

Electronic Supplementary Information (ESI)

Nonbenzenoid *N*-Aryl Oxalamide: Synthesis of *troponyl*-Oxalamide Peptides by Pd(II)-Catalyzed C(sp³)-H Functionalization of Glycinamides

Chinmay K. Jena,^{a,b} and Nagendra K. Sharma^{*a,b}

^aSchool of Chemical Sciences, National Institute of Science Education and Research (NISER), Jatani-752050, Odisha, India.

^b Homi Bhabha National Institute (HBNI)-Mumbai, Anushaktinagar, Mumbai, 400 094, India

*Corresponding author. Tel.: 0674-249-4141; fax: +91 (674) 2494004; E-mail: nagendra@niser.ac.in

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1. NMR and Mass Spectra of Troponyl Glycine Derivatives (2/ 3a- 3o)

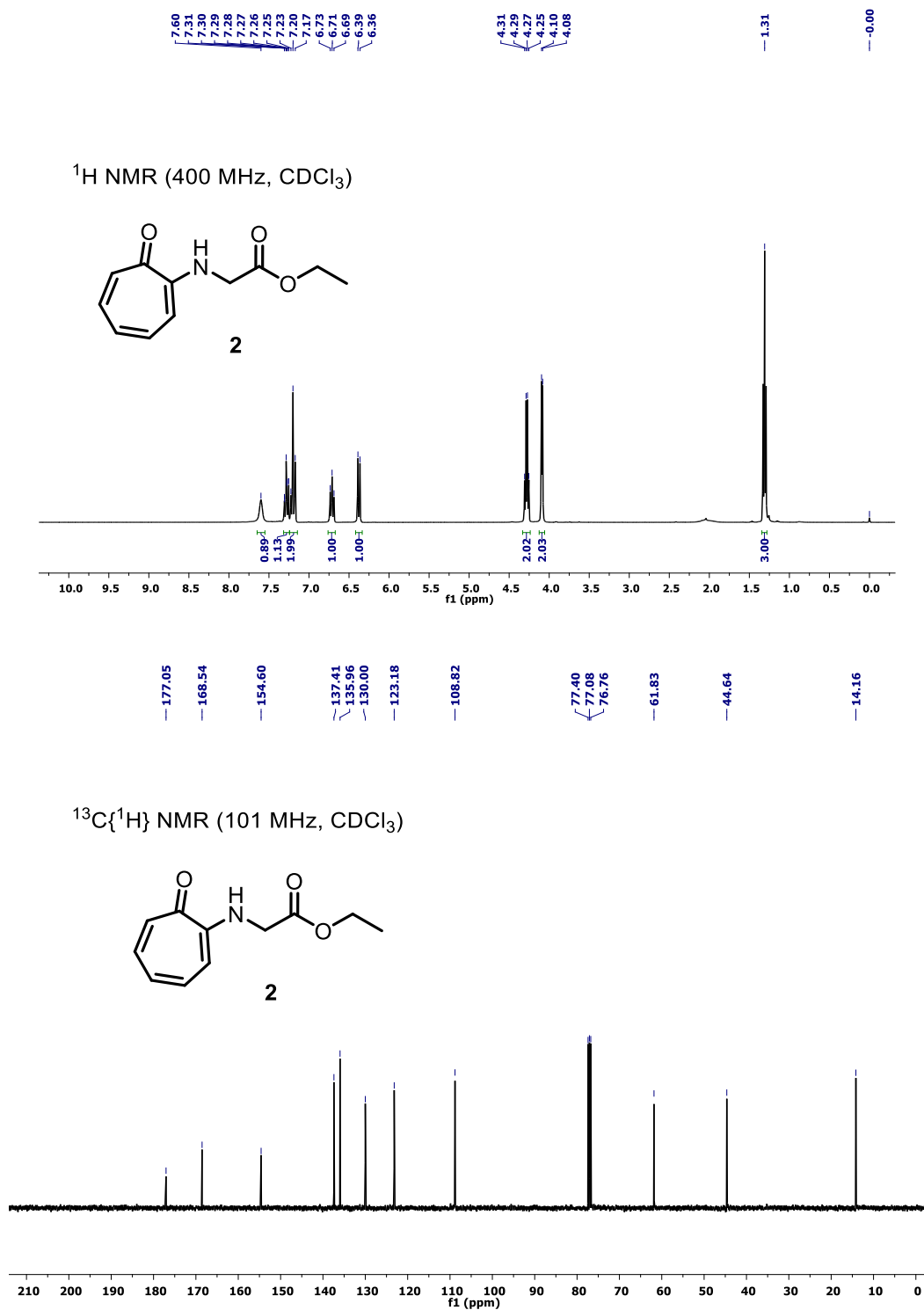


Fig S1. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **2**.

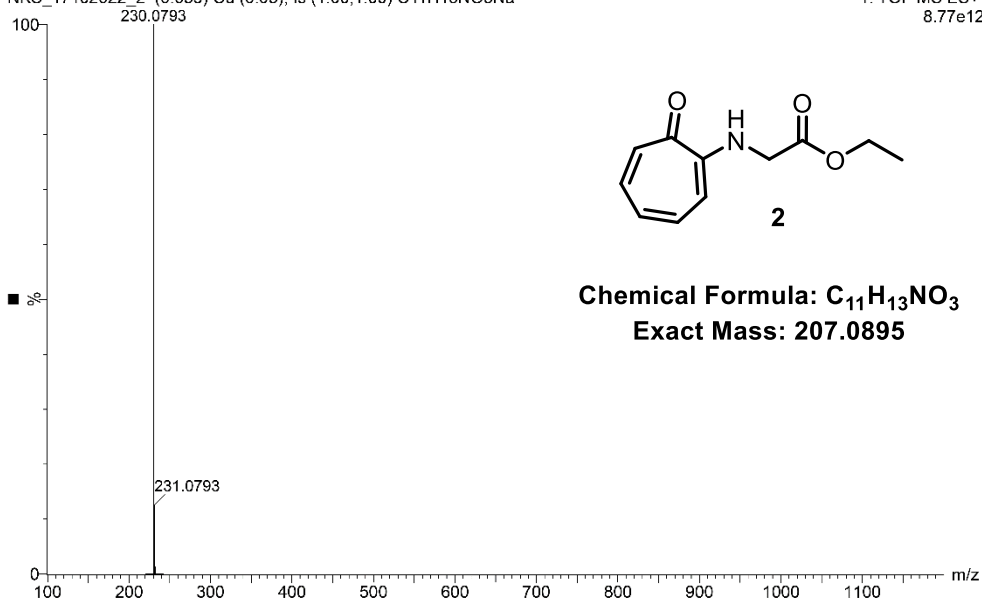
NKS-CKJ-942

17-Oct-2022
19:30:20

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8.77e12



NKS_17102022_2 41 (0.829) Cm (41:48)

1: TOF MS ES+
6.15e6

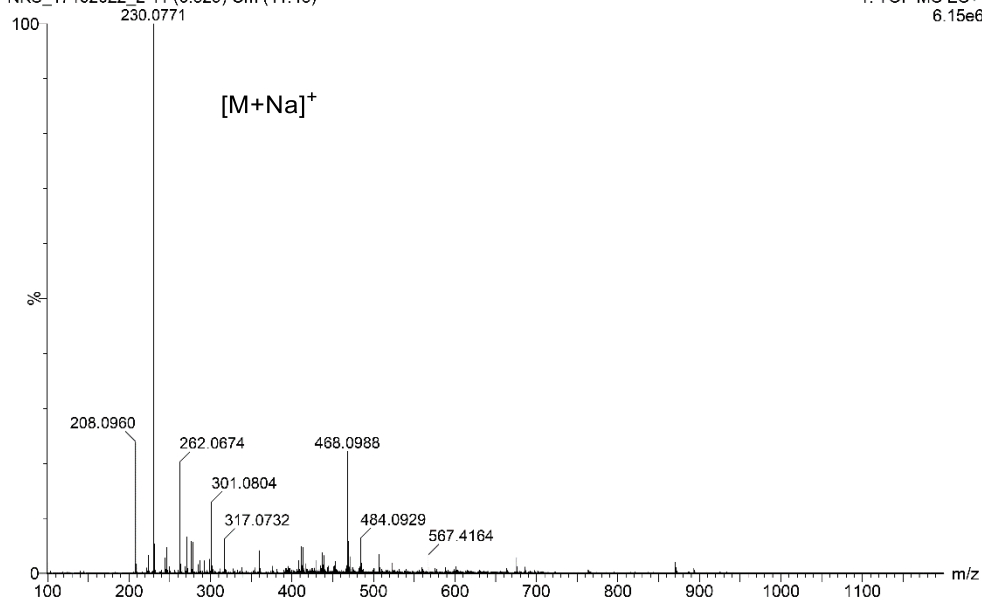


Fig S2. ESI-HRMS spectra of troponyl glycine derivative **2**.

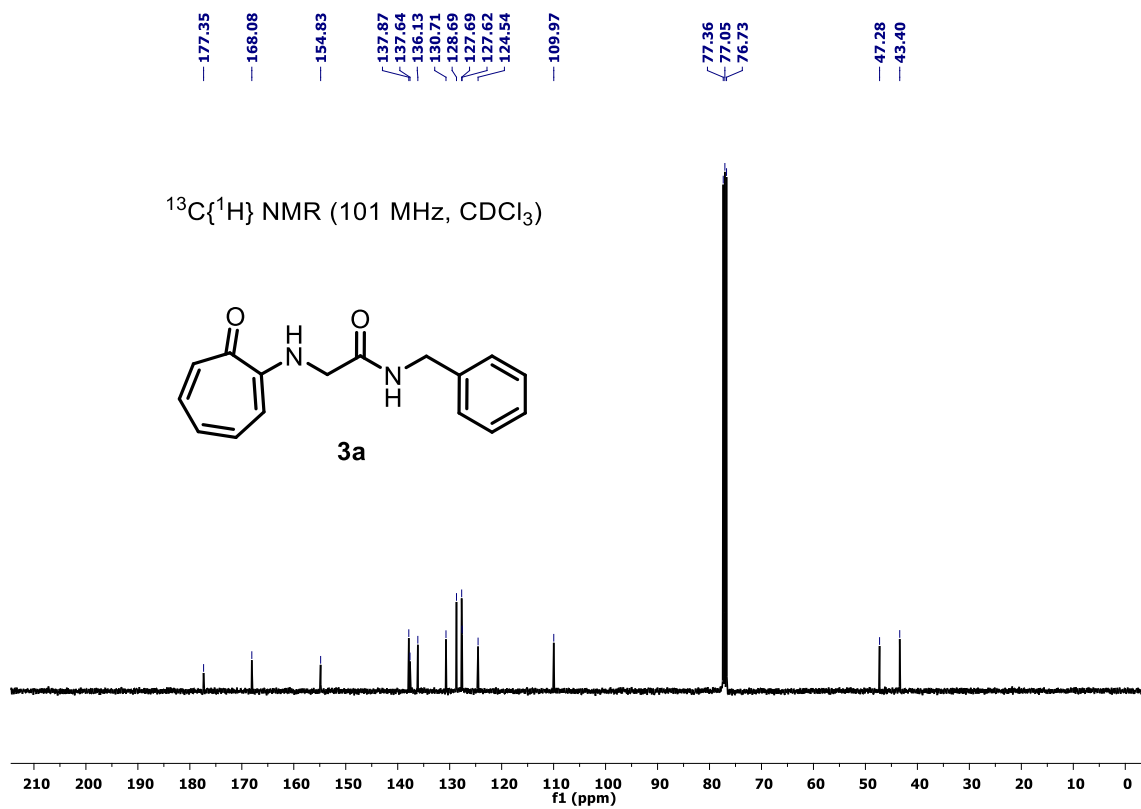
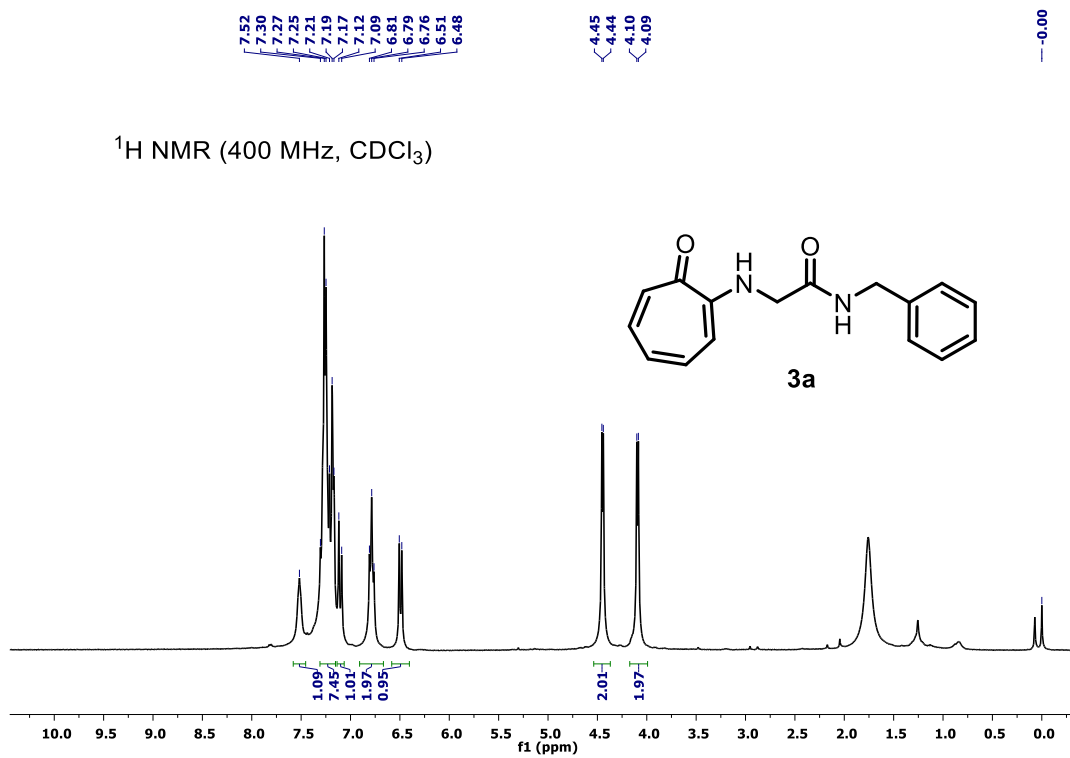


Fig S3. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3a**.

NKS-CKJ-403

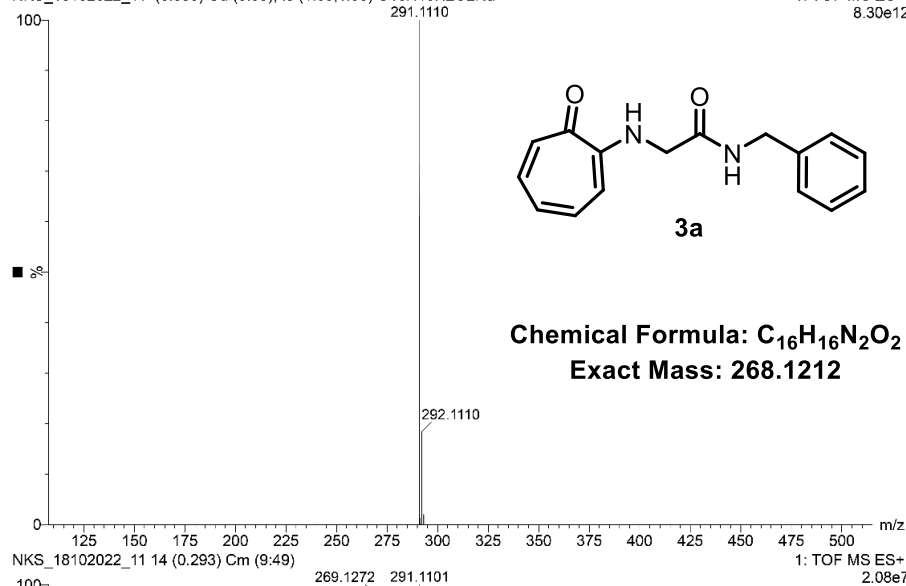
18-Oct-2022

19:42:40

XEVO-G2XSQTOF#YFA1739

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1: TOF MS ES+
8.30e12



NKS_18102022_11 14 (0.293) Cm (9:49)

1: TOF MS ES+
2.08e7

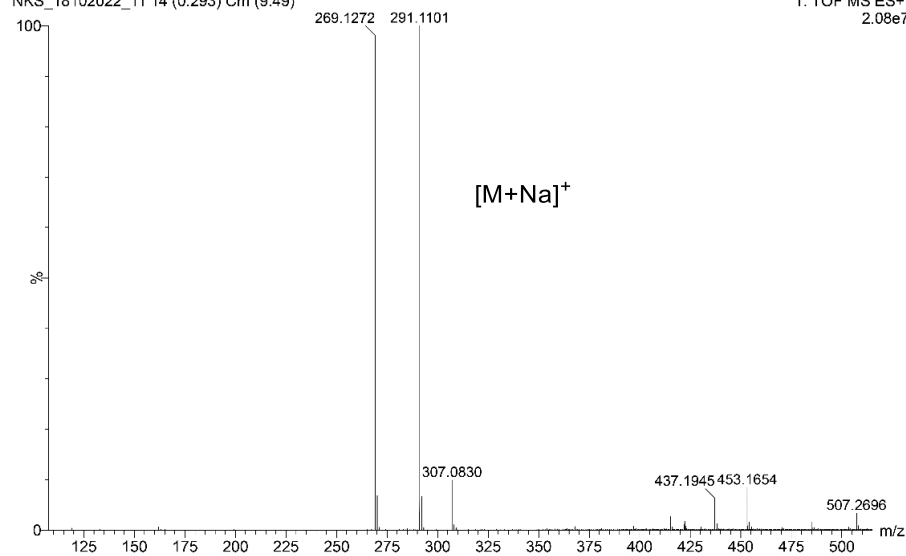


Fig S4. ESI-HRMS spectra of troponyl glycine derivative **3a**.

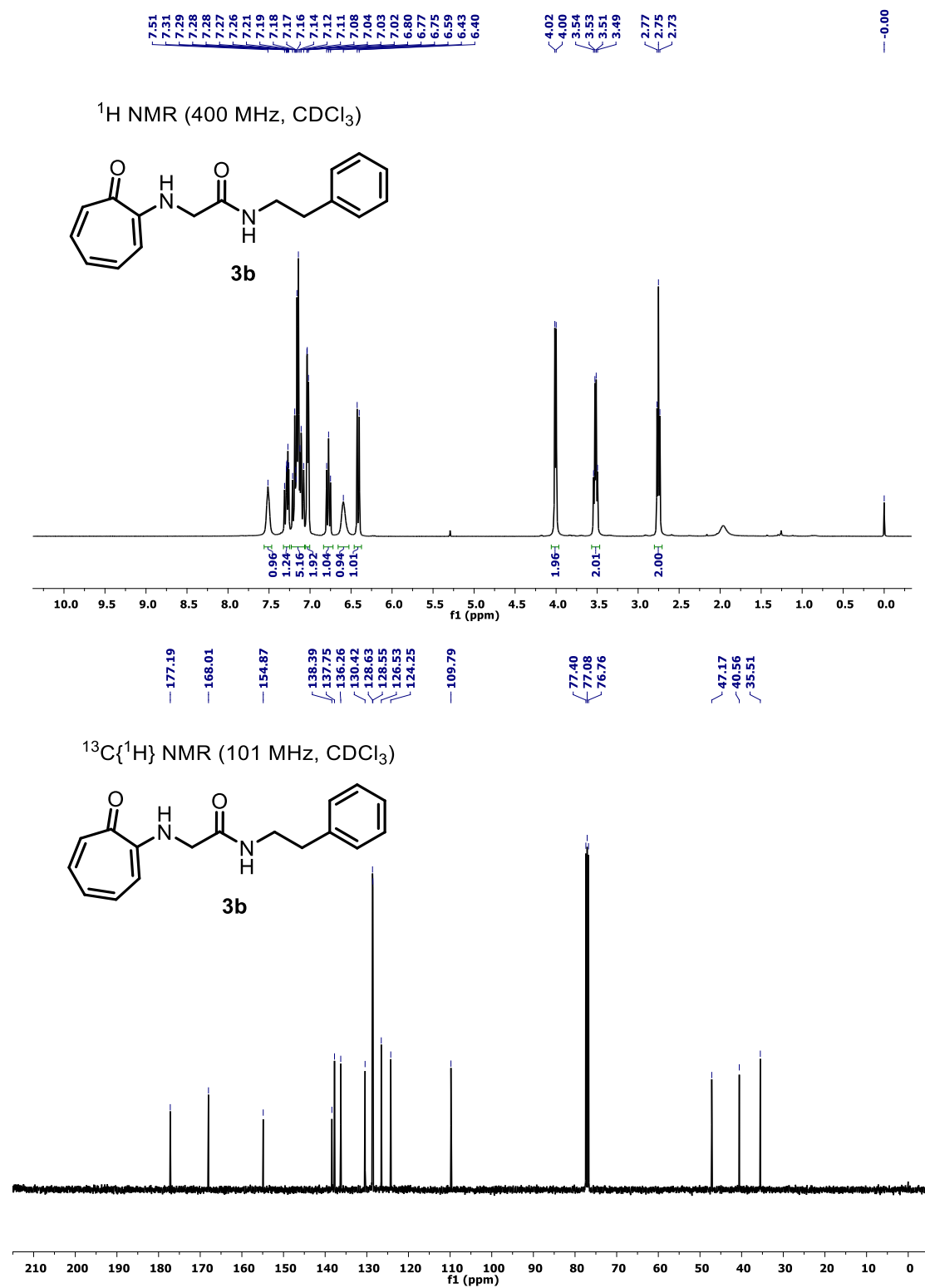


Fig S5. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3b**.

Display Report

Analysis Info

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Method Pos_tune_low_05122019.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/17/2021 5:24:25 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

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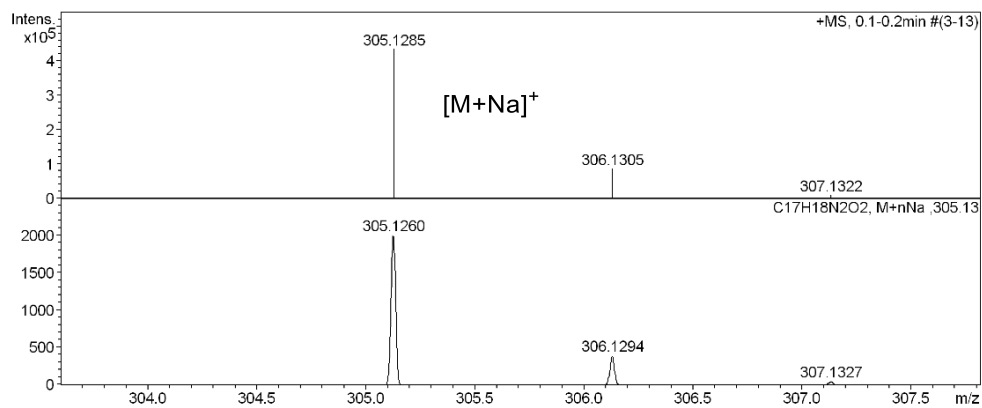
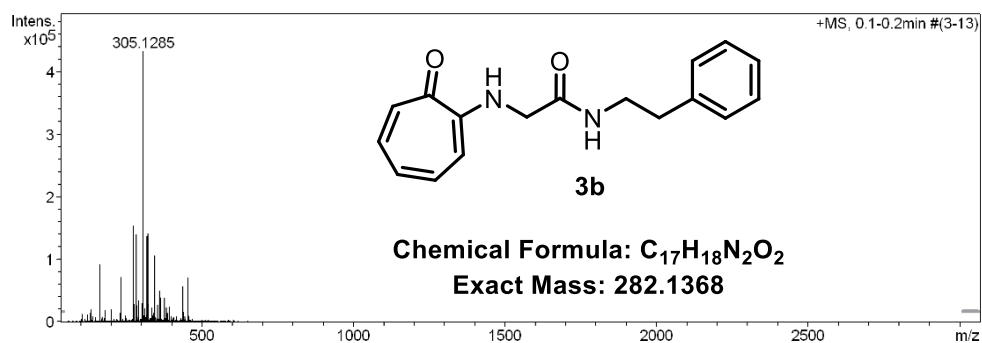
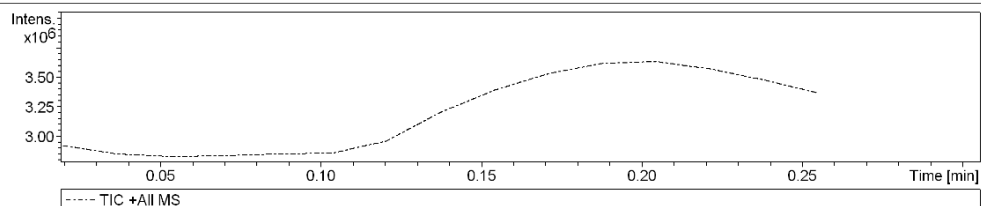
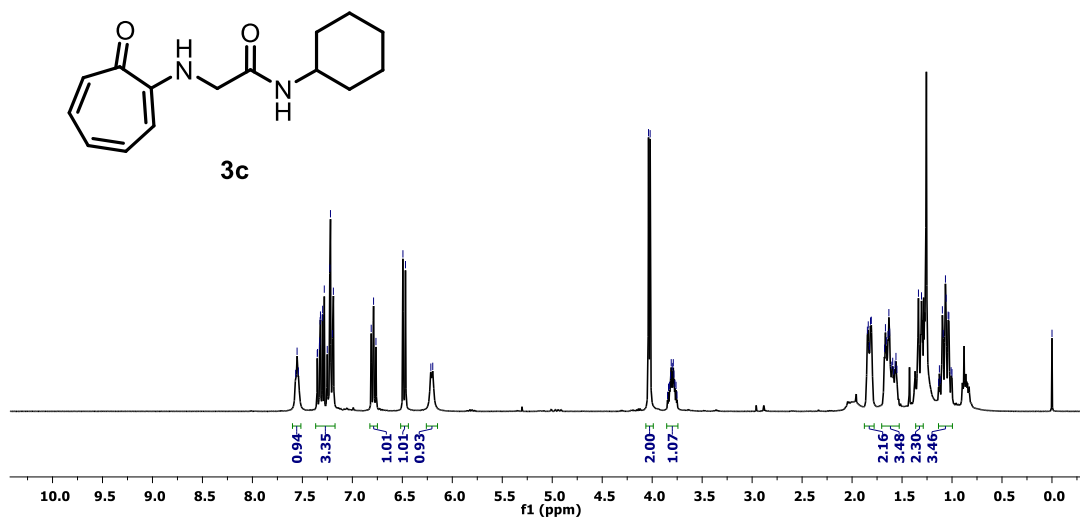


Fig S6. ESI-HRMS spectra of troponyl glycine derivative **3b**.

7.57
7.55
7.54
7.35
7.35
7.33
7.32
7.32
7.30
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7.20
7.19
6.81
6.79
6.76
6.49
6.47
6.21
6.20
6.20
4.02
3.83
3.82
3.81
3.80
3.79
3.78
3.77
3.76
1.85
1.84
1.83
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1.55
1.34
1.31
1.13
1.13
1.12
1.10
1.09
1.07
1.06
1.04
1.03
1.01
1.00
-0.00

^1H NMR (400 MHz, CDCl_3)



177.44
166.88
155.03
137.85
136.16
130.68
124.41
109.88
77.44
77.12
76.80
48.47
47.48
33.00
25.39
24.86

$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3)

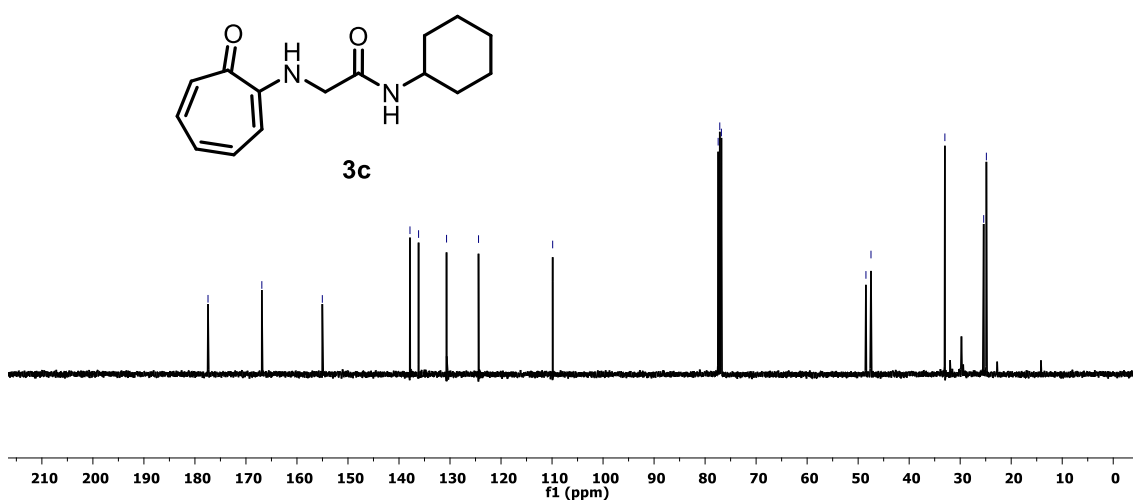


Fig S7. ^1H , $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of troponyl glycine derivative **3c**.

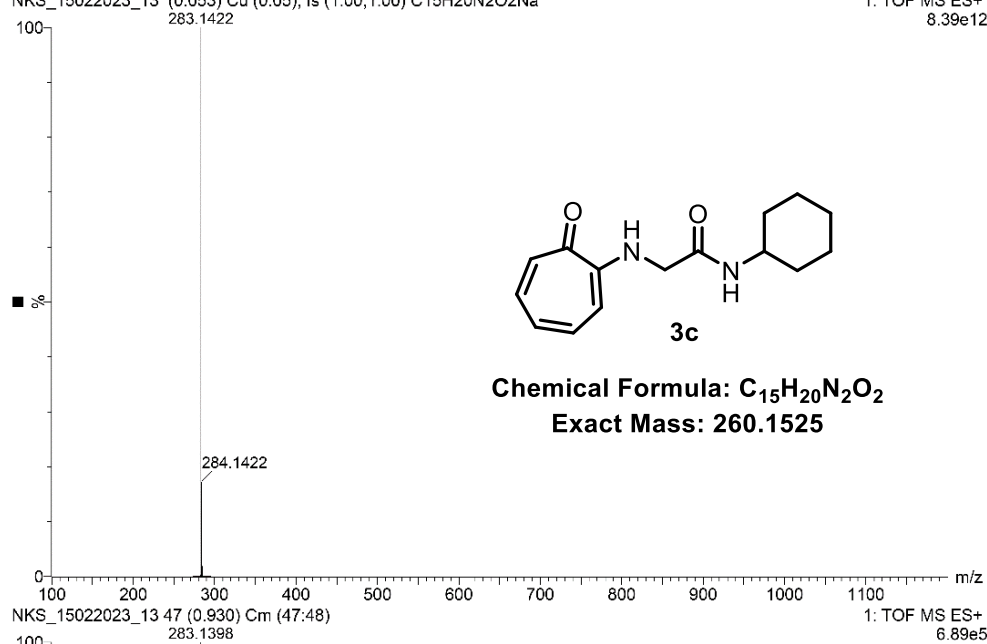
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16-Feb-2023
01:46:53

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8.39e12



NKS_15022023_13 47 (0.930) Cm (47:48)

1: TOF MS ES+
6.89e5

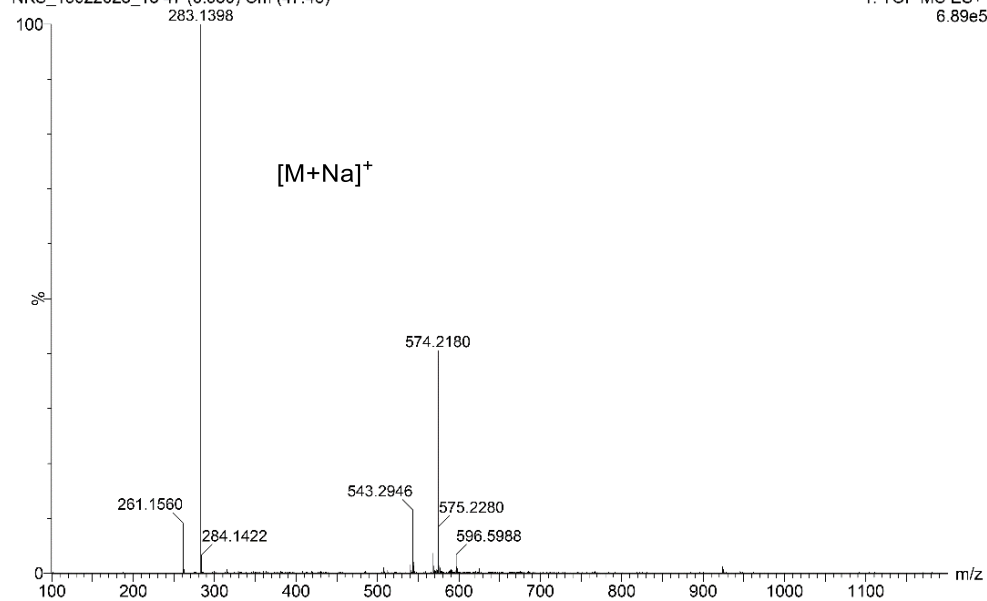


Fig S8. ESI-HRMS spectra of troponyl glycine derivative **3c**.

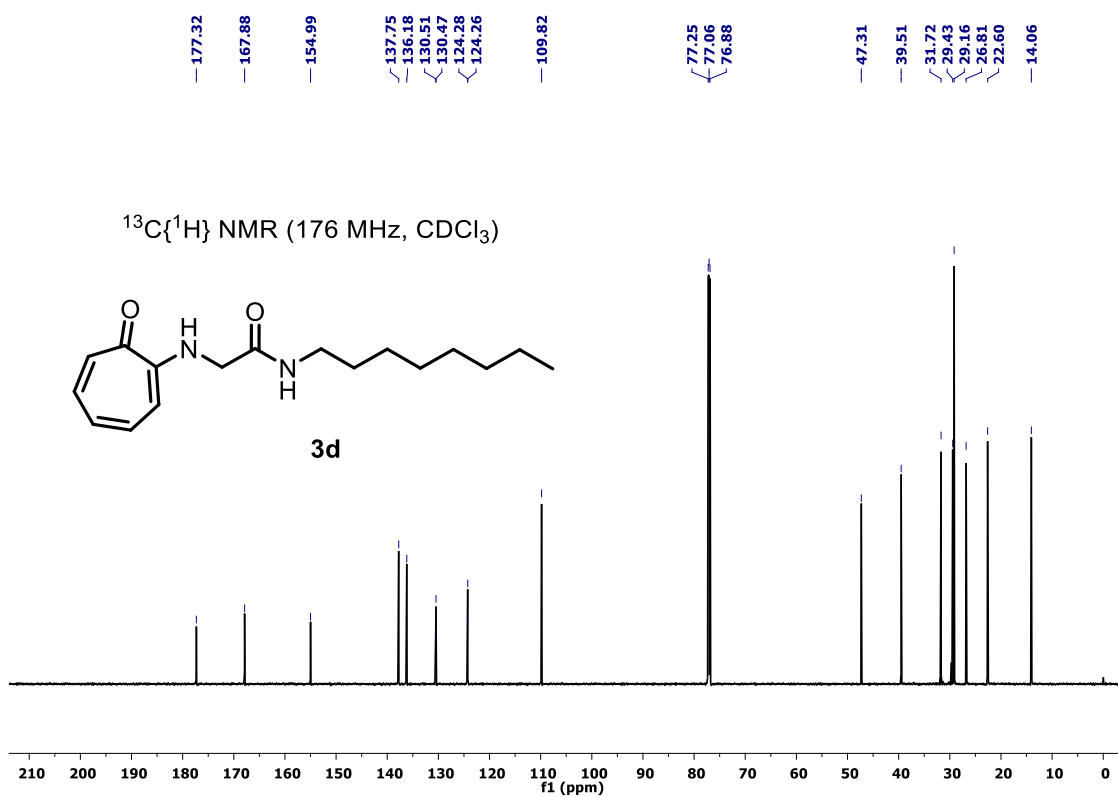
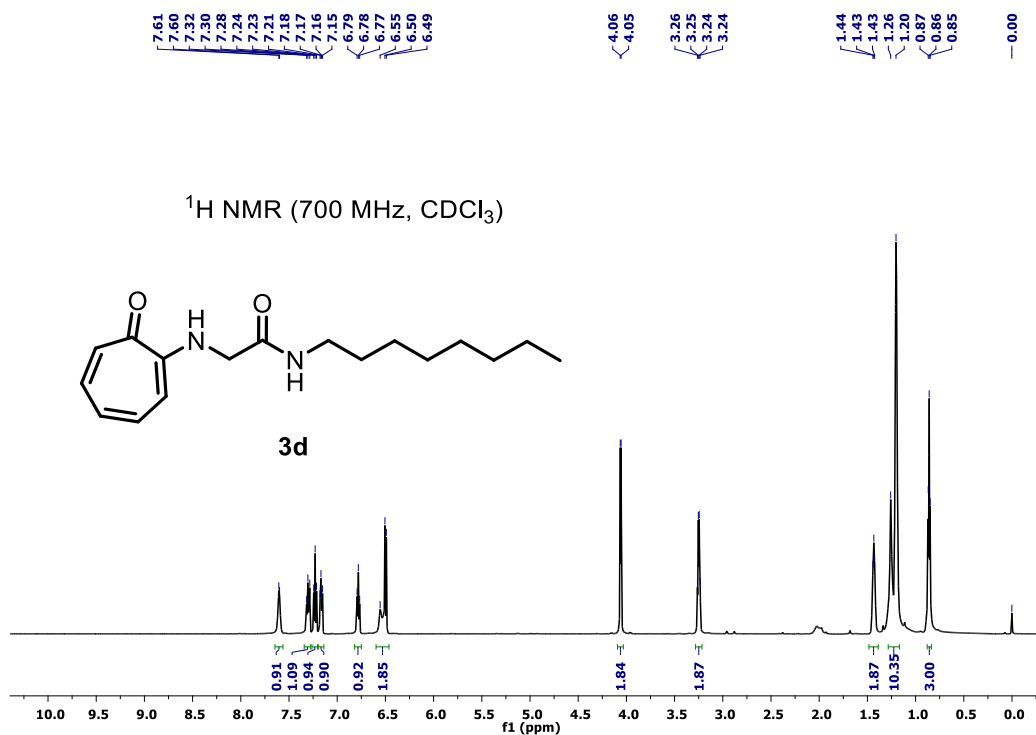


Fig S9. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3d**.

