

Supplementary Information

Pd-Catalyzed C(sp²)-H Olefination: Synthesis of *N*-alkylated Isoindolinone Scaffolds from Arylamide of Amino acid esters

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

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

& HBNI-Mumbai, Mumbai-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 (3a-3l, 6a-6r)

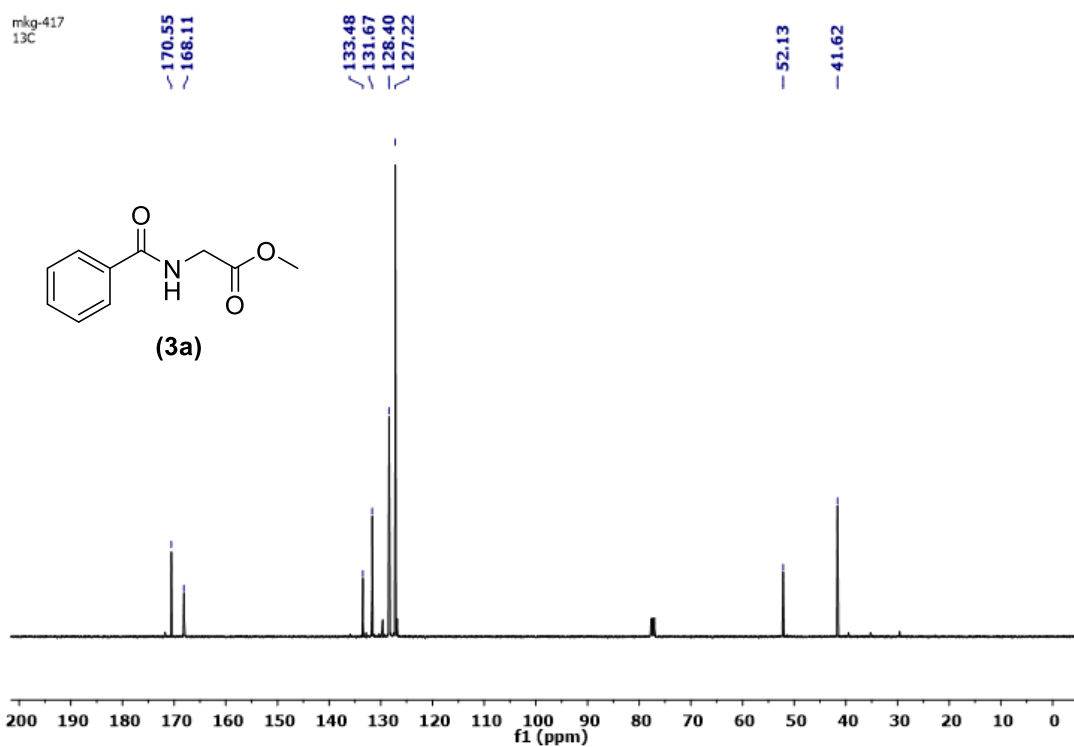
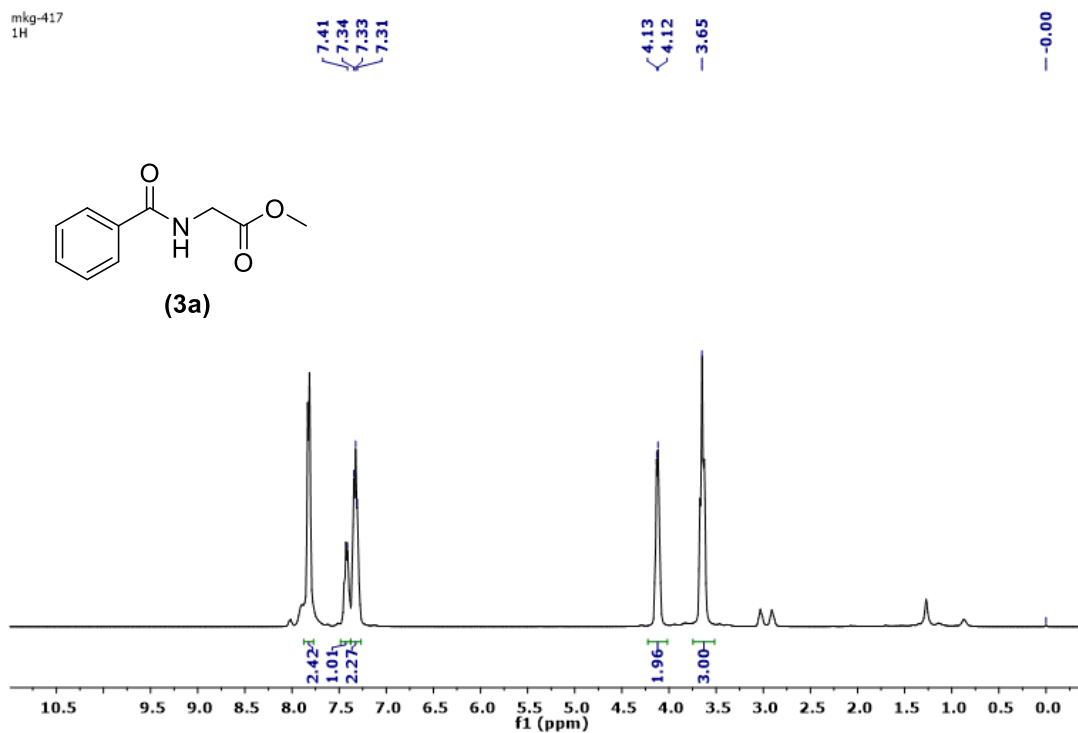


Figure. S1. ^1H , ^{13}C NMR spectra of Benzamide 3a

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Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

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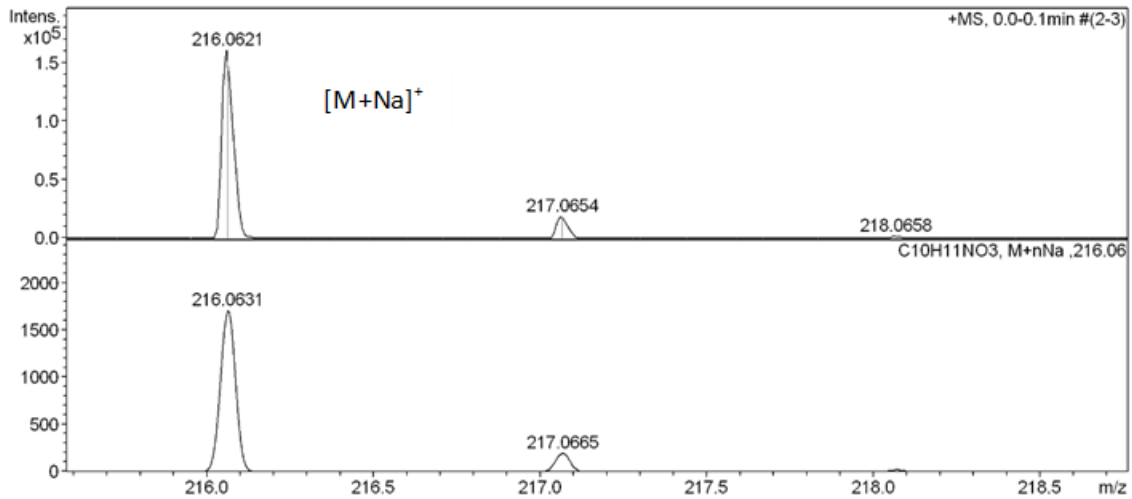
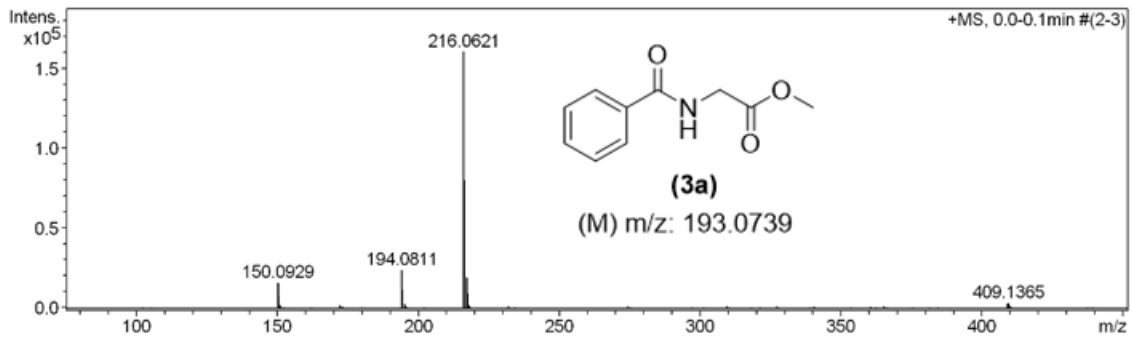
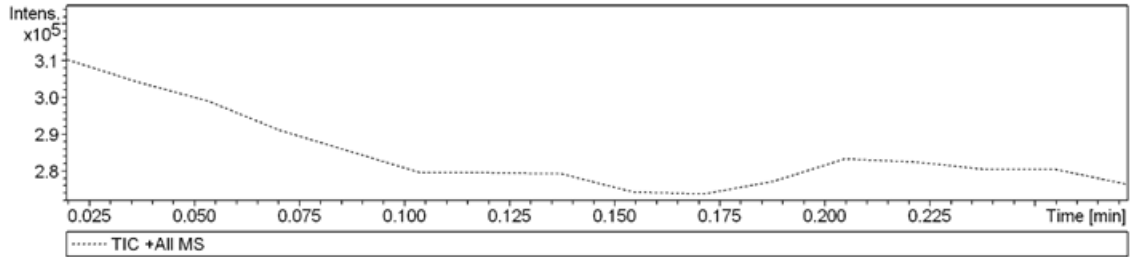


Figure. S2. ESI-HRMS spectra of Benzamide **3a**

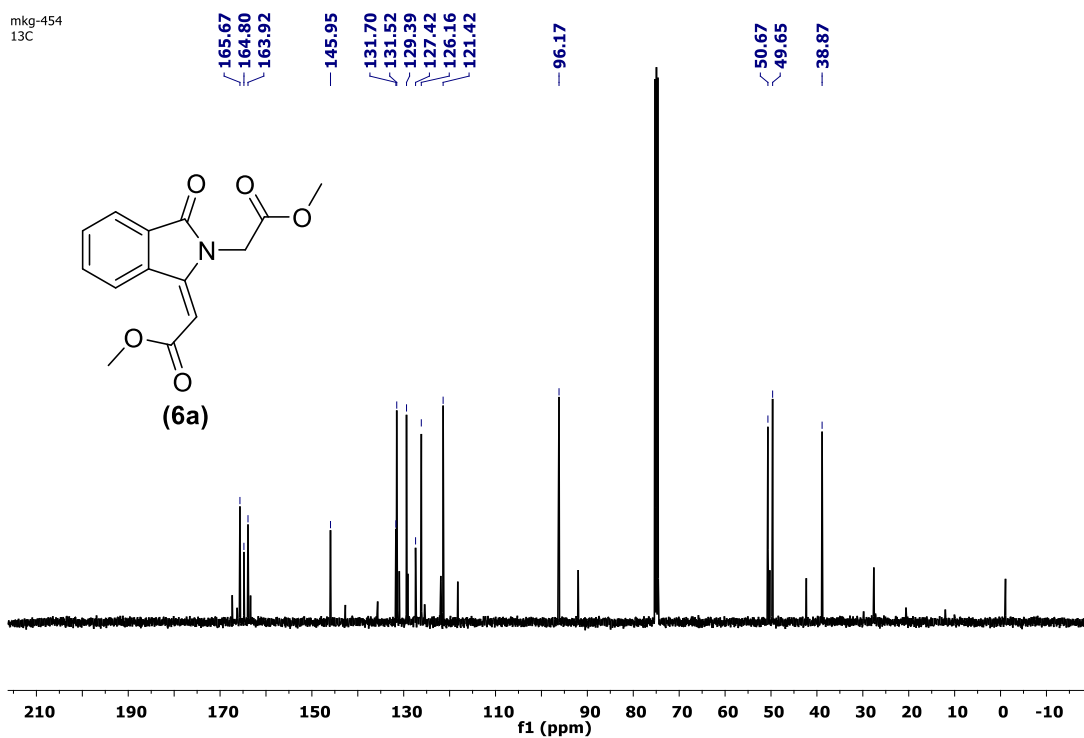
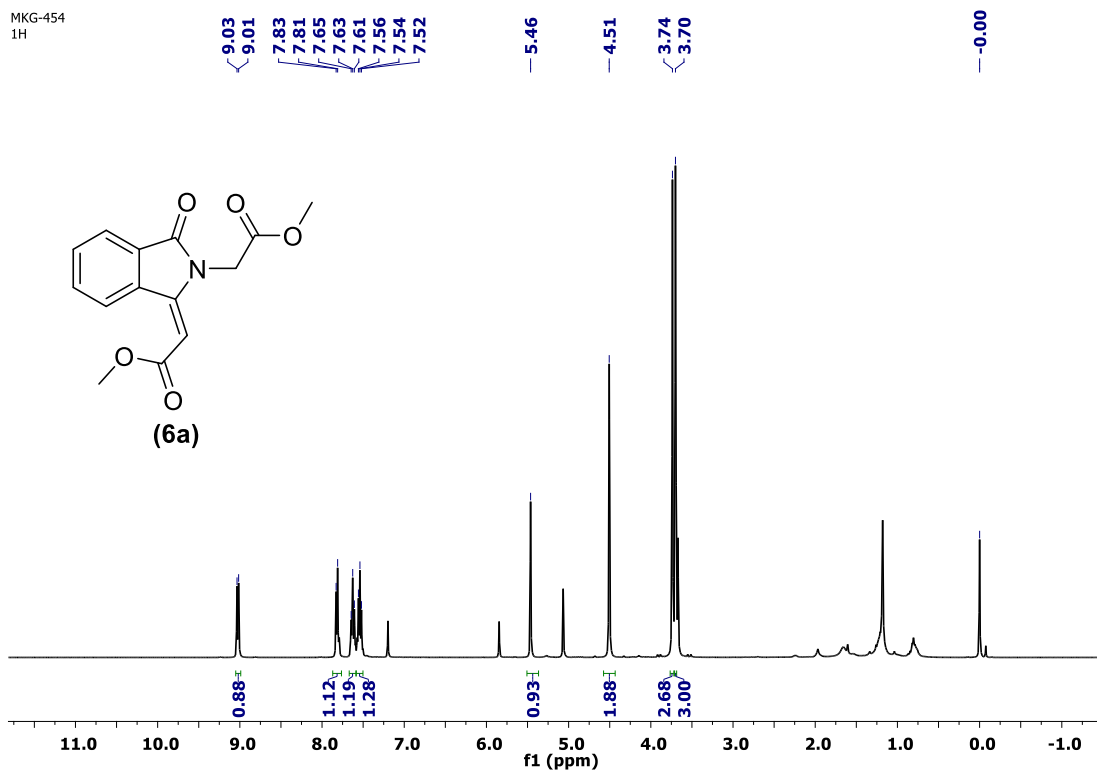


Figure. S3. ^1H , ^{13}C NMR spectra of indolinone **6a**

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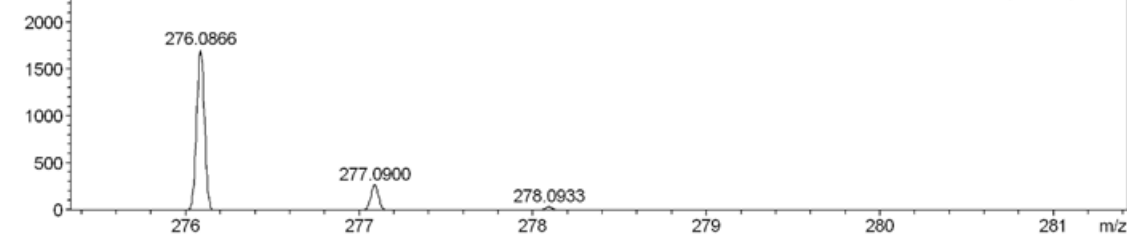
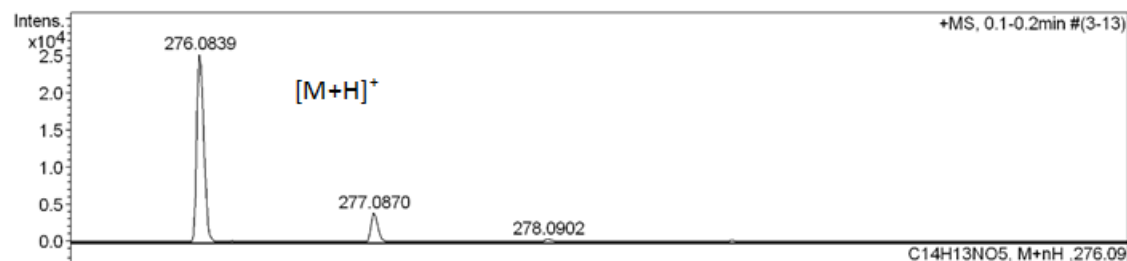
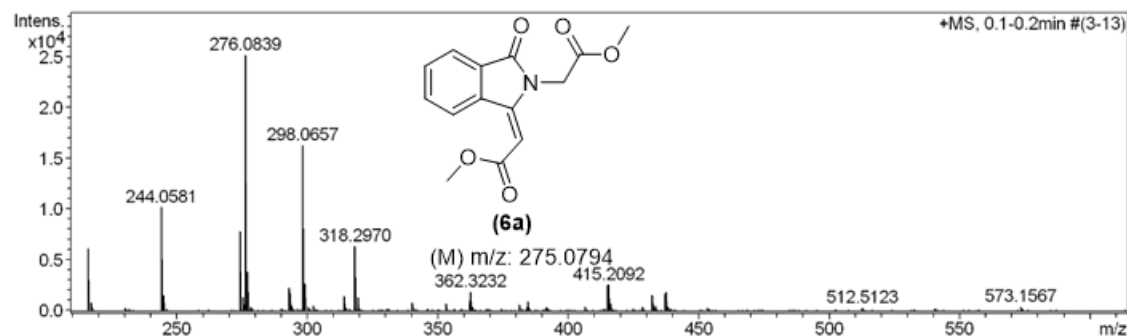
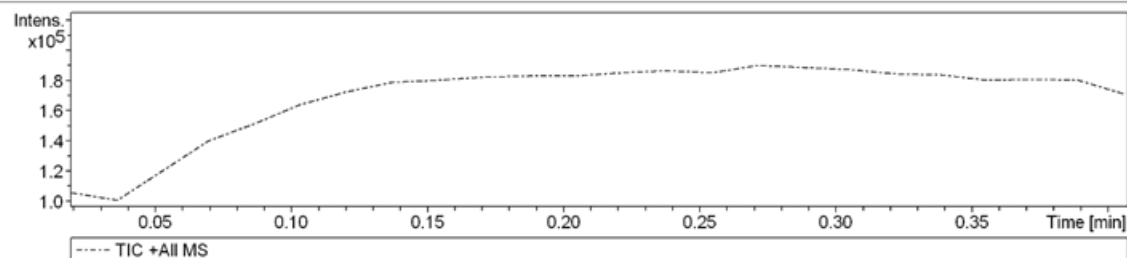


Figure. S4. ESI-HRMS spectra of indolinone **6a**

mkg-459
1H

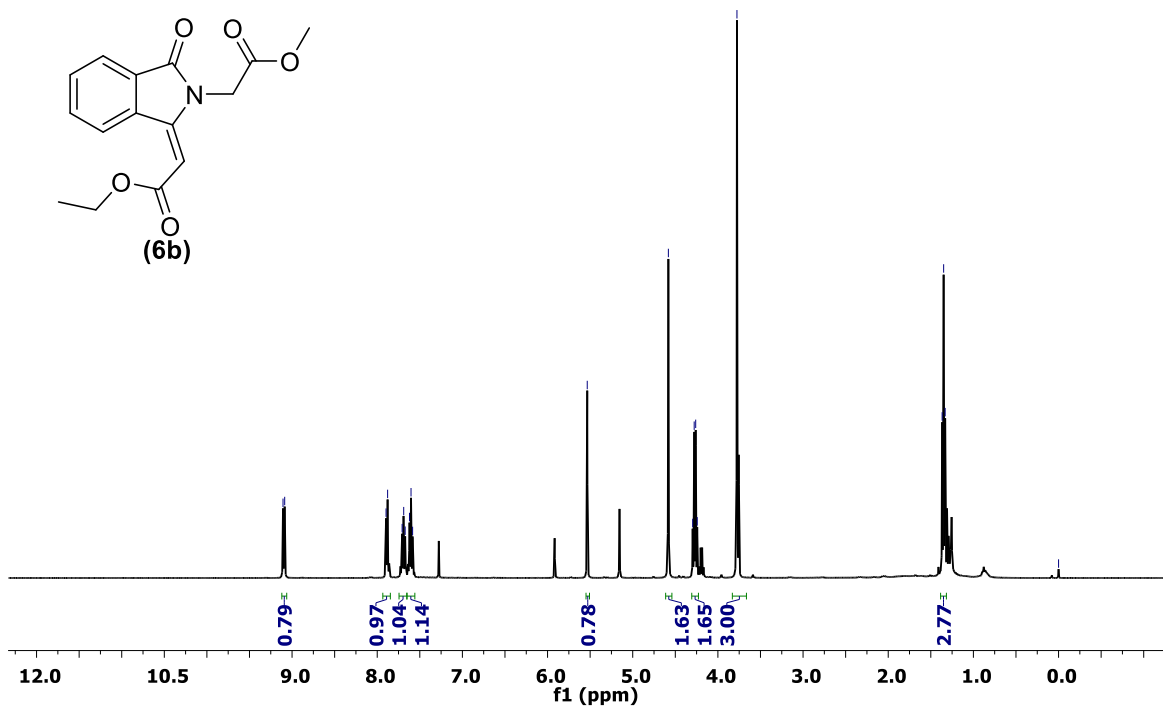
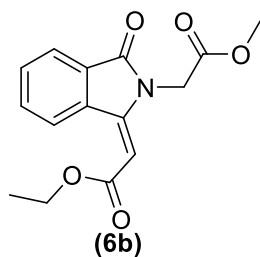
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9.09
7.90
7.88
7.71
7.69
7.67
7.62
7.60
7.58

5.53

4.58
4.30
4.28
4.26
4.24
3.78

1.37
1.35
1.33

0.00



mkg-459
13C

167.84
166.96
165.64

147.84

133.88
133.59
131.43
129.55
128.32
123.51

98.86

60.68

52.79

40.99

14.31

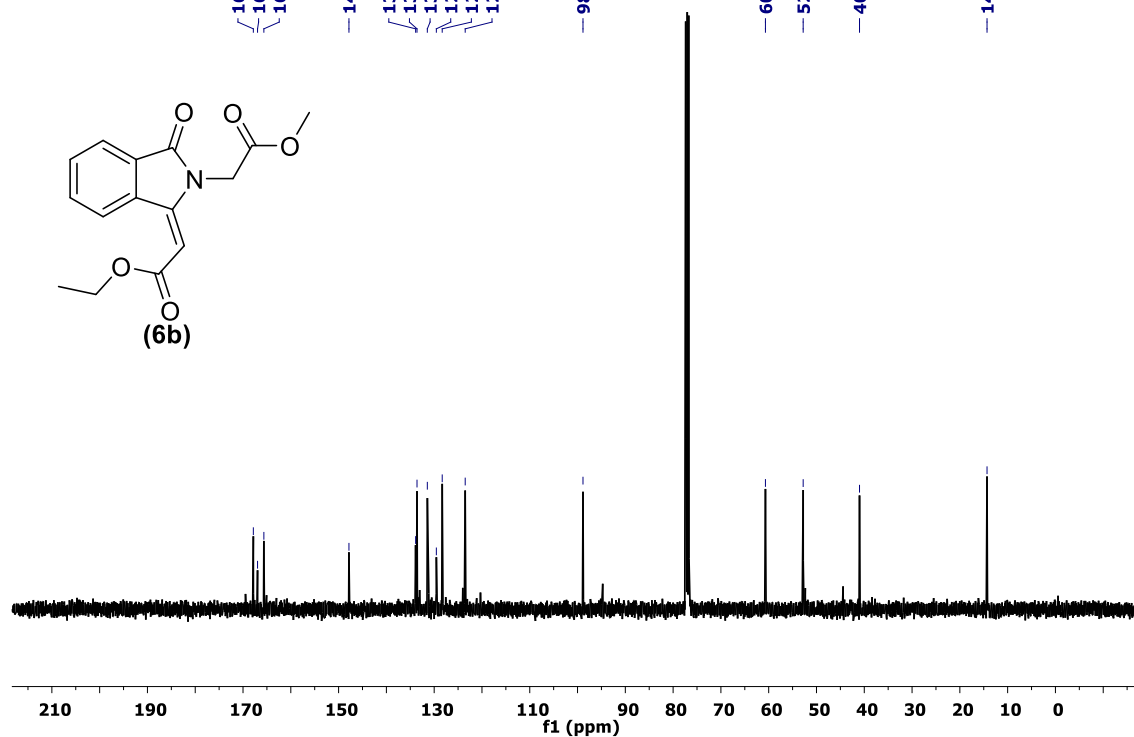
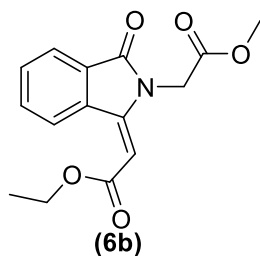


Figure. S5. ^1H , ^{13}C NMR spectra of indolinone **6b**

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Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

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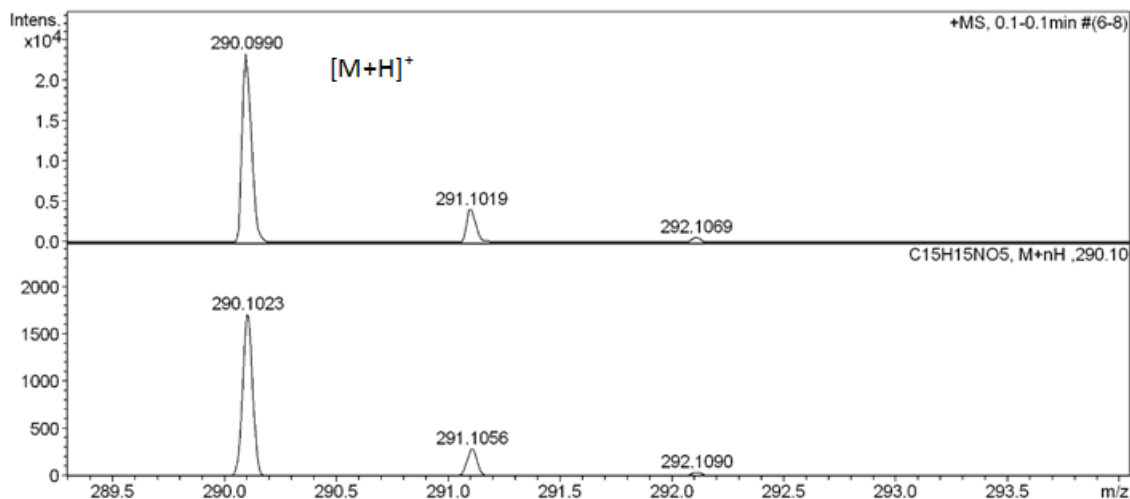
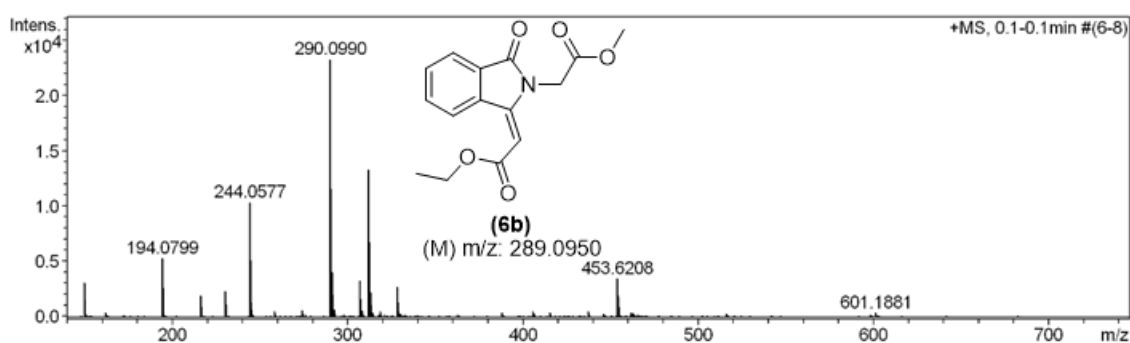
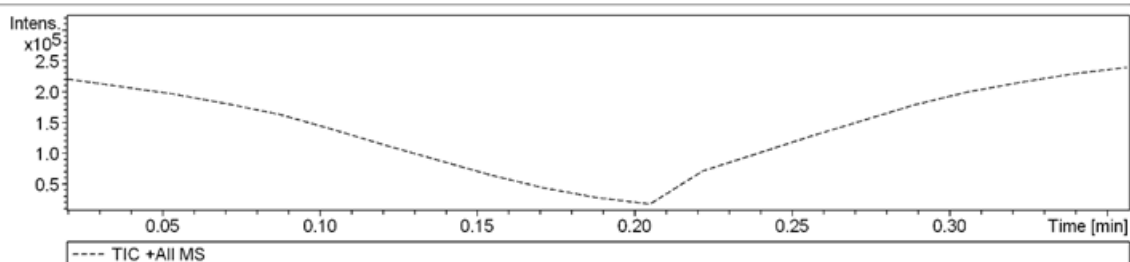


