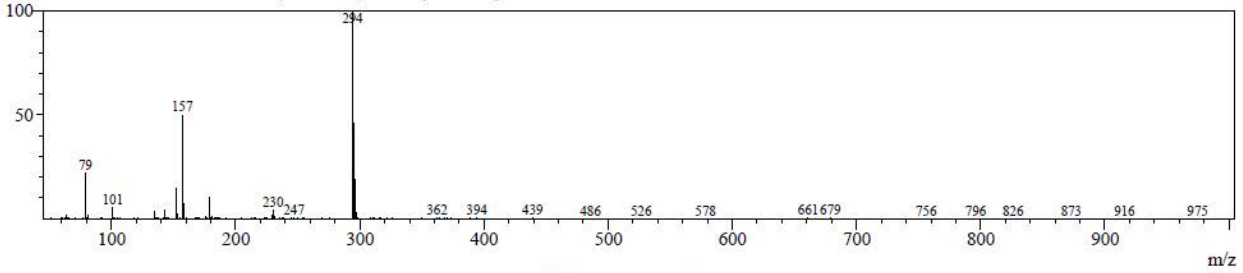






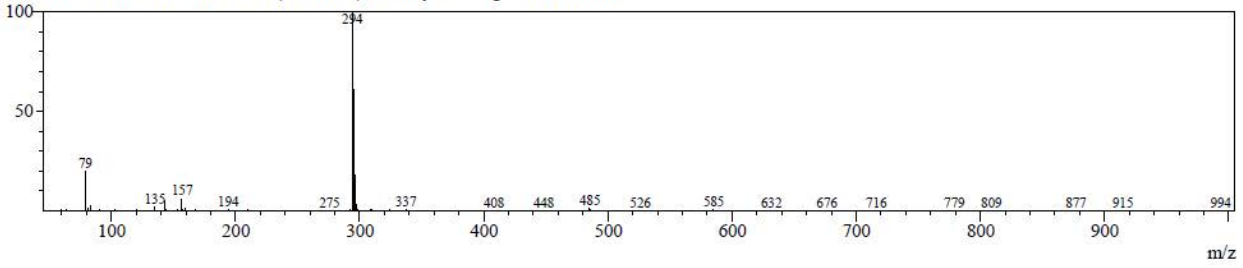
MS Spectrum Graph

Ret.Time:1.967(Scan#:59)
BG Mode:?
Mass Peaks:926 Base Peak:294.00(19477116) Polarity:Pos Segment1 - Event1



MS Spectrum Graph

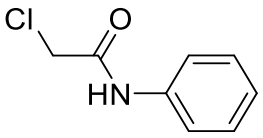
Ret.Time:7.167(Scan#:371)
BG Mode:None
Mass Peaks:930 Base Peak:293.95(18349773) Polarity:Pos Segment1 - Event1



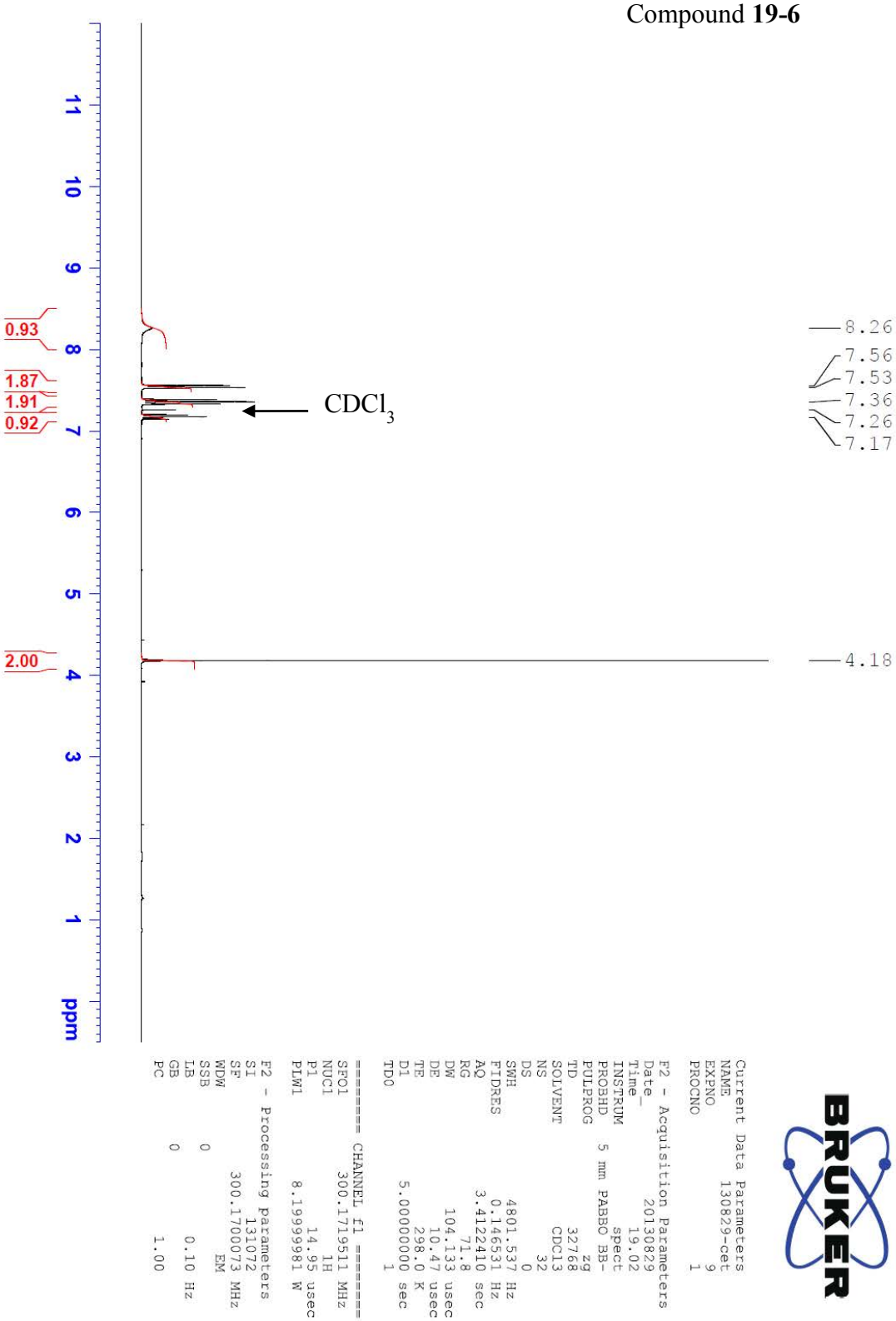
Spectra for Compound 19

^{1}H NMR Compound 19-6

2-chloro-N-phenylacetamide

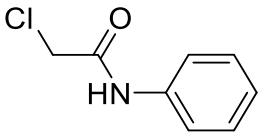


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **19-6**

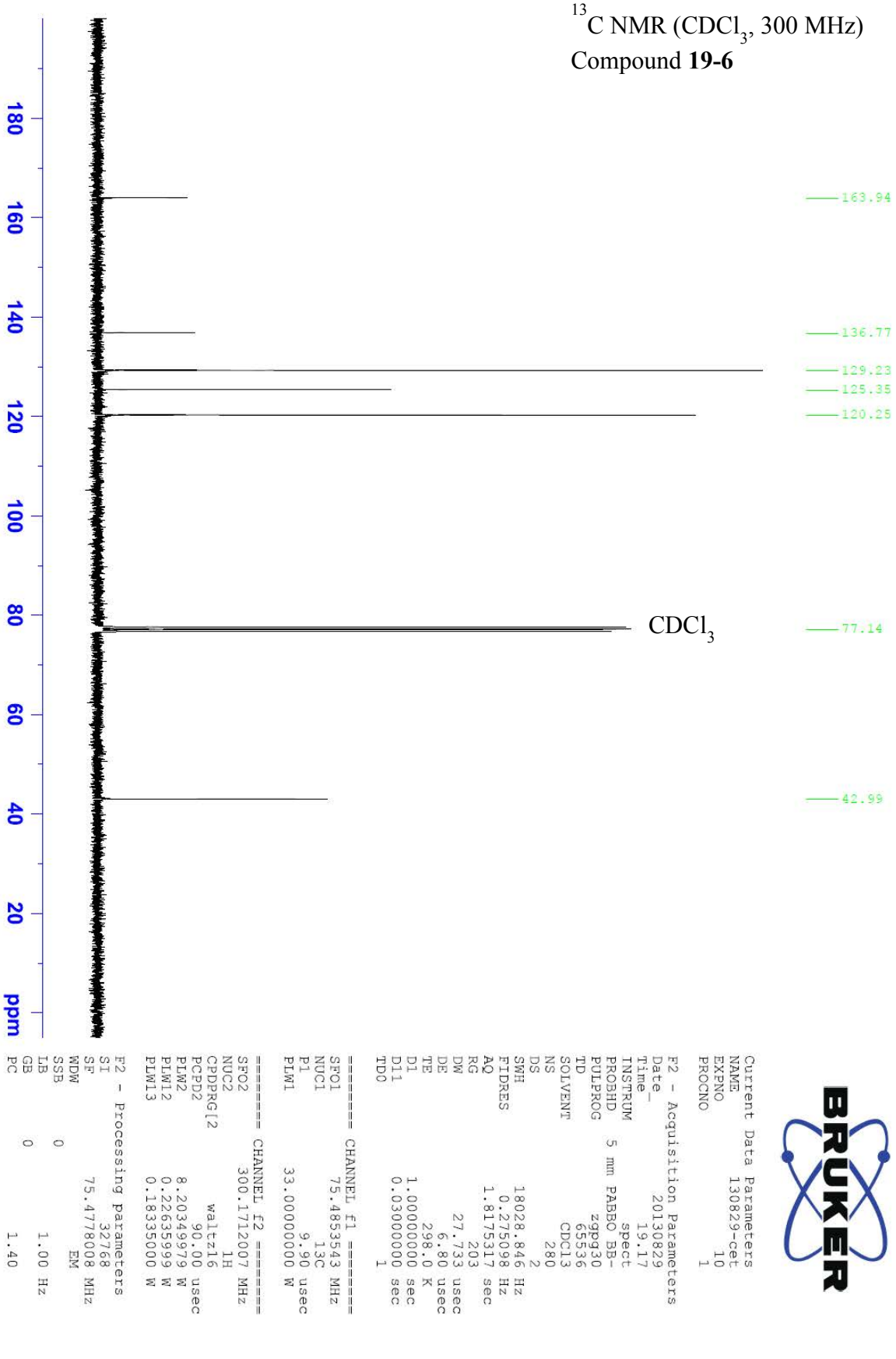


^{13}C NMR Compound 19-6

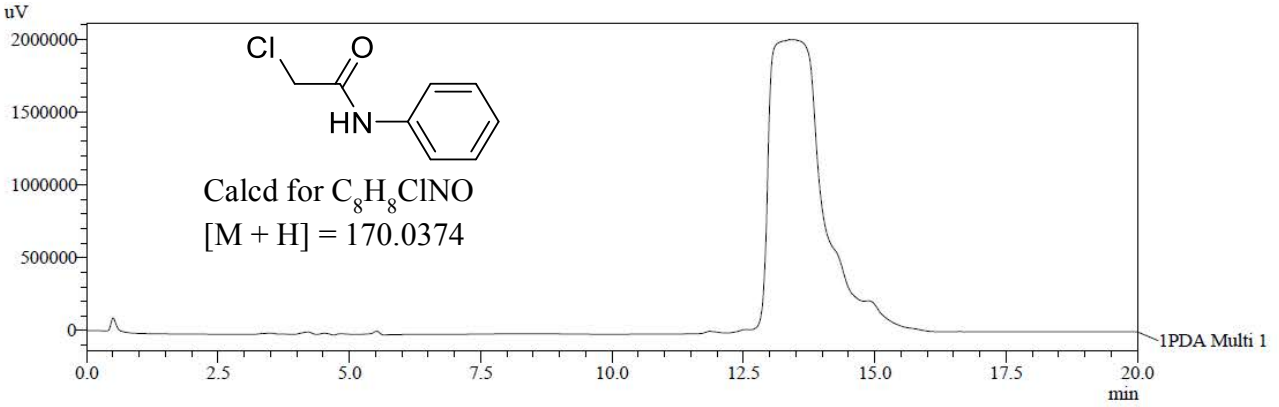
2-chloro-N-phenylacetamide

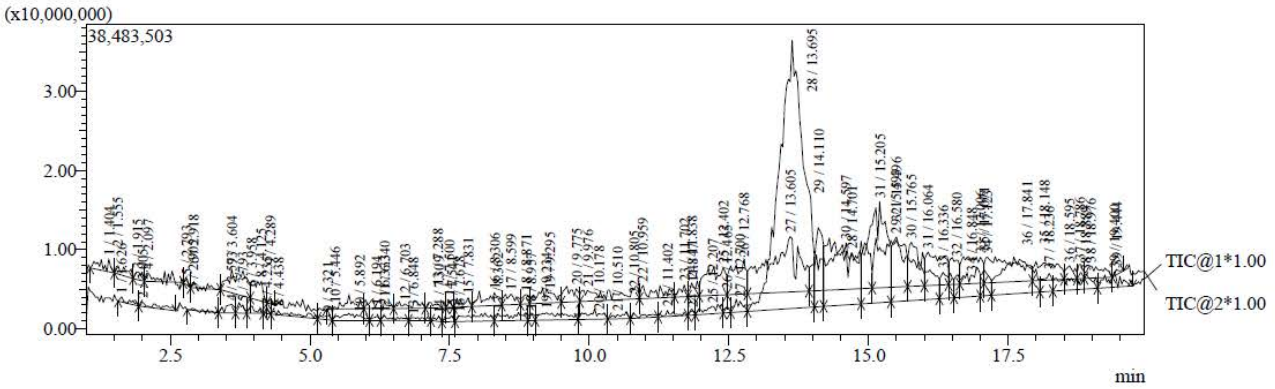


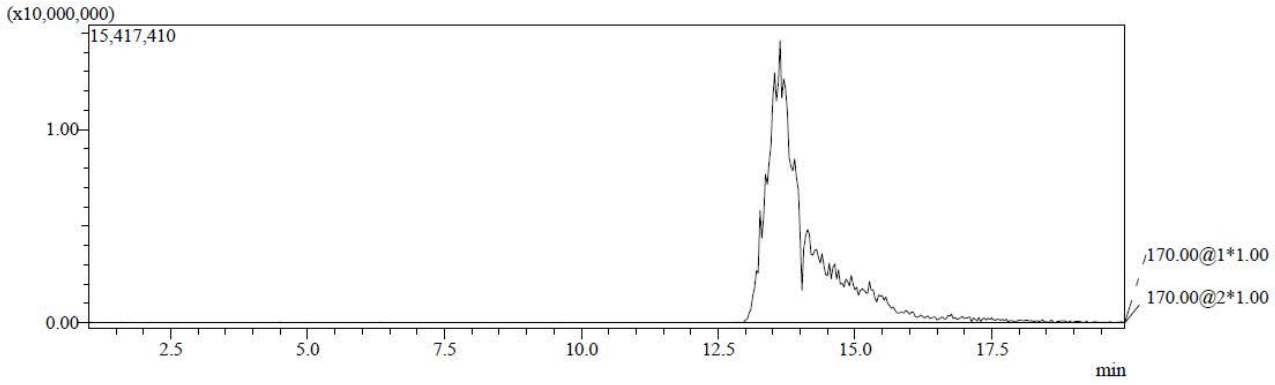
^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **19-6**



LCMS Compound 19-6

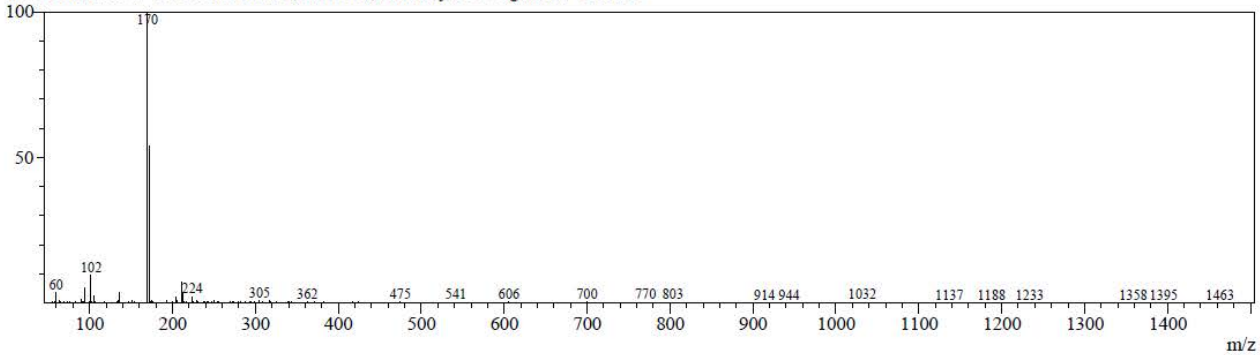




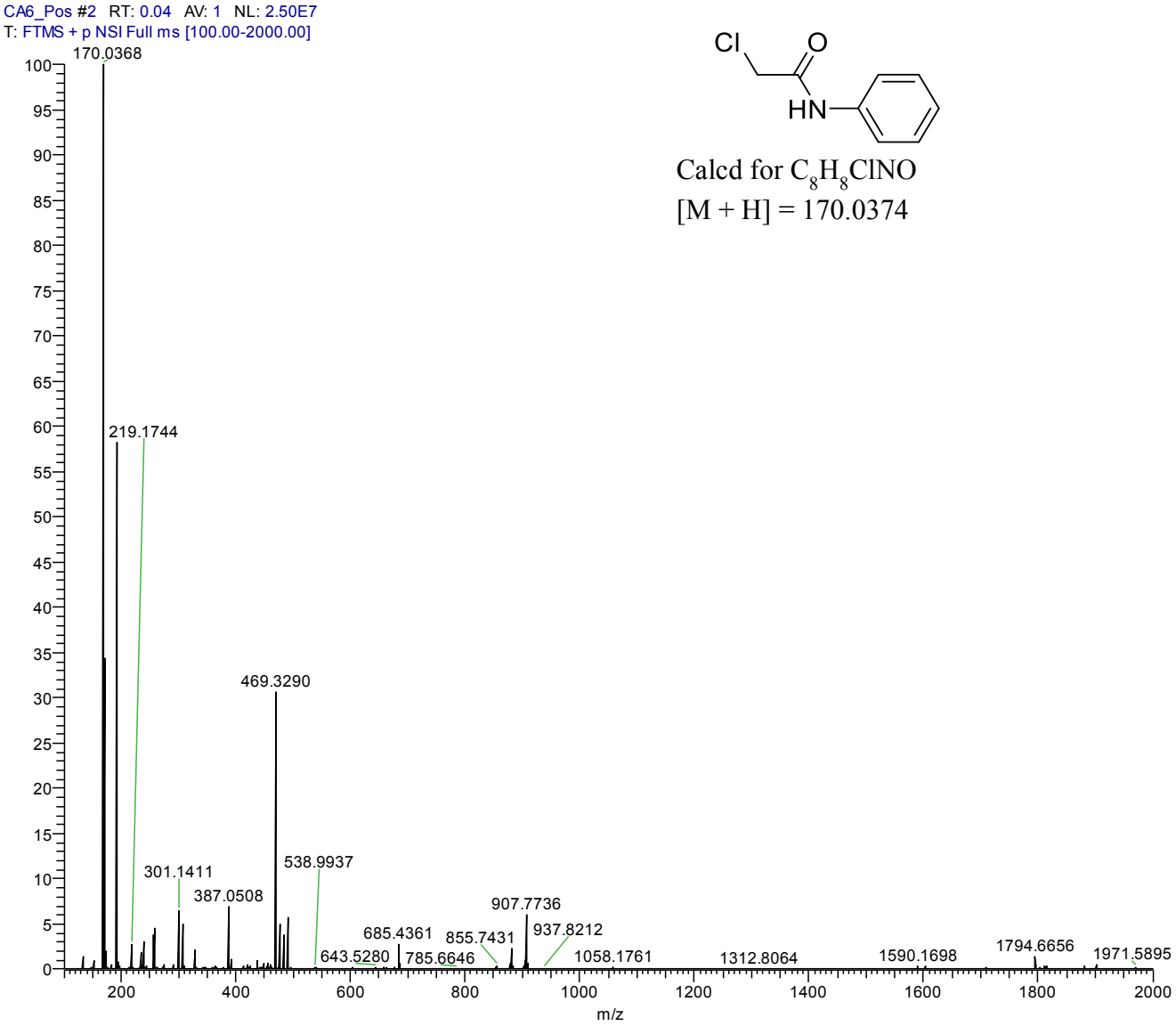


MS Spectrum Graph

Ret.Time:13.633(Scan#:759)
BG Mode:None
Mass Peaks:1234 Base Peak:169.75(14605968) Polarity:Pos Segment1 - Event1



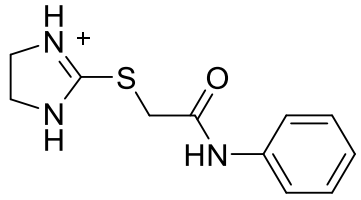
HRMS(ESI) Compound 19-6



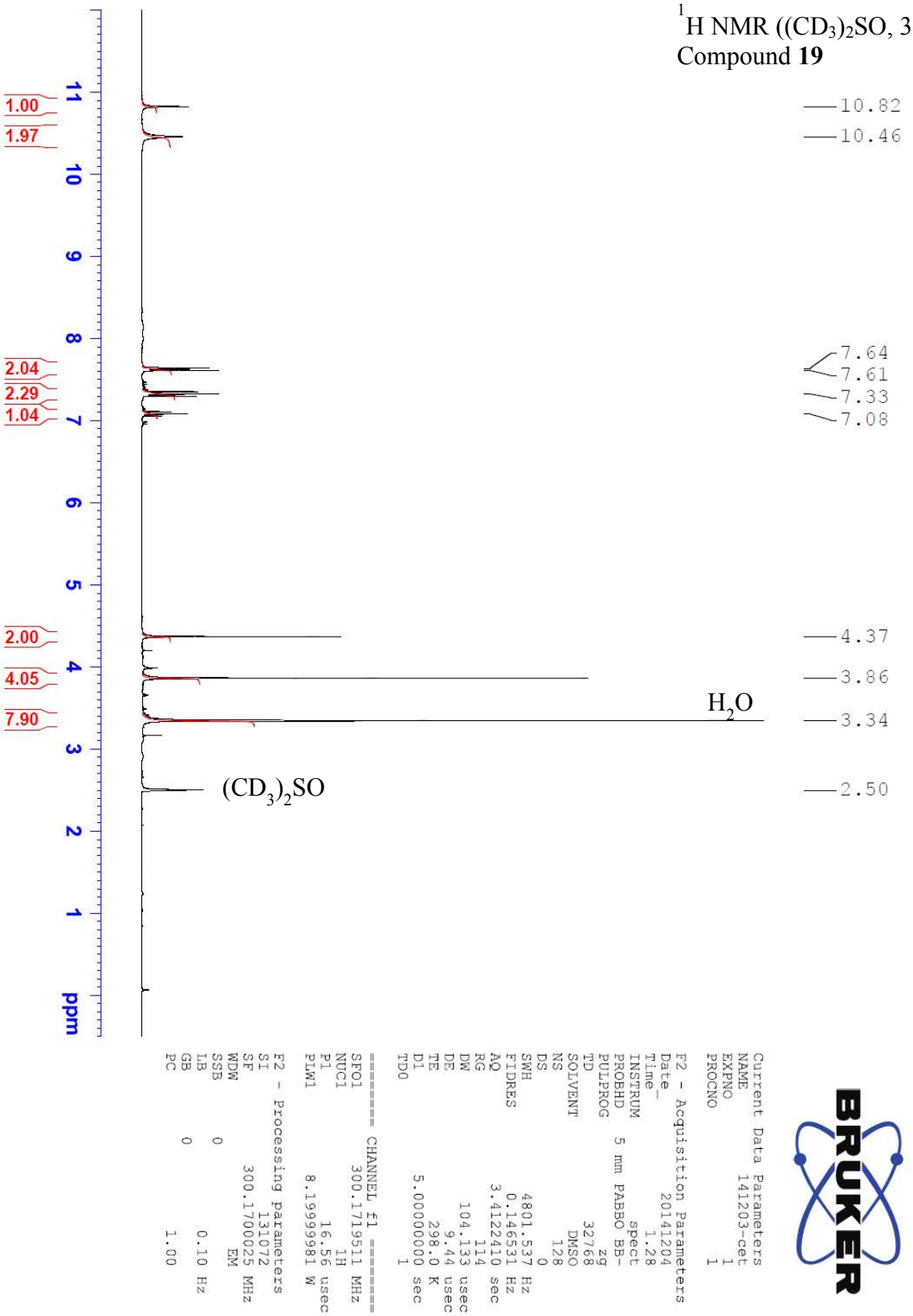
Calcd for C_8H_8ClNO
[M + H] = 170.0374

^{1}H NMR Compound 19

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenylacetamide

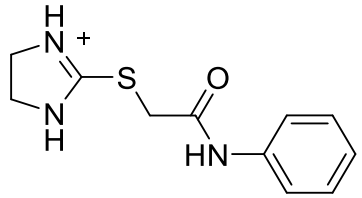


^{1}H NMR ($(CD_3)_2SO$, 300 MHz)
Compound **19**

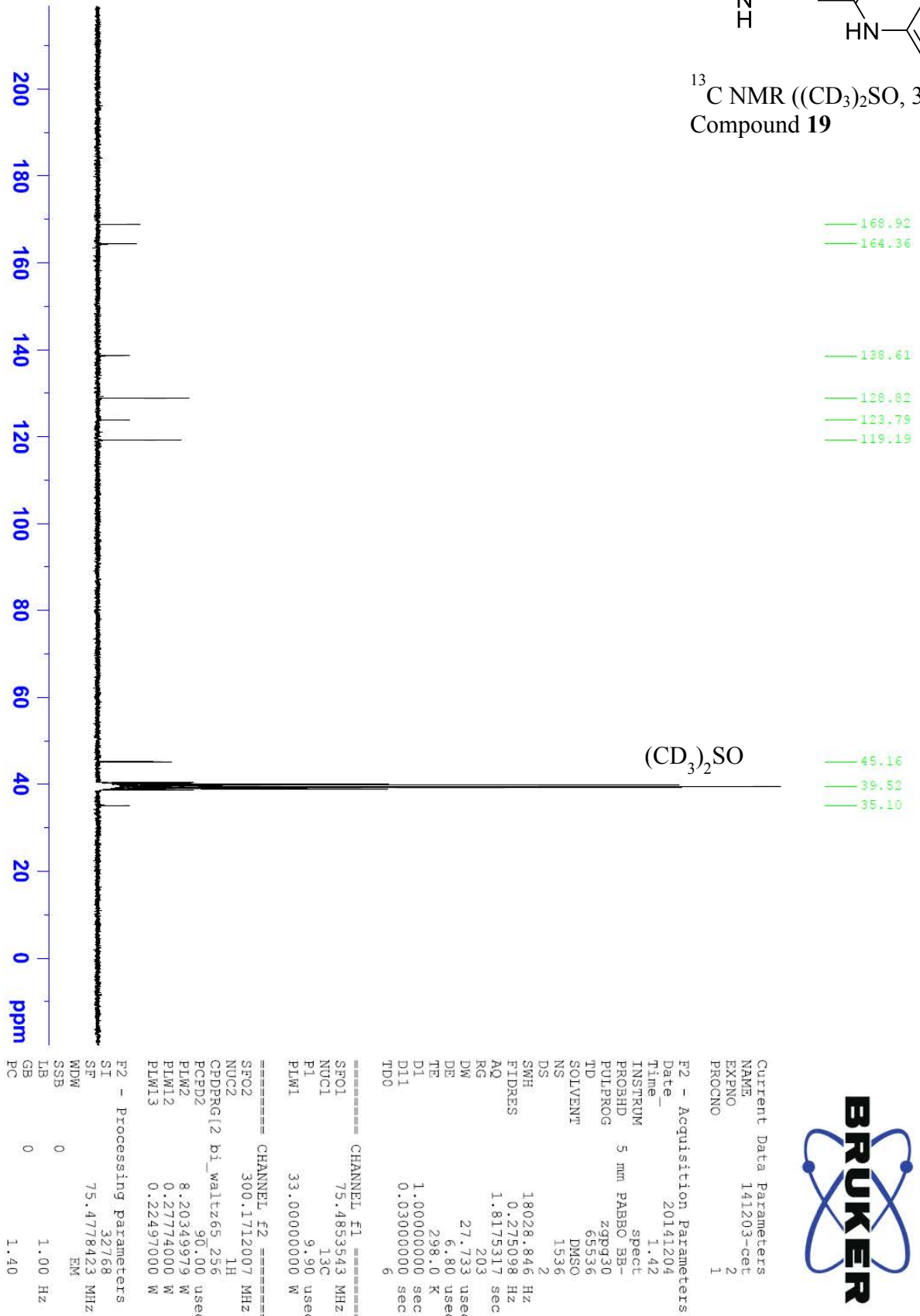


^{13}C NMR Compound 19

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenylacetamide



^{13}C NMR ($(CD_3)_2SO$, 300 MHz)
Compound **19**

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Current Data Parameters
NAME                 141203-cet
EXPNO                         2
PROCNO                        1

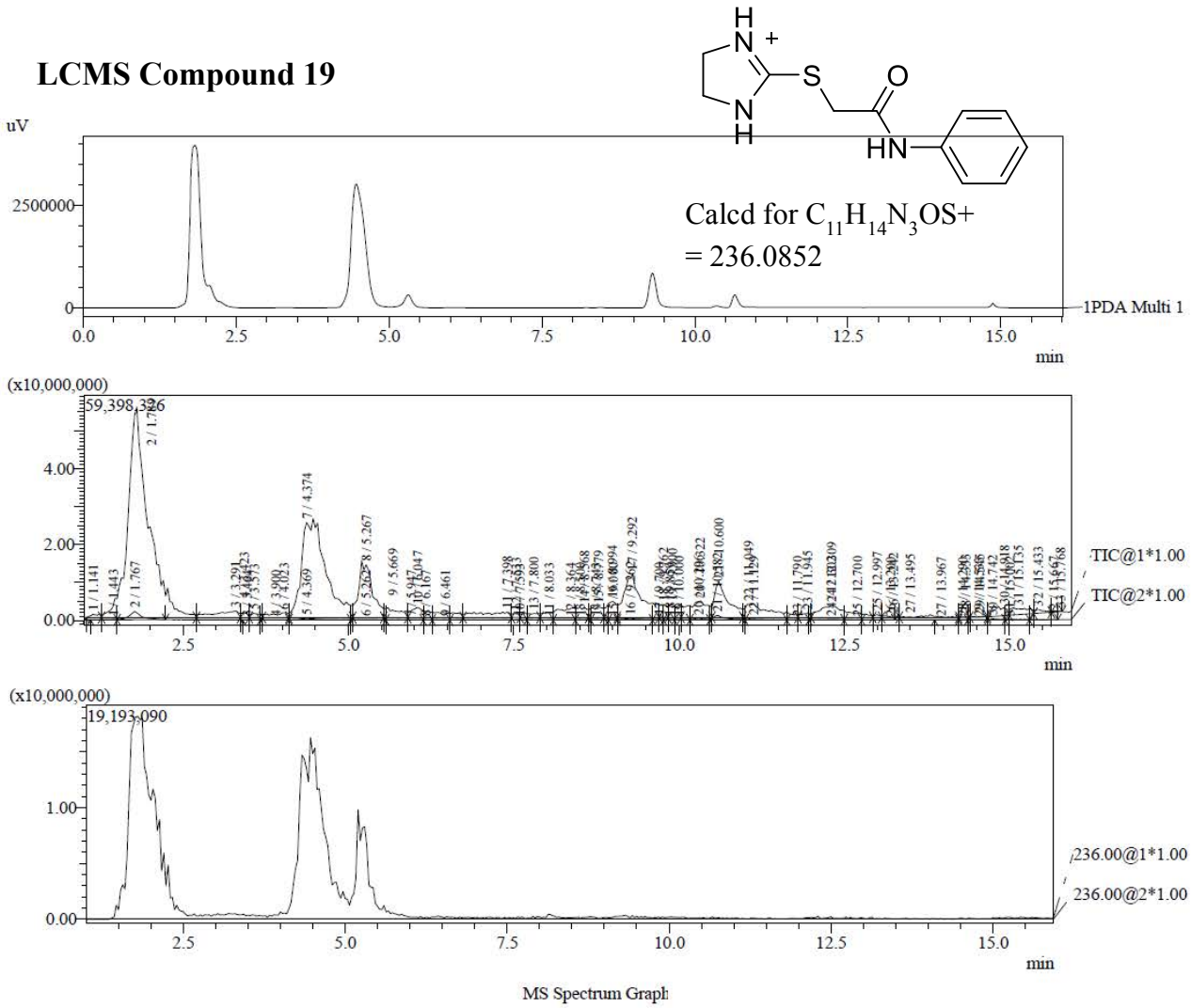
F2 - Acquisition Parameters
Date_               20141204
Time_                   1.42
INSTRUM                 spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                       65536
SOLVENT                  DMSO
NS                        1536
DS                           2
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                          203
DW                    27.733 usec
DE                     6.80 usec
TE                     298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                           6

========== CHANNEL f1 ========
SFO1                75.4853543 MHz
NUC1                       13C
P1                        9.90 usec
PLW1           33.00000000 W

========== CHANNEL f2 ========
SFO2                300.1712007 MHz
NUC2                       1H
CPDPRG[2 bi_waltz65_256
PCPD2                   90.00 usec
PLW2           8.20349979 W
PLW12          0.27774000 W
PLW13          0.22497000 W