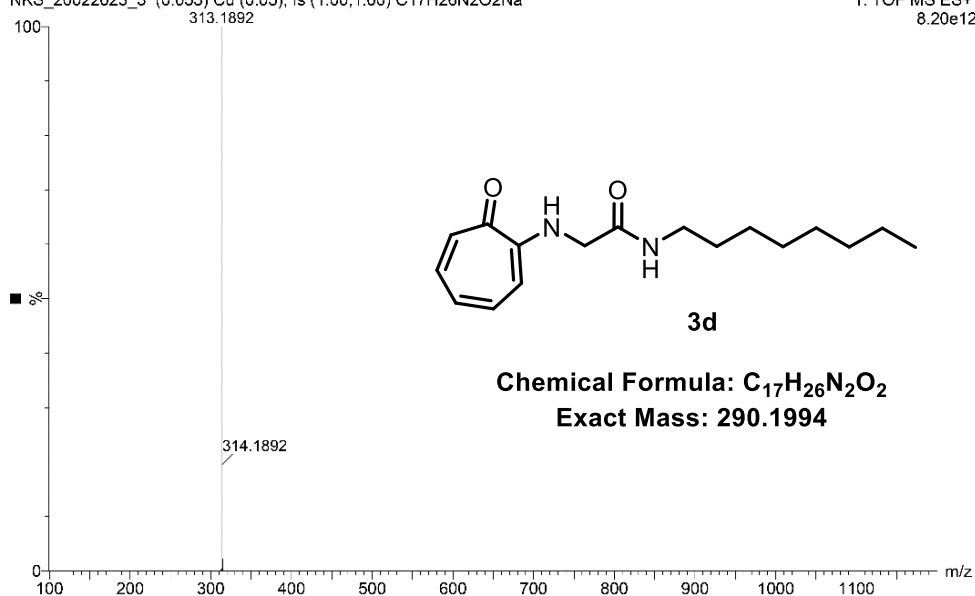
NKS_CKJ_1104

20-Feb-2023
18:10:25

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1: TOF MS ES+
8.20e12



NKS_20022023_3 47 (0.930) Cm (47.48)

1: TOF MS ES+
2.28e6

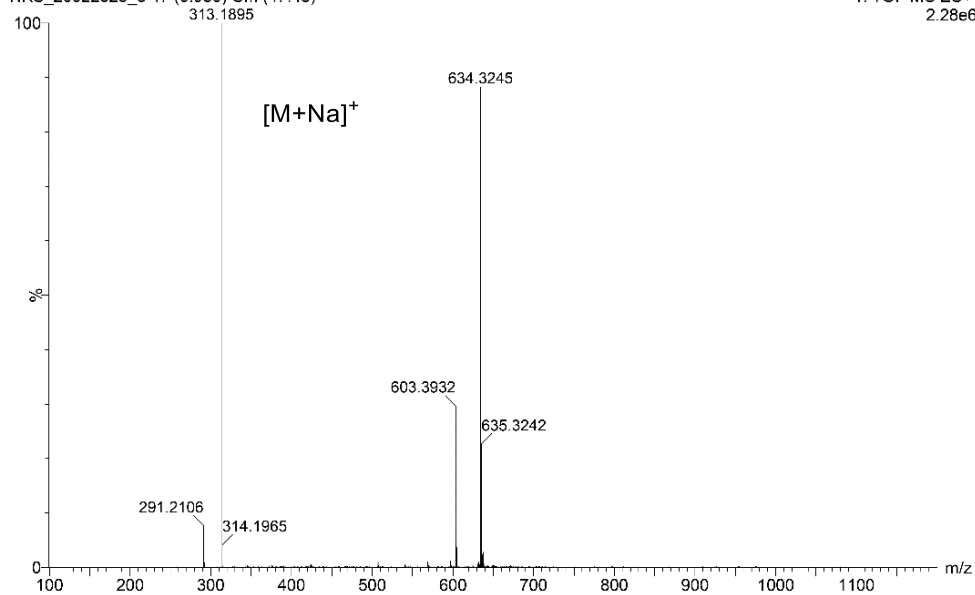


Fig S10. ESI-HRMS spectra of troponyl glycine derivative **3d**.

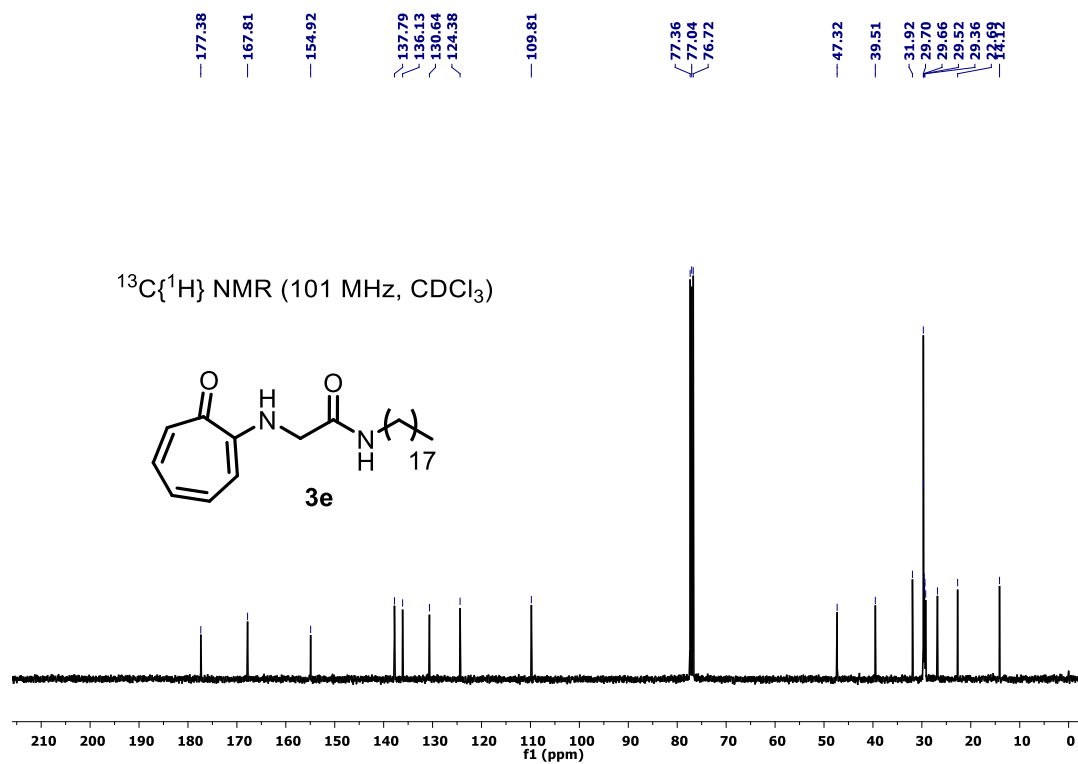
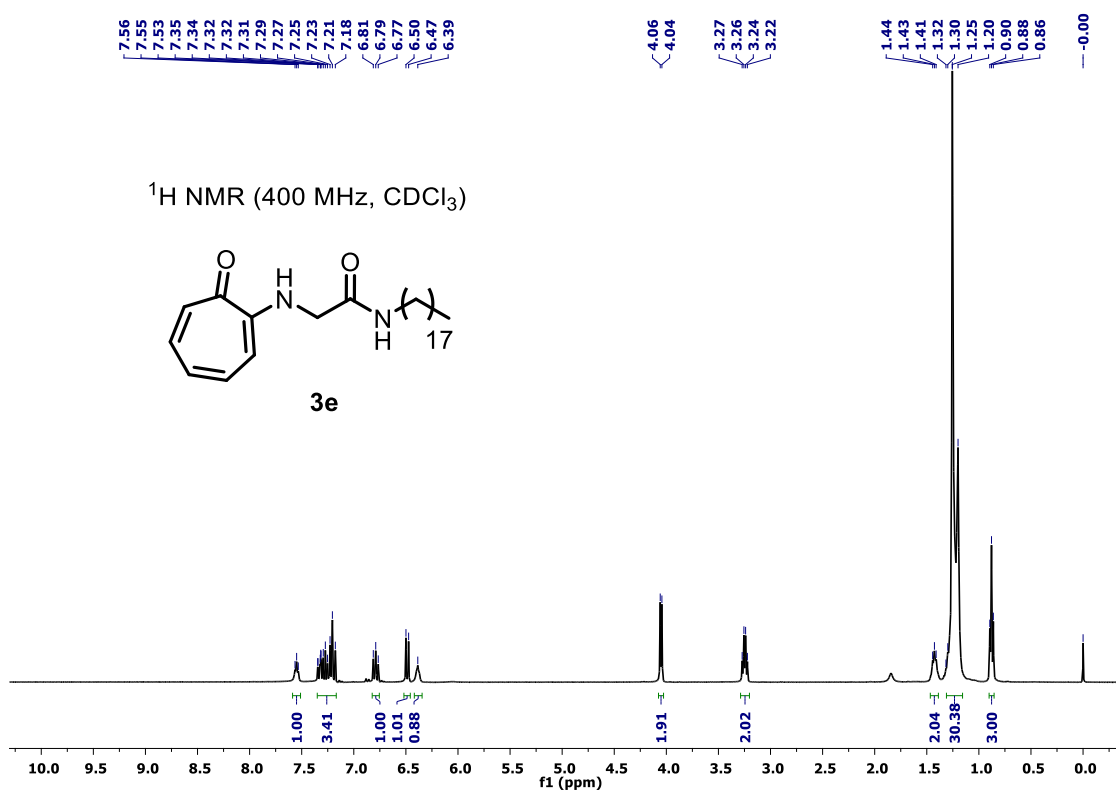


Fig S11. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3e**.

NKS_CKJ_1115

07-Mar-2023

21:03:46

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1: TOF MS ES+
7.35e12

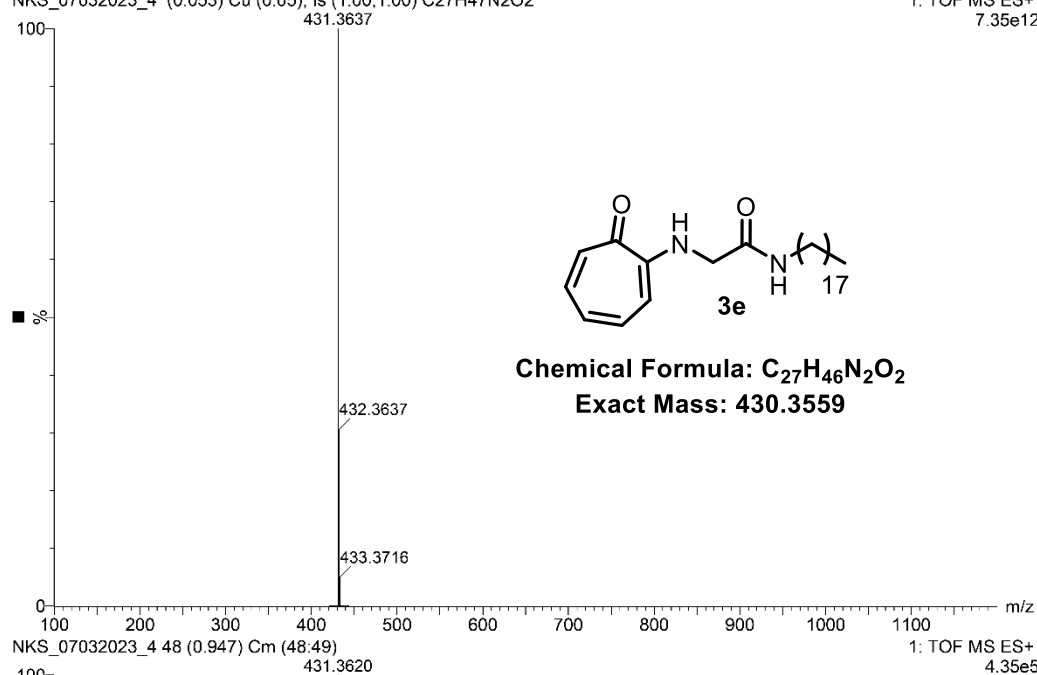


Fig S12. ESI-HRMS spectra of troponyl glycine derivative **3e**.

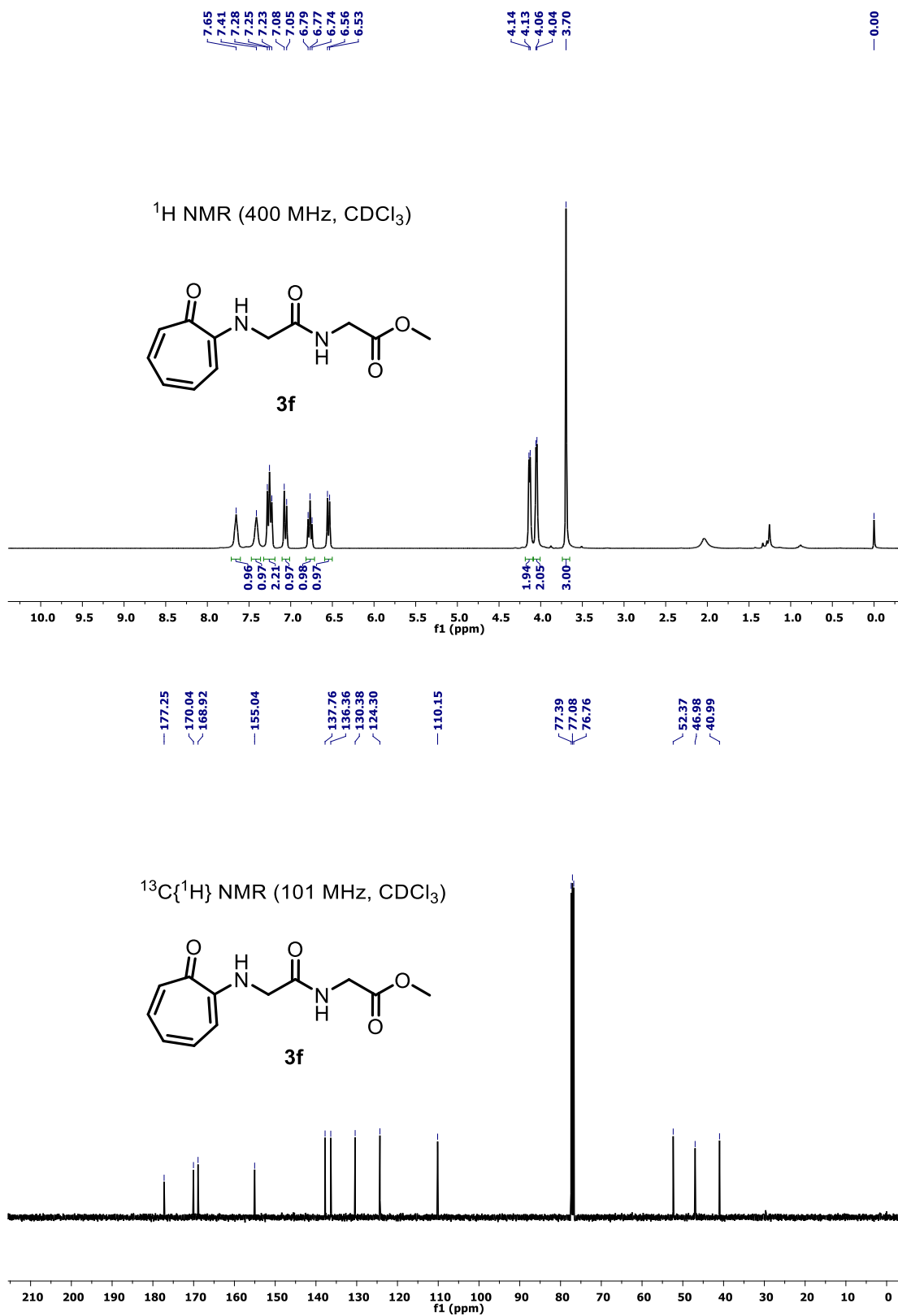


Fig S13. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3f**.

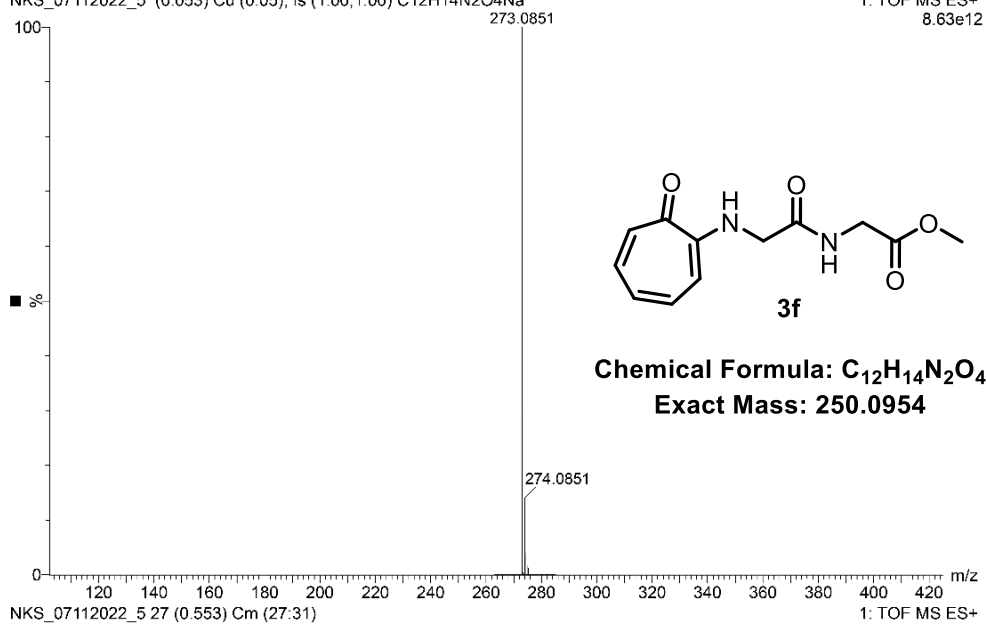
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07-Nov-2022
17:38:07

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1: TOF MS ES+
8.63e12



NKS_07112022_5 27 (0.553) Cm (27:31)

1: TOF MS ES+
1.96e7

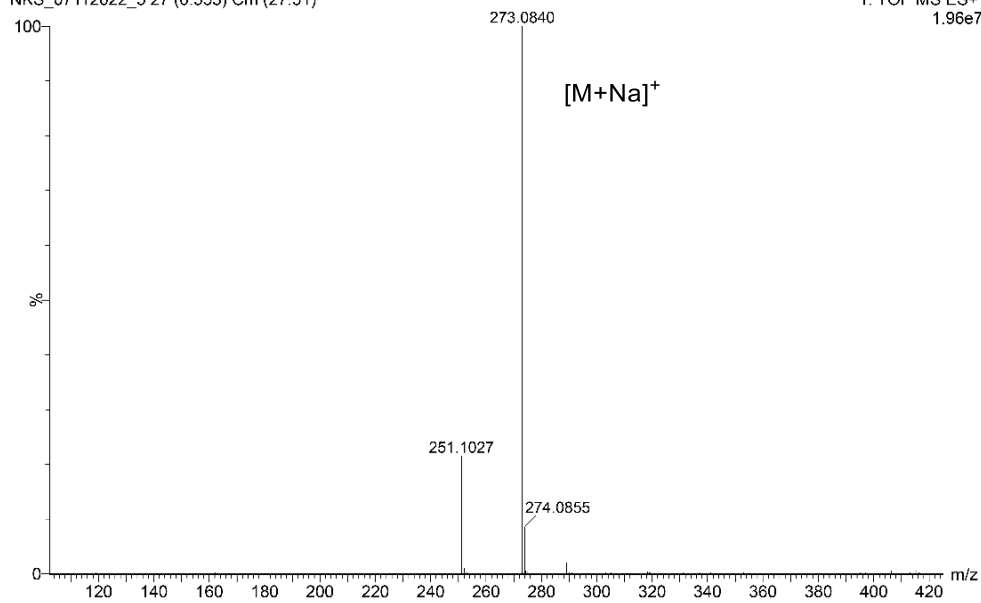


Fig S14. ESI-HRMS spectra of troponyl glycine derivative **3f**.

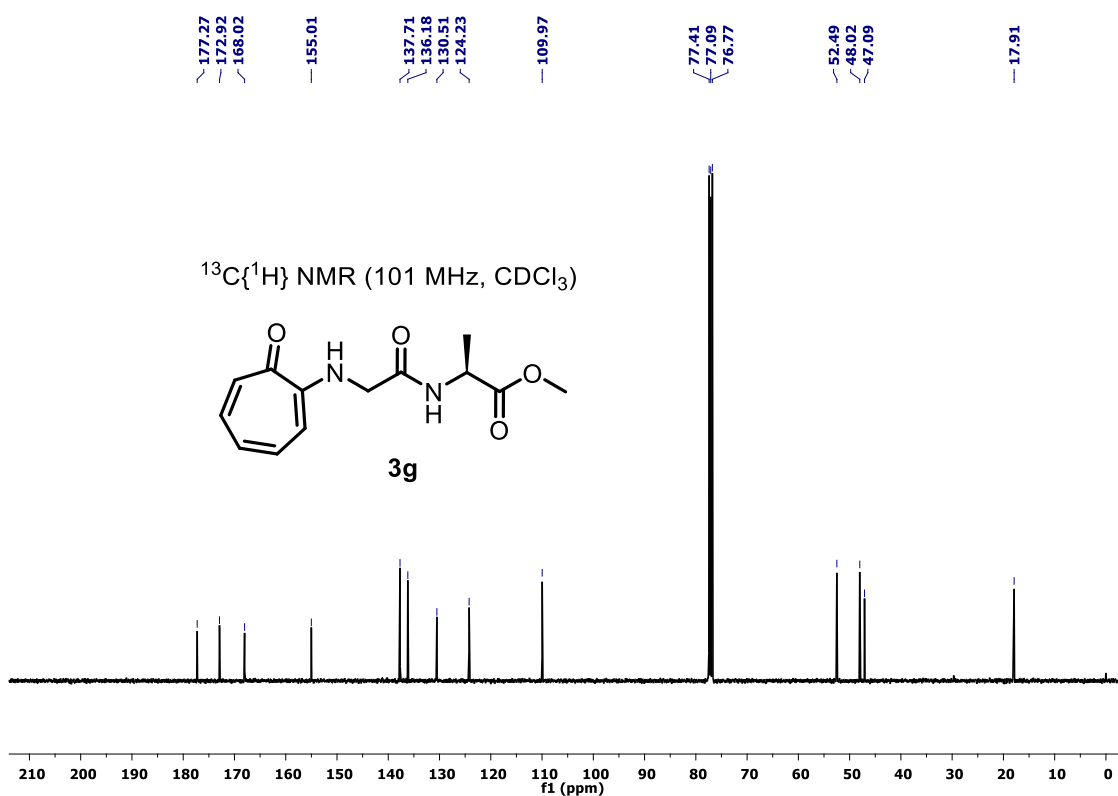
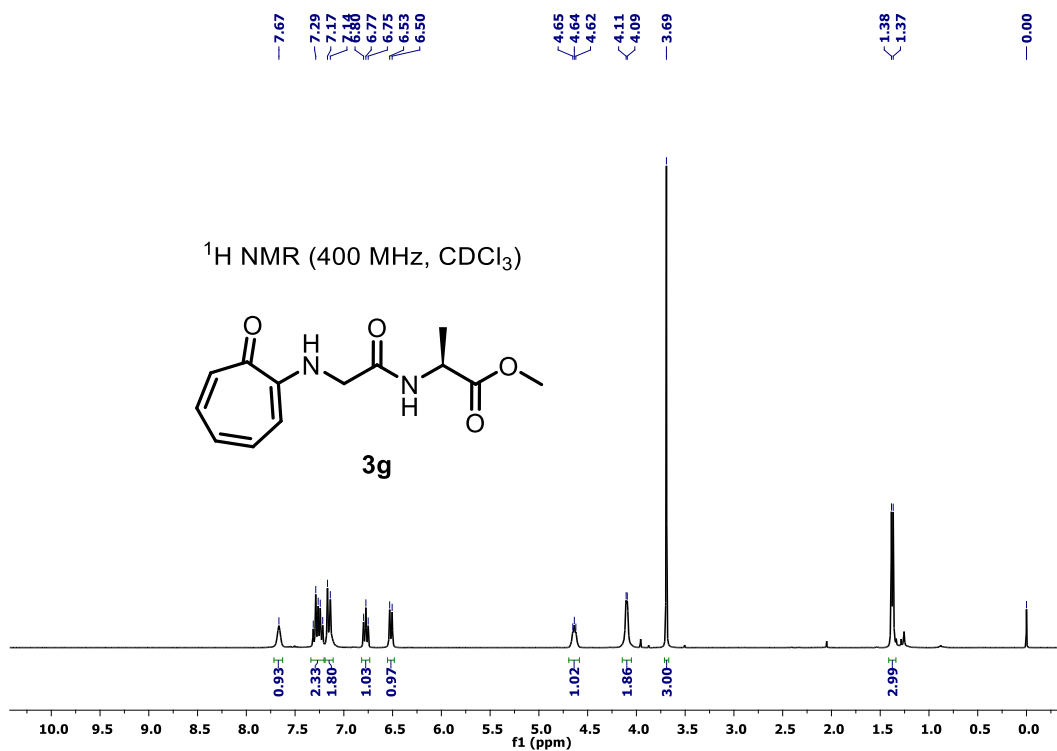


Fig S15. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3g**.

NKS_CKJ_1018

01-Dec-2022
00:54:04

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NKS_30112022_3 (0.053) Cu (0.05); Is (1.00,1.00) C₁₃H₁₆N₂O₄Na

1: TOF MS ES+
8.53e12

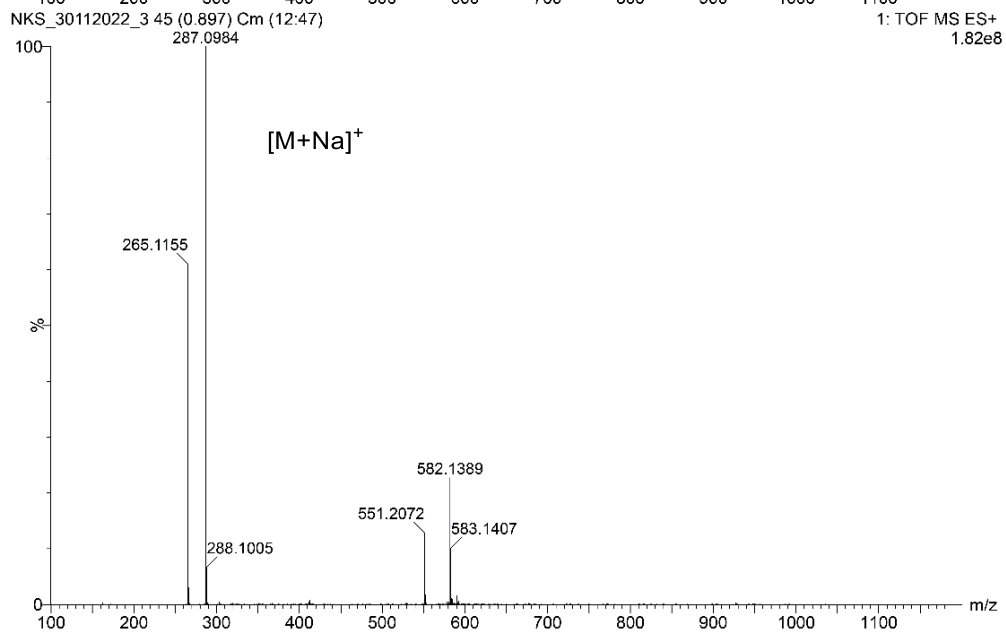
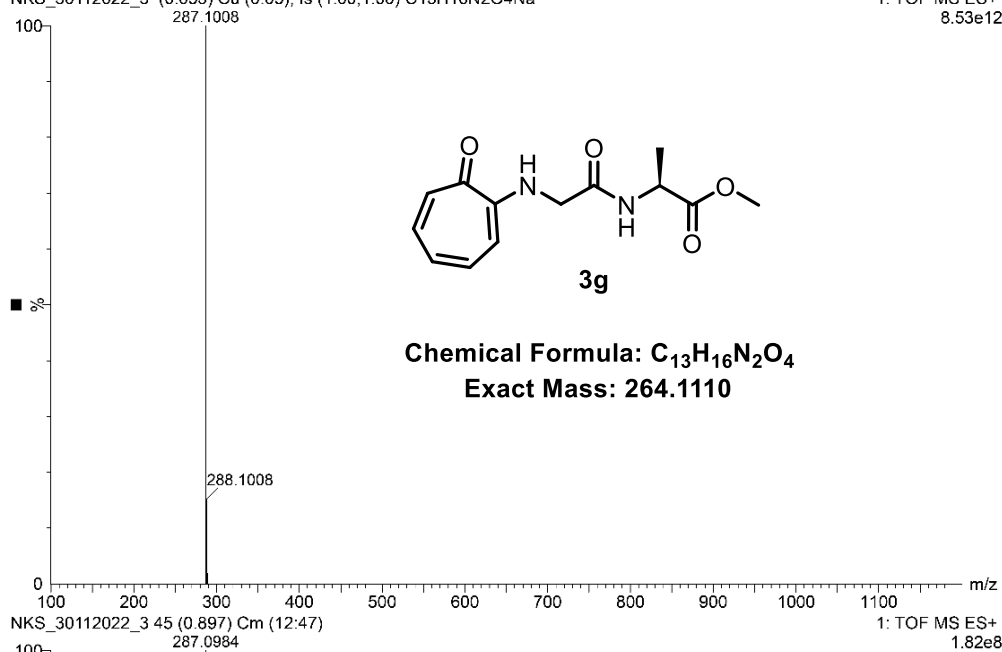


Fig S16. ESI-HRMS spectra of troponyl glycine derivative **3g**.

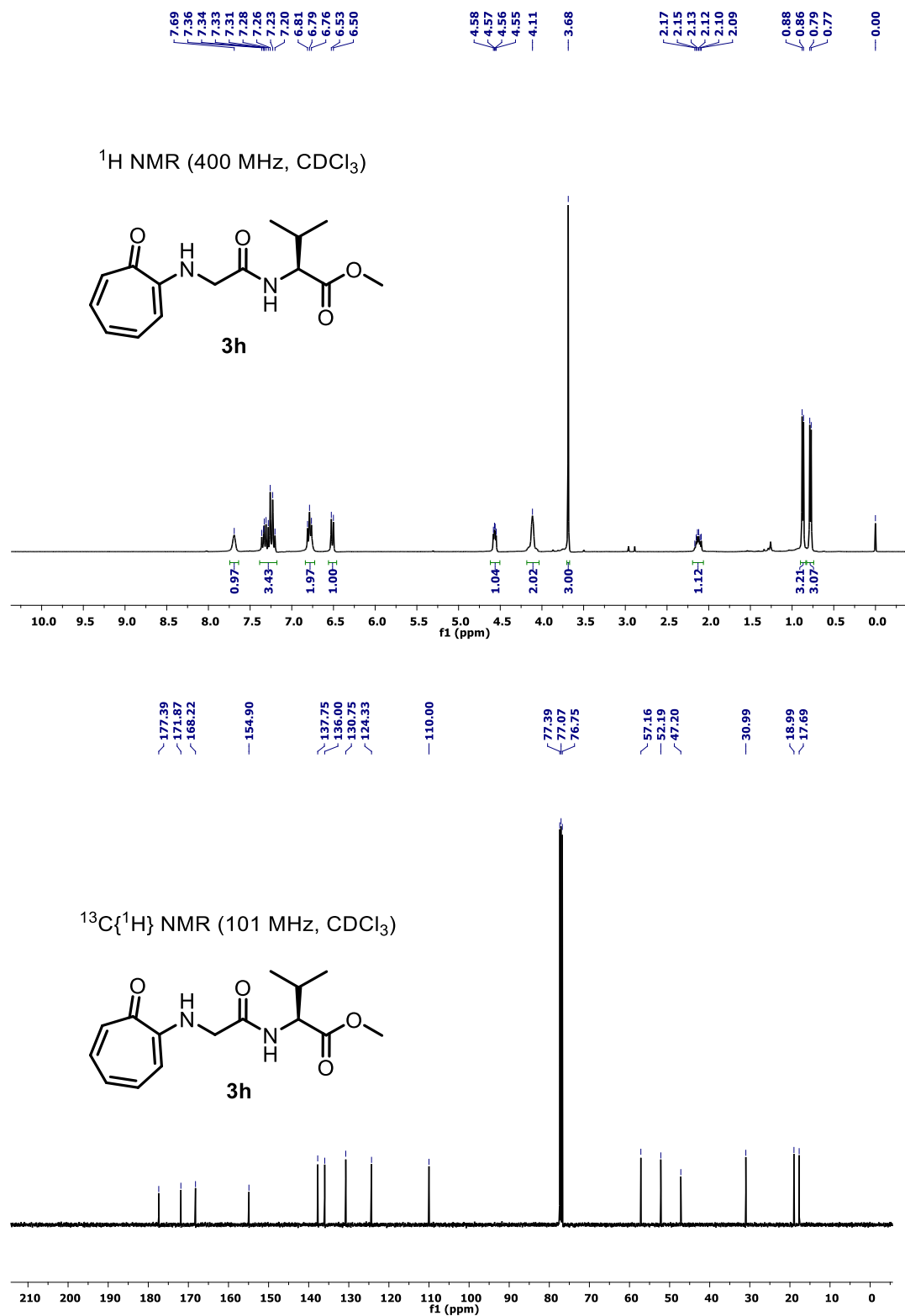


Fig S17. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3h**.

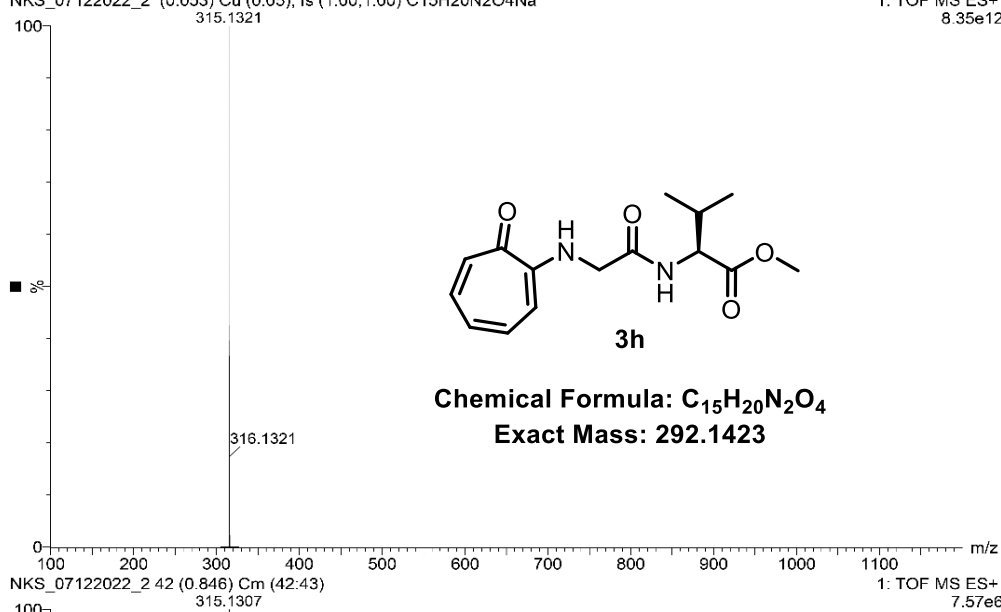
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07-Dec-2022
20:10:26

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1: TOF MS ES+
8.35e12



NKS_07122022_2 42 (0.846) Cm (42.43)

1: TOF MS ES+
7.57e6

Fig S18. ESI-HRMS spectra of troponyl glycine derivative **3h**.