Figure. S6. ESI-HRMS spectra of indolinone **6b**

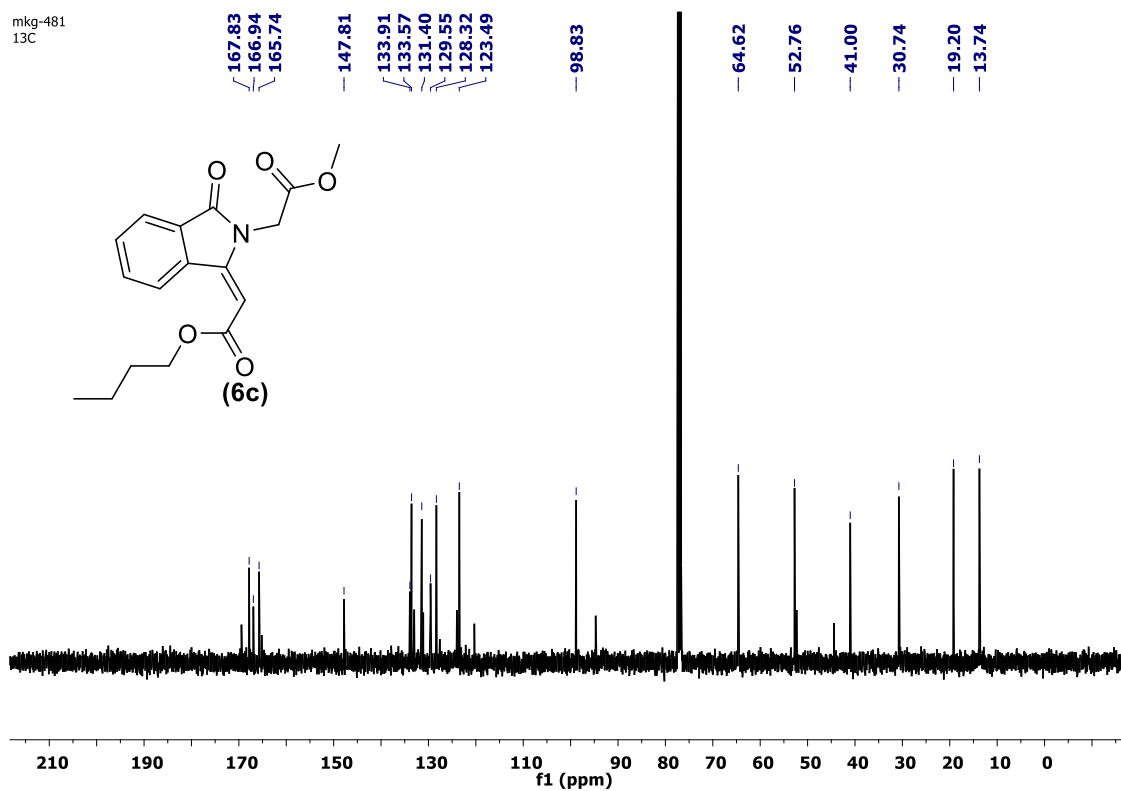
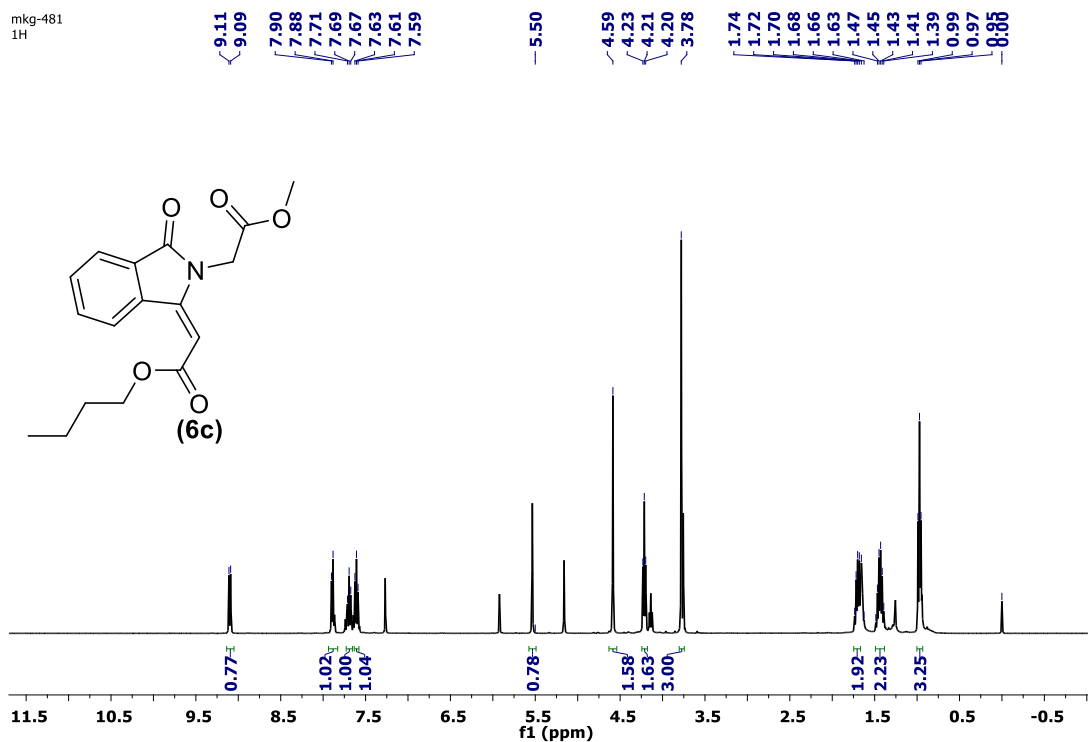


Figure. S7. ^1H , ^{13}C NMR spectra of indolinone **6c**

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Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

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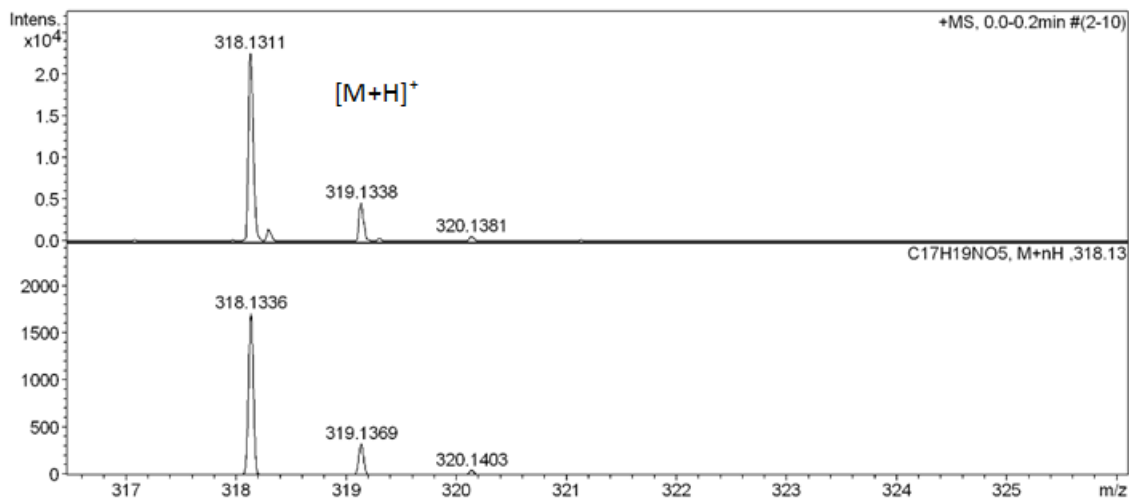
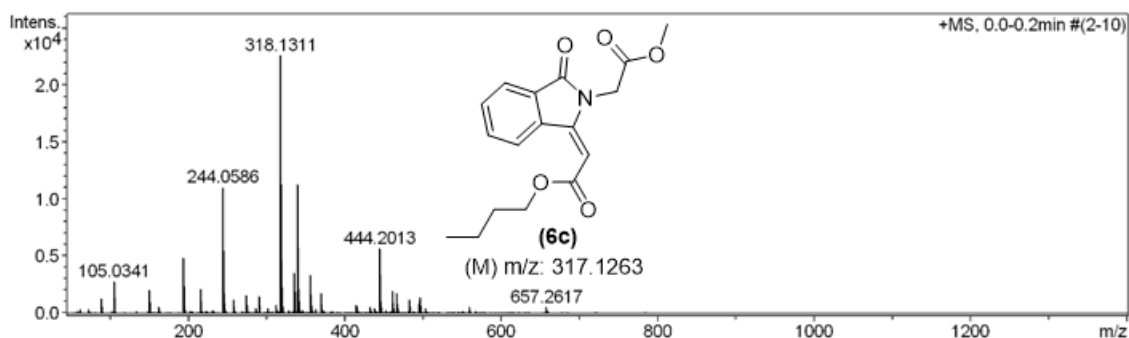
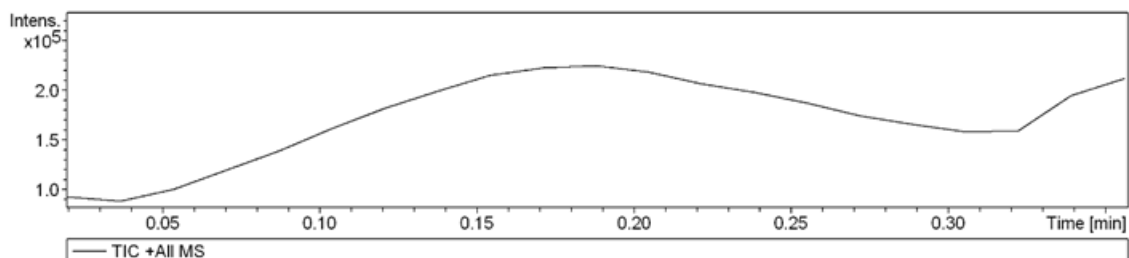
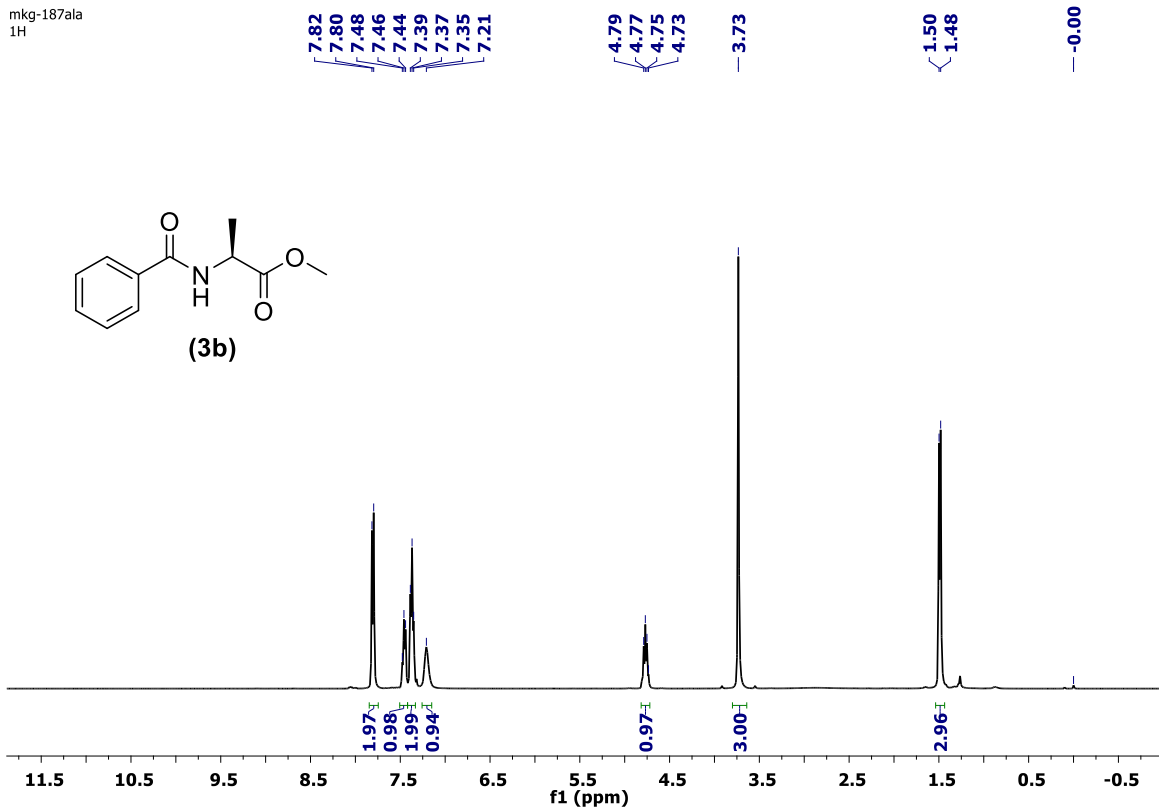


Figure. S8. ESI-HRMS spectra of indolinone **6c**

mkg-187ala
1H



mkg-187ala
13C

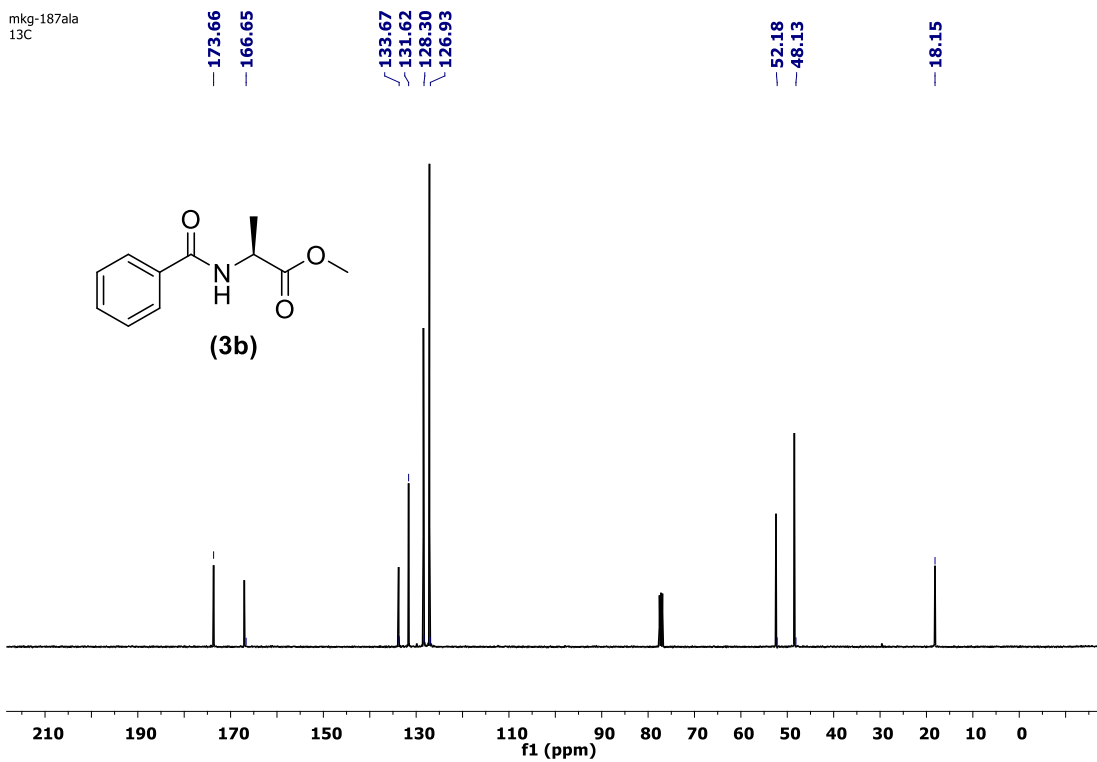


Figure. S9. ¹H, ¹³C NMR spectra of benzamide 3b

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Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

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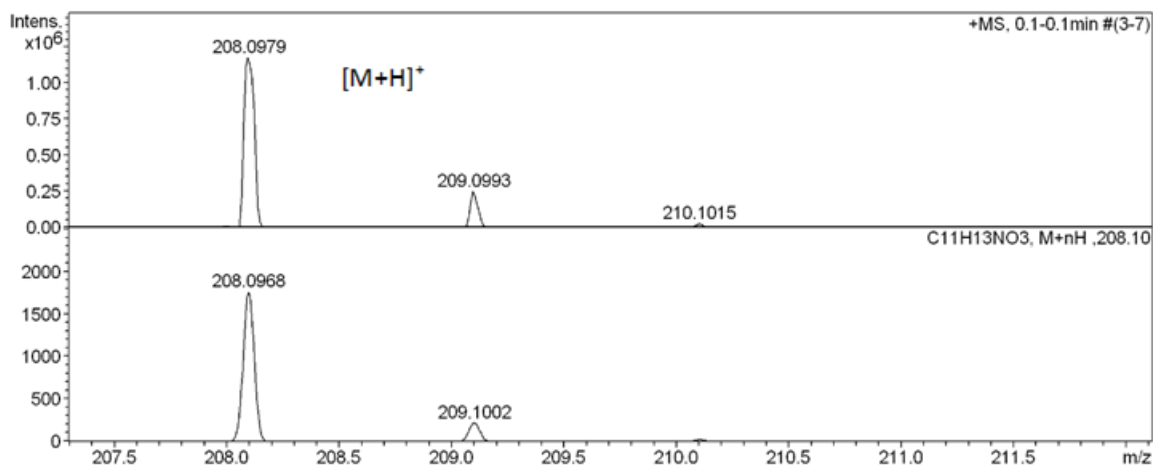
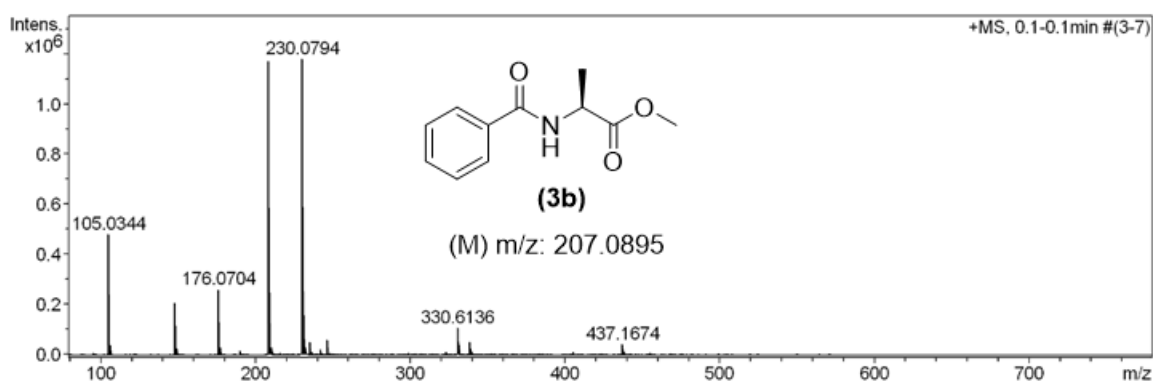
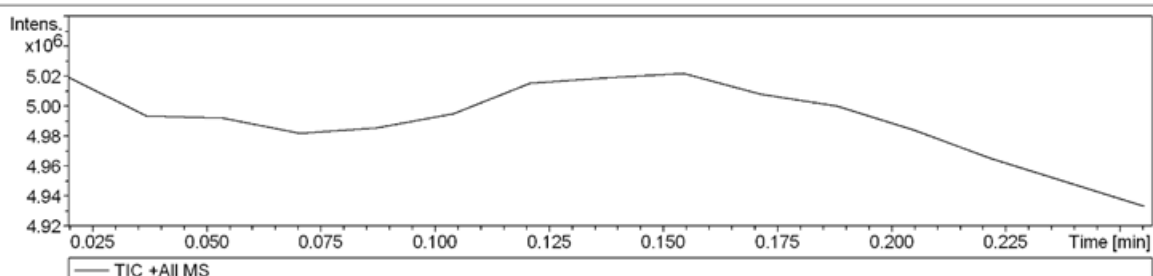


Figure. S10. ESI-HRMS spectra of benzamide **3b**

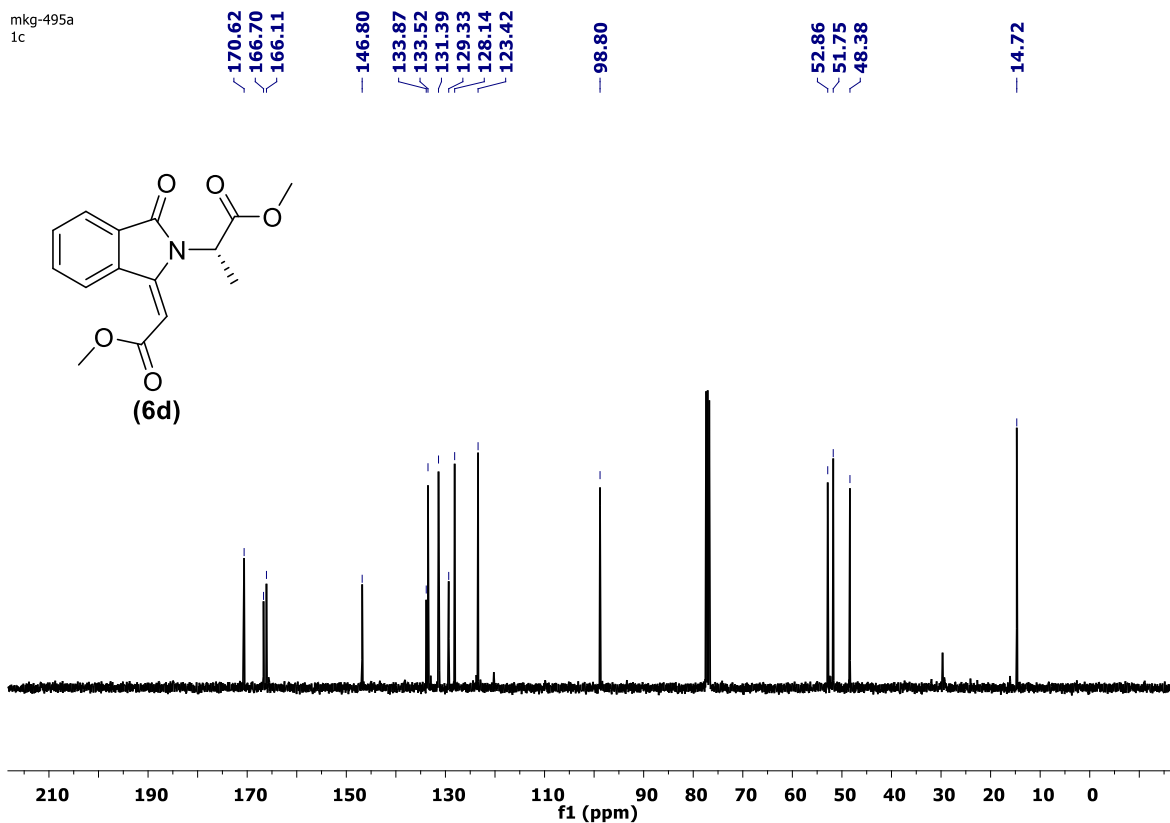
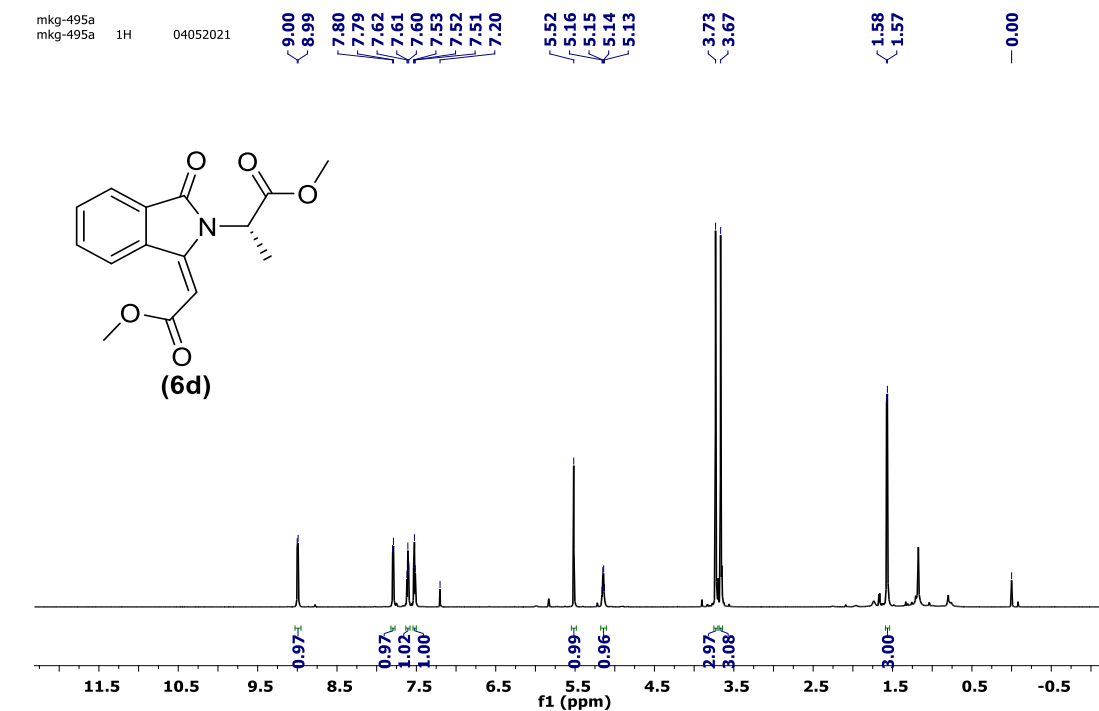


Figure. S11. ^1H , ^{13}C NMR spectra of indolinone **6d**

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Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

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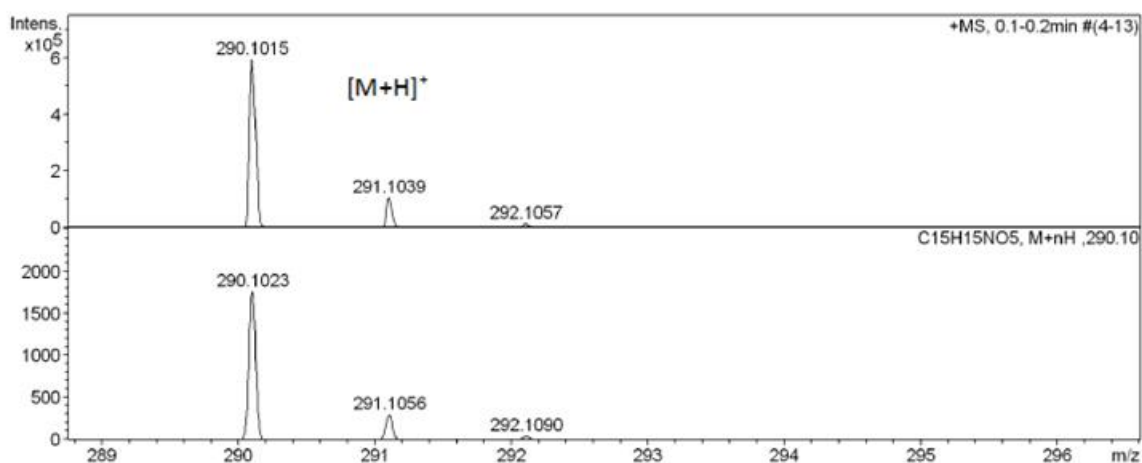
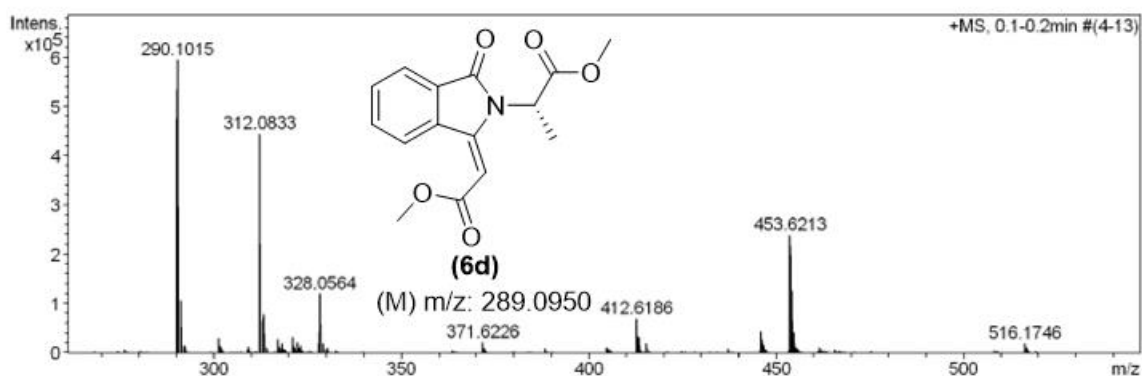
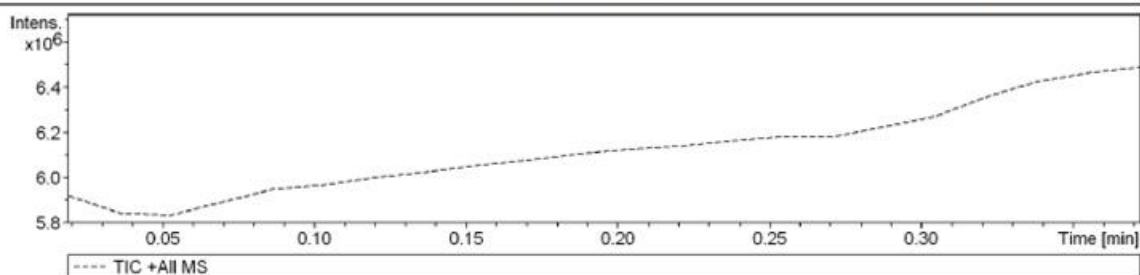
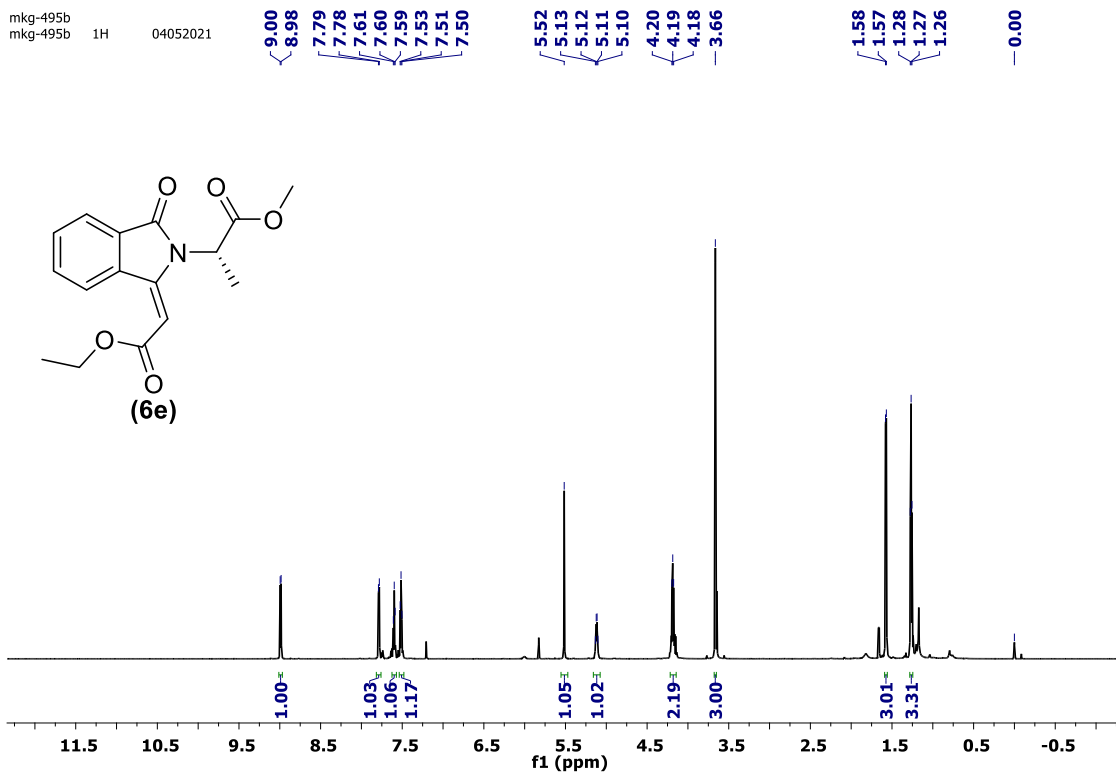