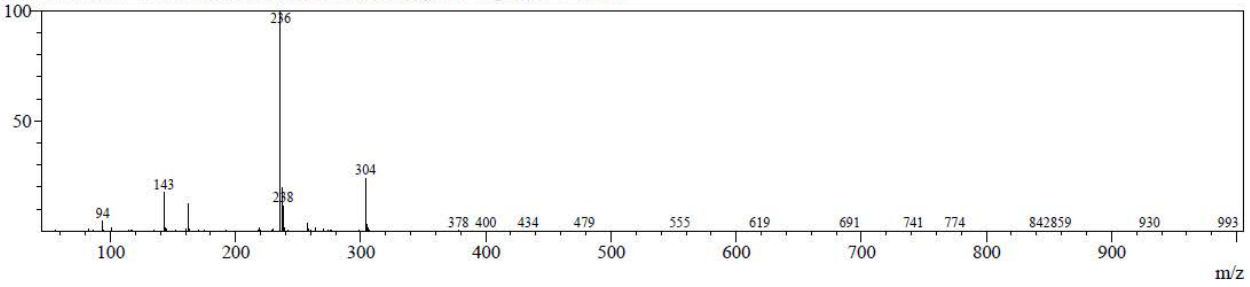
F2 - Processing parameters
SI                        32768
SF                75.4778423 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                    1.40
```

LCMS Compound 19



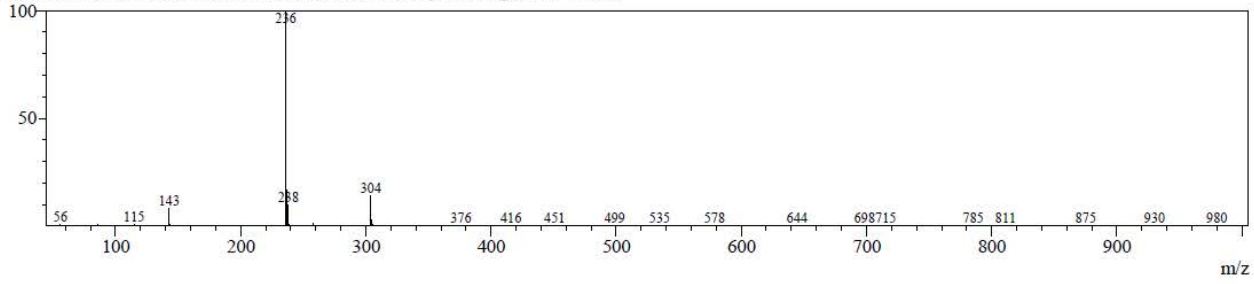
MS Spectrum Graph

Ret.Time:1.867(Scan#:53)
BG Mode:None
Mass Peaks:916 Base Peak:235.95(18058738) Polarity:Pos Segment1 - Event1

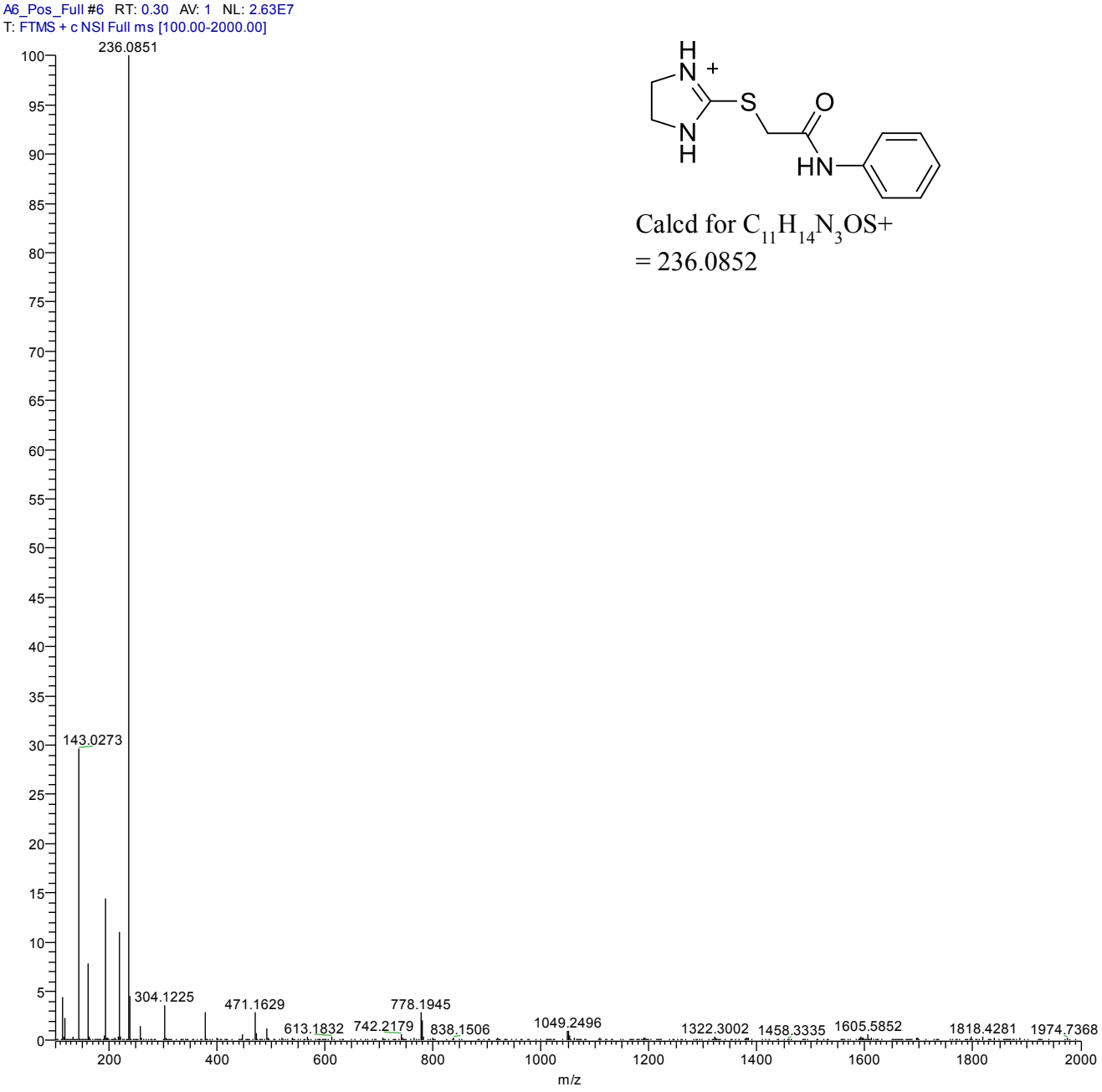


MS Spectrum Graph

Ret.Time:4.500(Scan#:211)
BG Mode:?
Mass Peaks:854 Base Peak:235.95(14842926) Polarity:Pos Segment1 - Event1



HRMS(ESI) Compound 19

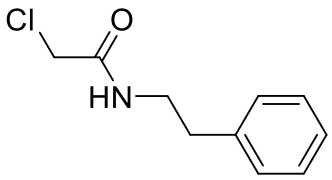


Calcd for $C_{11}H_{14}N_3OS^+$
= 236.0852

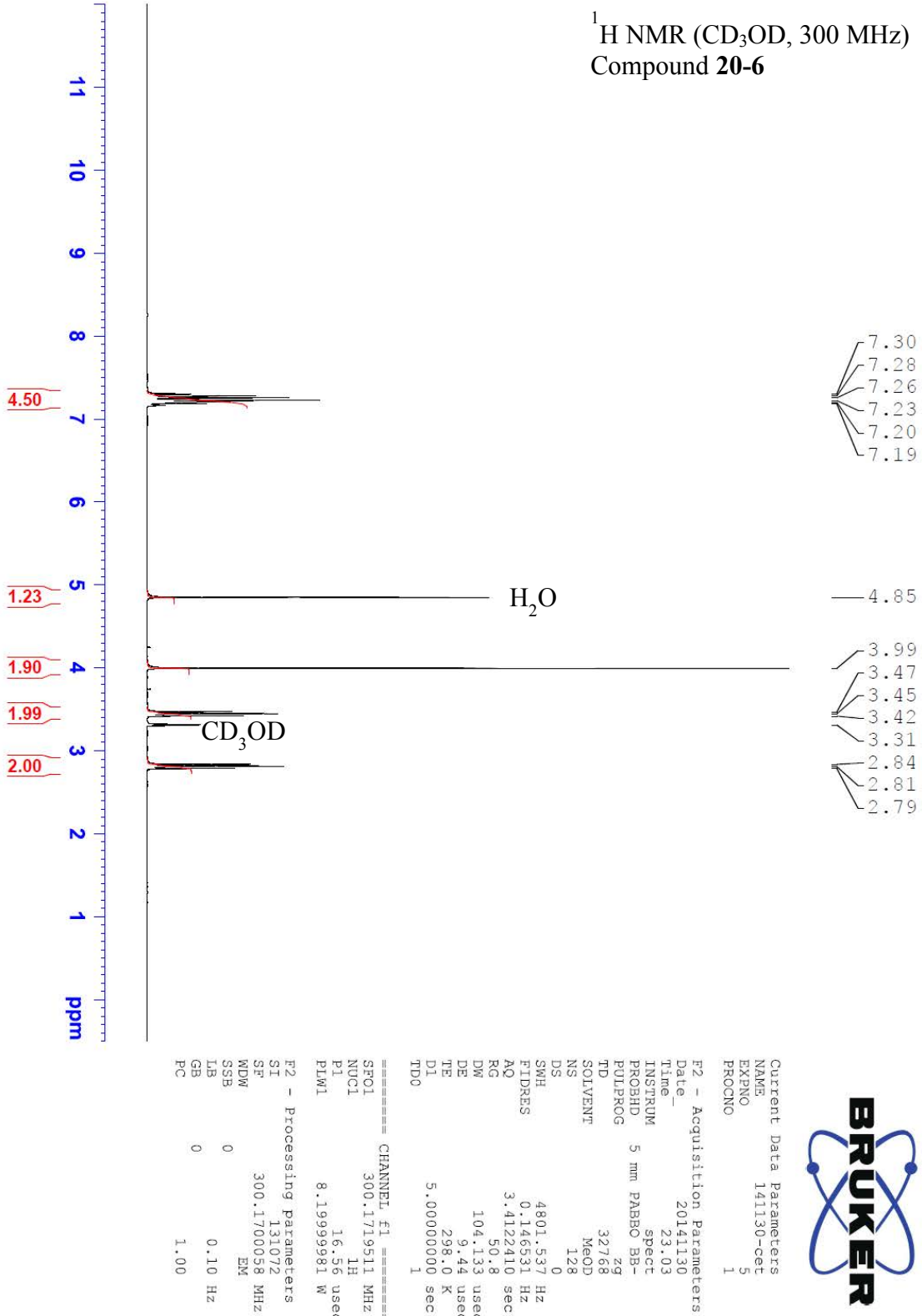
Spectra for Compound 20

^{1}H NMR Compound 20-6

2-chloro-N-phenethylacetamide

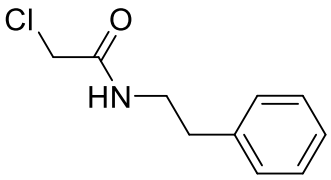


^{1}H NMR (CD_3OD, 300 MHz)
Compound **20-6**

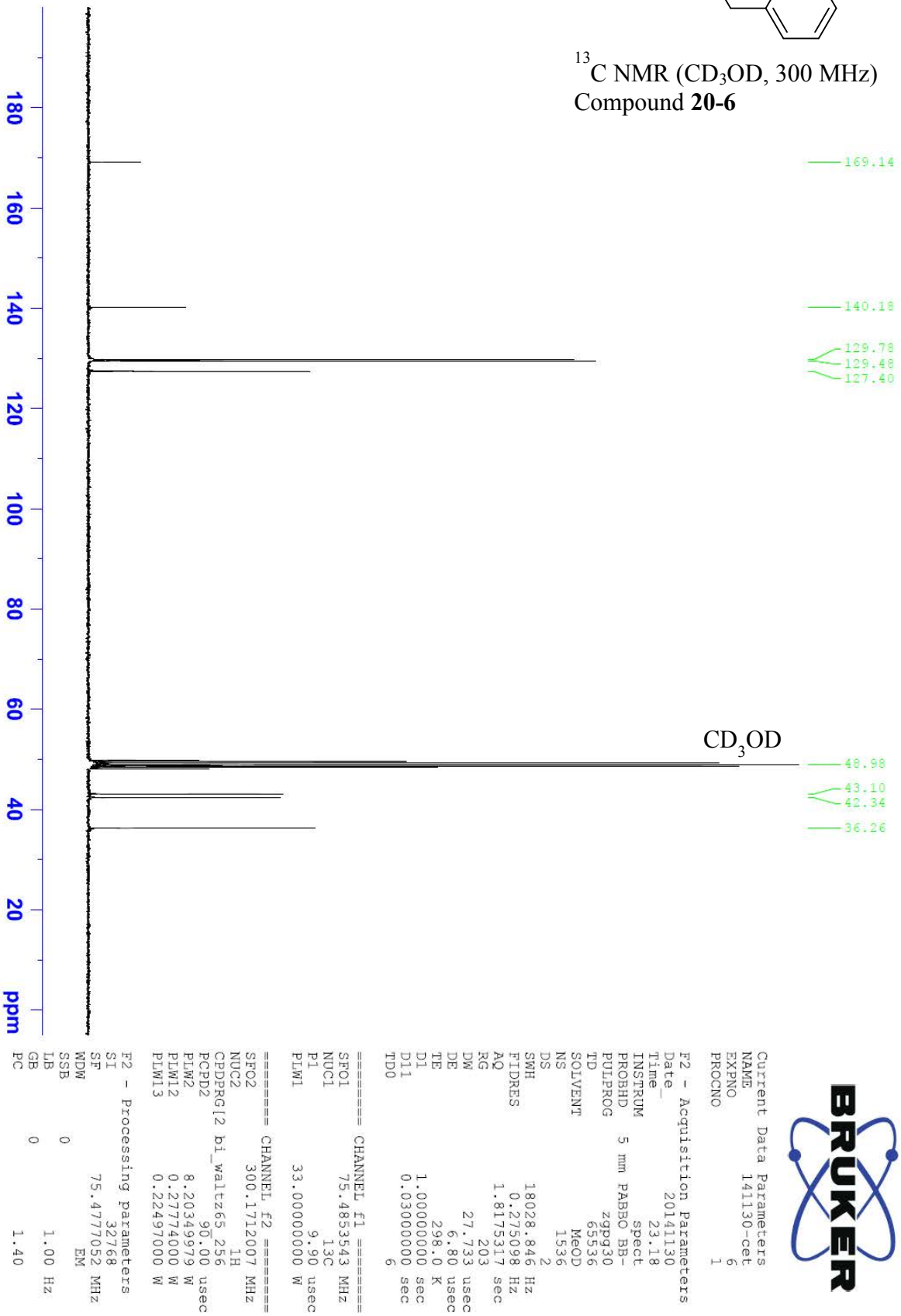


^{13}C NMR Compound 20-6

2-chloro-N-phenethylacetamide



^{13}C NMR (CD_3OD, 300 MHz)
Compound **20-6**

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Current Data Parameters
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EXPNO                          6
PROCNO                         1

F2 - Acquisition Parameters
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Time                       23.18
INSTRUM                     spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                   MeOD
NS                        1536
DS                           2
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                         203
DW                      27.733 usec
DE                       6.80 usec
TE                      298.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                            6

======== CHANNEL f1 ========
SFO1                 75.4853543 MHz
NUC1                        13C
P1                         9.90 usec
PLW1                 33.00000000 W

========= CHANNEL f2 ========
SFO2                 300.1712007 MHz
NUC2                        1H
CPDPRG[2 bi_waltz65_256
PCPD2                   90.00 usec
PLW2                 8.20349979 W
PLW12                0.27774000 W
PLW13                0.22497000 W

F2 - Processing parameters
SI                         32768
SF                  75.4777052 MHz
WDW                            EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```
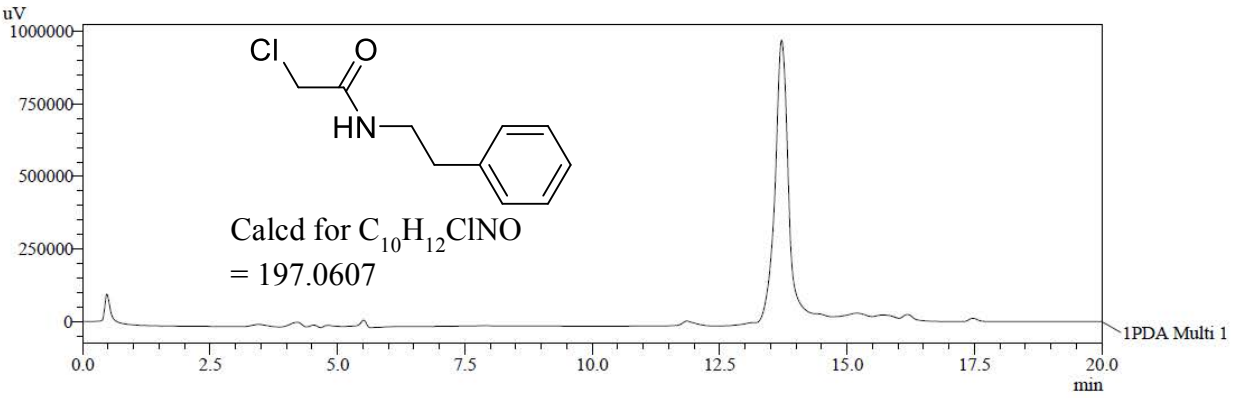
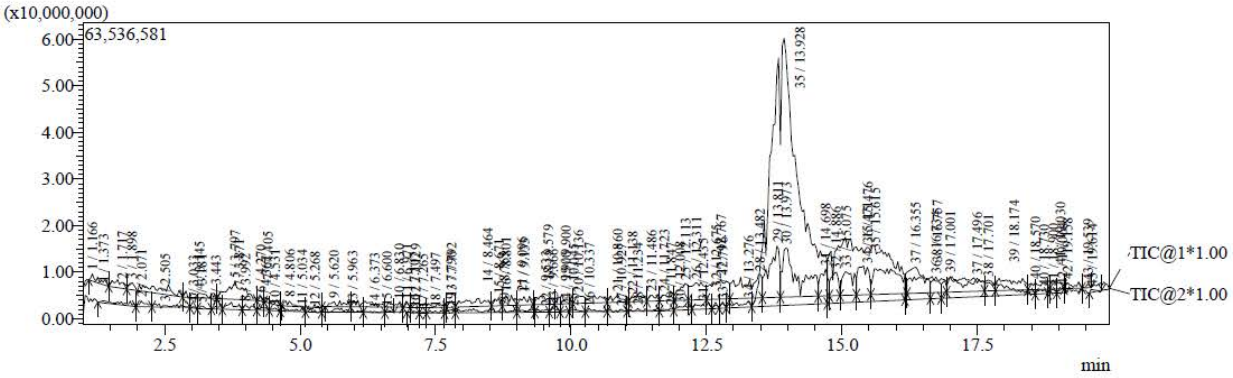
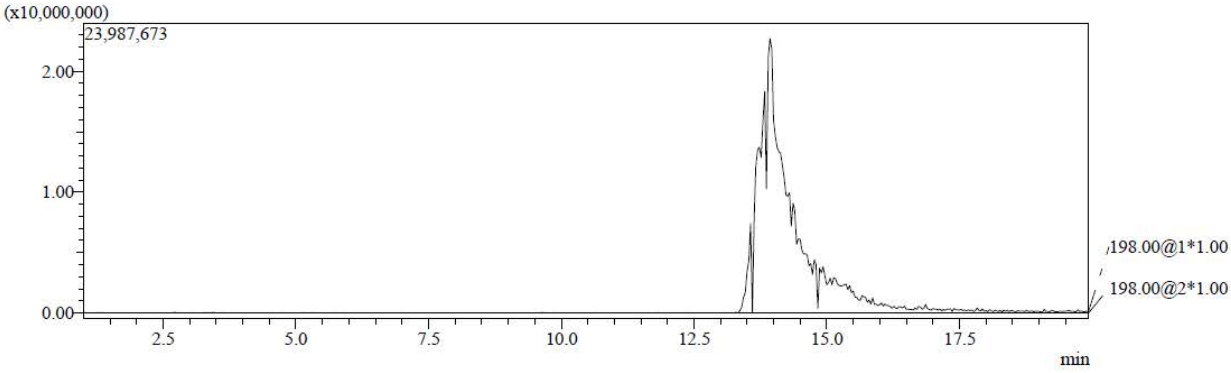

LCMS Compound 20-6