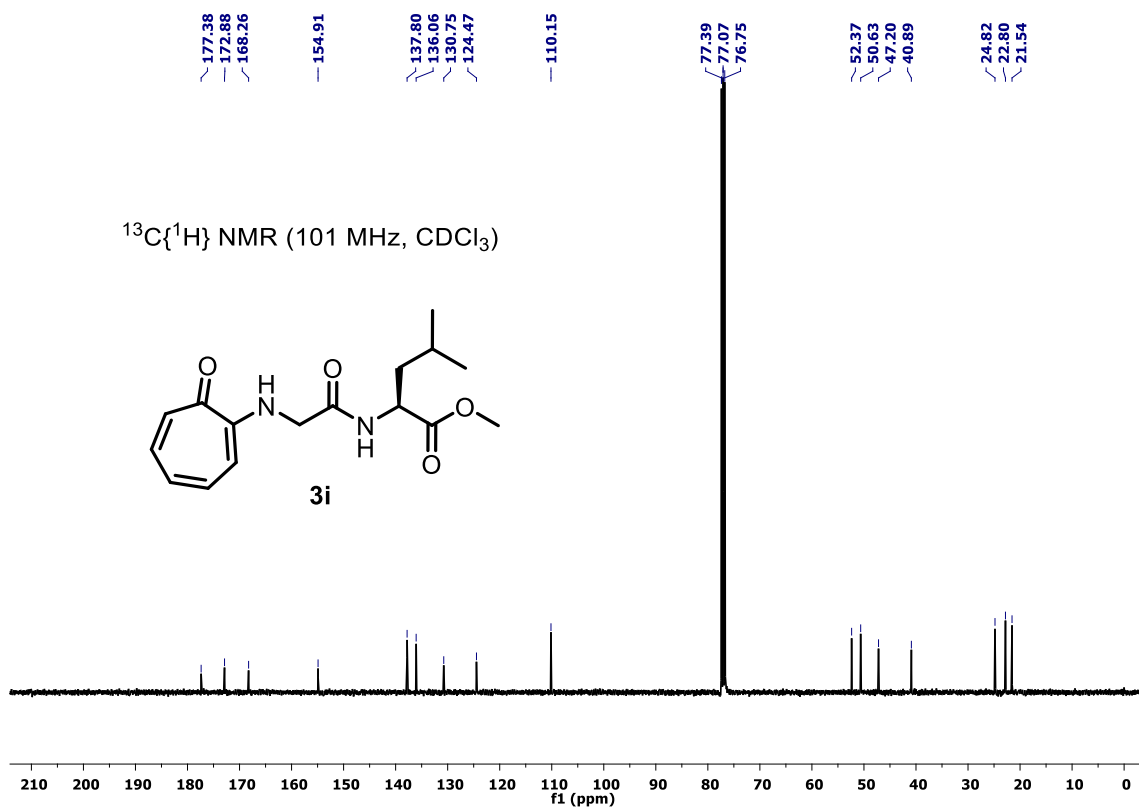
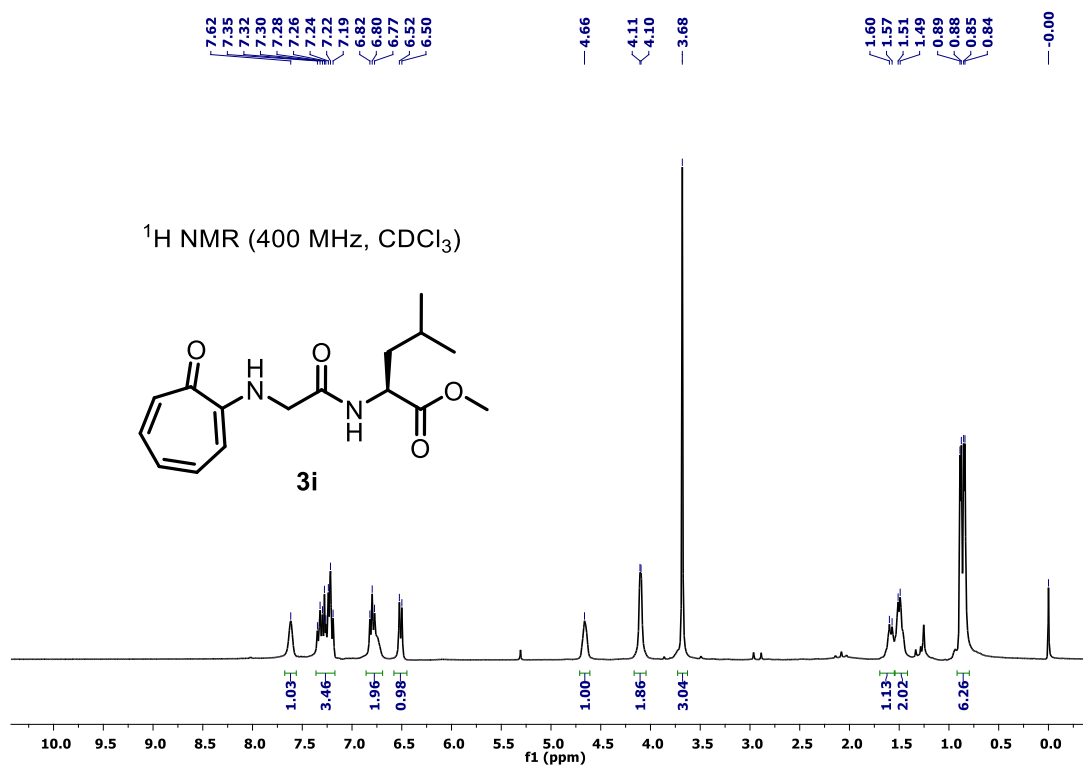


Fig S19. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3i**.

NKS_CKJ_1021

29-Nov-2022
17:51:09

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1: TOF MS ES+
8.25e12

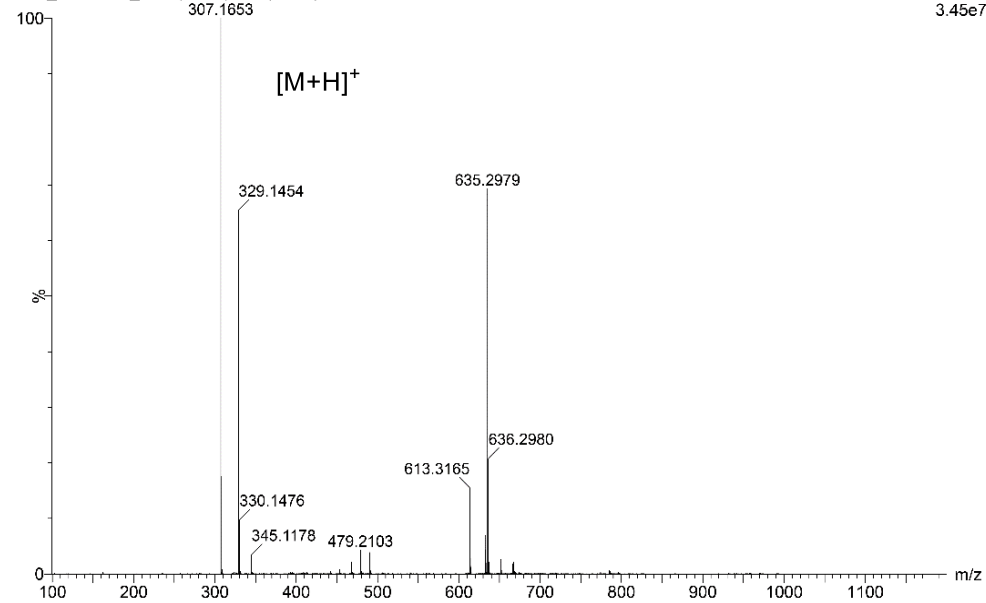
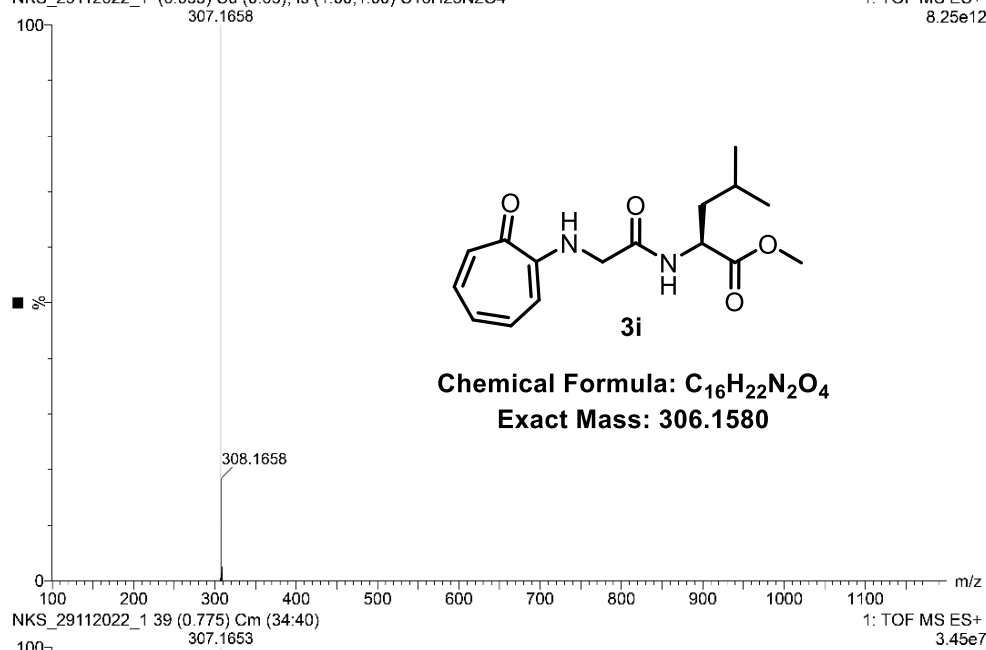


Fig S20. ESI-HRMS spectra of troponyl glycine derivative **3i**.

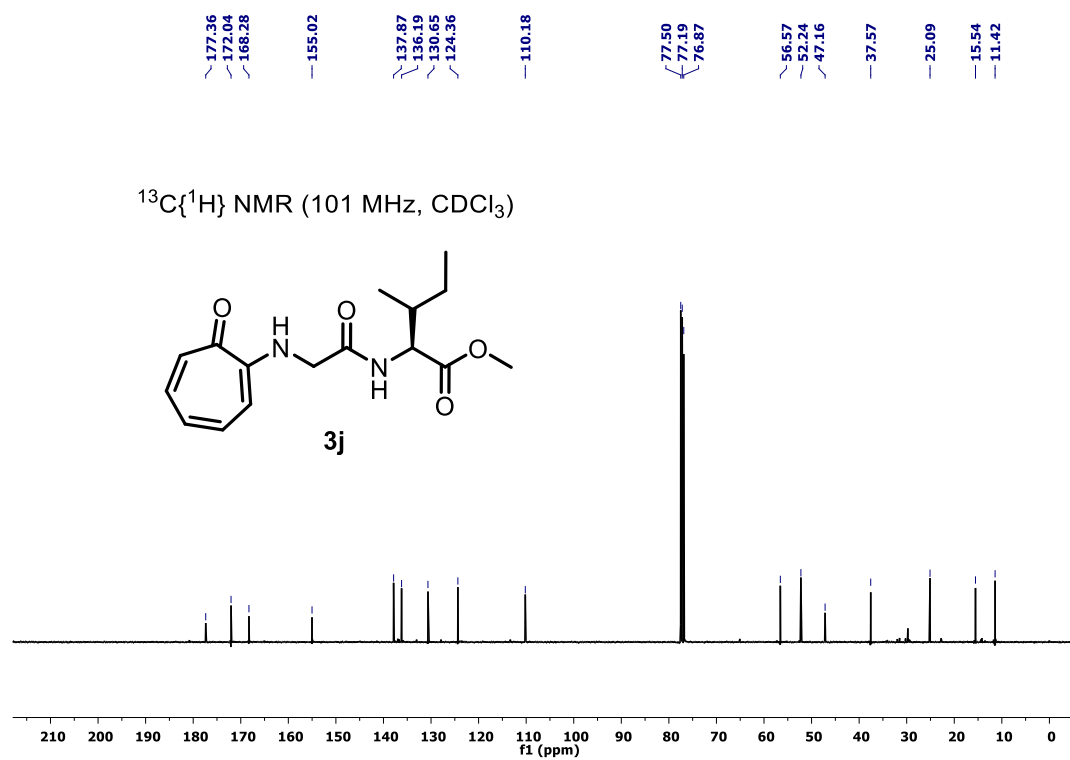
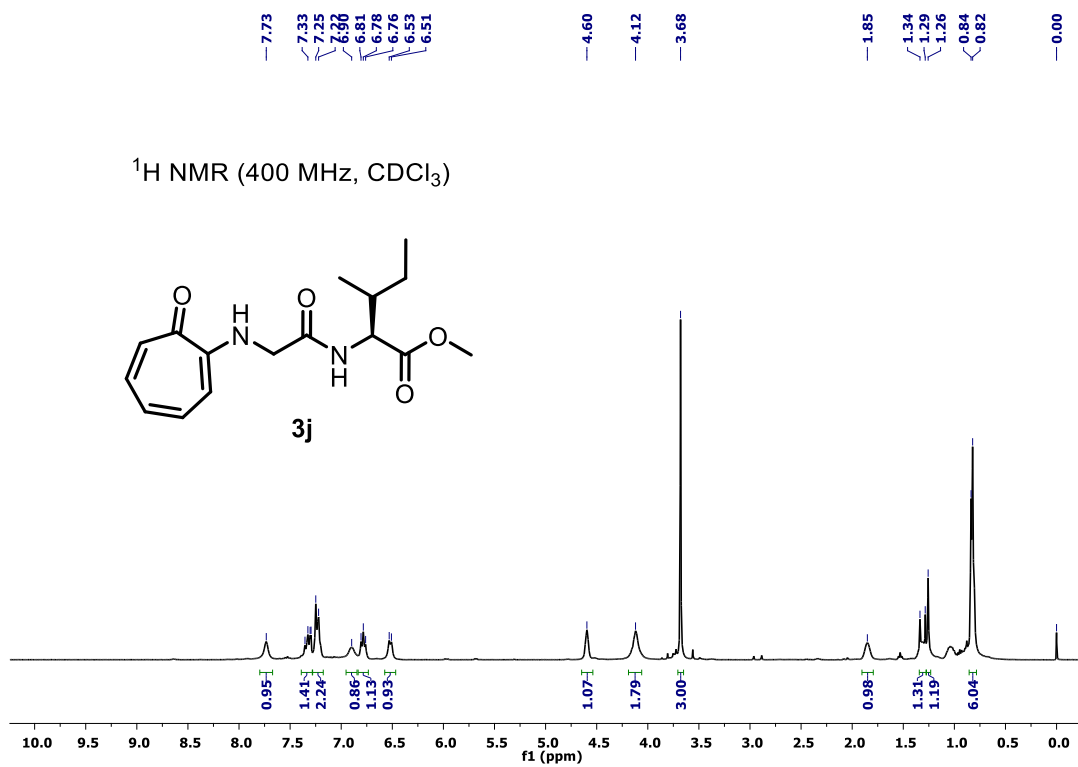


Fig S21. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3j**.

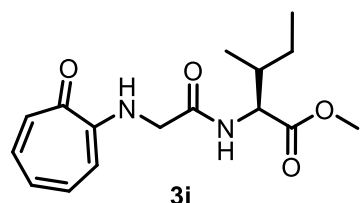
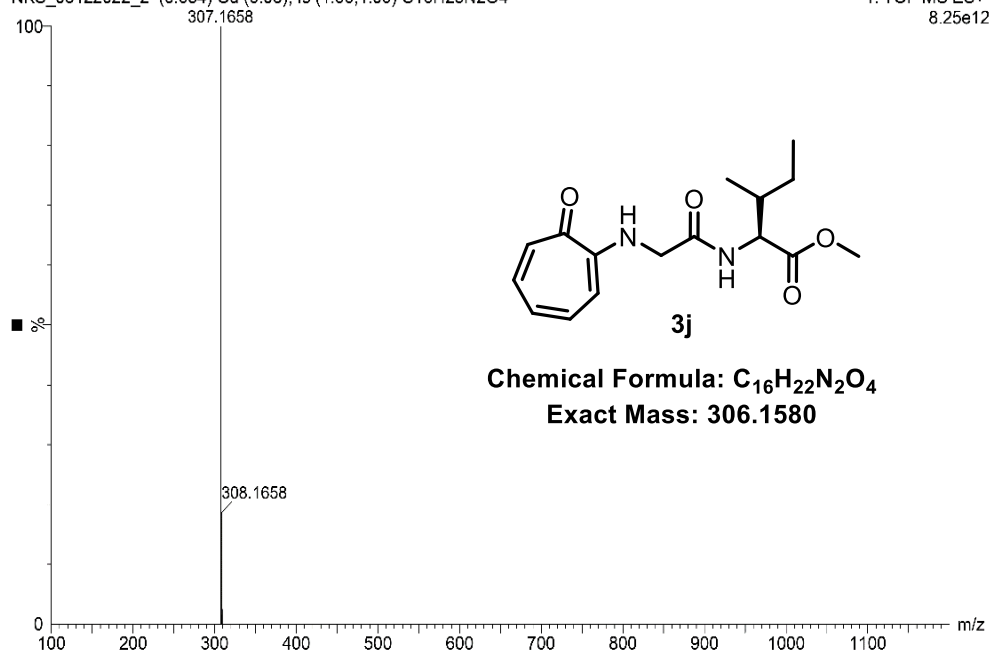
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03-Dec-2022
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1: TOF MS ES+
8.25e12



Chemical Formula: C₁₆H₂₂N₂O₄
Exact Mass: 306.1580

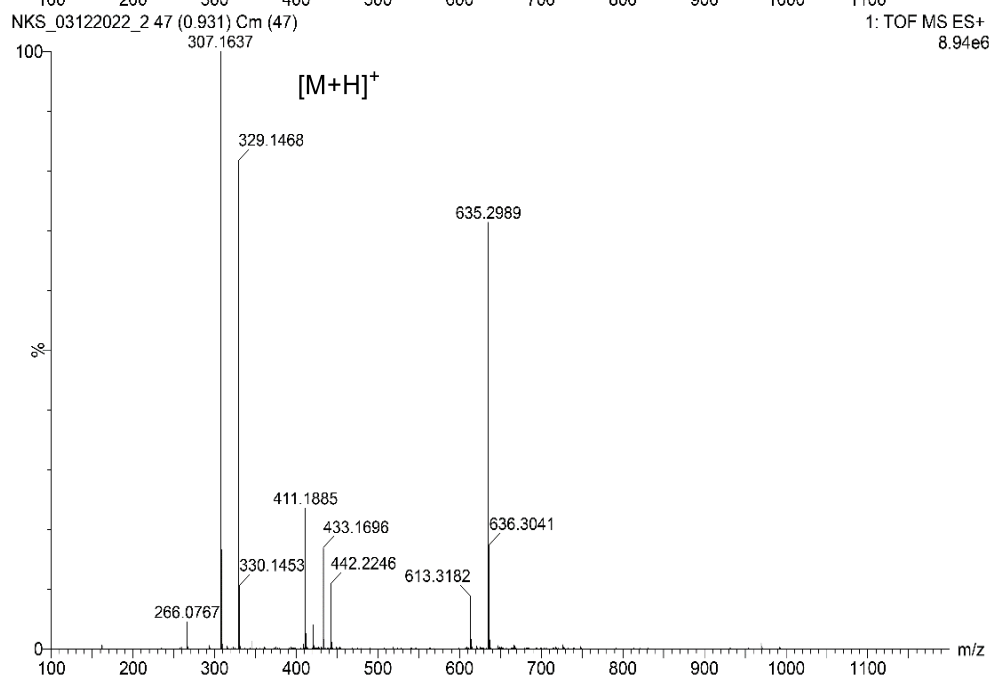


Fig S22. ESI-HRMS spectra of troponyl glycine derivative **3j**.

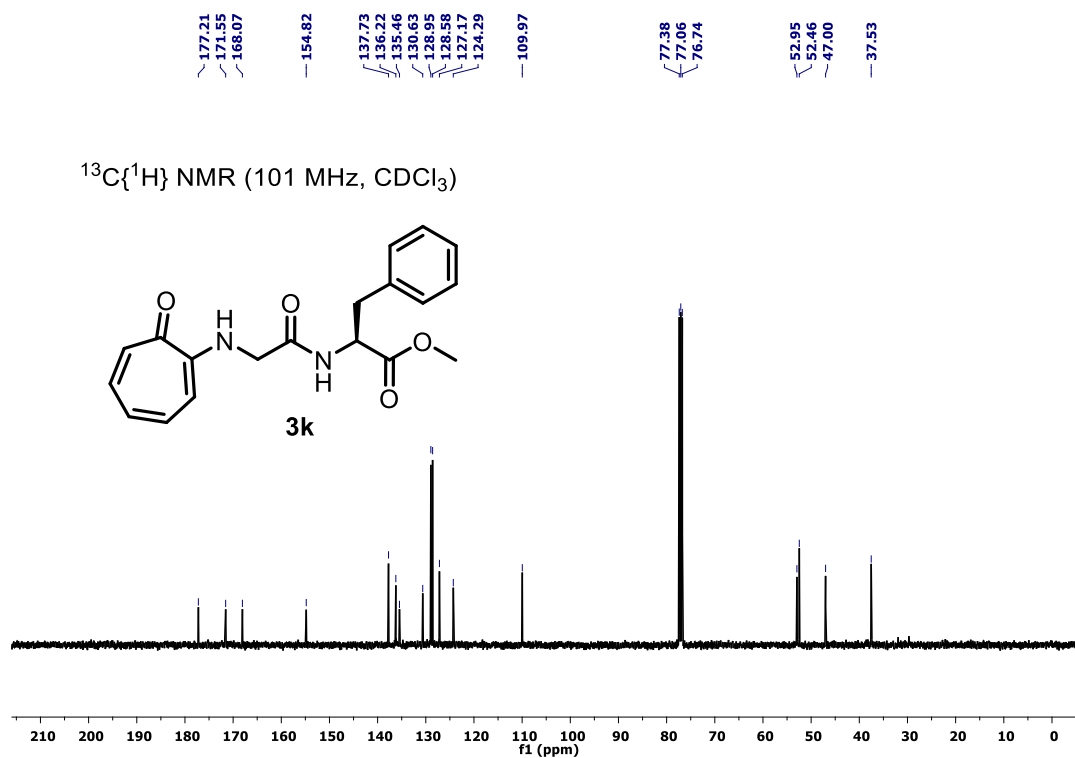
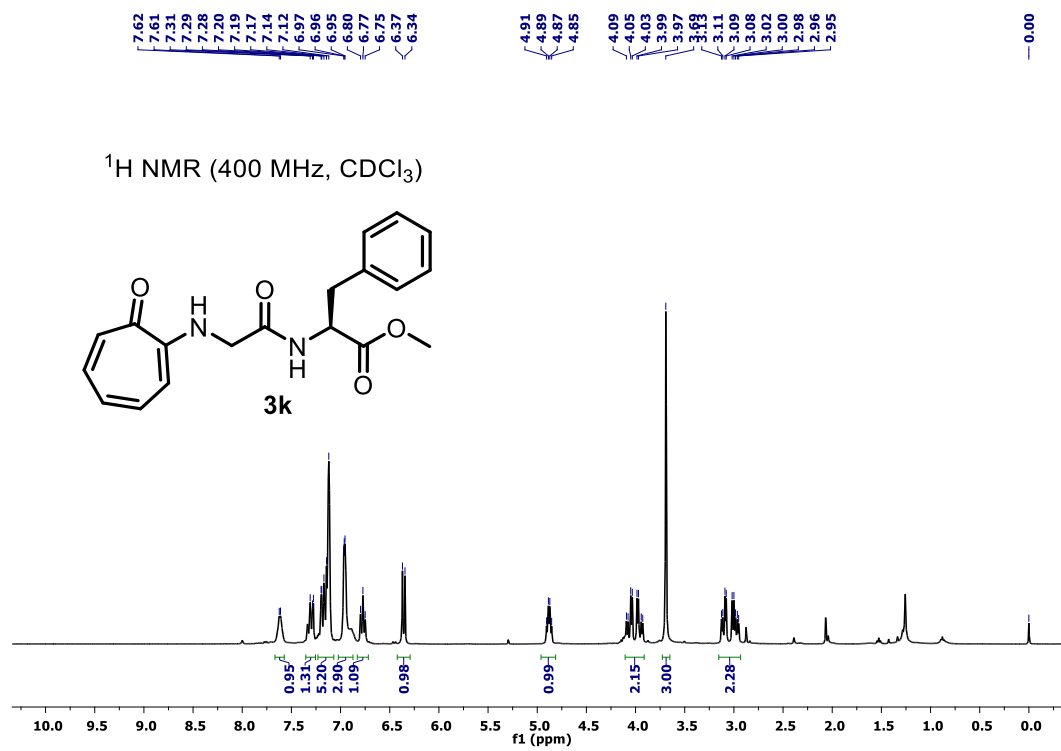


Fig S23. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3k**.

Display Report

Analysis Info

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Method Pos_tune_low_05122019.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/17/2021 4:13:18 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

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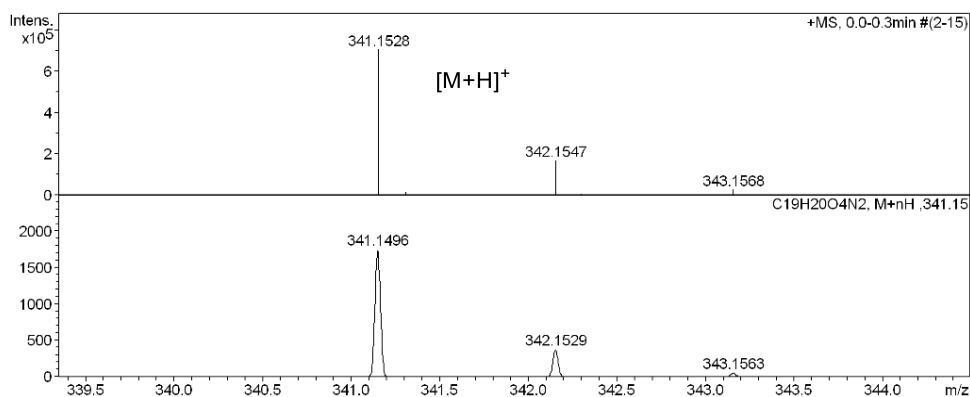
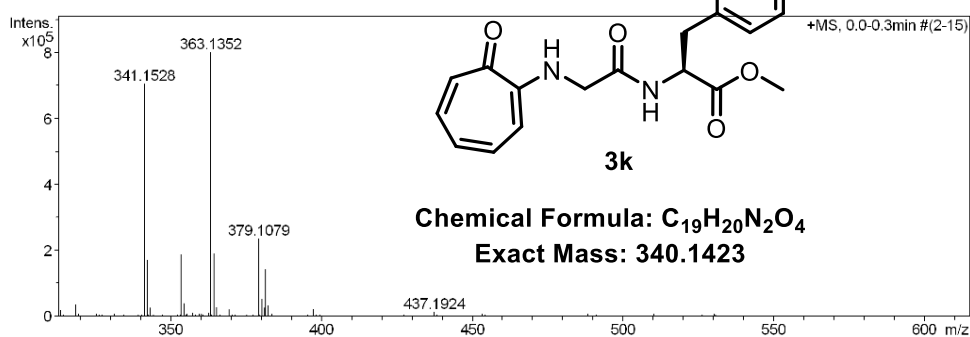
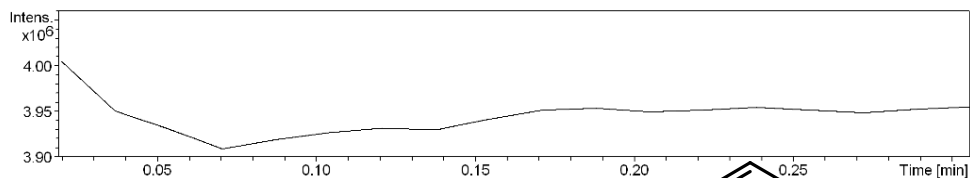


Fig S24. ESI-HRMS spectra of troponyl glycine derivative **3k**.

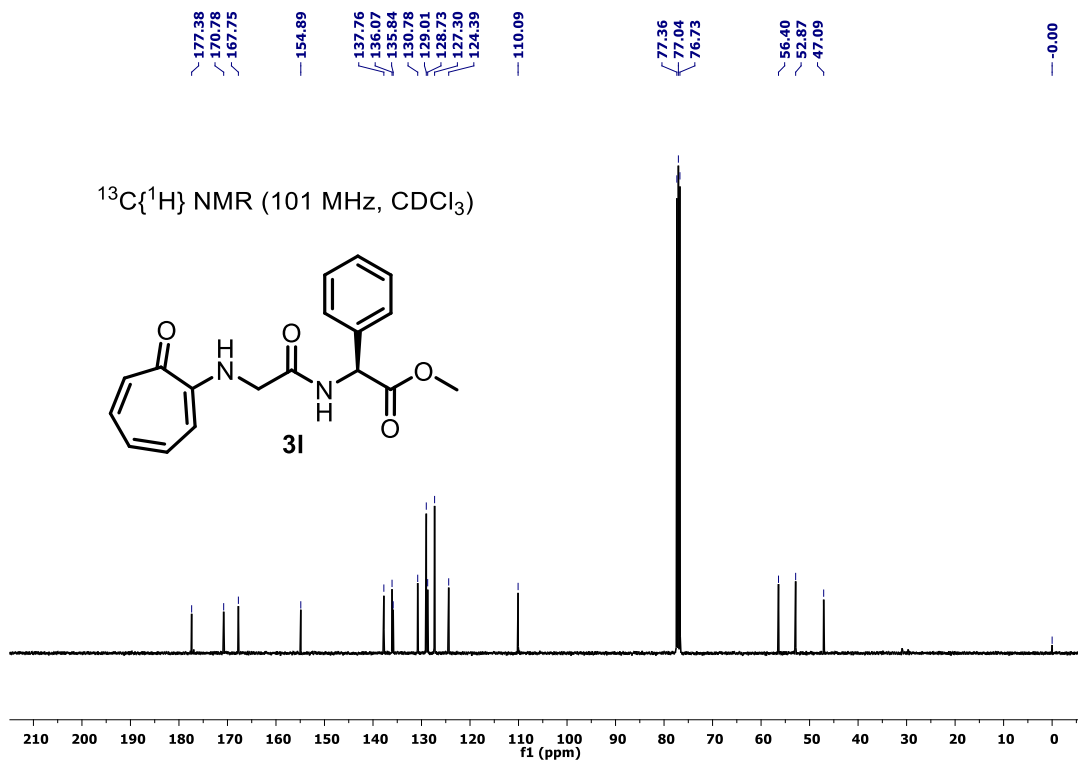
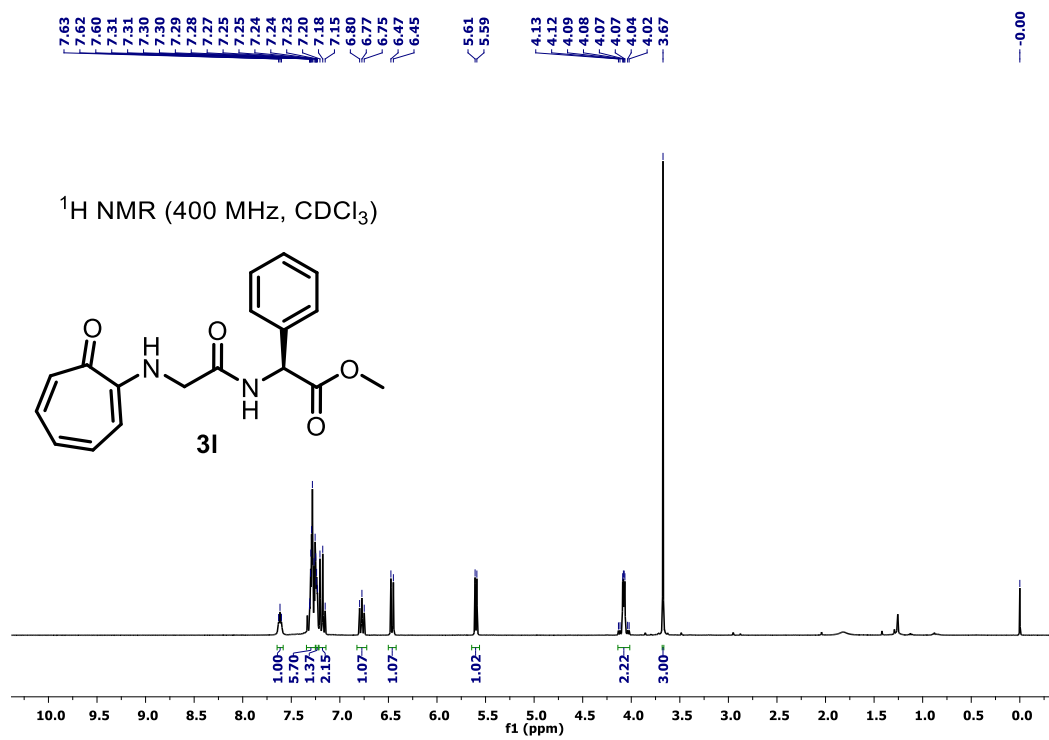


Fig S25. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3I**.

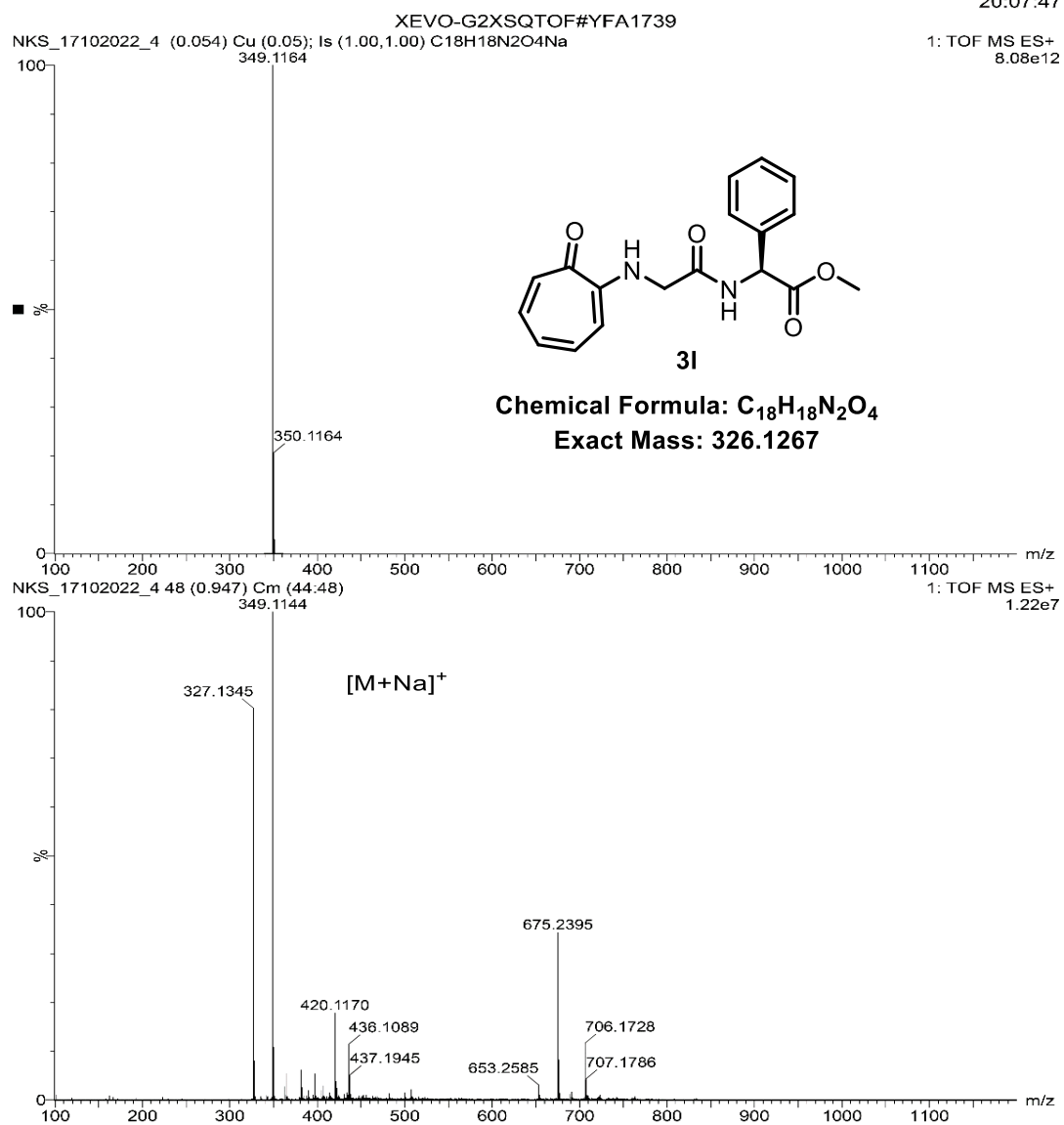


Fig S26. ESI-HRMS spectra of troponyl glycine derivative **3I**.

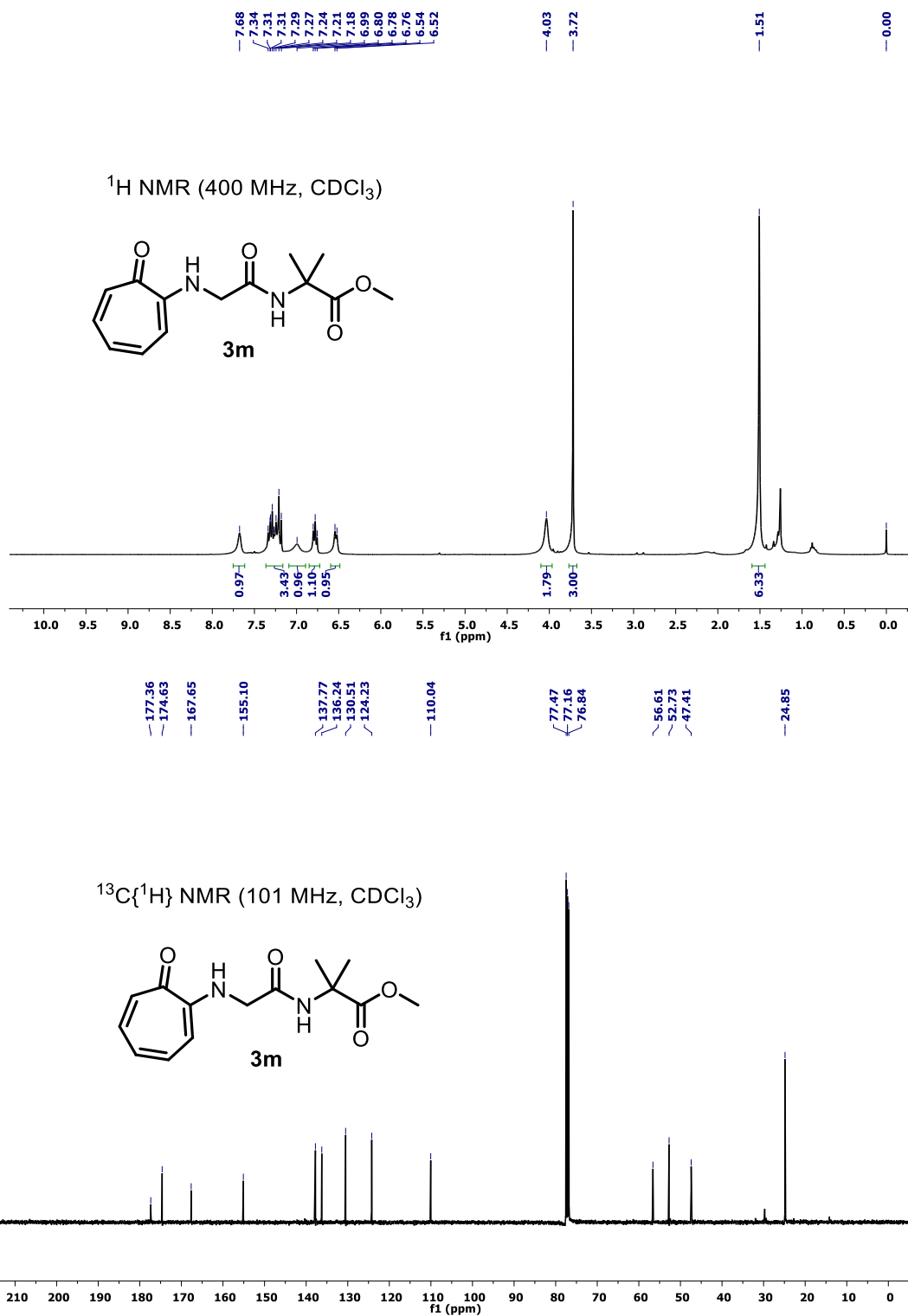


Fig S27. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3m**.