Figure. S12. ESI-HRMS spectra of indolinone **6d**

mkg-495b
mkg-495b 1H 04052021



MKG-495B
13C

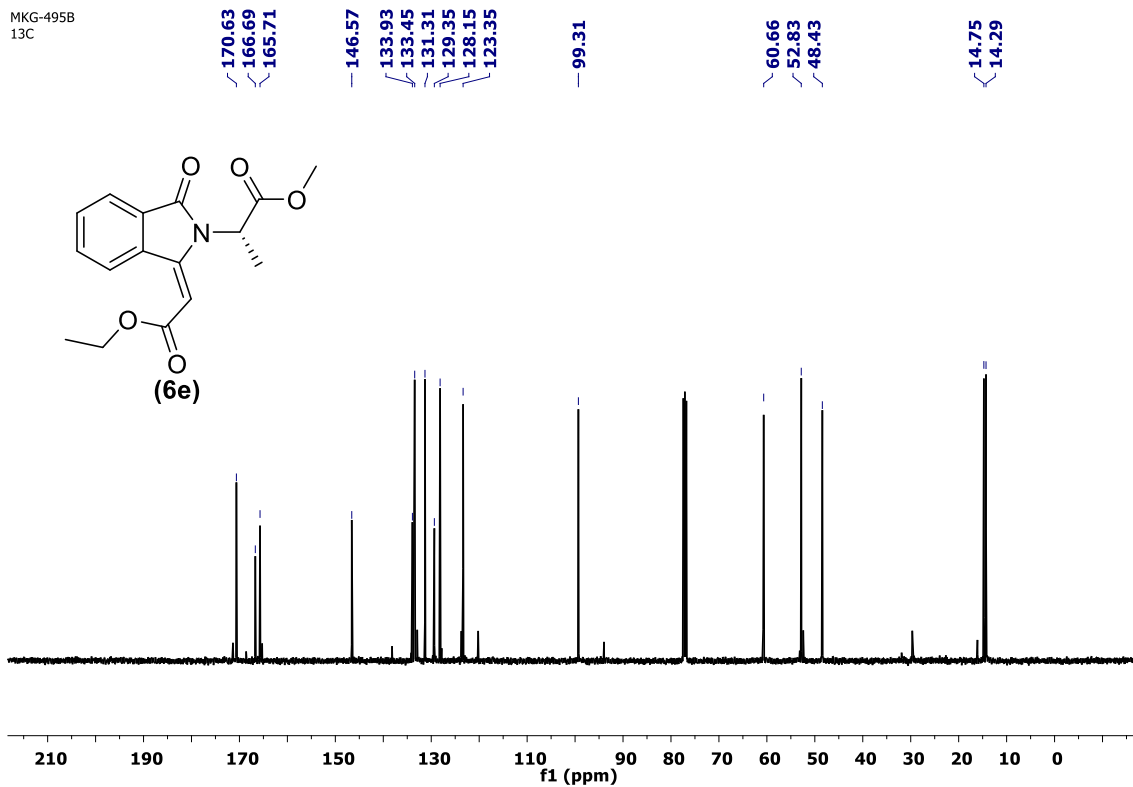


Figure. S13. ¹H, ¹³C NMR spectra of indolinone **6e**

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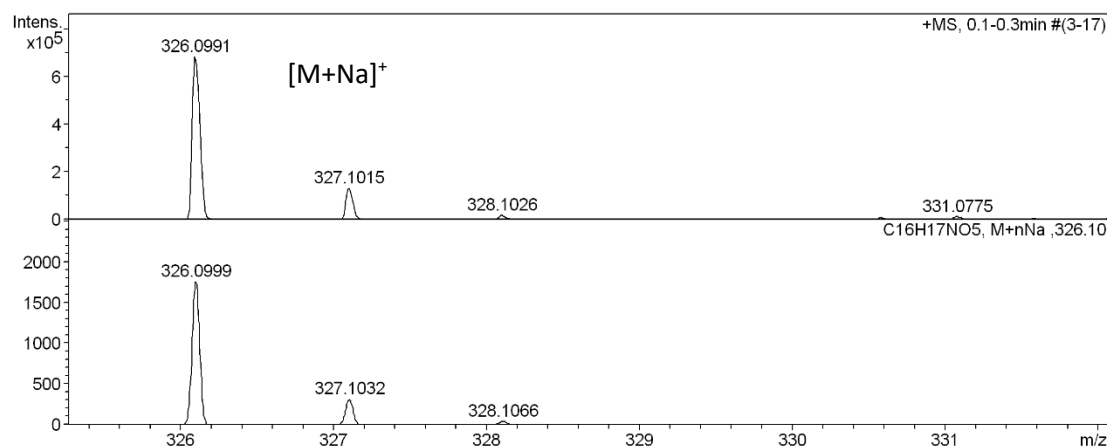
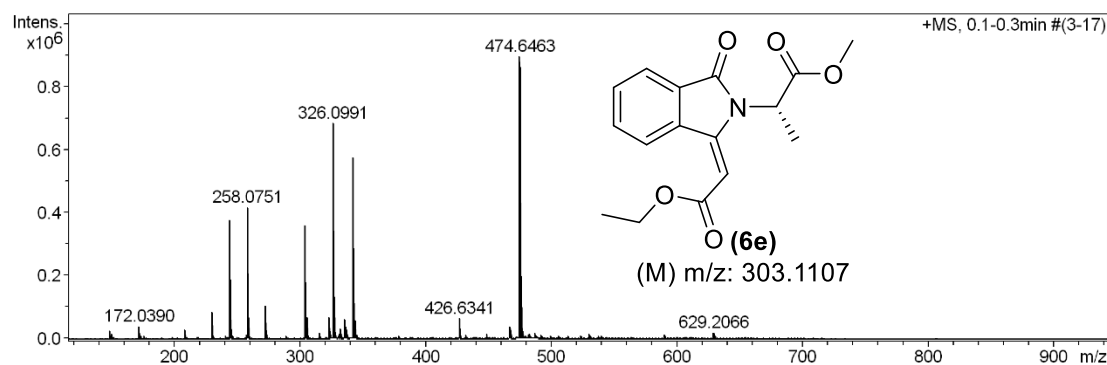
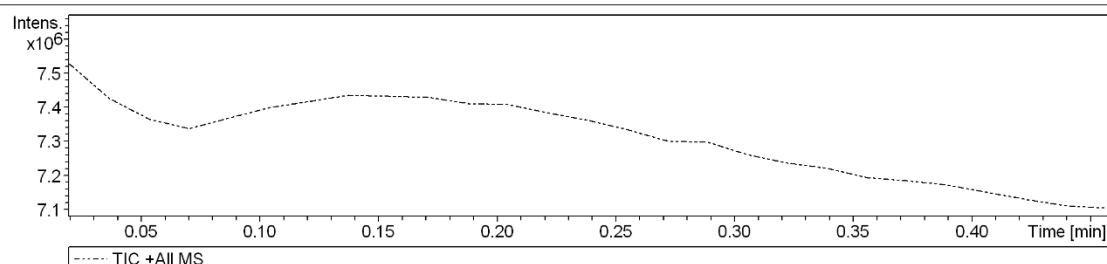
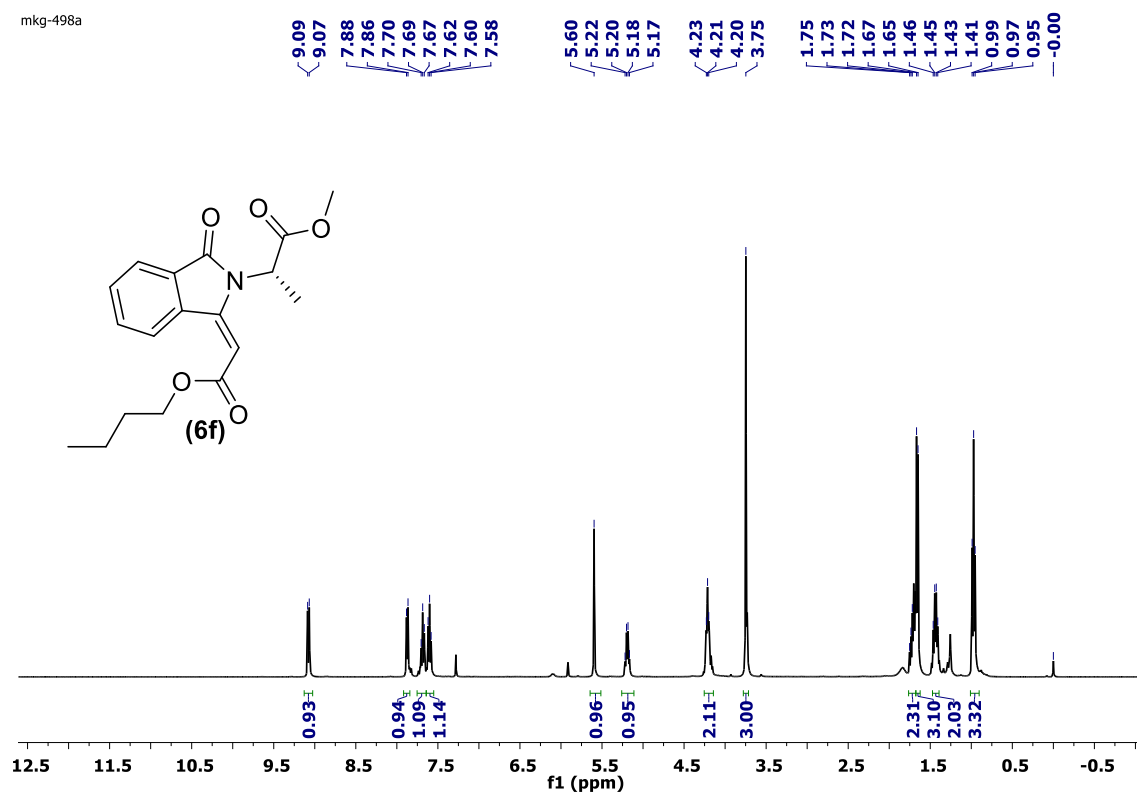


Figure. S14. ESI-HRMS spectra of indolinone **6e**

mkg-498a



mkg-498a
13c

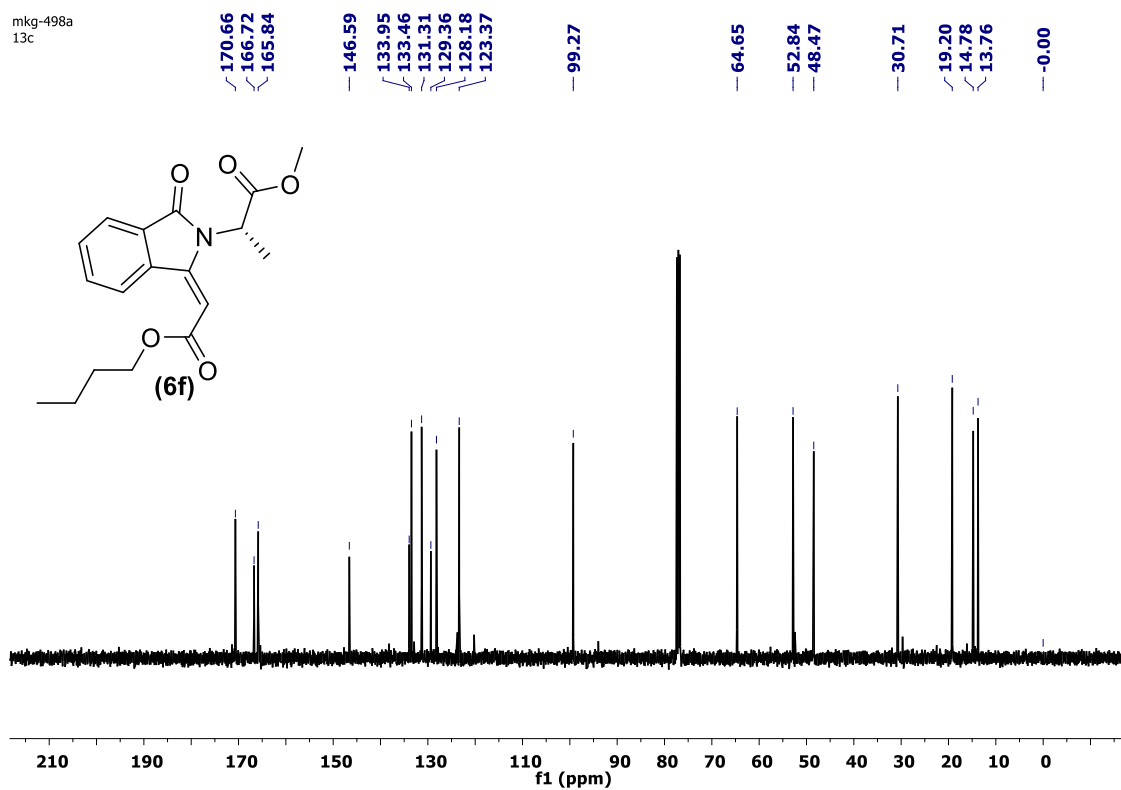


Figure. S15. ¹H, ¹³C NMR spectra of indolinone 6f

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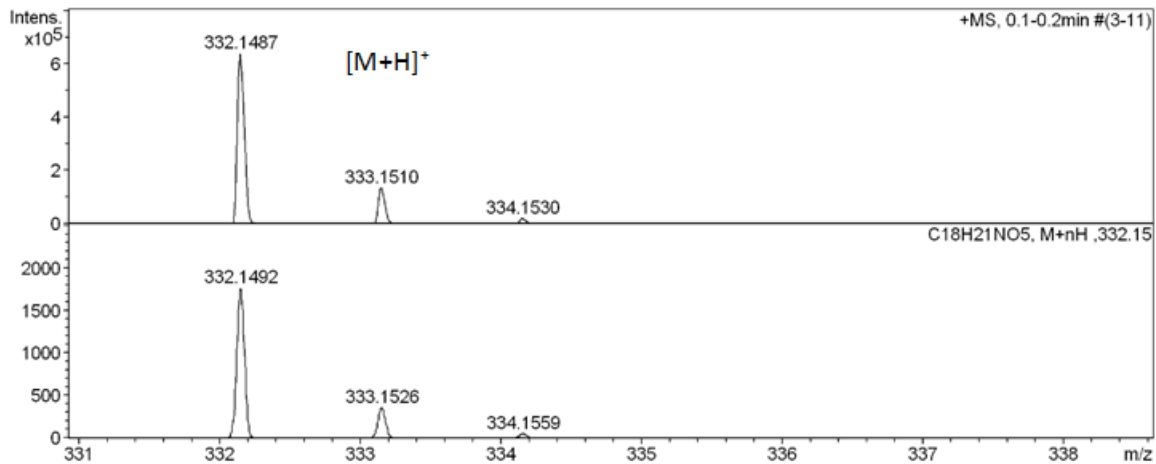
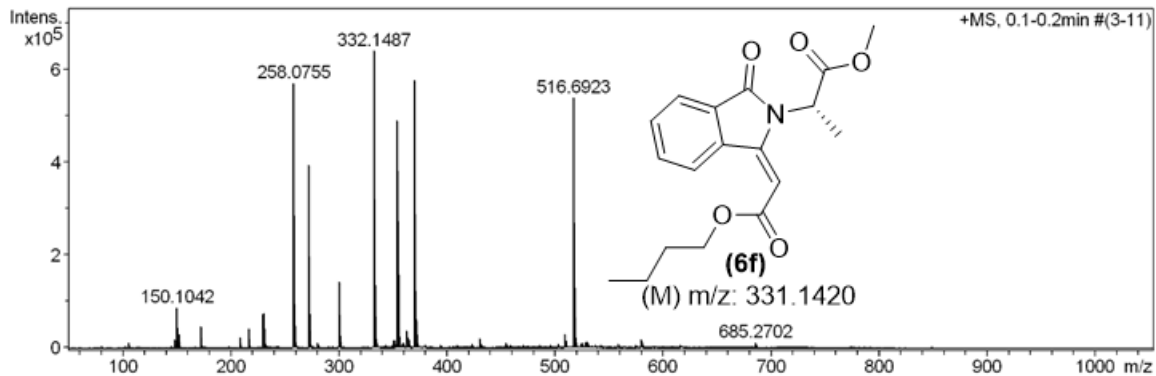
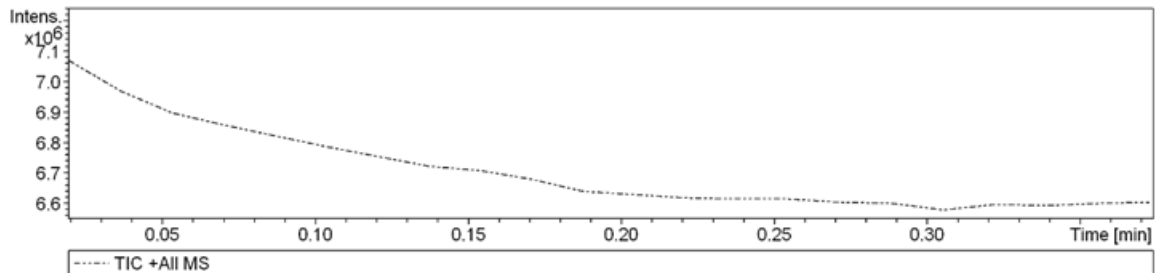


Figure. S16. ESI-HRMS spectra of indolinone **6f**

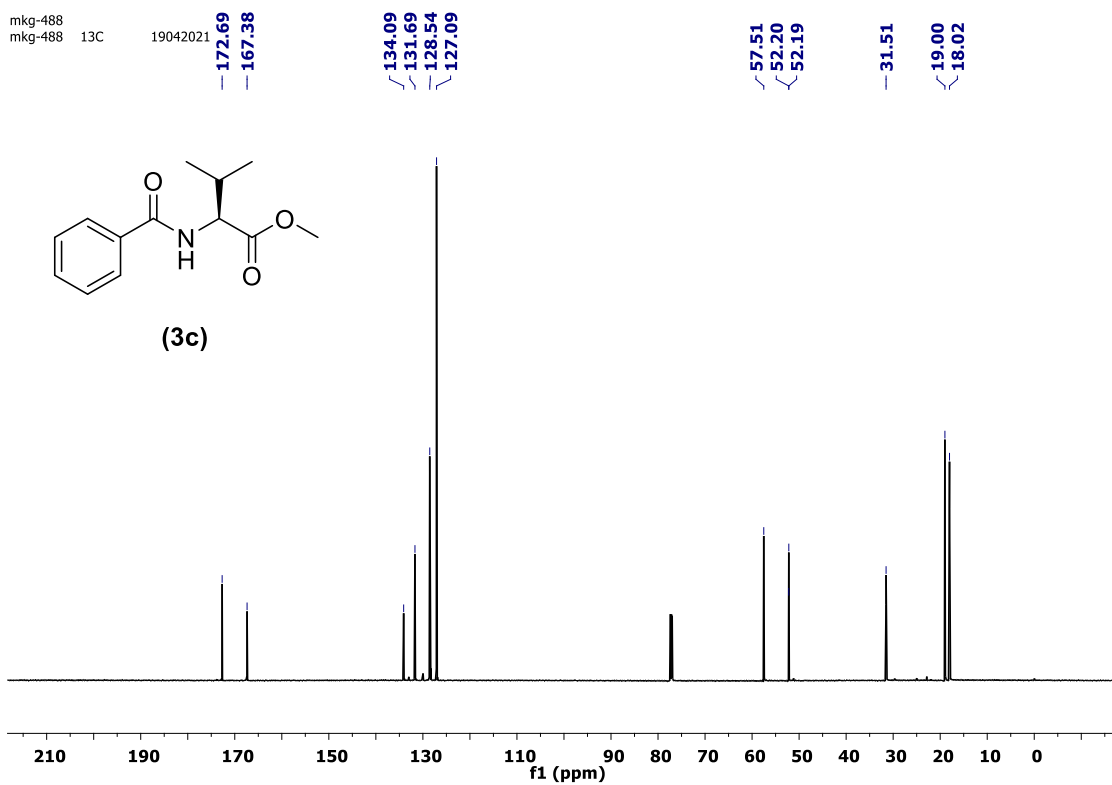
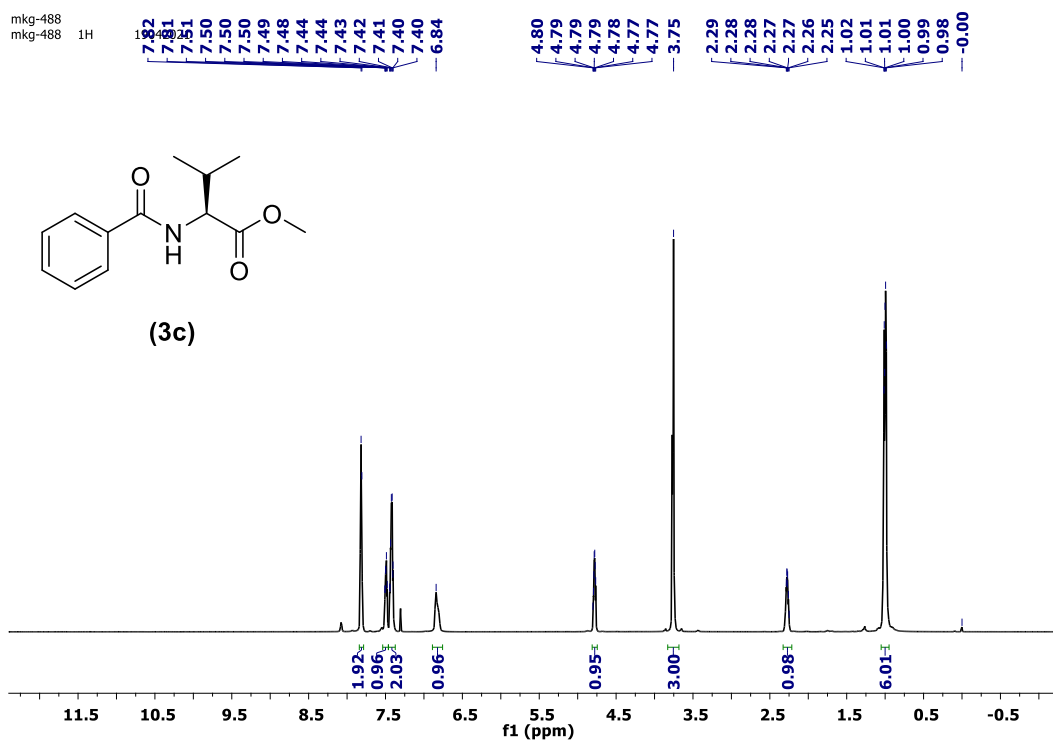


Figure. S17. ¹H, ¹³C NMR spectra of benzamide 3c

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Operator Amit S.Sahu
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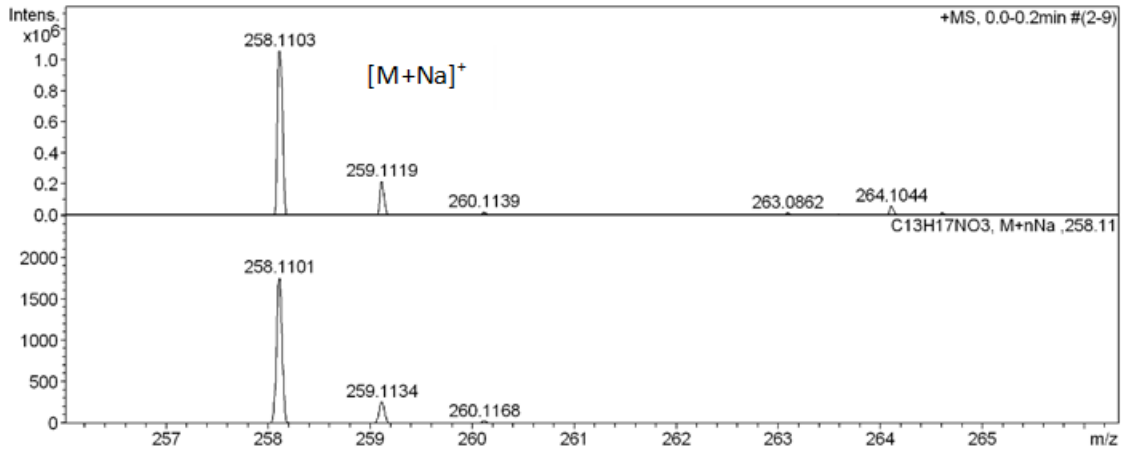
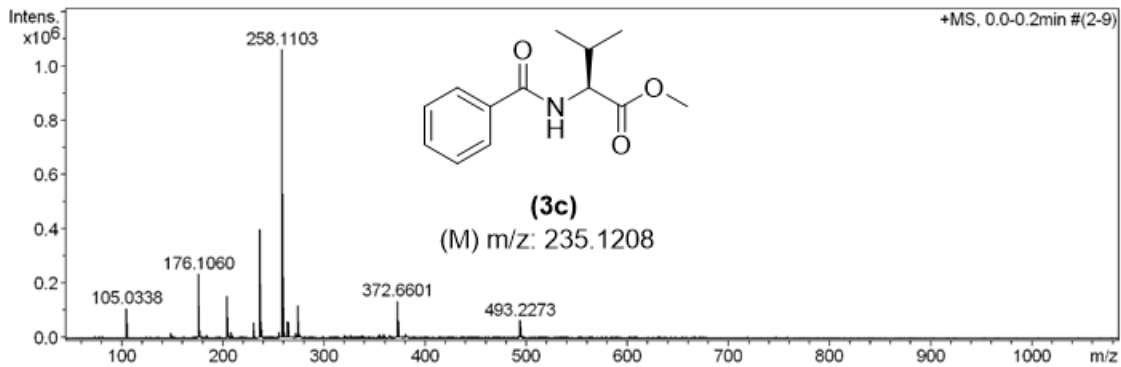
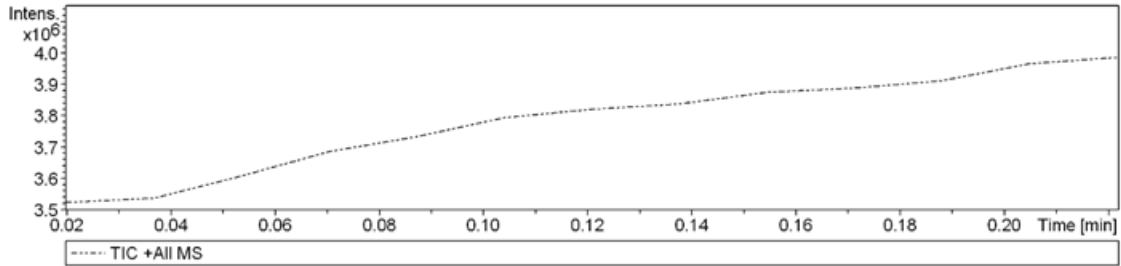


Figure. S18. ESI-HRMS spectra of benzamide 3c

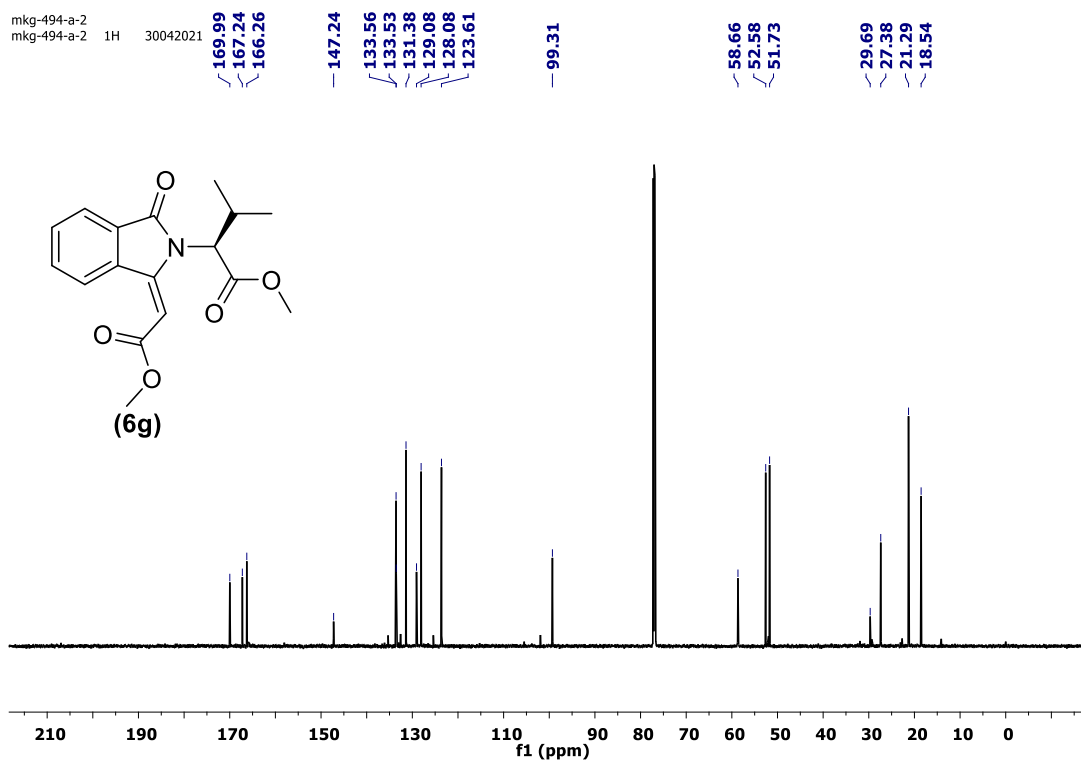
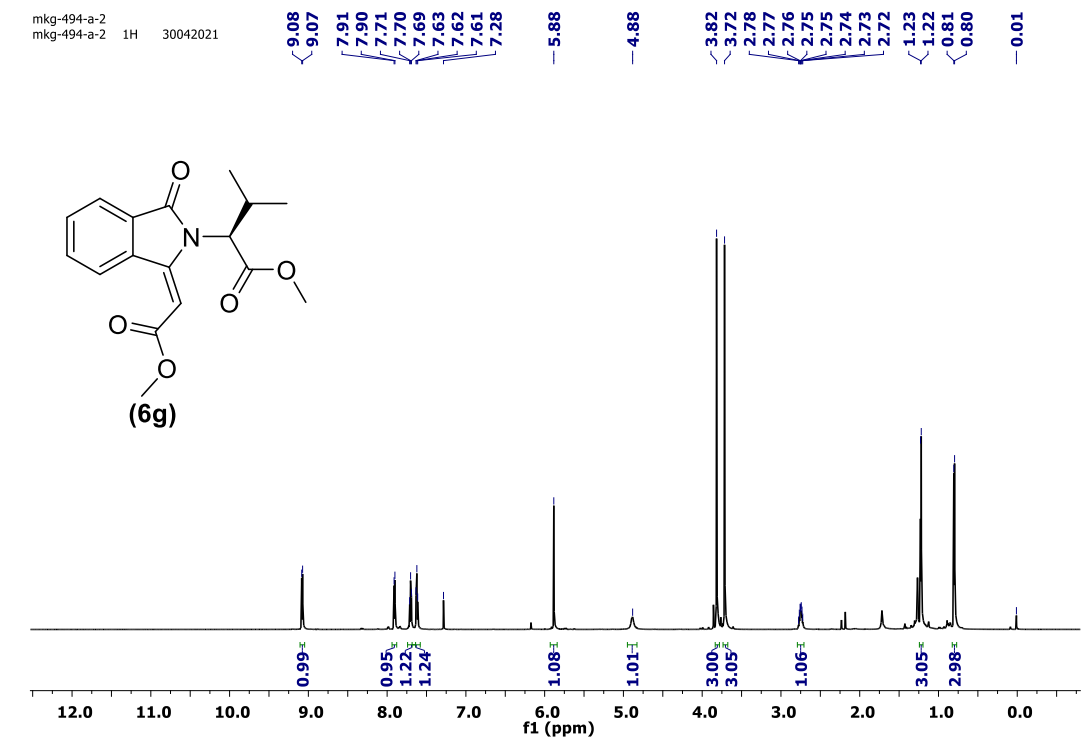


Figure. S19. ^1H , ^{13}C NMR spectra of indolinone **6g**

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Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