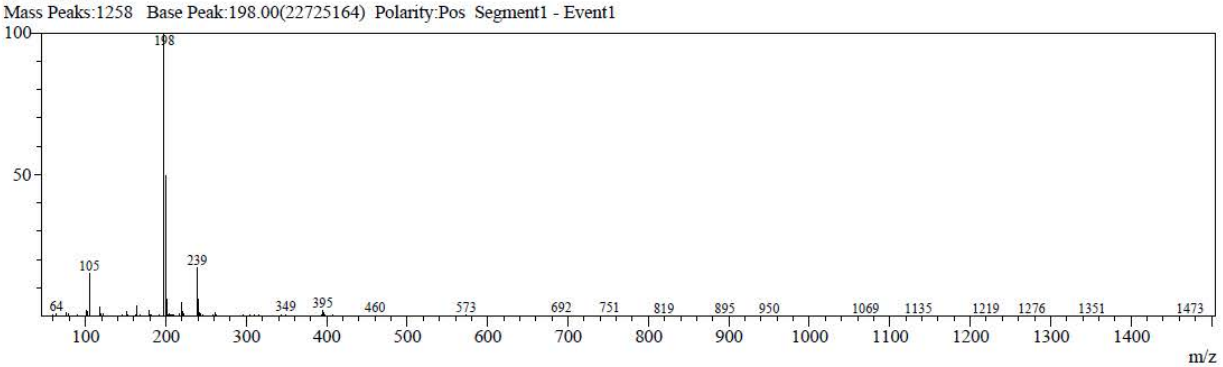




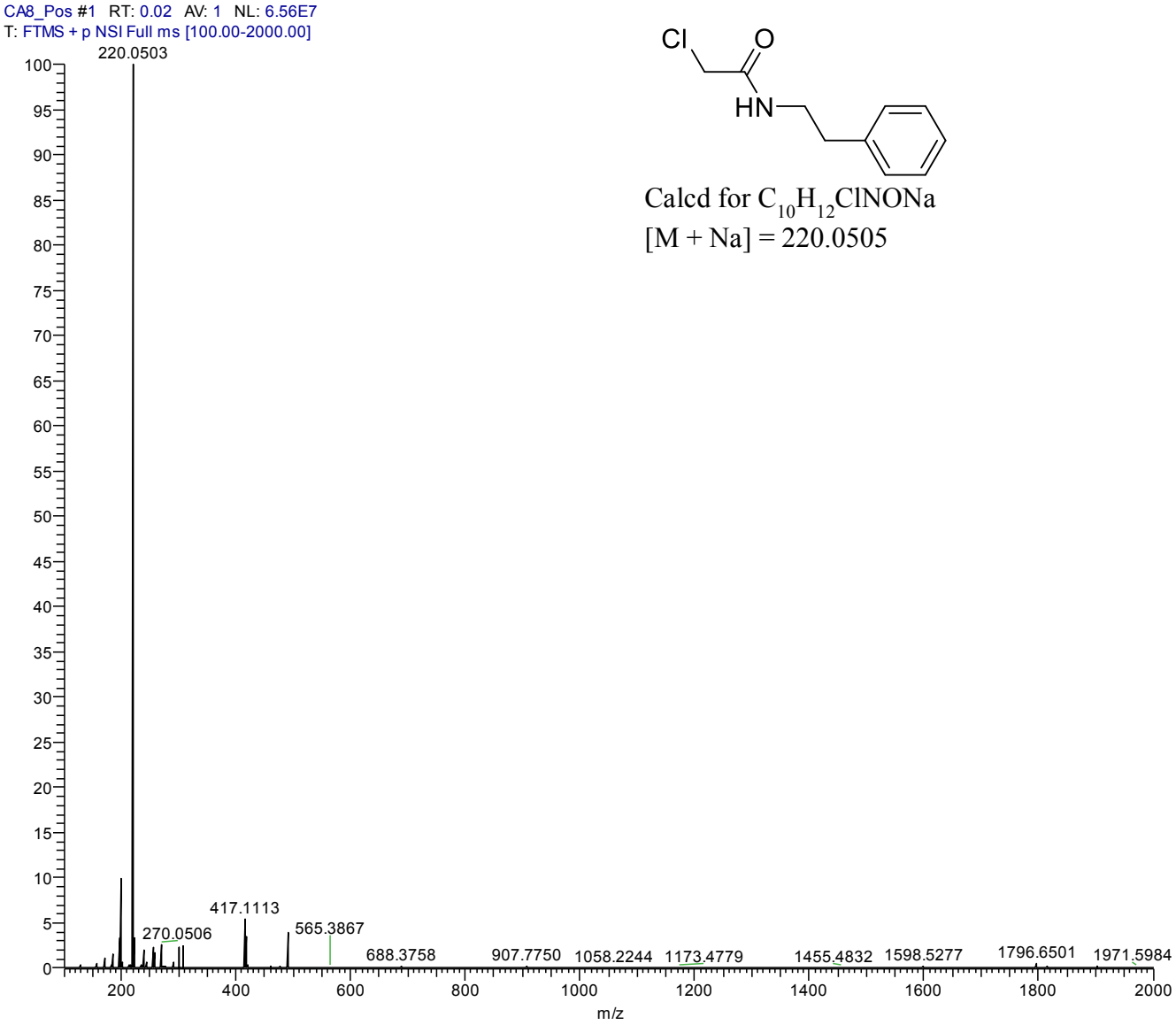


MS Spectrum Graph

Ret.Time:13.933(Scan#:777)
BG Mode:None
Mass Peaks:1258 Base Peak:198.00(22725164) Polarity:Pos Segment1 - Event1



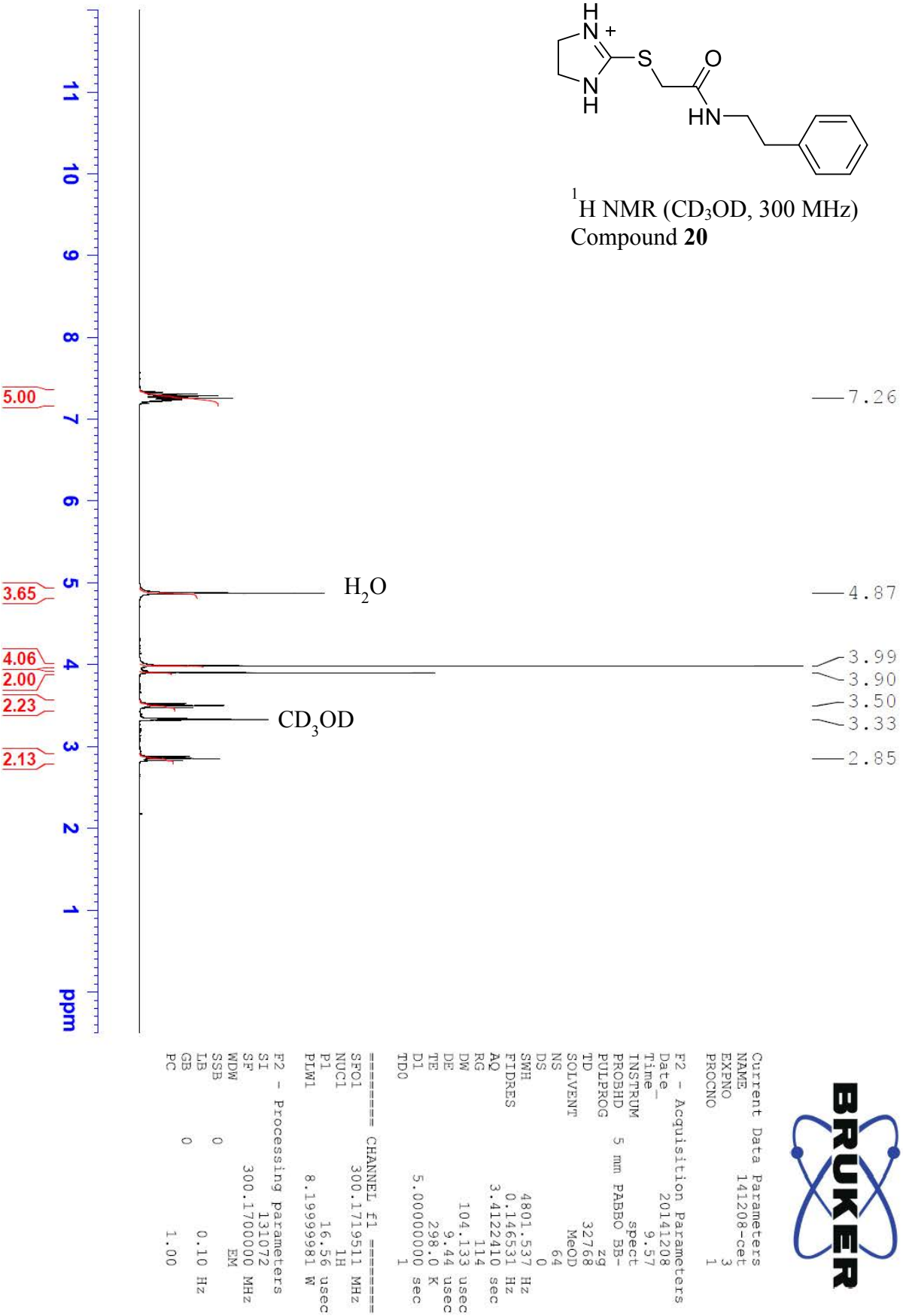
HRMS(ESI) Compound 20-6



Calcd for $C_{10}H_{12}ClNONa$
[M + Na] = 220.0505

^{1}H NMR Compound 20

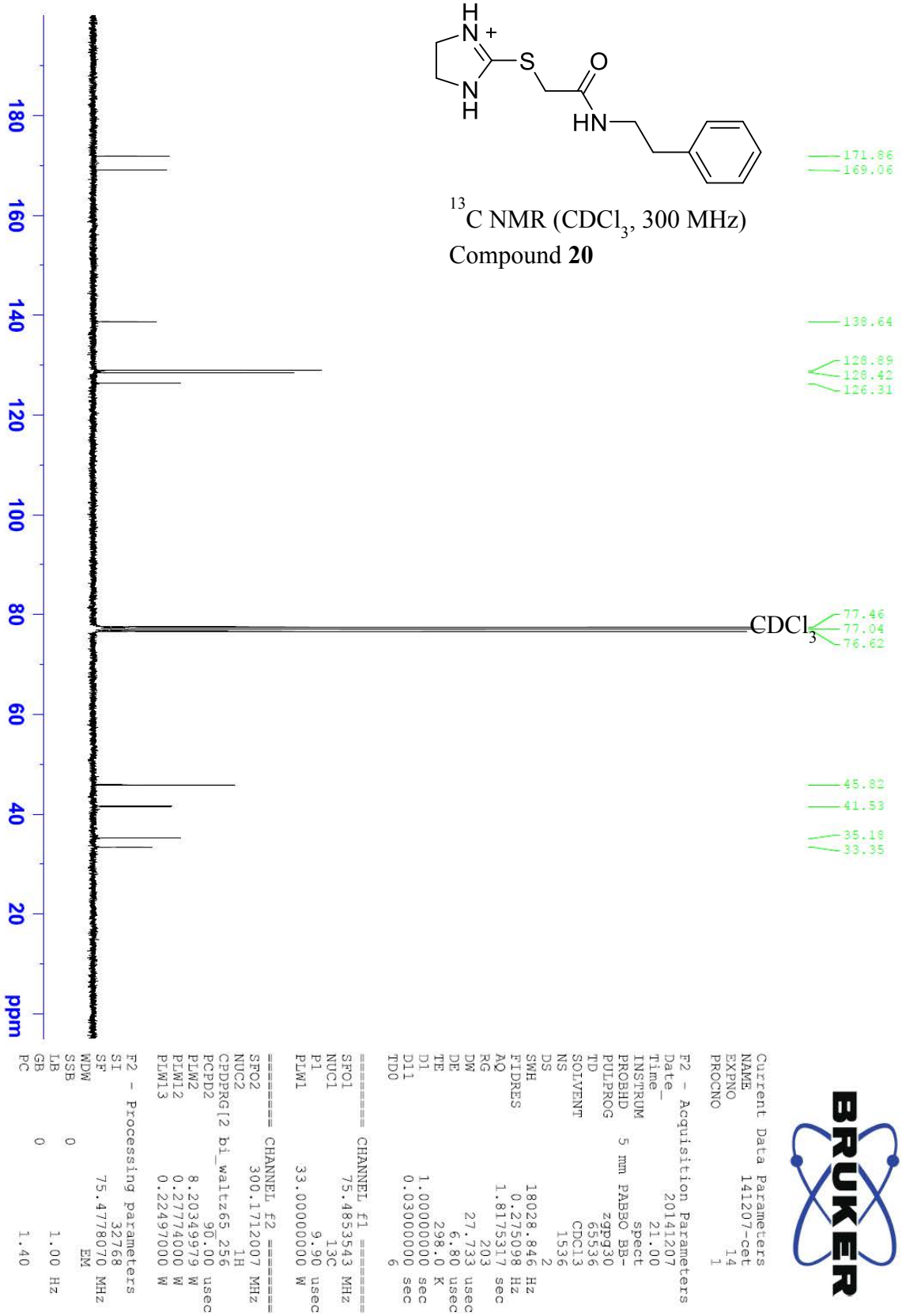
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenethylacetamide



^{1}H NMR (CD_3OD, 300 MHz)
Compound **20**

^{13}C NMR Compound 20

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenethylacetamide



^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **20**

Current Data Parameters
NAME 141207-cet
EXPNO 14
PROCNO 1

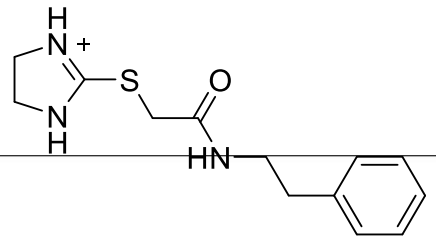
F2 - Acquisition Parameters
Date_ 20141207
Time_ 21.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1536
DS 2
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 203
DW 27.733 usec
DE 6.80 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 6

======== CHANNEL f1 ========
SFO1 75.4853543 MHz
NUC1 13C
P1 9.90 usec
PLW1 33.00000000 W

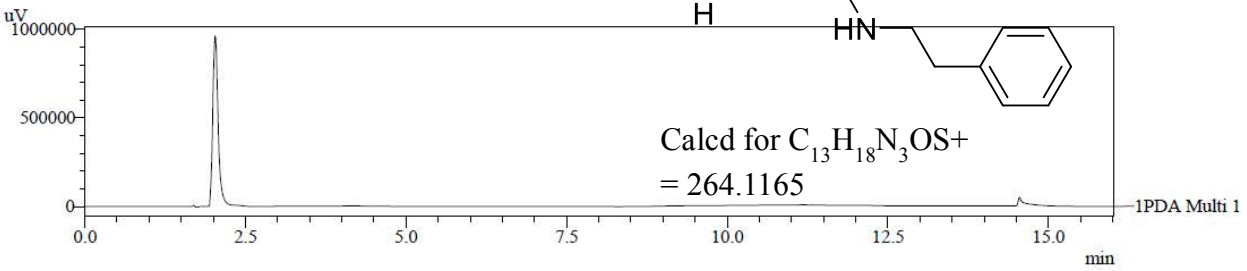
======== CHANNEL f2 ========
SFO2 300.1712007 MHz
NUC2 1H
CPDPRG[2 bi_waltz65_256
PCPD2 90.00 usec
PLW2 8.20349979 W
PLW12 0.27774000 W
PLW13 0.22497000 W

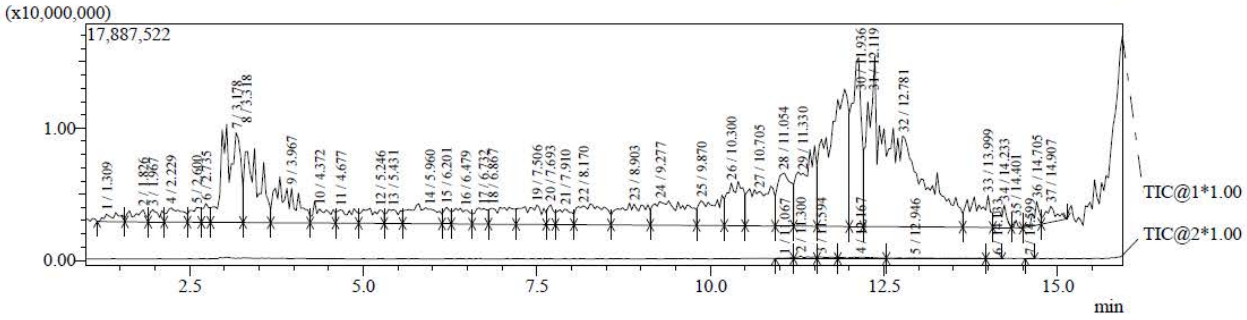
F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

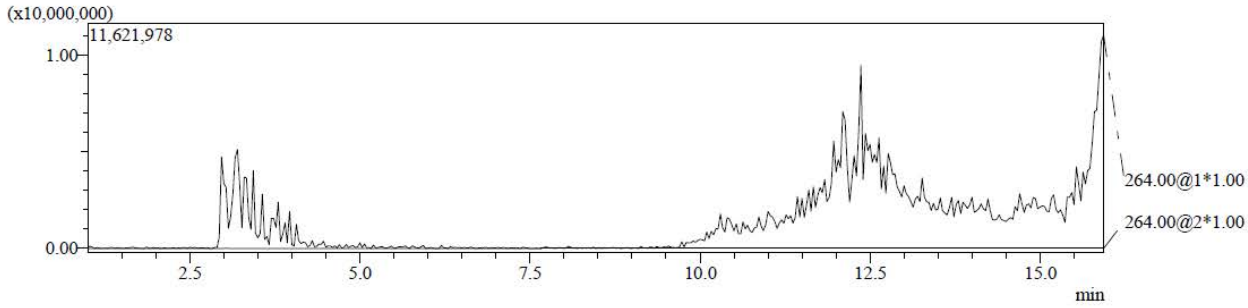
LCMS Compound 20



Calcd for $C_{13}H_{18}N_3OS^+$ = 264.1165

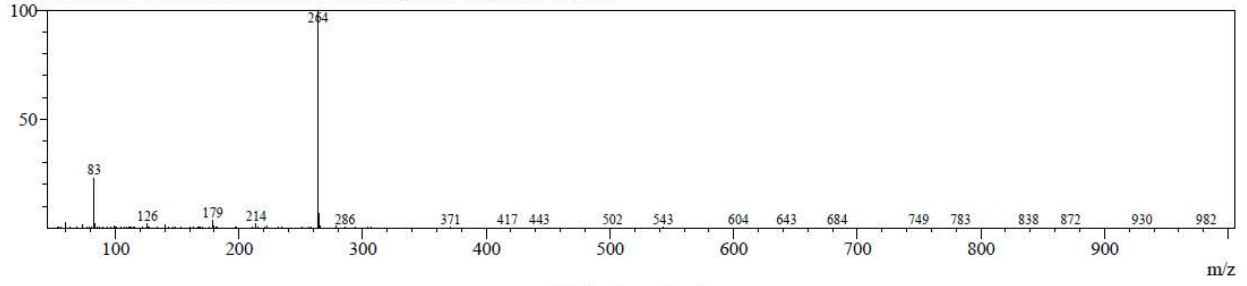






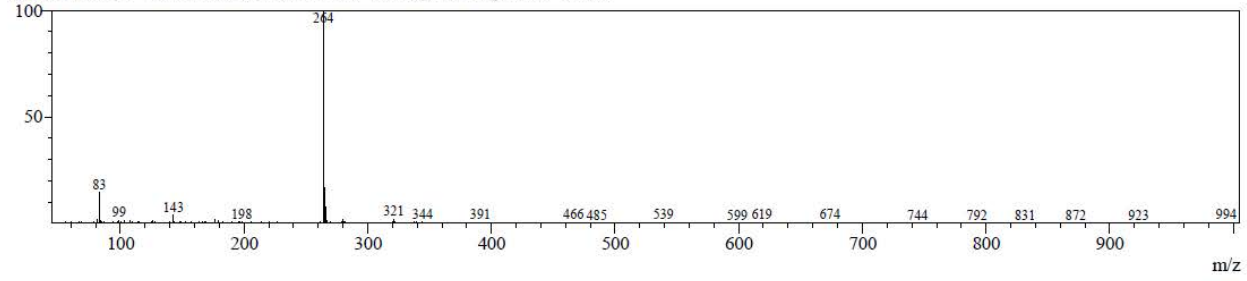
MS Spectrum Graph

Ret.Time:3.200(Scan#:133)
BG Mode:?
Mass Peaks:911 Base Peak:263.90(5107273) Polarity:Pos Segment1 - Event1

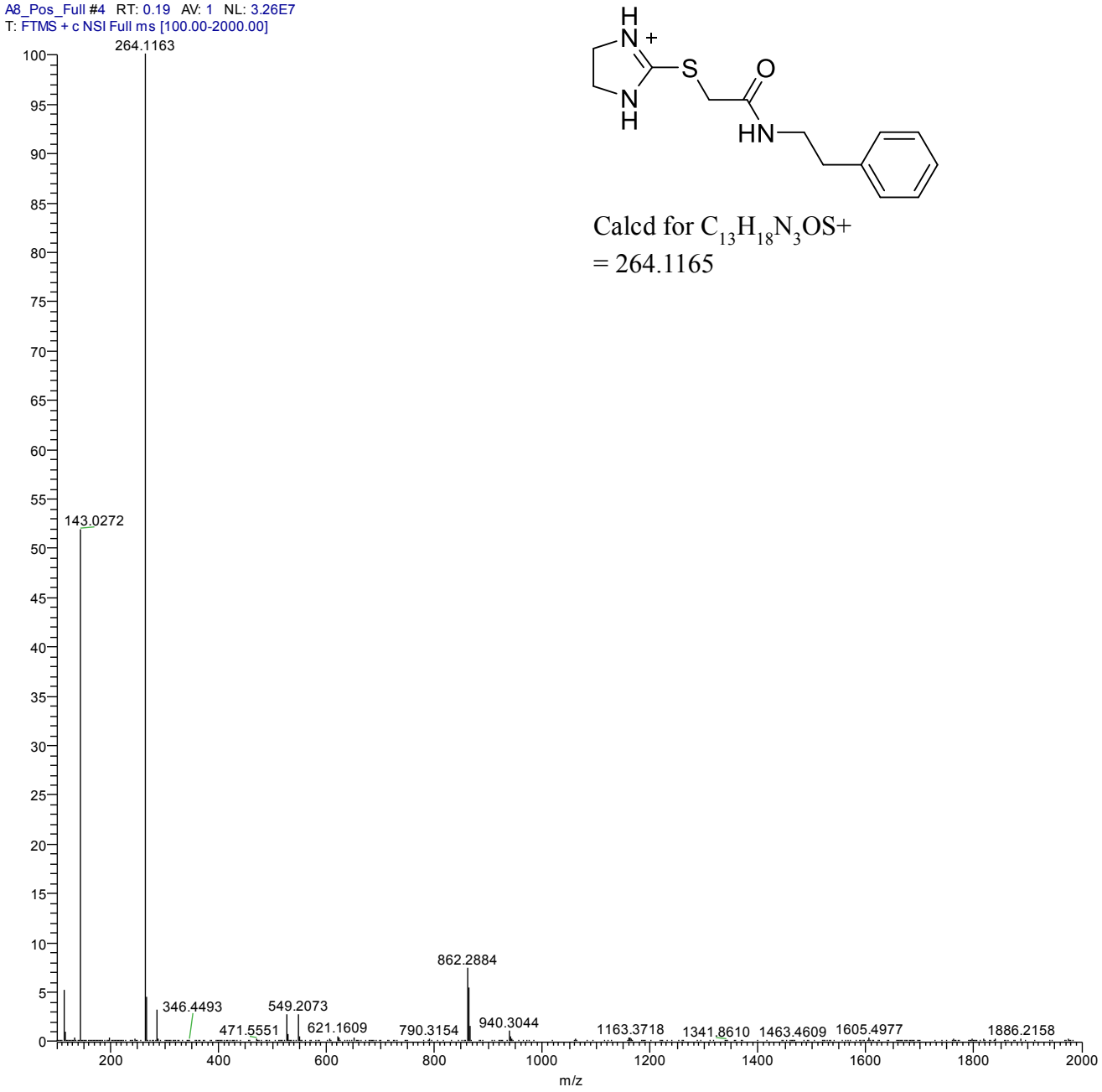


MS Spectrum Graph

Ret.Time:12.133(Scan#:669)
BG Mode:?
Mass Peaks:905 Base Peak:263.95(6616477) Polarity:Pos Segment1 - Event1



HRMS(ESI) Compound 20

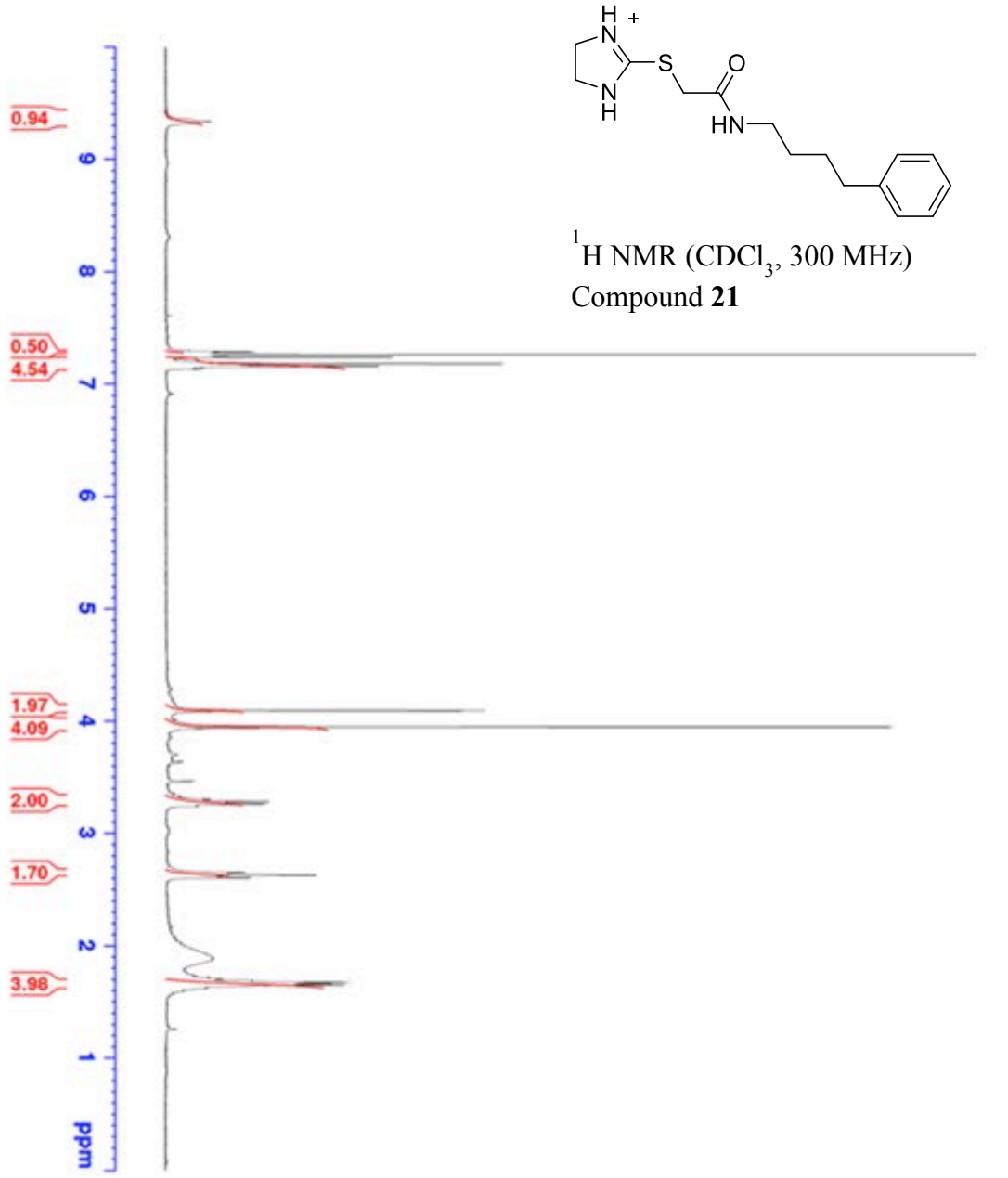


Calcd for $C_{13}H_{18}N_3OS^+$
= 264.1165

Spectra for Compound 21

^{1}H NMR Compound 21

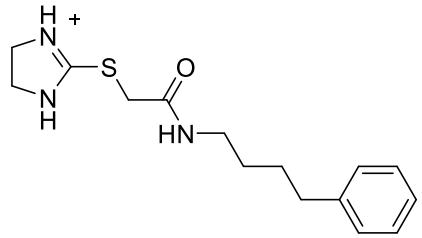
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(4-phenylbutyl)acetamide

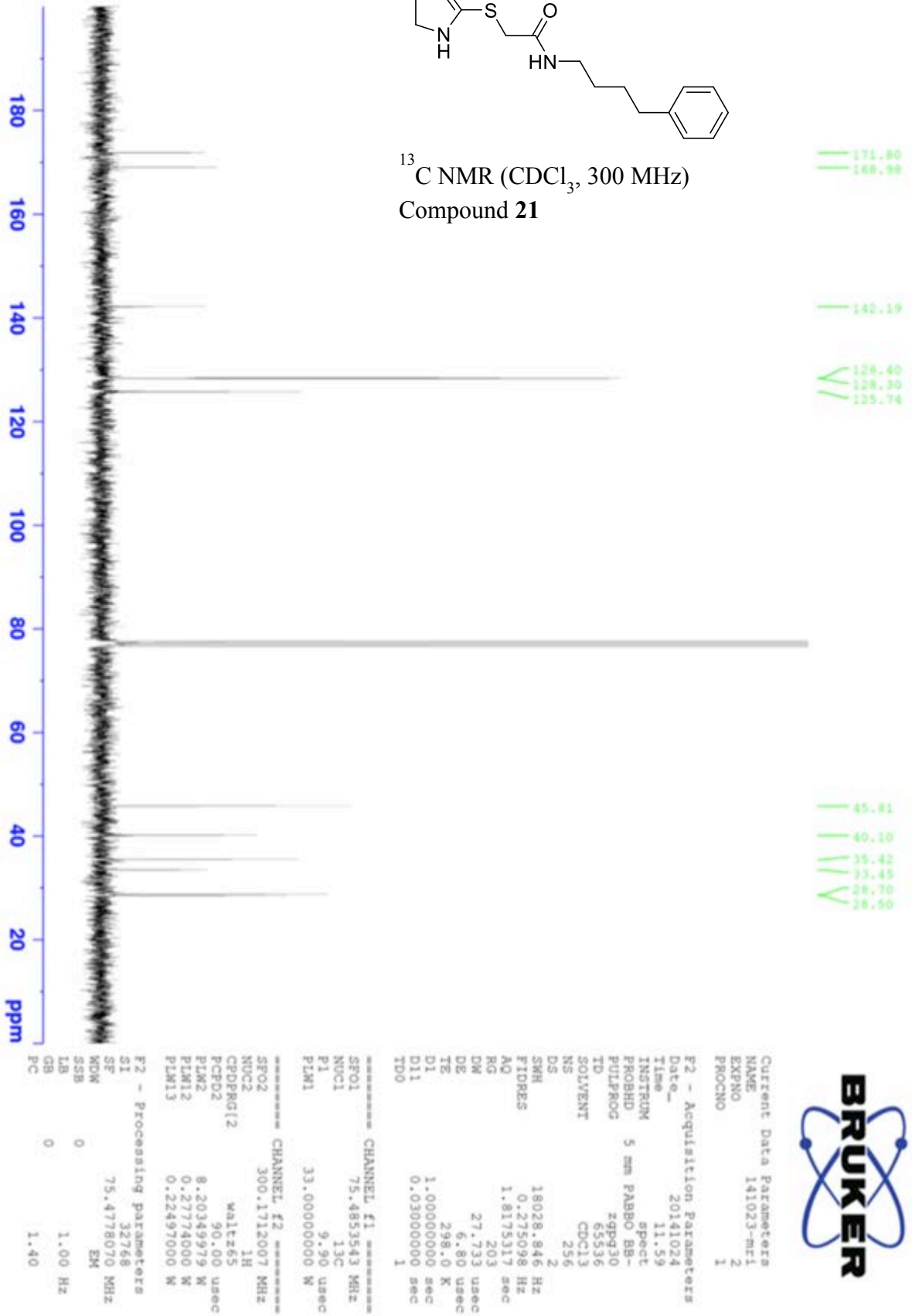


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **21**

^{13}C NMR Compound 21

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(4-phenylbutyl)acetamide

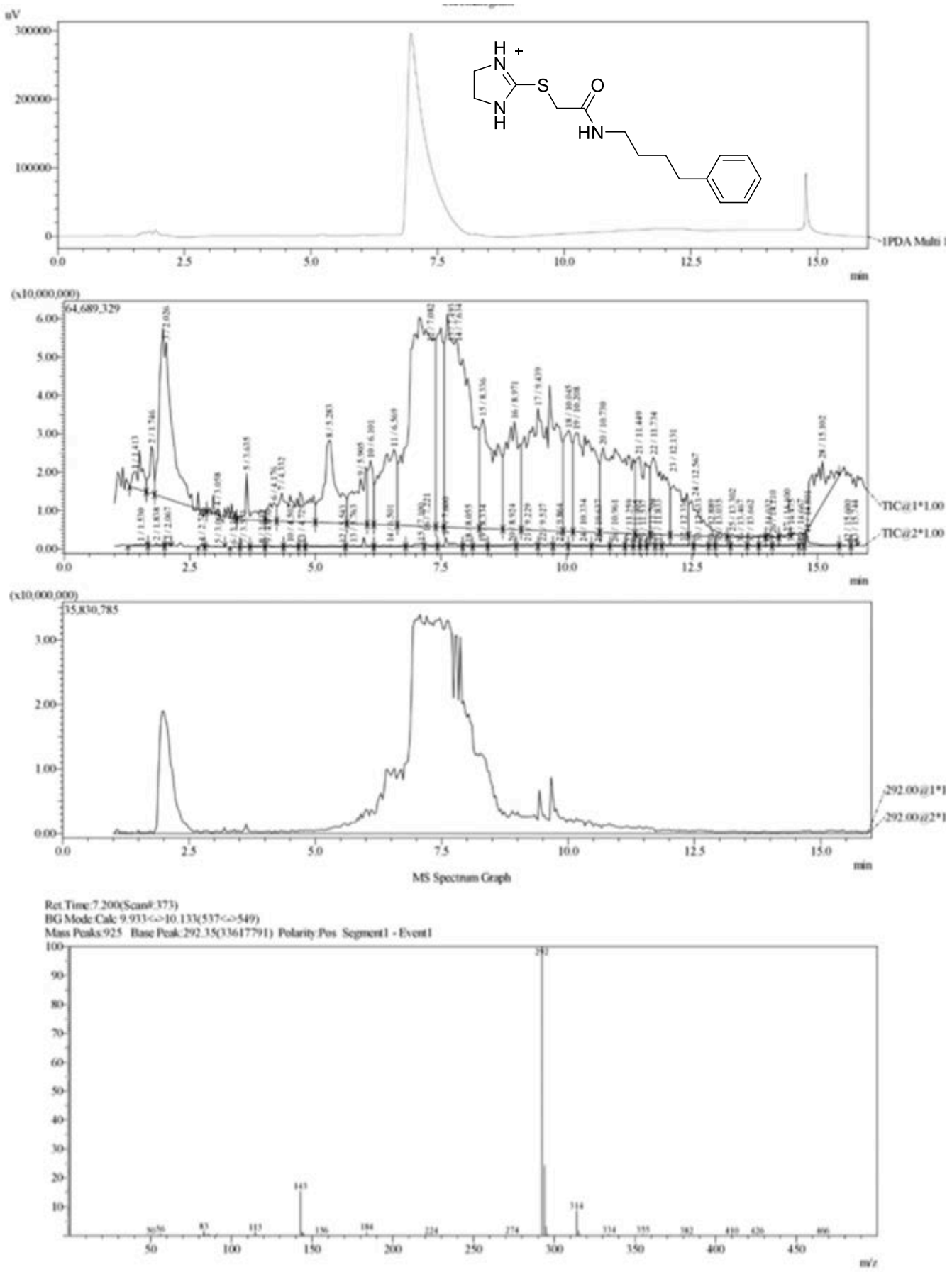




^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **21**

LCMS Compound 21

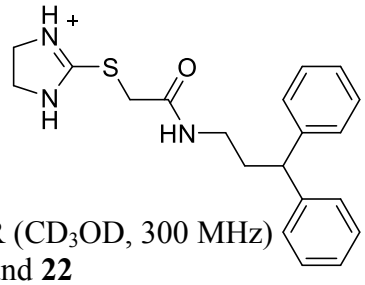