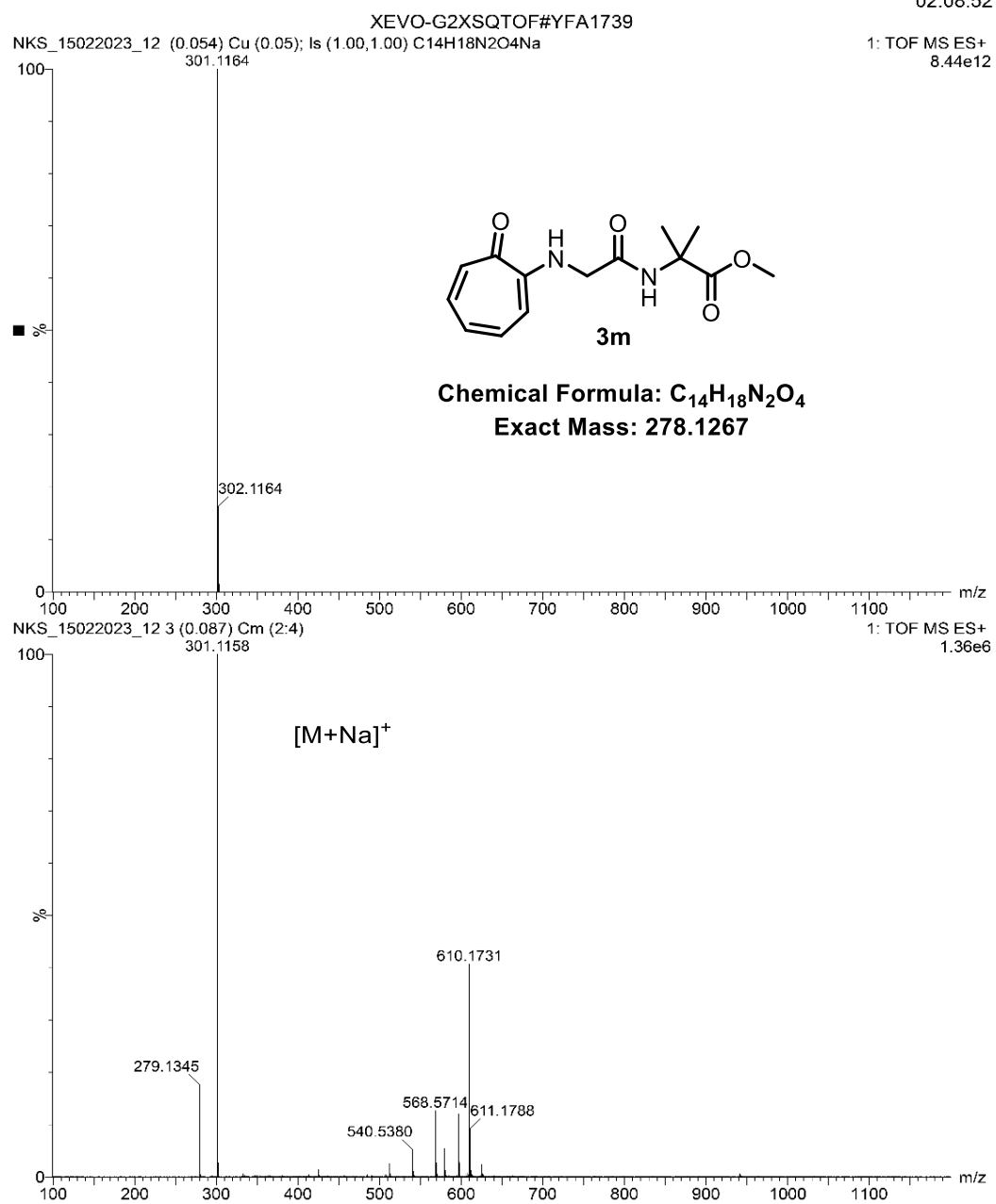


Fig S28. ESI-HRMS spectra of troponyl glycine derivative **3m**.

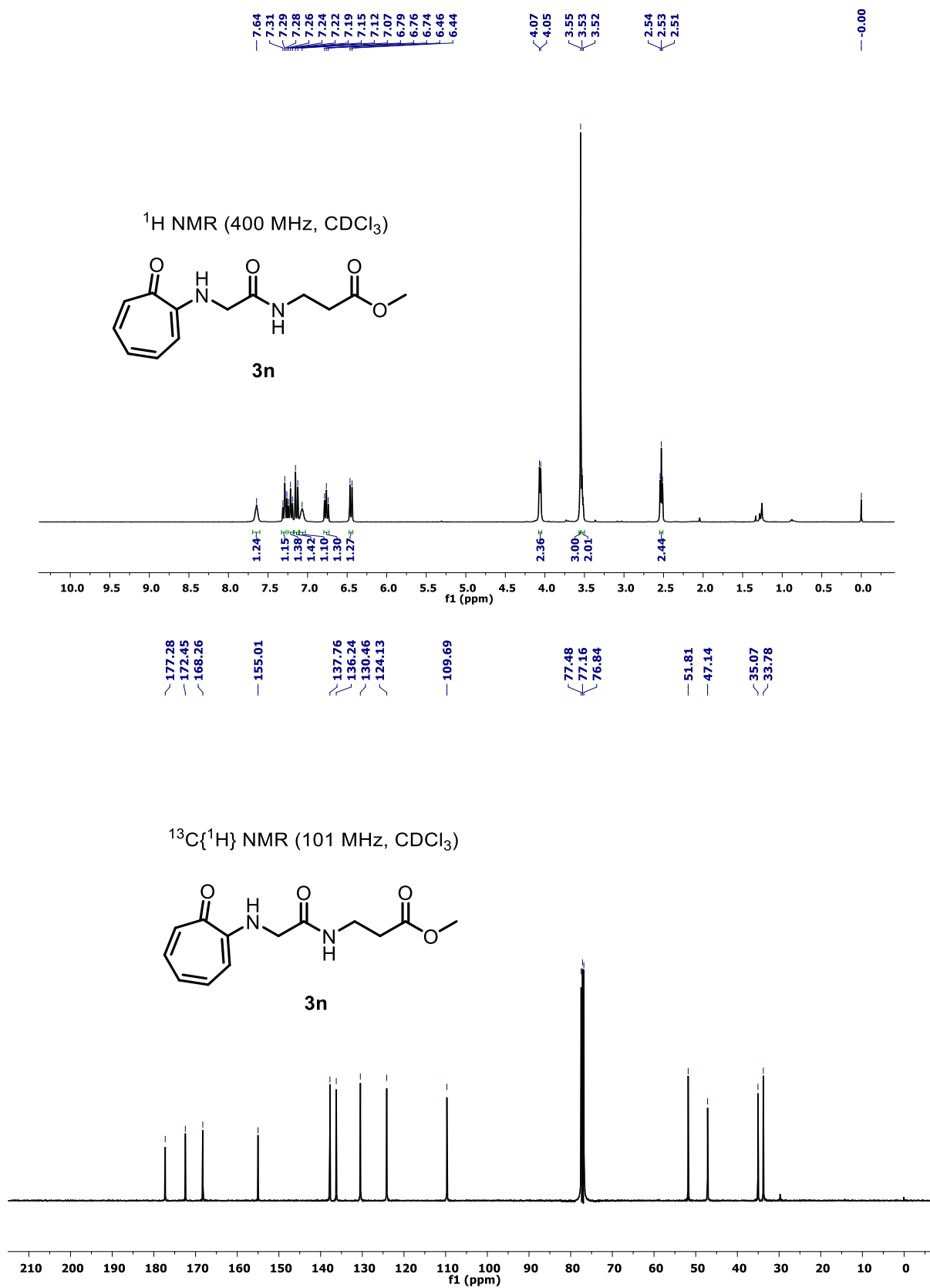


Fig S29. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3n**.

NKS_CKJ_1025 A

04-Dec-2022
00:42:46

XEVO-G2XSQTOF#YFA1739

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8.53e12

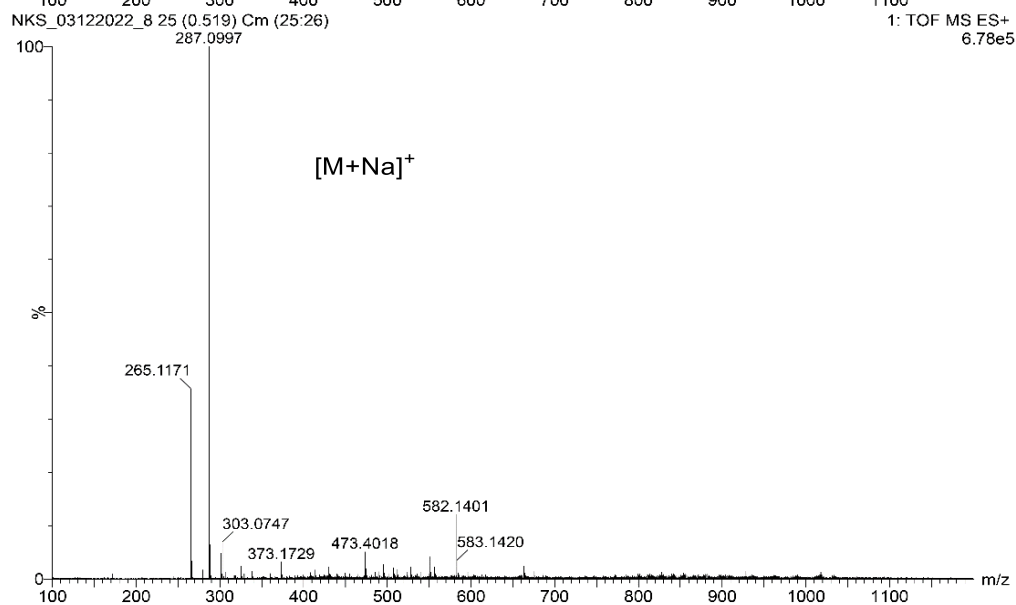
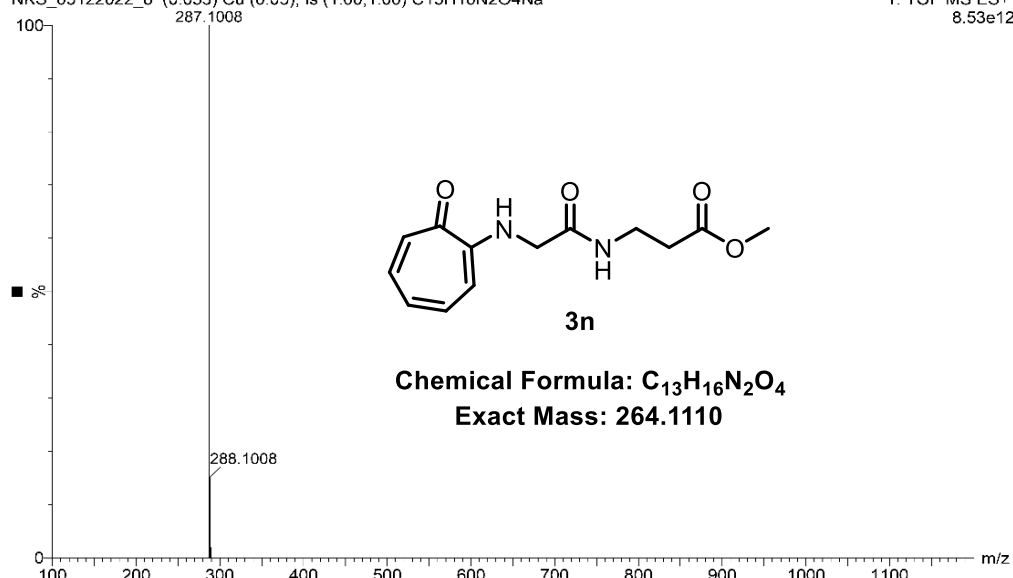


Fig S30. ESI-HRMS spectra of troponyl glycine derivative **3n**.

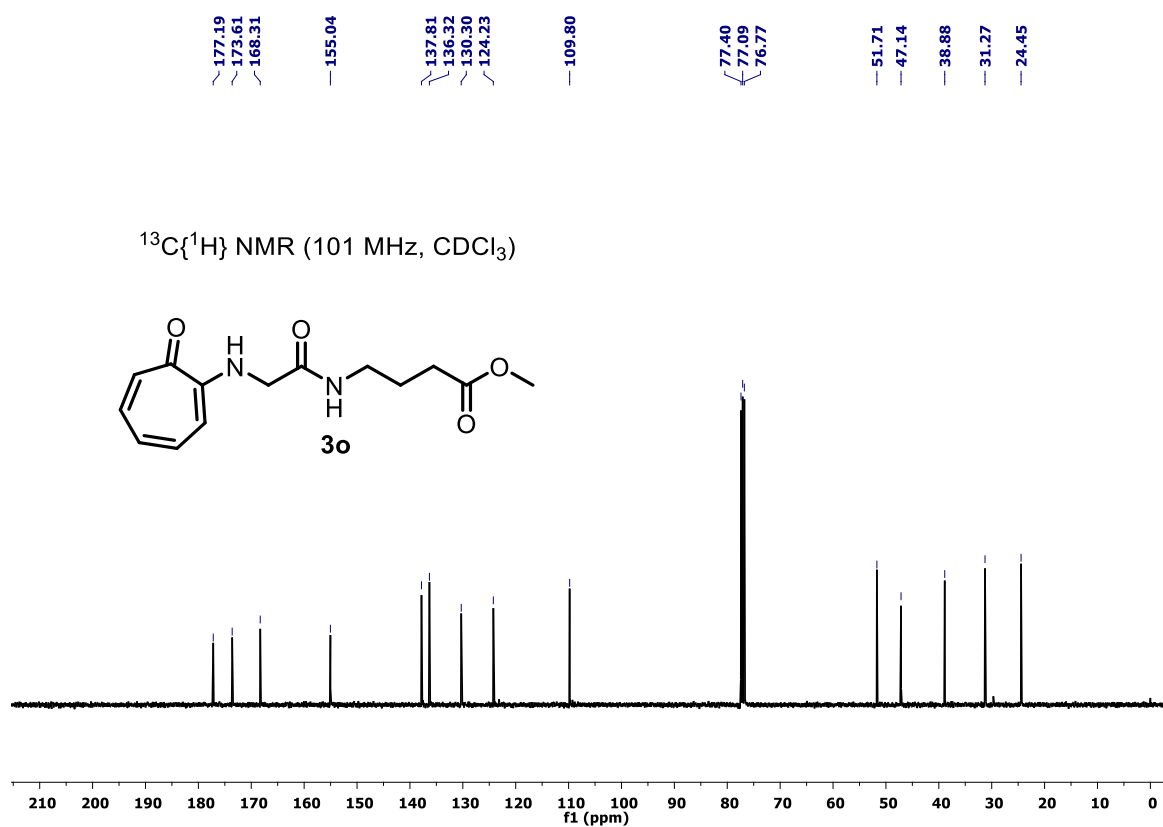
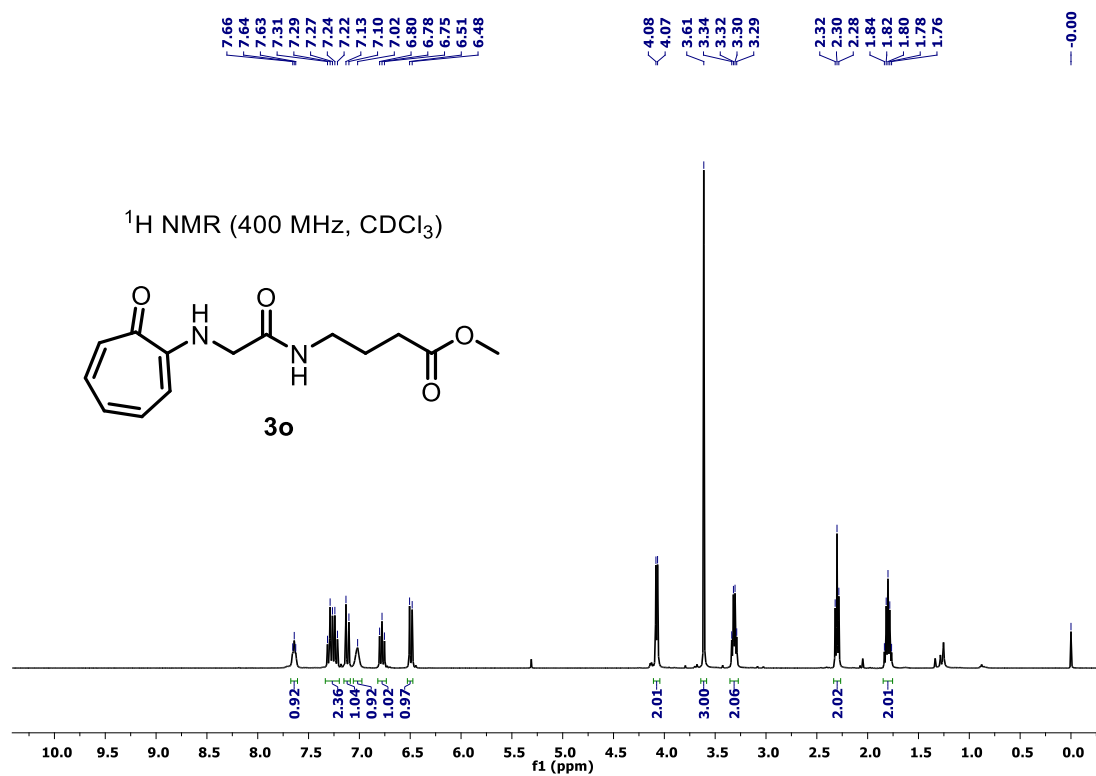


Fig S31. ¹H, ¹³C {¹H} NMR spectra of troponyl glycine derivative **3o**.

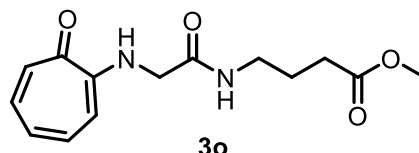
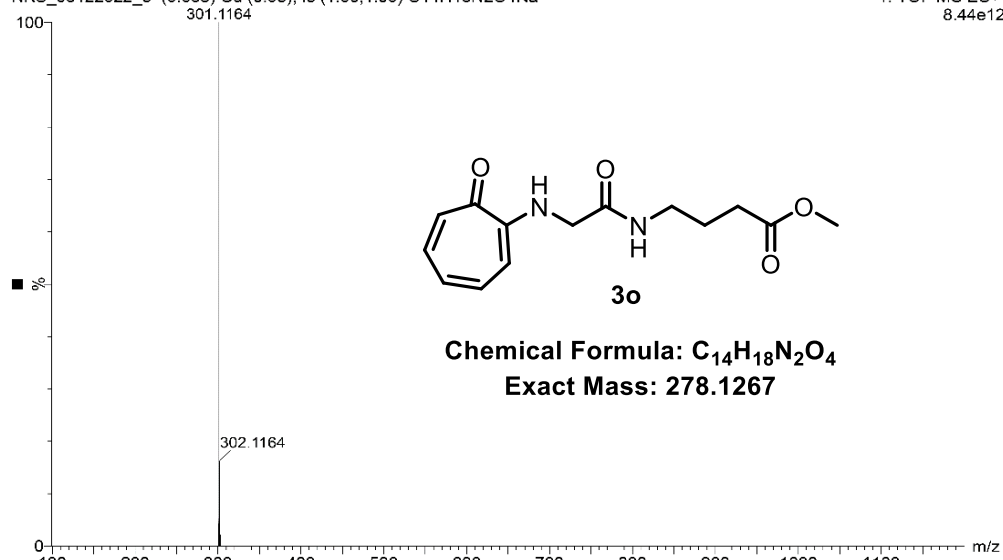
NKS_CKJ_1023 A

04-Dec-2022
00:08:18

XEVO-G2XSQTOF#YFA1739

NKS_03122022_5 (0.053) Cu (0.05); Is (1.00,1.00) C₁₄H₁₈N₂O₄Na

1: TOF MS ES+
8.44e12



Chemical Formula: C₁₄H₁₈N₂O₄
Exact Mass: 278.1267

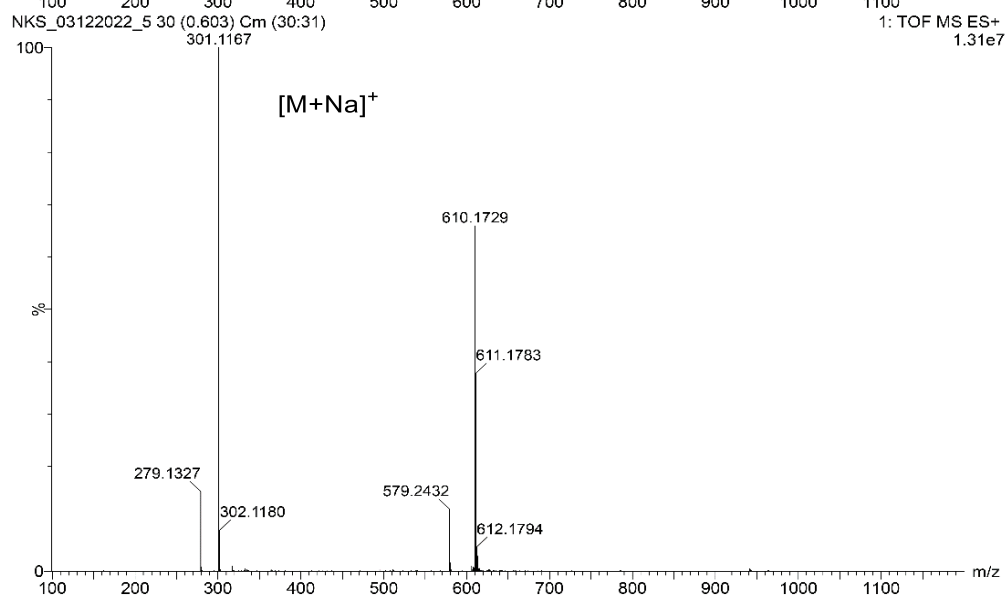


Fig S32. ESI-HRMS spectra of troponyl glycine derivative **3o**.

2. NMR and Mass Spectra of Oxidized Troponyl Glycine Derivatives (2a/ 4a-4o)

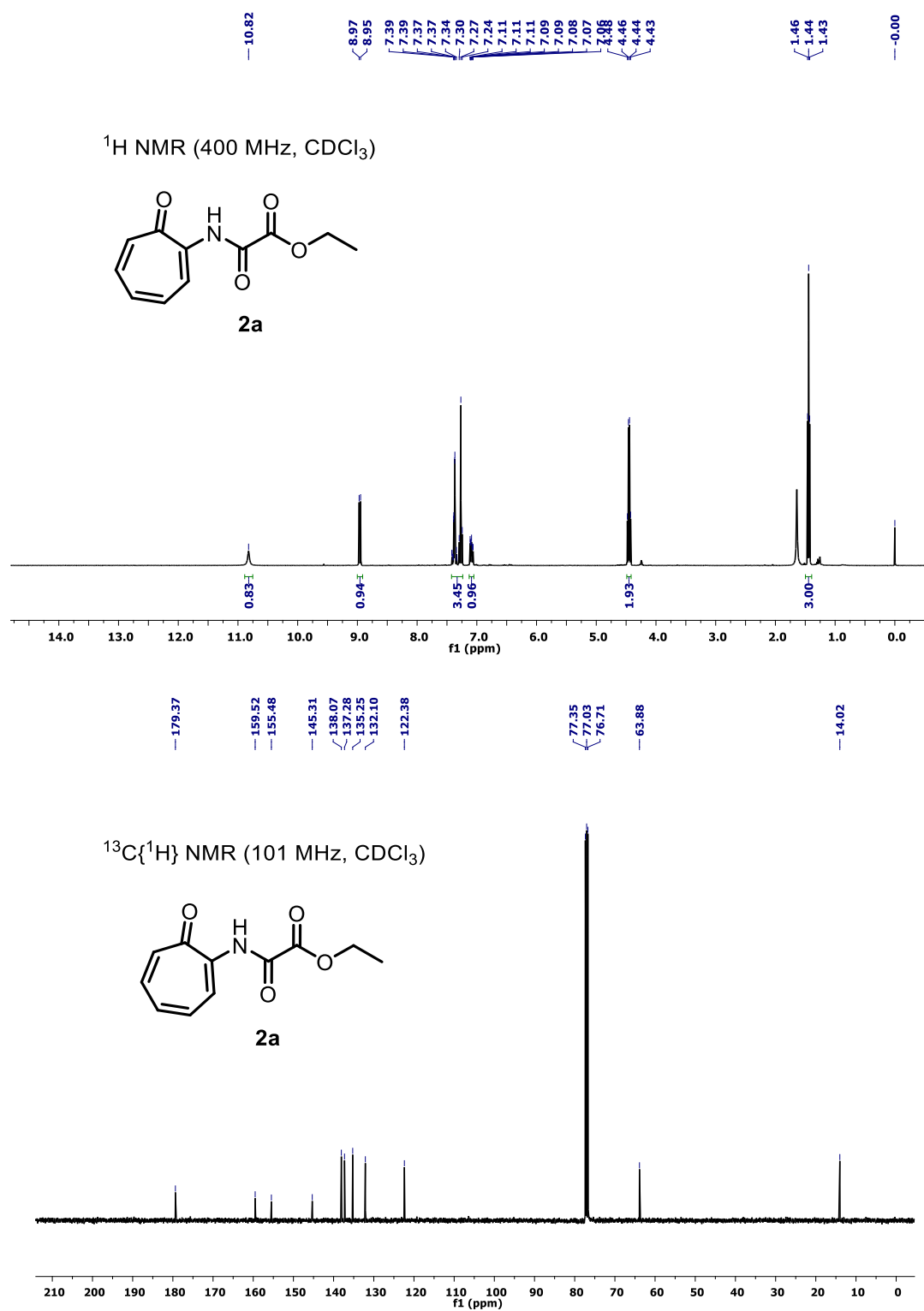


Fig S33. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **2a**.

NKS-CKJ-880

17-Oct-2022
18:58:06

XEVO-G2XSQTOF#YFA1739

NKS_17102022_1 (0.053) Cu (0.05); Is (1.00,1.00) C₁₁H₁₁NO₄Na

1: TOF MS ES+
8.75e12

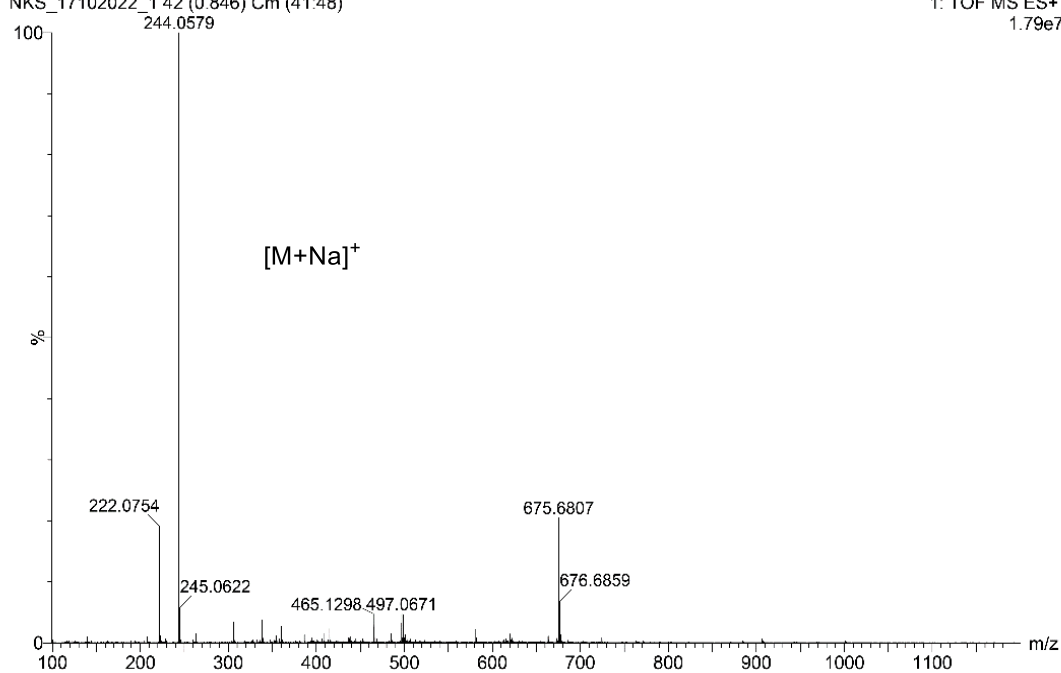
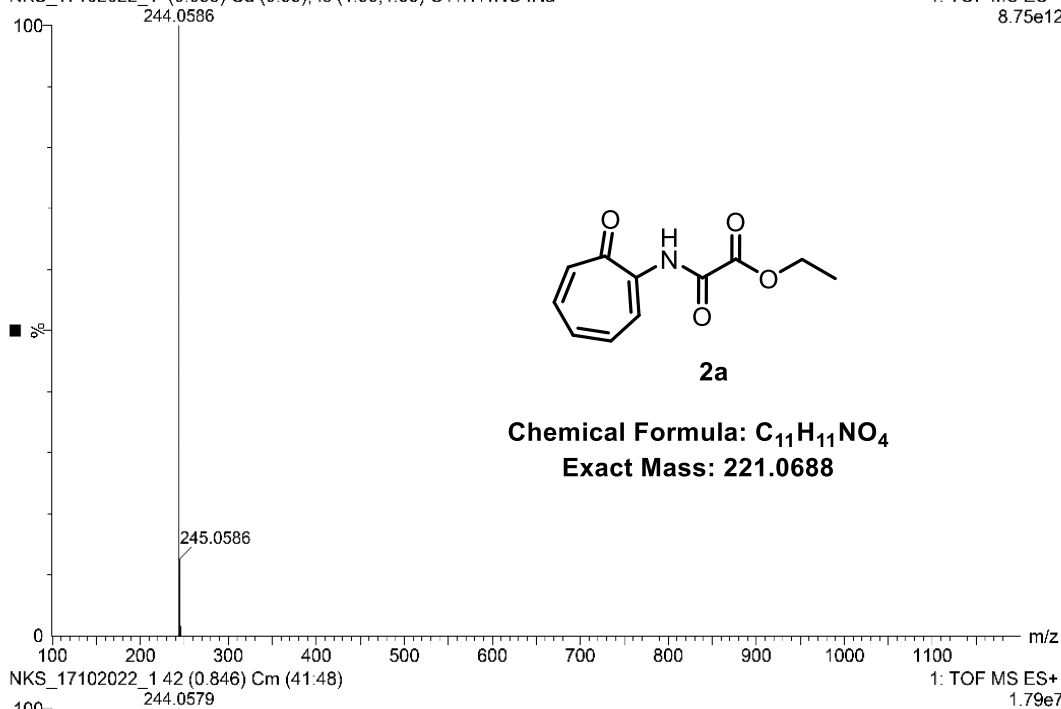


Fig S34. ESI-HRMS spectra of oxidized troponyl glycine derivative **2a**.

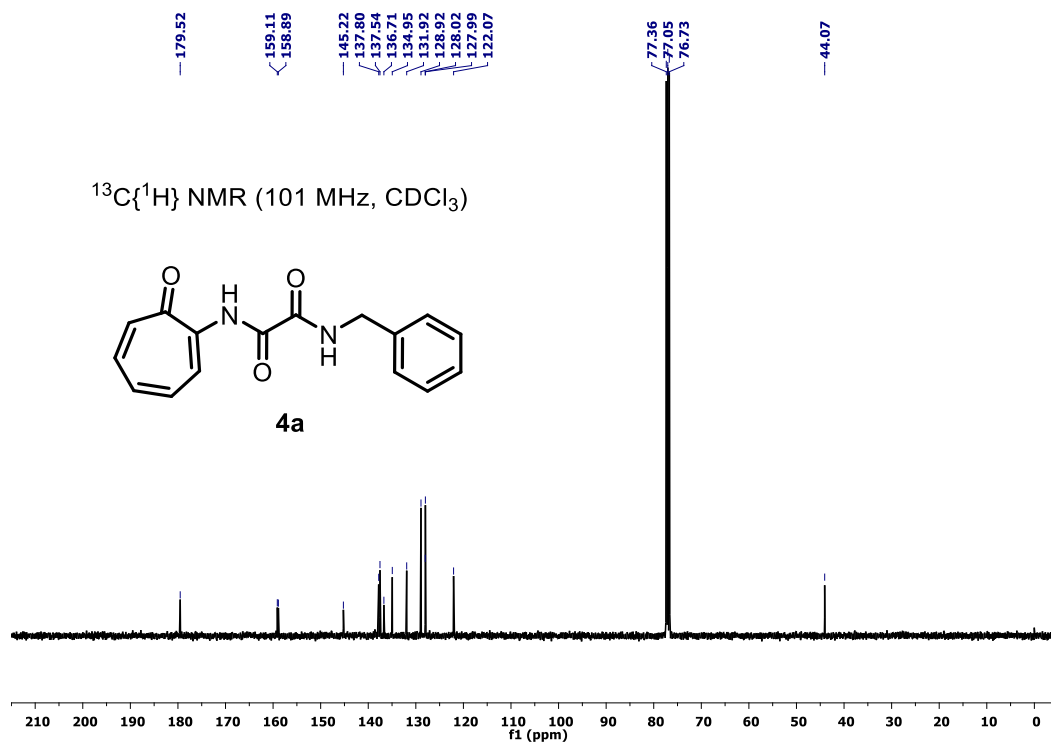
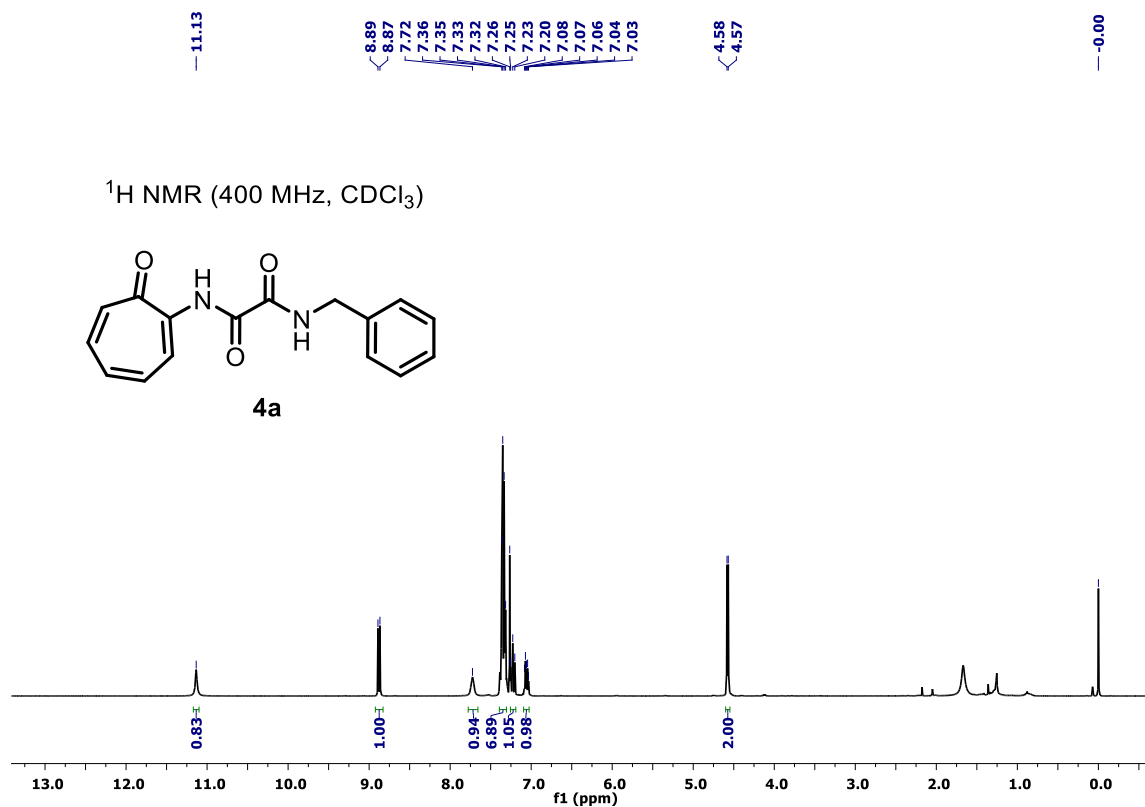


Fig S35. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4a**.

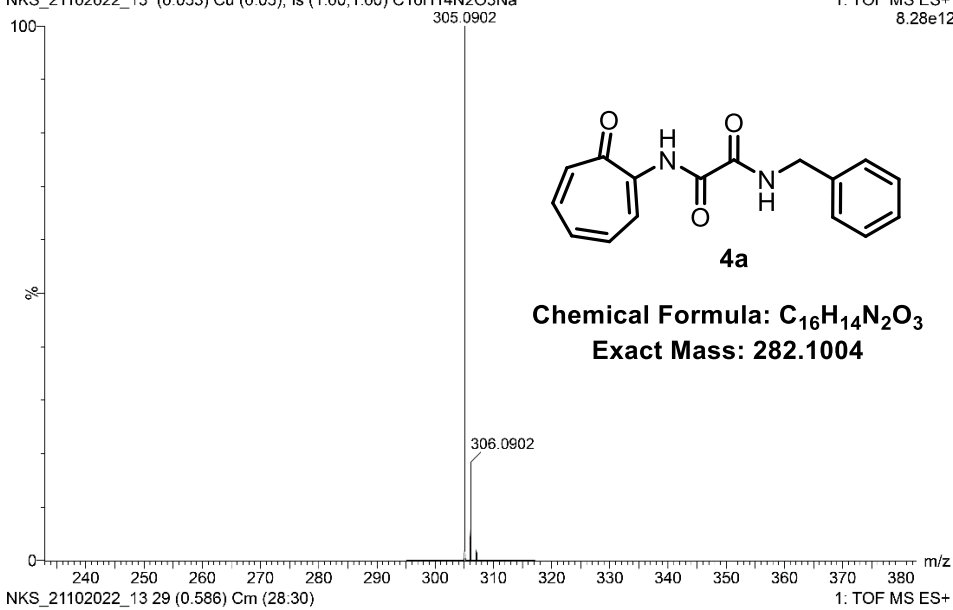
NKS_CKJ_988 A

21-Oct-2022
17:44:55

XEVO-G2XSQTOF#YFA1739

NKS_21102022_13 (0.053) Cu (0.05); Is (1.00,1.00) C₁₆H₁₄N₂O₃Na

1: TOF MS ES+
8.28e12



NKS_21102022_13 29 (0.586) Cm (28.30)

1: TOF MS ES+
1.43e7

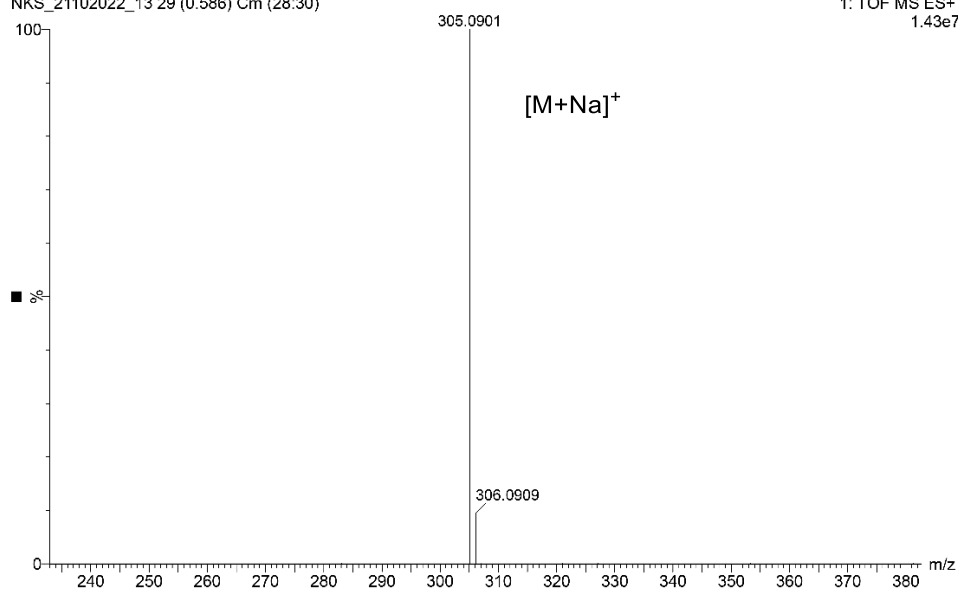


Fig S36. ESI-HRMS spectra of oxidized troponyl glycine derivative **4a**.