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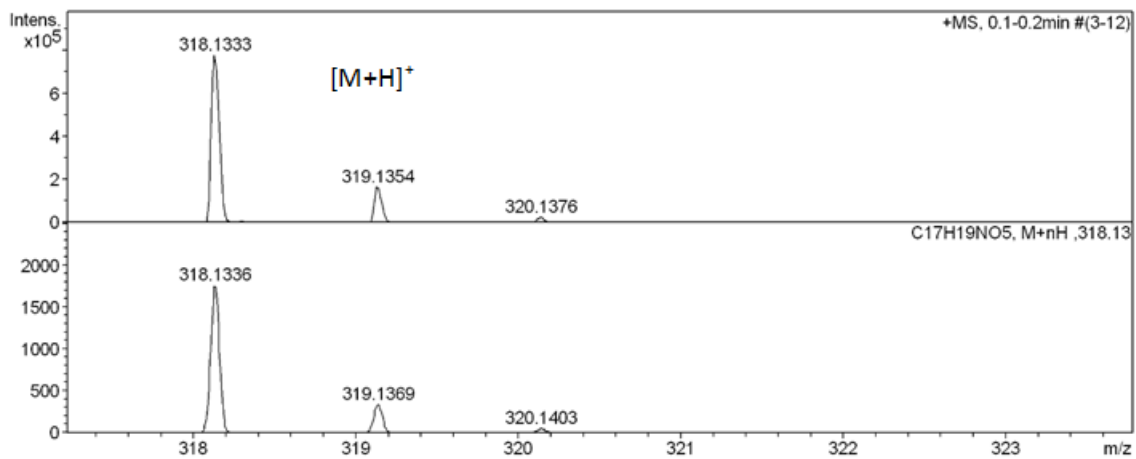
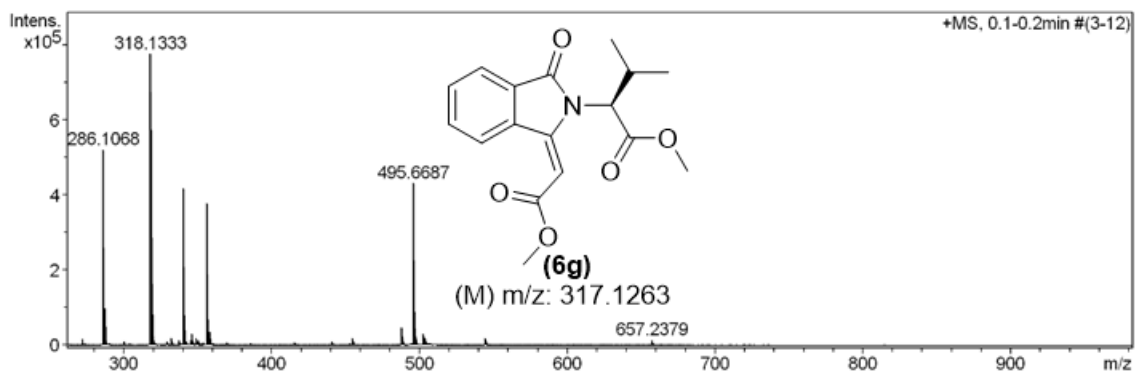
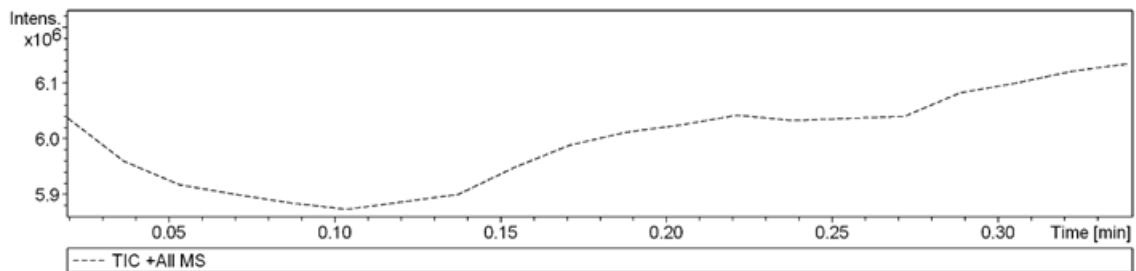
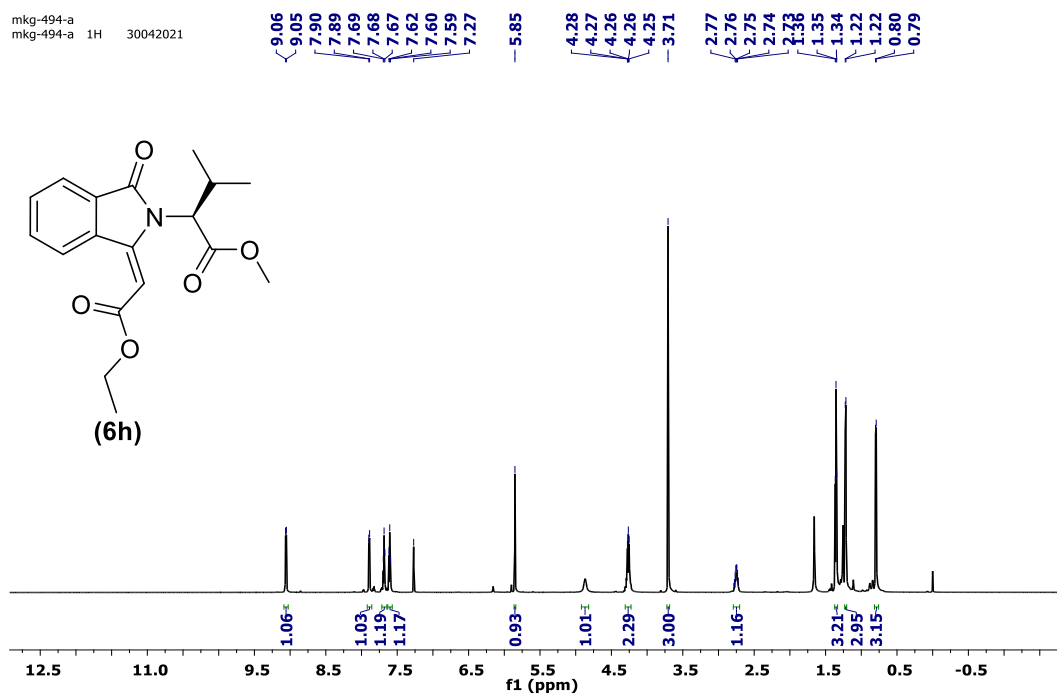


Figure. S20. ESI-HRMS spectra of indolinone **6g**

mkg-494-a
mkg-494-a 1H 30042021



mkg-494-a
mkg-494-a 1H 30042021

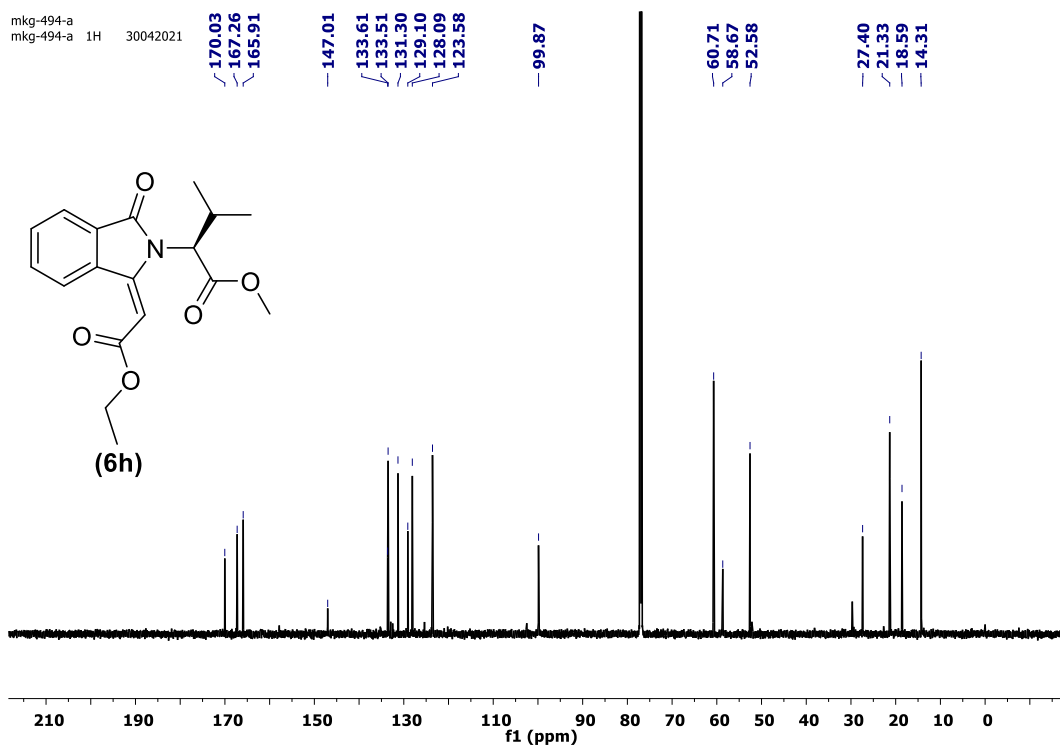


Figure. S21. ^1H , ^{13}C NMR spectra of indolinone **6h**

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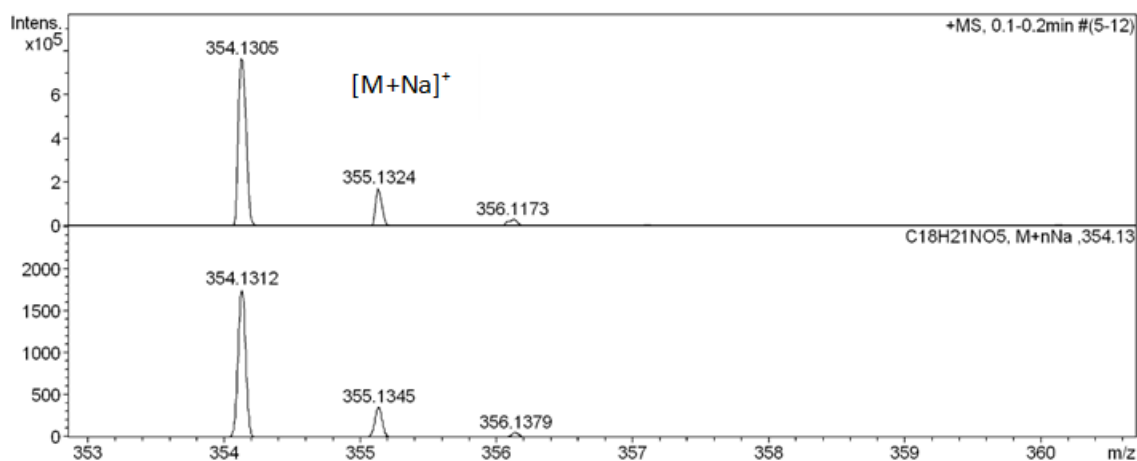
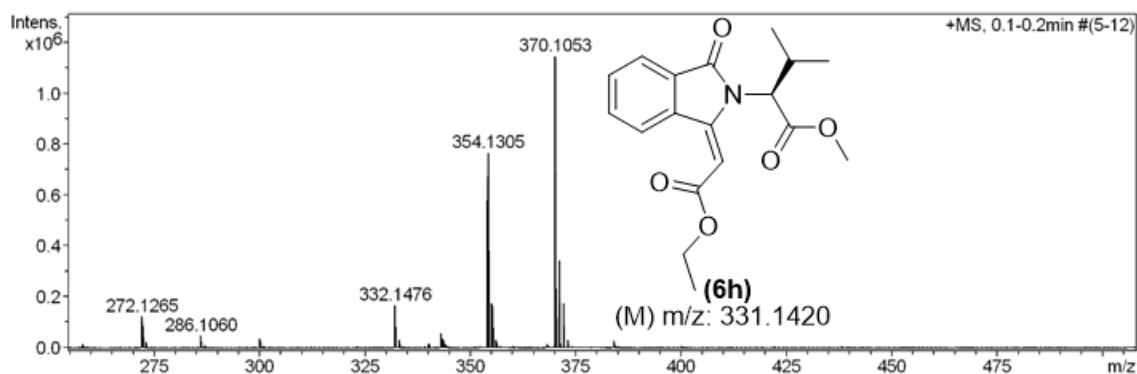
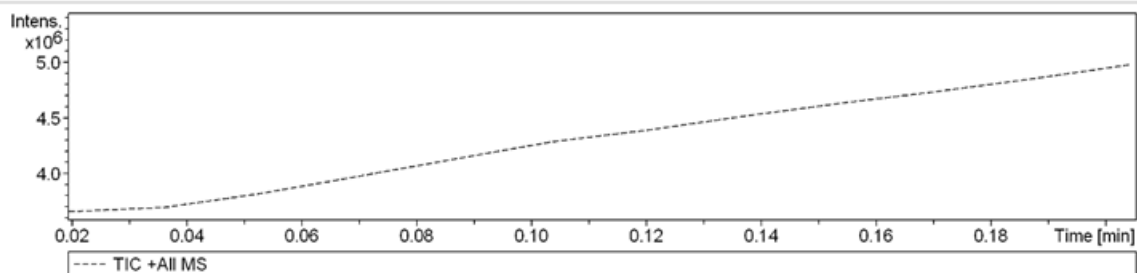
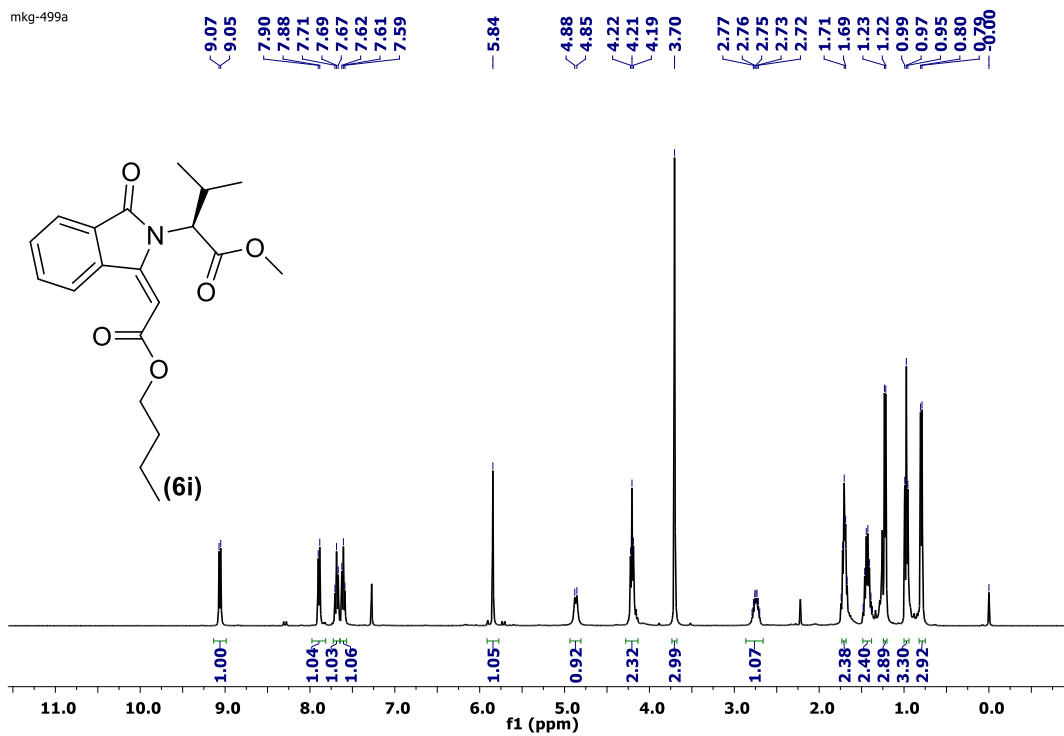


Figure. S22. ESI-HRMS spectra of indolinone **6h**

mkg-499a



mkg-499a
13c

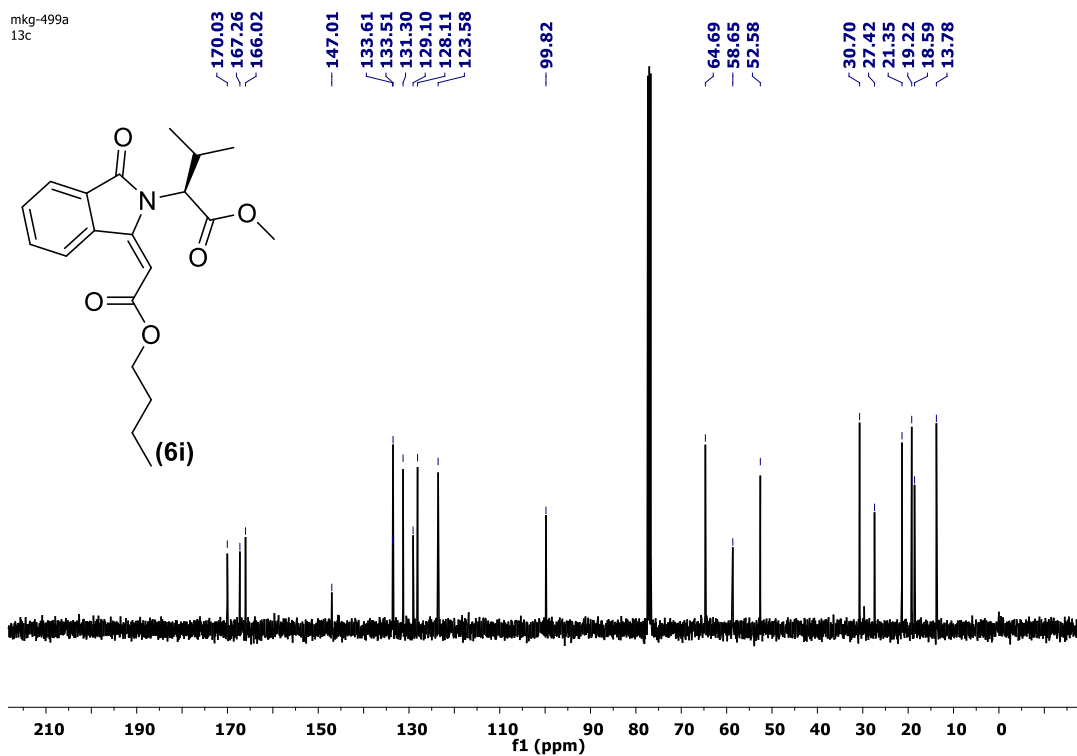


Figure. S23. ^1H , ^{13}C NMR spectra of indolinone **6i**

Display Report

Analysis Info

Analysis Name D:\Data\JUN-2021\NKS\09062021_NKS_MKG_499-A.d
Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/11/2021 8:27:31 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

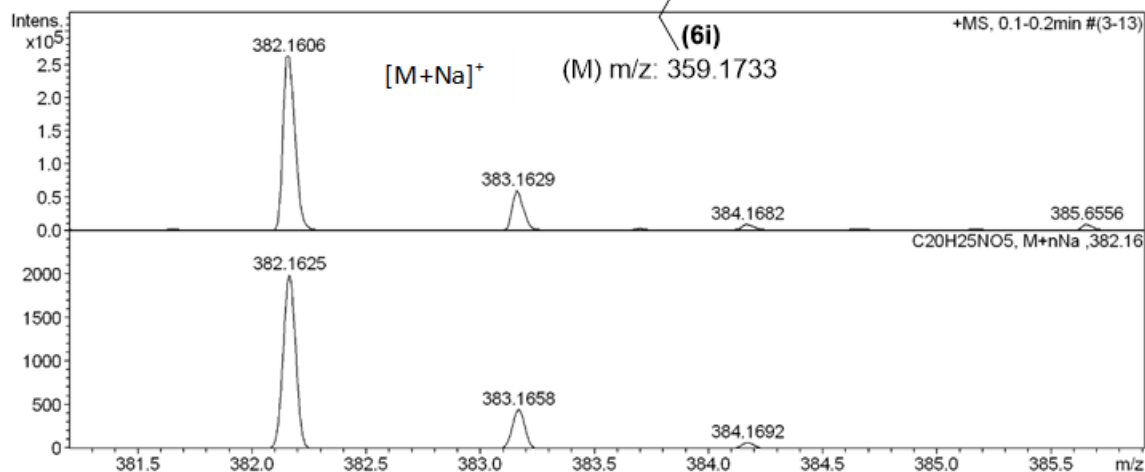
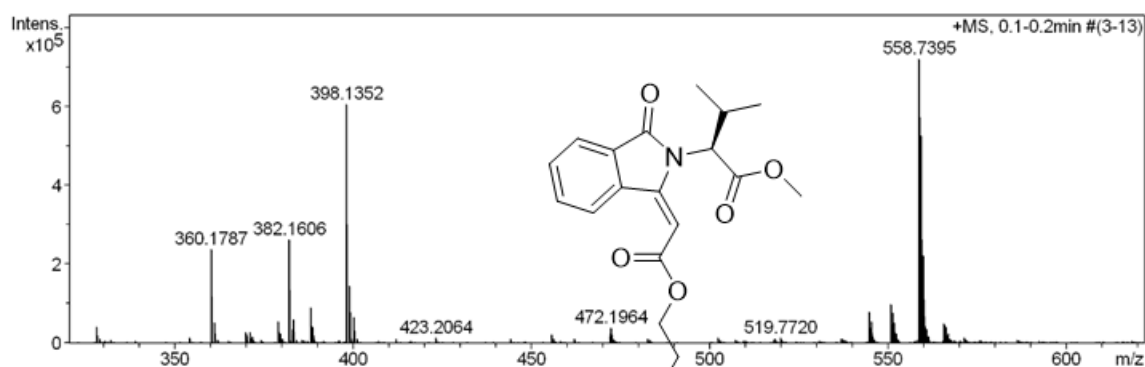
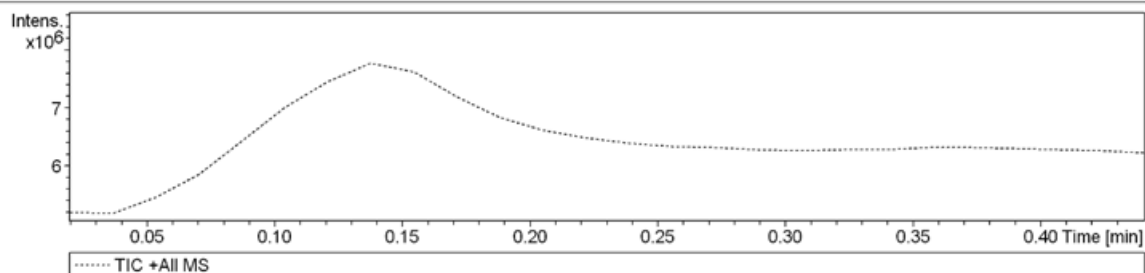


Figure. S24. ESI-HRMS spectra of indolinone **6i**

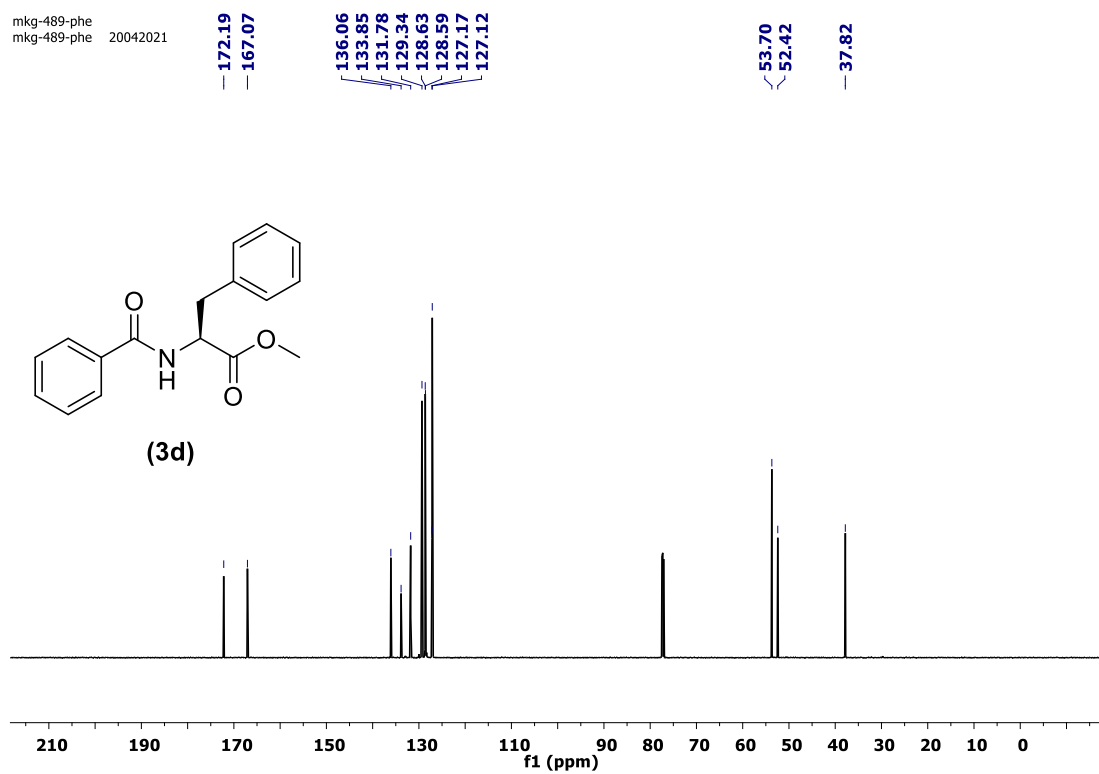
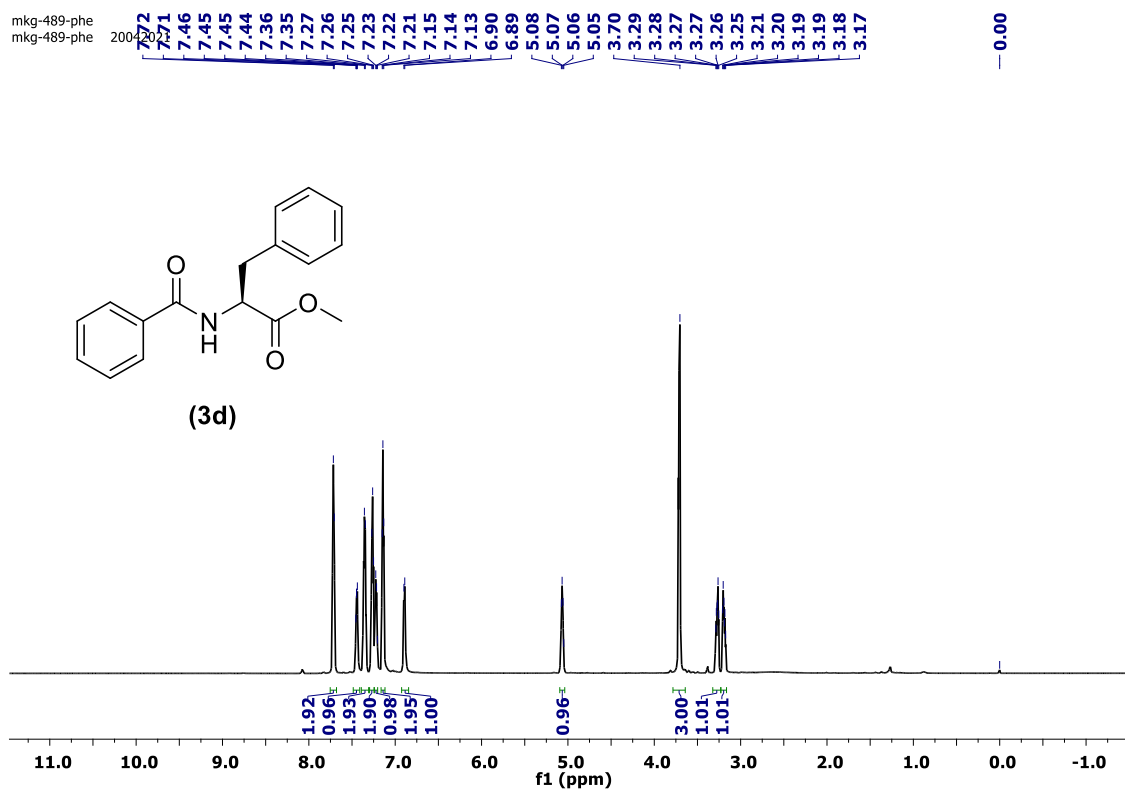


Figure. S25. ^1H , ^{13}C NMR spectra of benzamide **3d**

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:14:41 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

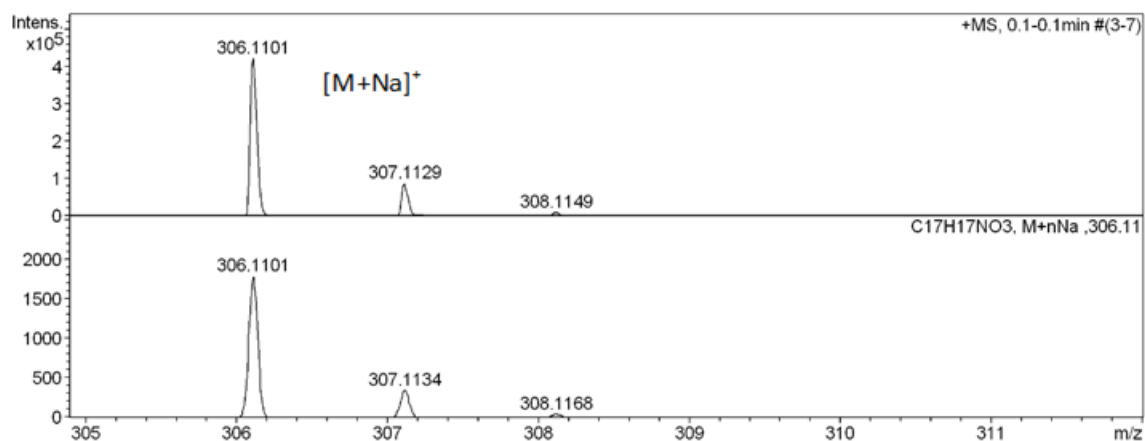
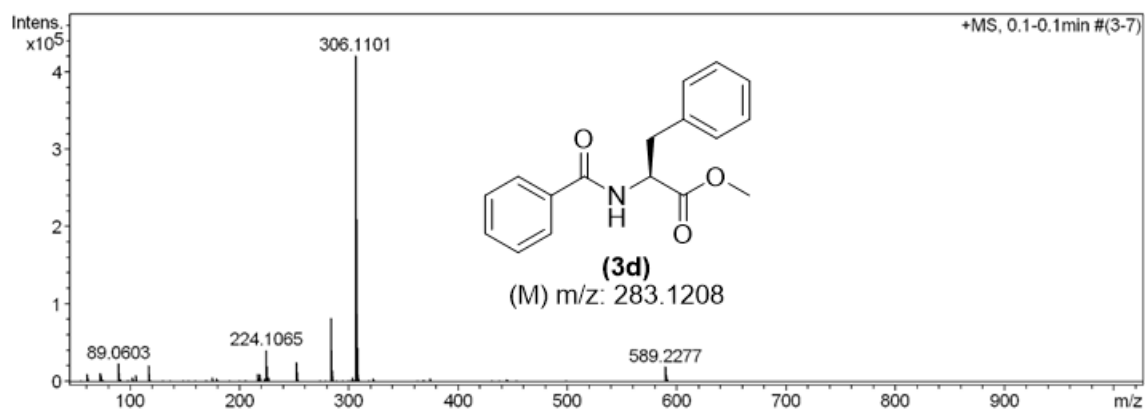
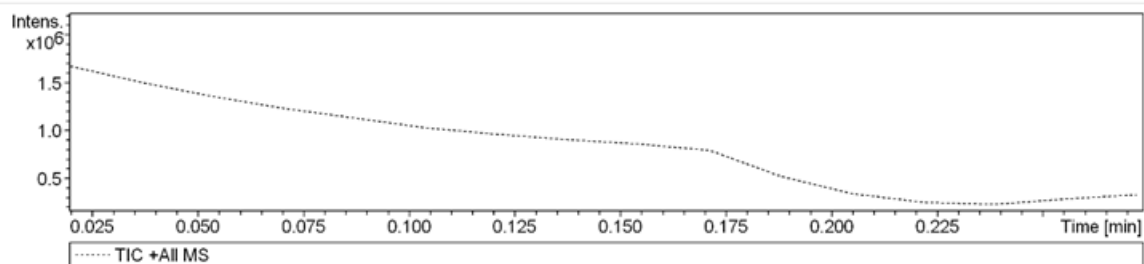
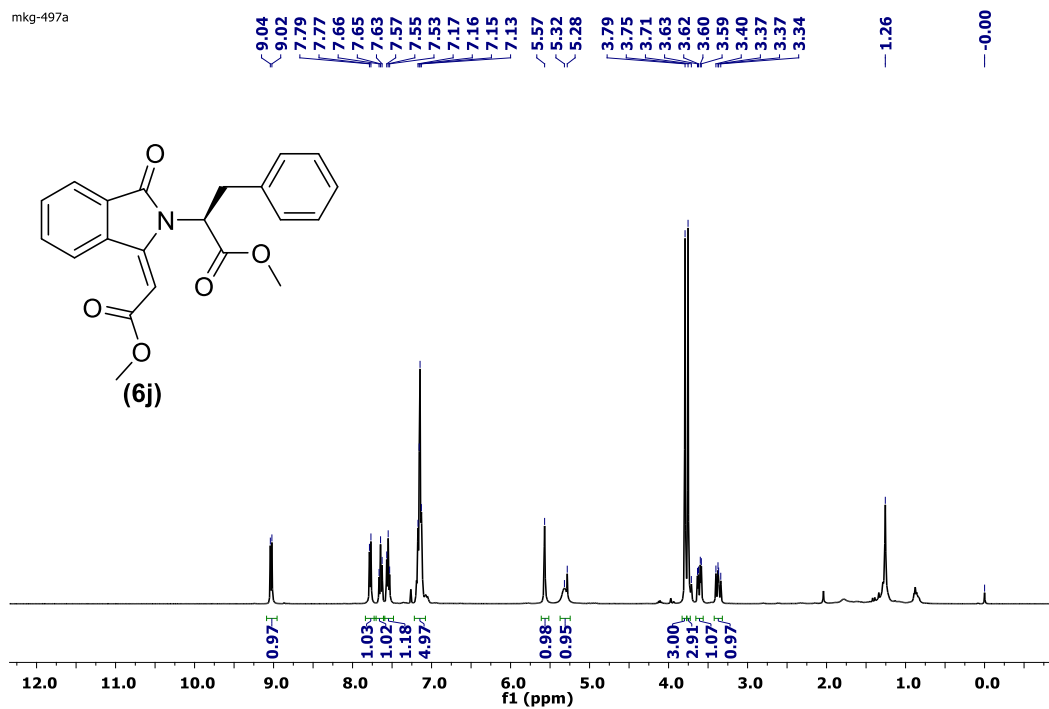