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(4-phenylbutyl)acetamide



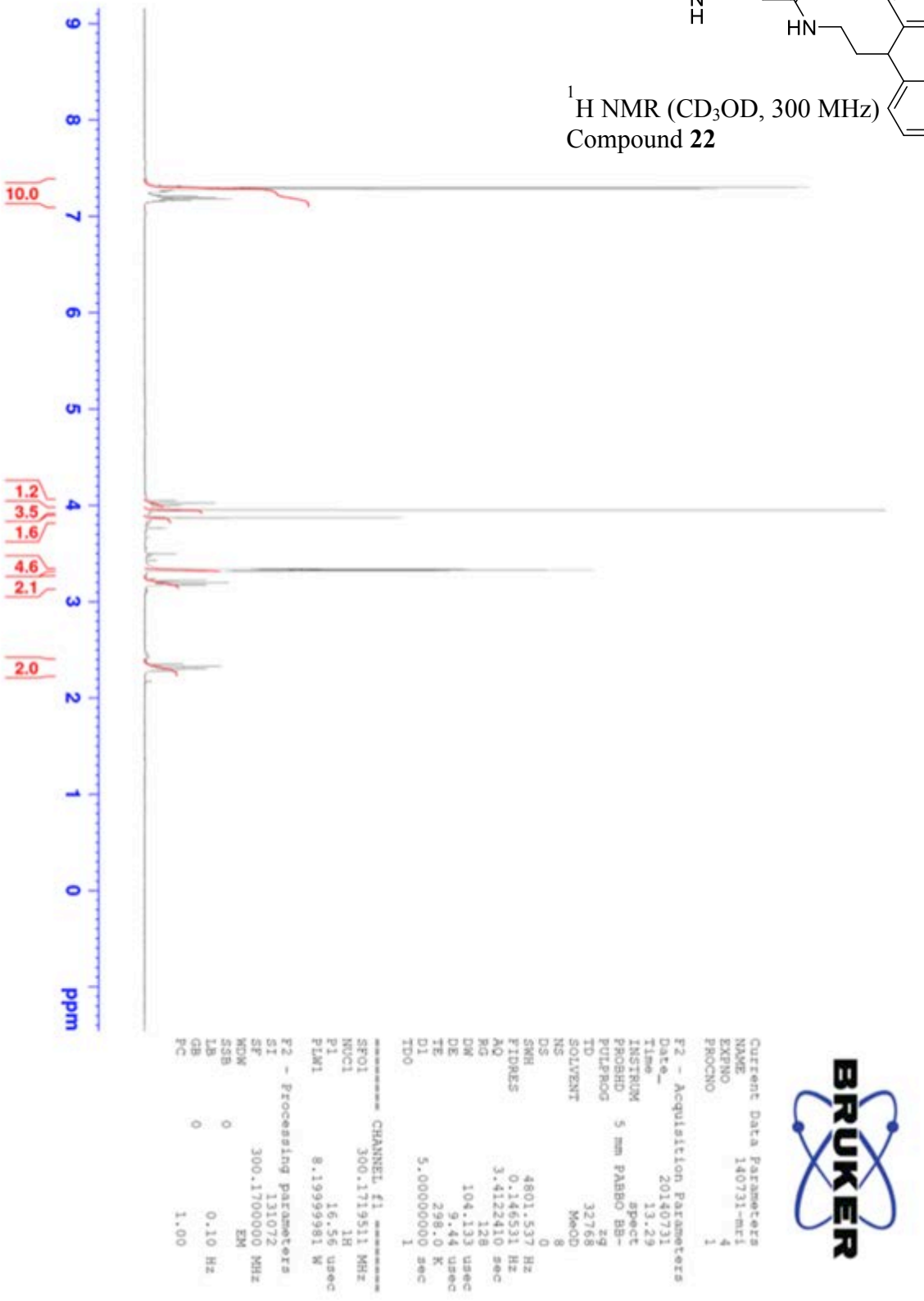
Spectra for Compound 22

^{1}H NMR Compound 22

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(3,3-diphenylpropyl)acetamide

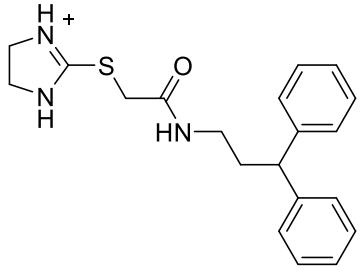


^{1}H NMR (CD_3OD, 300 MHz)
Compound **22**



^{13}C NMR Compound 22

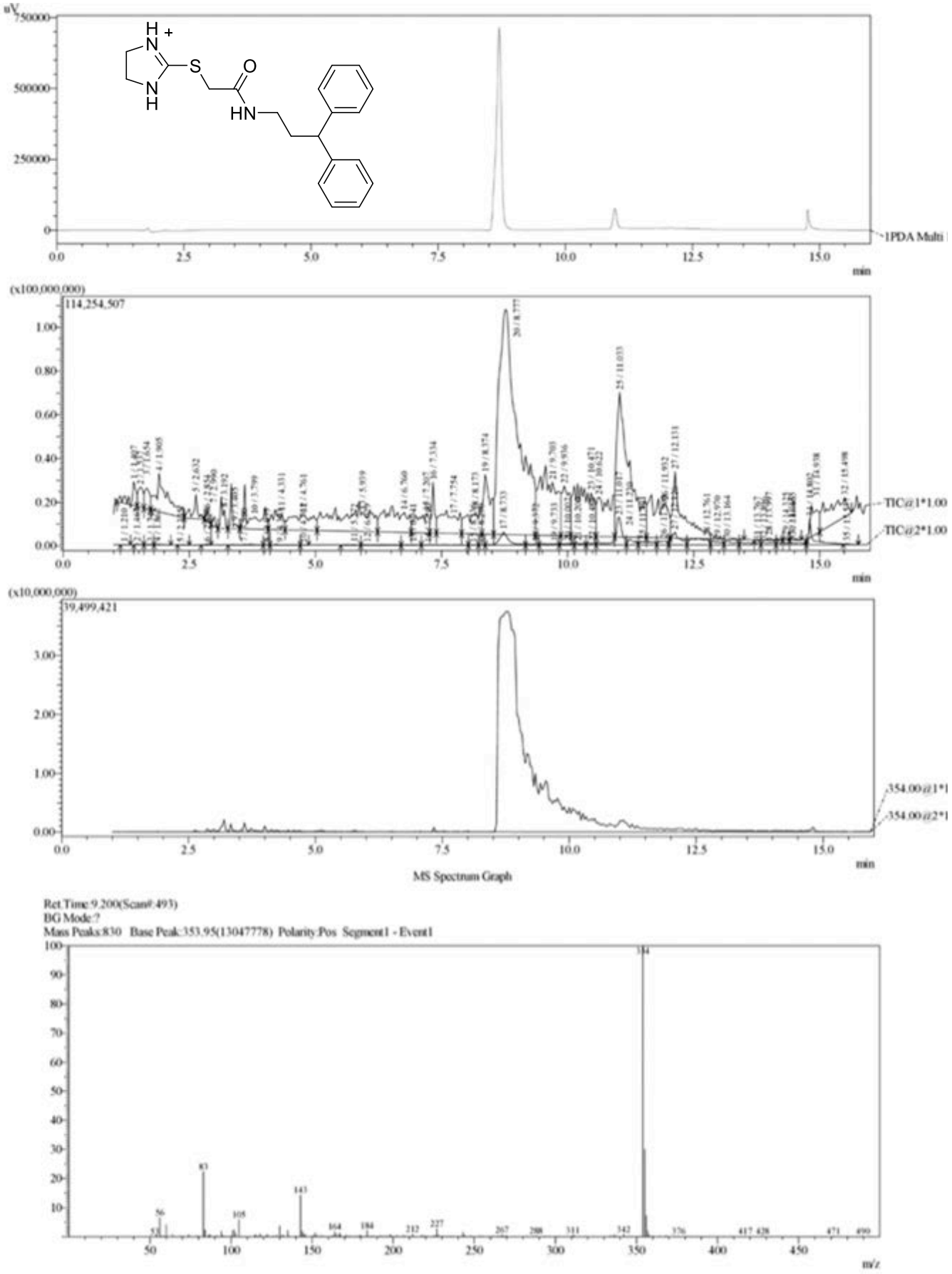
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(3,3-diphenylpropyl)acetamide



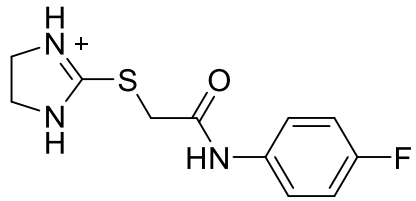
^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **22**

LCMS of Compound 22

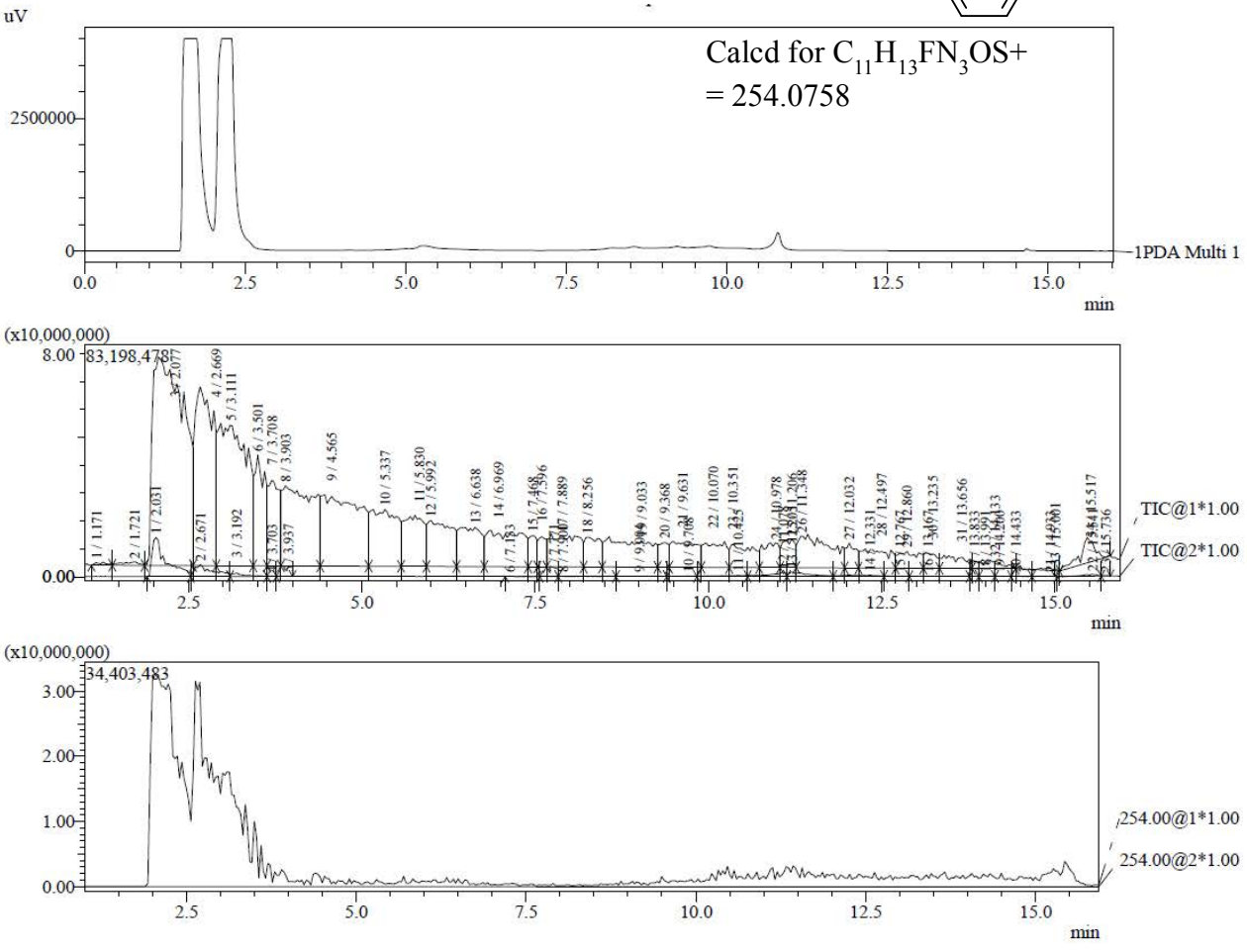
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(3,3-diphenylpropyl)acetamide



Spectra for Compound 23

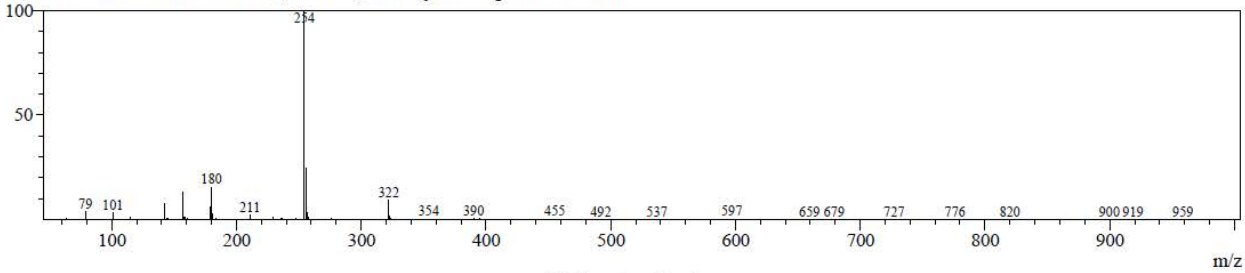


LCMS Compound 23



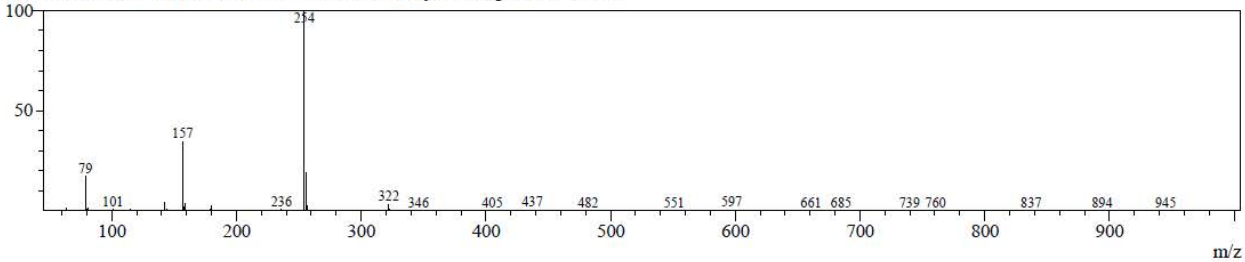
MS Spectrum Graph

Ret.Time:2.033(Scan#:63)
BG Mode:None
Mass Peaks:913 Base Peak:254.30(32592774) Polarity:Pos Segment1 - Event1

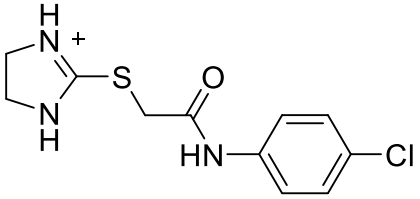


MS Spectrum Graph

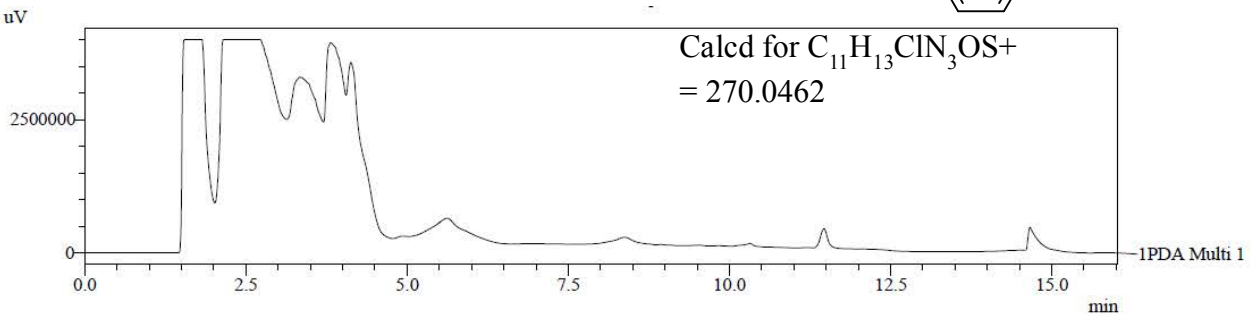
Ret.Time:2.700(Scan#:103)
BG Mode:?
Mass Peaks:920 Base Peak:254.30(31377512) Polarity:Pos Segment1 - Event1

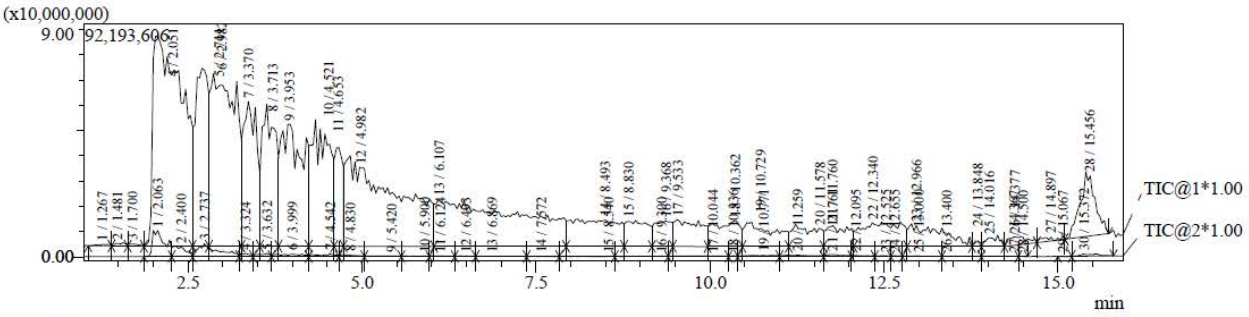


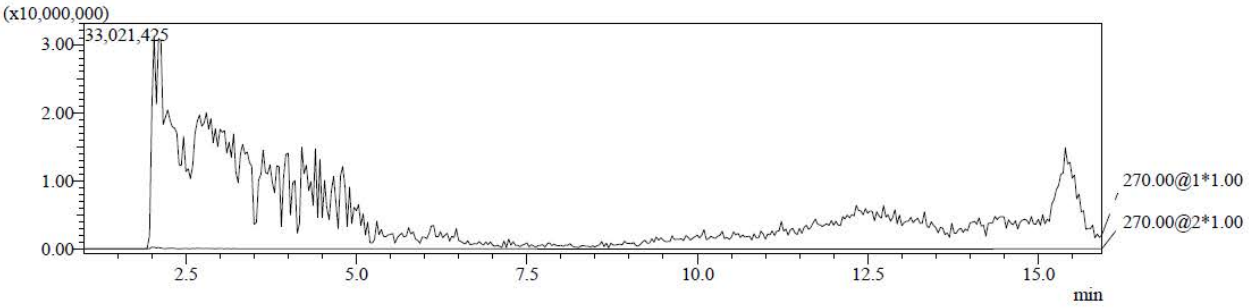
Spectra for Compound 24



LCMS Compound 24

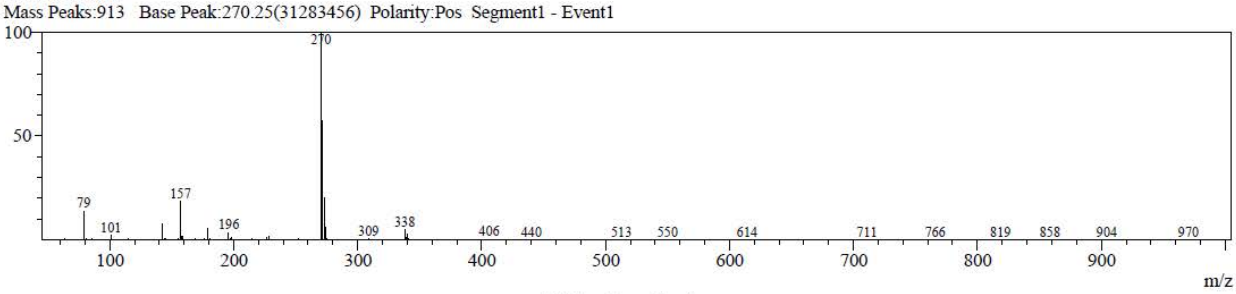






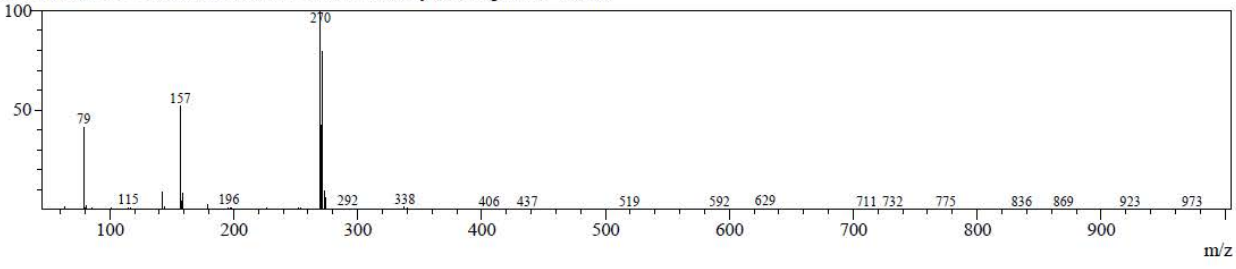
MS Spectrum Graph

Ret.Time:2.033(Scan#:63)
BG Mode:None
Mass Peaks:913 Base Peak:270.25(31283456) Polarity:Pos Segment1 - Event1

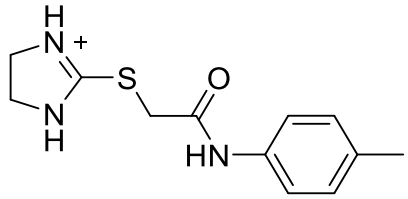


MS Spectrum Graph

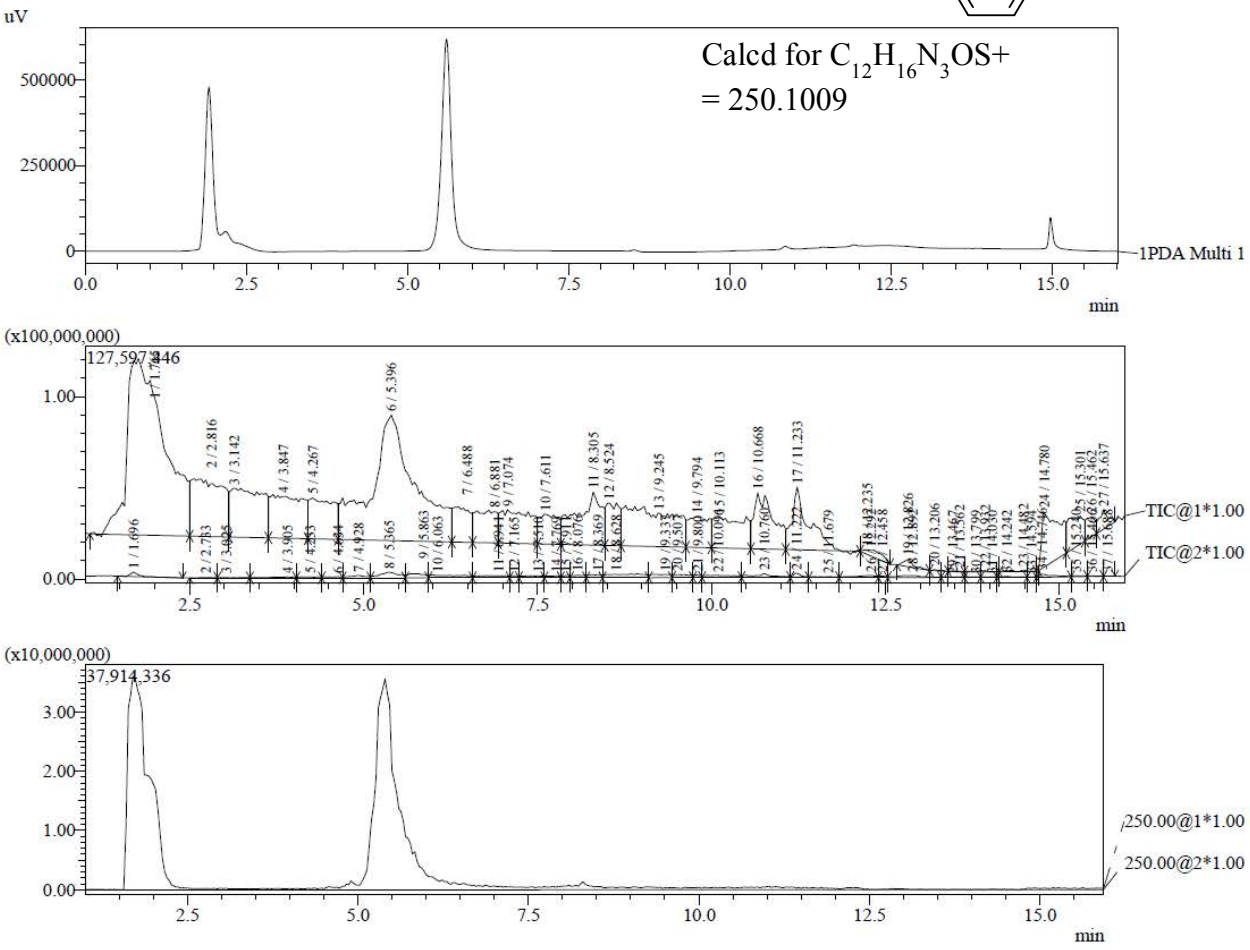
Ret.Time:2.767(Scan#:107)
BG Mode:?
Mass Peaks:922 Base Peak:269.85(18518087) Polarity:Pos Segment1 - Event1



Spectra for Compound 25

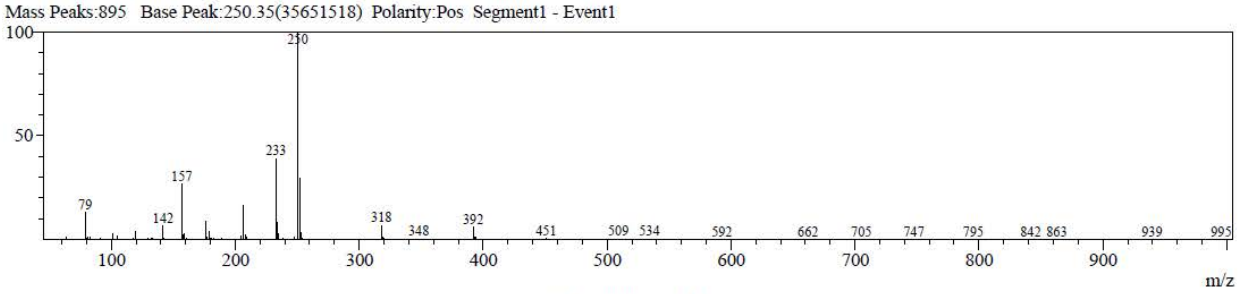


LCMS Compound 25



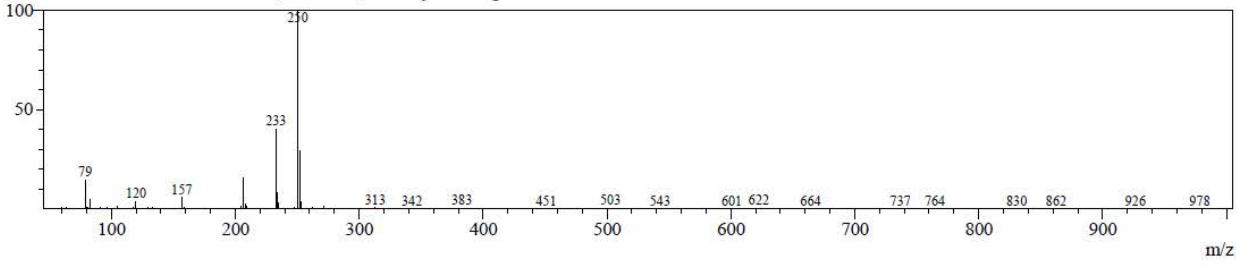
MS Spectrum Graph

Ret.Time:1.733(Scan#:45)
BG Mode:?
Mass Peaks:895 Base Peak:250.35(35651518) Polarity:Pos Segment1 - Event1

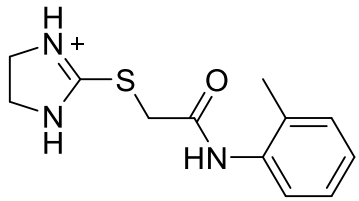


MS Spectrum Graph

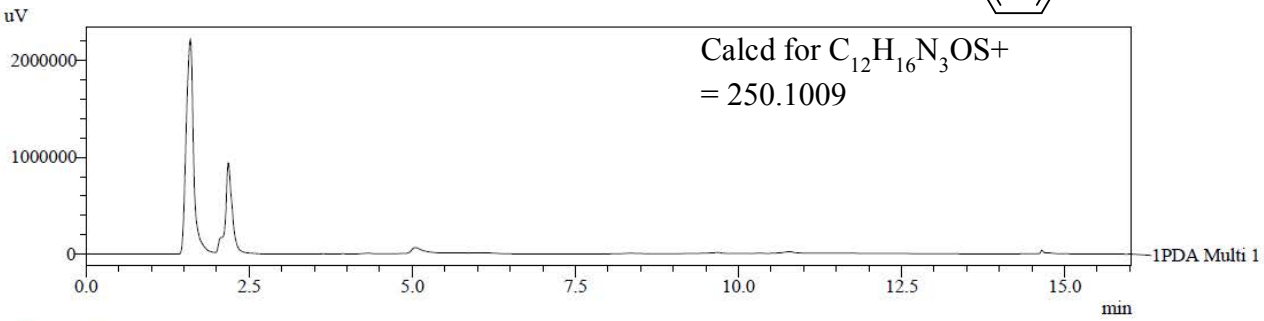
Ret.Time:5.367(Scan#:263)
BG Mode:?
Mass Peaks:903 Base Peak:250.30(33991177) Polarity:Pos Segment1 - Event1

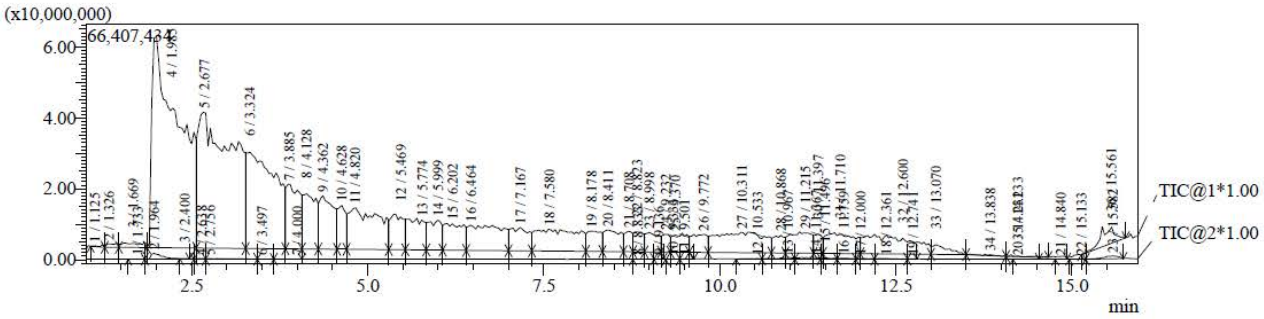


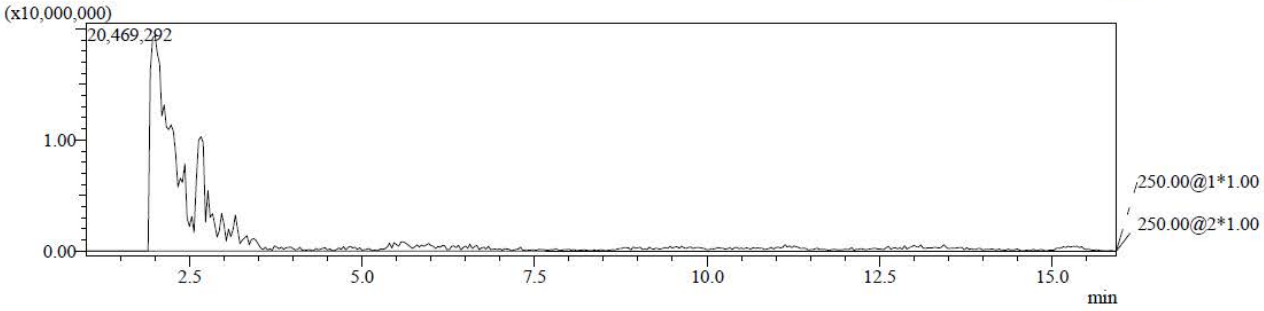
Spectra for Compound 26



LCMS Compound 26

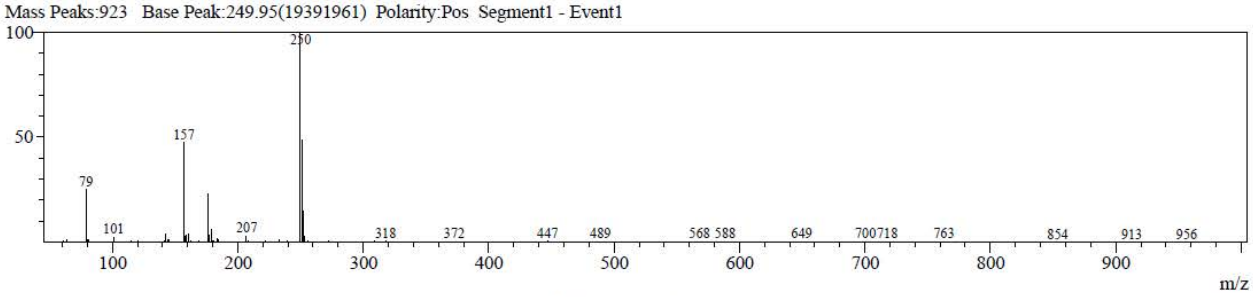






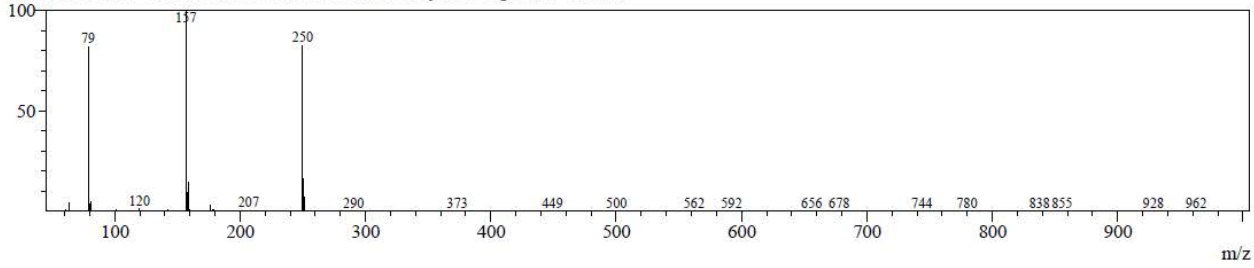
MS Spectrum Graph

Ret.Time:2.000(Scan#:61)
BG Mode:None