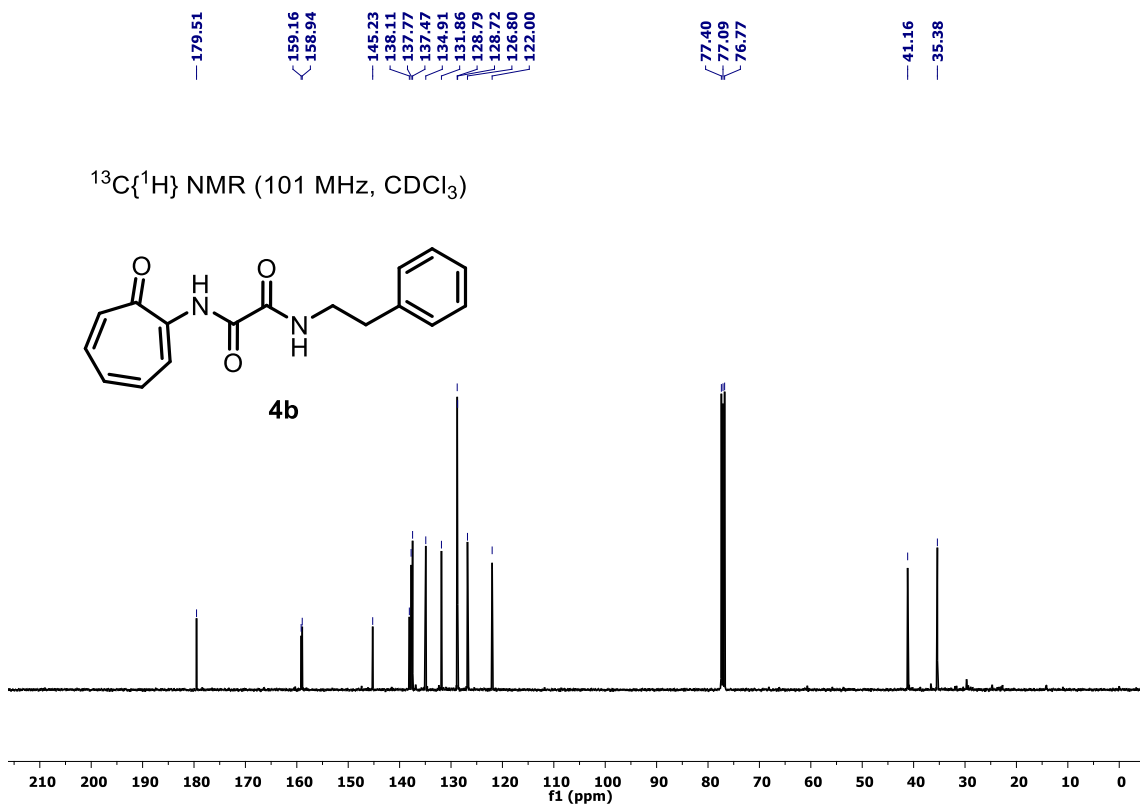
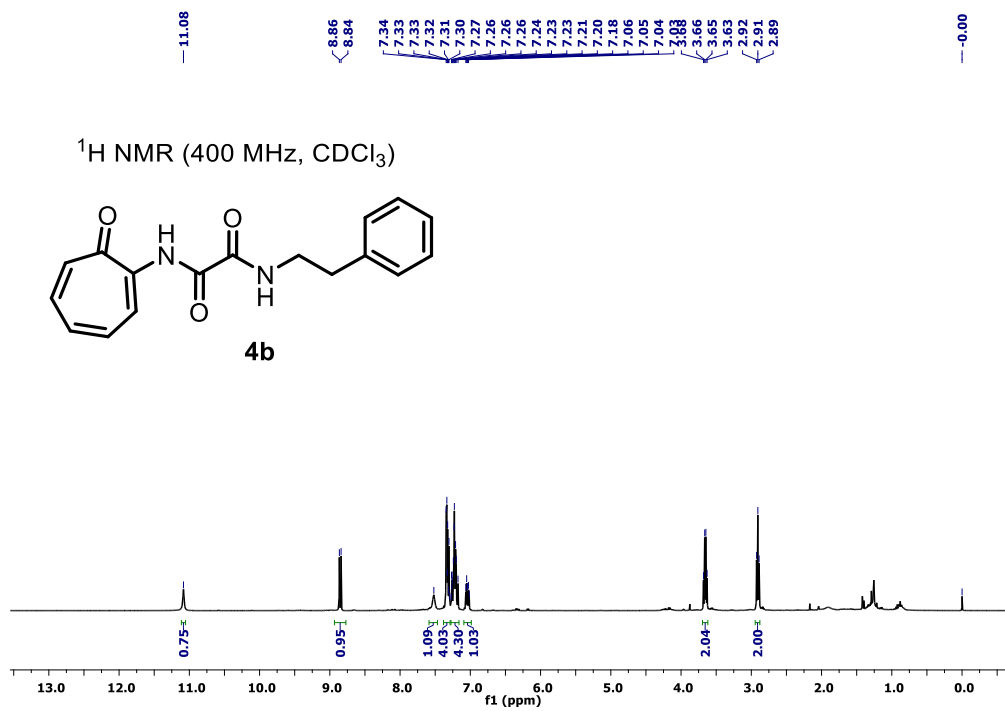


Fig S37. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4b**.

Display Report

Analysis Info

Analysis Name D:\Data\JULY-2021\NKS\17072021_-NKS-CKJ-420(A)-RE.d
Method Pos_tune_low_05122019.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/17/2021 5:20:50 PM
Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

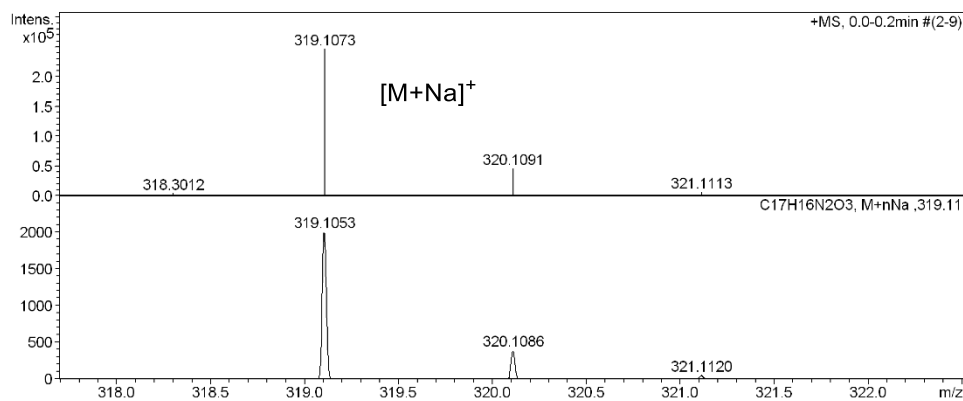
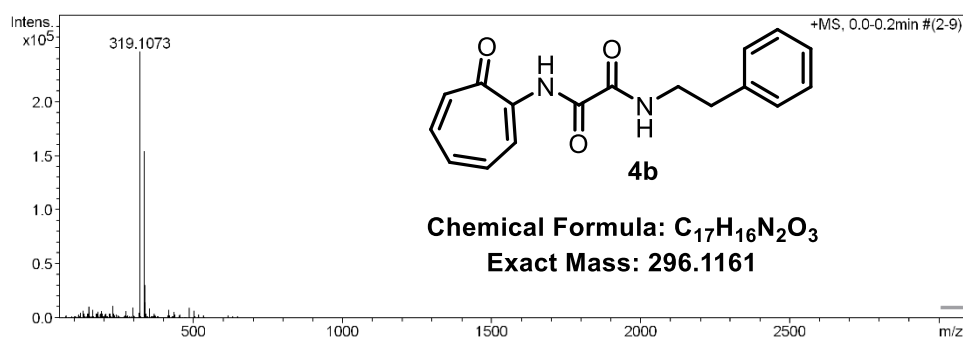
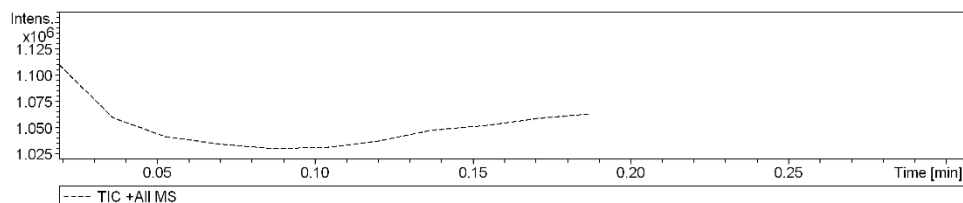


Fig S38. ESI-HRMS spectra of oxidized troponyl glycine derivative **4b**.

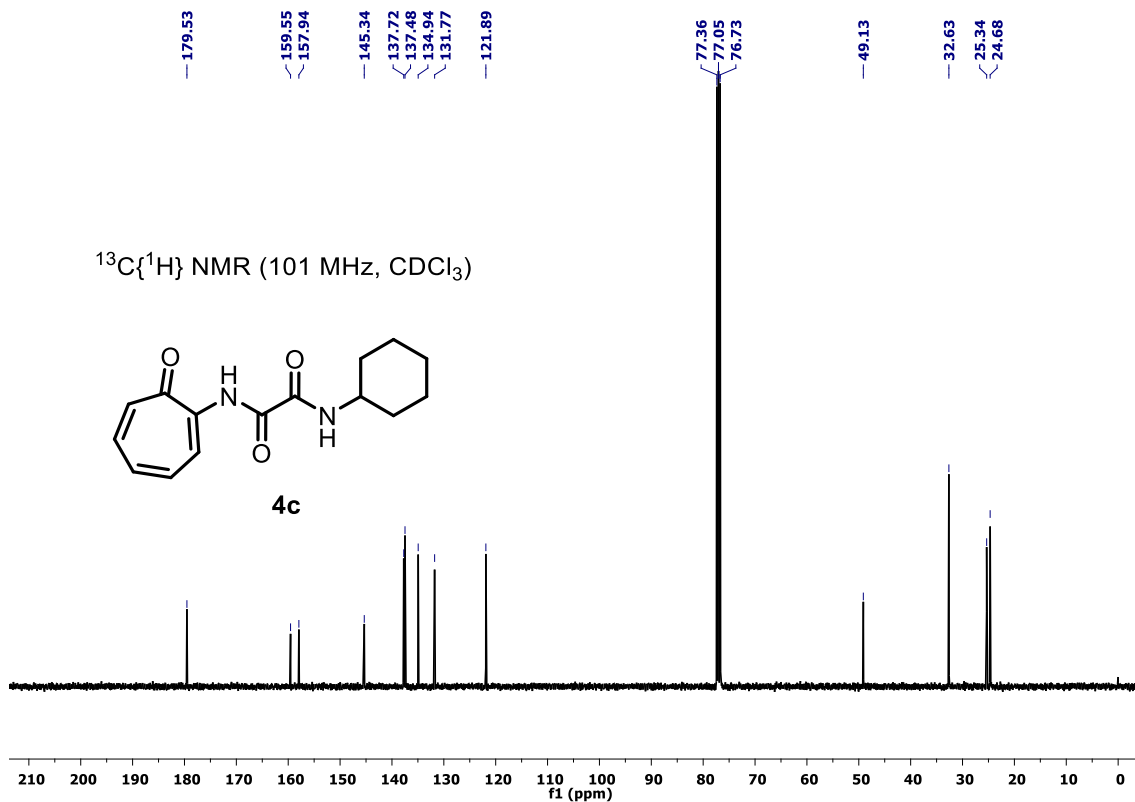
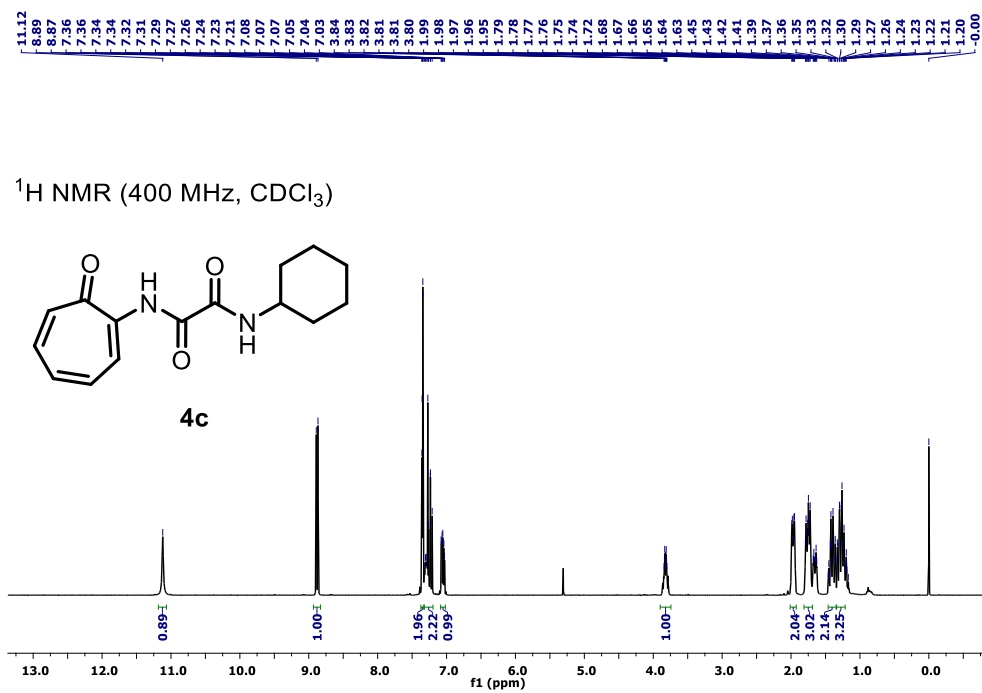


Fig S39. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4c**.

NKS_CKJ_1072

15-Feb-2023
23:16:54

XEVO-G2XSQTOF#YFA1739

NKS_15022023_9 (0.053) Cu (0.05); Is (1.00,1.00) C₁₅H₁₈N₂O₃Na

1: TOF MS ES+
8.37e12

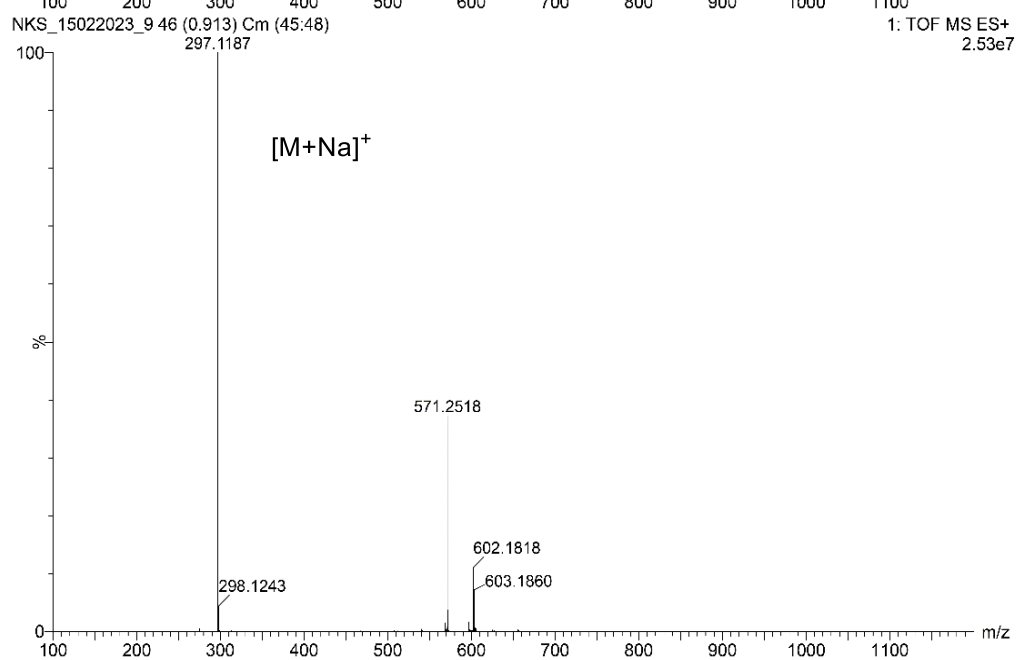
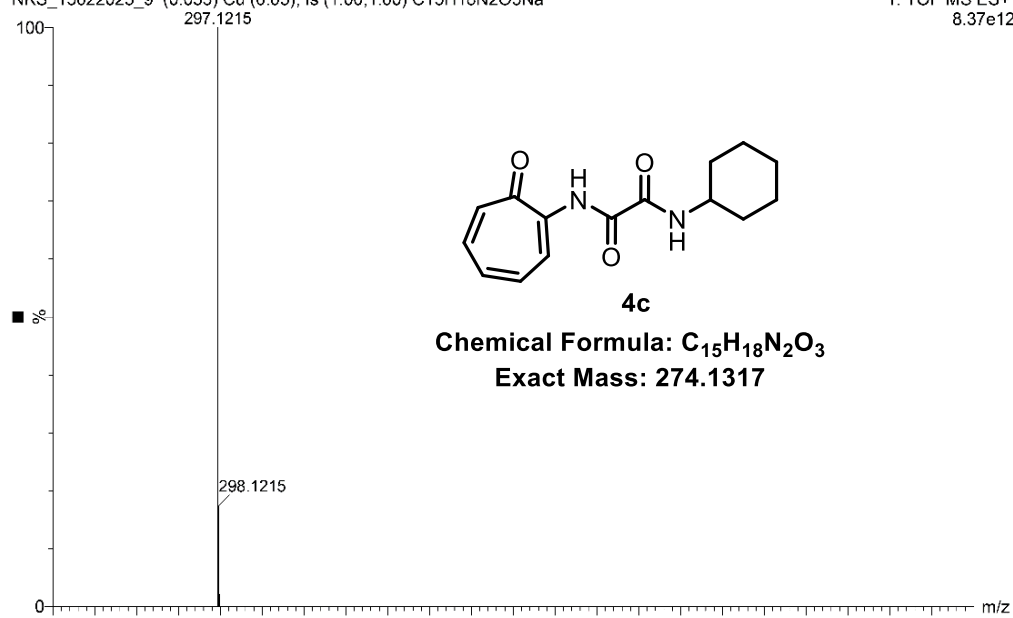


Fig S40. ESI-HRMS spectra of oxidized troponyl glycine derivative **4c**.

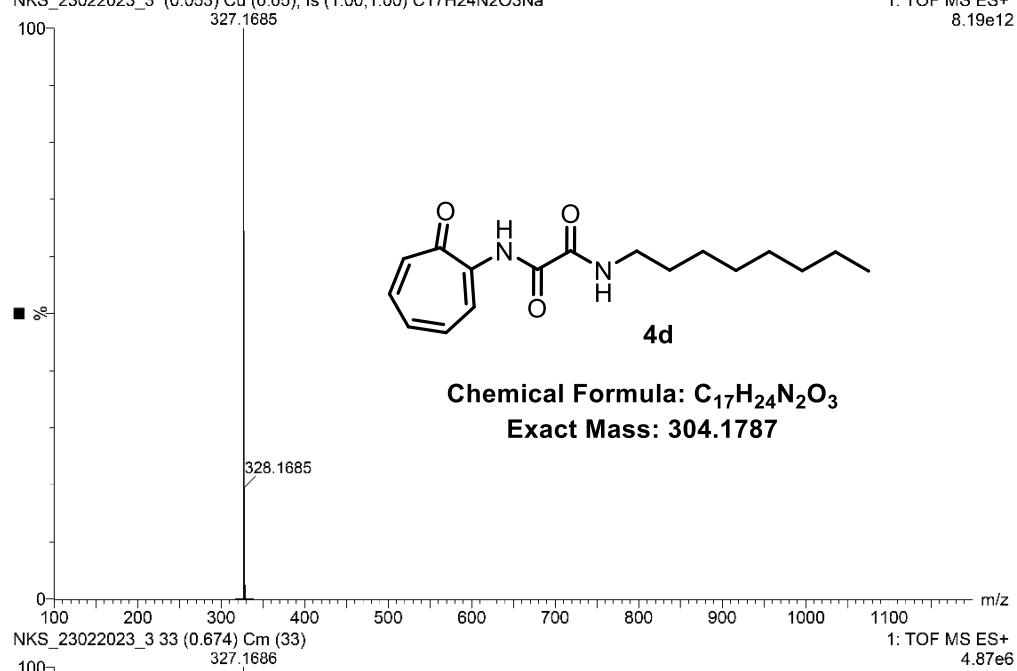


Fig S42. ESI-HRMS spectra of oxidized troponyl glycine derivative **4d**.

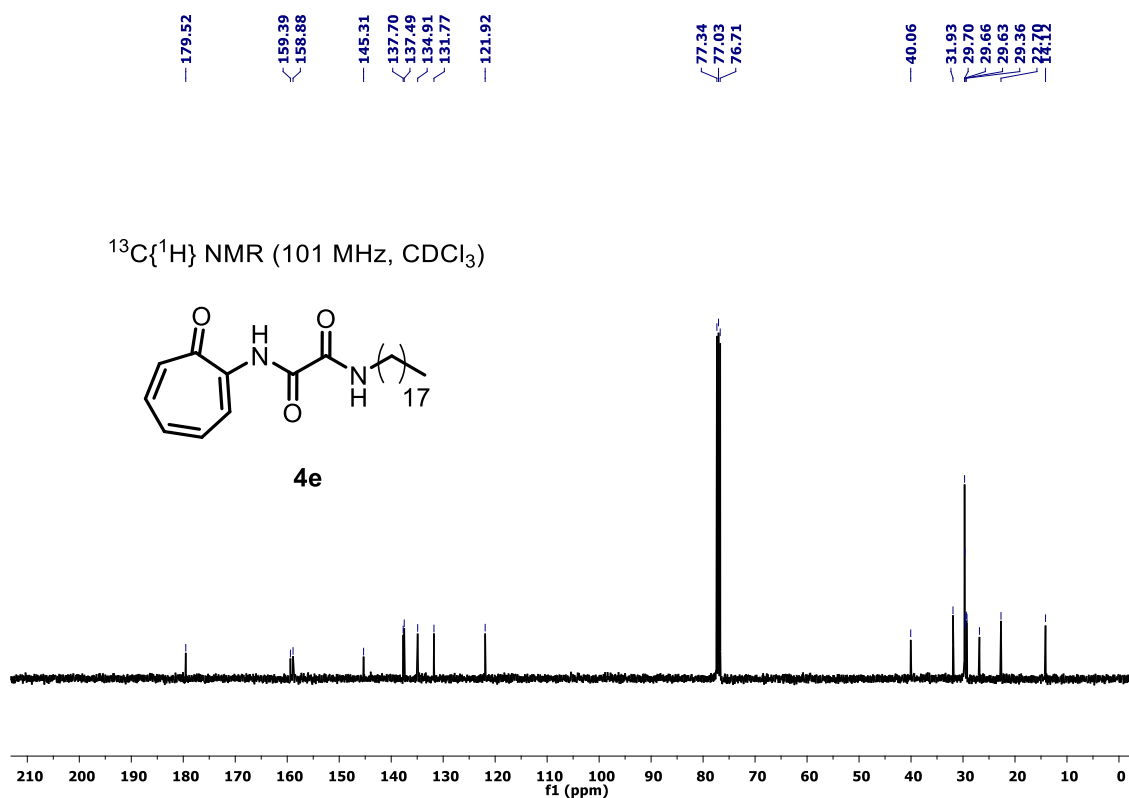
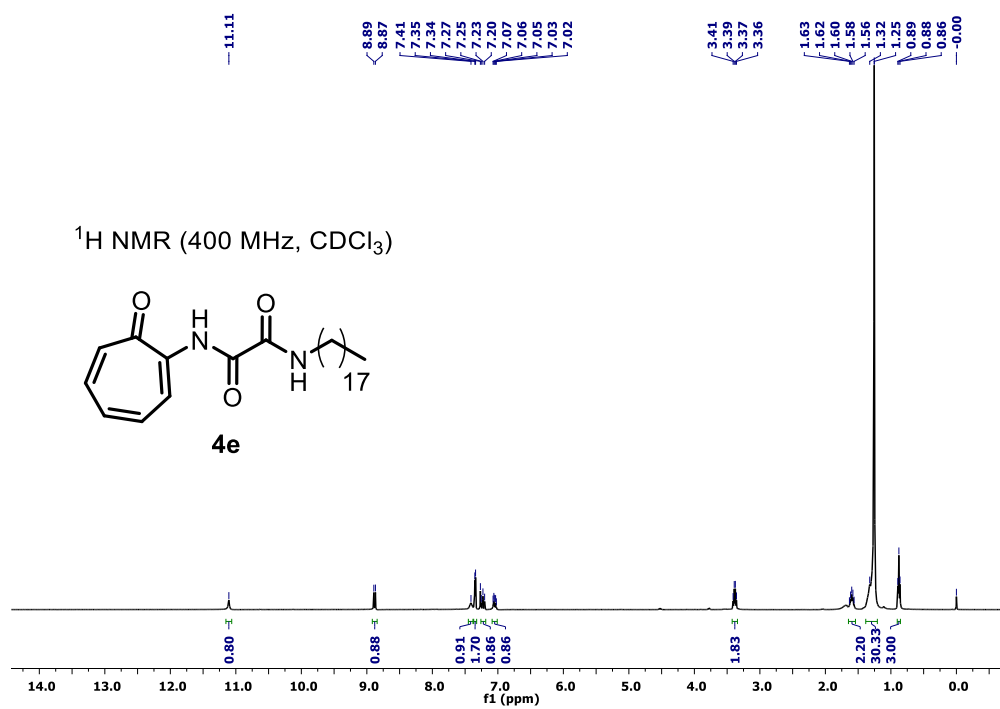


Fig S43. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4e**.

NKS_CKJ_1119 A

23-Mar-2023
20:31:56

XEVO-G2XSQTOF#YFA1739

NKS_23032023_1 (0.053) Cu (0.05); Is (1.00,1.00) C27H44N2O3Na

1: TOF MS ES+
7.33e12

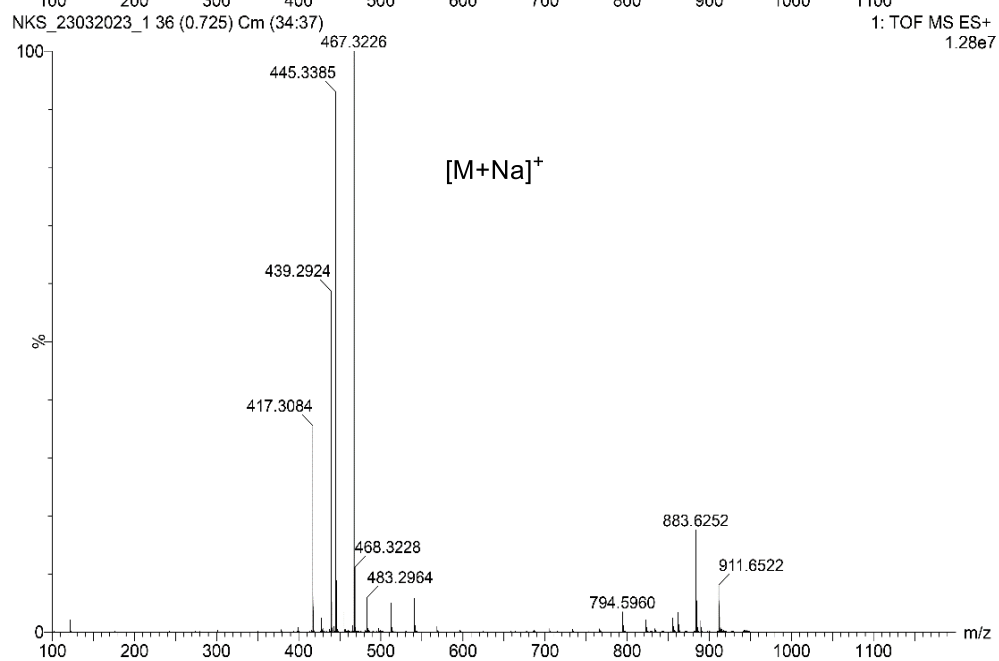
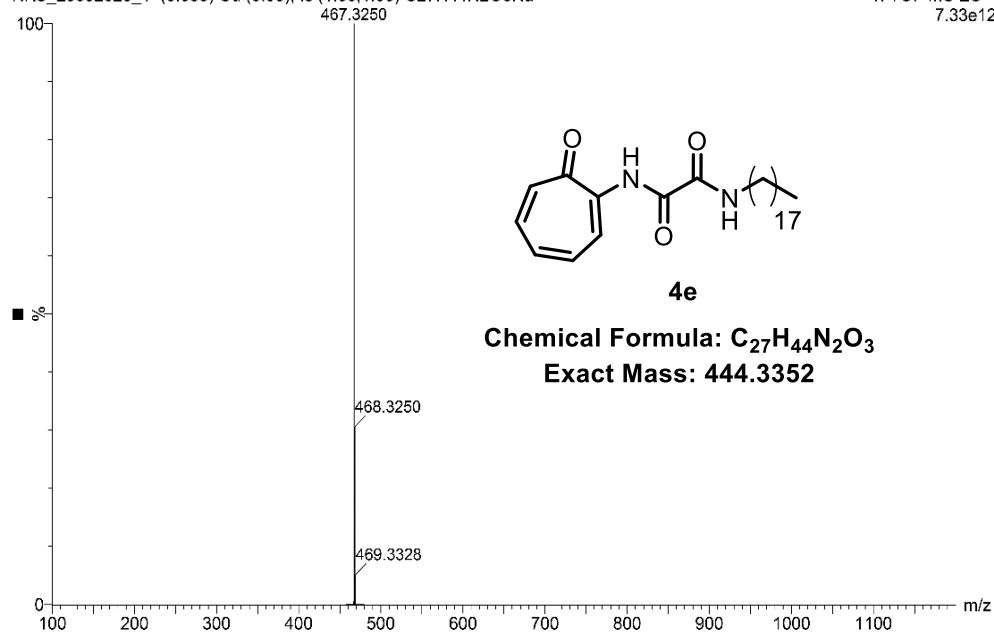


Fig S44. ESI-HRMS spectra of oxidized troponyl glycine derivative **4e**.

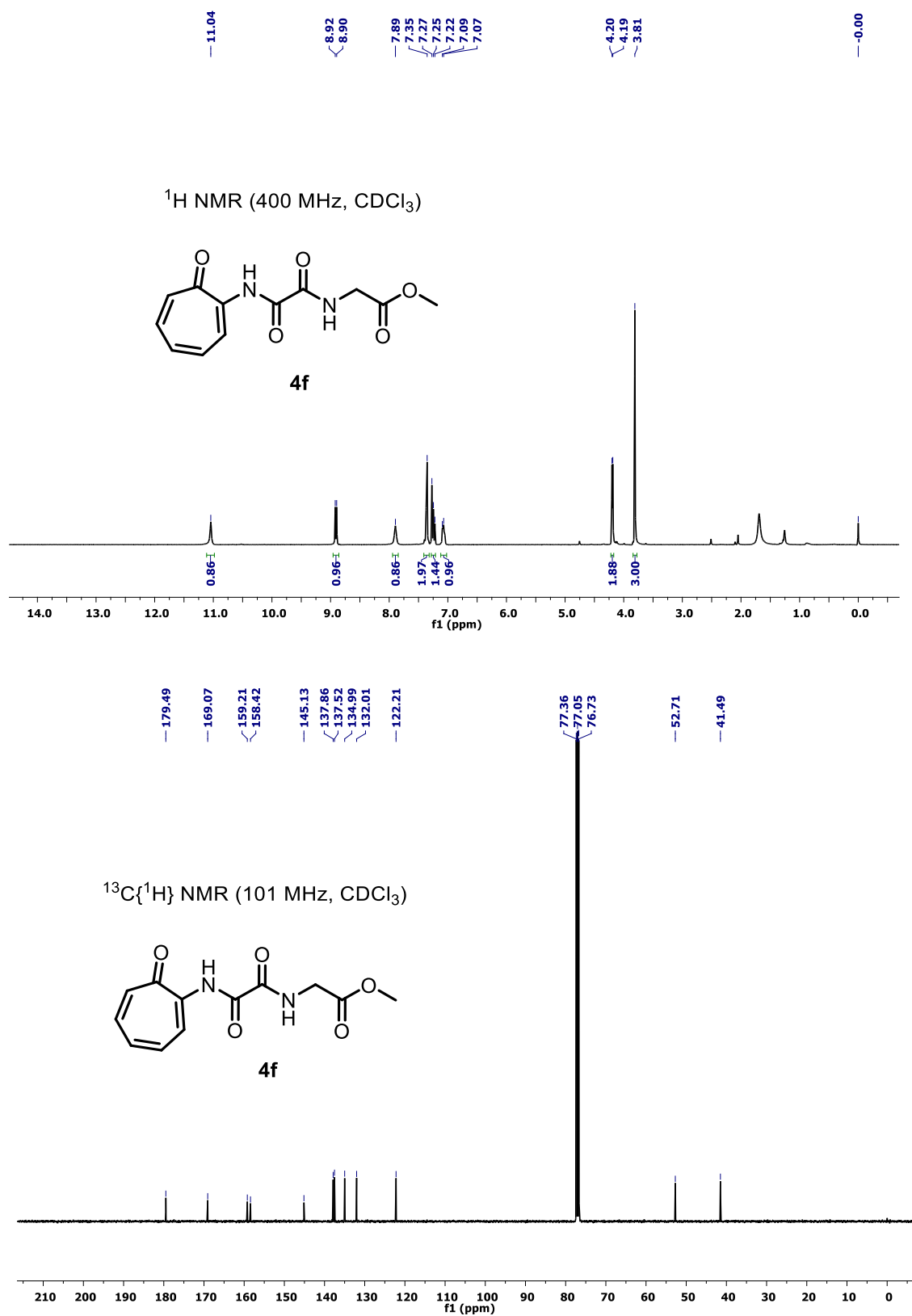


Fig S45. ^1H , $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of oxidized troponyl glycine derivative **4f**.