Figure. S26. ESI-HRMS spectra of benzamide **3d**

mkg-497a



mkg-497a
13C

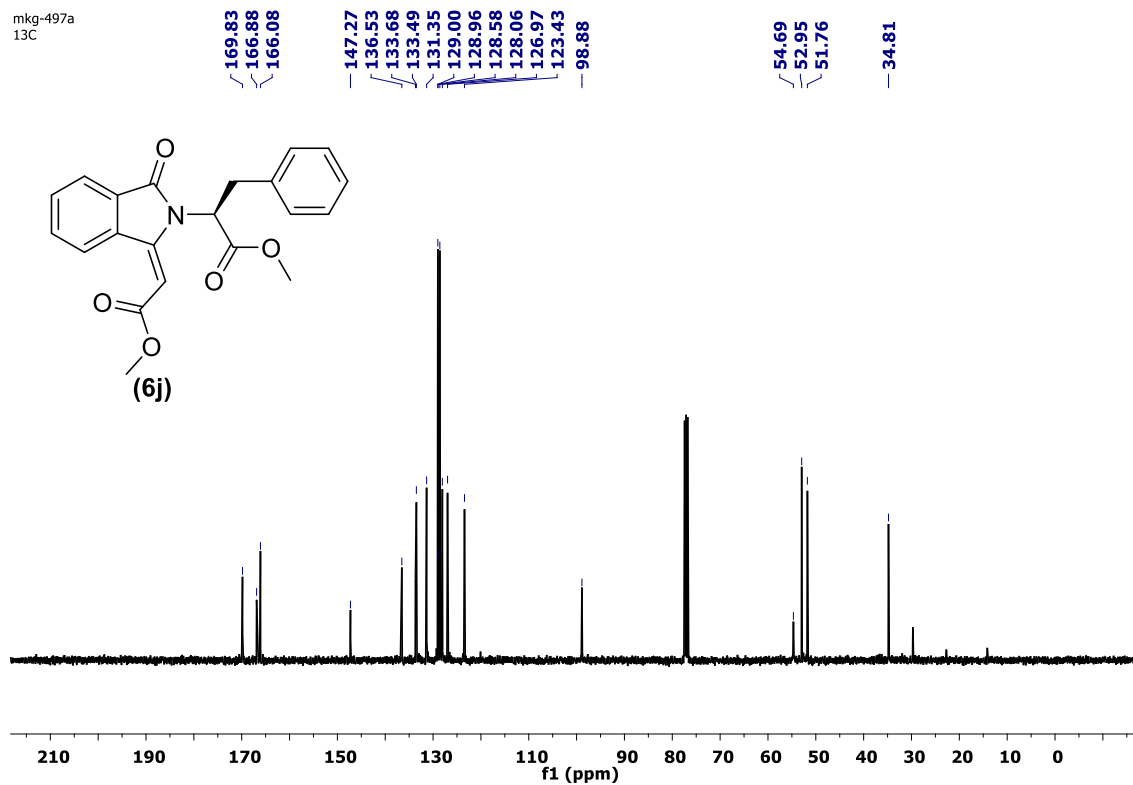


Figure. S27. ¹H, ¹³C NMR spectra of indolinone **6j**

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:17:27 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

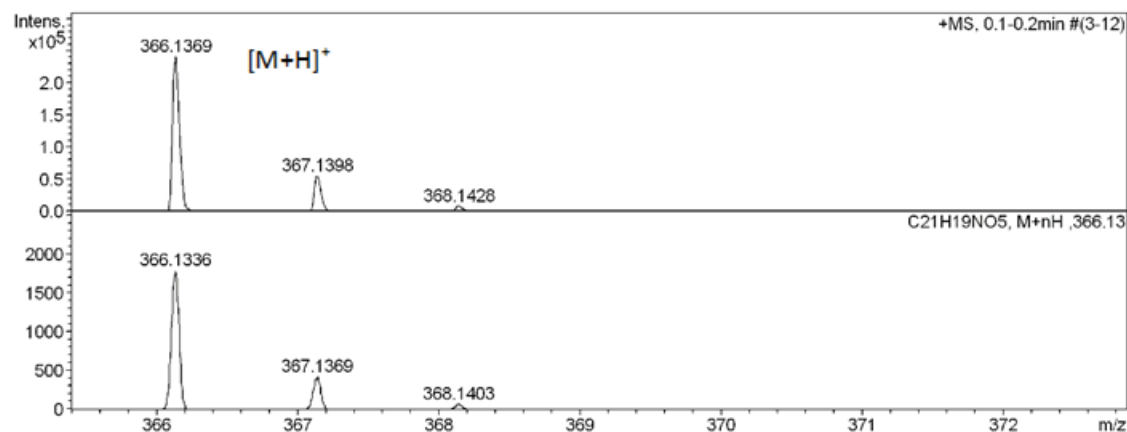
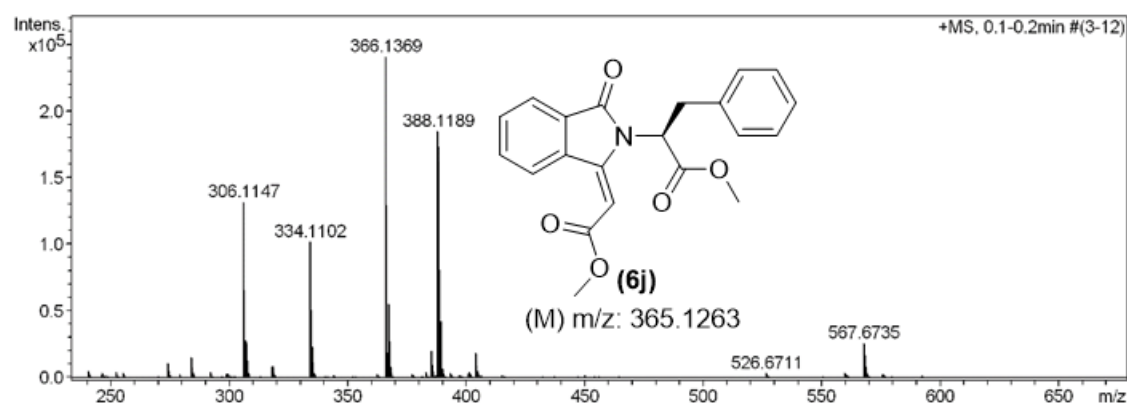
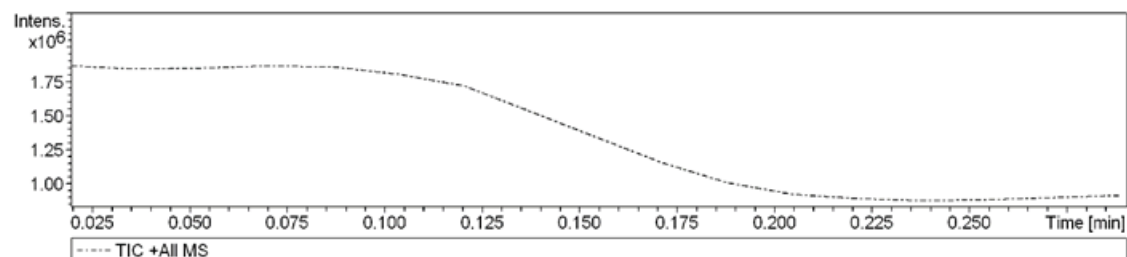
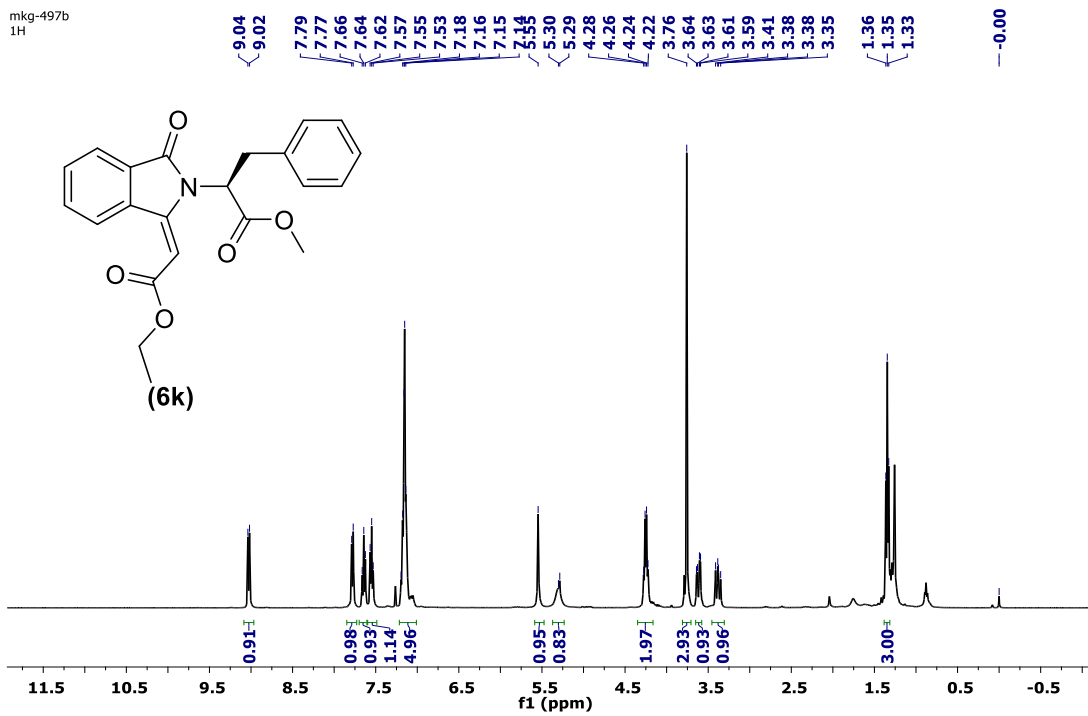


Figure. S28. ESI-HRMS spectra of indolinone 6j

mkg-497b
1H



mkg-497b
13C

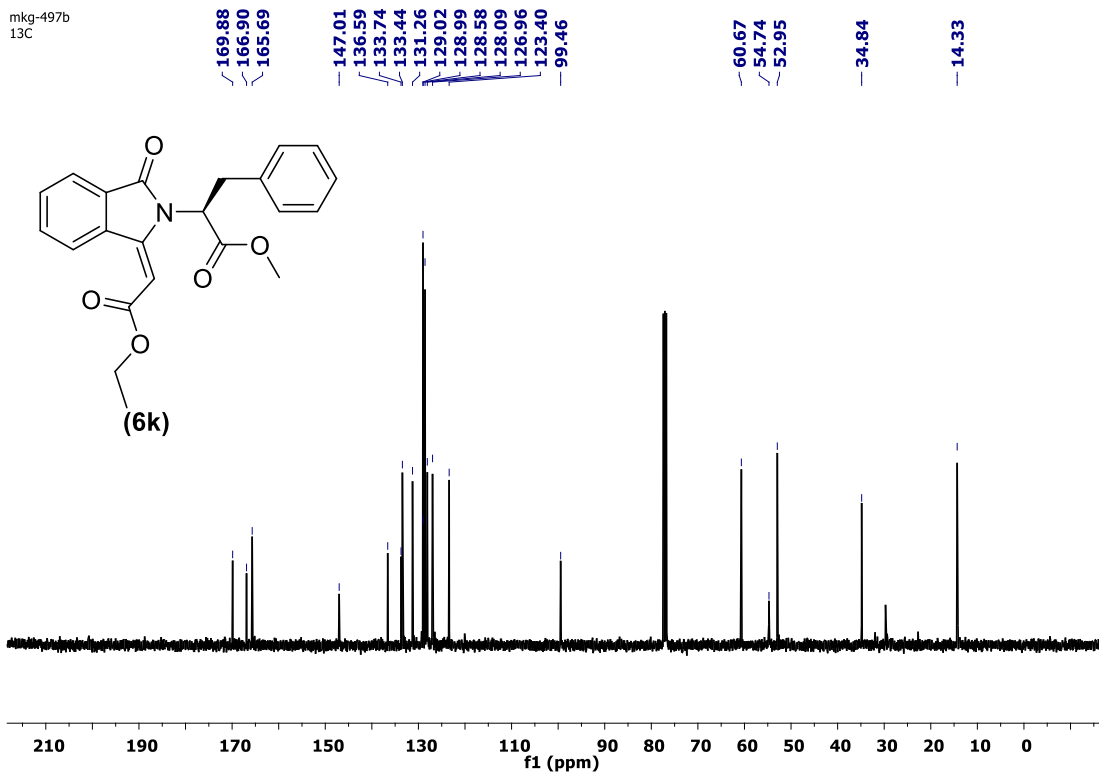


Figure. S29. ¹H, ¹³C NMR spectra of indolinone **6k**

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:20:21 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

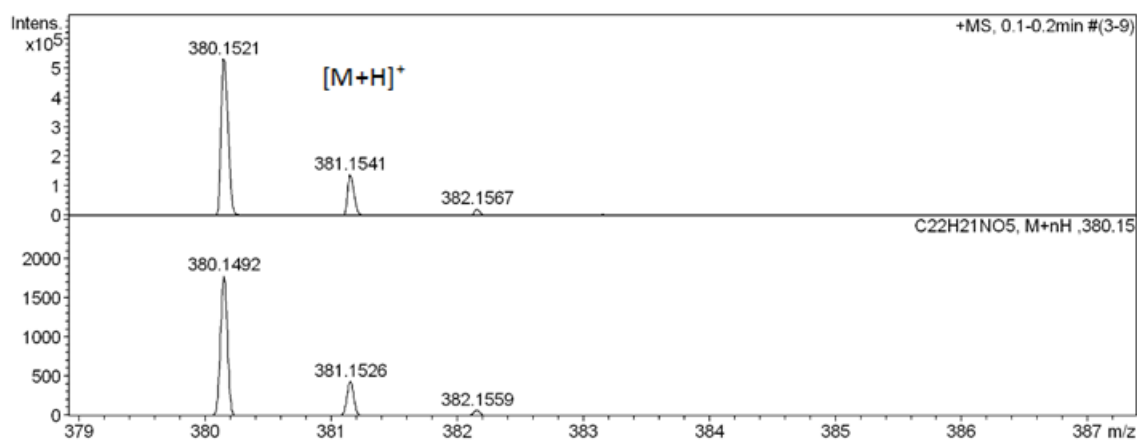
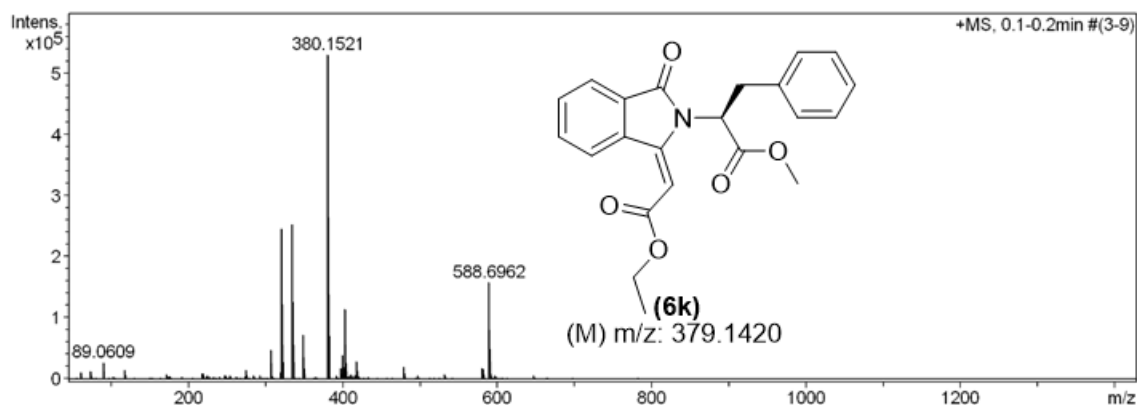
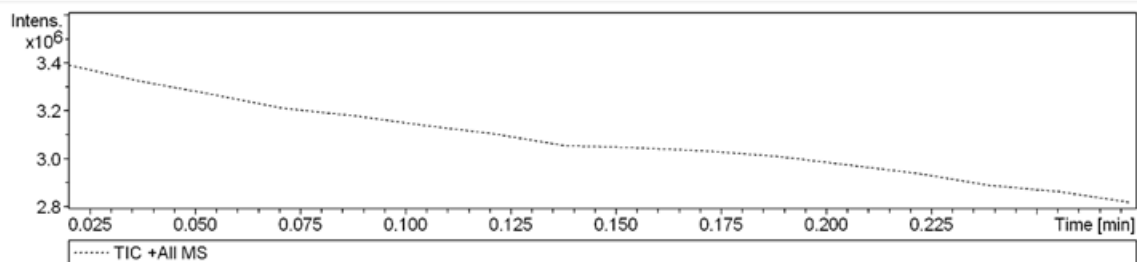
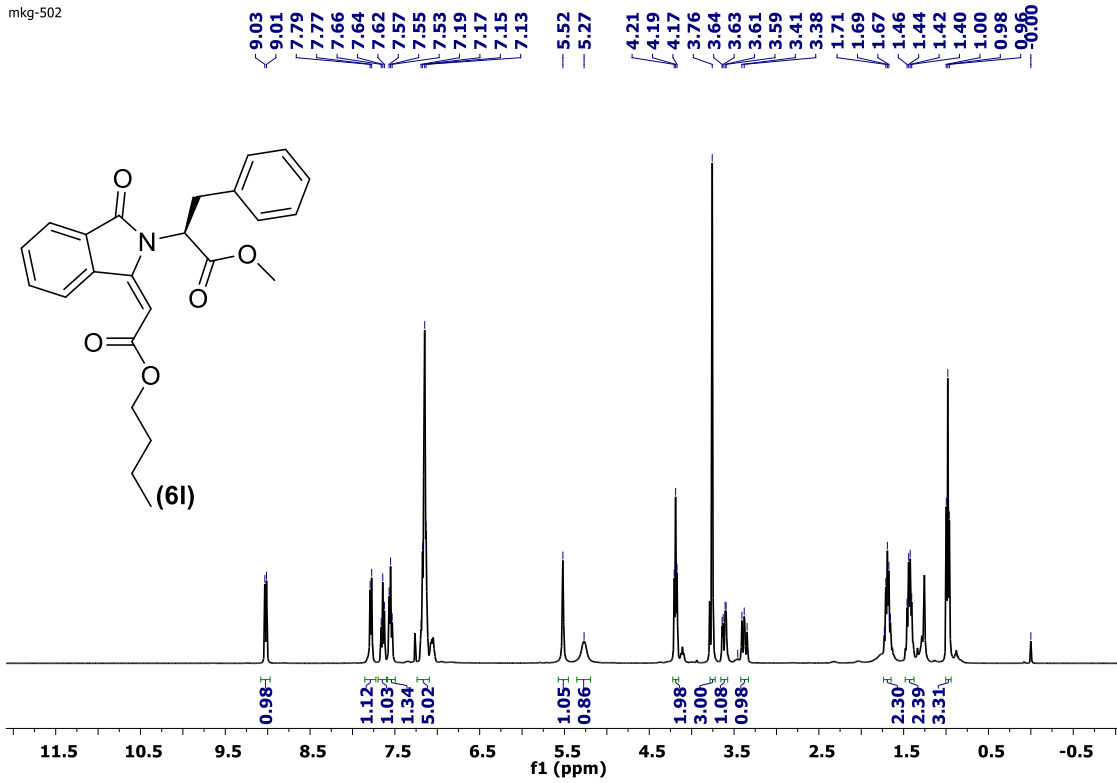


Figure. S30. ESI-HRMS spectra of indolinone **6k**

mkg-502



mkg-502
¹³C

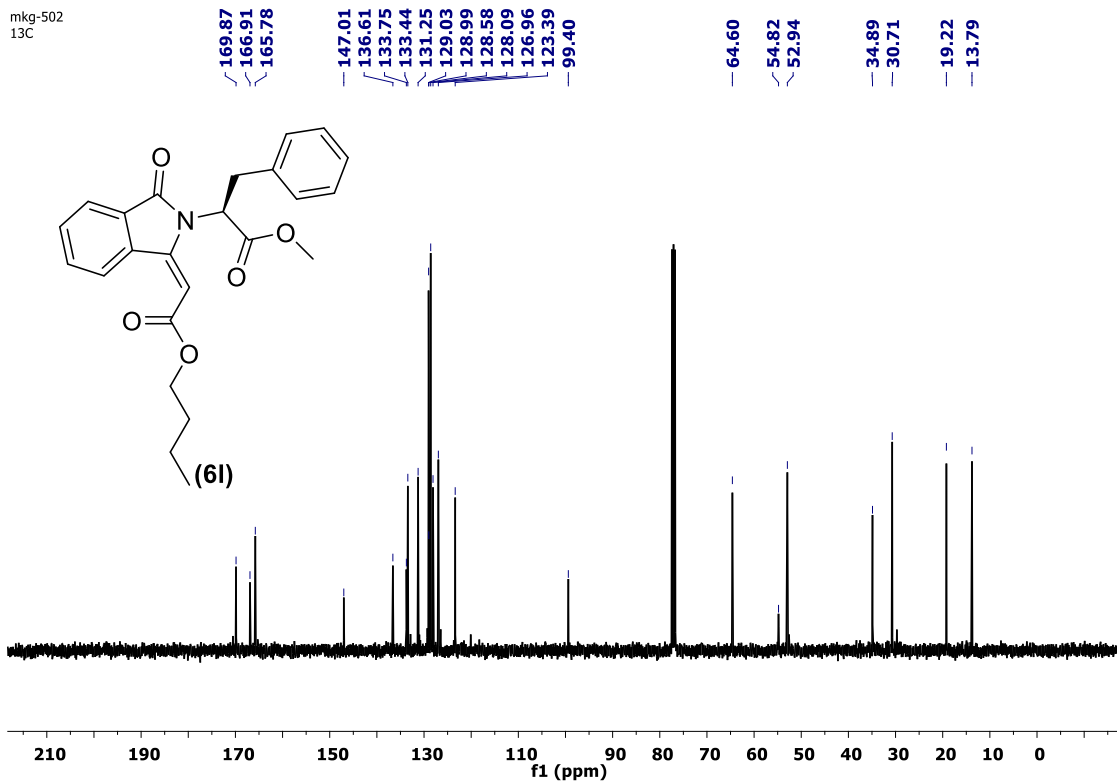


Figure. S31. ¹H, ¹³C NMR spectra of indolinone **6l**

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:22:56 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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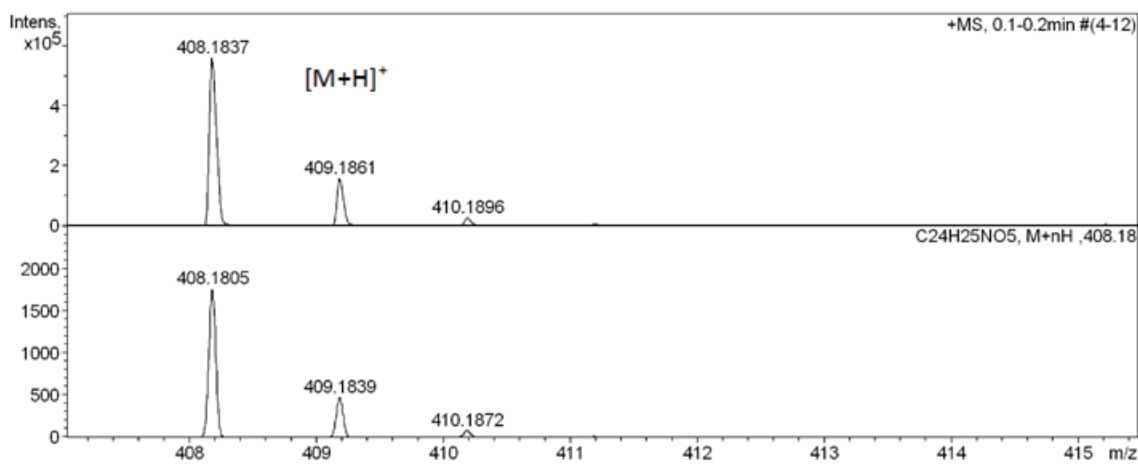
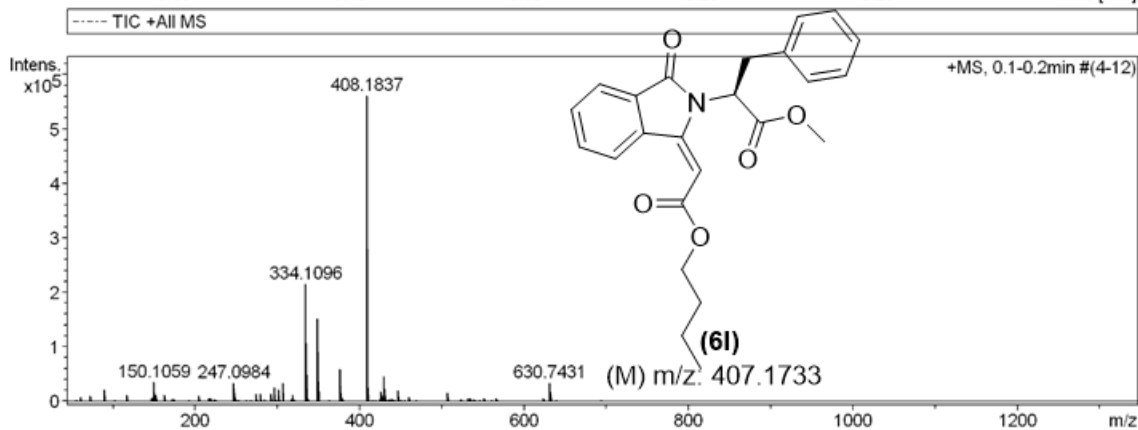
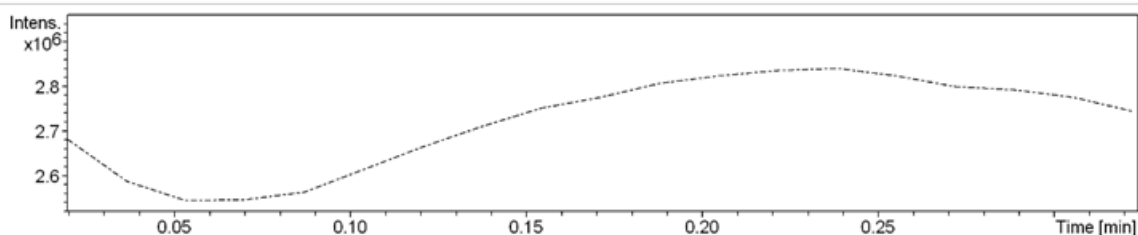


Figure. S32. ESI-HRMS spectra of indolinone **61**

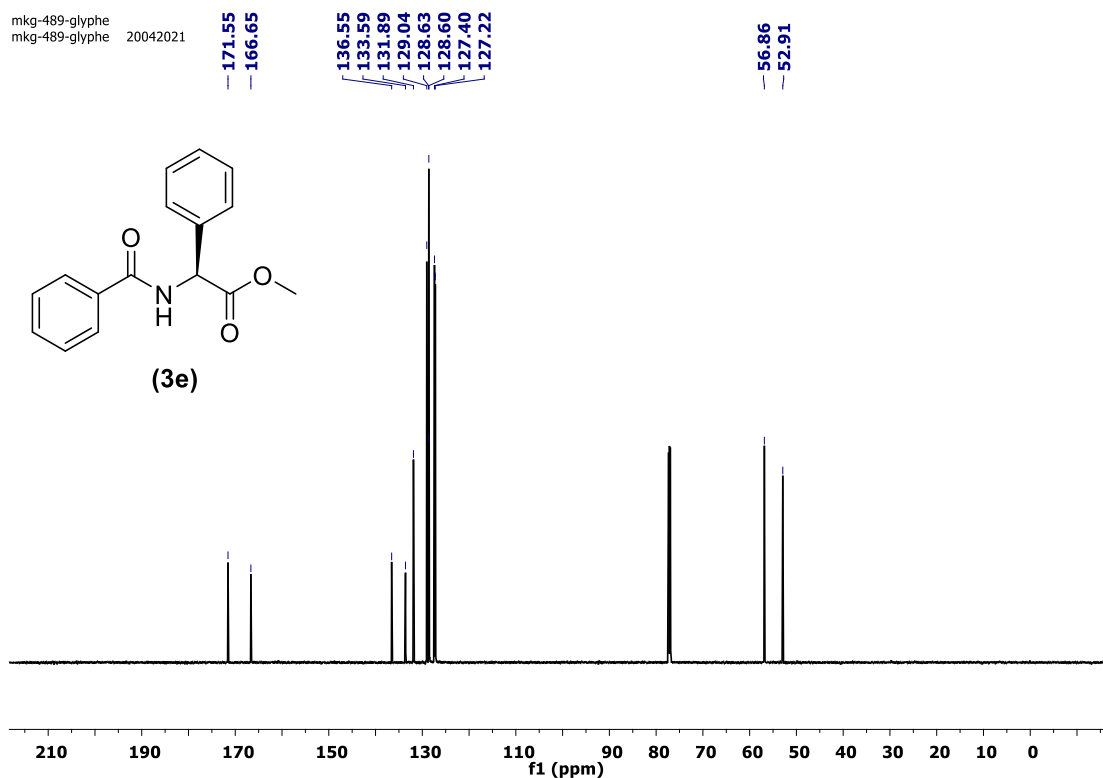
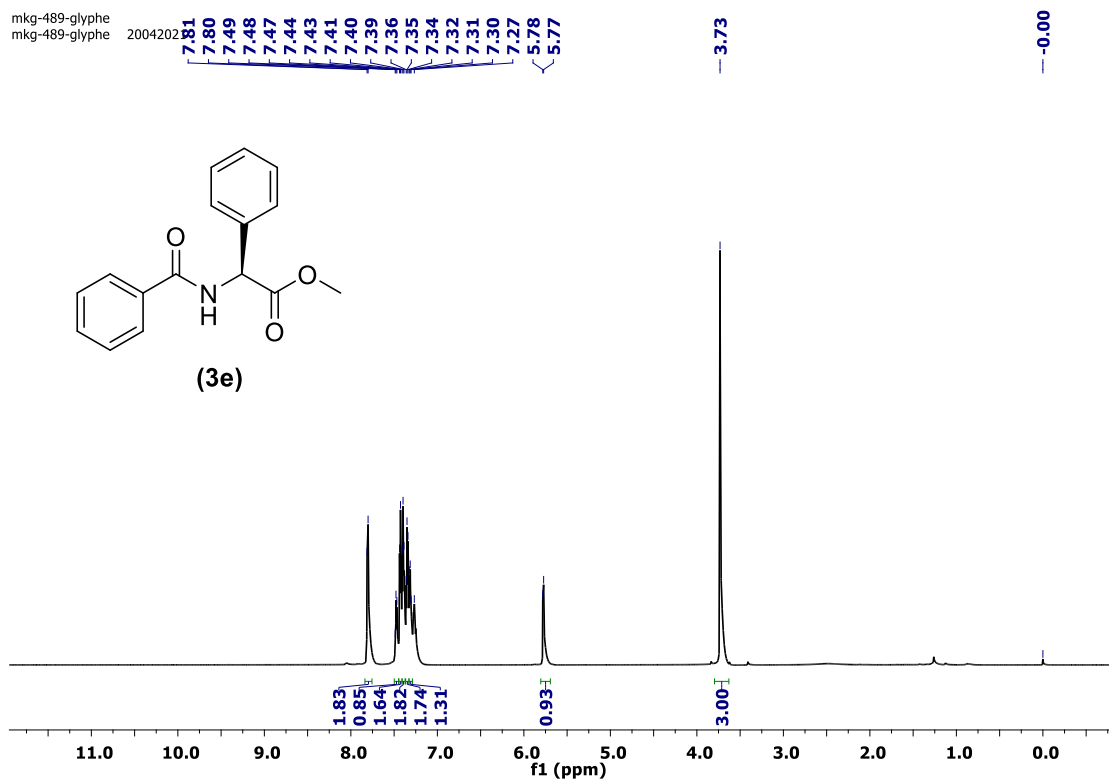