Mass Peaks:923 Base Peak:249.95(19391961) Polarity:Pos Segment1 - Event1



MS Spectrum Graph

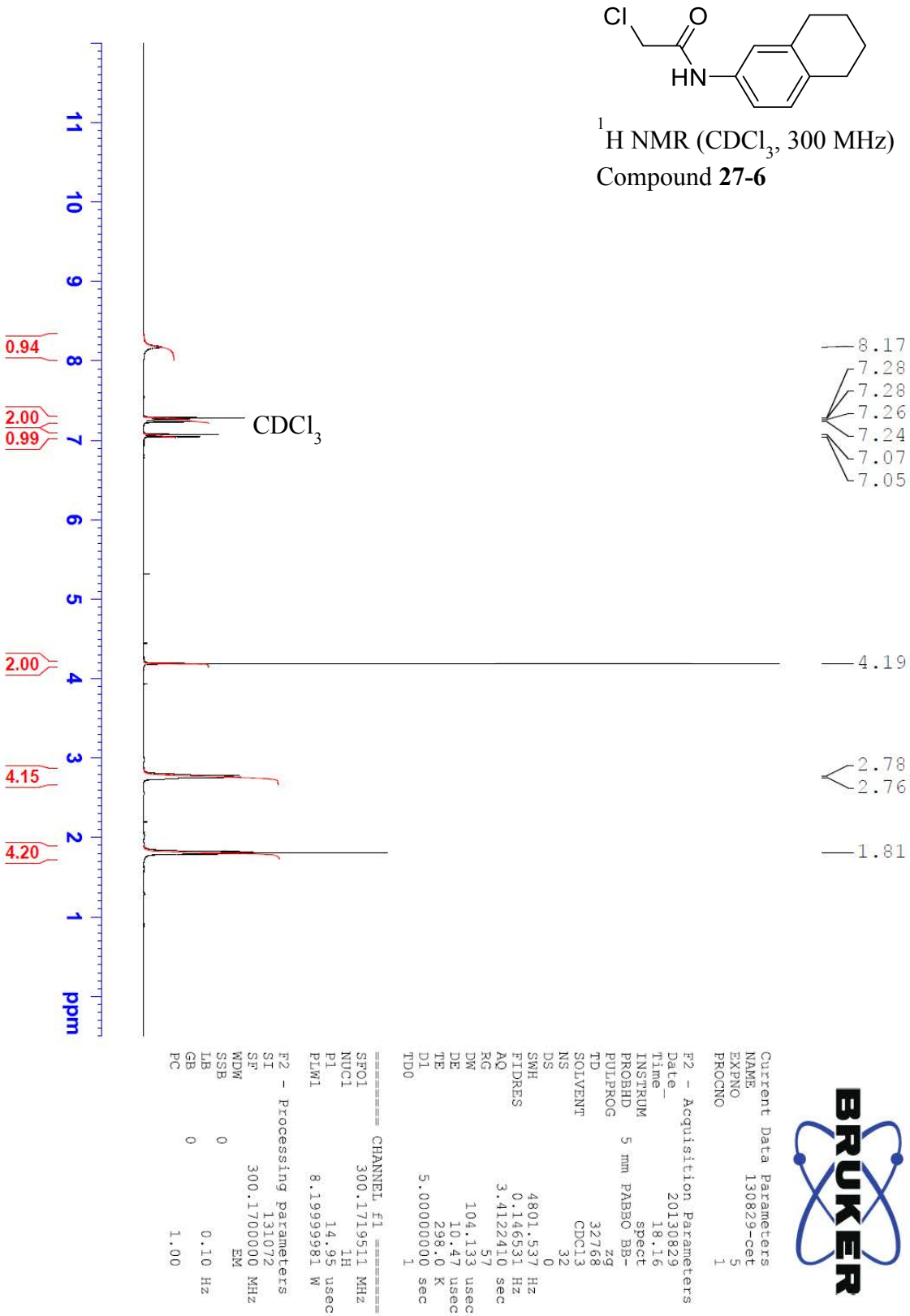
Ret.Time:2.700(Scan#:103)
BG Mode:?
Mass Peaks:890 Base Peak:156.80(11829281) Polarity:Pos Segment1 - Event1



Spectra for Compound 27

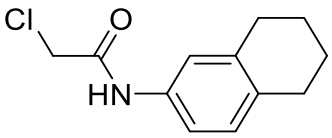
^{1}H NMR Compound 27-6

2-chloro-N-(5,6,7,8-tetrahydro-2-naphthyl)acetamide

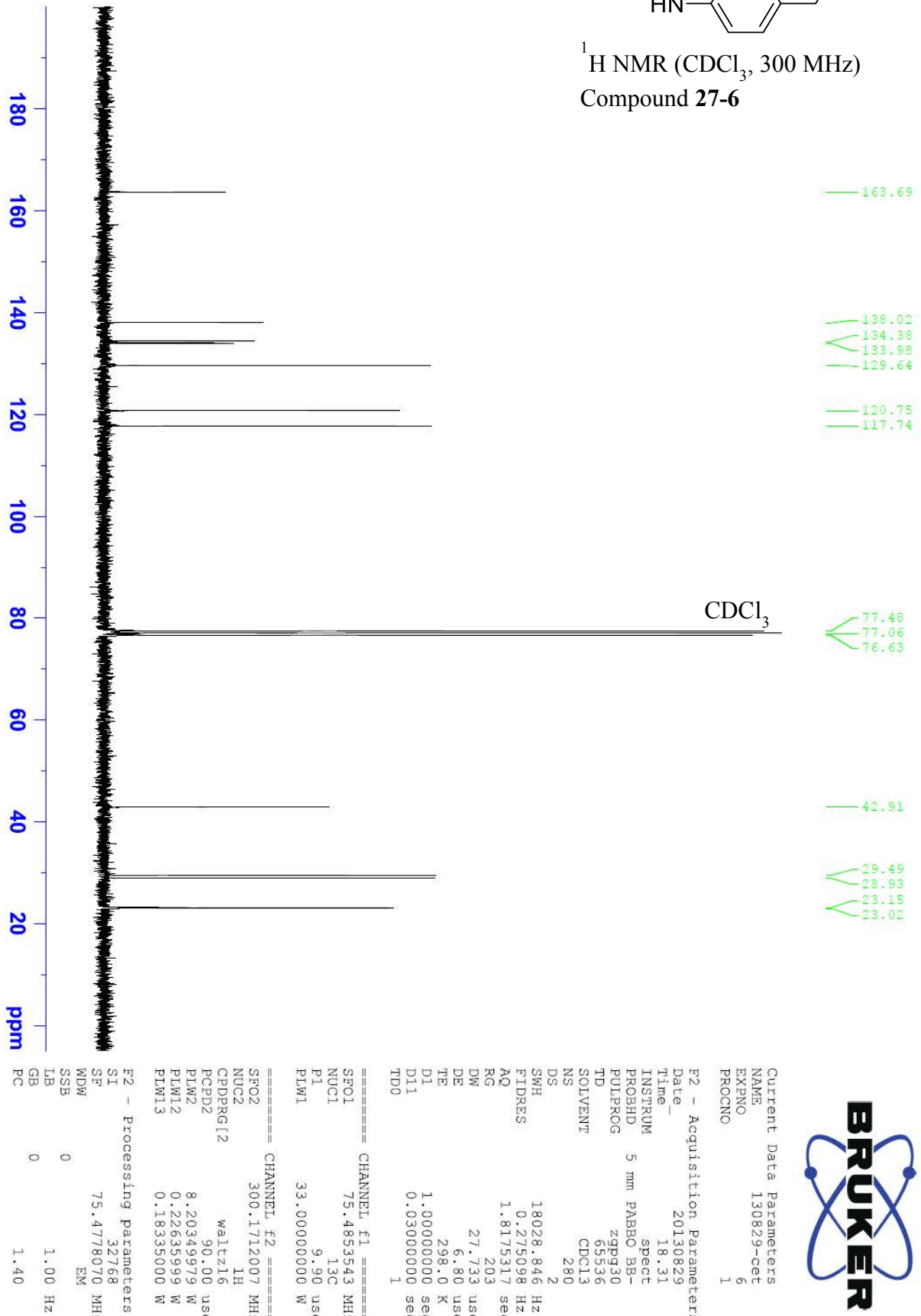


^{13}C NMR Compound 27-6

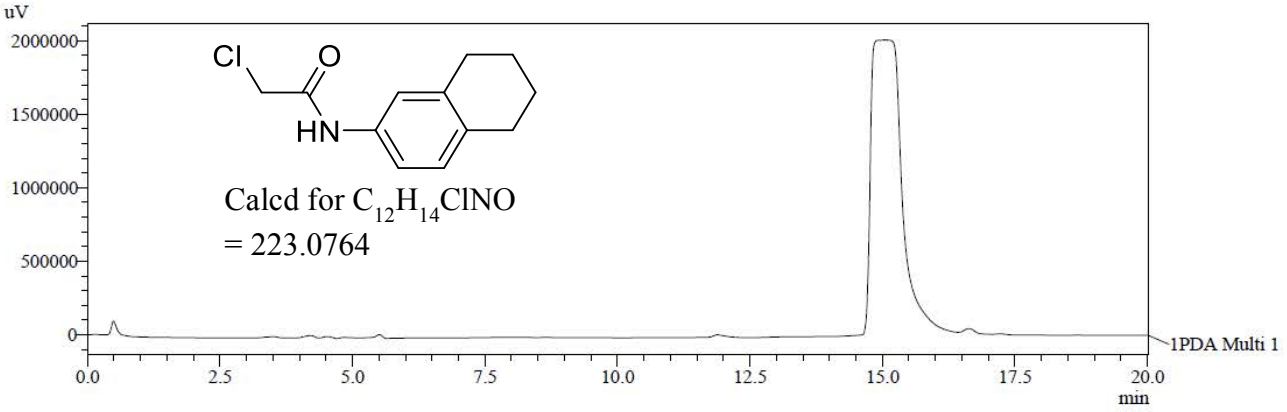
2-chloro-N-(5,6,7,8-tetrahydro-2-naphthyl)acetamide

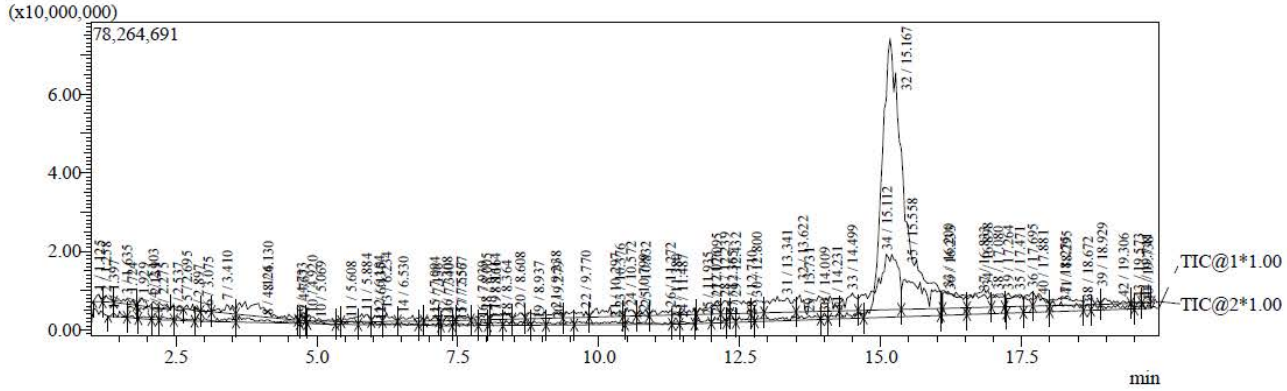


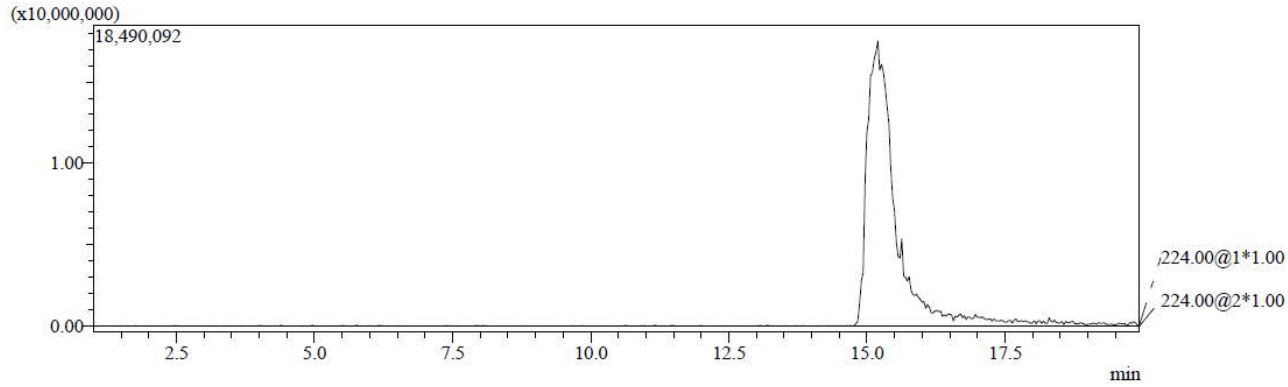
^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **27-6**



LCMS Compound 27-6

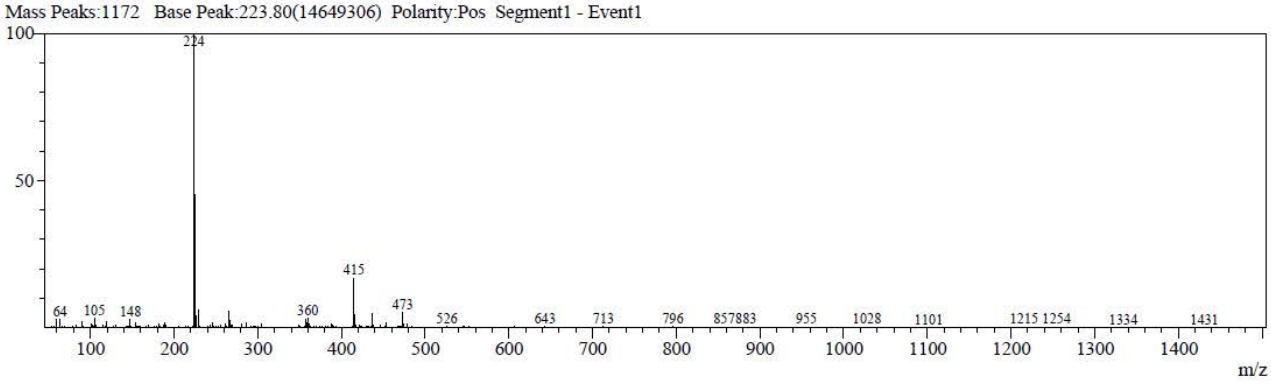




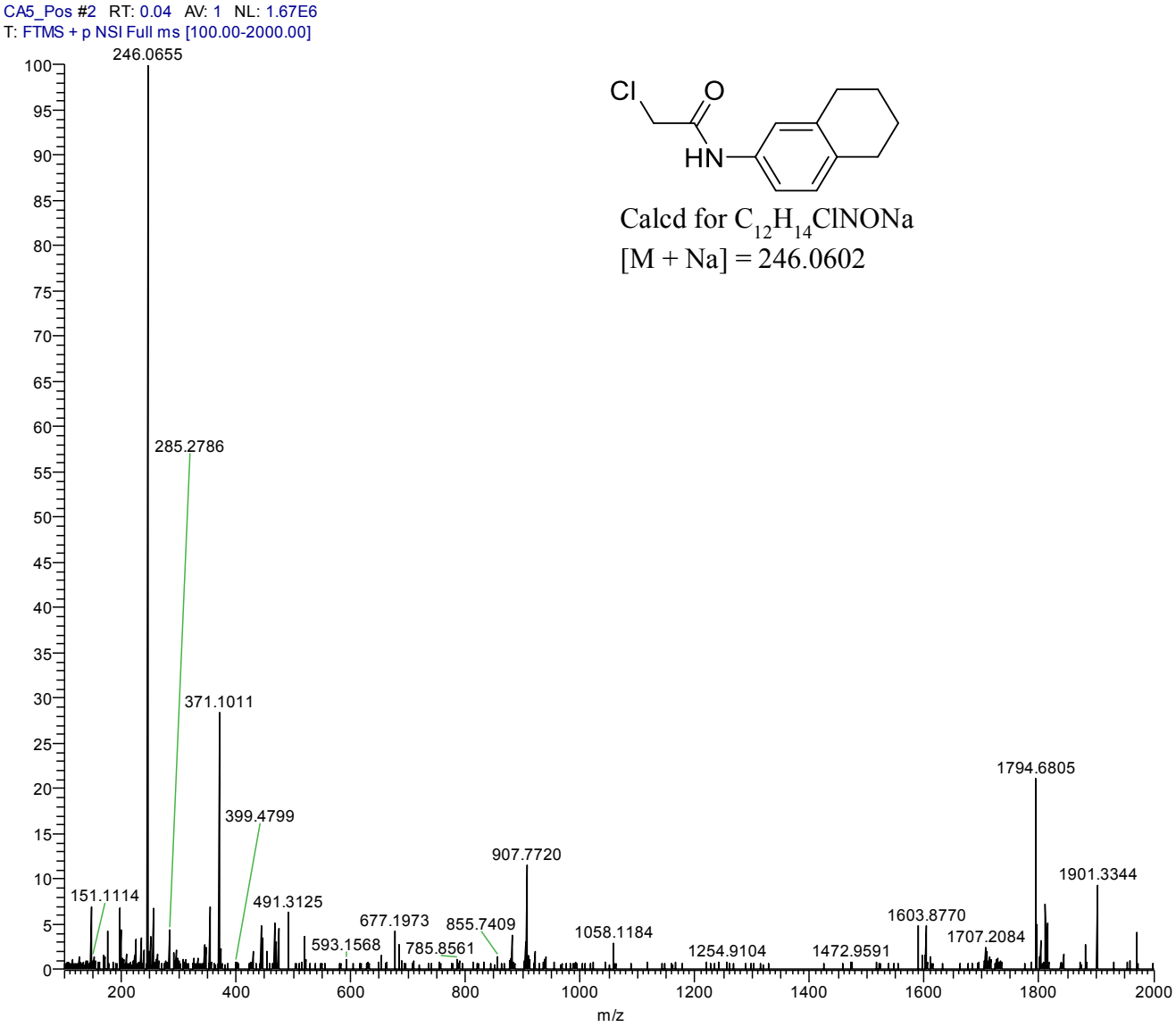


MS Spectrum Graph

Ret.Time:15.333(Scan#:861)
BG Mode:None
Mass Peaks:1172 Base Peak:223.80(14649306) Polarity:Pos Segment1 - Event1



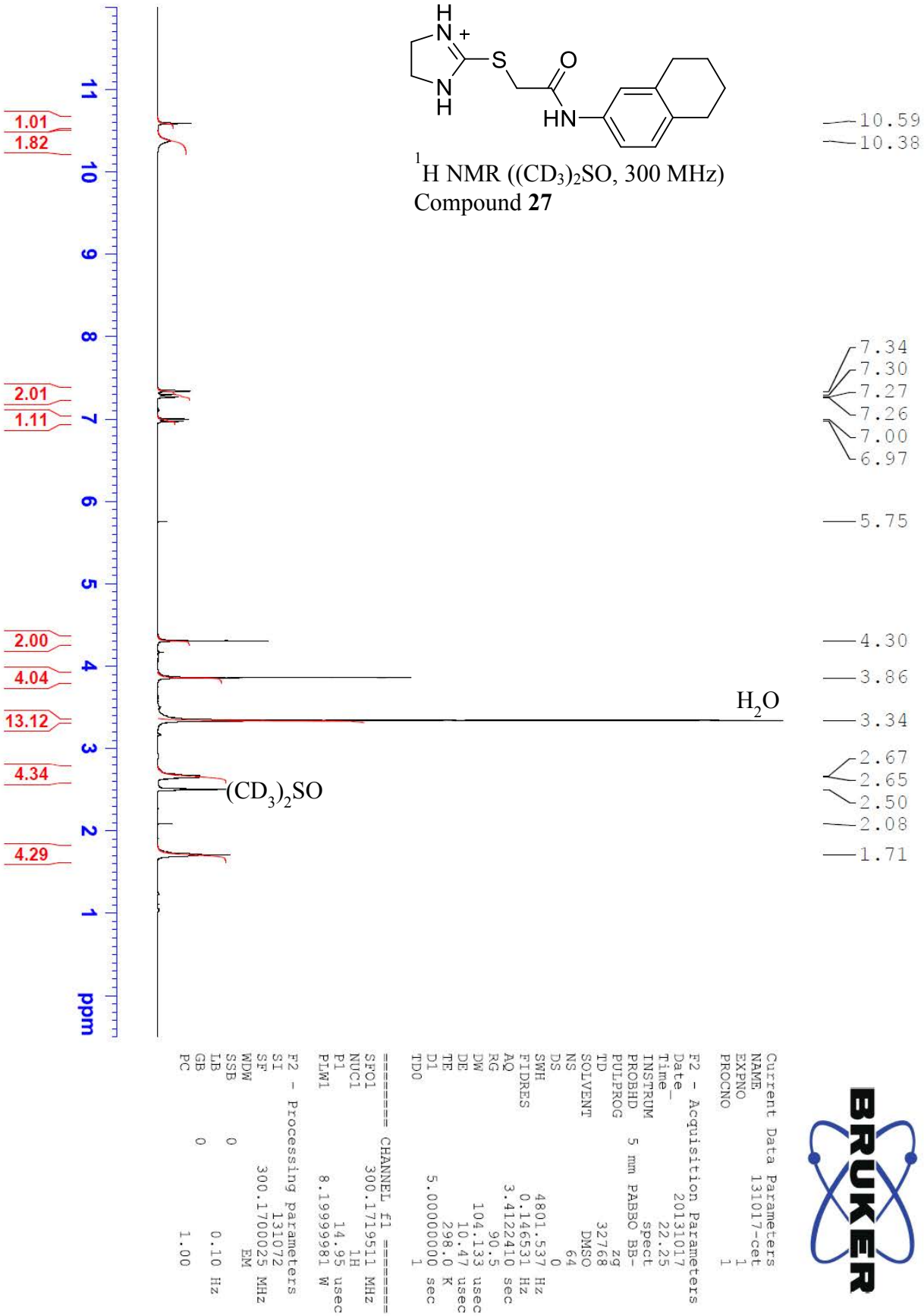
HRMS(ESI) Compound 27-6



Calcd for $C_{12}H_{14}ClNONa$
[M + Na] = 246.0602

^{1}H NMR Compound 27

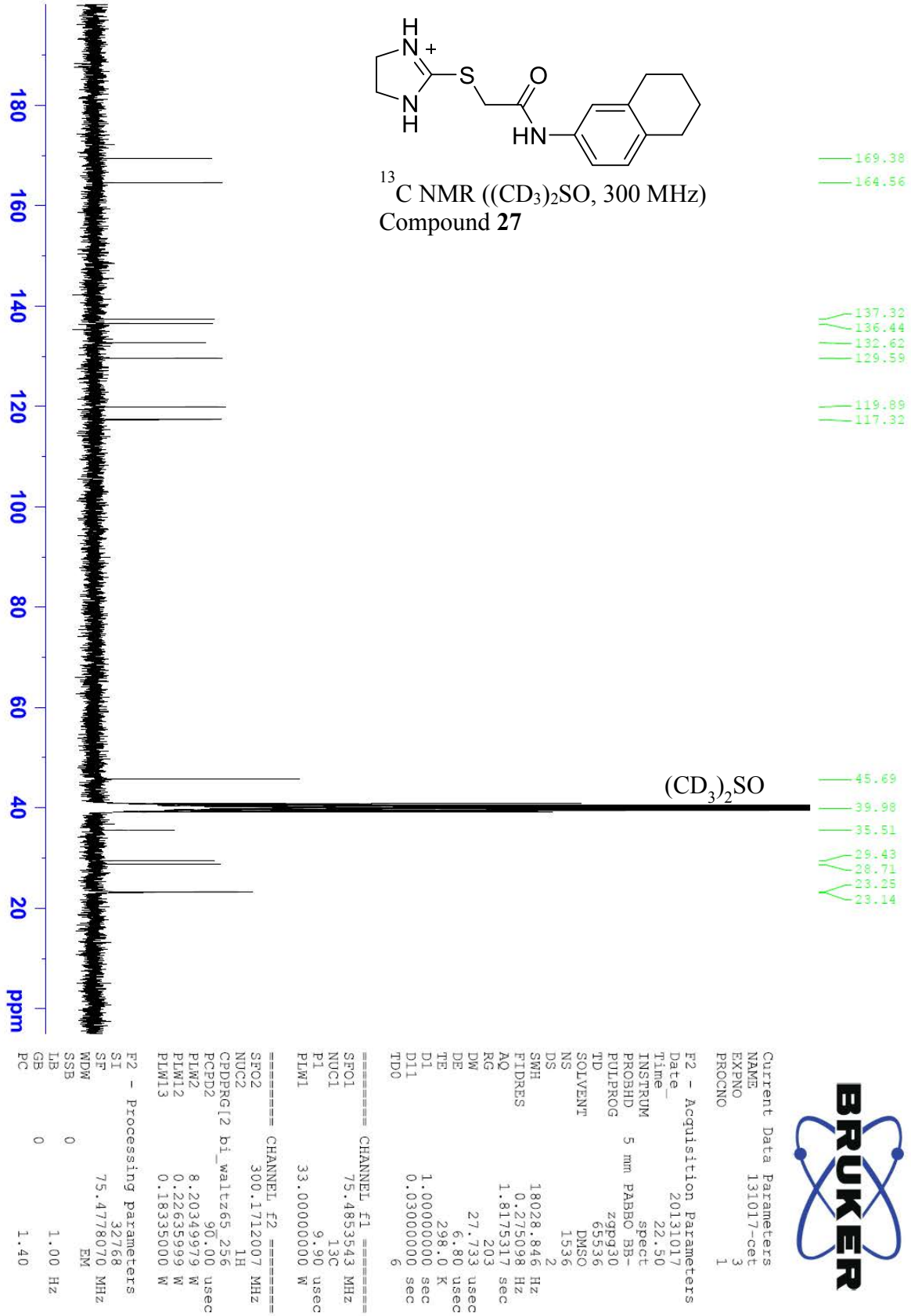
2-((4,5-dihydro-1H-imidazol-2-yl)thio)- N-(5,6,7,8-tetrahydro-2-naphthyl)acetamide



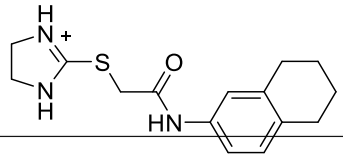
^{1}H NMR ((CD$_3$)$_2$SO, 300 MHz)
Compound **27**

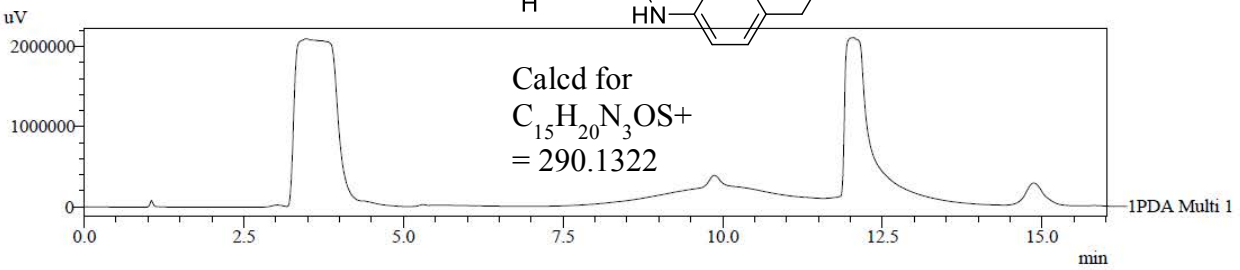
^{13}C NMR Compound 27

2-((4,5-dihydro-1H-imidazol-2-yl)thio)- N-(5,6,7,8-tetrahydro-2-naphthyl)acetamide

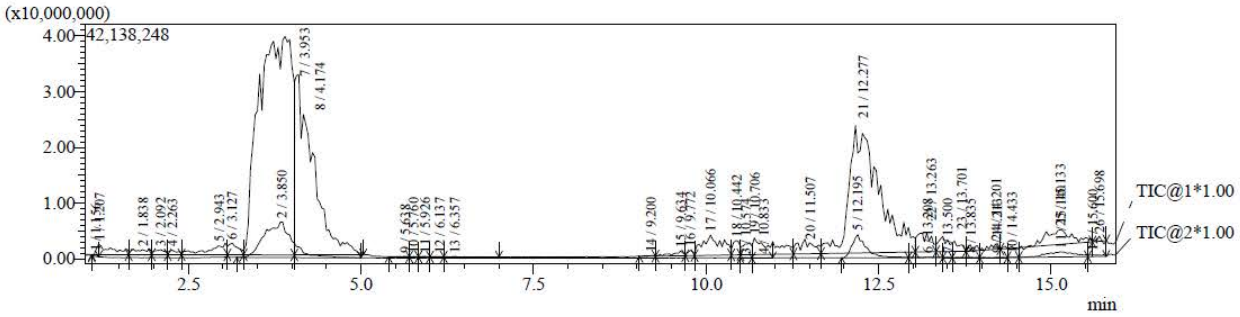


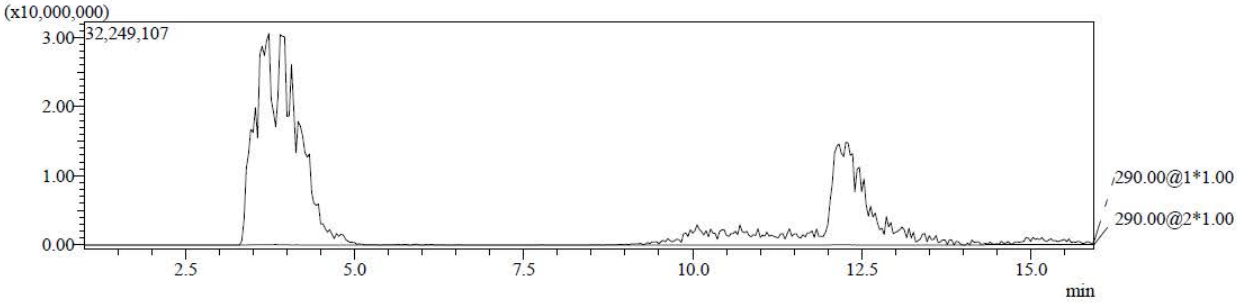
LCMS Compound 27





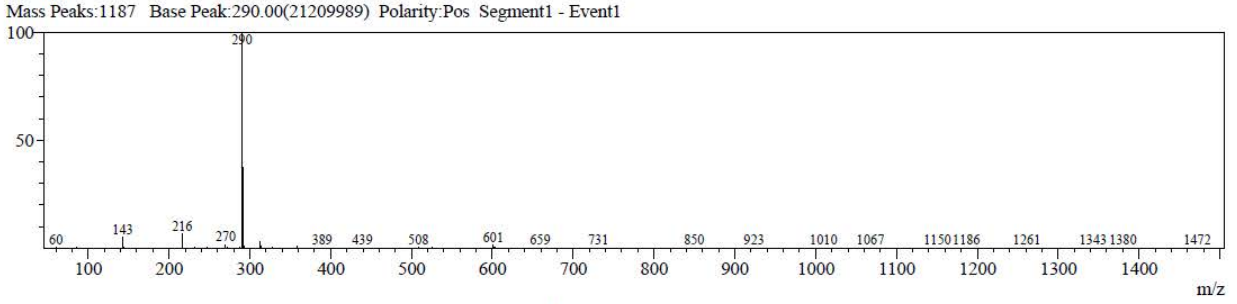
Calcd for
$C_{15}H_{20}N_3OS^+$
= 290.1322





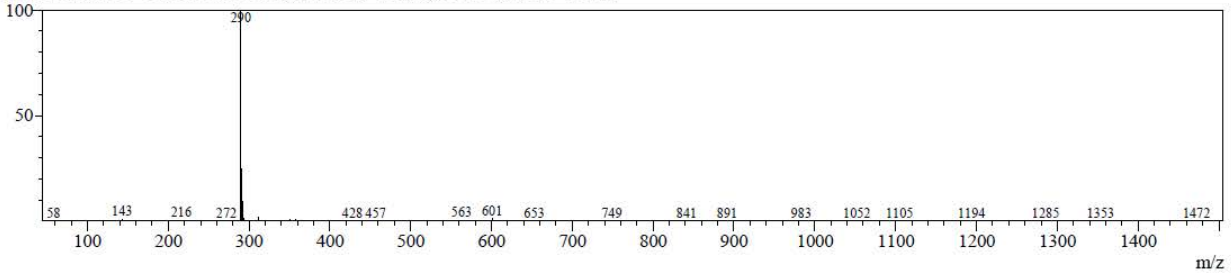
MS Spectrum Graph

Ret.Time:3.867(Scan#:173)
BG Mode:?
Mass Peaks:1187 Base Peak:290.00(21209989) Polarity:Pos Segment1 - Event1

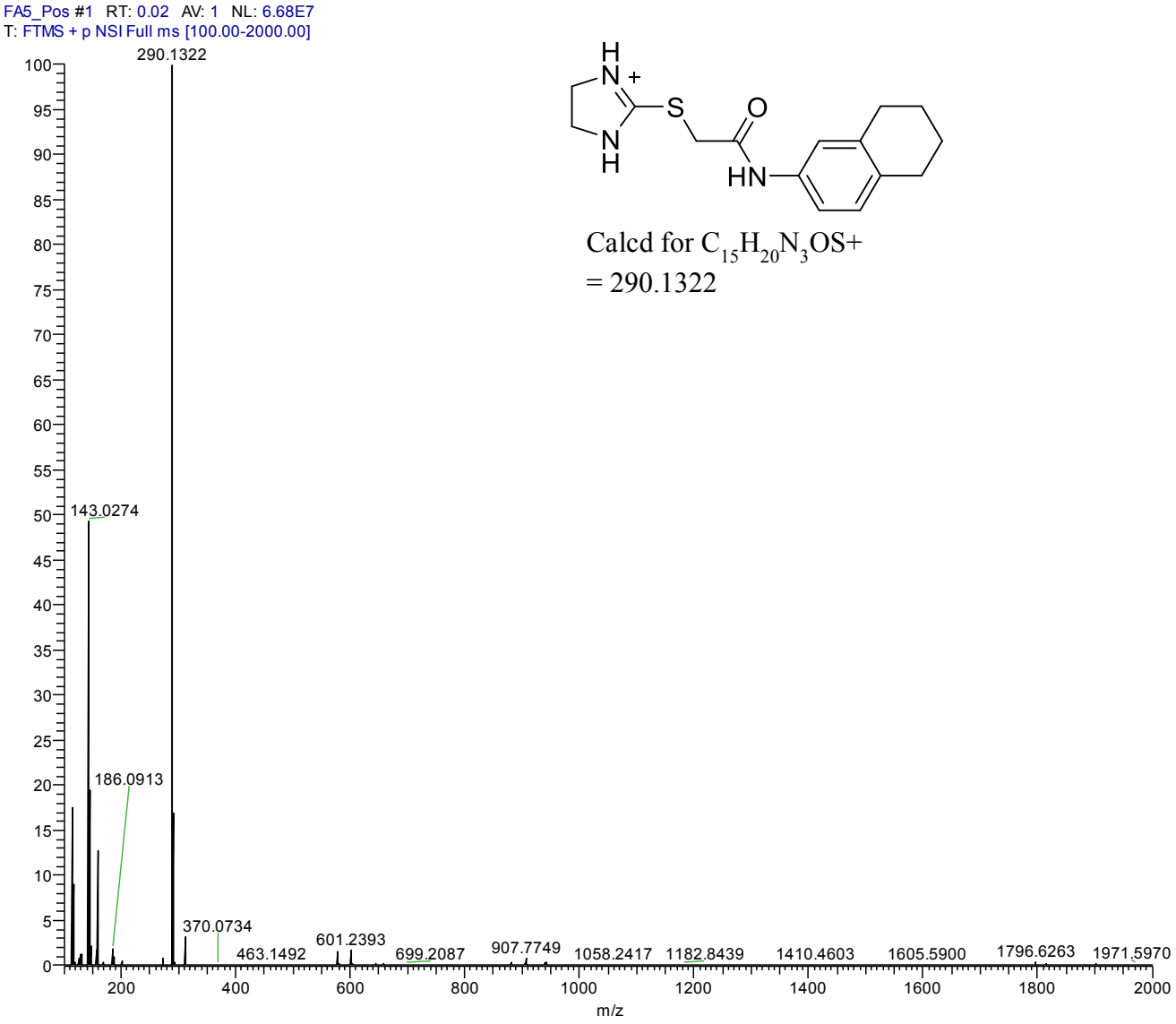


MS Spectrum Graph

Ret.Time:12.267(Scan#:677)
BG Mode:?
Mass Peaks:1109 Base Peak:289.90(14852321) Polarity:Pos Segment1 - Event1



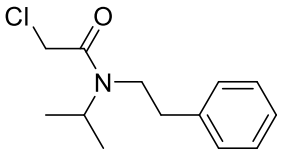
HRMS(ESI) Compound 27



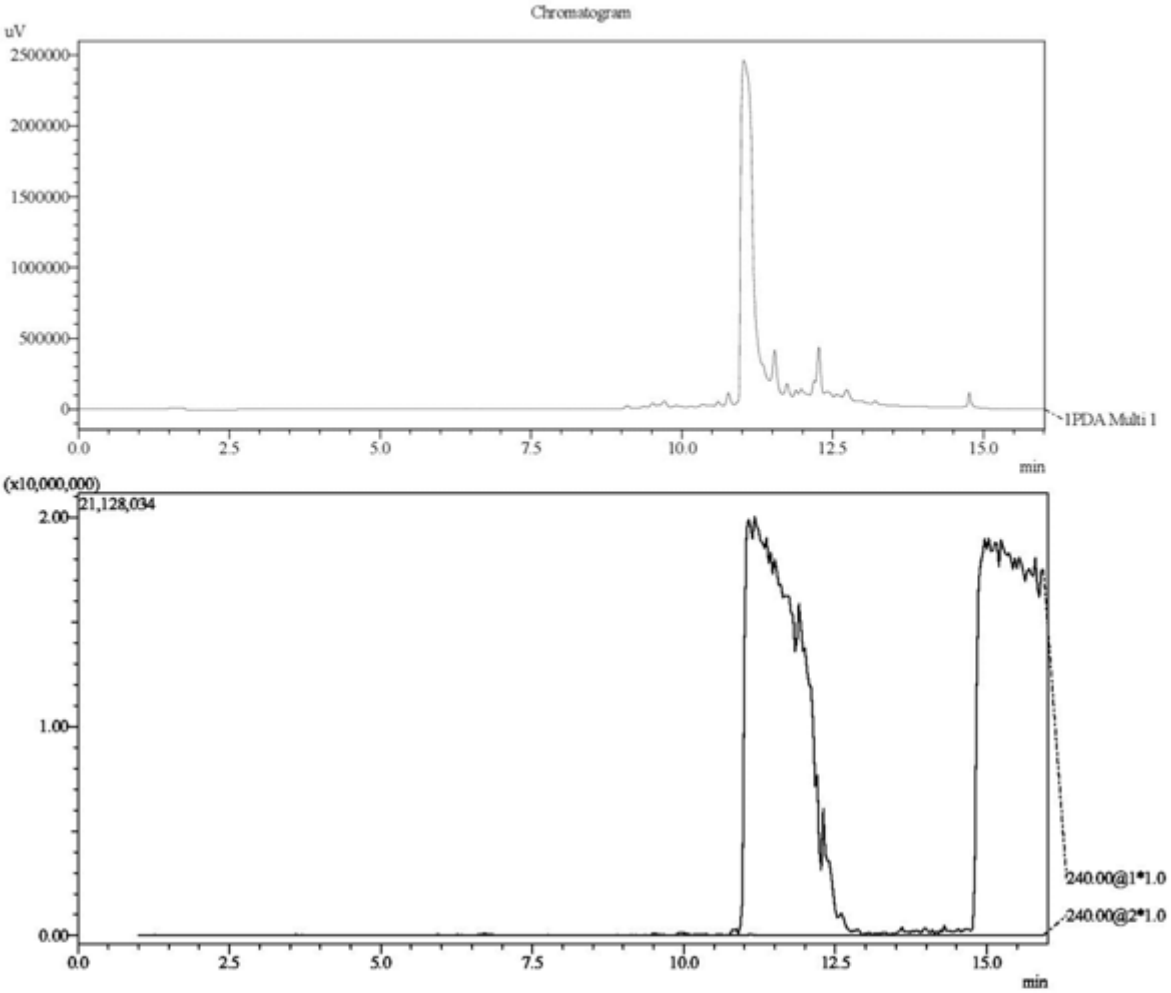
Calcd for $C_{15}H_{20}N_3OS^+$
= 290.1322

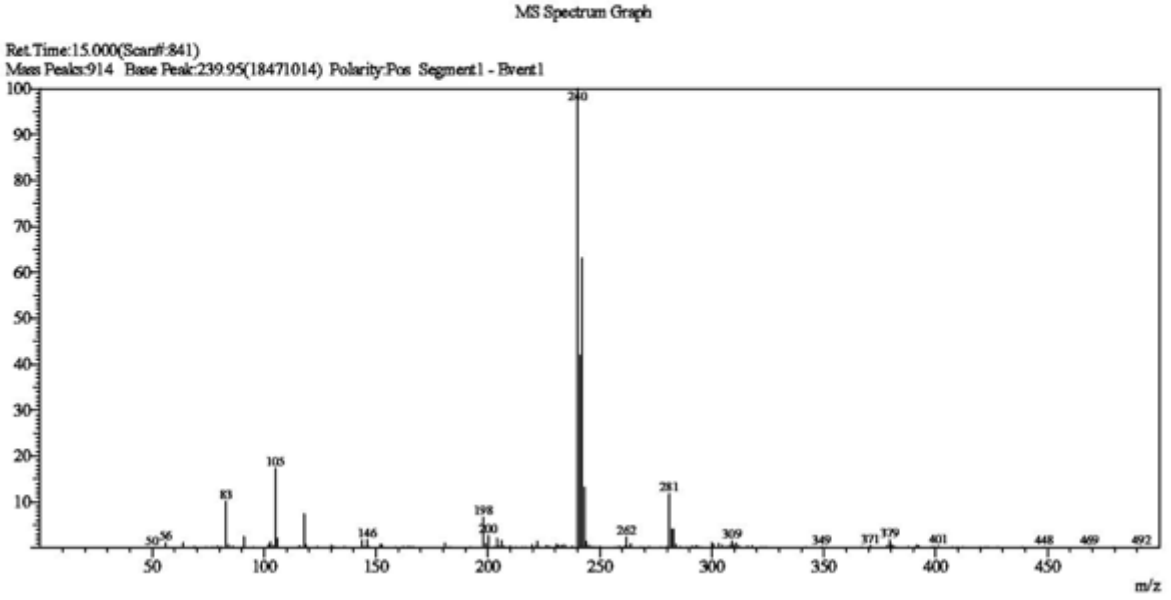
Spectra for Compound 28

LCMS of Compound 28-37



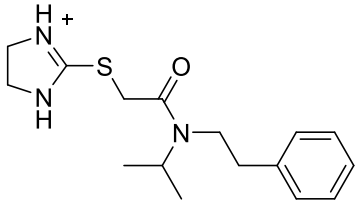
2-chloro-N-isopropyl-N-phenethylacetamide



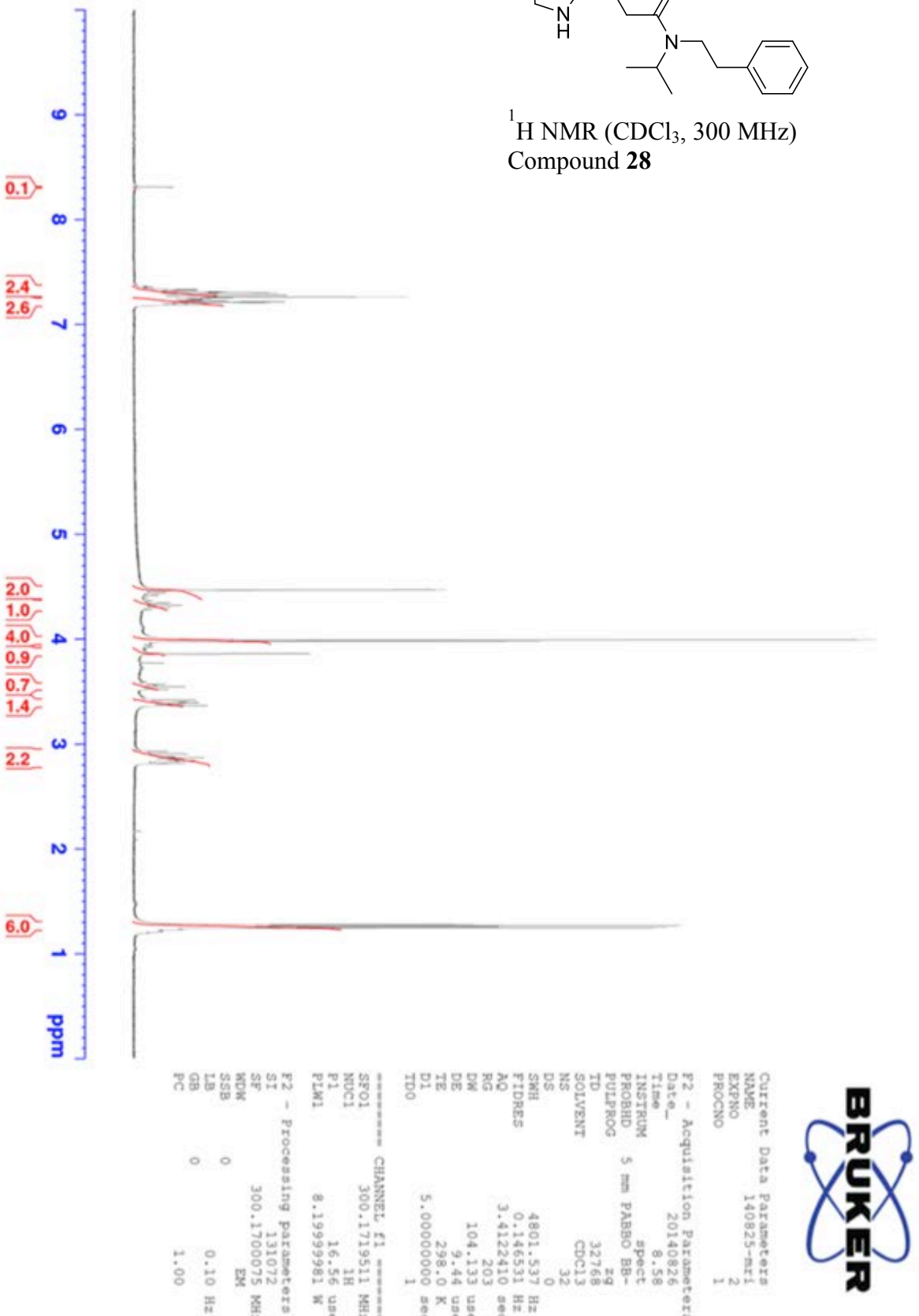


^{1}H NMR Compound 28

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-phenethylacetamide

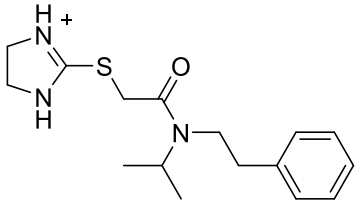


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **28**