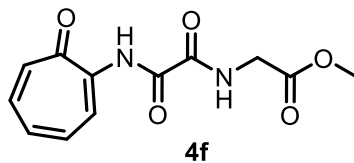
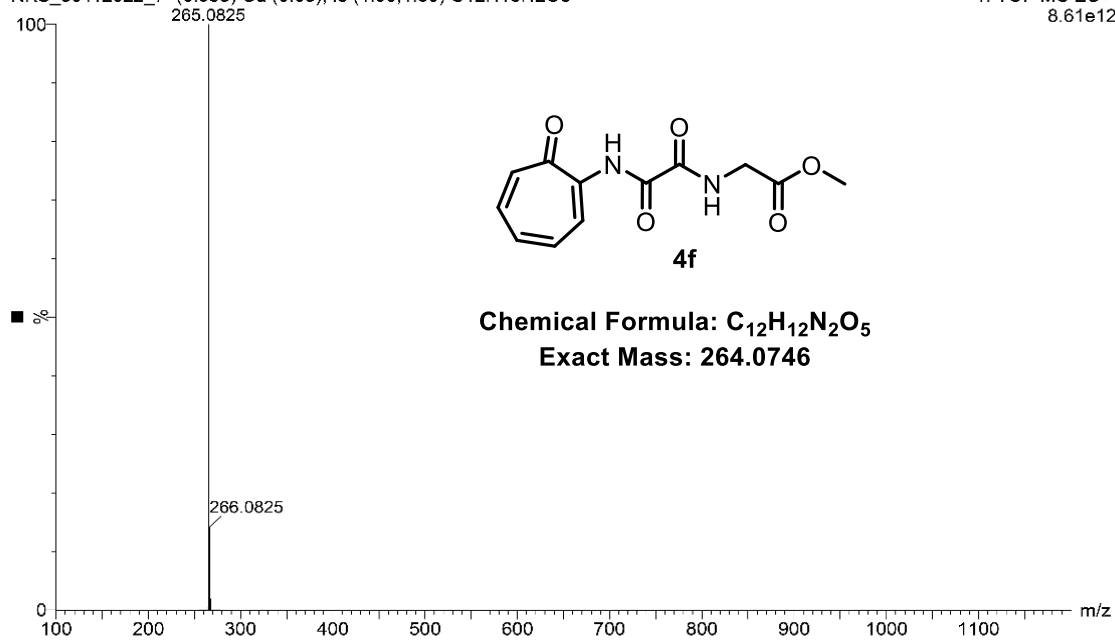
NKS_CKJ_999 RE

01-Dec-2022
02:11:24

XEVO-G2XSQTOF#YFA1739

NKS_30112022_7 (0.053) Cu (0.05); Is (1.00,1.00) C₁₂H₁₃N₂O₅

1: TOF MS ES+
8.61e12



Chemical Formula: C₁₂H₁₂N₂O₅
Exact Mass: 264.0746

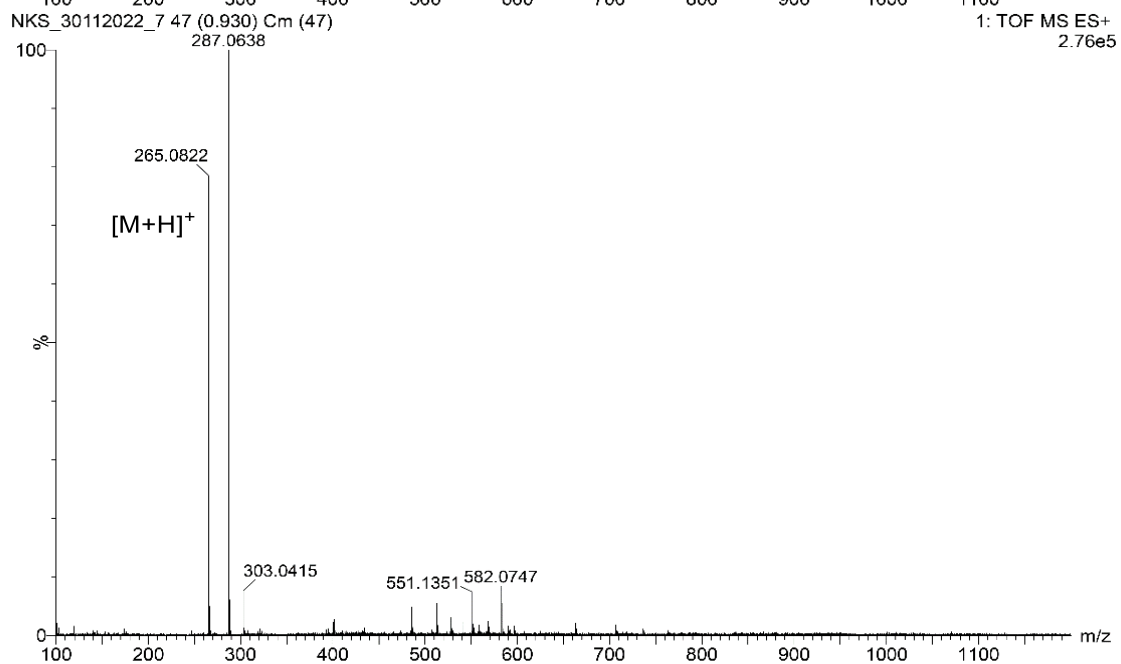


Fig S46. ESI-HRMS spectra of oxidized troponyl glycine derivative **4f**.

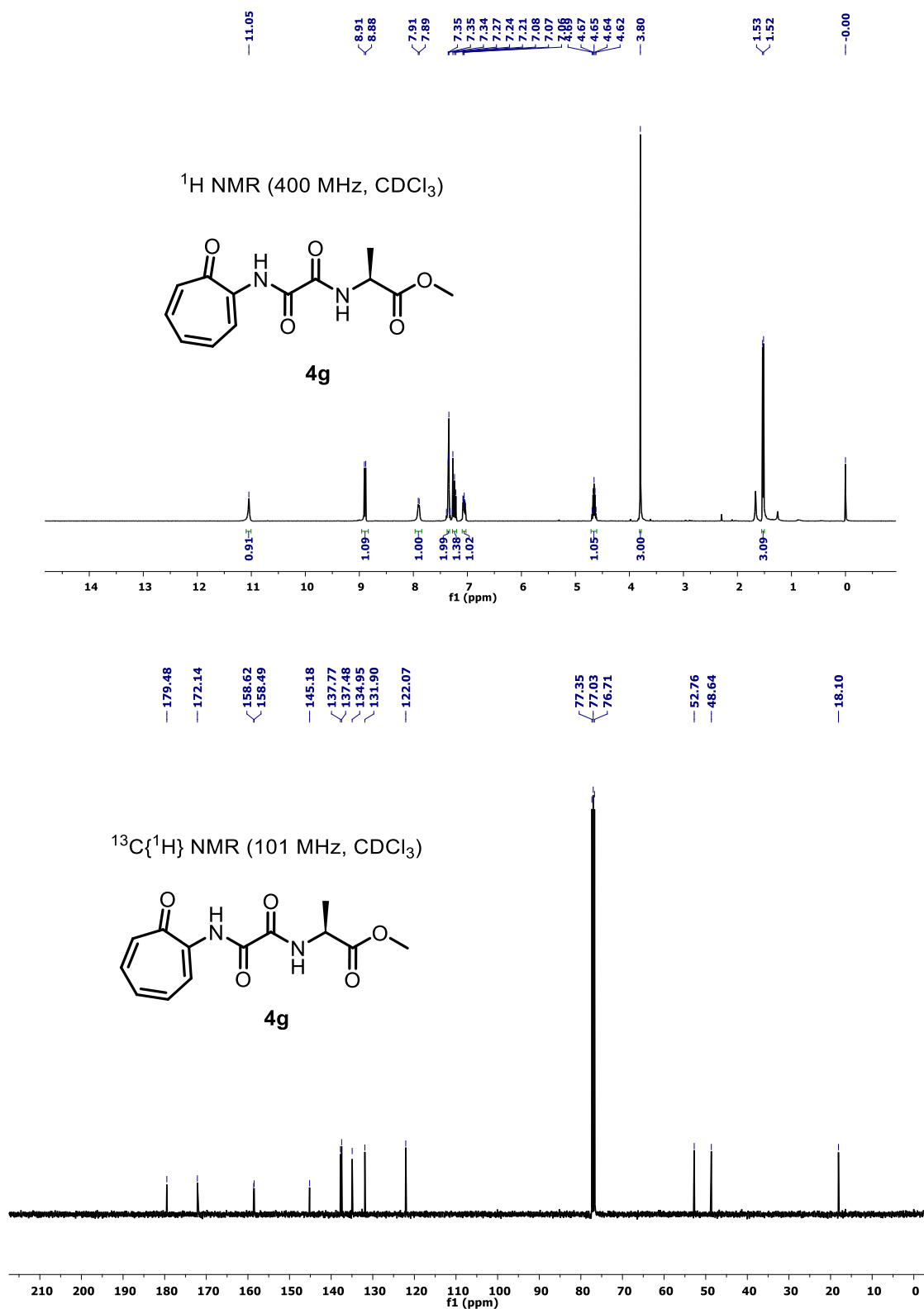


Fig S47. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4g**.

NKS_CKJ_1030 A

17-Feb-2023
00:01:32

XEVO-G2XSQTOF#YFA1739

NKS_16022023_6 (0.053) Cu (0.05); Is (1.00,1.00) C₁₃H₁₄N₂O₅Na

1: TOF MS ES+
8.51e12

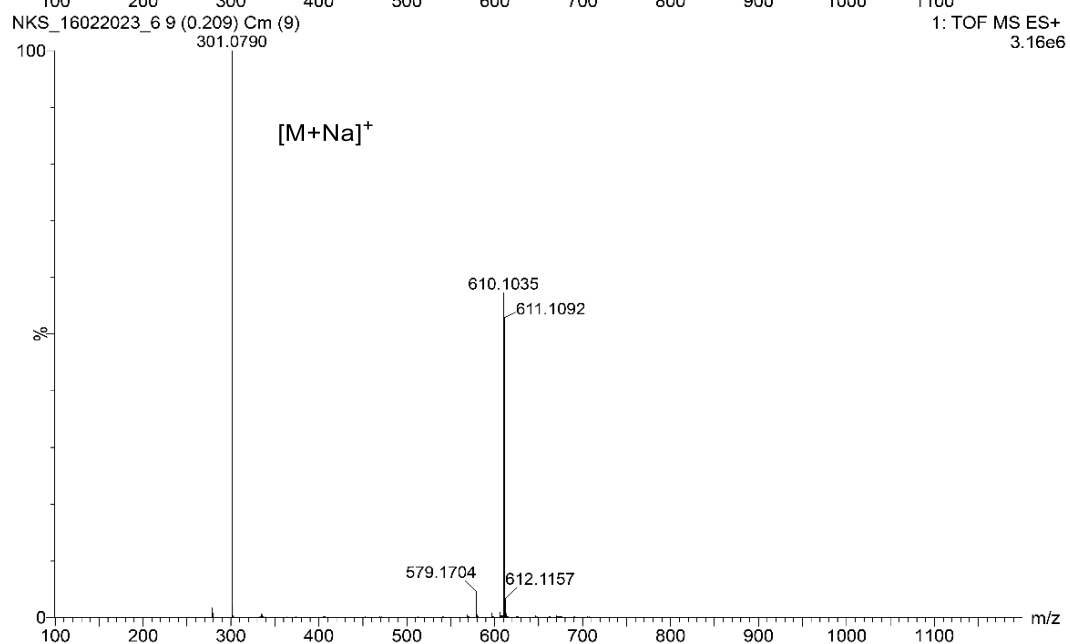
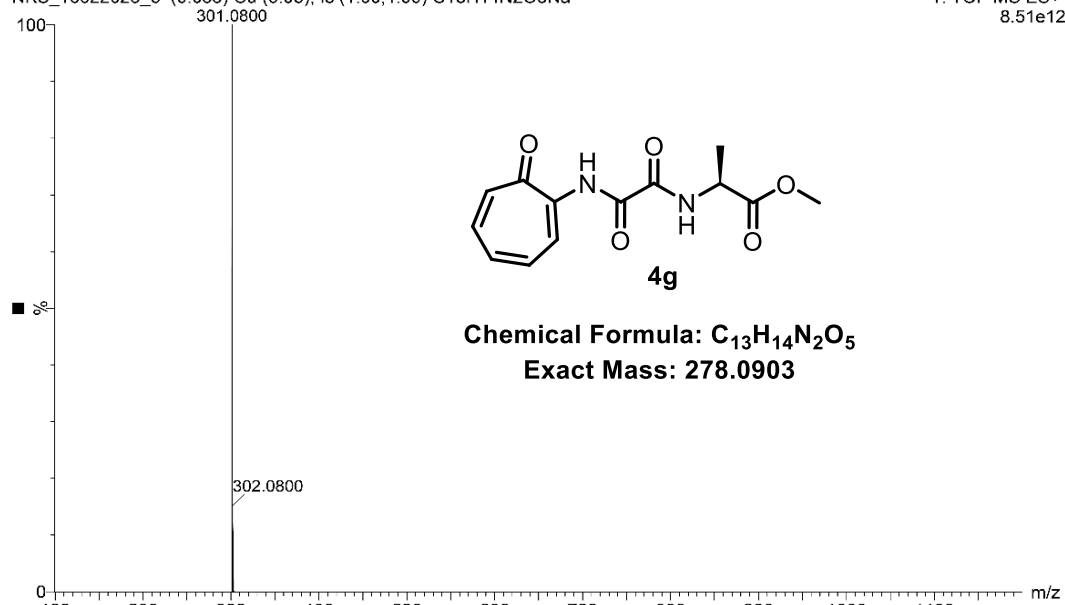


Fig S48. ESI-HRMS spectra of oxidized troponyl glycine derivative **4g**.

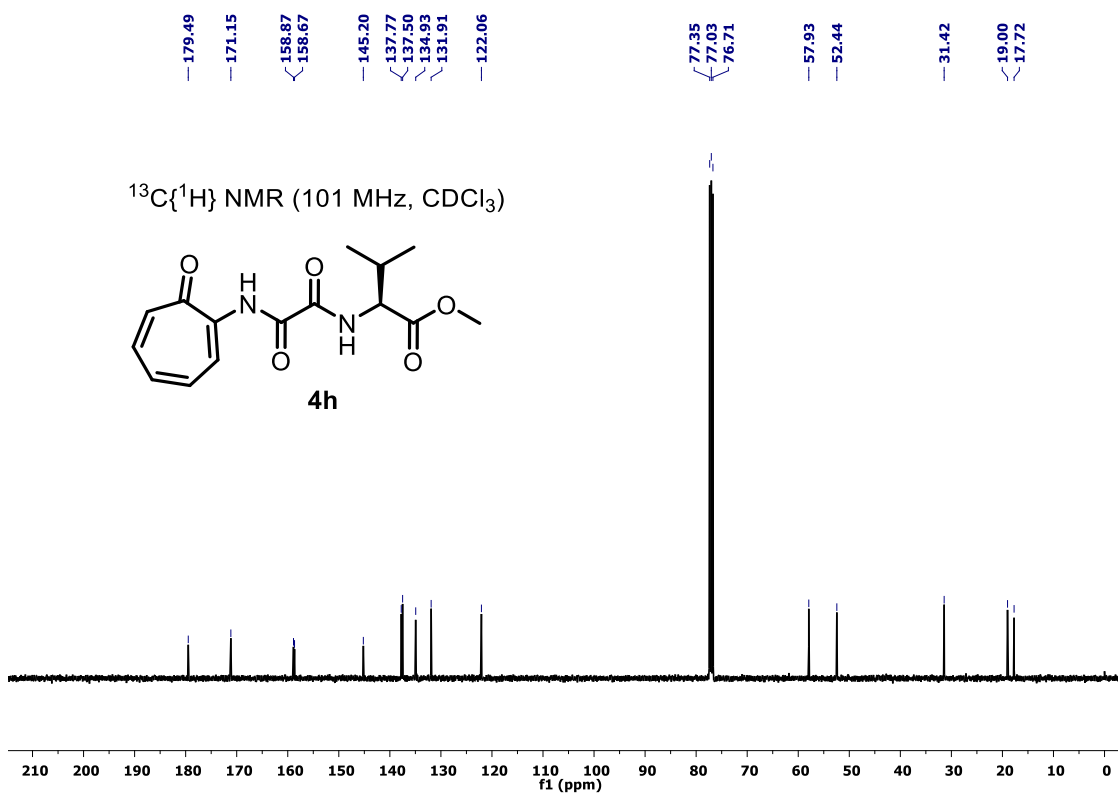
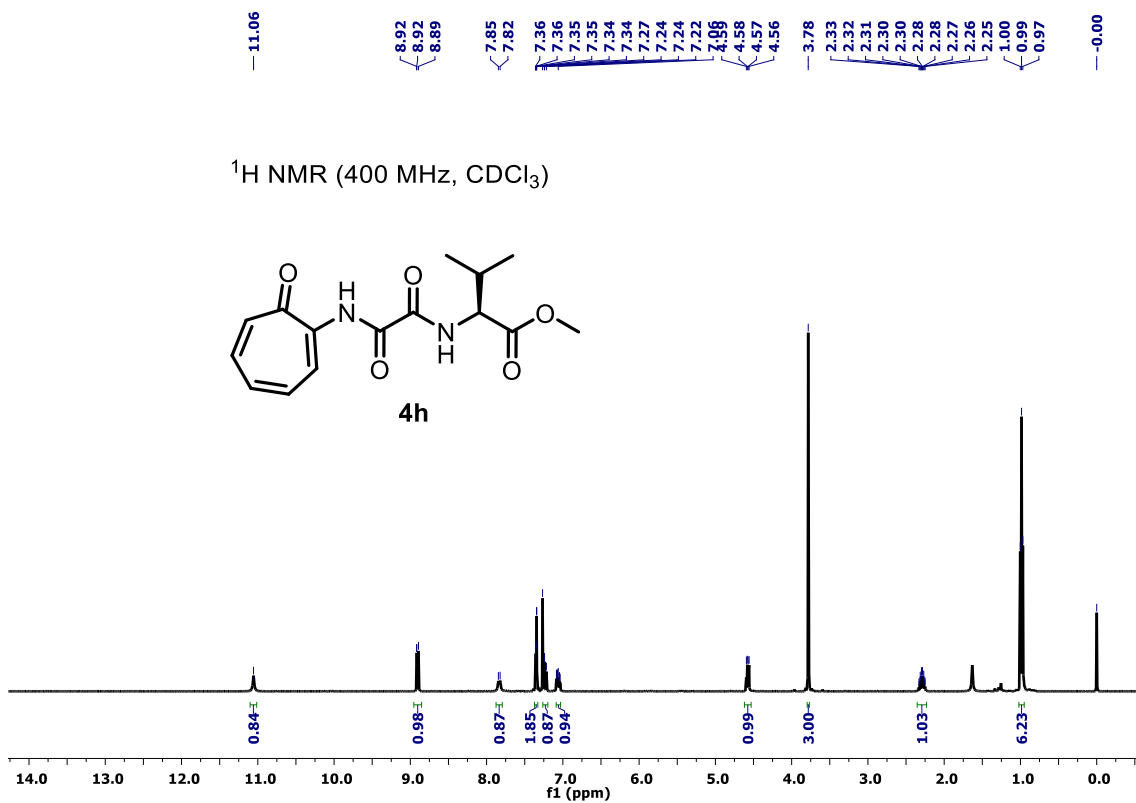


Fig S49. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4h**.

NKS_CKJ_1031

17-Feb-2023
00:15:10

XEVO-G2XSQTOF#YFA1739

NKS_16022023_7 (0.053) Cu (0.05); Is (1.00,1.00) C₁₅H₁₈N₂O₅Na

1: TOF MS ES+
8.33e12

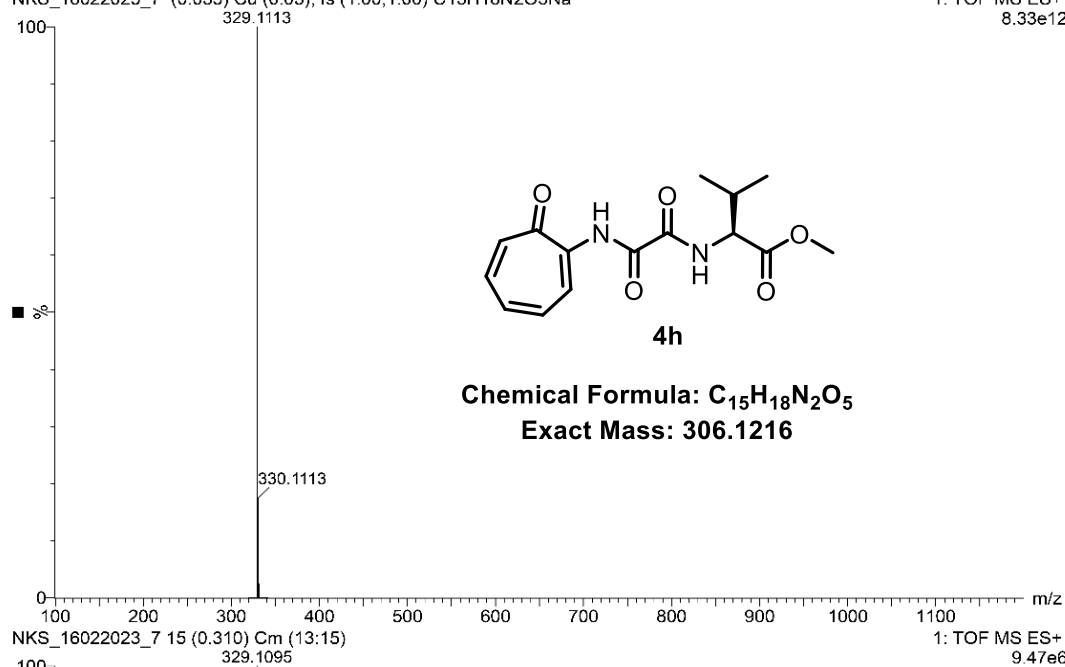


Fig S50. ESI-HRMS spectra of oxidized troponyl glycine derivative **4h**.

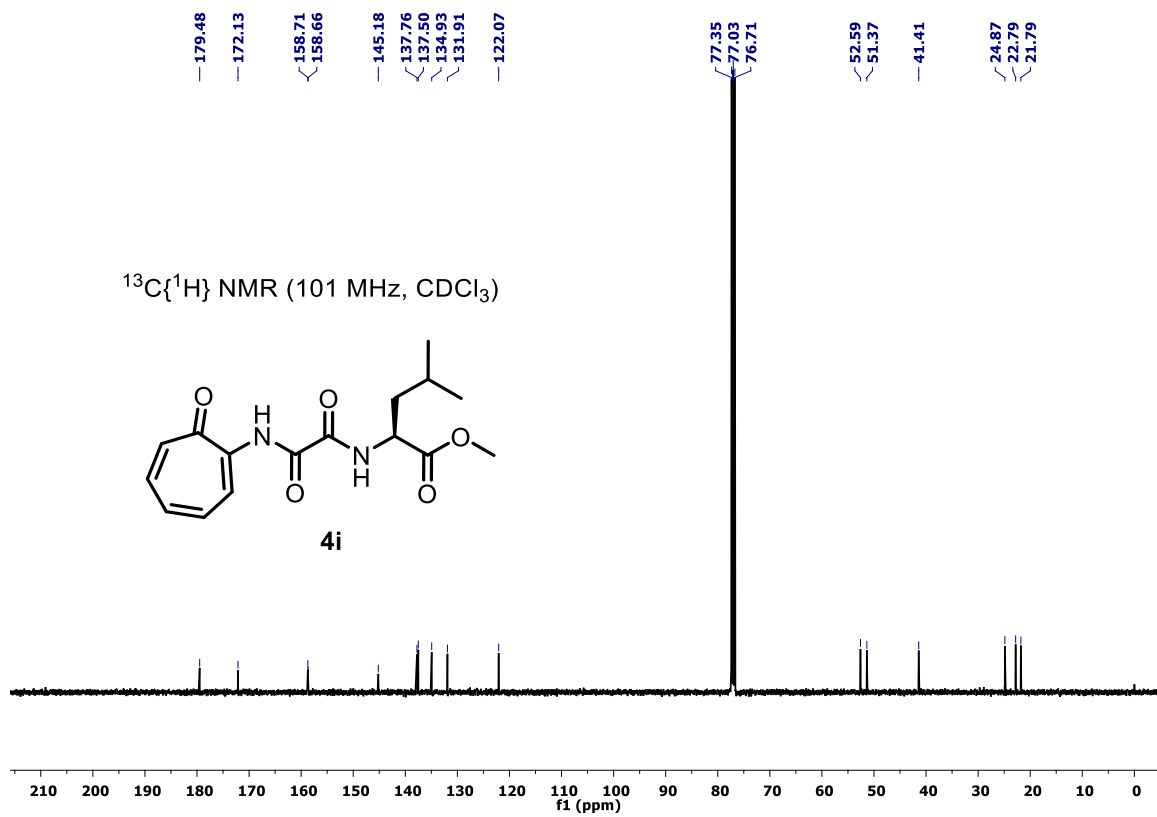
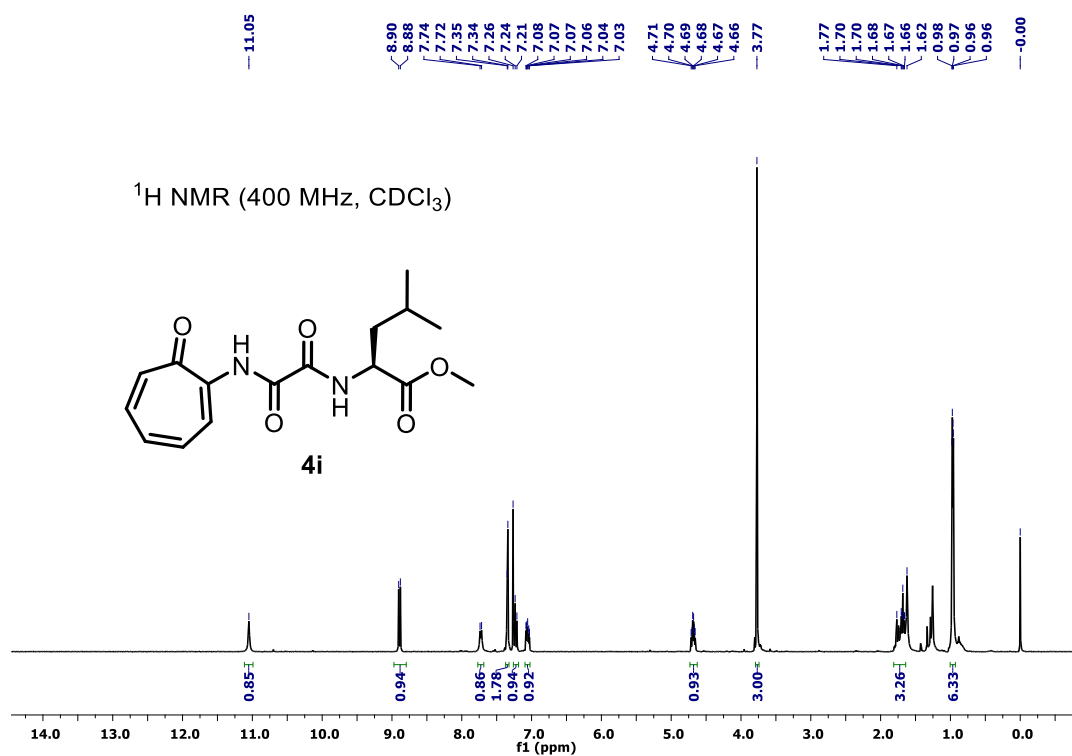


Fig S51. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4i**.

NKS_CKJ_1034

17-Feb-2023

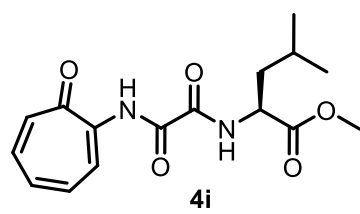
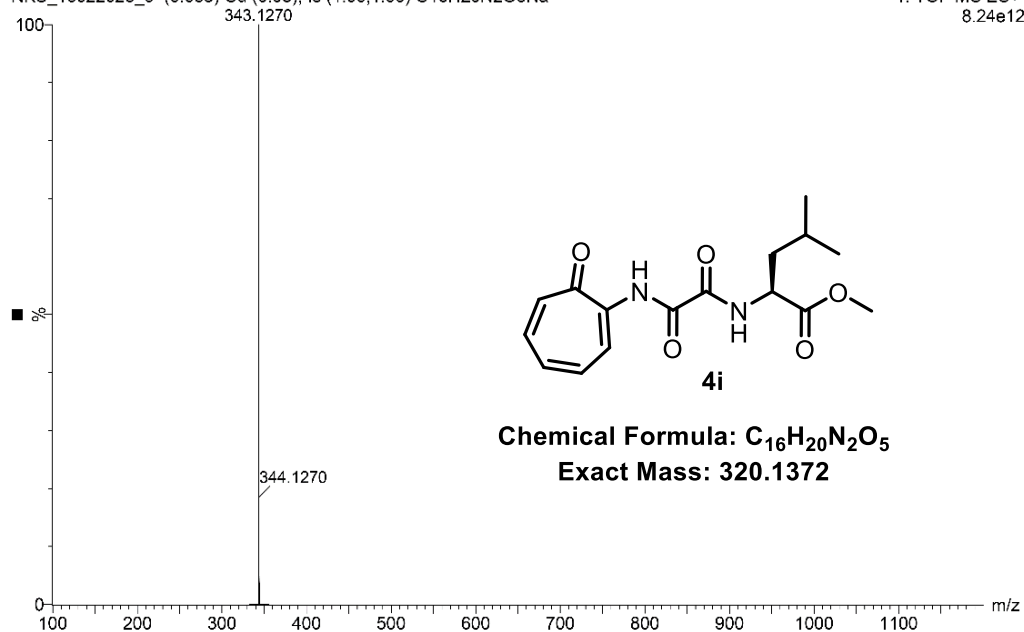
00:38:30

XEVO-G2XSQTOF#YFA1739

NKS_16022023_9 (0.053) Cu (0.05); Is (1.00,1.00) C₁₆H₂₀N₂O₅Na

1: TOF MS ES+

8.24e12



Chemical Formula: C₁₆H₂₀N₂O₅
Exact Mass: 320.1372

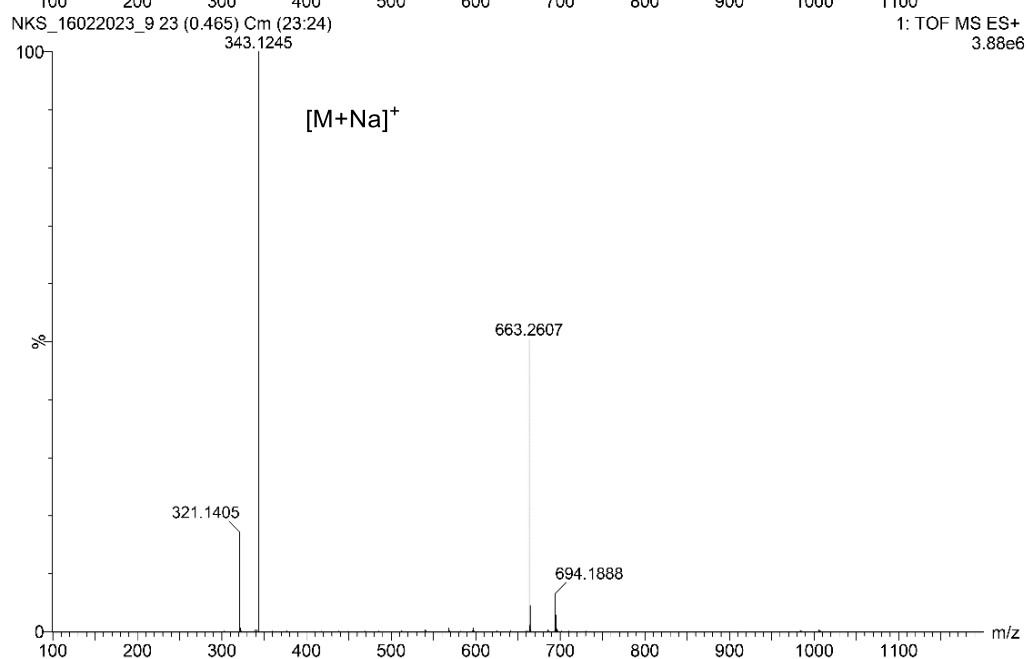


Fig S52. ESI-HRMS spectra of oxidized troponyl glycine derivative **4i**.

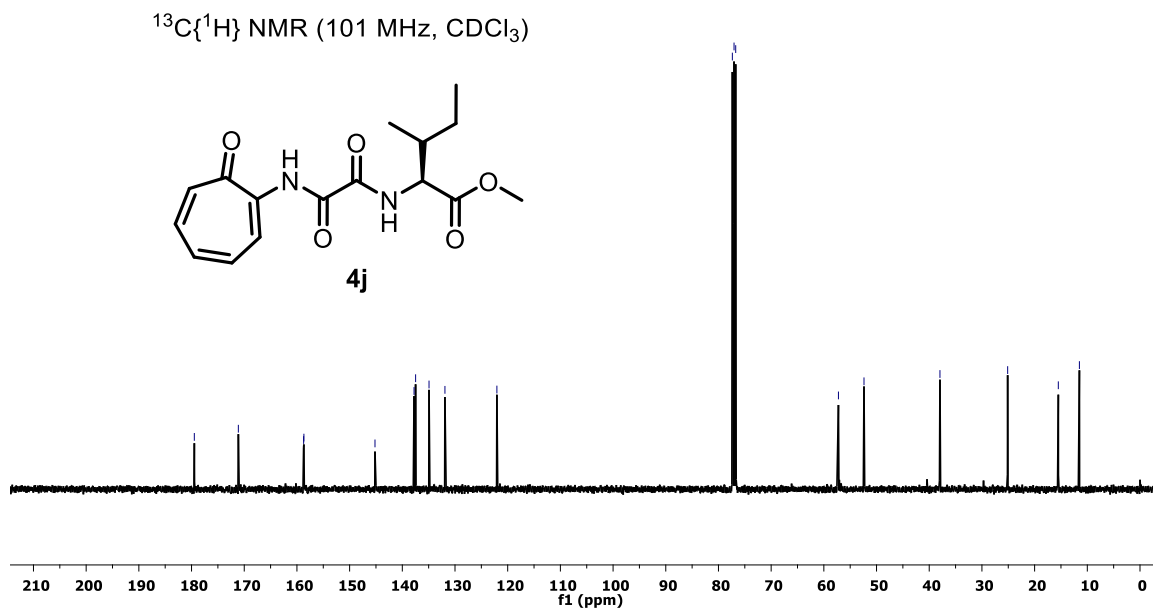
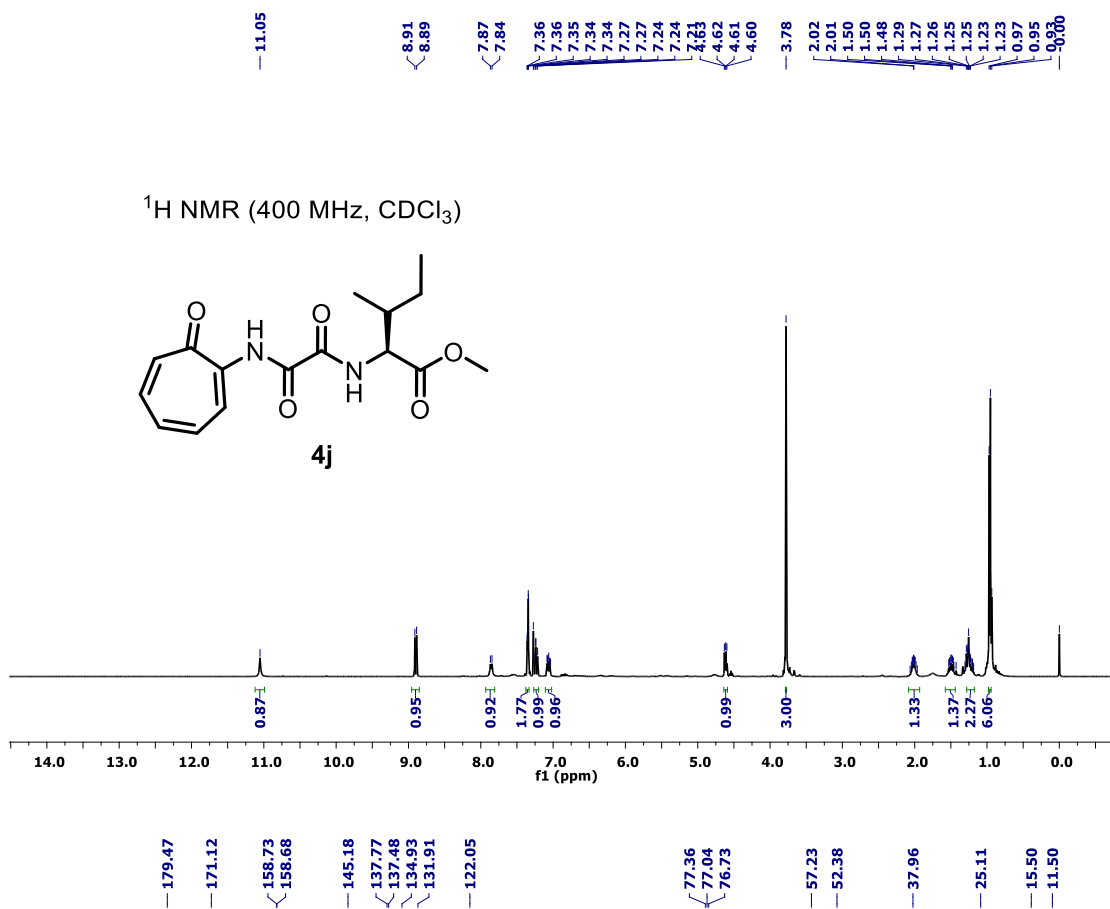


Fig S53. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4j**.

NKS_CKJ_1035

17-Feb-2023
00:50:43

XEVO-G2XSQTOF#YFA1739

NKS_16022023_10 (0.053) Cu (0.05); Is (1.00,1.00) C₁₆H₂₀N₂O₅Na

1: TOF MS ES+
8.24e12

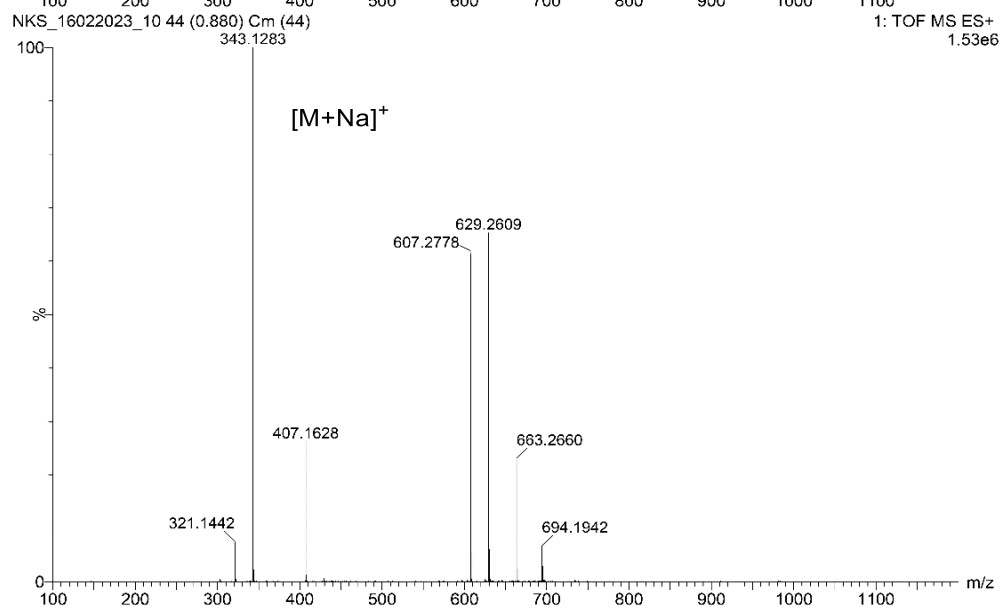
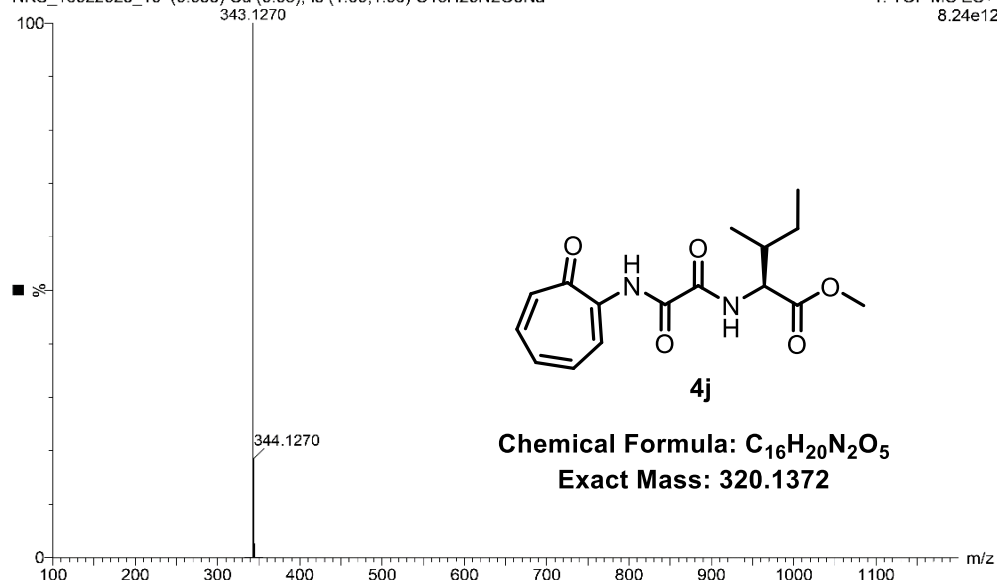


Fig S54. ESI-HRMS spectra of oxidized troponyl glycine derivative **4j**.