Figure. S33. ^1H , ^{13}C NMR spectra of benzamide **3e**

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:25:54 PM
Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

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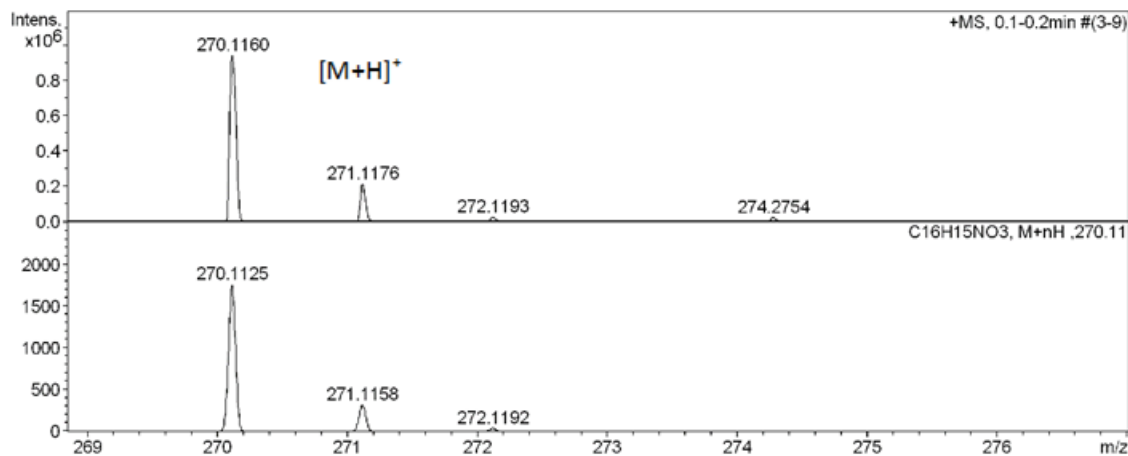
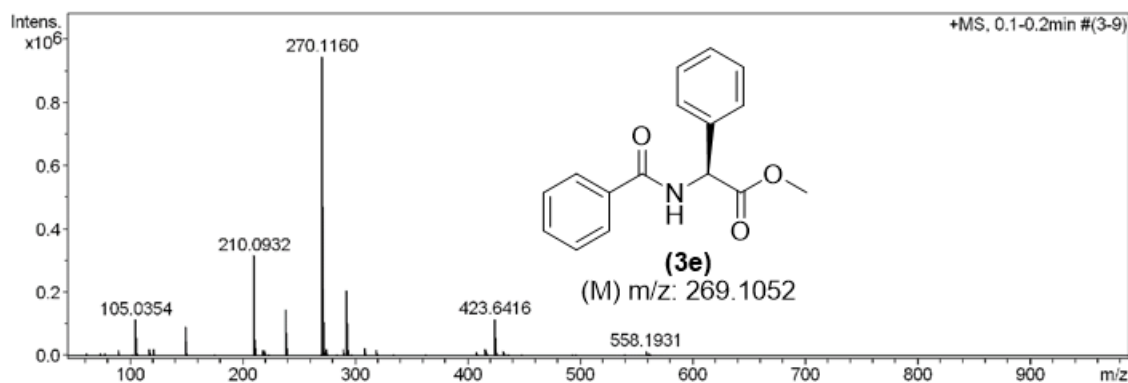
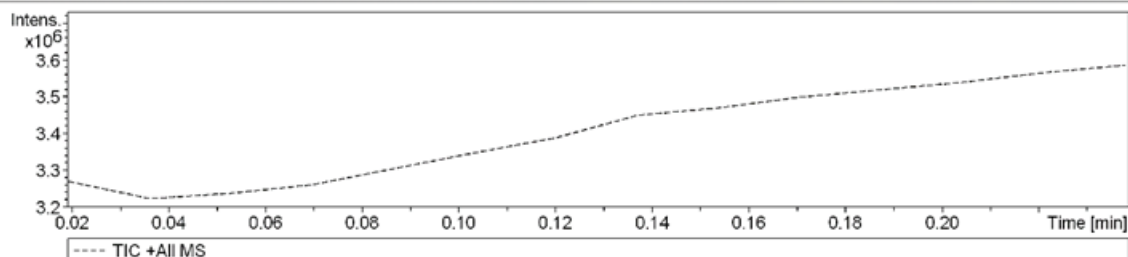
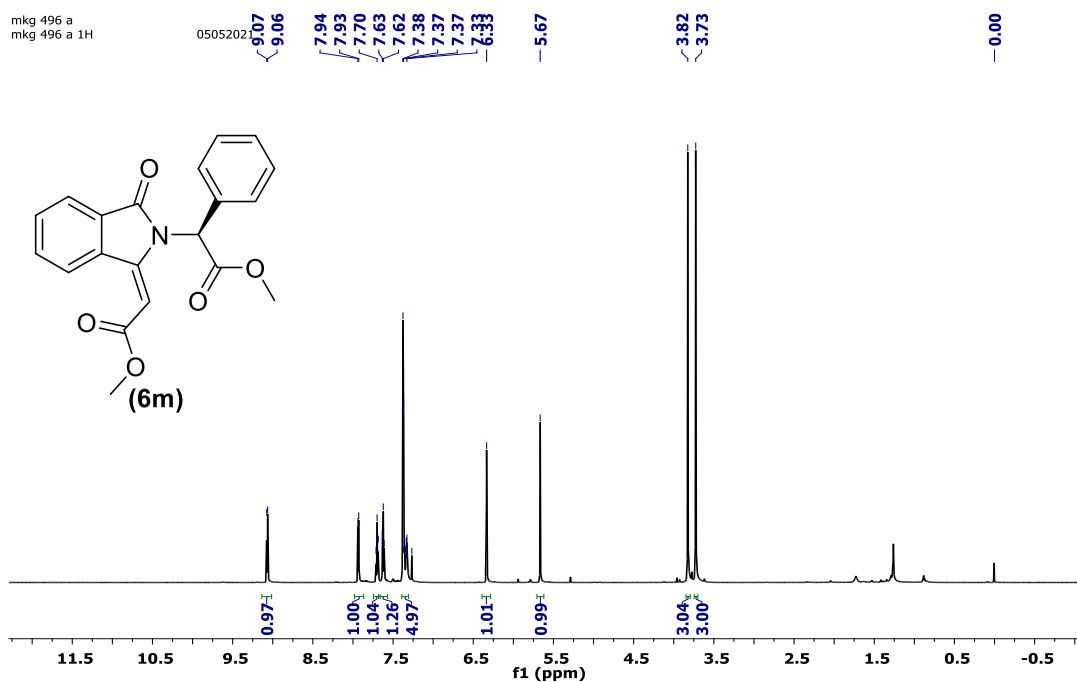


Figure. S34. ESI-HRMS spectra of benzamide **3e**

mkg 496 a
mkg 496 a 1H



496a
13C

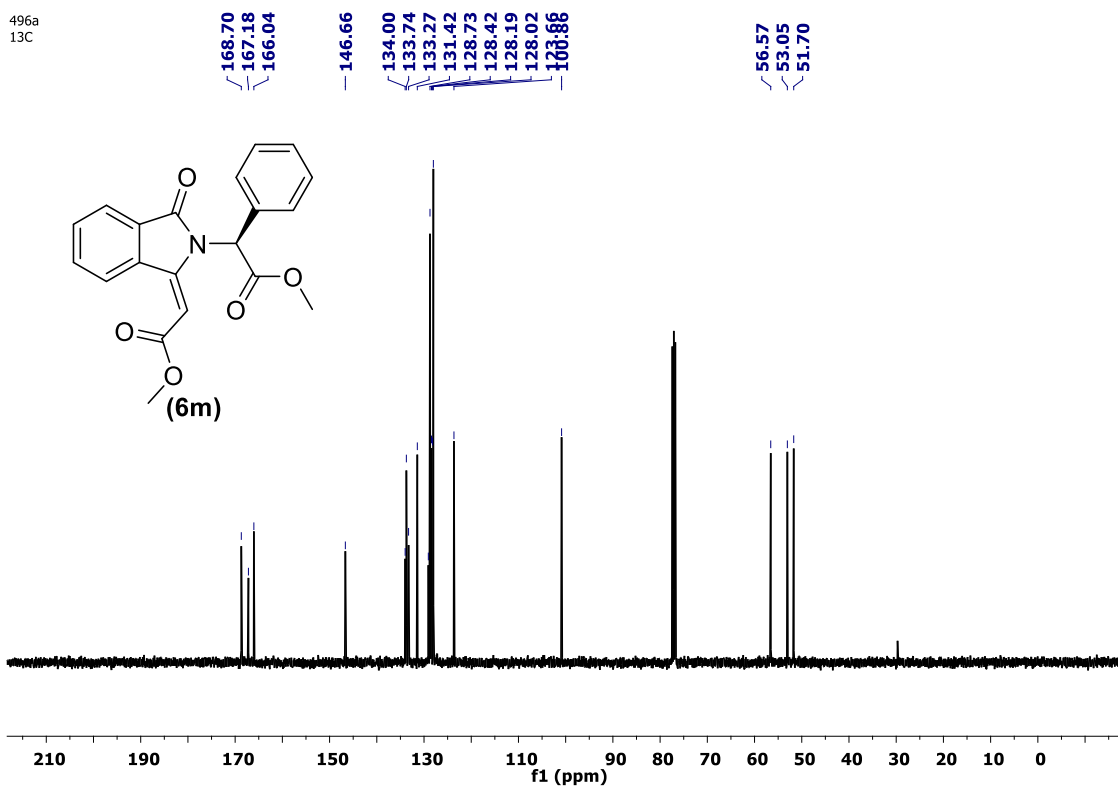


Figure. S35. ¹H, ¹³C NMR spectra of indolinone **6m**

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:32:38 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
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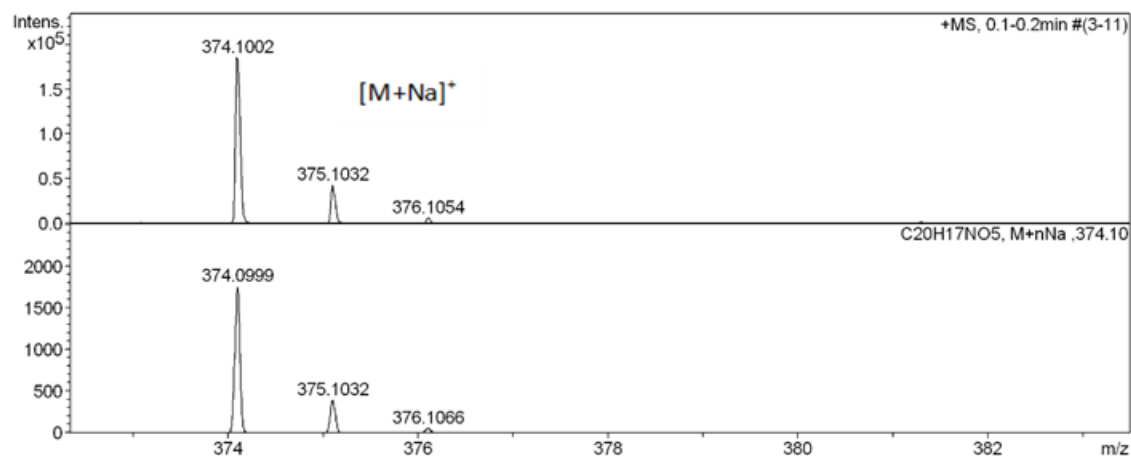
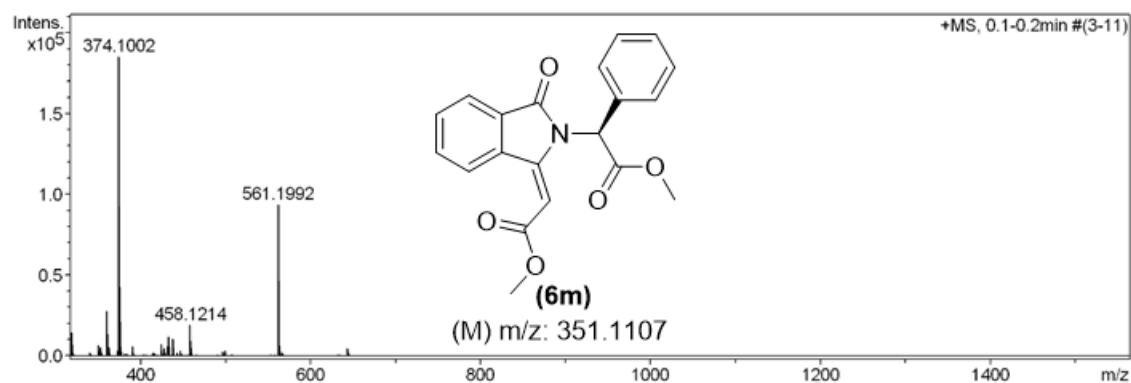
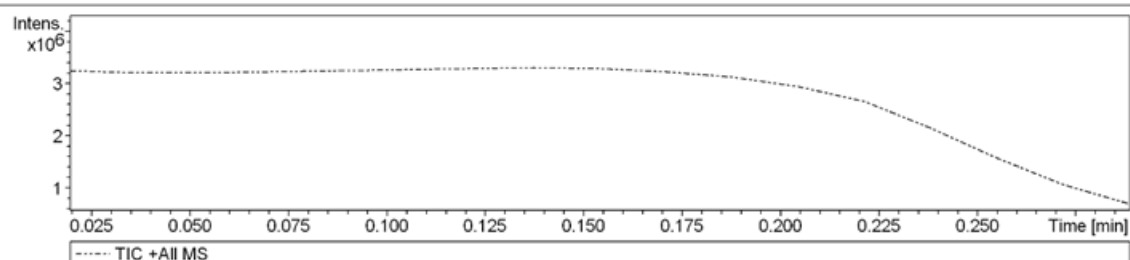


Figure. S36. ESI-HRMS spectra of indolinone **6m**

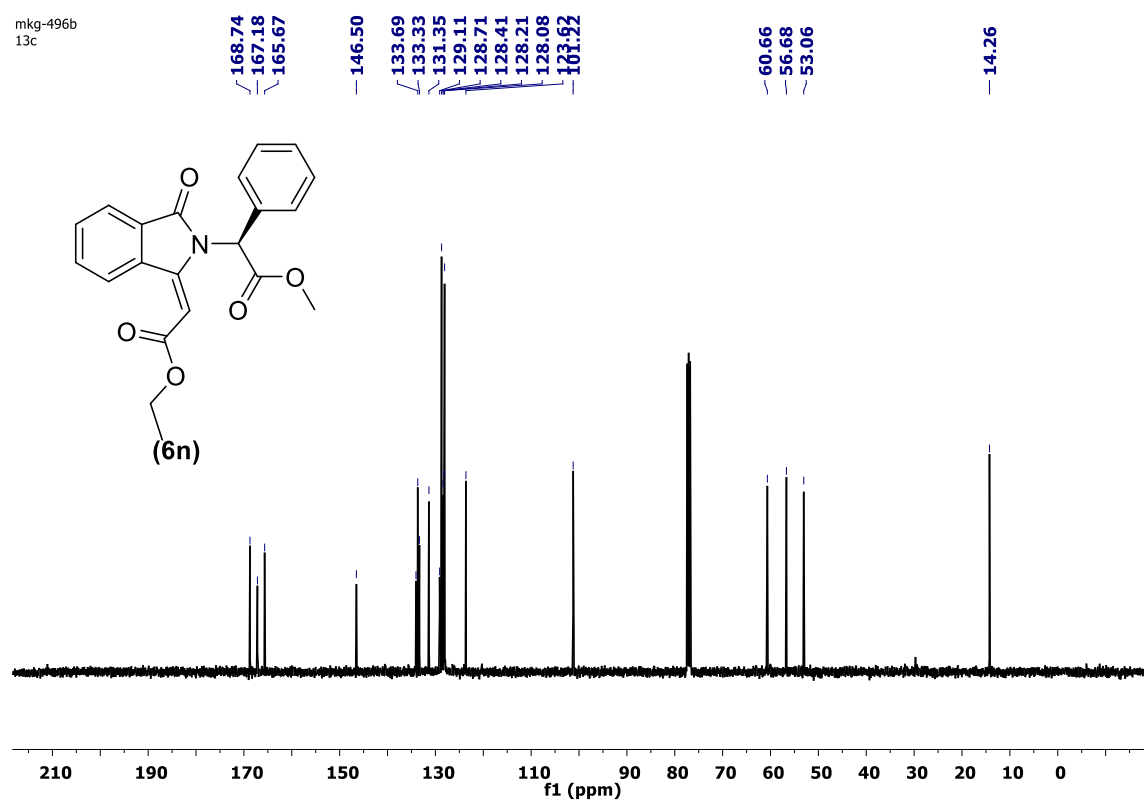
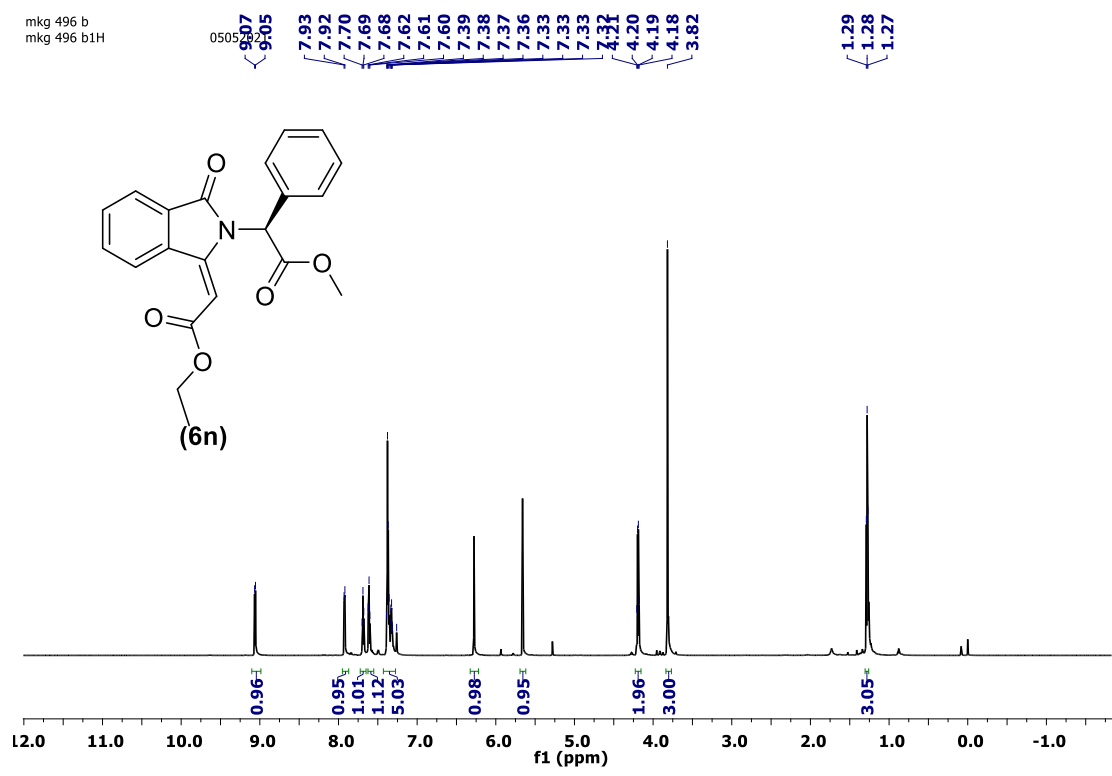


Figure. S37. ¹H, ¹³C NMR spectra of indolinone 6n

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:34:56 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

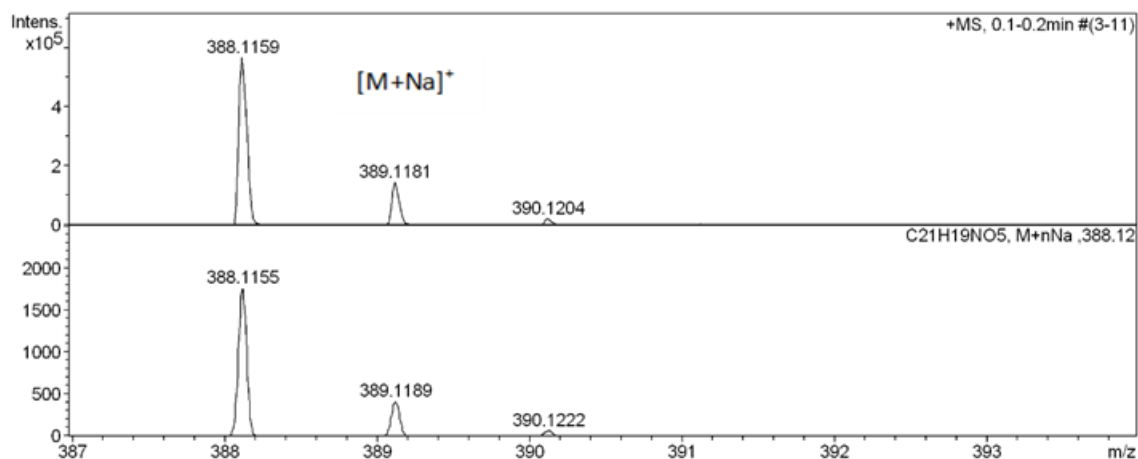
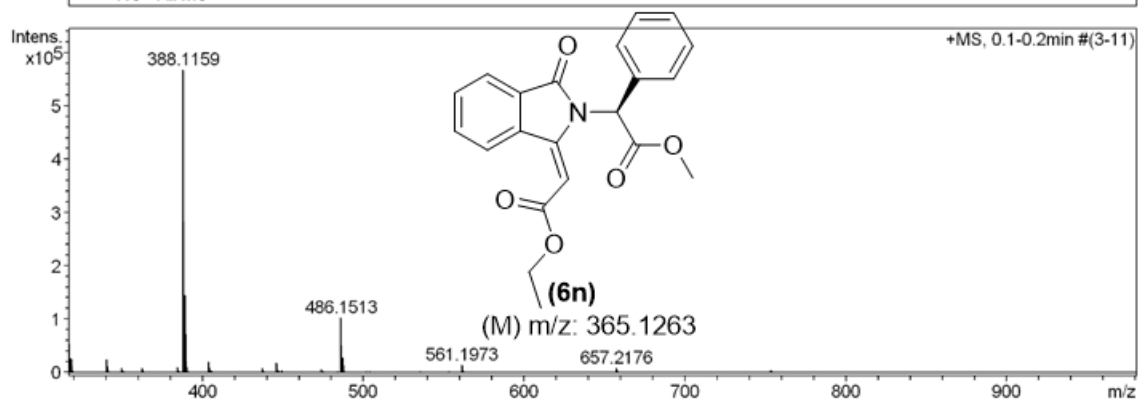
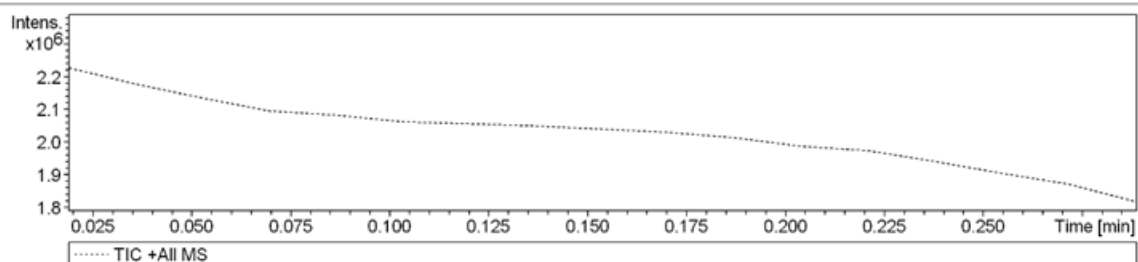
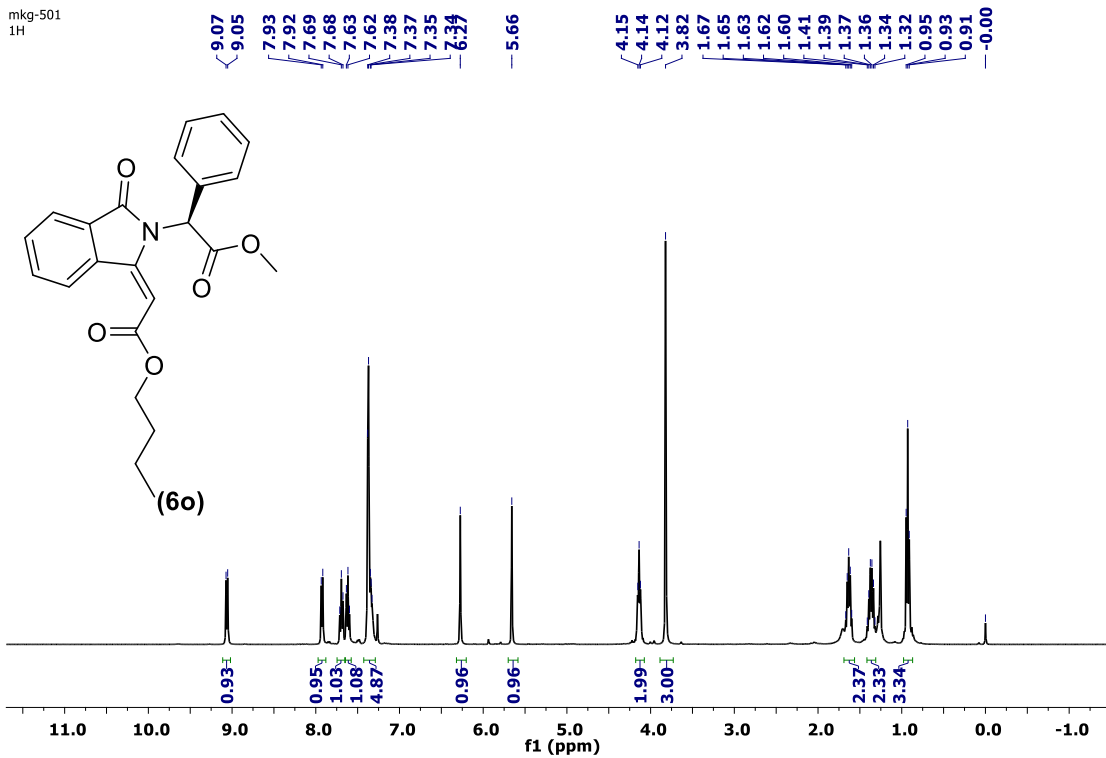


Figure. S38. ESI-HRMS spectra of indolinone 6n

mkg-501
1H



mkg-501
13C

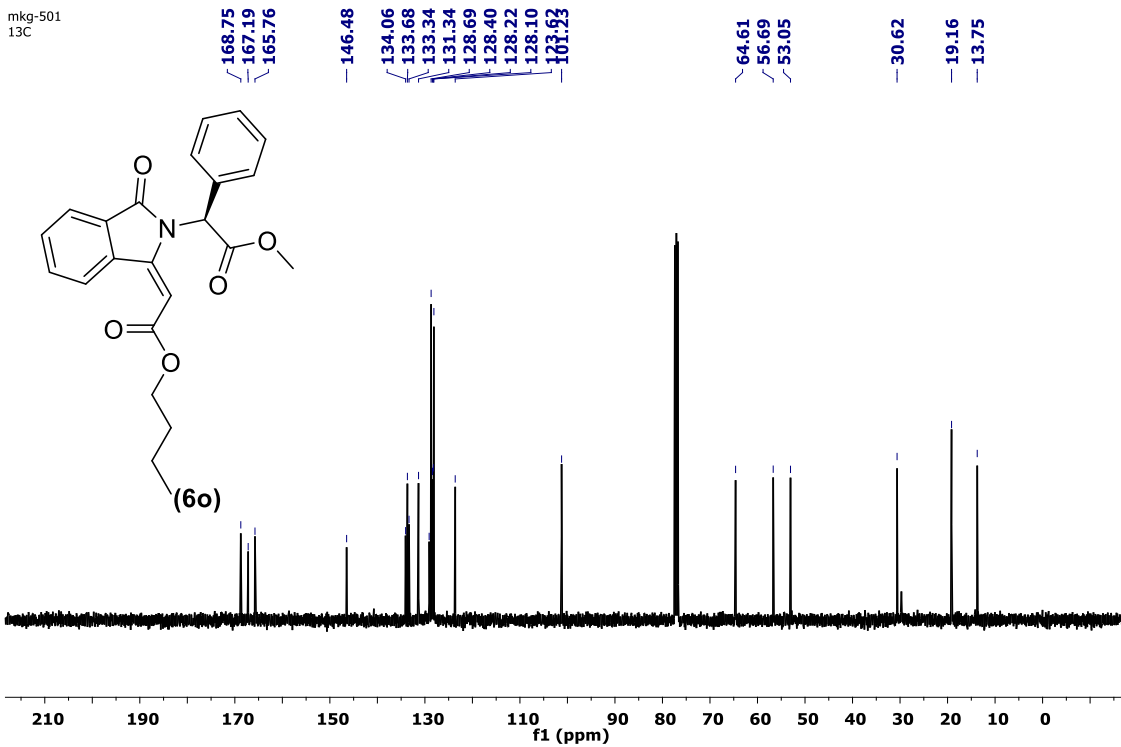


Figure. S39. ¹H, ¹³C NMR spectra of indolinone 6o

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:36:56 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