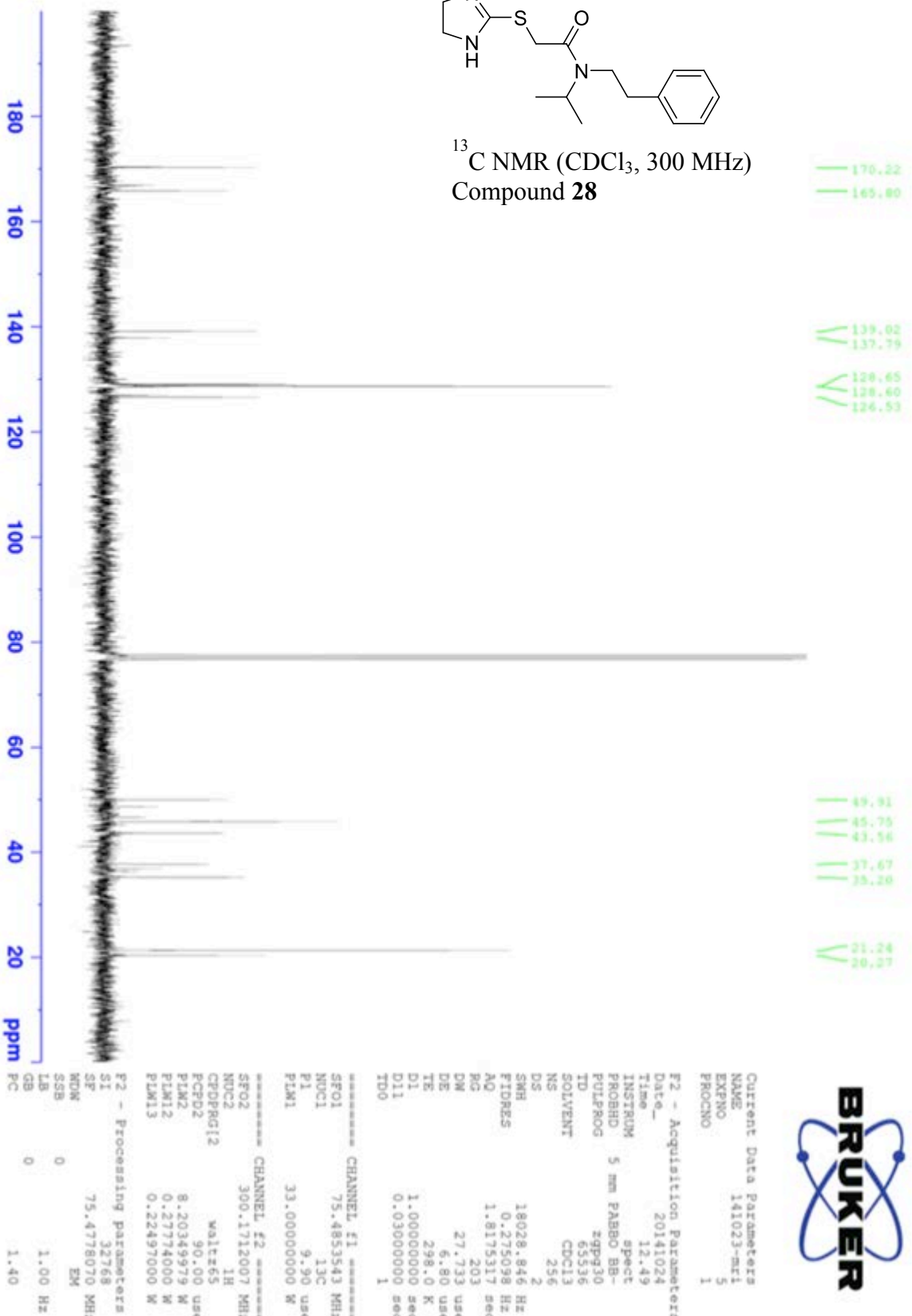


^{13}C NMR Compound 28

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-phenethylacetamide

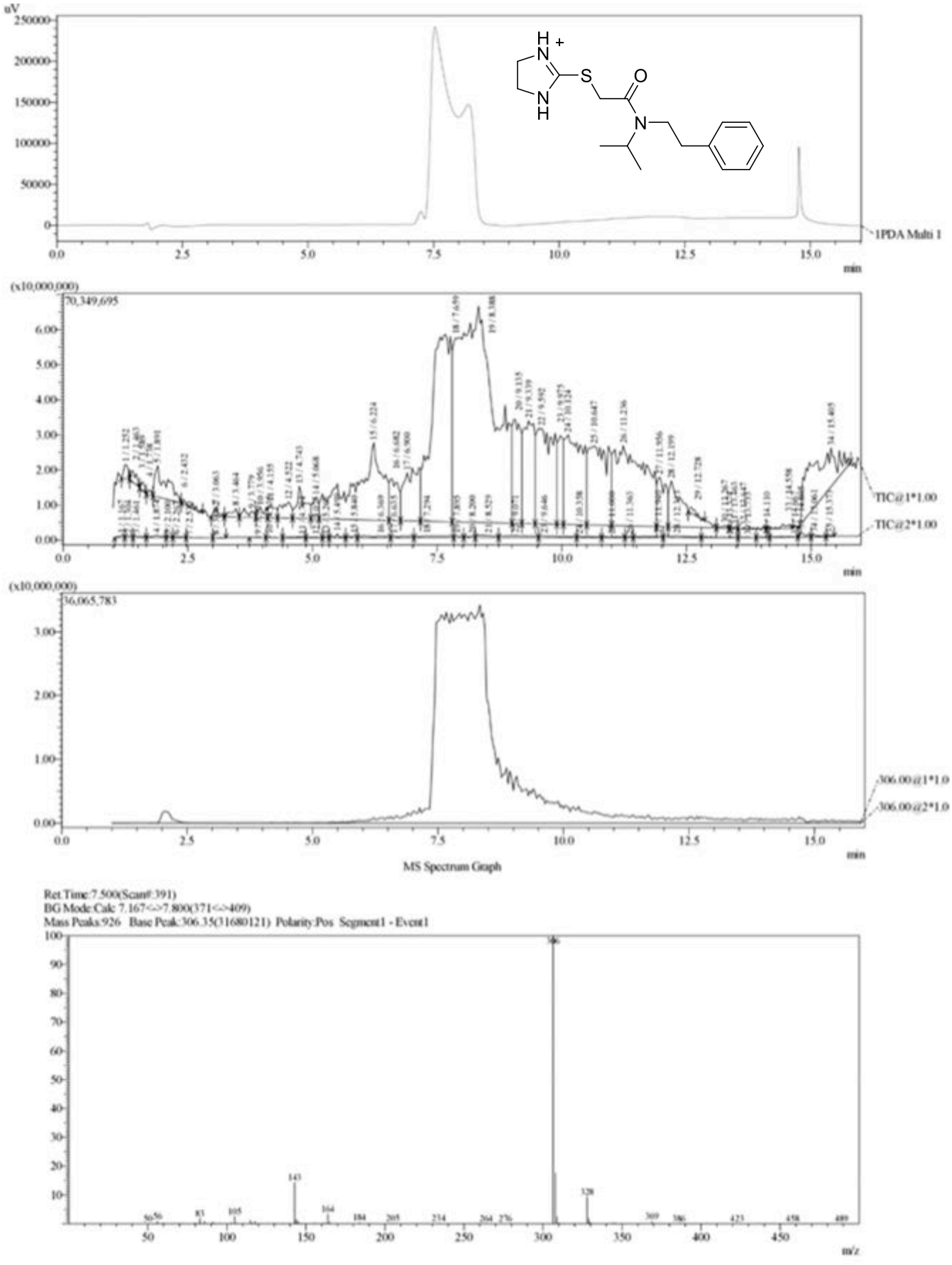


^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **28**



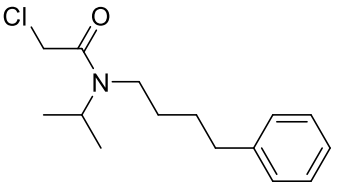
LCMS of Compound 28

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-phenethylacetamide

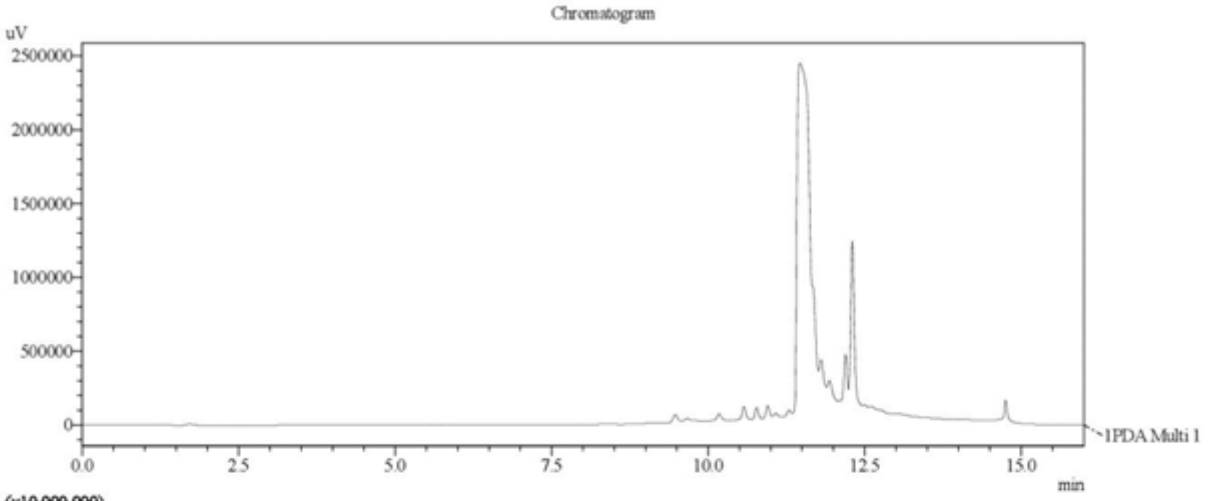


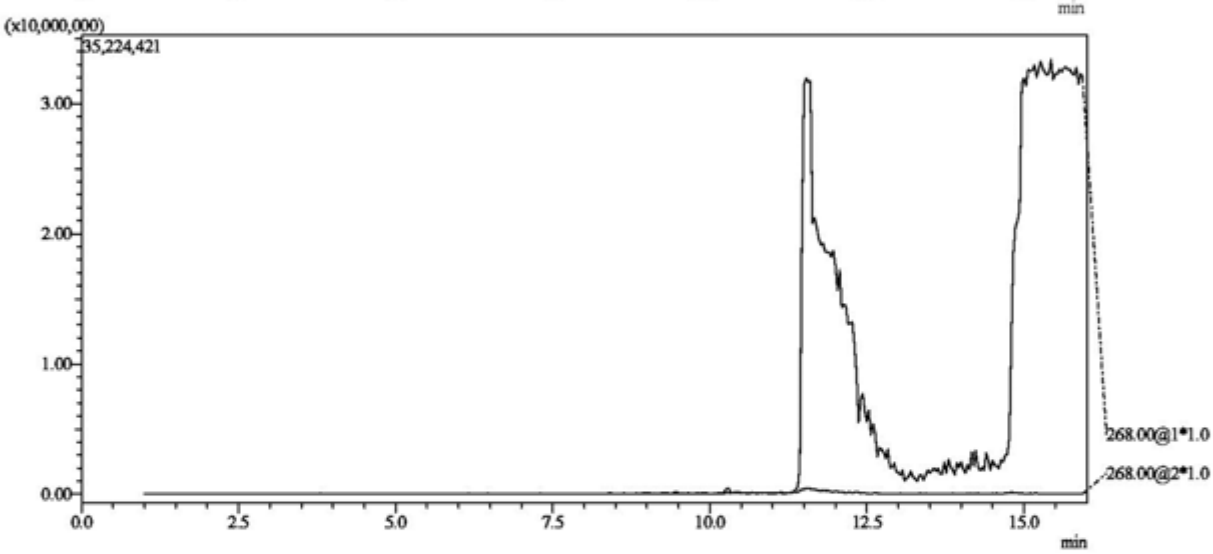
Spectra for Compound 29

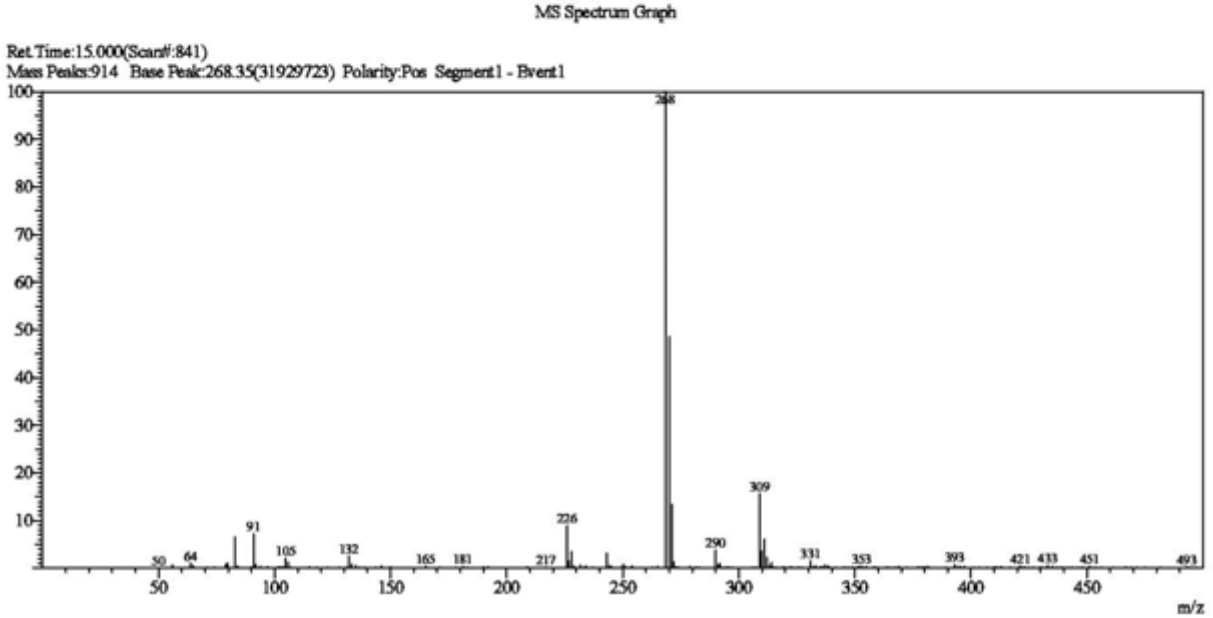
LCMS of Compound 29-37



2-chloro-N-isopropyl-N-(4-phenylbutyl)acetamide

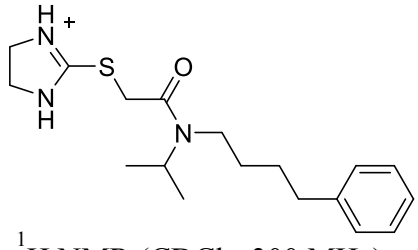




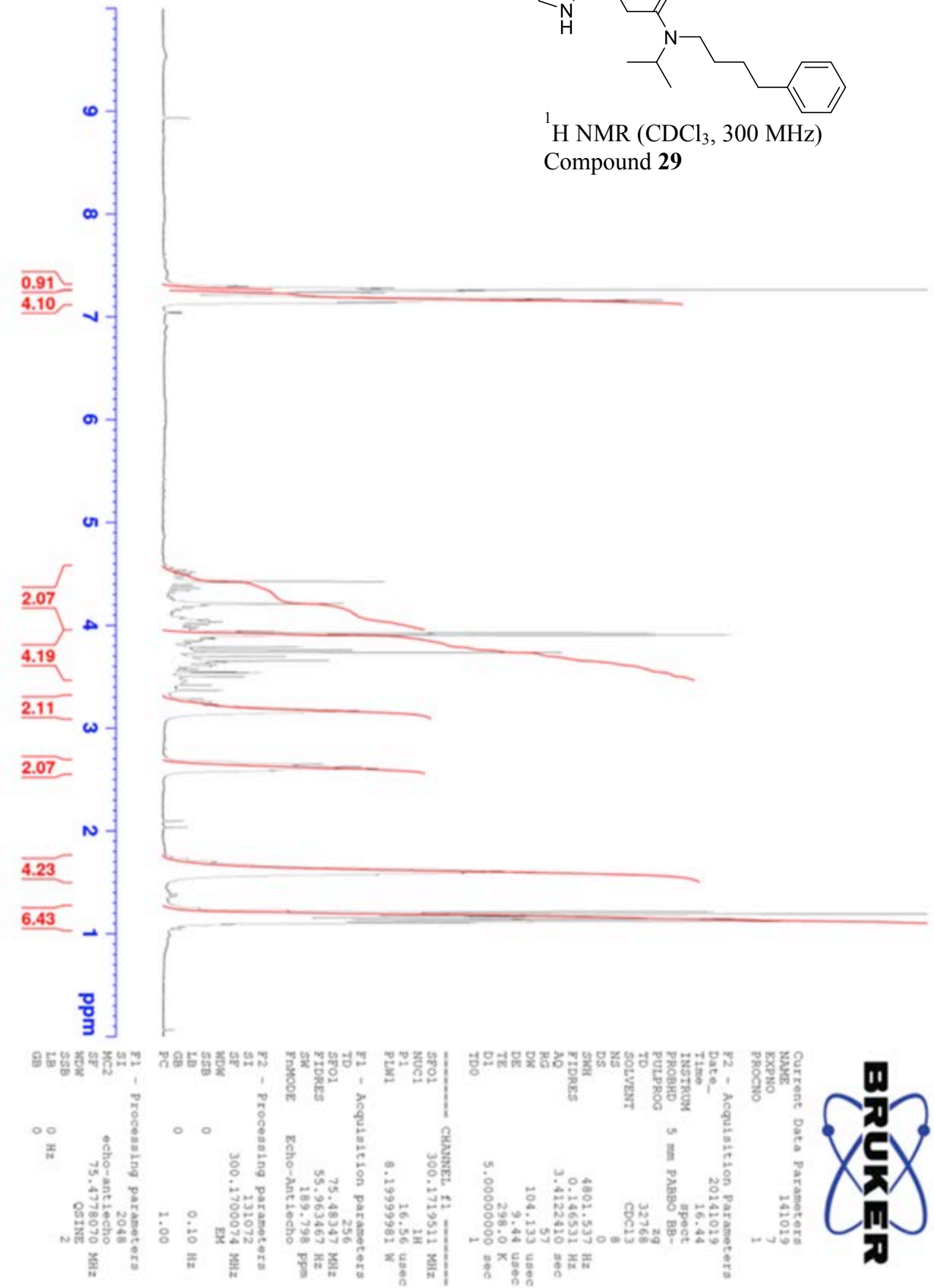


^{1}H NMR Compound 29

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-(4-phenylbutyl)acetamide

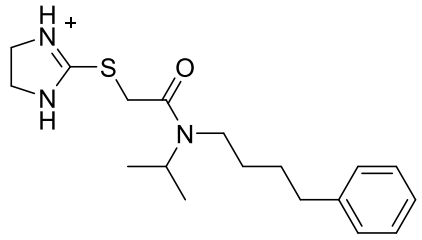


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **29**

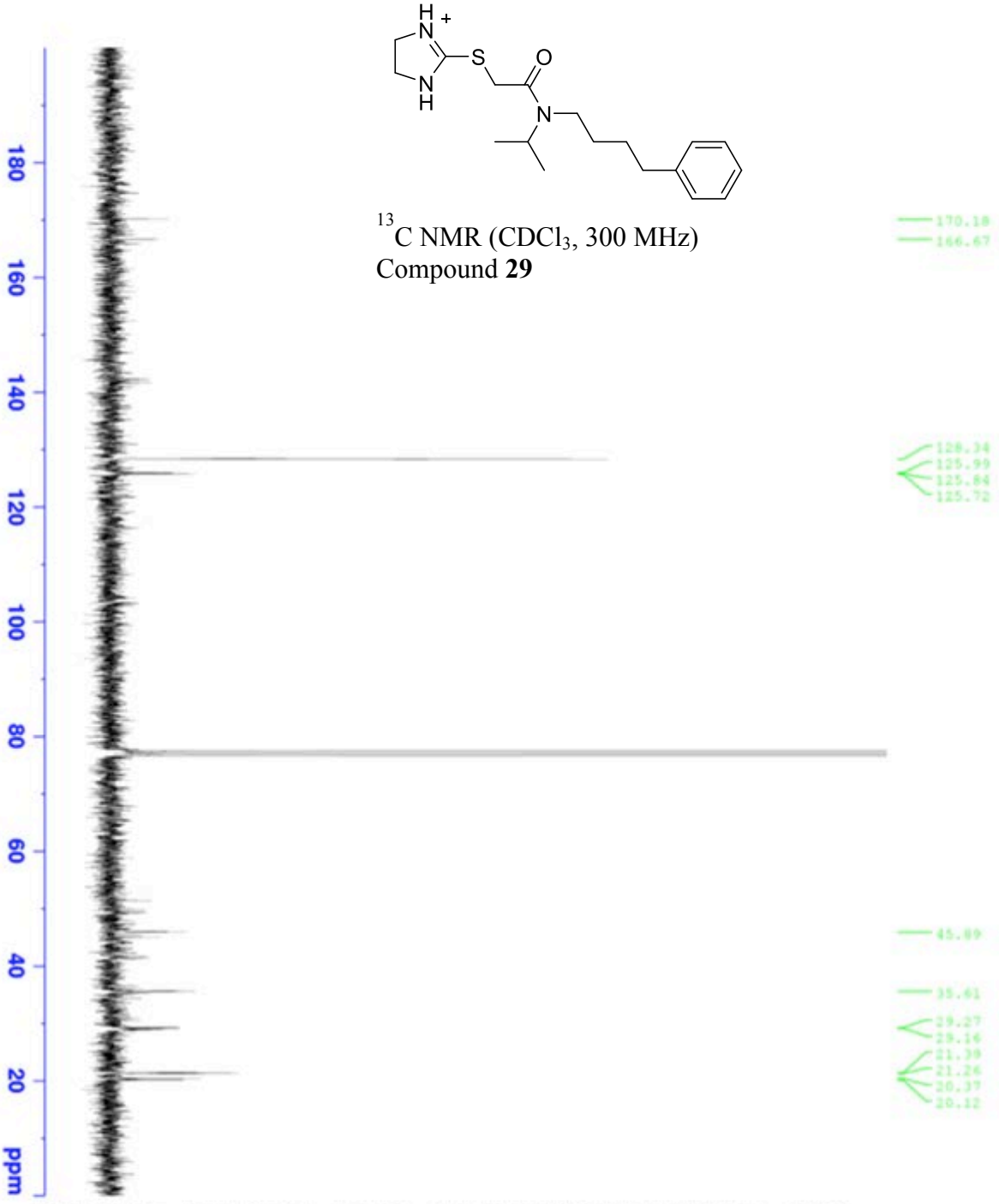


^{13}C NMR Compound 29

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-(4-phenylbutyl)acetamide

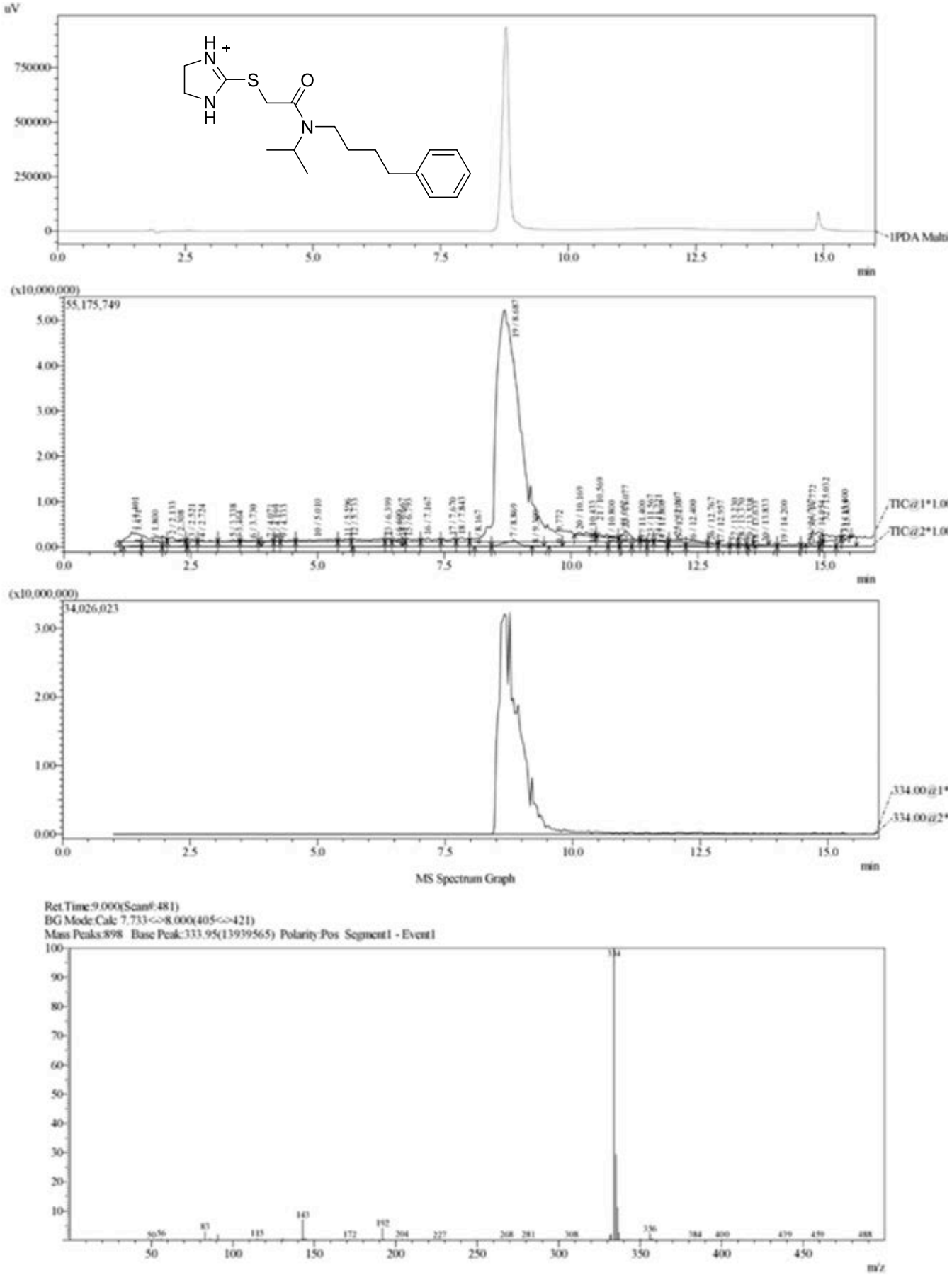


^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **29**



LCMS Compound 29

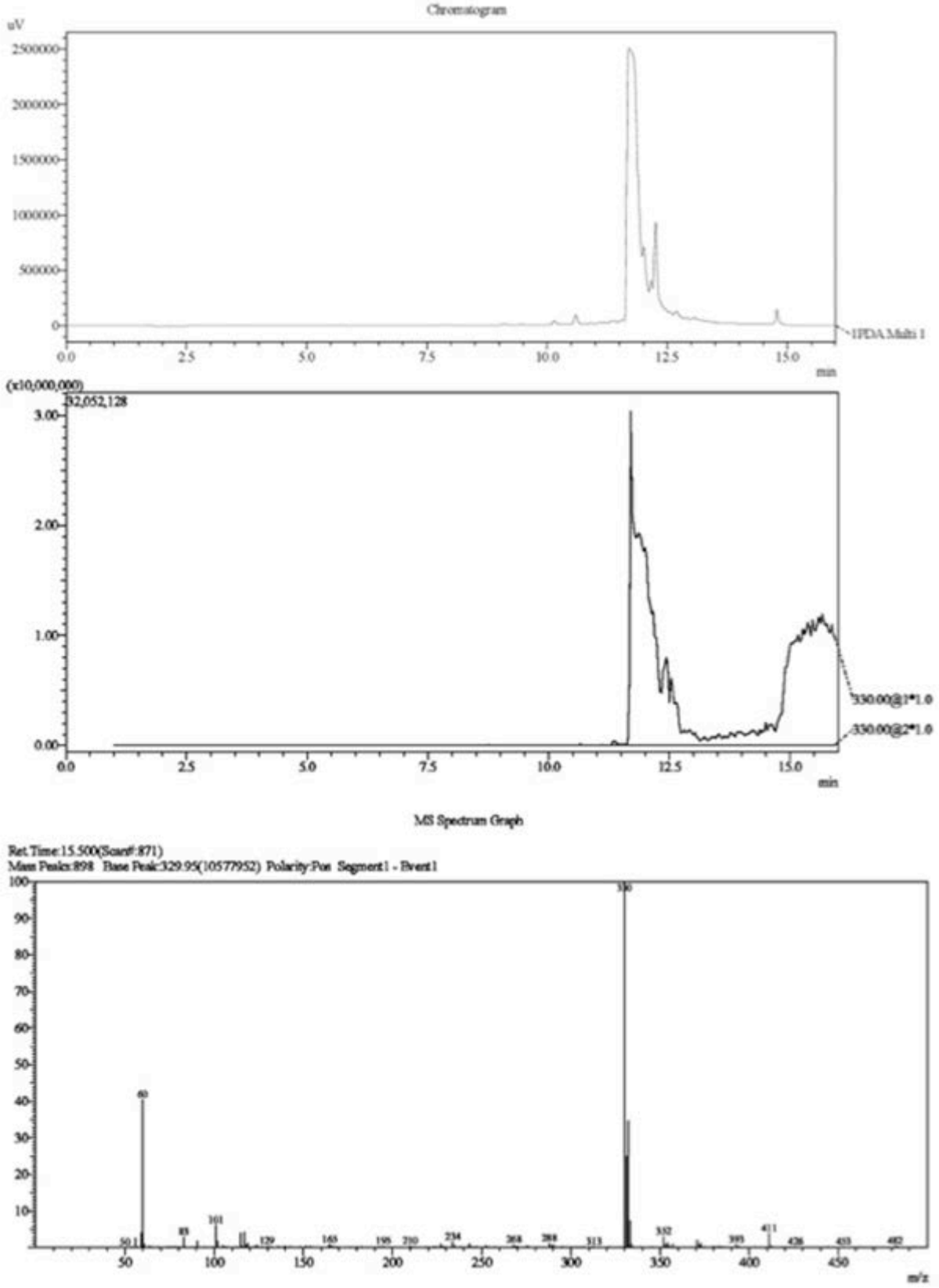
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-isopropyl-N-(4-phenylbutyl)acetamide



Spectra of Compound 30

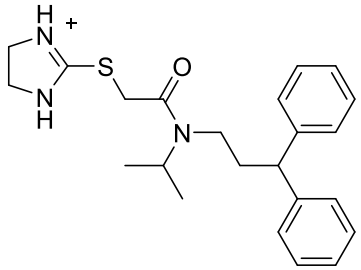
LCMS of Compound 30-37

2-chloro-N-(3,3-diphenylpropyl)-N-isopropylacetamide

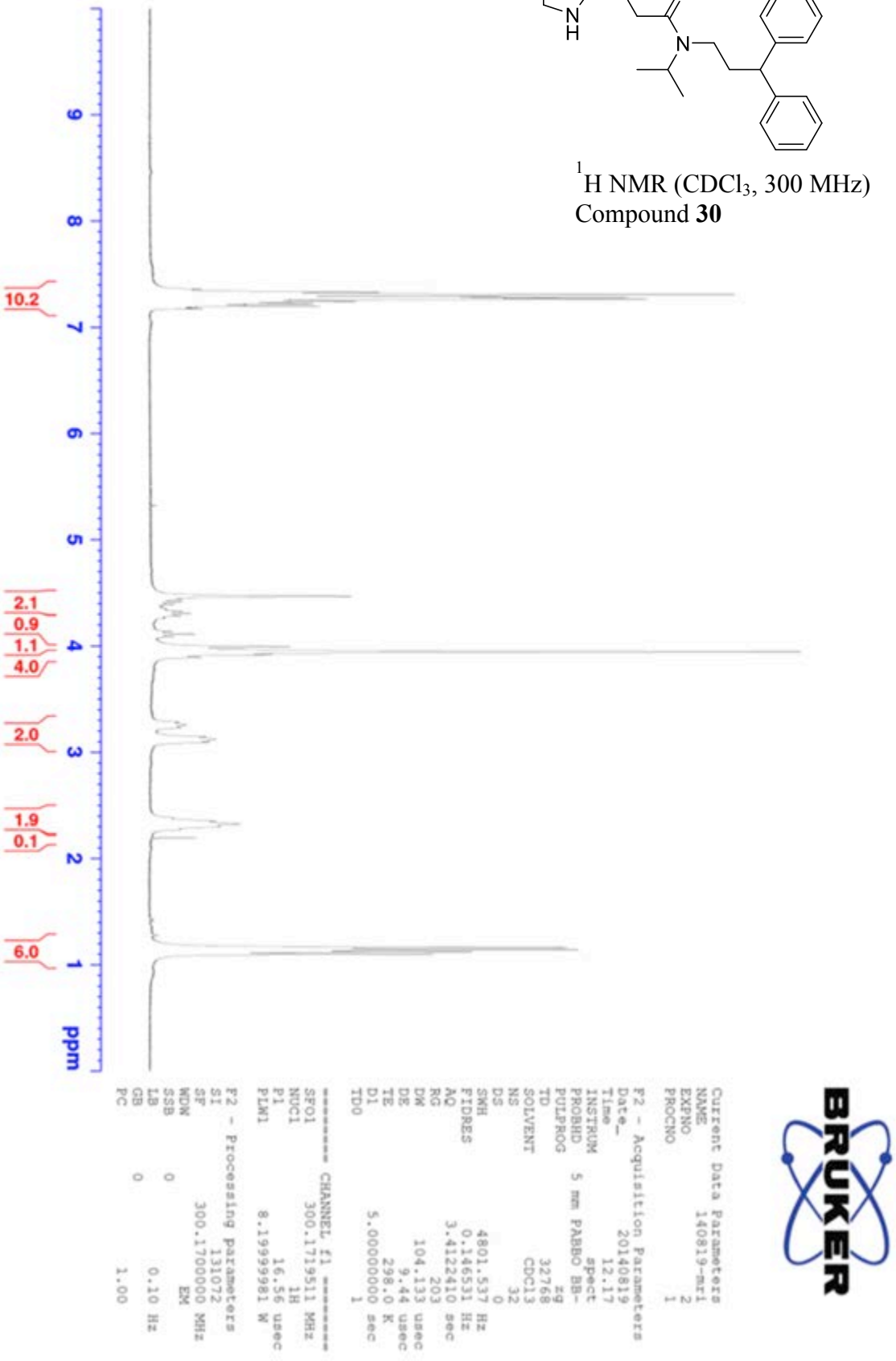


^{1}H NMR Compound 30

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(3,3-diphenylpropyl)-N-isopropylacetamide

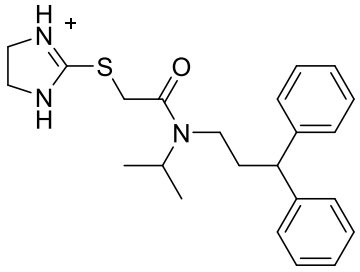


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **30**

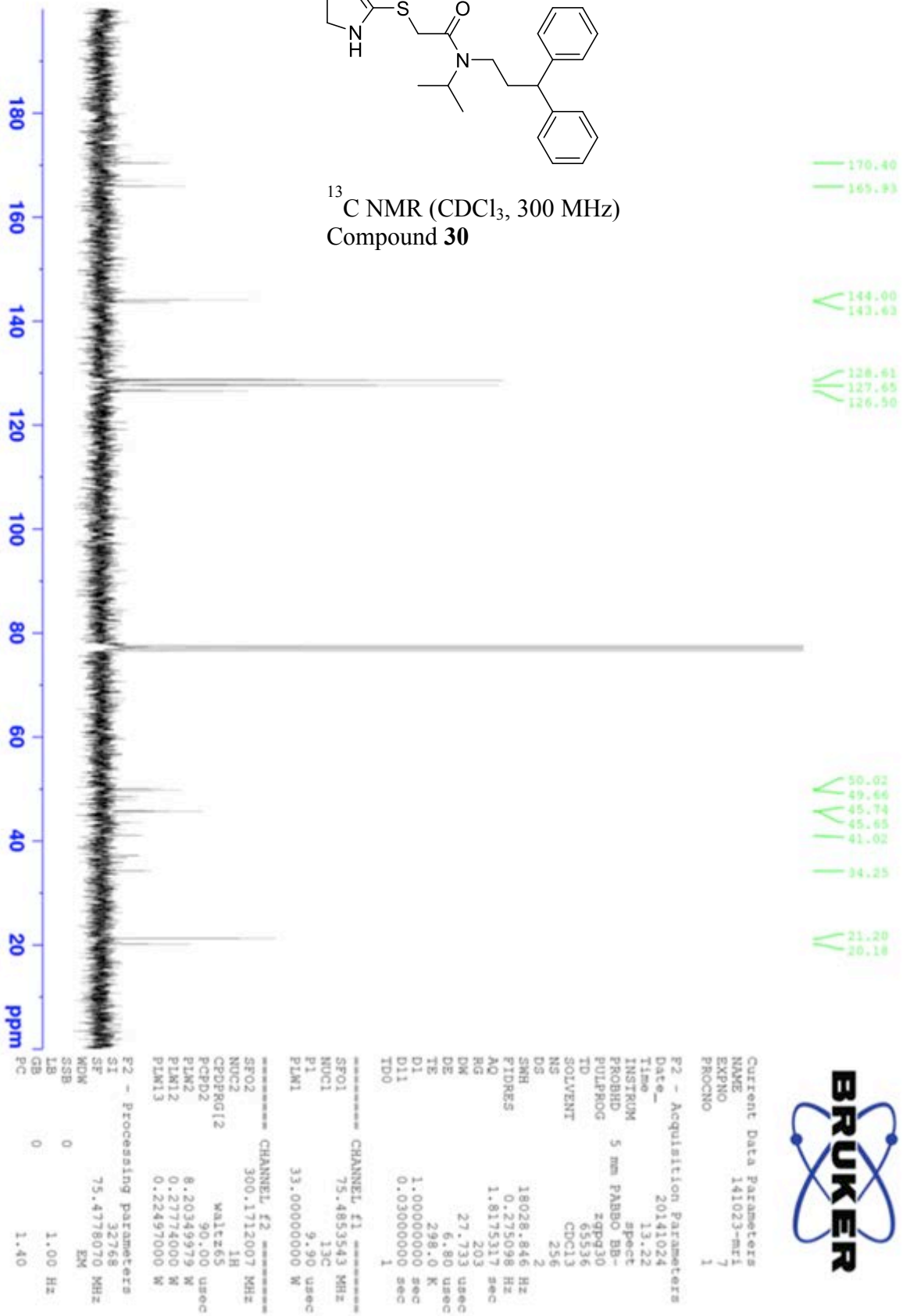


^{13}C NMR Compound 30

2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(3,3-diphenylpropyl)-N-isopropylacetamide

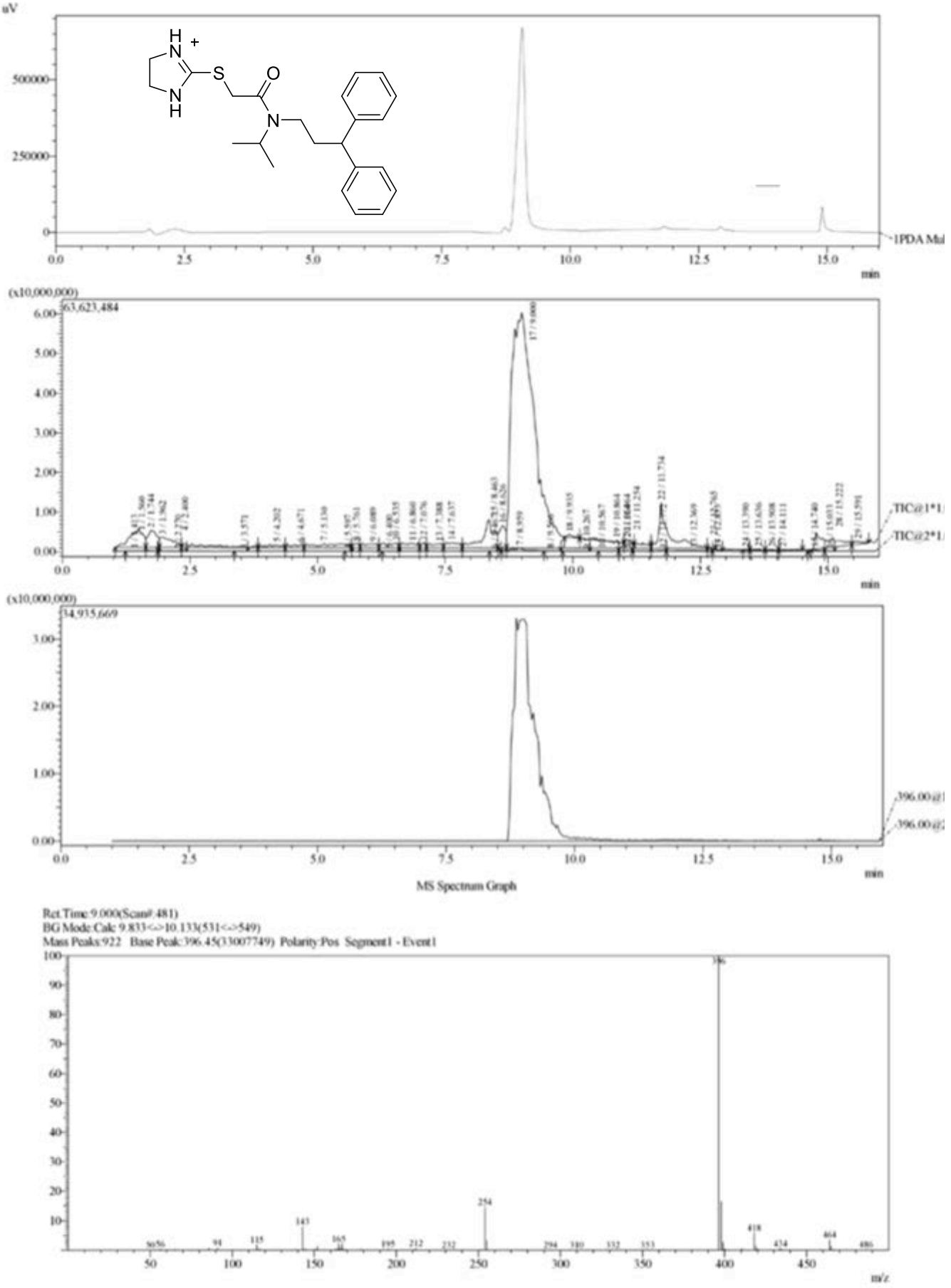


^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **30**



LCMS of Compound 30

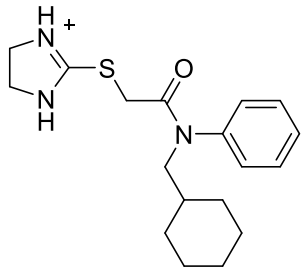
2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(3,3-diphenylpropyl)-N-isopropylacetamide

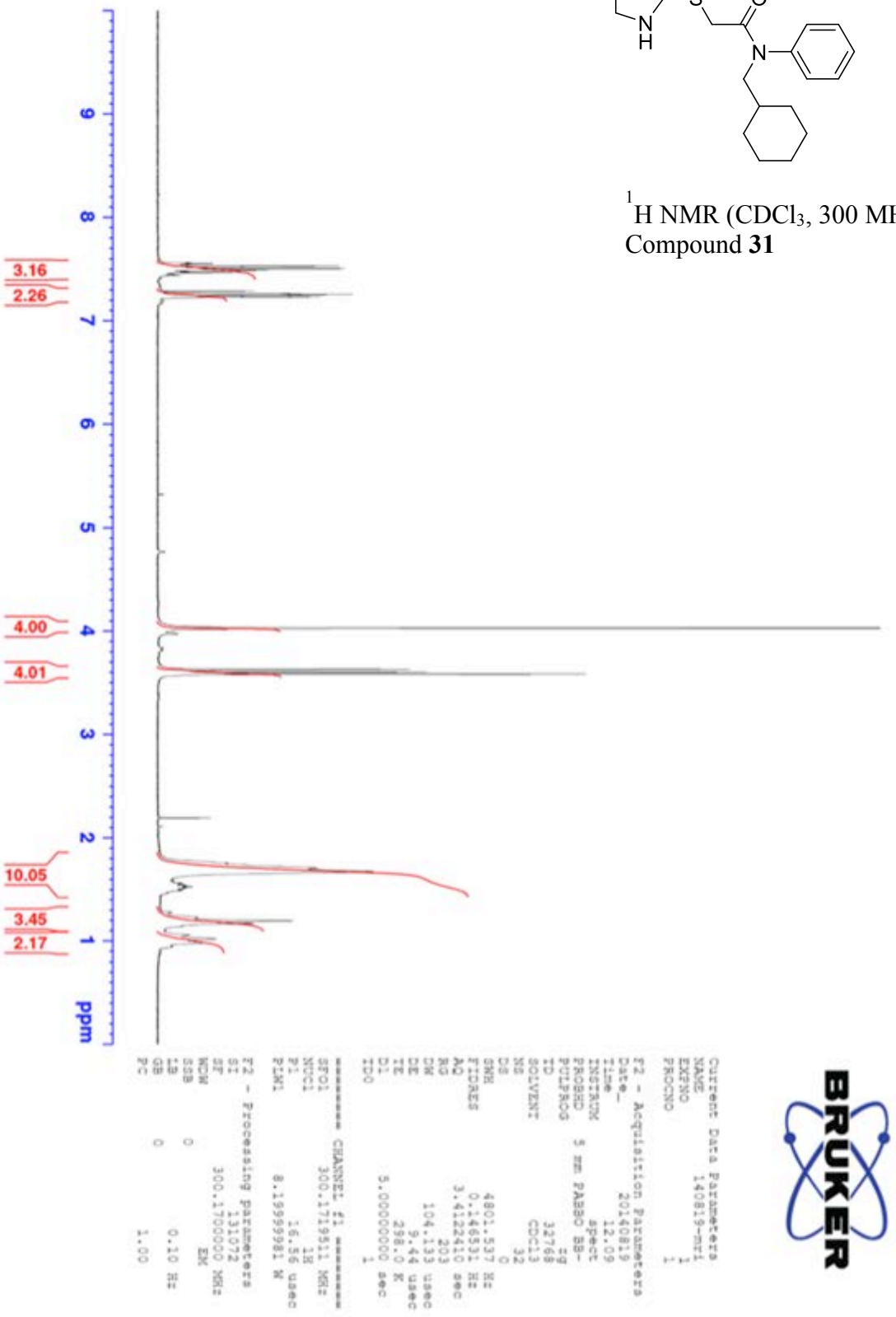


Spectra for Compound 31

^{1}H NMR Compound 31

N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenylacetamide

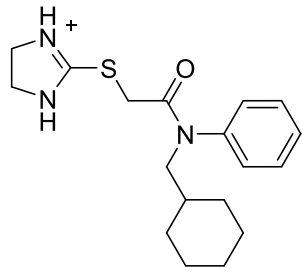


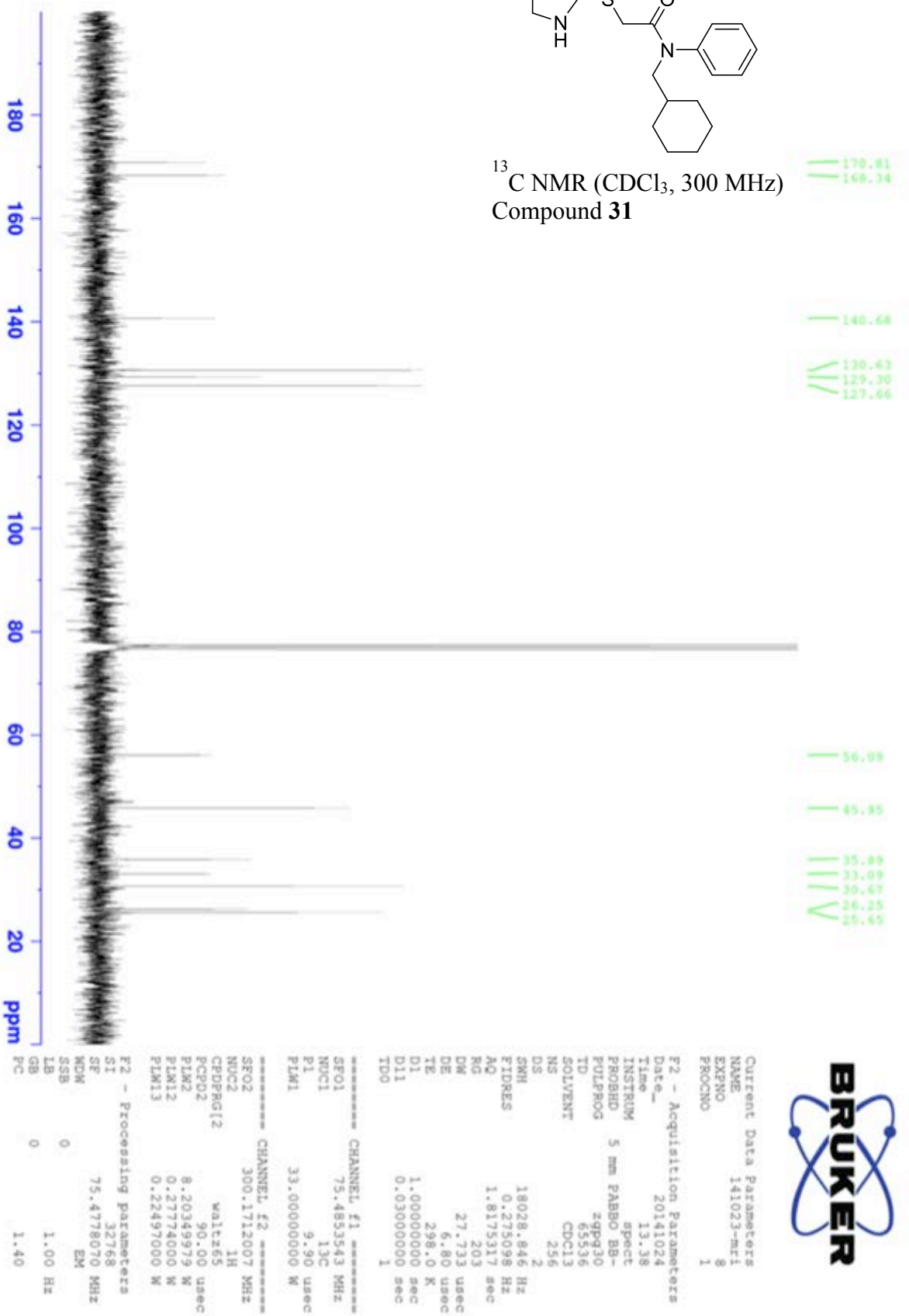


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **31**

^{13}C NMR Compound 31

N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenylacetamide