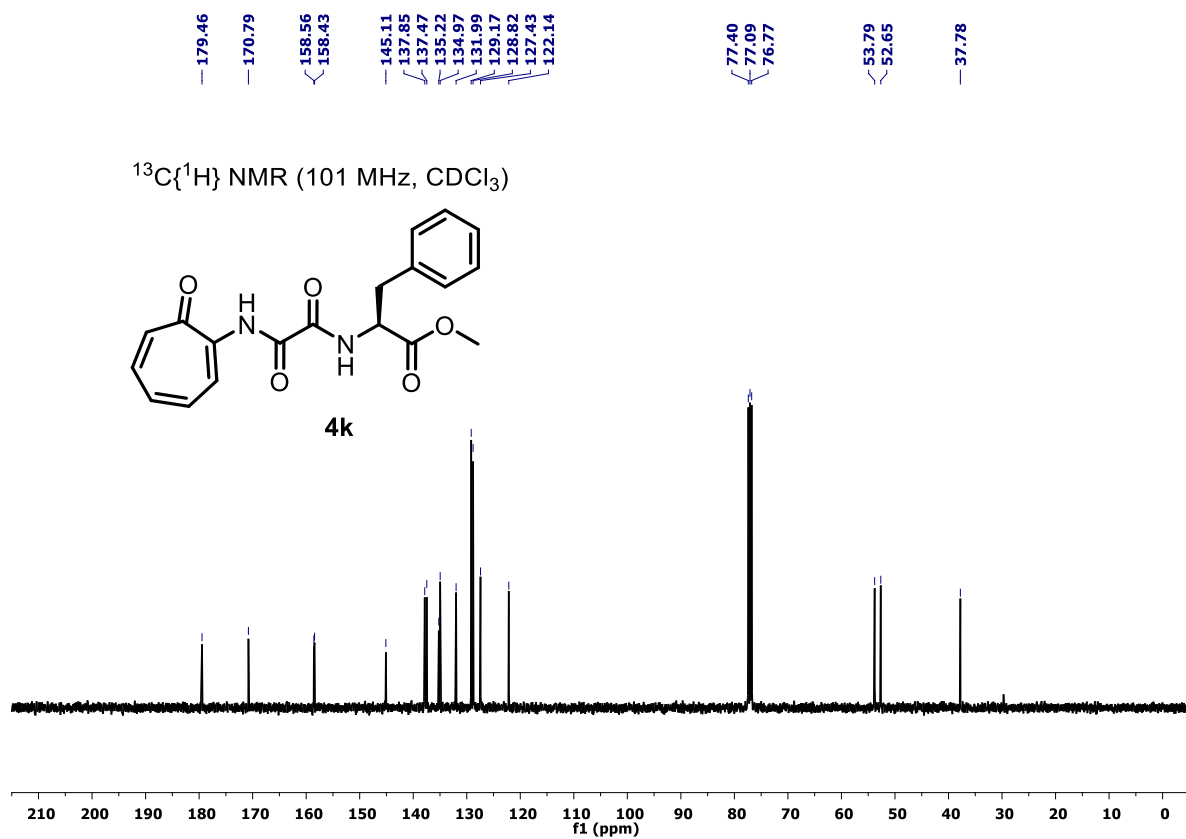
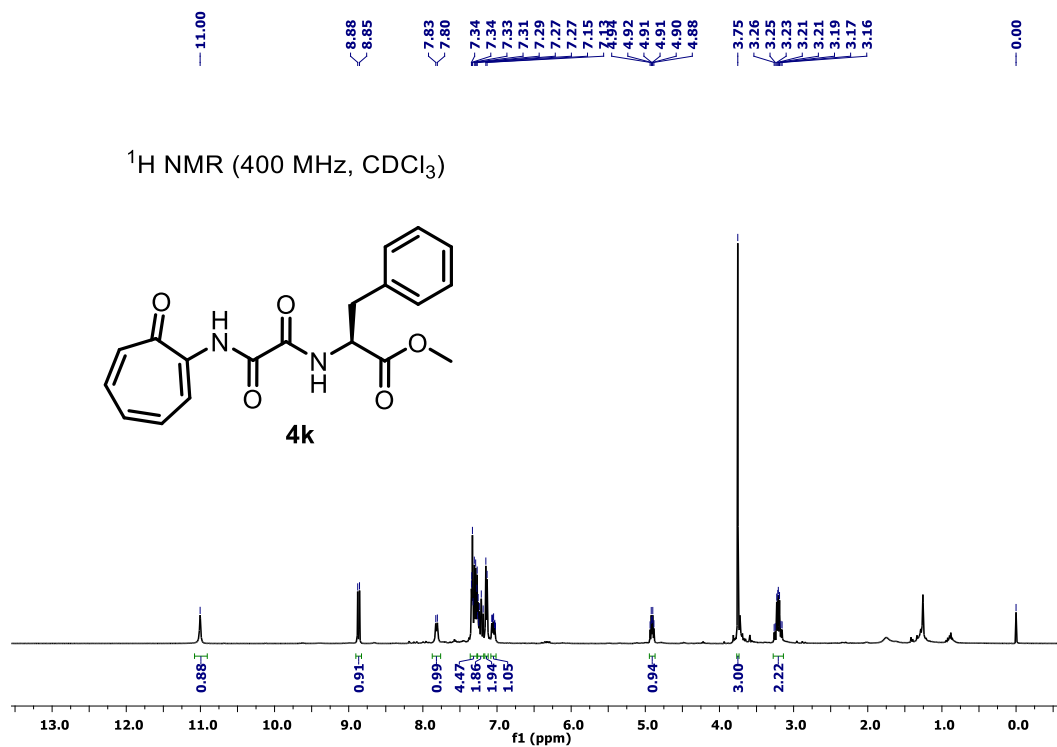


Fig S55. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4k**.

Display Report

Analysis Info

Analysis Name D:\Data\JULY-2021\NKS\17072021_-NKS-CKJ-342(A).d
Method Pos_tune_low_05122019.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/17/2021 4:33:22 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

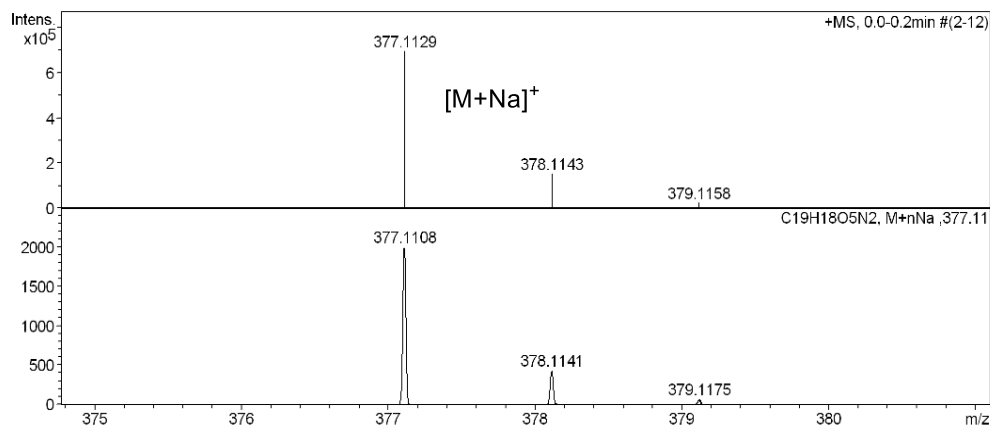
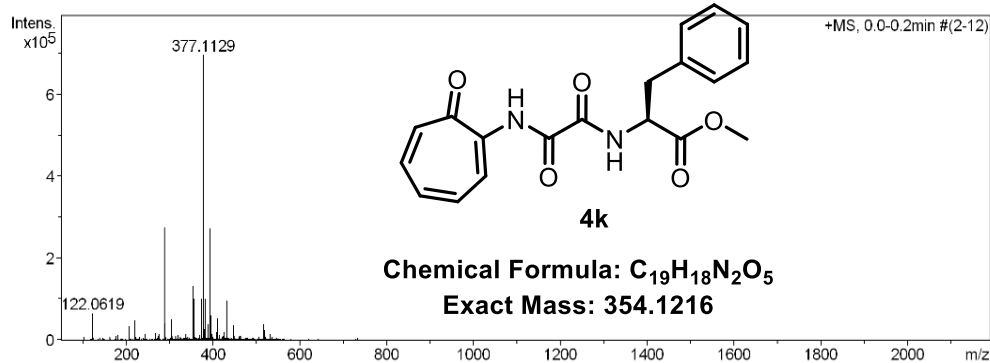
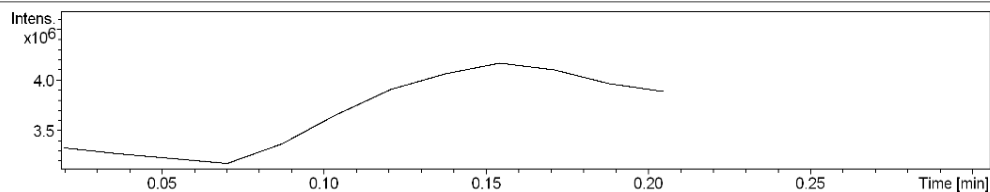


Fig S56. ESI-HRMS spectra of oxidized troponyl glycine derivative **4k**.

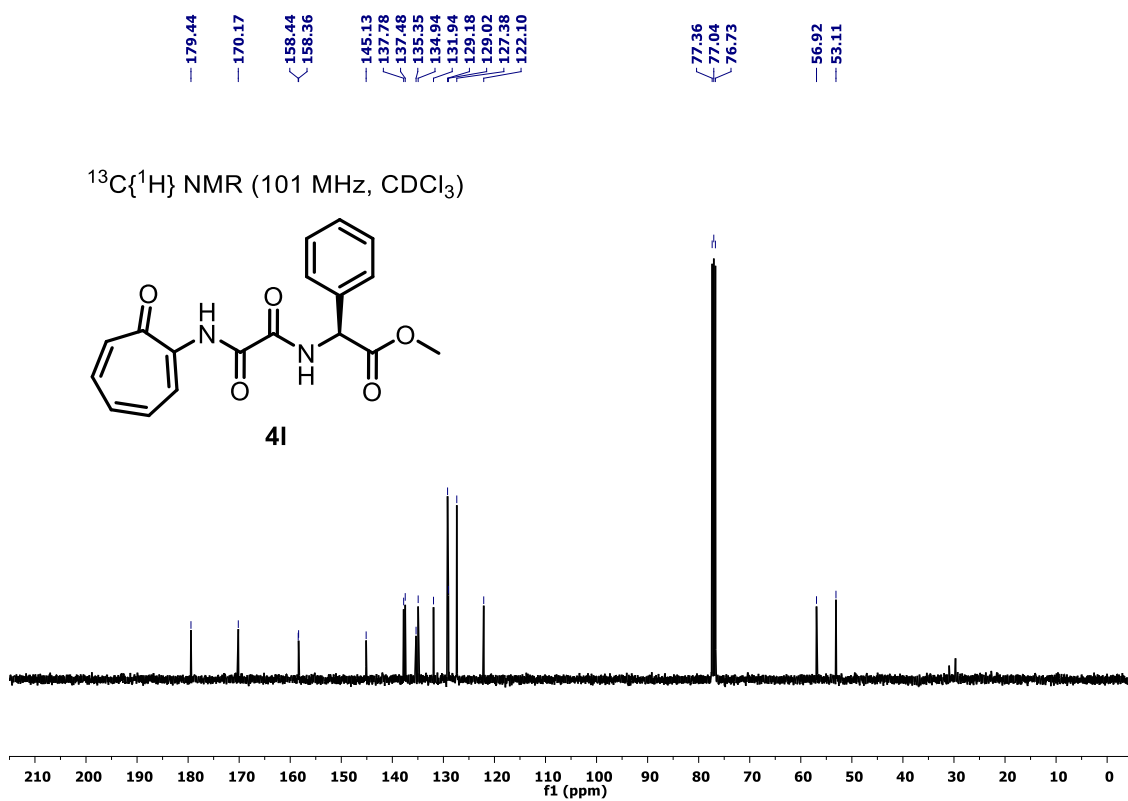
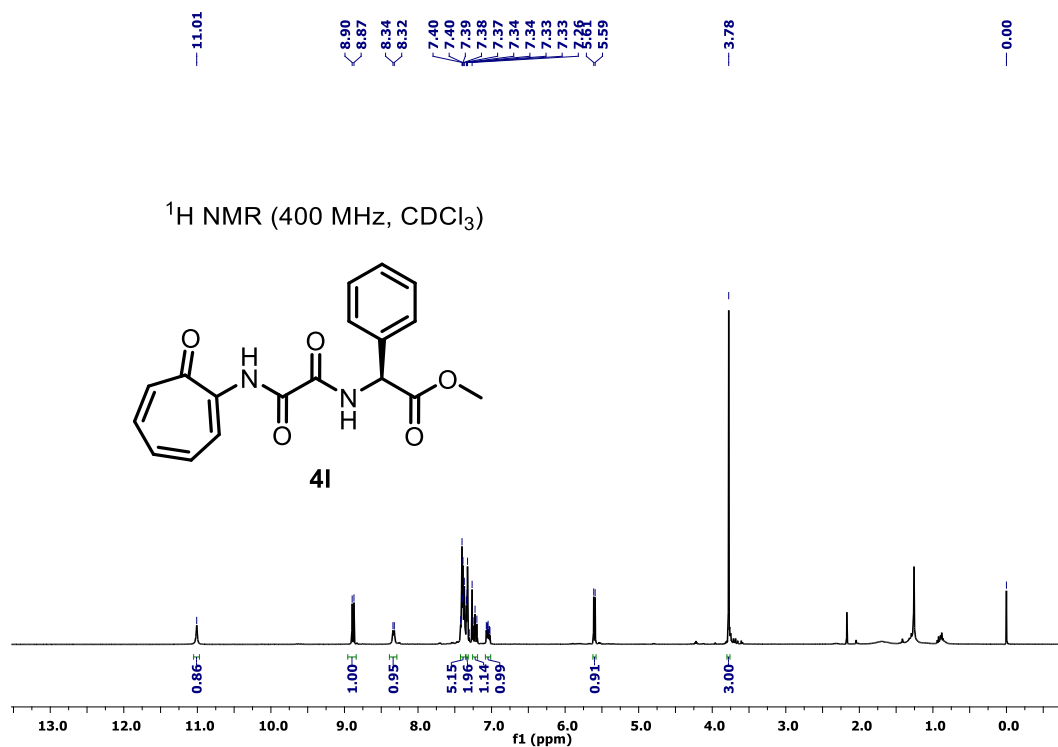


Fig S57. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4I**.

Display Report

Analysis Info

Analysis Name D:\Data\JULY-2021\NKS\17072021_-NKS-CKJ-374-A.d
Method Pos_tune_low_05122019.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/17/2021 5:32:57 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

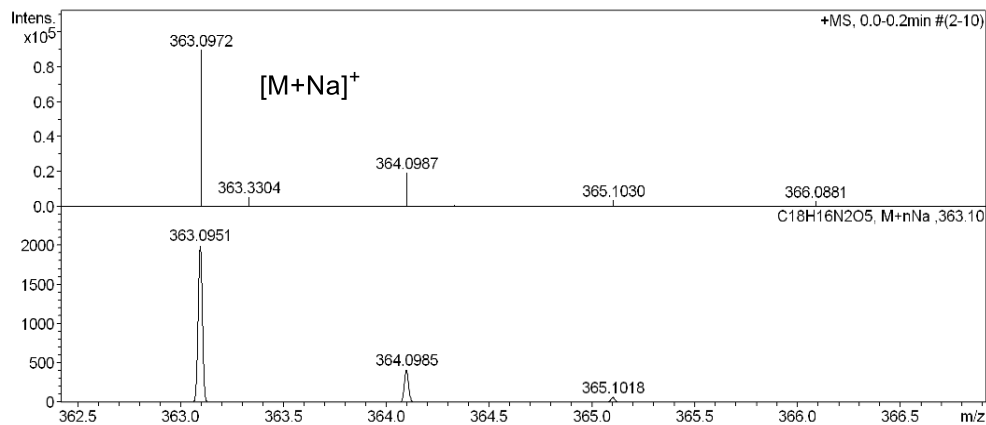
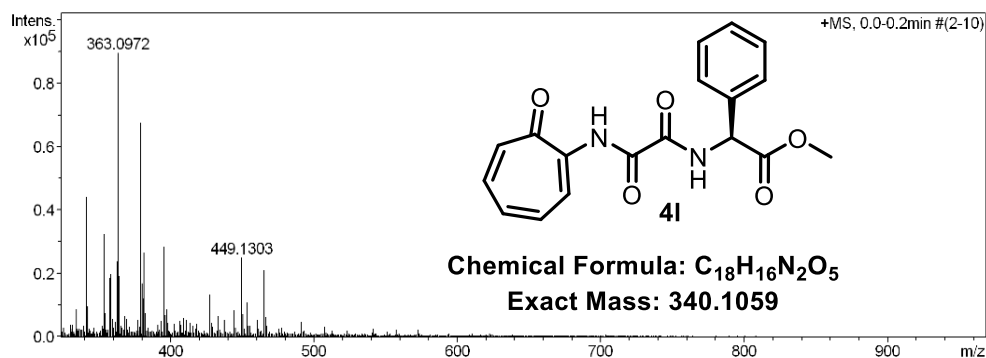
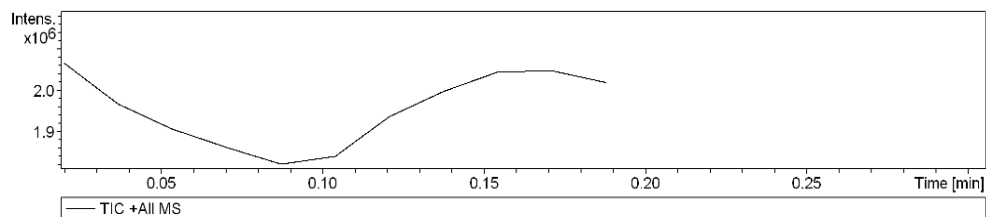


Fig S58. ESI-HRMS spectra of oxidized troponyl glycine derivative **4I**.

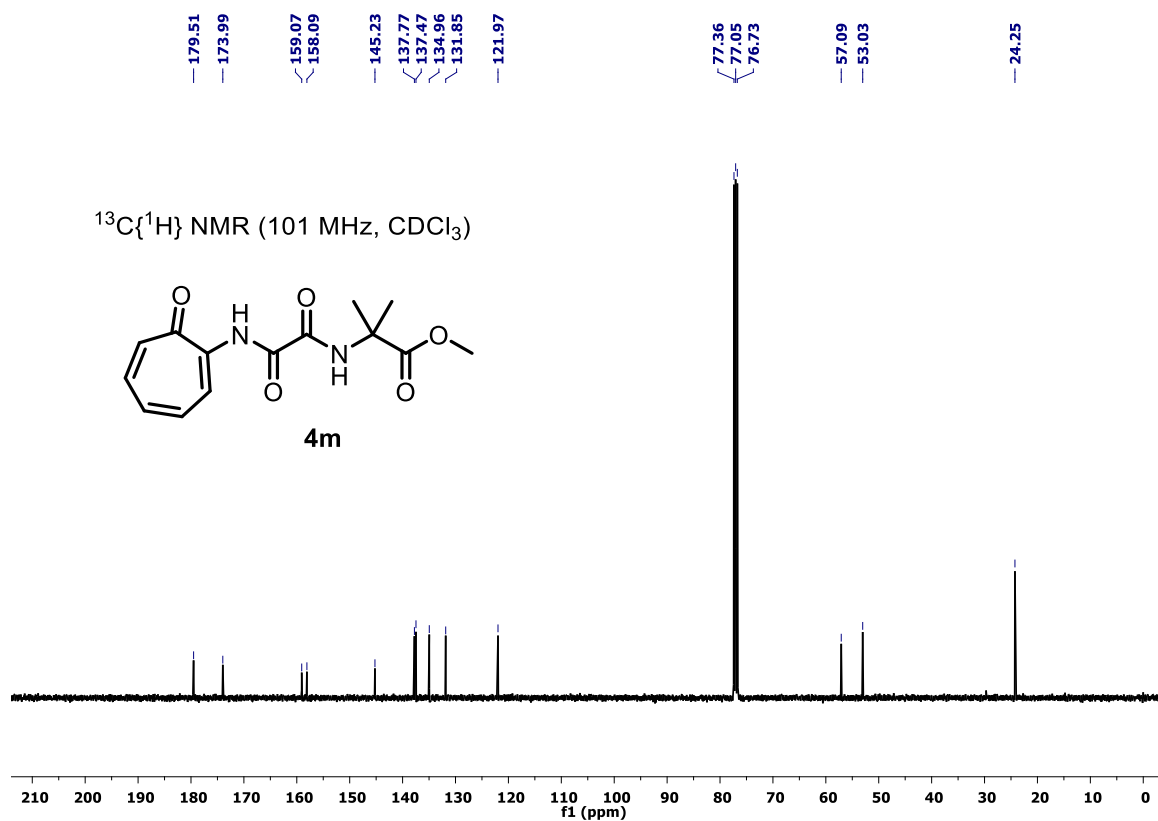
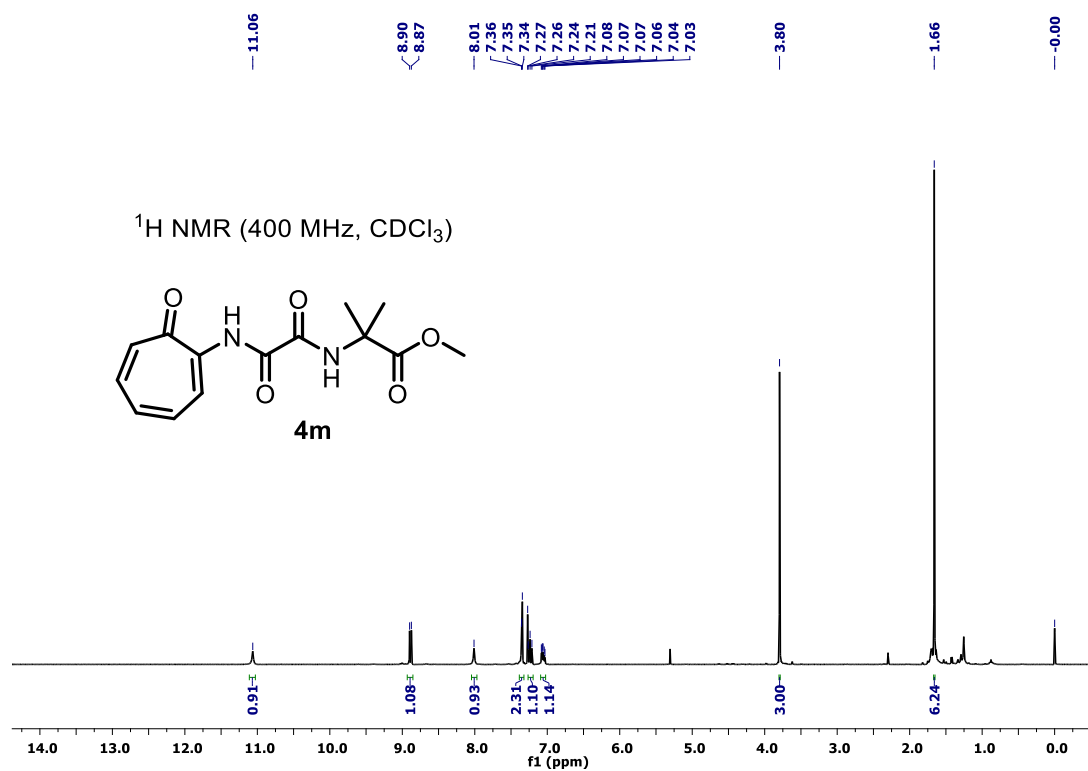


Fig S59. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4m**.

NKS_CKJ_1071

15-Feb-2023
23:04:52

XEVO-G2XSQTOF#YFA1739

NKS_15022023_8 (0.053) Cu (0.05); Is (1.00,1.00) C₁₄H₁₆N₂O₅Na

1: TOF MS ES+
8.42e12

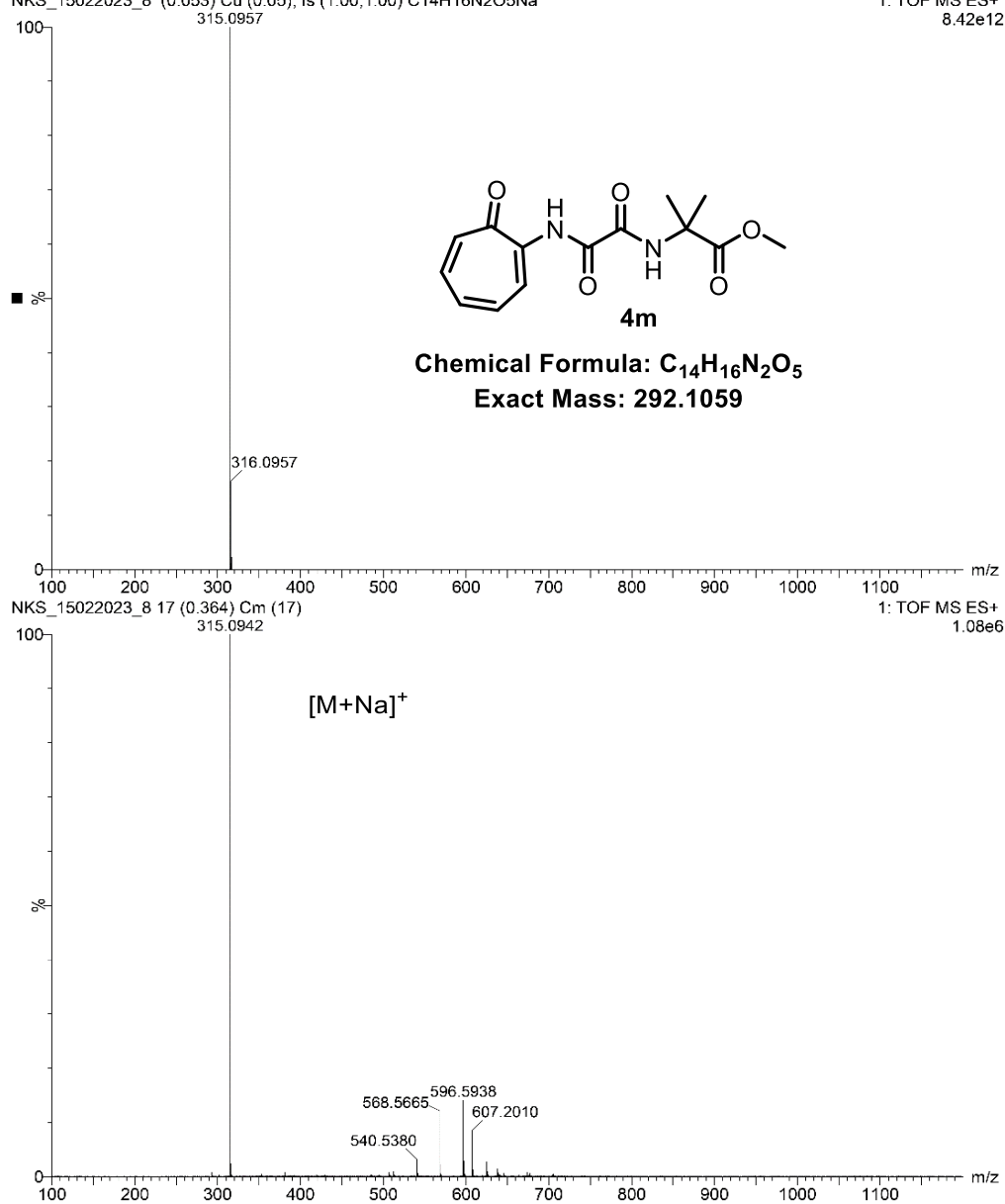


Fig S60. ESI-HRMS spectra of oxidized troponyl glycine derivative **4m**.

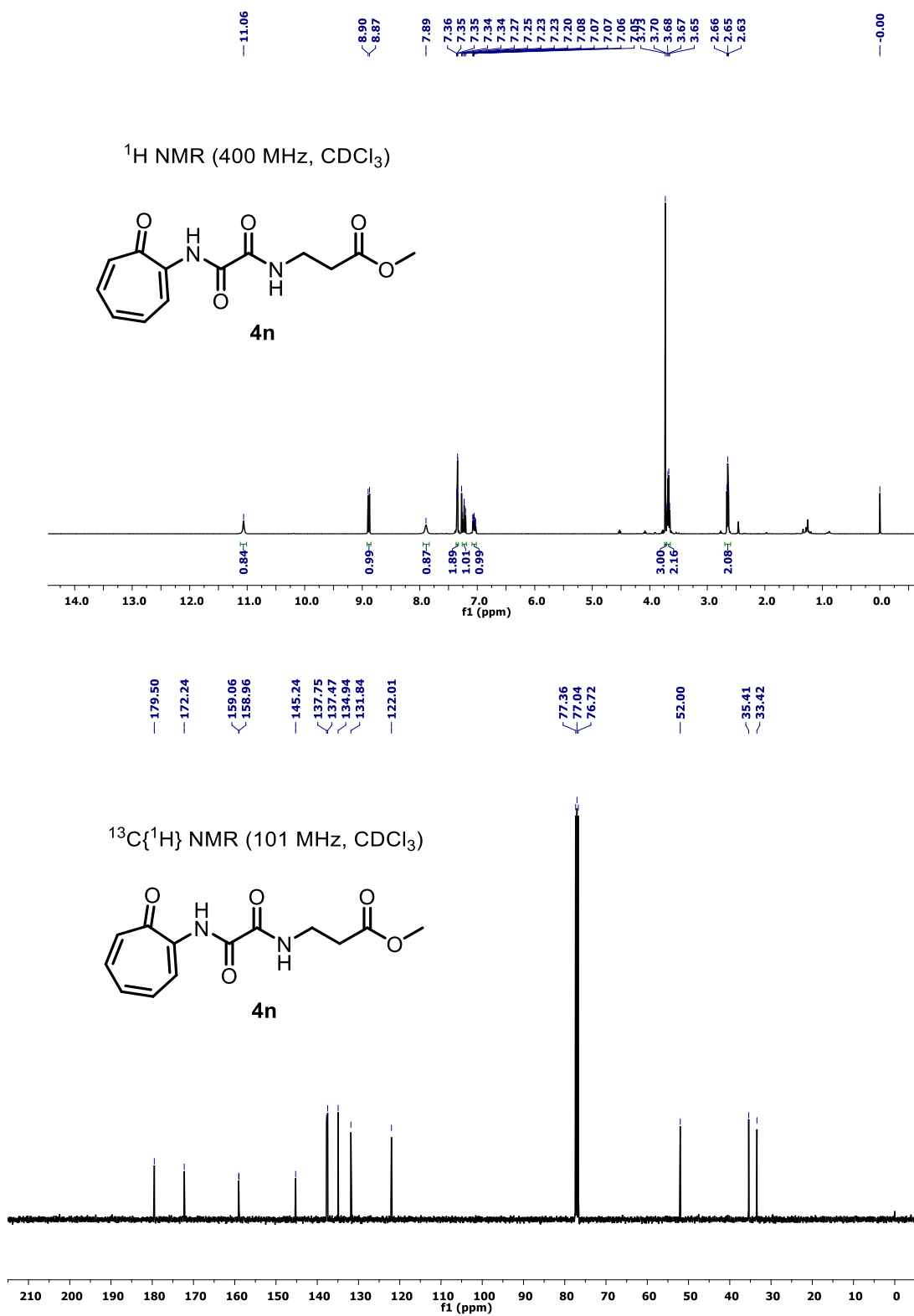


Fig S61. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4n**.

NKS_CKJ_1032

17-Feb-2023
00:23:57

XEVO-G2XSQTOF#YFA1739

NKS_16022023_8 (0.053) Cu (0.05); ls (1.00,1.00) C₁₃H₁₄N₂O₅Na

1: TOF MS ES+
8.51e12

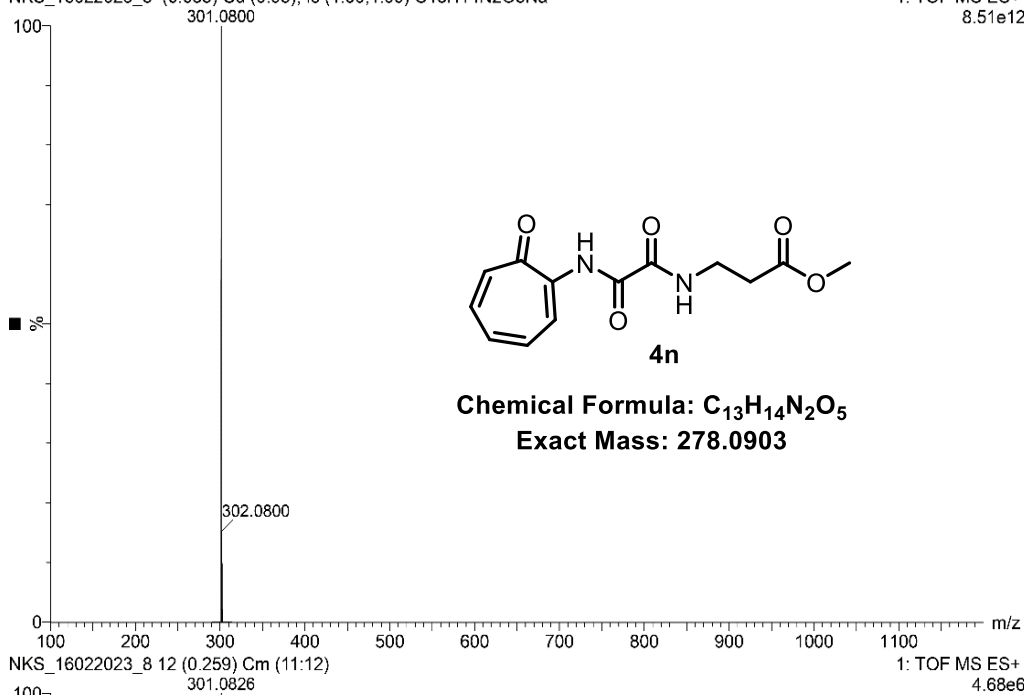


Fig S62. ESI-HRMS spectra of oxidized troponyl glycine derivative **4n**.

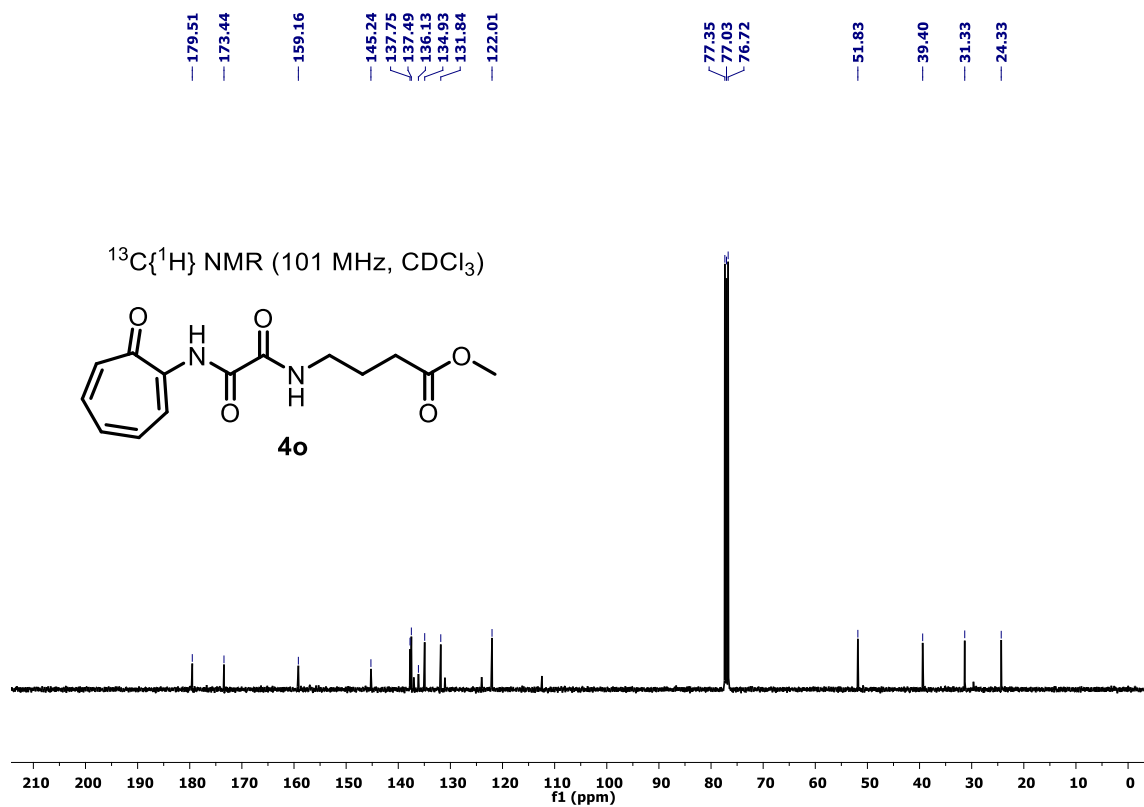
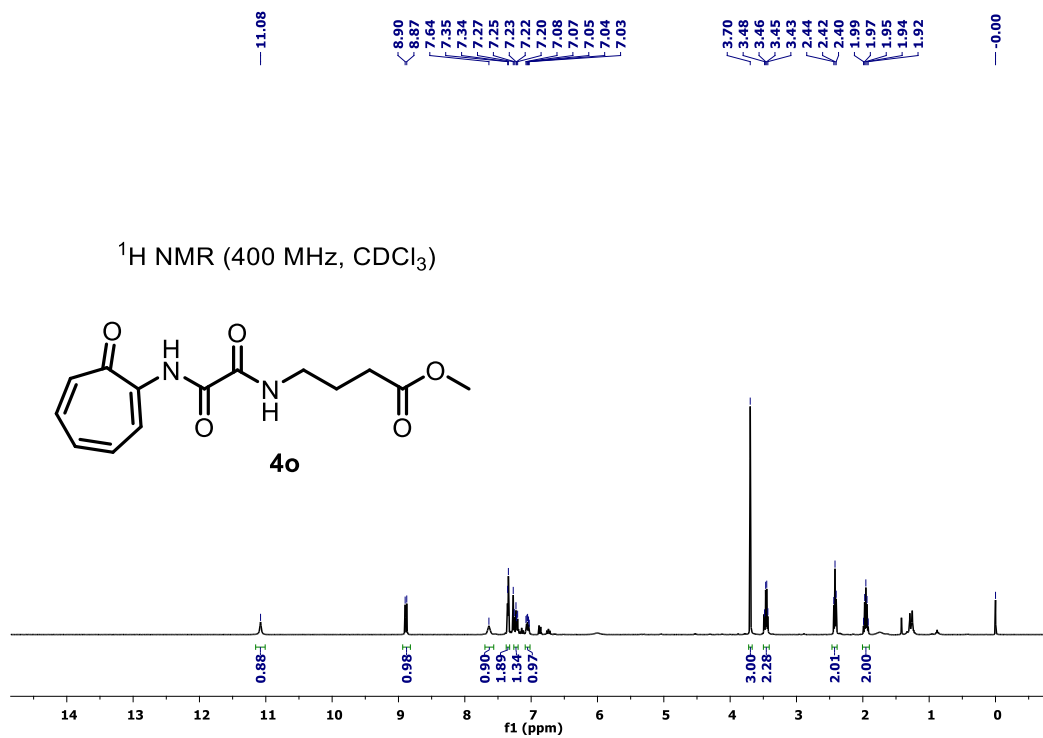


Fig S63. ¹H, ¹³C {¹H} NMR spectra of oxidized troponyl glycine derivative **4o**.