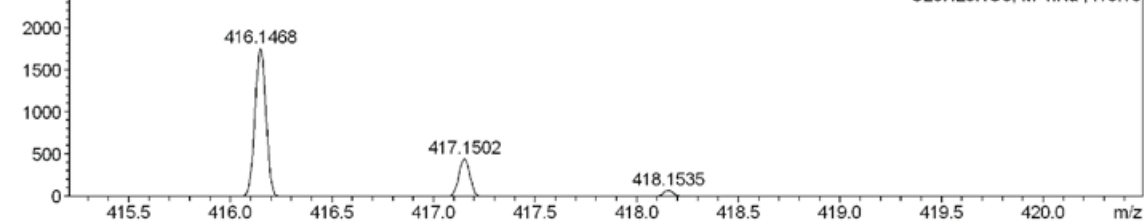
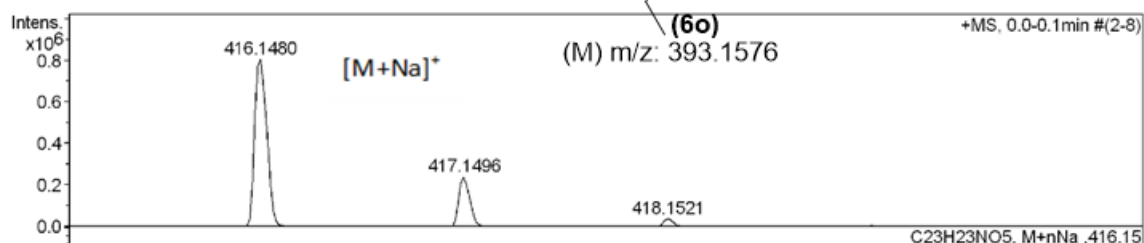
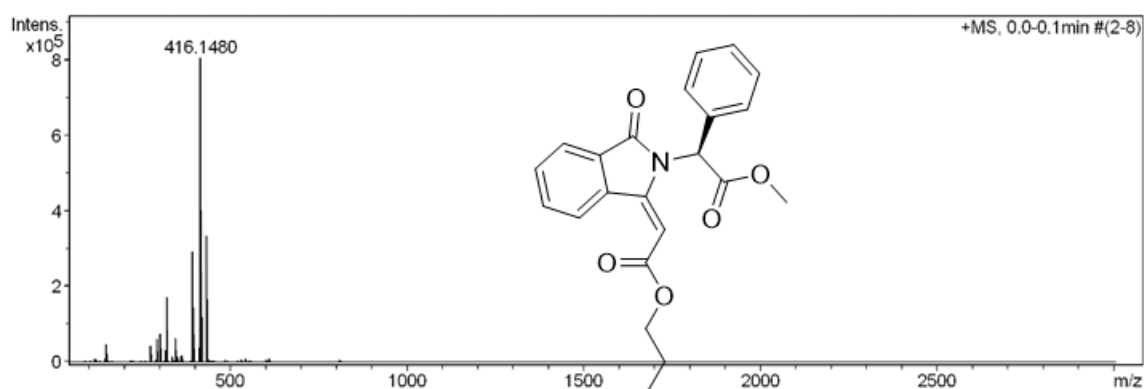
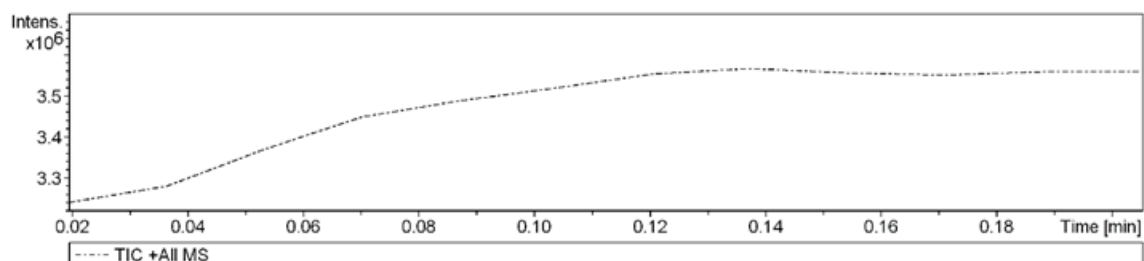
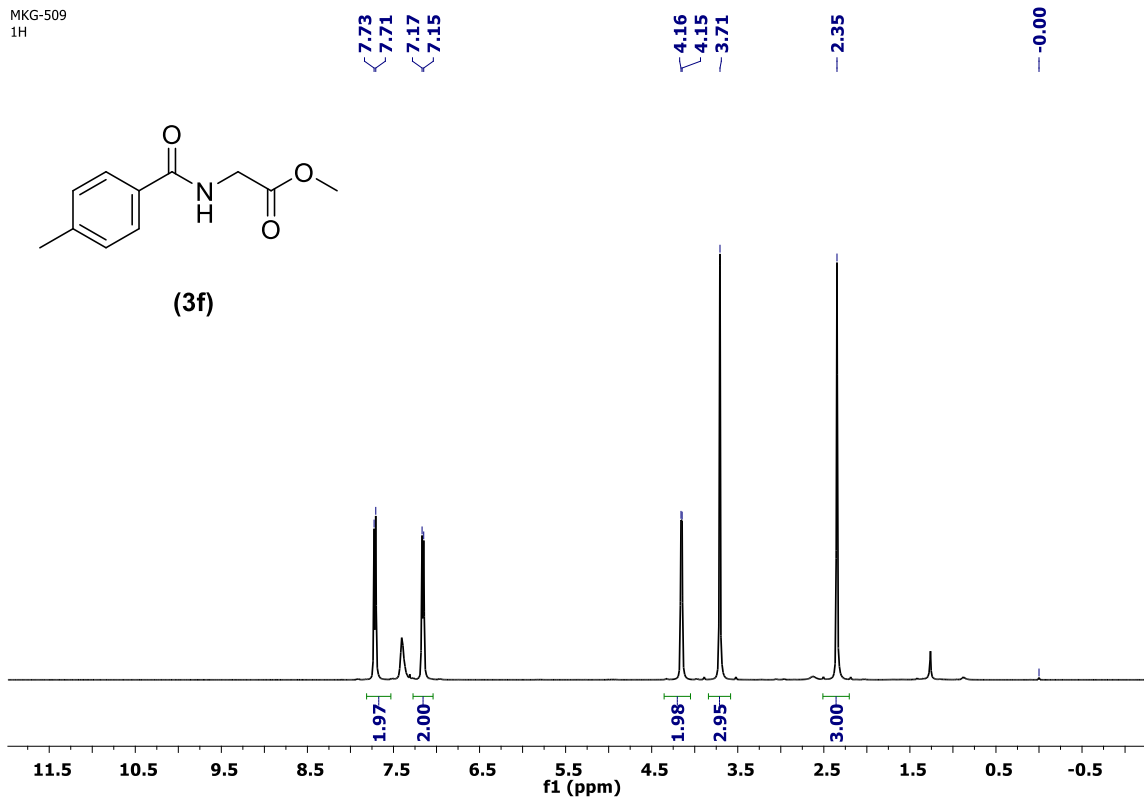


Figure. S40. ESI-HRMS spectra of indolinone **6o**

MKG-509
1H



MKG-509
13C

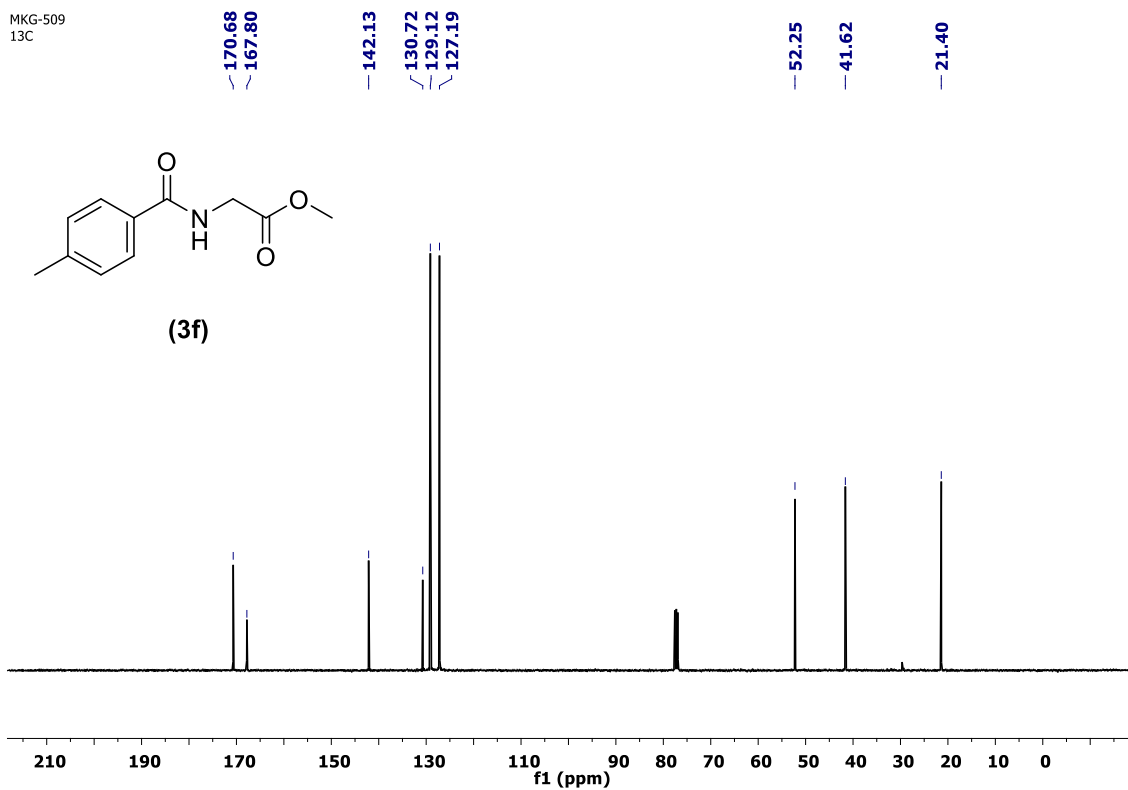


Figure. S41. ¹H, ¹³C NMR spectra of benzamide **3f**

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:39:46 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

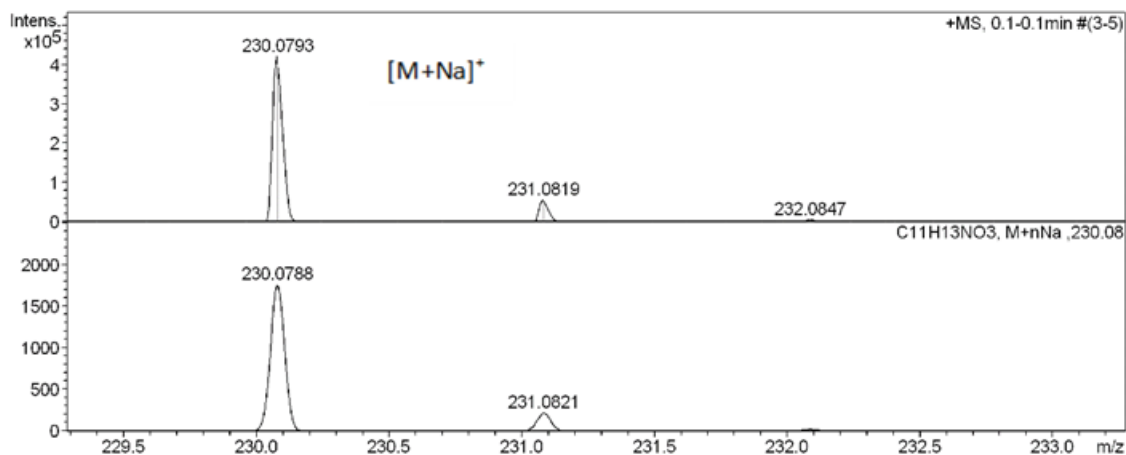
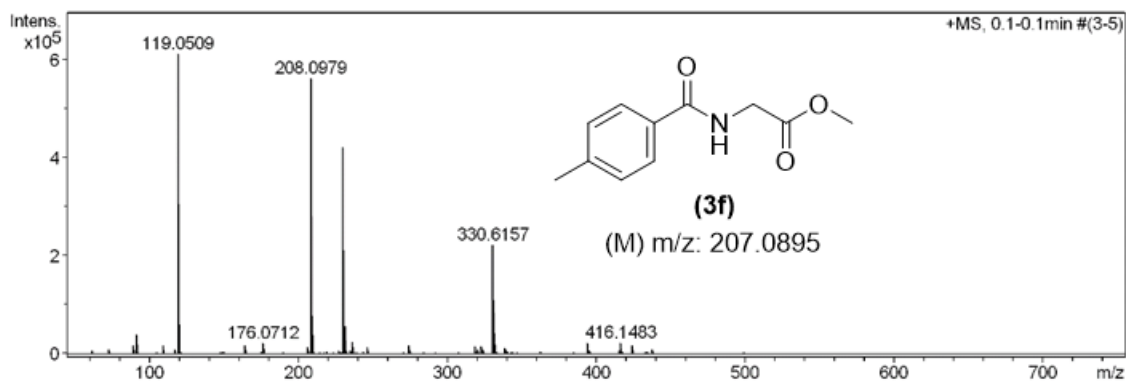
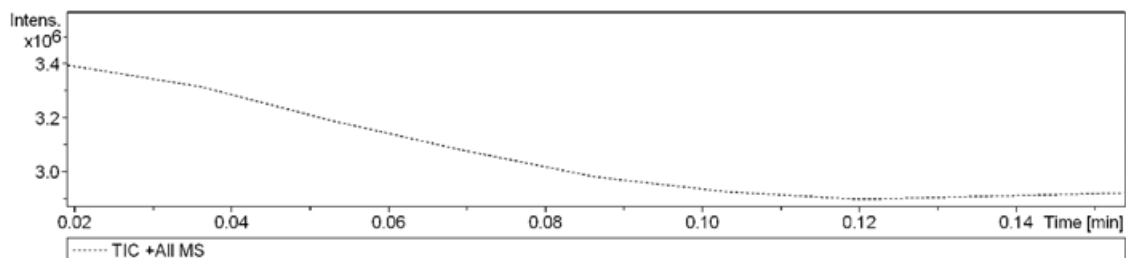
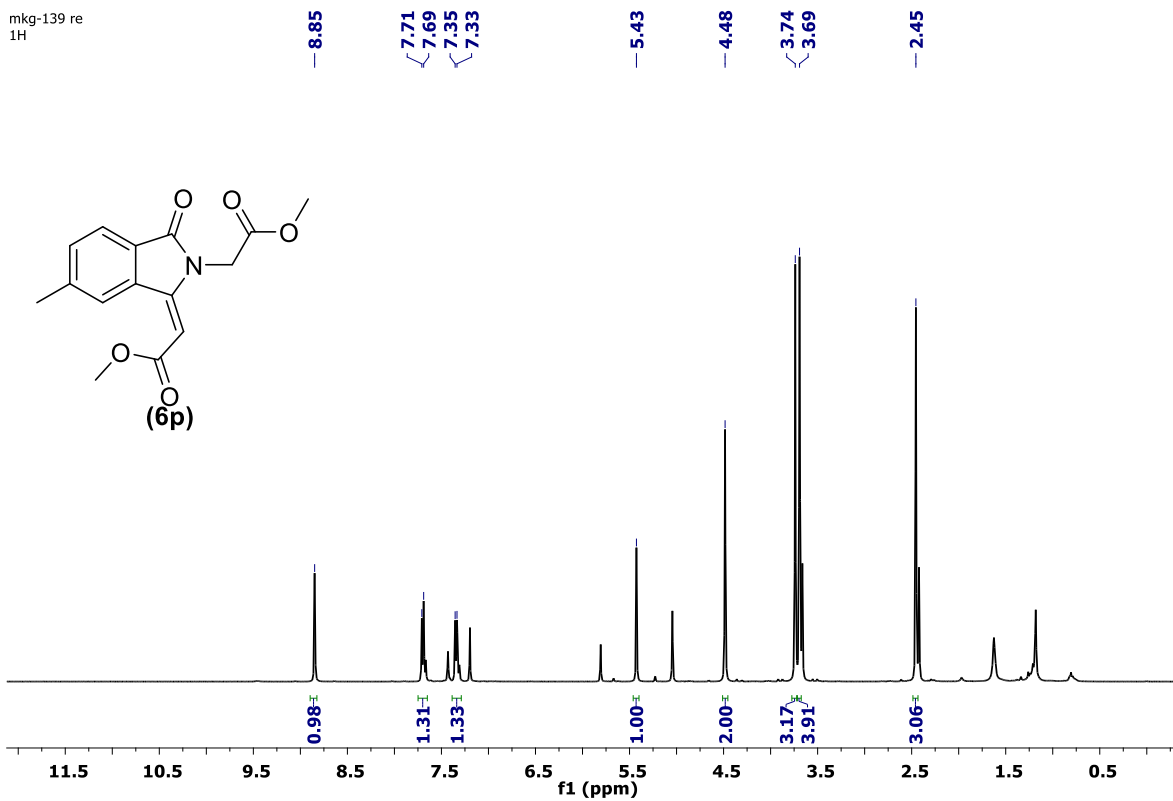


Figure. S42. ESI-HRMS spectra of benzamide **3f**

mkg-139 re
1H



mkg-139 re
13C

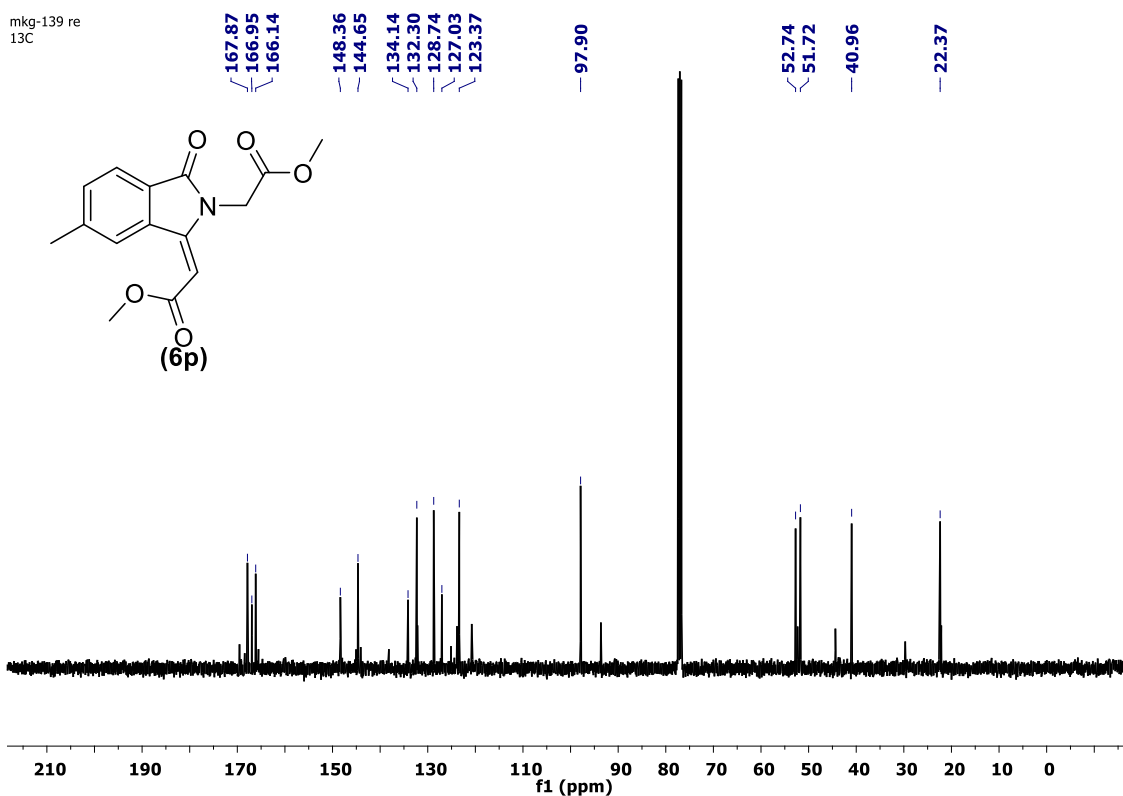


Figure.S43. ¹H, ¹³C NMR spectra of indolinone **6p**

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:43:46 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

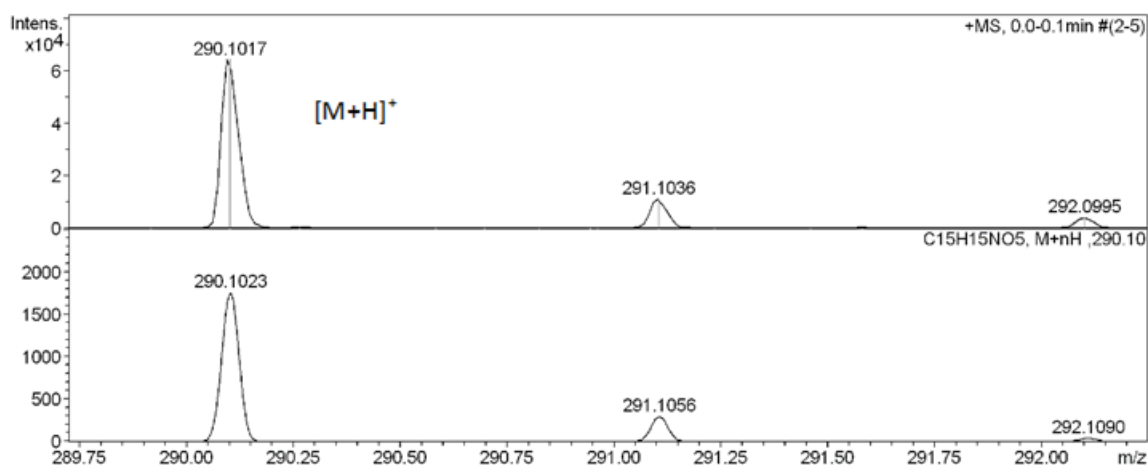
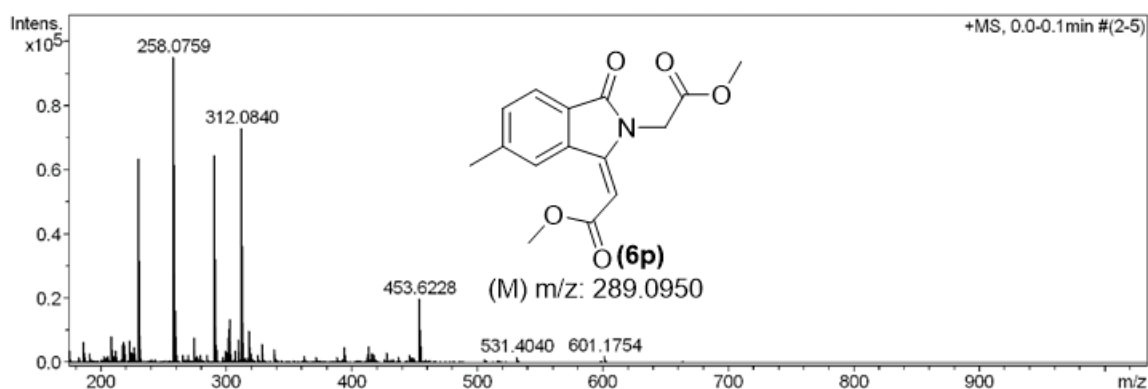
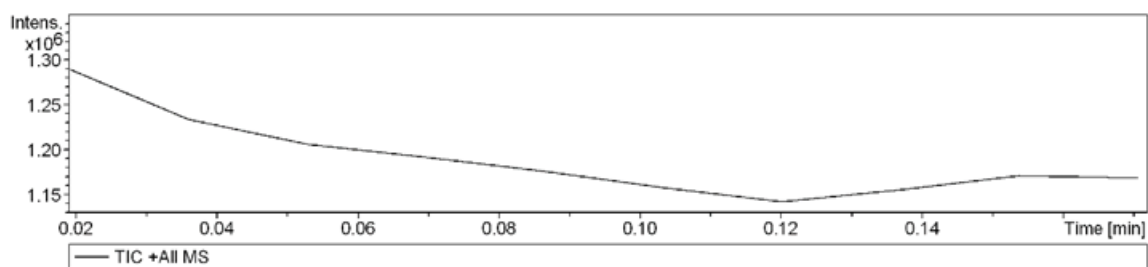
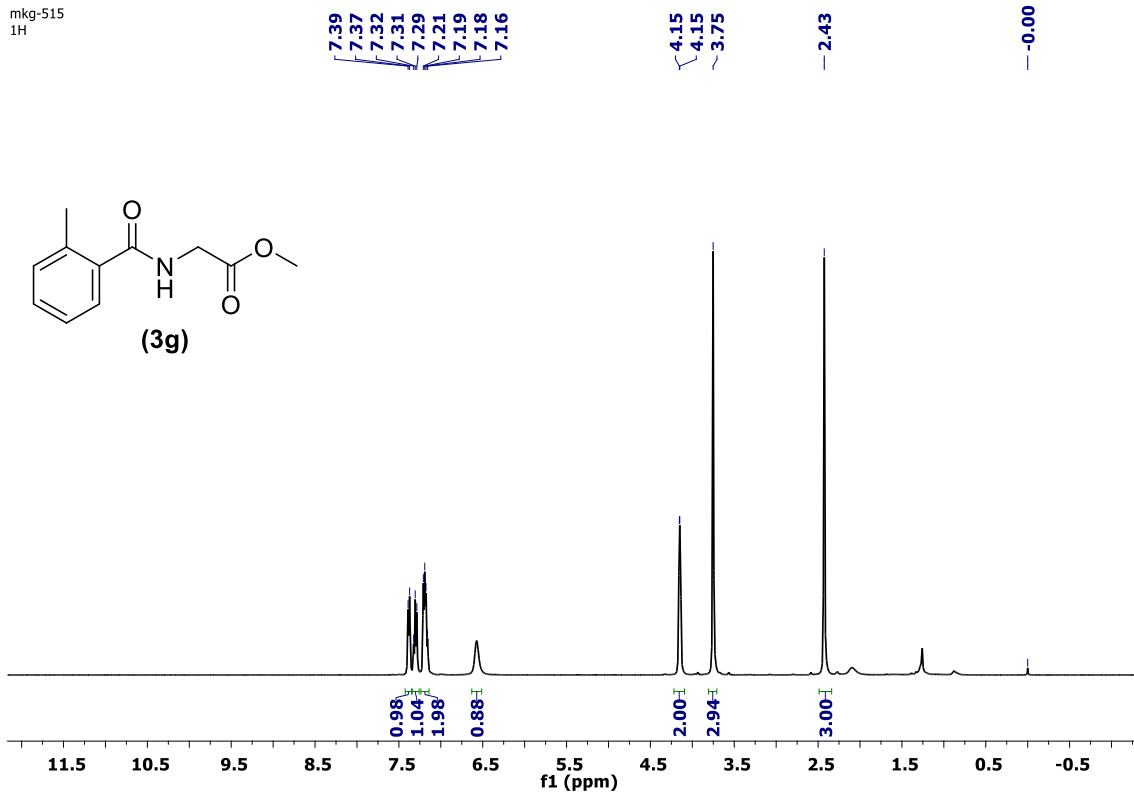


Figure. S44. ESI-HRMS spectra of indolinone 6p

mkg-515
1H



mkg-515
13c

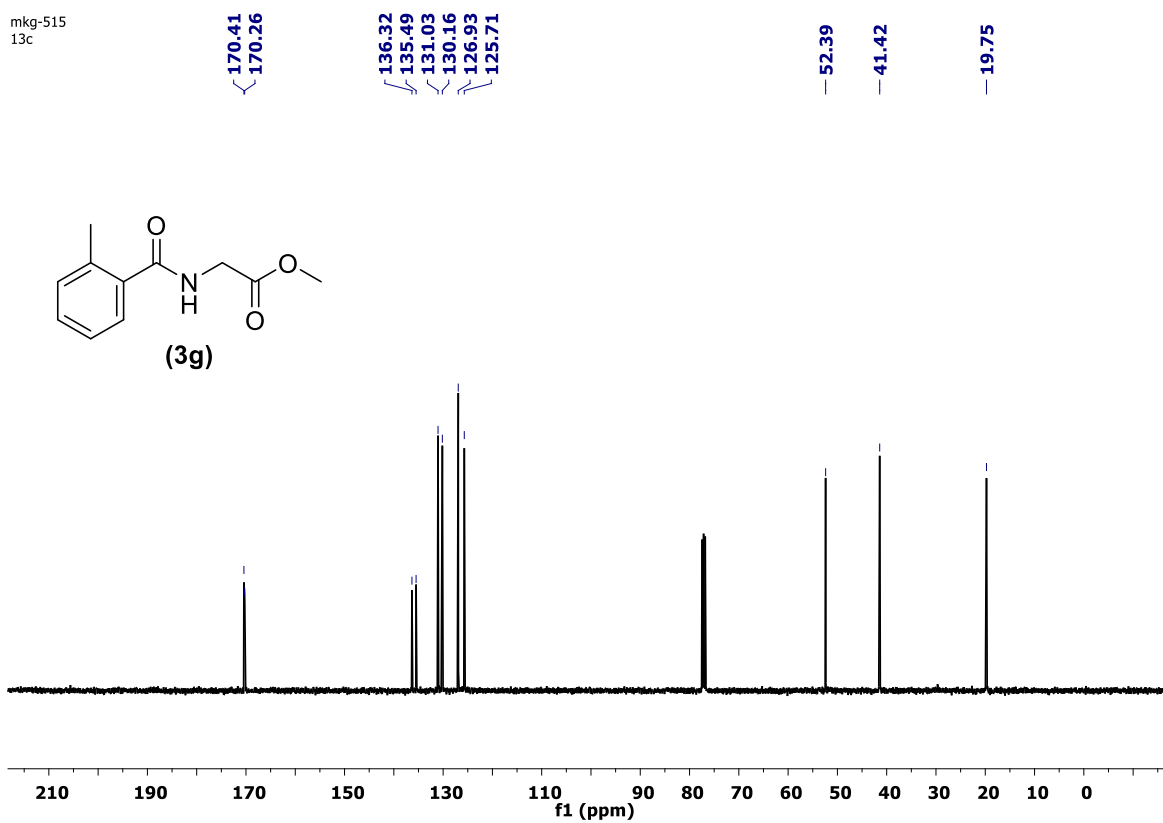


Figure. S45. ¹H, ¹³C NMR spectra of benzamide 3g

Display Report

Analysis Info

Analysis Name D:\Data\JUN-2021\NKS\21062021_NKS_MKG_515.d
Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:46:21 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