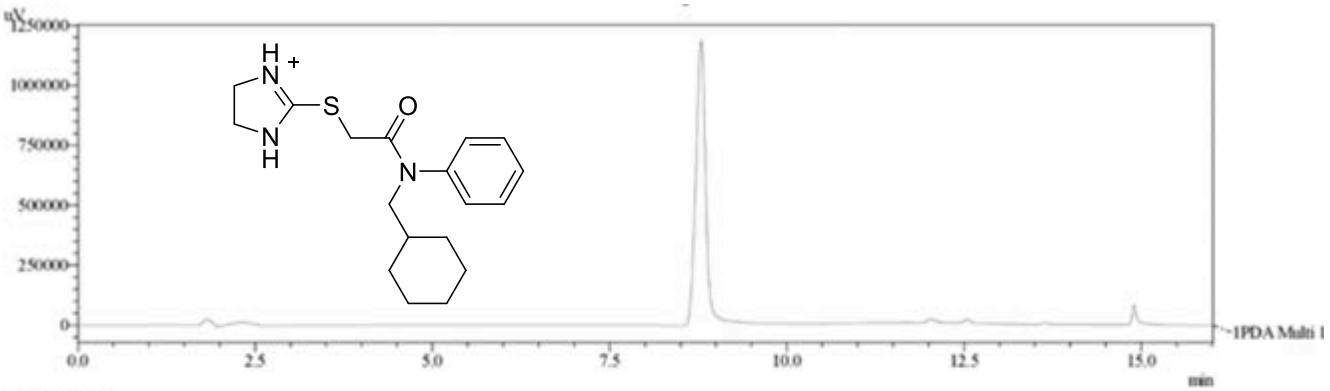


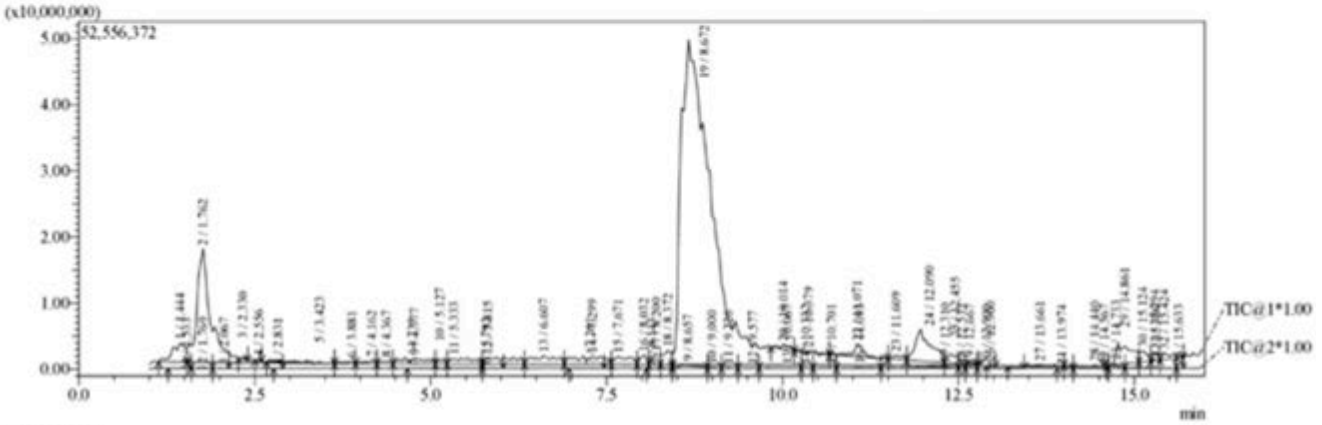


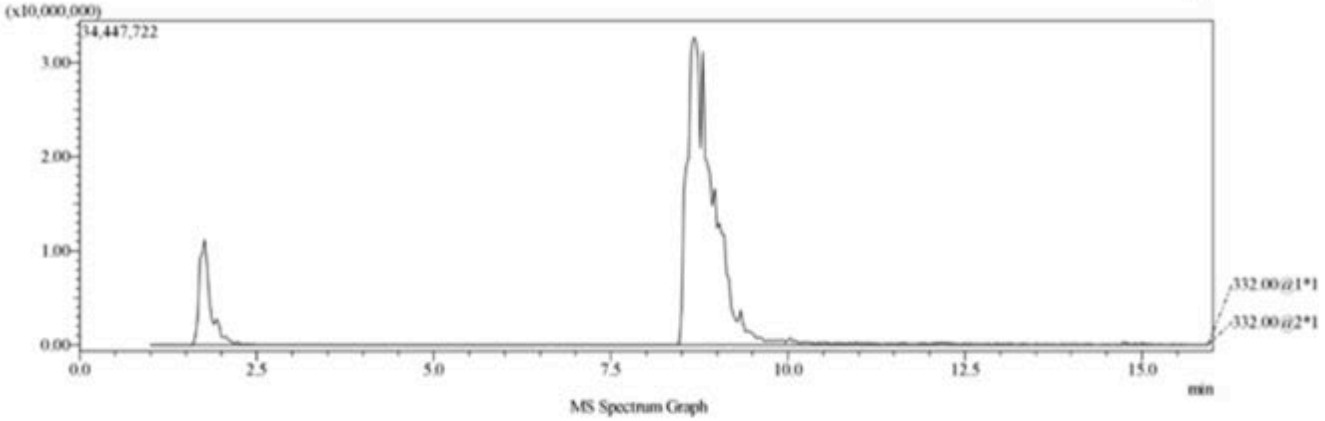
^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **31**

LCMS Compound 31

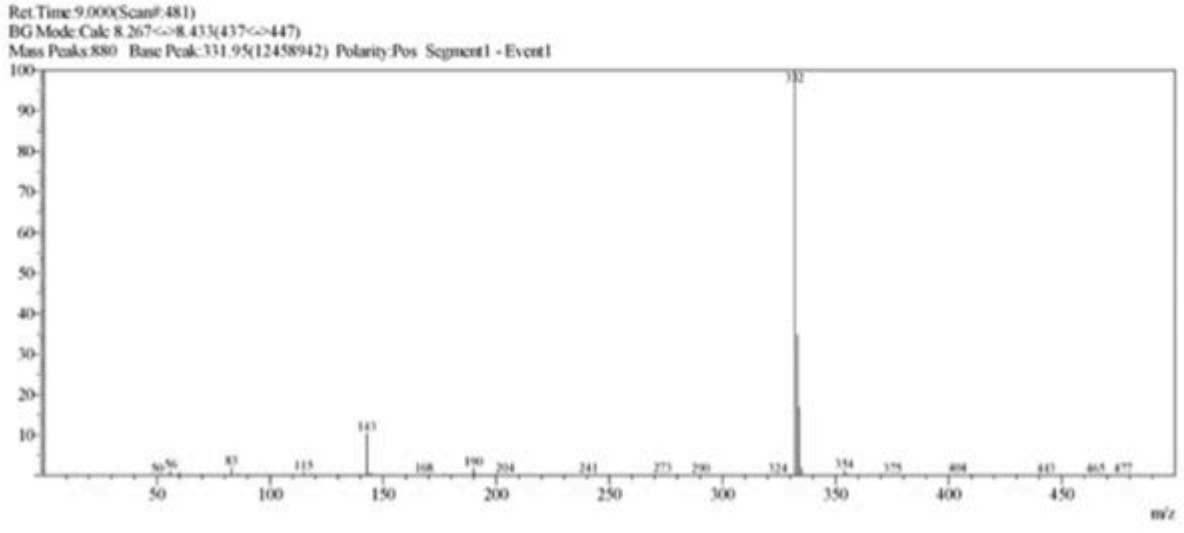
N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenylacetamide



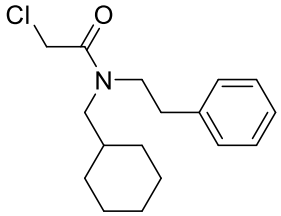




MS Spectrum Graph

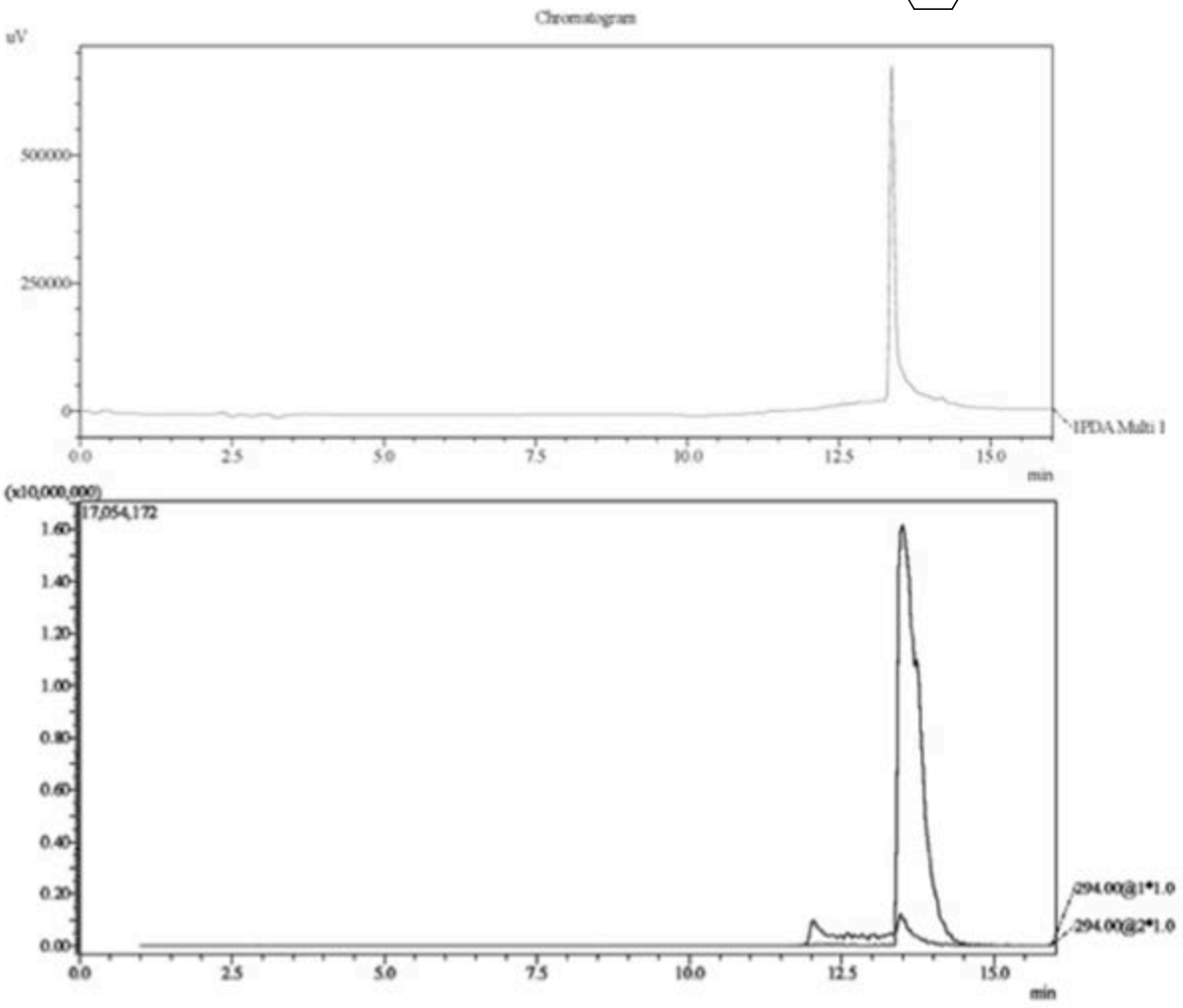


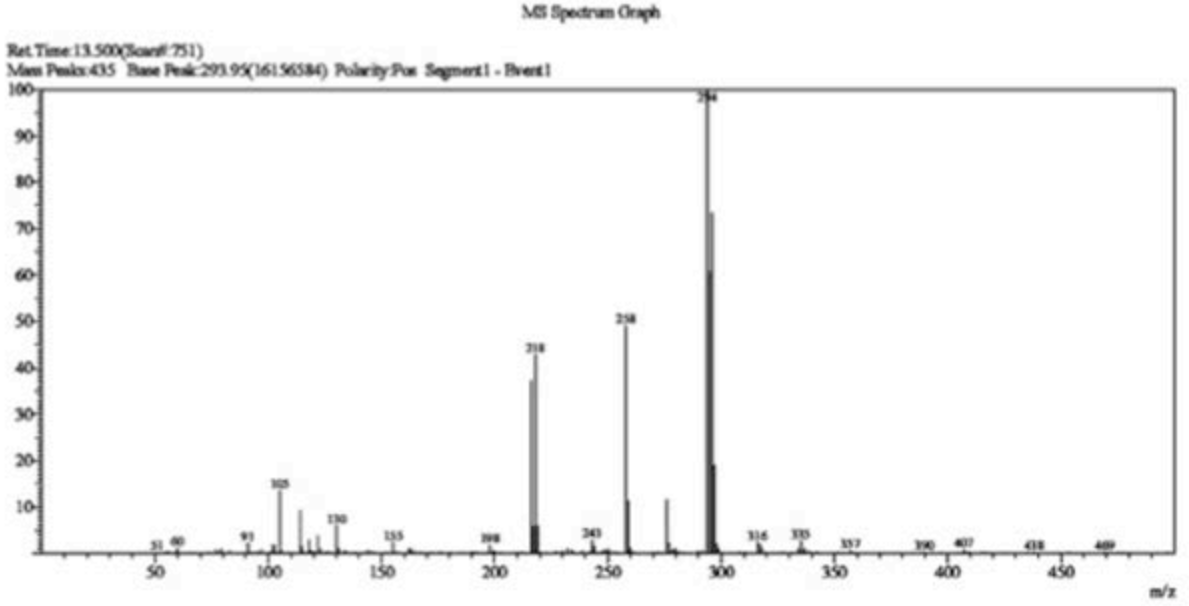
Spectra for Compound 32



LCMS of Compound 32-37

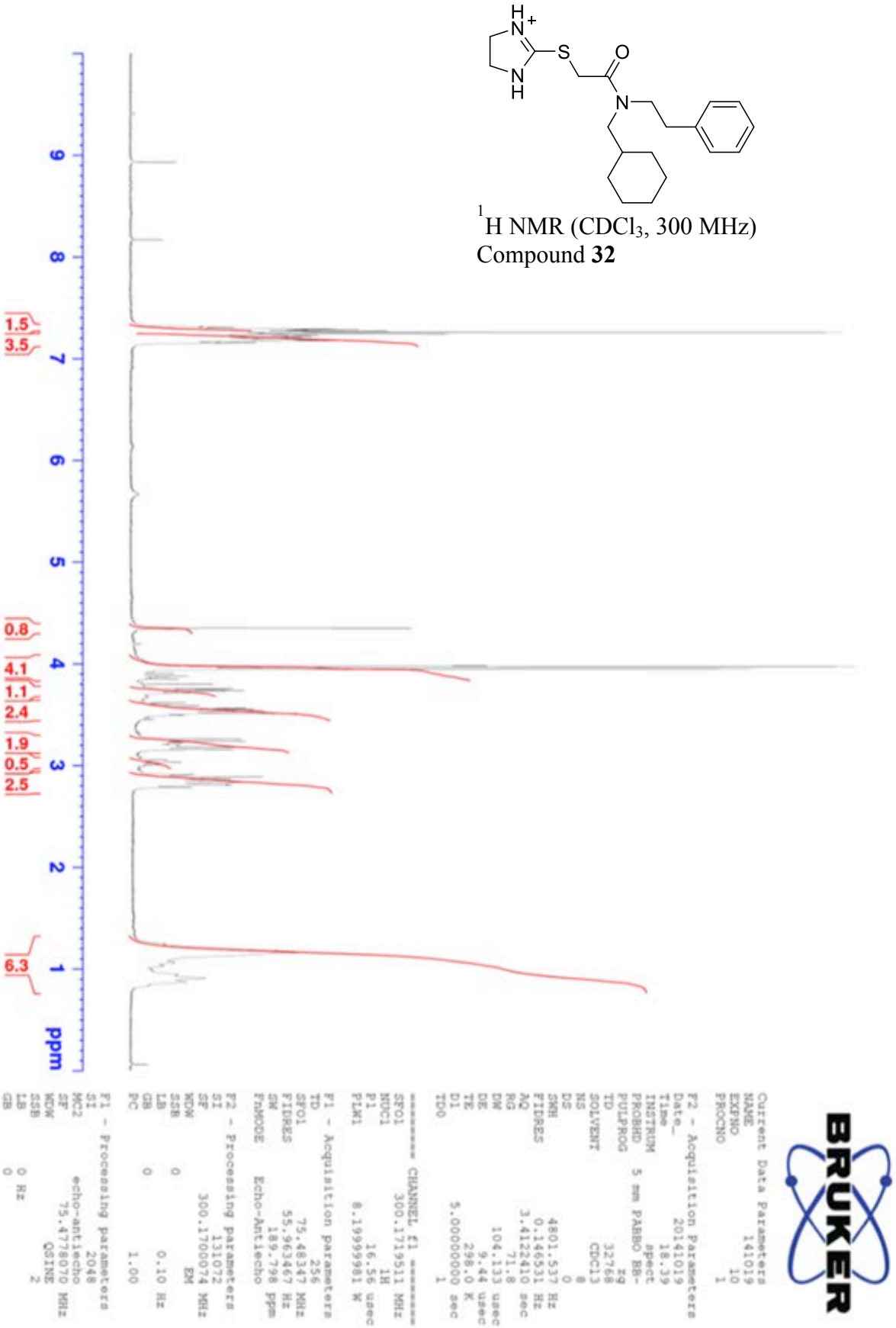
2-chloro-N-(cyclohexylmethyl)-N-phenethylacetamide





^{1}H NMR Compound 32

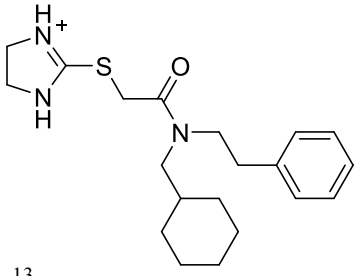
N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenethylacetamide



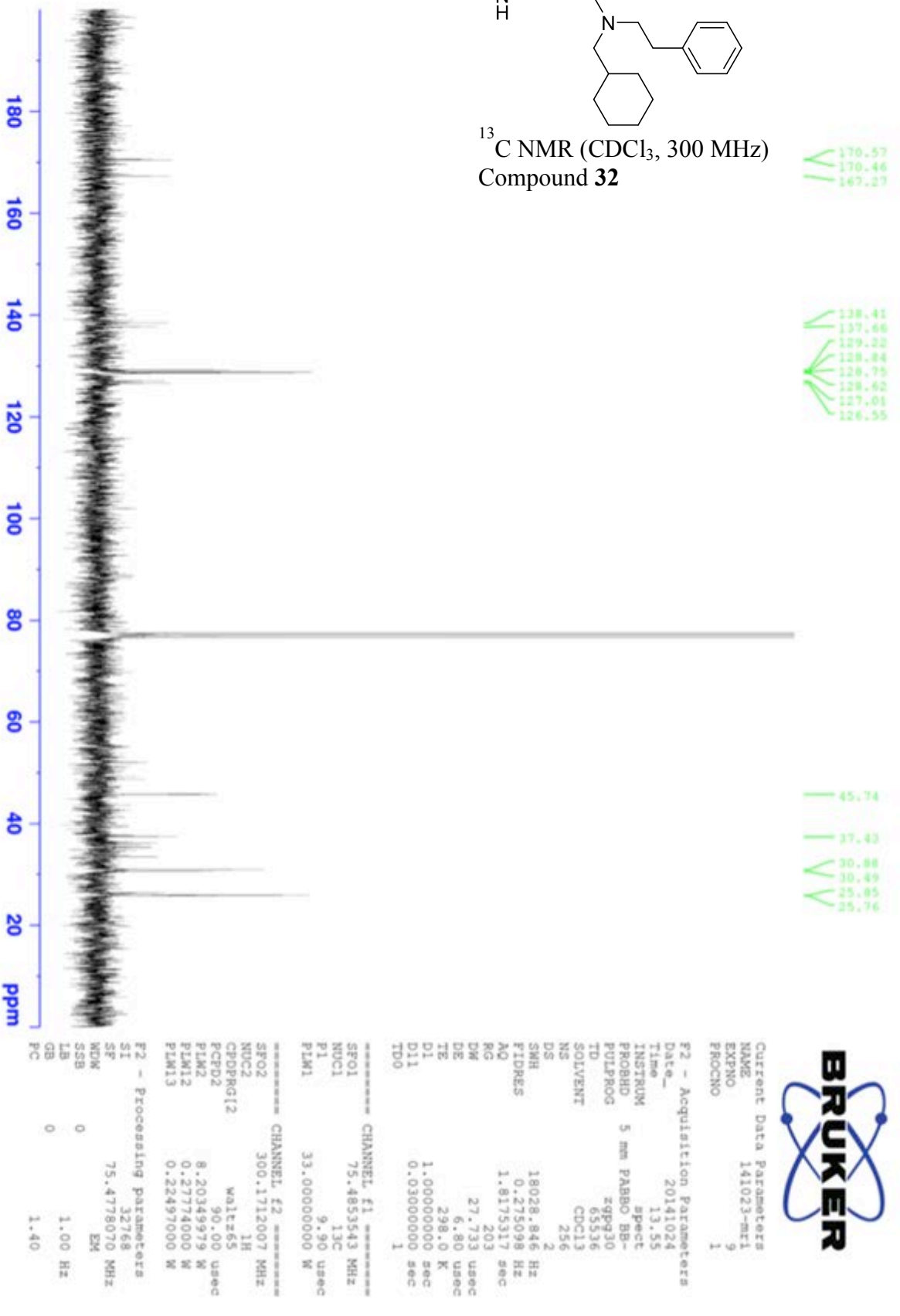
^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **32**

^{13}C NMR Compound 32

N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenethylacetamide

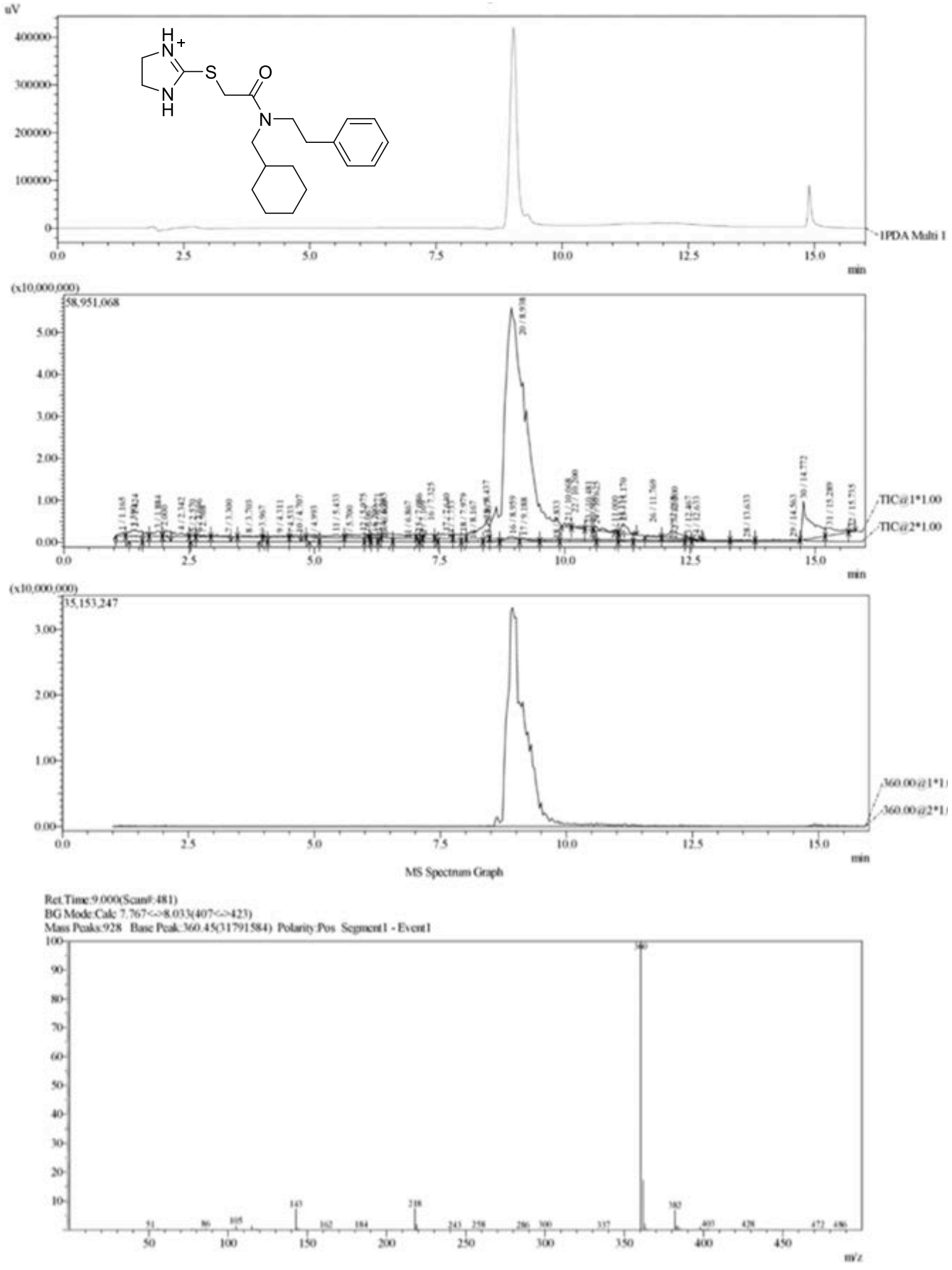


^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **32**



LCMS Compound 32

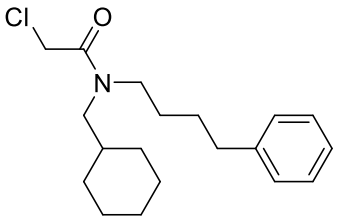
N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-phenethylacetamide

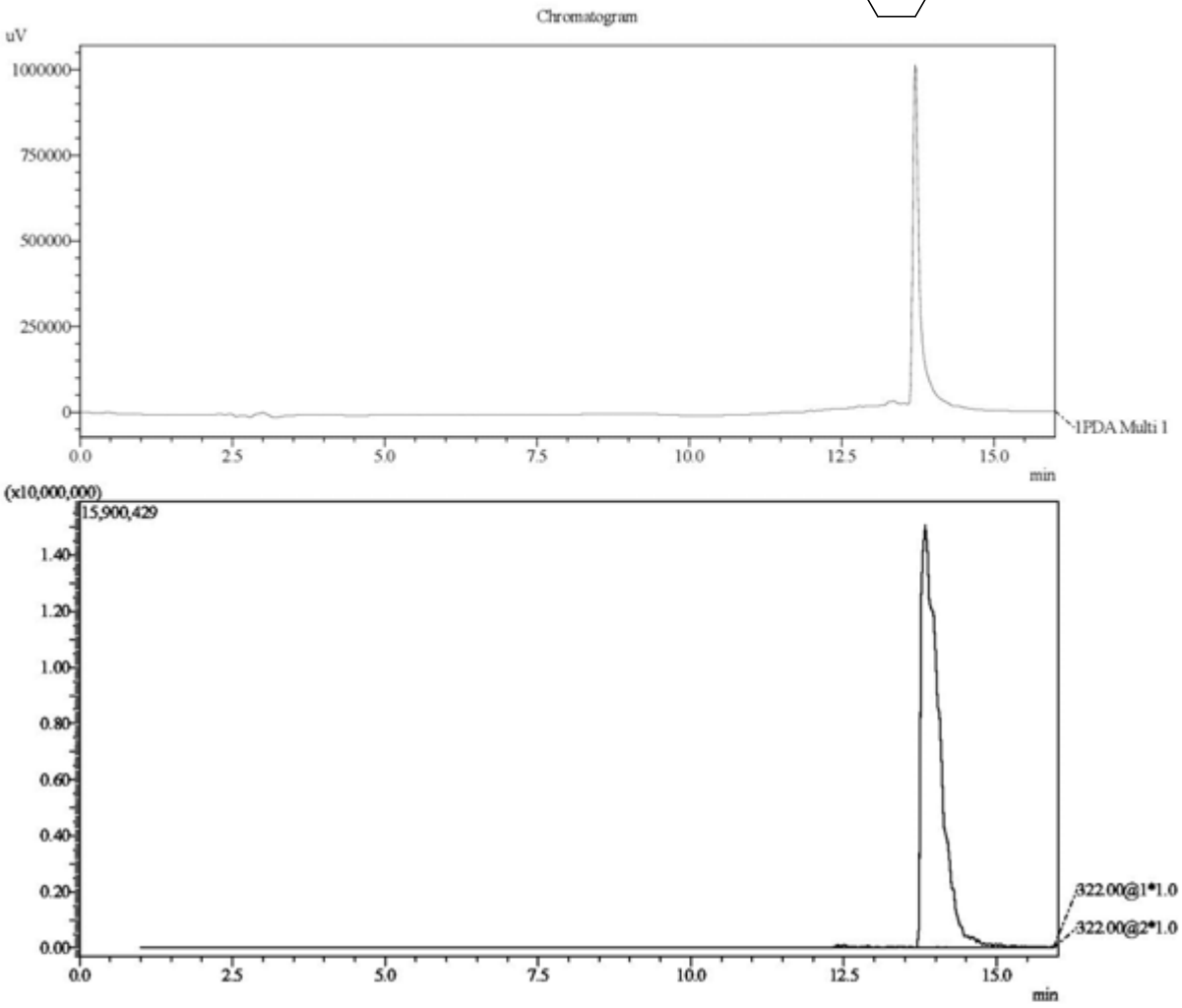


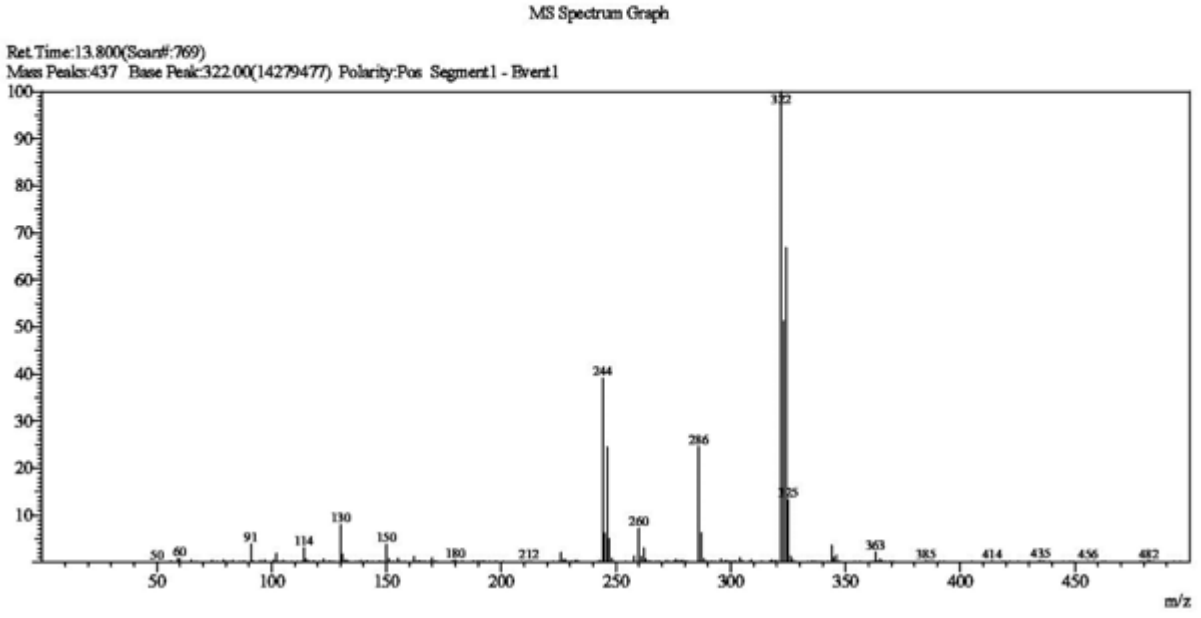
Spectra of Compound 33

LCMS of Compound 33-37

2-chloro-N-(cyclohexylmethyl)-N-(4-phenylbutyl)acetamide

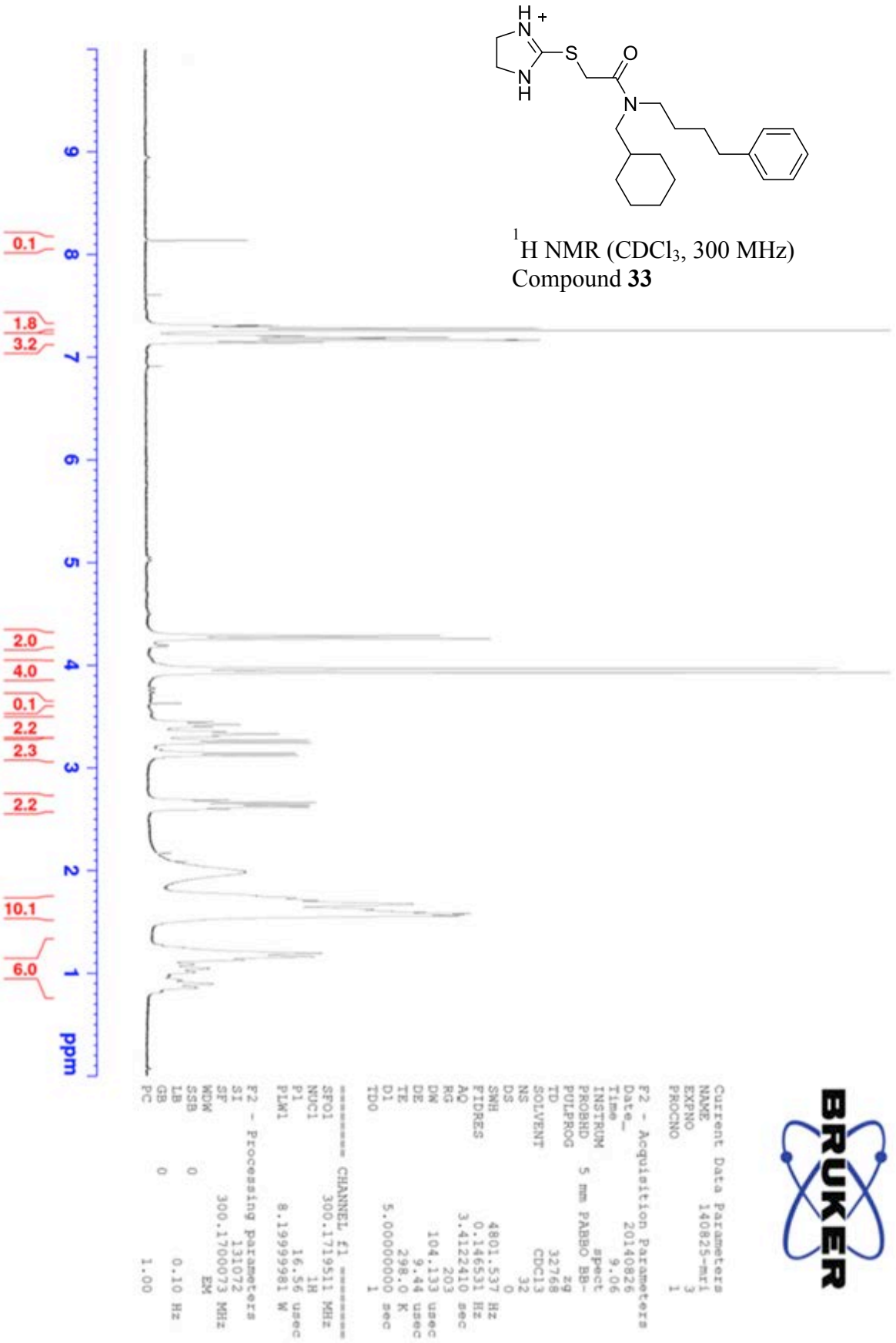






^{1}H NMR Compound 33

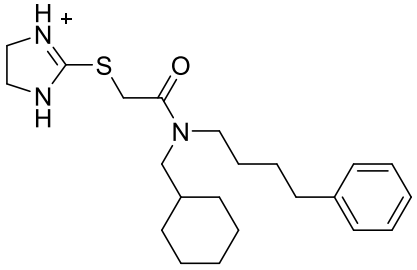
N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(4-phenylbutyl)acetamide



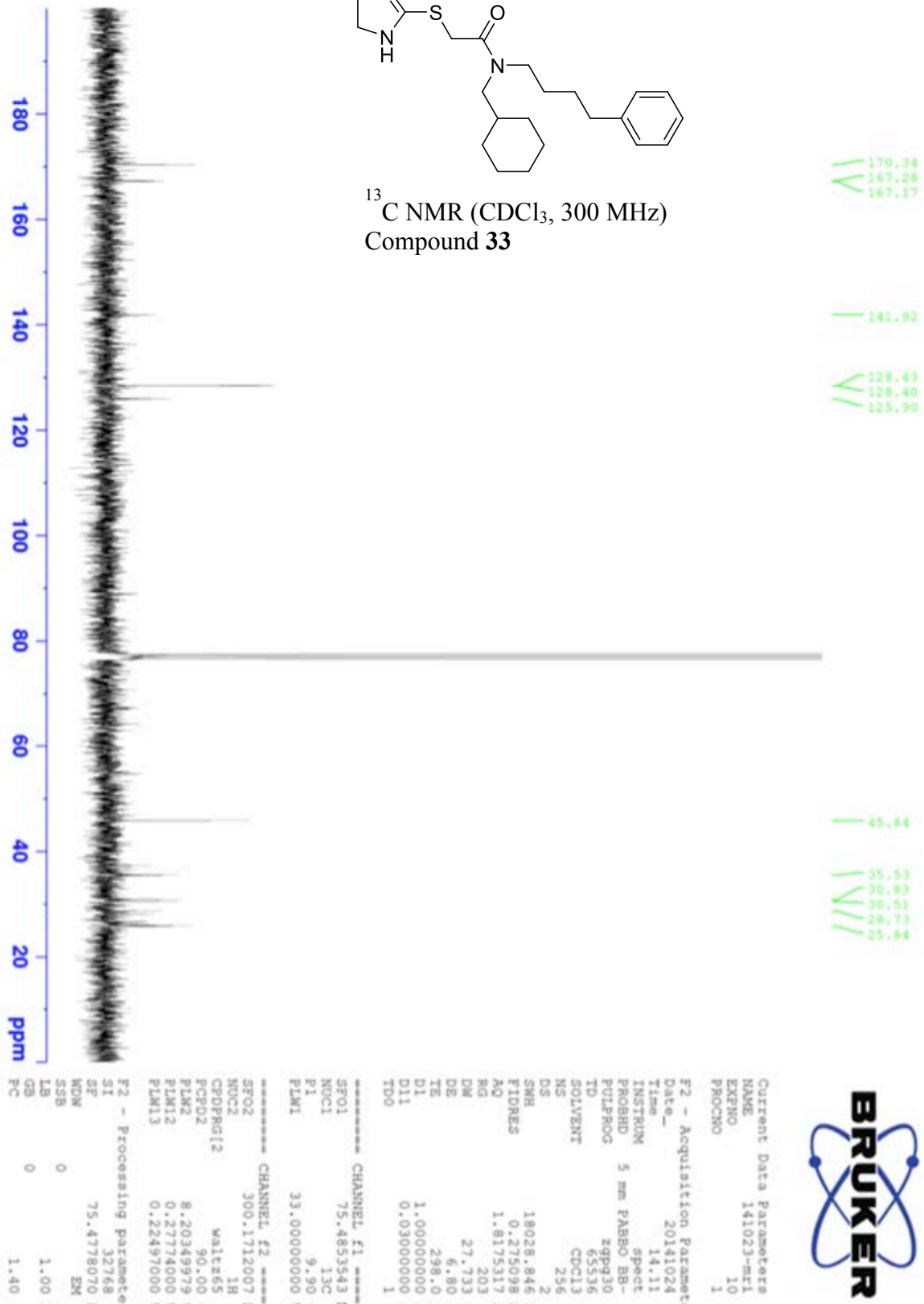
^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **33**

^{13}C NMR Compound 33

N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(4-phenylbutyl)acetamide

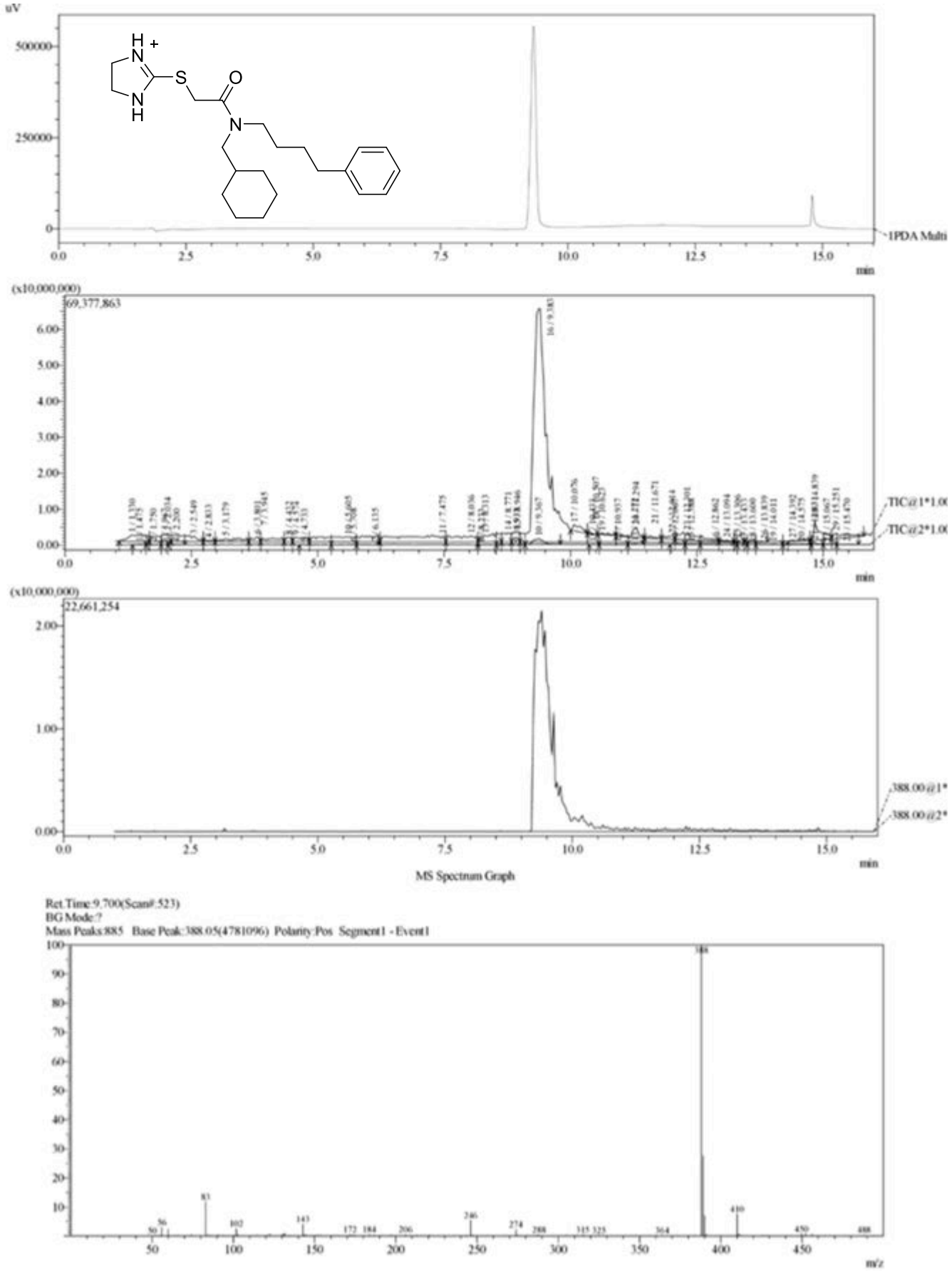


^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **33**



LCMS Compound 33

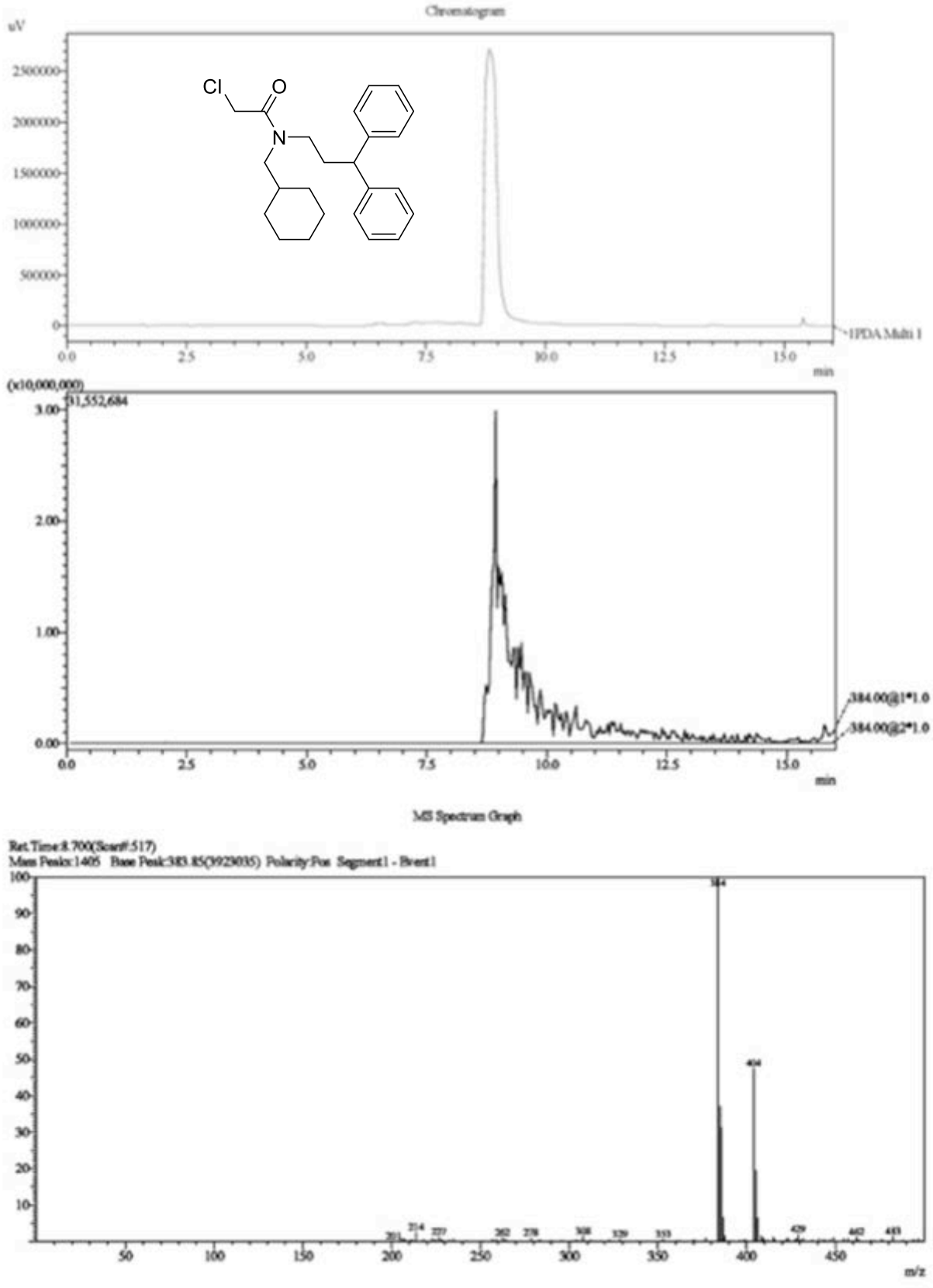
N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(4-phenylbutyl)acetamide



Spectra for Compound 34

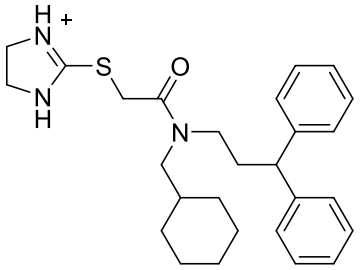
LCMS Compound 34-37

2-chloro-N-(cyclohexylmethyl)-N-(3,3-diphenylpropyl)acetamide

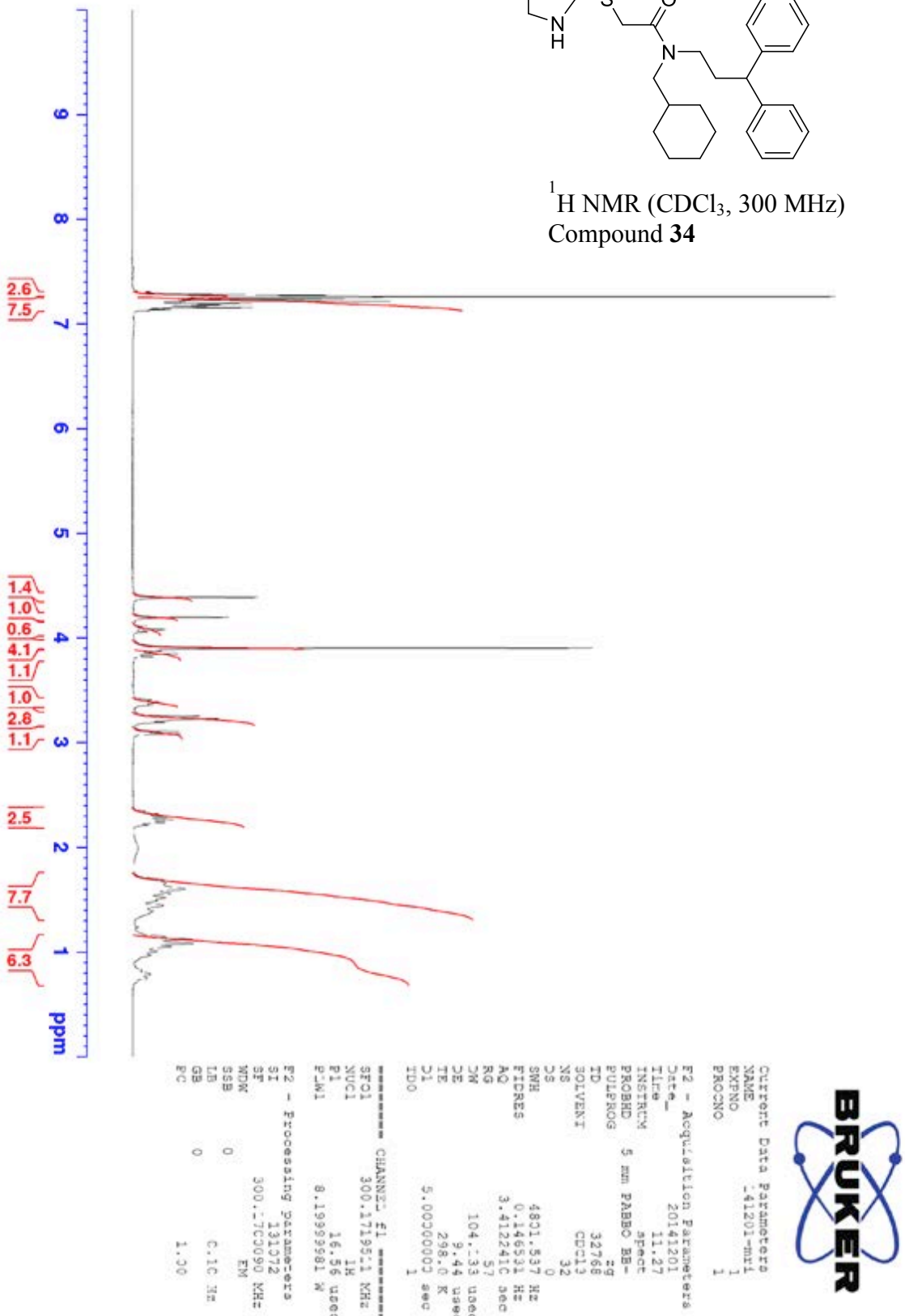


^{1}H NMR Compound 34

N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(3,3-diphenylpropyl)acetamide

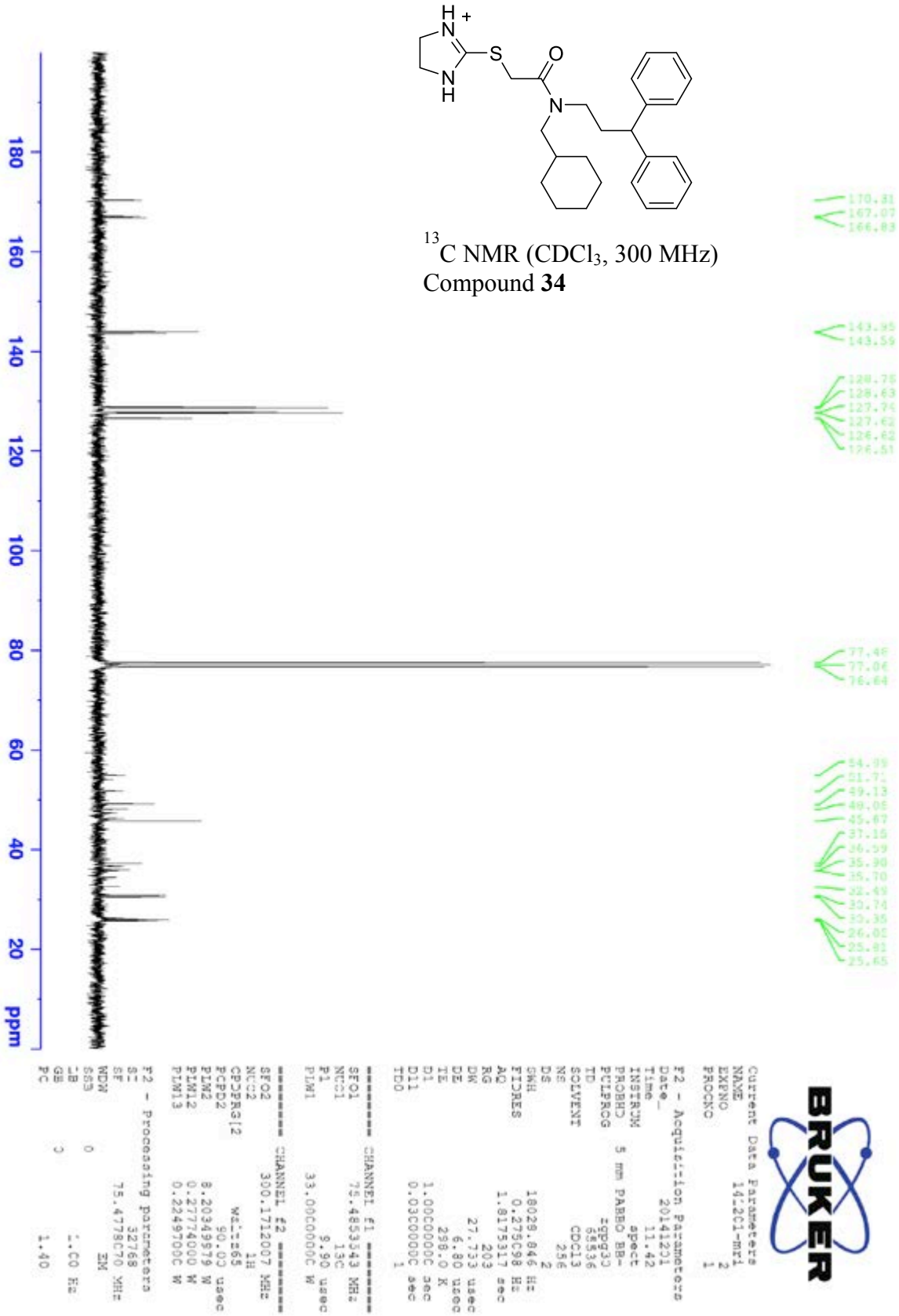


^{1}H NMR ($CDCl_3$, 300 MHz)
Compound **34**



^{13}C NMR Compound 34

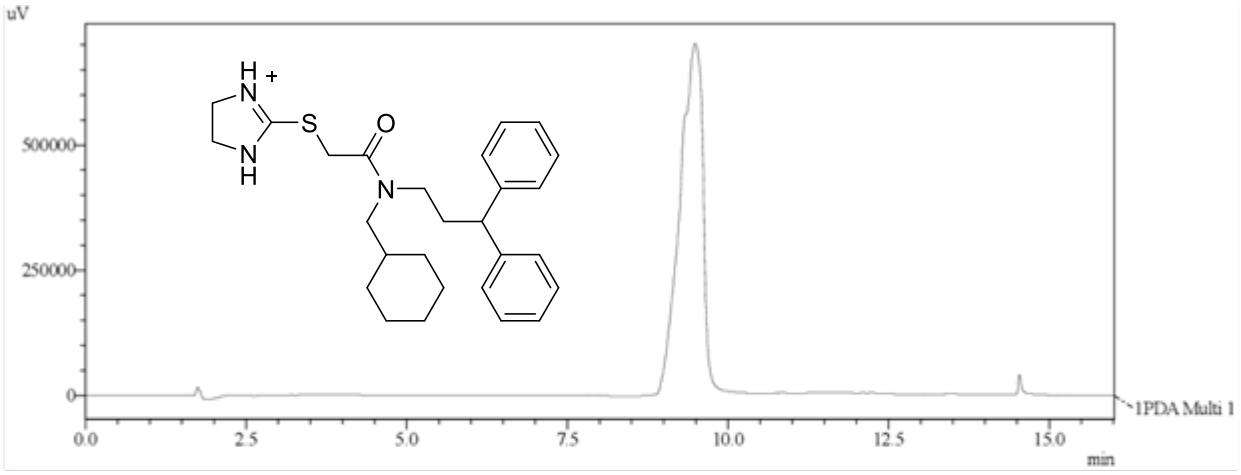
N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(3,3-diphenylpropyl)acetamide