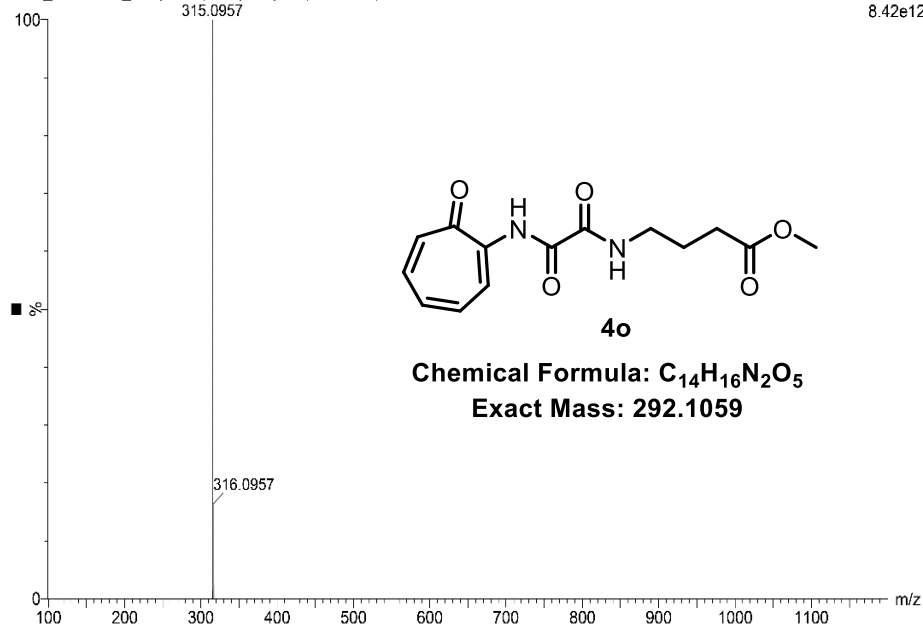
NKS_CKJ_1033

17-Feb-2023
18:46:26

XEVO-G2XSQTOF#YFA1739

NKS_17022023_14 (0.053) Cu (0.05); Is (1.00,1.00) C₁₄H₁₆N₂O₅Na

1: TOF MS ES+
8.42e12



NKS_17022023_14 49 (0.984) Cm (47:49)

1: TOF MS ES+
2.56e7

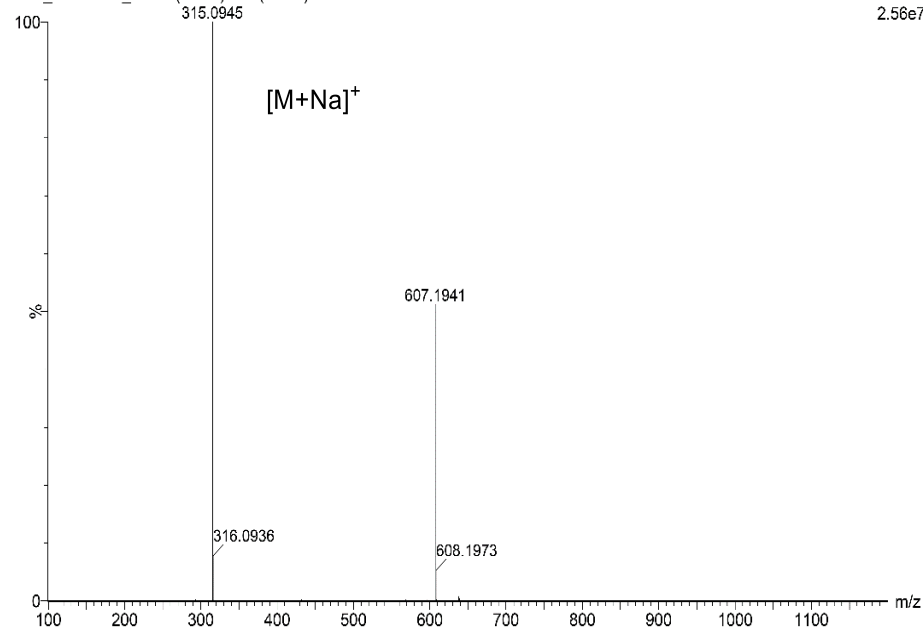


Fig S64. ESI-HRMS spectra of oxidized troponyl glycine derivative **4o**.

3. NMR Spectra of Non-troponyl (*N*-Aryl and *N*- Alkyl) Glycine Derivatives (*N*-phenyl glycinate and *N,N*- diethyl glycinate)

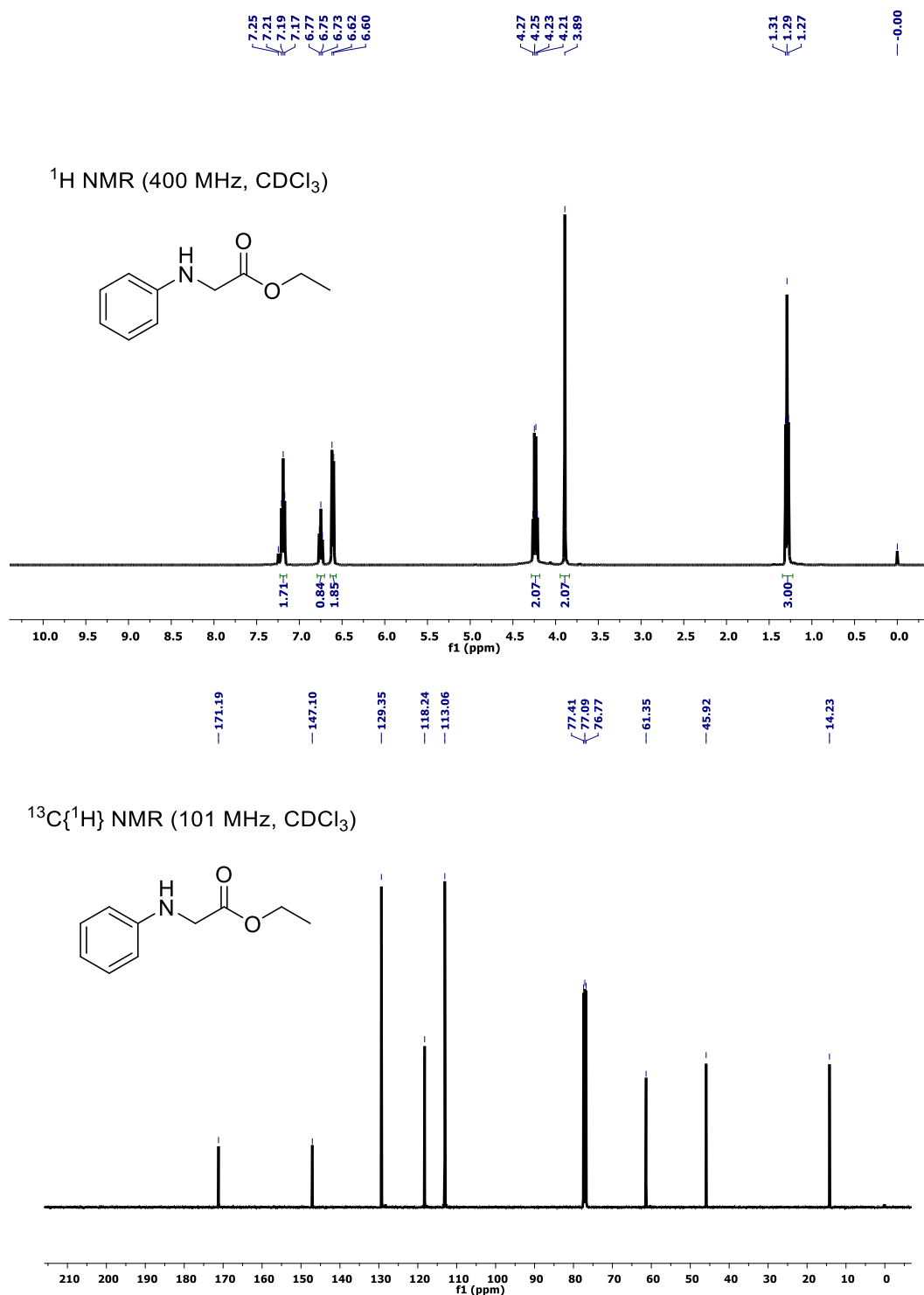


Fig S65. ¹H, ¹³C {¹H} NMR spectra of *N*-phenyl glycinate.

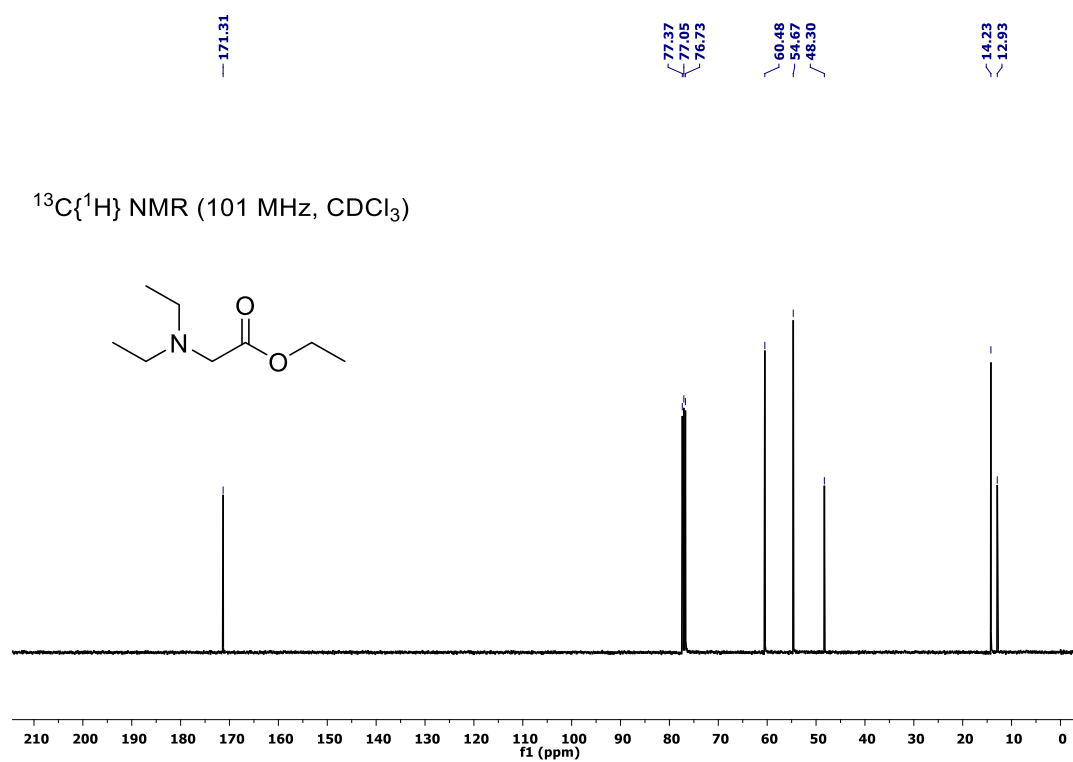
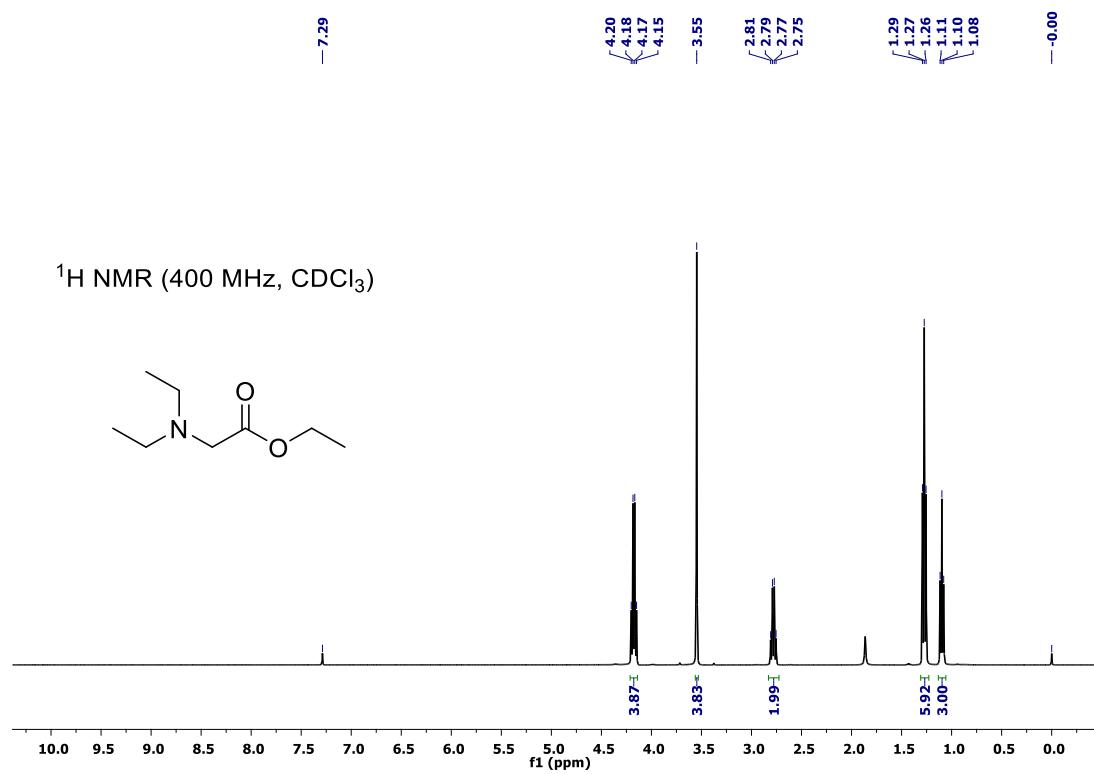


Fig S66. ¹H, ¹³C {¹H} NMR spectra of *N, N*- diethyl glycinate.

4. X-Ray Studies of Single Crystals (**2a/4a/4b/4f/ 4g/2-Pd**)

Compound 2a. Single crystal of oxidized troponyl glycine derivative **2a** was obtained in solvent mixture ethylacetate and hexane by slow evaporation method. The crystal data of oxidized troponyl glycine derivative **2a** was collected on a Rigaku Oxford diffractometer at 100 K. Selected collection parameters and other crystallographic results are summarized below. The program package SHELXTL1 and Olex2 was used for structure solution and ORTEP diagram carried out by DIAMOND 3.2.

Table S1 Crystal data and structure refinement for 2a.

Identification code	2a
Empirical formula	C ₁₁ H ₁₁ NO ₄
Formula weight	221.21
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	13.3680(14)
b/Å	4.2874(5)
c/Å	19.184(2)
α/°	90
β/°	110.238(13)
γ/°	90
Volume/Å ³	1031.6(2)
Z	4
ρ _{calc} /cm ³	1.424
μ/mm ⁻¹	0.110
F (000)	464.0
Crystal size/mm ³	0.01 × 0.01 × 0.001
Radiation	Mo Kα (λ = 0.71073)
2θ range for data collection/°	6.496 to 60.238
Index ranges	-17 ≤ h ≤ 17, -5 ≤ k ≤ 5, -21 ≤ l ≤ 26
Reflections collected	6699
Independent reflections	2388 [R _{int} = 0.0549, R _{sigma} = 0.0757]
Data/restraints/parameters	2388/0/146
Goodness-of-fit on F ²	1.001
Final R indexes [I >= 2σ (I)]	R ₁ = 0.0702, wR ₂ = 0.1823
Final R indexes [all data]	R ₁ = 0.1037, wR ₂ = 0.2041
Largest diff. peak/hole / e Å ⁻³	0.44/-0.38

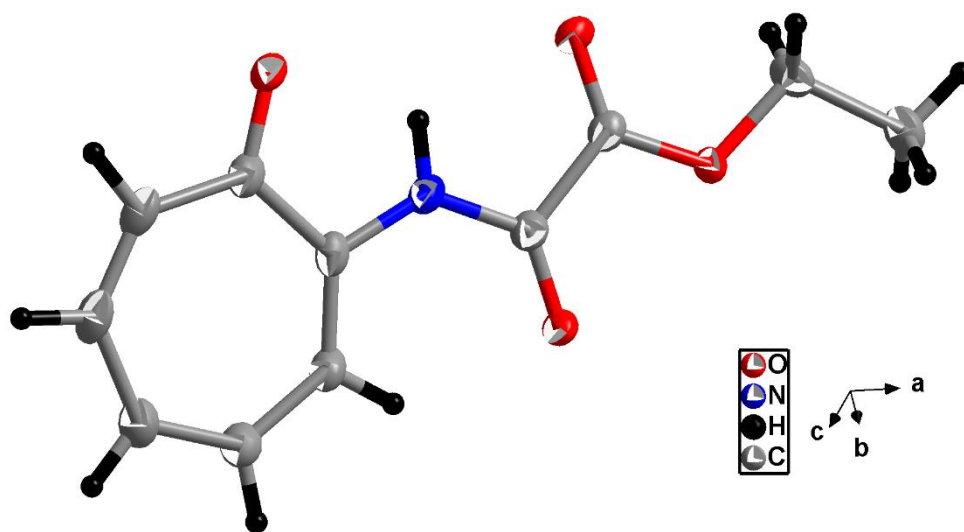


Fig S67. ORTEP diagram of oxidized troponyl glycine derivative **2a** [ellipsoid contour probability: 50%].

Compound 4a. Single crystal of oxidized troponyl glycine derivative **4a** was obtained in solvent mixture ethylacetate and hexane by slow evaporation method. The crystal data of oxalamide **4a** was collected on a Rigaku Oxford diffractometer at 292 K. Selected collection parameters and other crystallographic results are summarized below. The program package SHELXTL1 and Olex2 was used for structure solution and ORTEP diagram carried out by DIAMOND 3.2.

Table S2 Crystal data and structure refinement for 4a.

Identification code	4a
Empirical formula	C ₁₀ H ₂₂ N ₂ O ₇
Formula weight	282.29
Temperature/K	292(3)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	12.9666(11)
b/Å	13.3233(10)
c/Å	8.2903(6)
α/°	90
β/°	102.069(8)
γ/°	90
Volume/Å ³	1400.56(19)
Z	4
ρ _{calc} /cm ³	1.339
μ/mm ⁻¹	0.113
F (000)	608.0
Crystal size/mm ³	0.021 × 0.012 × 0.001
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	6.91 to 52.732
Index ranges	-16 ≤ h ≤ 15, -16 ≤ k ≤ 15, -9 ≤ l ≤ 10
Reflections collected	12306
Independent reflections	2846 [R _{int} = 0.0297, R _{sigma} = 0.0262]
Data/restraints/parameters	2846/0/190
Goodness-of-fit on F ²	0.791
Final R indexes [I >= 2σ (I)]	R ₁ = 0.0417, wR ₂ = 0.1687
Final R indexes [all data]	R ₁ = 0.0563, wR ₂ = 0.1942
Largest diff. peak/hole / e Å ⁻³	0.15/-0.16

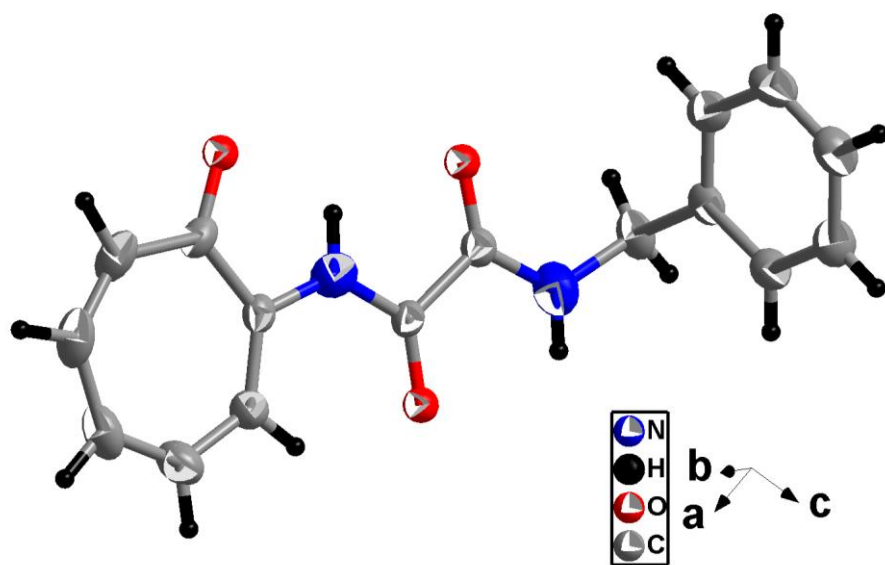


Fig S68. ORTEP diagram of oxidized troponyl glycine derivative **4a** [ellipsoid contour probability: 50%].

Compound 4b. Single crystal of oxidized troponyl glycine derivative **4b** was obtained in solvent mixture ethylacetate and hexane by slow evaporation method. The crystal data of oxalamide **4b** was collected on a Rigaku Oxford diffractometer at 248 K. Selected - collection parameters and other crystallographic results are summarized below. The program package SHELXTL1 and Olex2 was used for structure solution and ORTEP diagram carried out by DIAMOND 3.2.

Table S3 Crystal data and structure refinement for 4b.

Identification code	4b
Empirical formula	C ₁₇ H ₁₆ N ₂ O ₃
Formula weight	296.32
Temperature/K	248(50)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	13.8803(10)
b/Å	13.7121(9)
c/Å	8.2130(5)
α/°	90
β/°	104.208(7)
γ/°	90
Volume/Å ³	1515.35(18)
Z	4
ρ _{calc} /cm ³	1.299
μ/mm ⁻¹	0.090
F (000)	624.0
Crystal size/mm ³	0.01 × 0.01 × 0.001
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	6.67 to 60.264
Index ranges	-18 ≤ h ≤ 16, -17 ≤ k ≤ 19, -11 ≤ l ≤ 10
Reflections collected	14346
Independent reflections	3667 [R _{int} = 0.0384, R _{sigma} = 0.0361]
Data/restraints/parameters	3667/0/199
Goodness-of-fit on F ²	1.045
Final R indexes [I>=2σ (I)]	R ₁ = 0.0433, wR ₂ = 0.1122
Final R indexes [all data]	R ₁ = 0.0697, wR ₂ = 0.1252
Largest diff. peak/hole / e Å ⁻³	0.14/-0.14

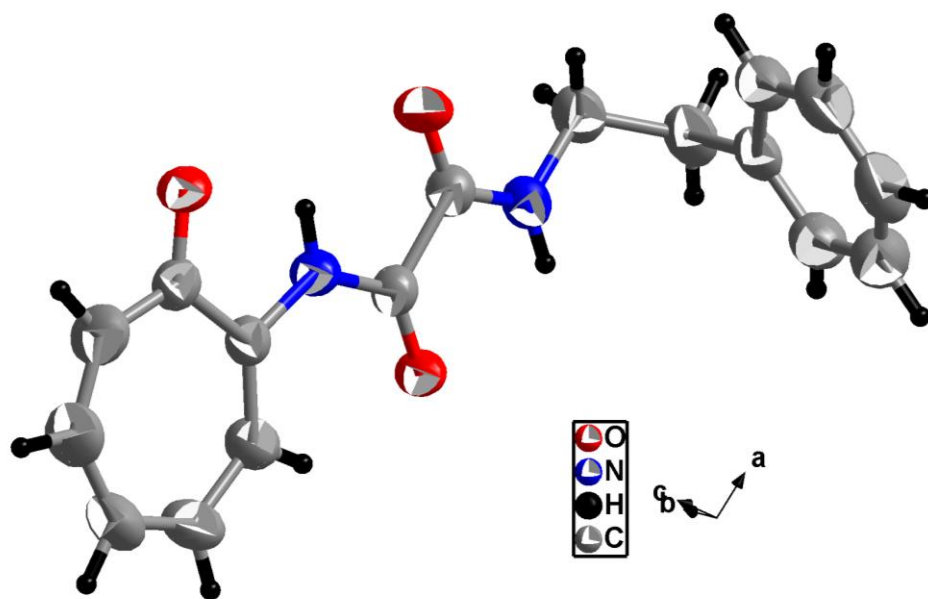


Fig S69. ORTEP diagram of oxidized troponyl glycine derivative **4b** [ellipsoid contour probability: 50%].

Compound 4f. Single crystal of oxidized troponyl glycine derivative **4f** was obtained in solvent mixture ethylacetate and hexane by slow evaporation method. The crystal data of oxalamide **4f** was collected on a Rigaku Oxford diffractometer at 205 K. Selected - collection parameters and other crystallographic results are summarized below. The program package SHELXTL1 and Olex2 was used for structure solution and ORTEP diagram carried out by DIAMOND 3.2.

Table S4 Crystal data and structure refinement for 4f.

Identification code	4f
Empirical formula	C _{2.18} H _{2.18} N _{0.36} O _{0.91}
Formula weight	48.04
Temperature/K	205(6)
Crystal system	triclinic
Space group	P-1
a/Å	8.1779(9)
b/Å	9.0116(6)
c/Å	10.0761(8)
α/°	113.570(7)
β/°	95.895(8)
γ/°	112.019(9)
Volume/Å ³	602.44(10)
Z	11
ρ _{calc} /cm ³	1.457
μ/mm ⁻¹	0.115
F (000)	276.0
Crystal size/mm ³	0.01 × 0.01 × 0.001
Radiation	Mo Kα (λ = 0.71073)
2θ range for data collection/°	6.812 to 60.938
Index ranges	-10 ≤ h ≤ 10, -11 ≤ k ≤ 11, -13 ≤ l ≤ 13
Reflections collected	10853
Independent reflections	2925 [R _{int} = 0.0514, R _{sigma} = 0.0472]
Data/restraints/parameters	2925/0/173
Goodness-of-fit on F ²	1.033
Final R indexes [I >= 2σ (I)]	R ₁ = 0.0499, wR ₂ = 0.1214
Final R indexes [all data]	R ₁ = 0.0764, wR ₂ = 0.1371
Largest diff. peak/hole / e Å ⁻³	0.22/-0.31

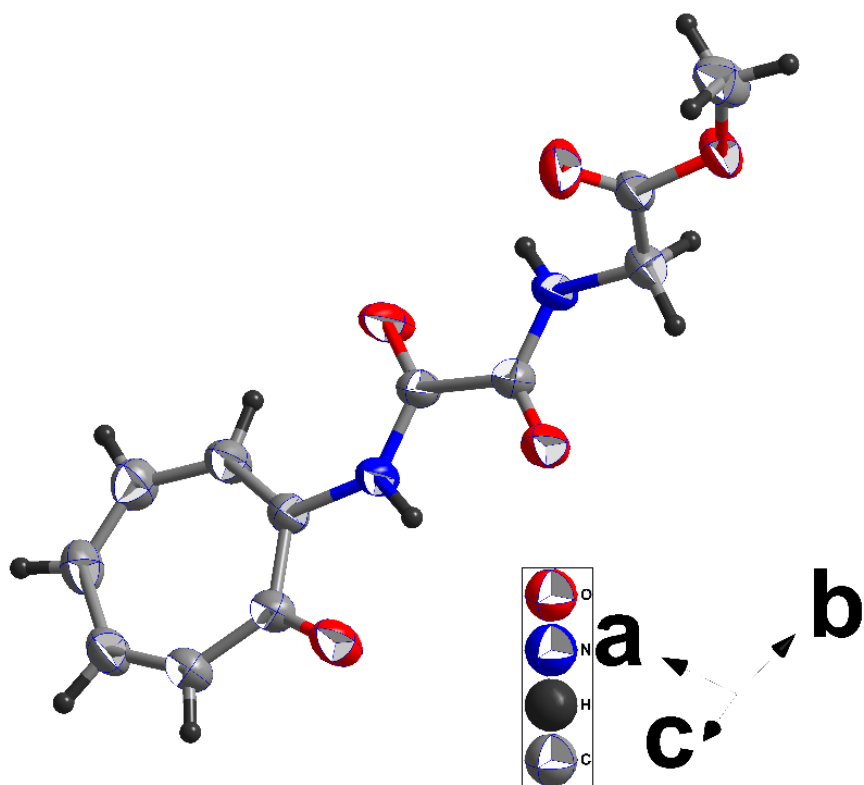


Fig S70. ORTEP diagram of oxidized troponyl glycine derivative **4f** [ellipsoid contour probability: 50%].

Compound 4g. Single crystal of oxidized troponyl glycine derivative **4g** was obtained in solvent mixture ethylacetate and hexane by slow evaporation method. The crystal data of oxalamide **4g** was collected on a Rigaku Oxford diffractometer at 193 K. Selected - collection parameters and other crystallographic results are summarized below. The program package SHELXTL1 and Olex2 was used for structure solution and ORTEP diagram carried out by DIAMOND 3.2.

Table S5 Crystal data and structure refinement for 4g.

Identification code	4g
Empirical formula	C ₂₆ H ₂₈ N ₄ O ₁₀
Formula weight	556.52
Temperature/K	193(14)
Crystal system	orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁
a/Å	7.0174(2)
b/Å	19.6228(4)
c/Å	19.9031(5)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	2740.68(12)
Z	4
ρ _{calc} /cm ³	1.349
μ/mm ⁻¹	0.889
F (000)	1168.0
Crystal size/mm ³	0.01 × 0.01 × 0.001
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	8.886 to 151.168
Index ranges	-8 ≤ h ≤ 4, -24 ≤ k ≤ 24, -25 ≤ l ≤ 24
Reflections collected	12621
Independent reflections	5077 [R _{int} = 0.0389, R _{sigma} = 0.0477]
Data/restraints/parameters	5077/0/365
Goodness-of-fit on F ²	1.040
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0434, wR ₂ = 0.1179
Final R indexes [all data]	R ₁ = 0.0497, wR ₂ = 0.1230
Largest diff. peak/hole / e Å ⁻³	0.15/-0.16
Flack parameter	0.21(16)

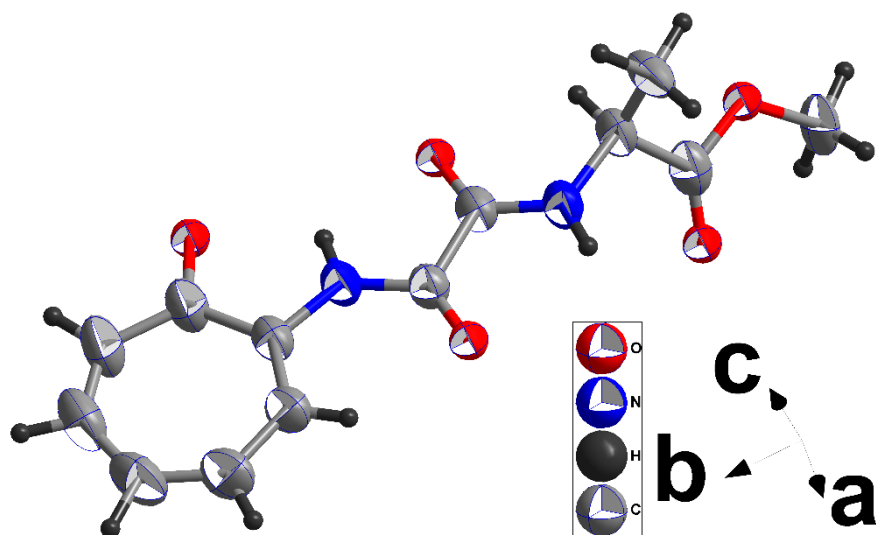


Fig S71. ORTEP diagram of oxidized troponyl glycine derivative **4g** [ellipsoid contour probability: 50%].

Compound 2-Pd(II) complex. Single crystal of *troponyl* glycine derivative- Pd complex (2- Pd) was obtained in solvent mixture ethylacetate and hexane by slow evaporation method. The crystal data of palladacycle (2- Pd) was collected on a Rigaku Oxford diffractometer at 270 K. Selected collection parameters and other crystallographic results are summarized below. The program package SHELXTL1 and Olex2 was used for structure solution and ORTEP diagram carried out by DIAMOND 3.2.

Table S6 Crystal data and structure refinement for 2- Pd.

Identification code	2- Pd
Empirical formula	C ₁₁ H ₁₂ NO ₃ Pd _{0.5}
Formula weight	259.42
Temperature/K	270(3)
Crystal system	monoclinic
Space group	P2 ₁ /n
a/Å	13.4678(9)
b/Å	5.0155(3)
c/Å	17.0923(10)
α/°	90
β/°	111.300(7)
γ/°	90
Volume/Å ³	1075.68(12)
Z	4
ρ _{calc} /cm ³	1.602
μ/mm ⁻¹	7.319
F (000)	528.0
Crystal size/mm ³	0.01 × 0.01 × 0.001
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	7.214 to 151.446
Index ranges	-16 ≤ h ≤ 16, -6 ≤ k ≤ 6, -21 ≤ l ≤ 21
Reflections collected	8238
Independent reflections	2126 [R _{int} = 0.1131, R _{sigma} = 0.0713]
Data/restraints/parameters	2126/0/143
Goodness-of-fit on F ²	1.084
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0756, wR ₂ = 0.2096
Final R indexes [all data]	R ₁ = 0.0915, wR ₂ = 0.2286
Largest diff. peak/hole / e Å ⁻³	1.53/-1.50

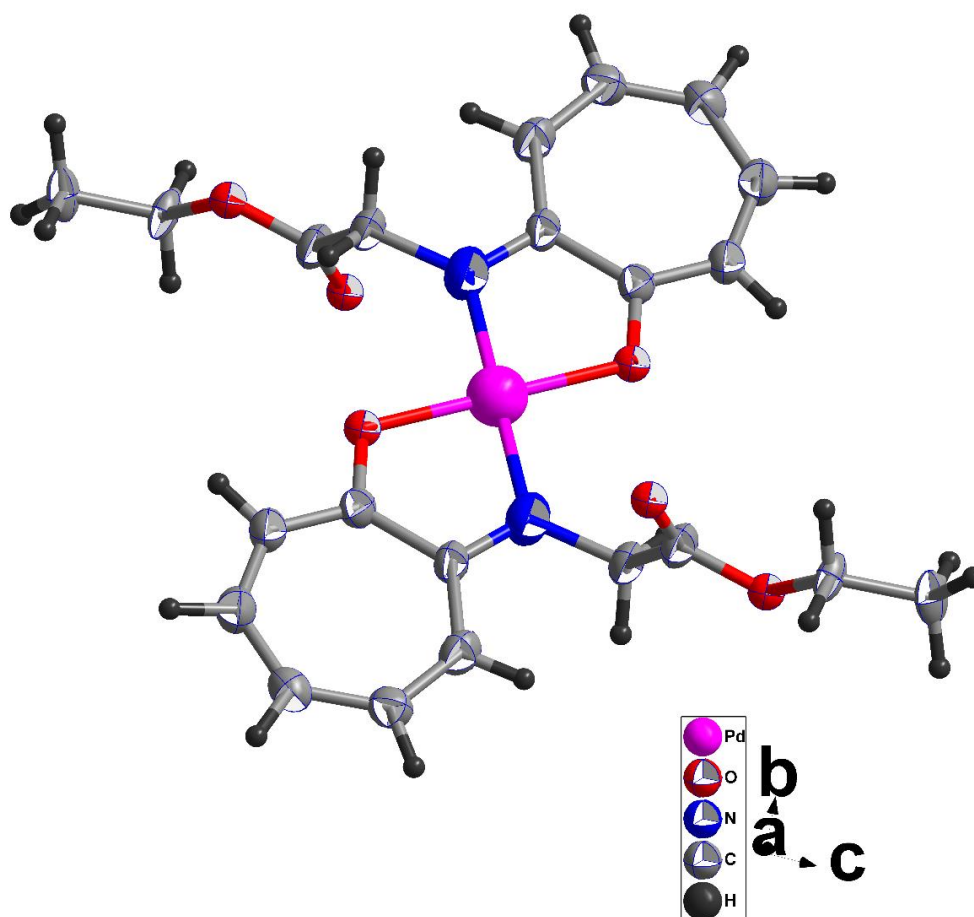


Fig S72. ORTEP diagram of *troponyl* glycine – Pd complex (**2- Pd**) [ellipsoid contour probability: 50%].