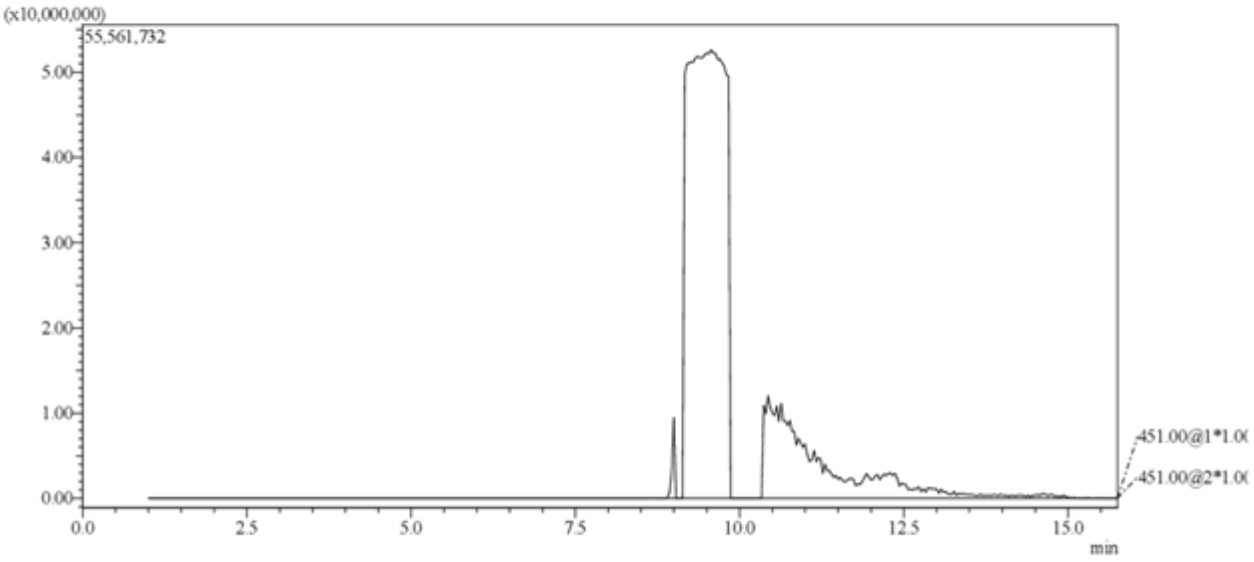


^{13}C NMR ($CDCl_3$, 300 MHz)
Compound **34**

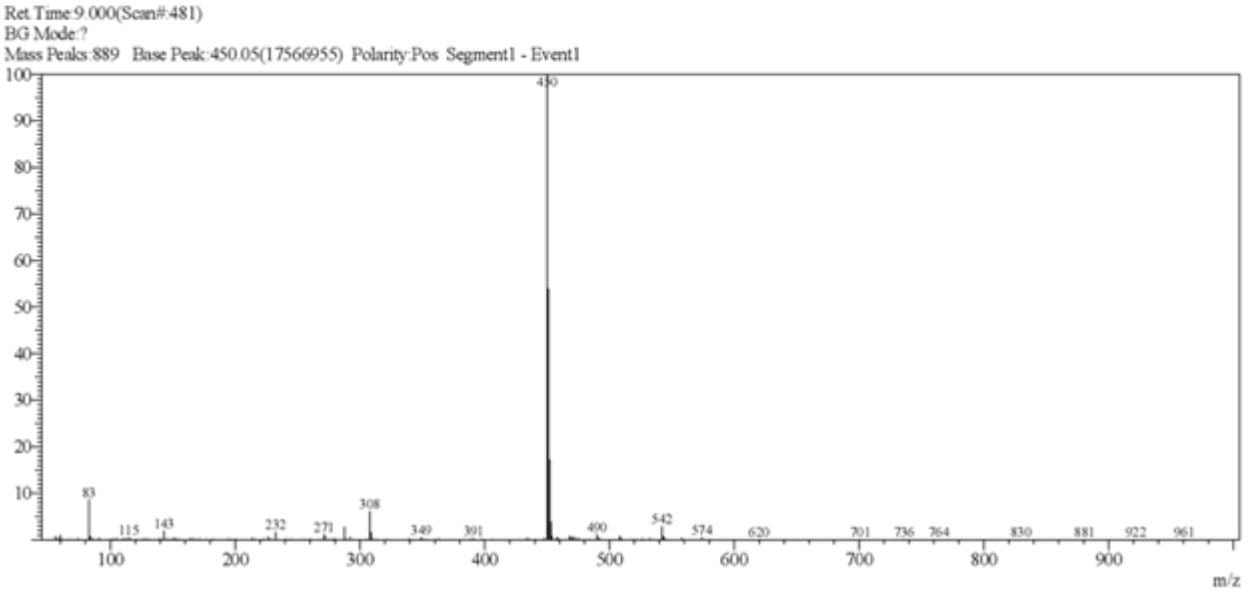
LCMS Compound 34

N-(cyclohexylmethyl)-2-((4,5-dihydro-1H-imidazol-2-yl)thio)-N-(3,3-diphenylpropyl)acetamide



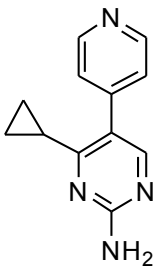


MS Spectrum Graph

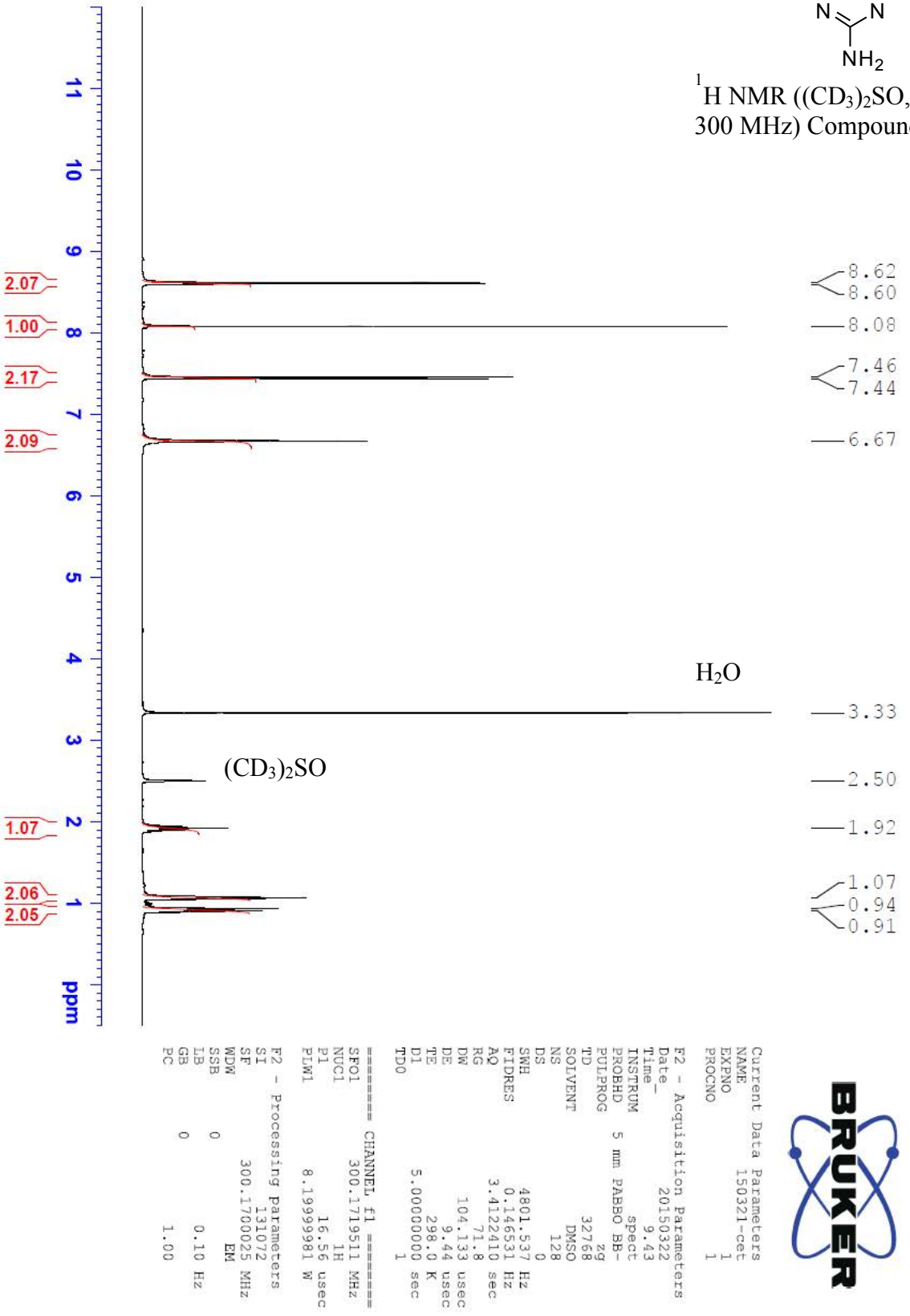


Spectra for Compound J9

^{1}H NMR Compound J9

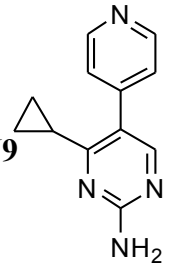


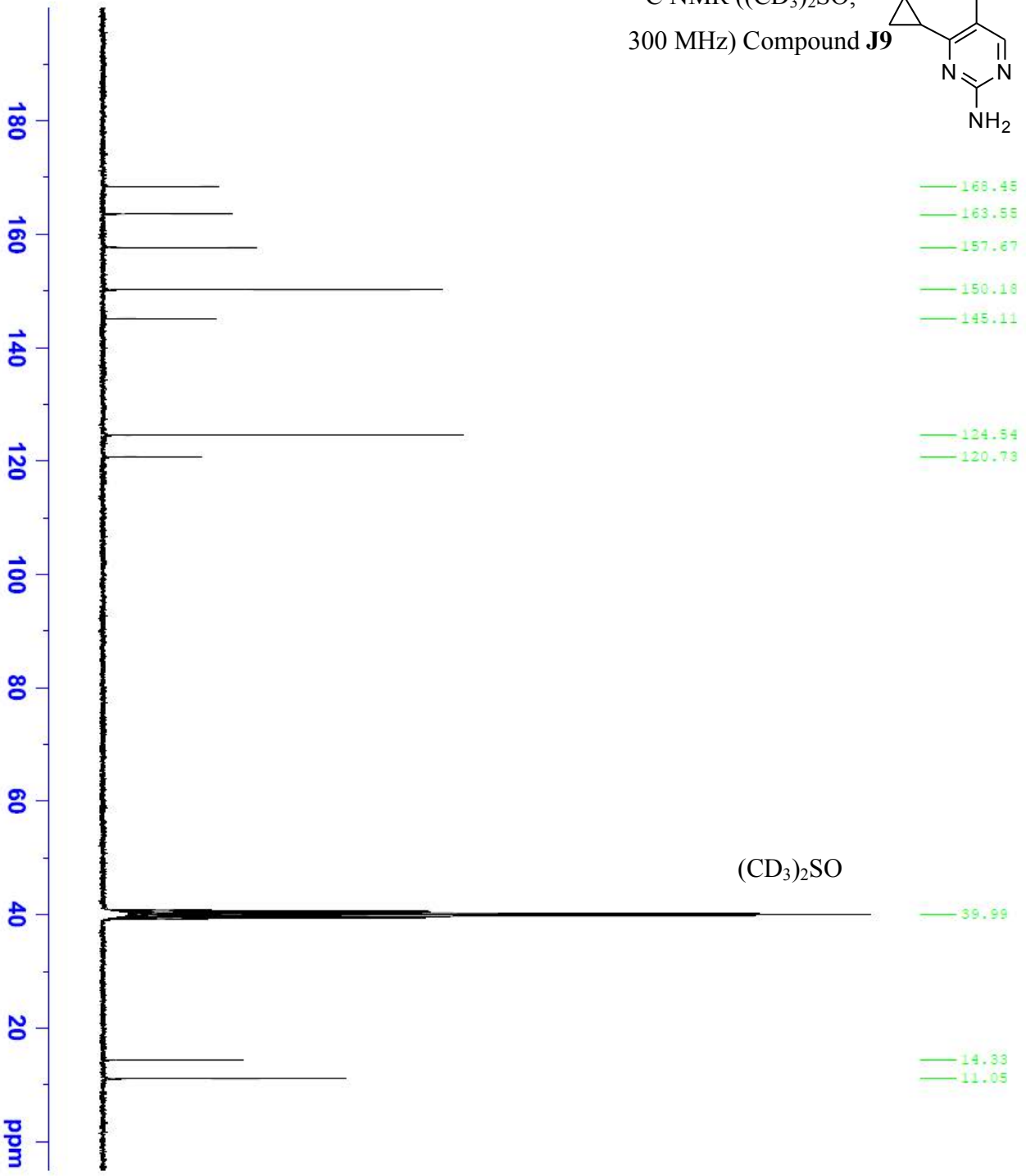
^{1}H NMR ($(CD_3)_2SO$, 300 MHz) Compound **J9**



^{13}C NMR Compound J9

^{13}C NMR ($(CD_3)_2SO$, 300 MHz) Compound **J9**



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EXPNO                          2
PROCNO                         1

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TD                  65536
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NS                     1536
DS                        2
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FIDRES                0.275098 Hz
AQ                1.8175317 sec
RG                       203
DW                  27.733 usec
DE                   6.80 usec
TE                  298.1 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                       6

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NUC1                      13C
P1                       9.90 usec
PLW1            33.00000000 W

======== CHANNEL f2 ========
SFO2            300.1712007 MHz
NUC2                      1H
CPDPRG[2 bi_waltz65_256
PCPD2                 90.00 usec
PLW2             8.20349979 W
PLW12            0.27774000 W
PLW13            0.22497000 W

F2 - Processing parameters
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SSB                       0
LB                             1.00 Hz
GB                        0
PC                             1.40
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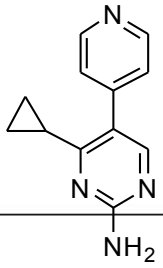
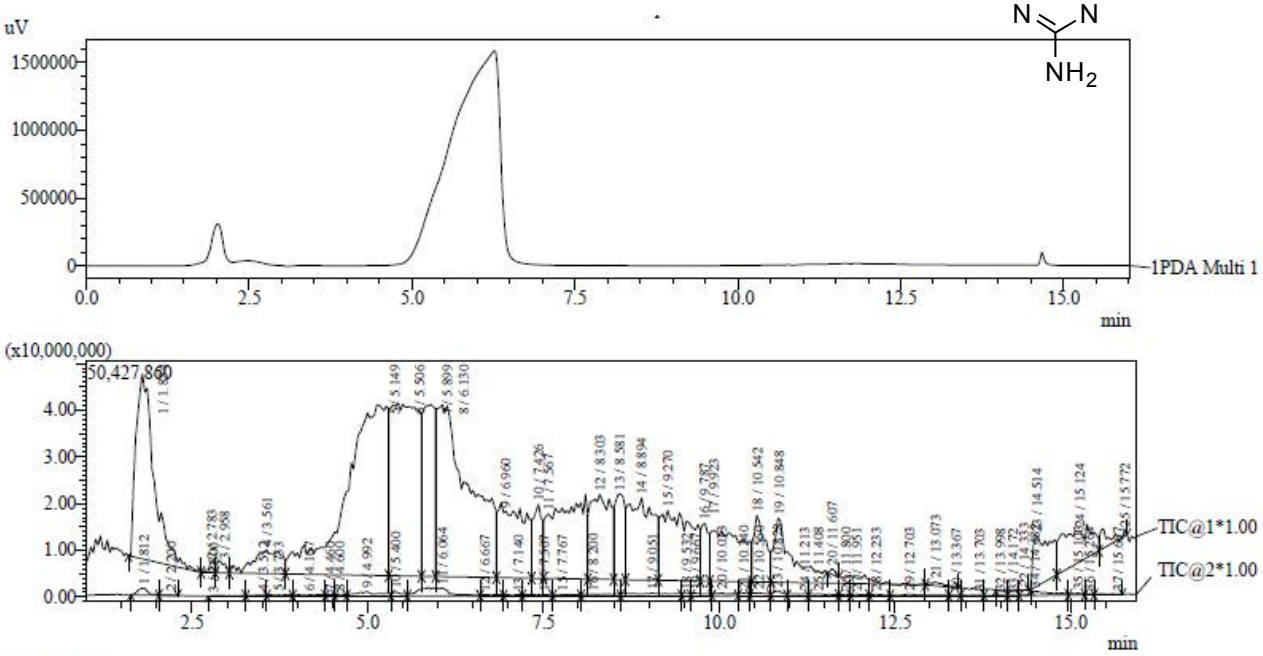
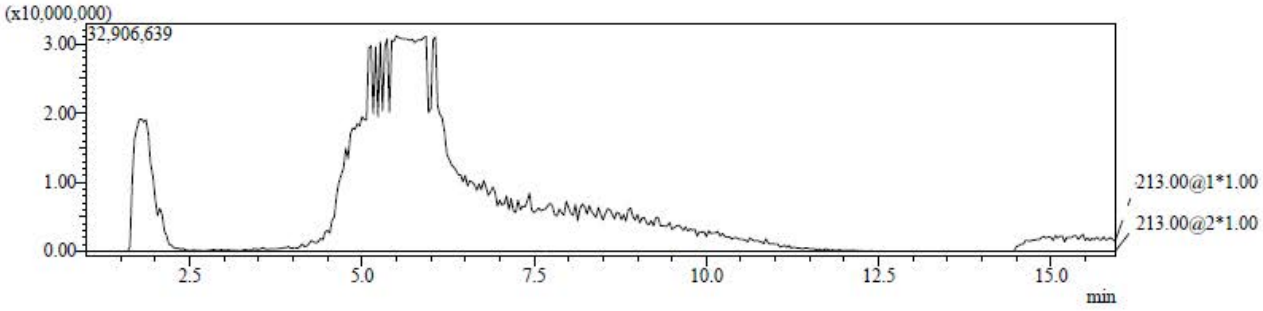
LCMS Compound J9

Calcd for $C_{12}H_{12}N_4$ + H
= 213.1142
Compound **J9**

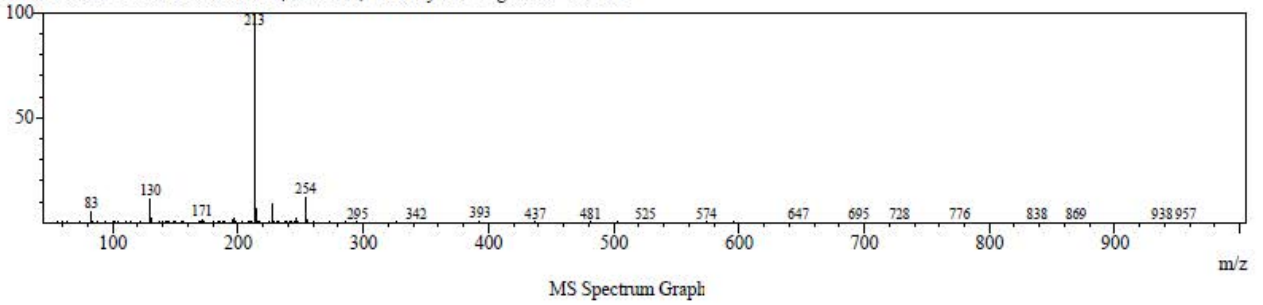






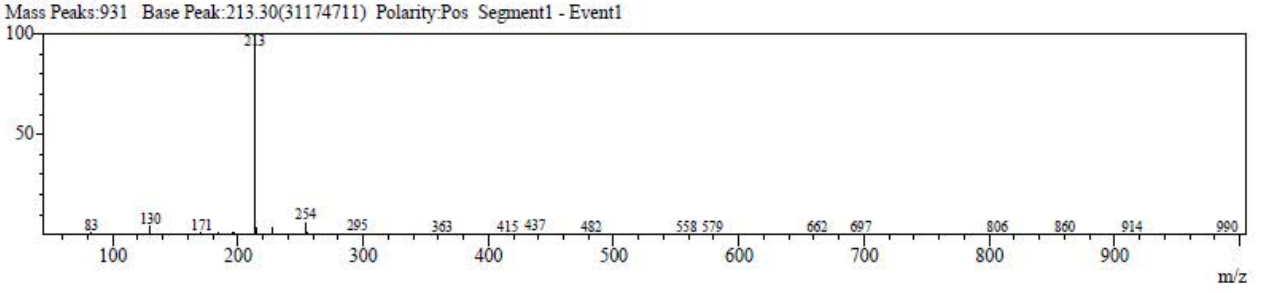
MS Spectrum Graph

Ret.Time:1.800(Scan#:49)
BG Mode:?
Mass Peaks:912 Base Peak:213.00(19169314) Polarity:Pos Segment1 - Event1



MS Spectrum Graph

Ret.Time:5.500(Scan#:271)
BG Mode:?
Mass Peaks:931 Base Peak:213.30(31174711) Polarity:Pos Segment1 - Event1



HRMS(ESI) Compound J9

Calcd for $C_{12}H_{12}N_4$ + H
= 213.1142
Compound **J9**