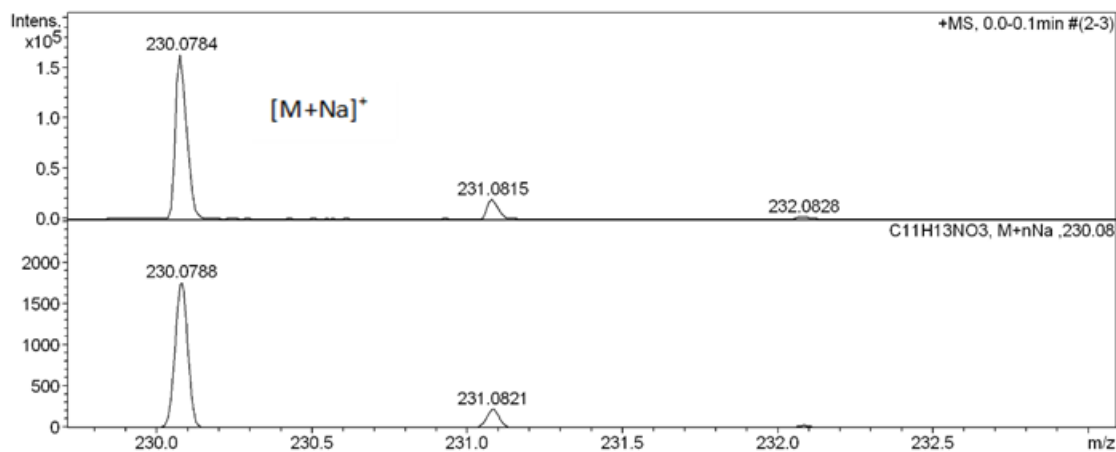
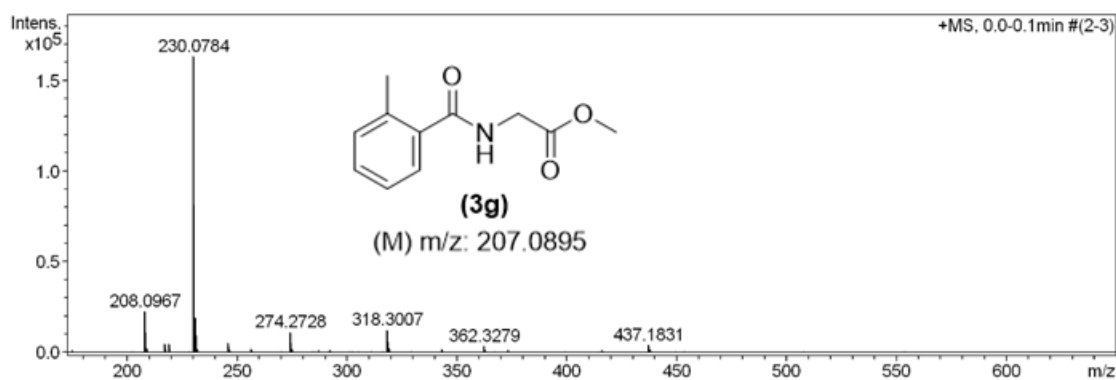
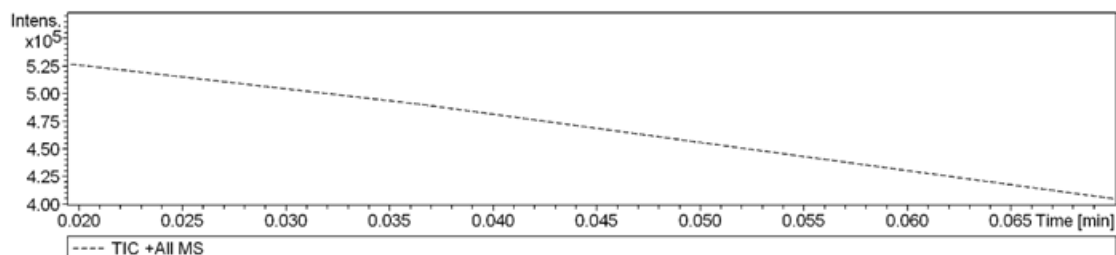
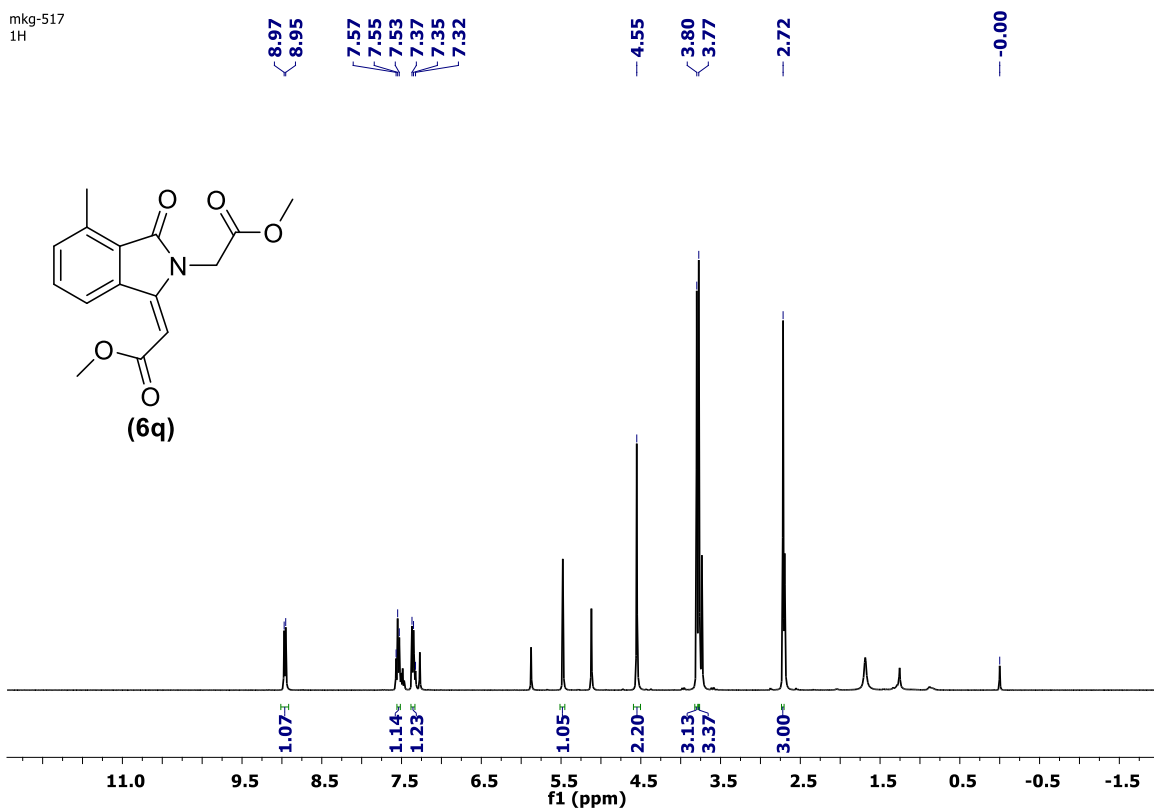


Figure. S46. ESI-HRMS spectra of benzamide **3g**

mkg-517
1H



mkg-517
13C

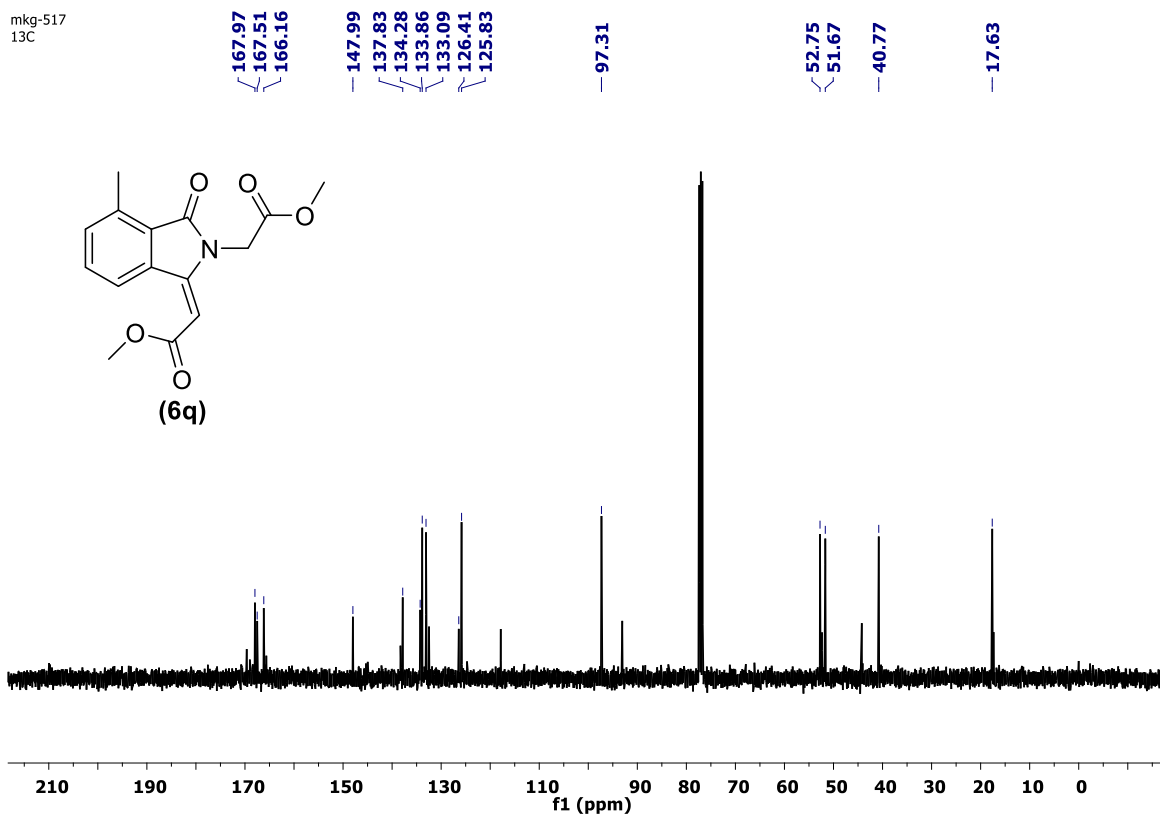


Figure. S47. ¹H, ¹³C NMR spectra of indolinone **6q**

Display Report

Analysis Info

Analysis Name D:\Data\JUN-2021\NKS\21062021_NKS_MKG_517.d
Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 6/21/2021 5:48:18 PM

Operator Amit S.Sahu
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

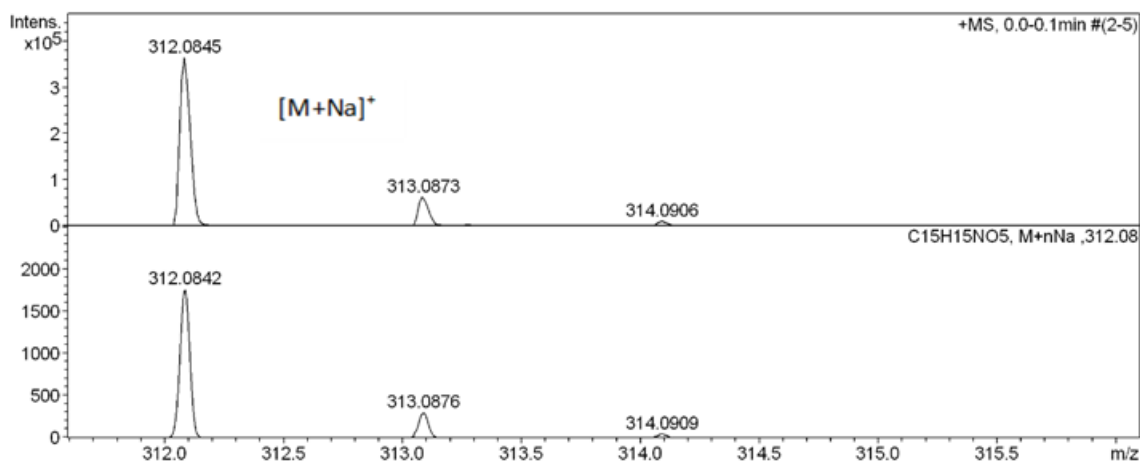
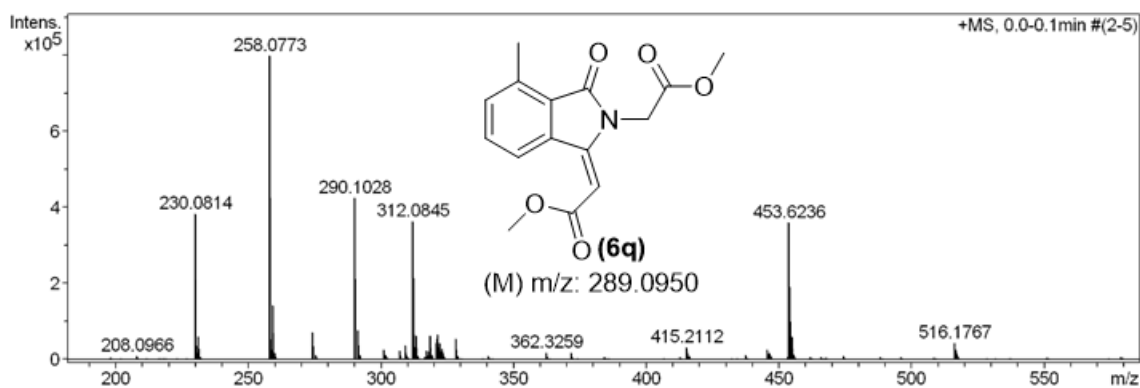
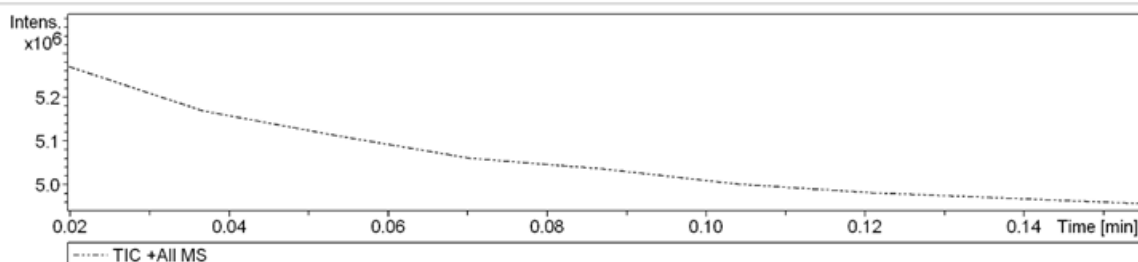
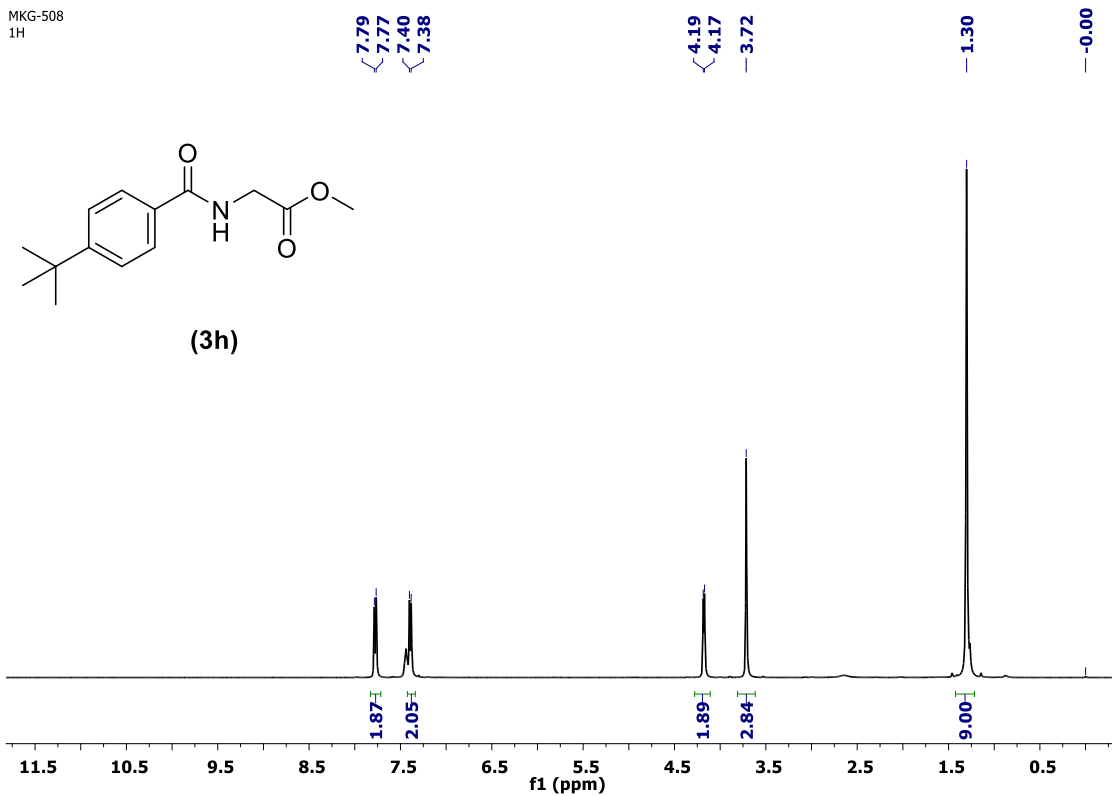


Figure. S48. ESI-HRMS spectra of indolinone 6q

MKG-508
1H



MKG-508
13C

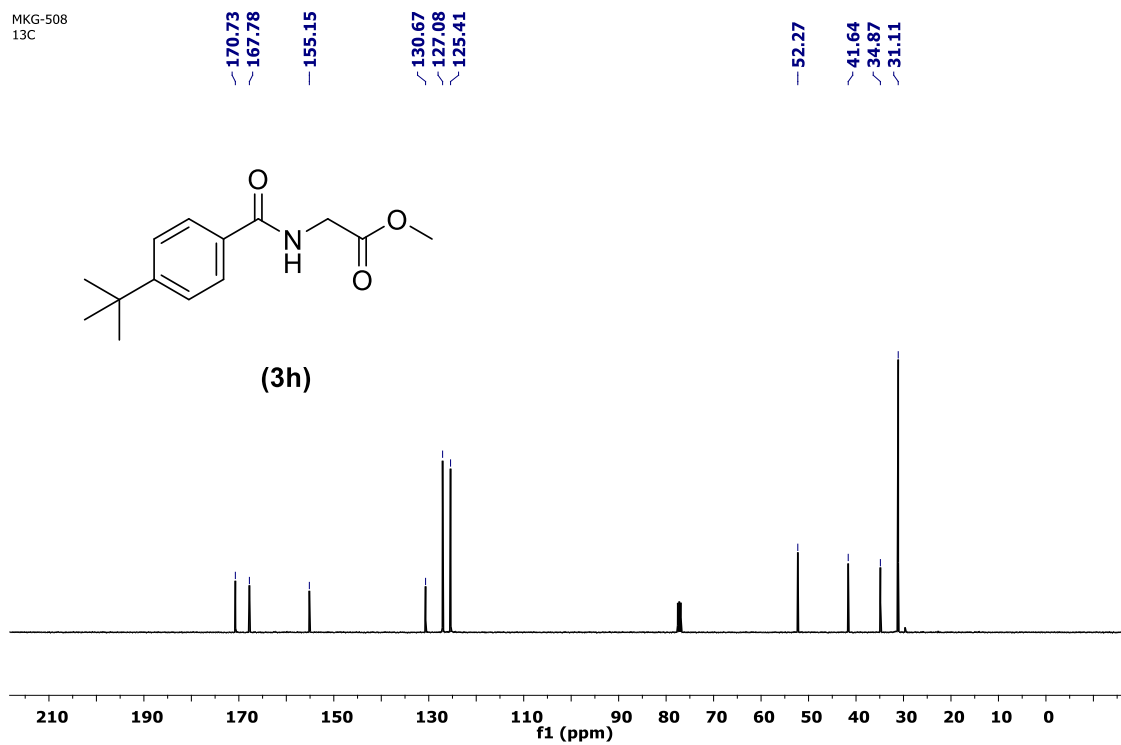


Figure. S49. ¹H, ¹³C NMR spectra of benzamide **3h**

Display Report

Analysis Info

Analysis Name D:\Data\JULY-2021\NKS\MKG\13072021_NKS-MKG-508-RE.d
Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/13/2021 5:51:31 PM
Operator PRAKASH BEHERA
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

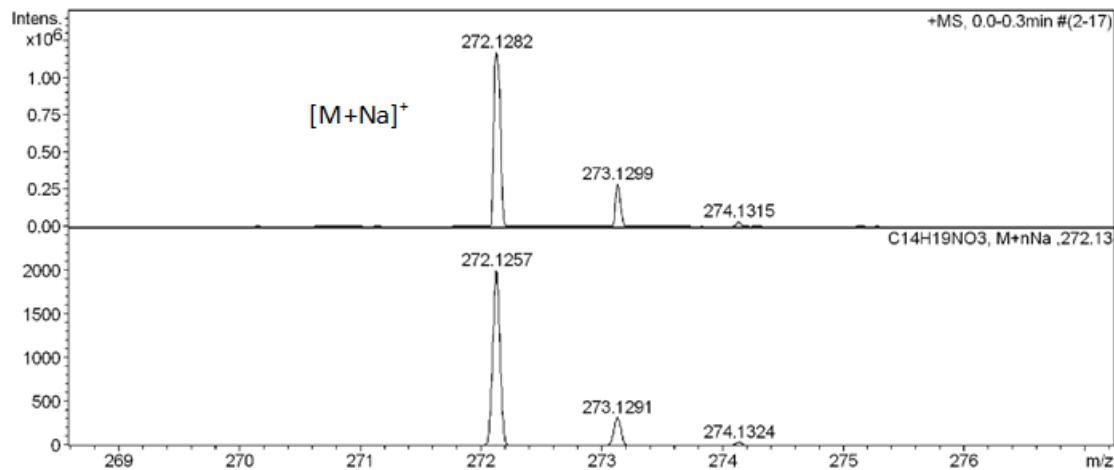
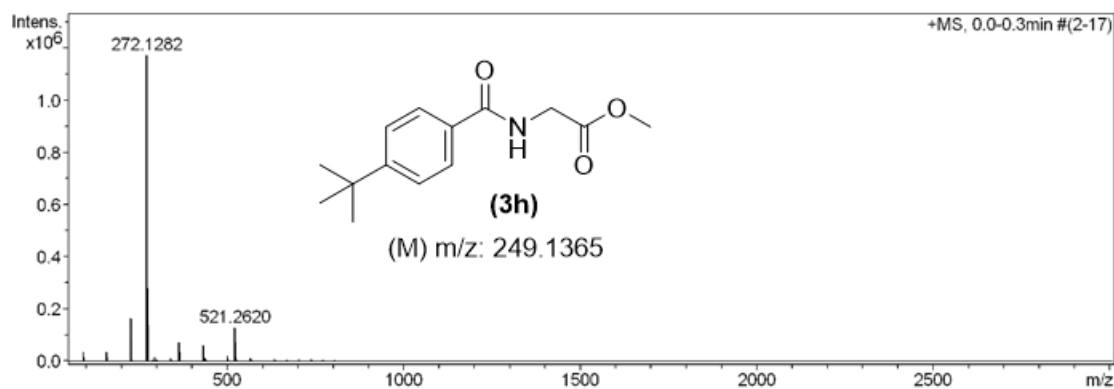
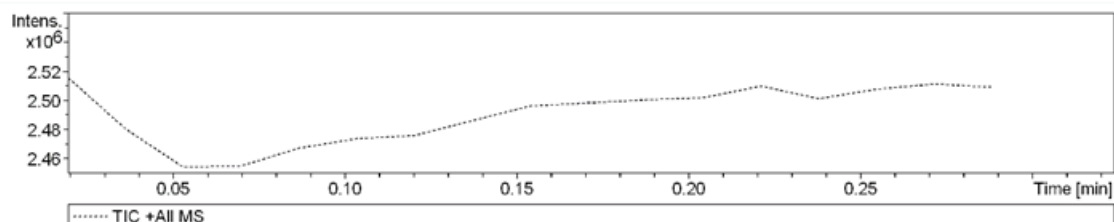


Figure. S50. ESI-HRMS spectra of benzamide **3h**

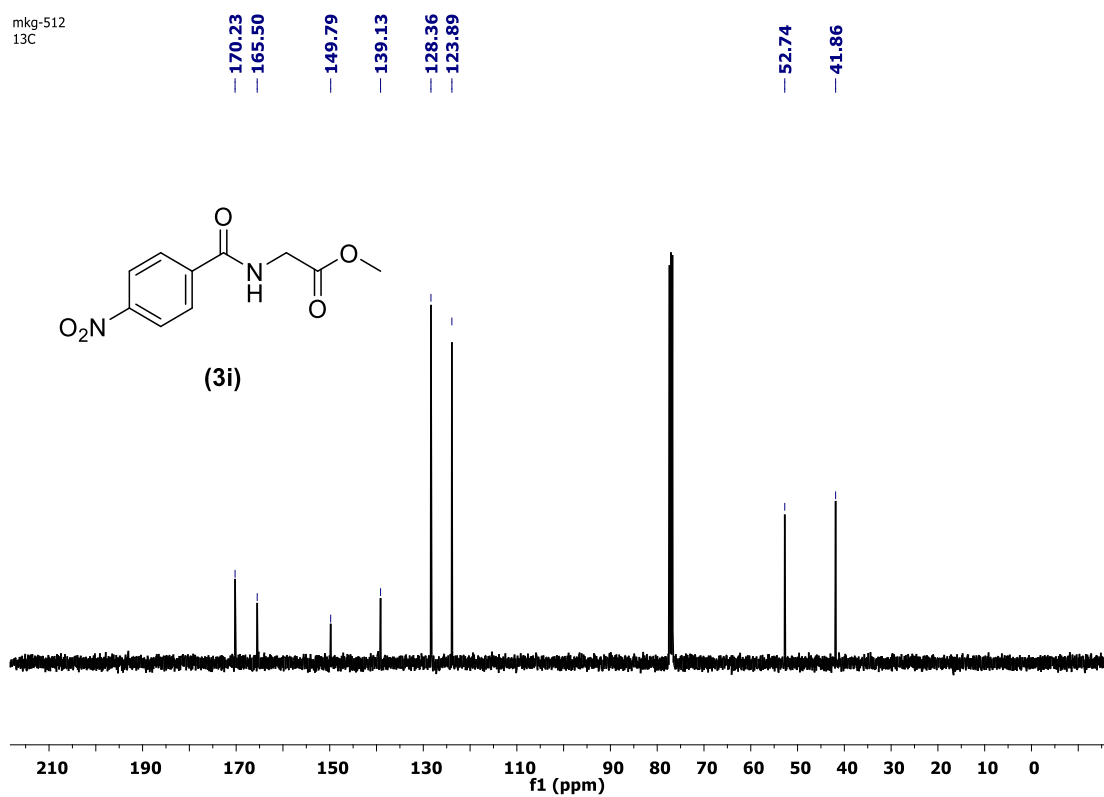
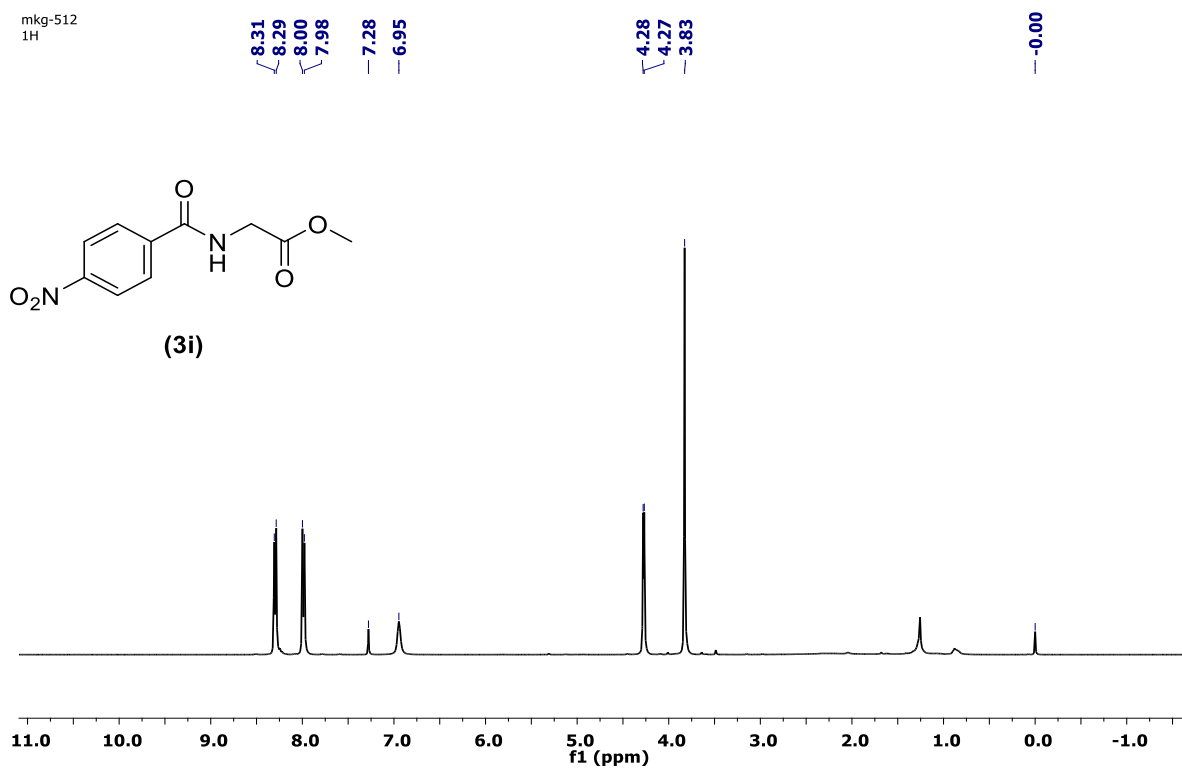


Figure. S51. ¹H, ¹³C NMR spectra of benzamide 3i

Display Report

Analysis Info

Analysis Name D:\Data\JULY-2021\NKS\MKG\13072021_NKS-MKG-512.d
Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/13/2021 5:56:32 PM

Operator PRAKASH BEHERA
Instrument micOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

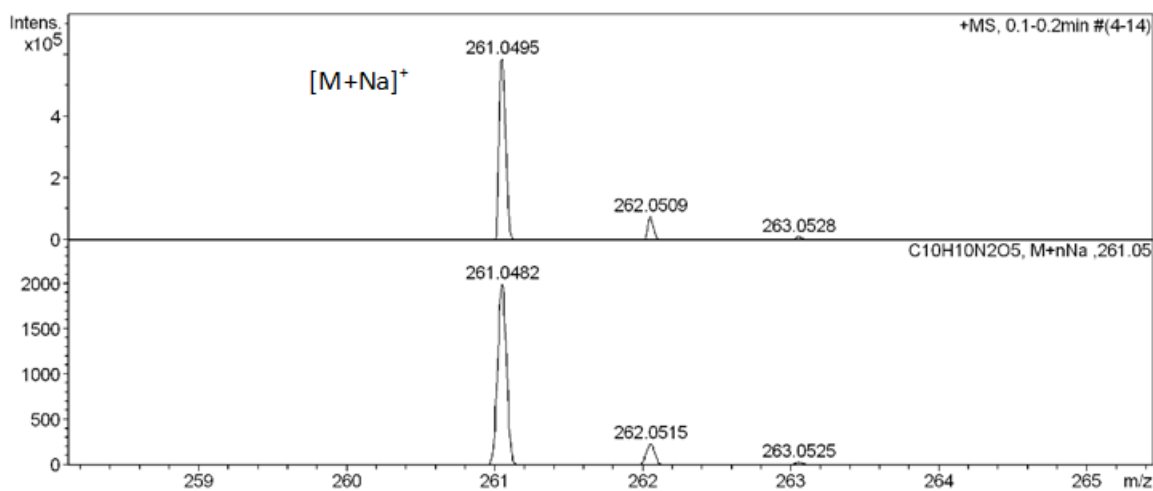
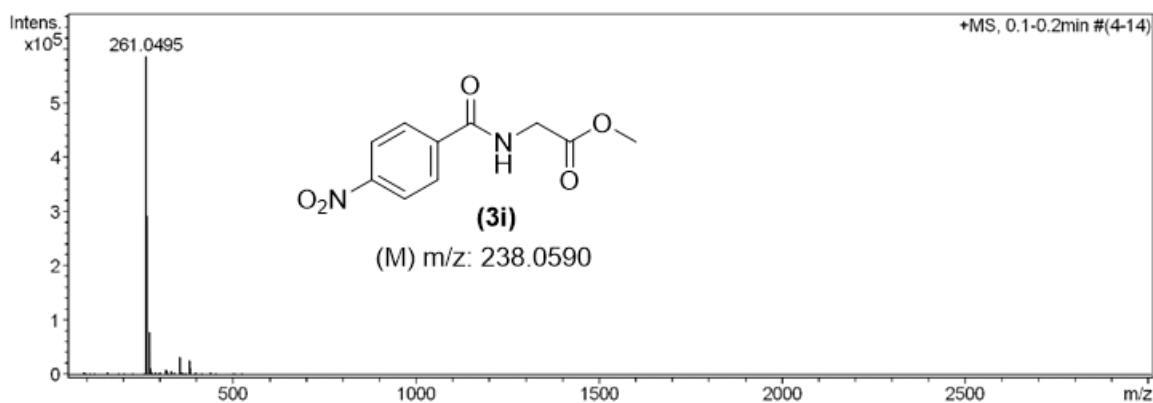
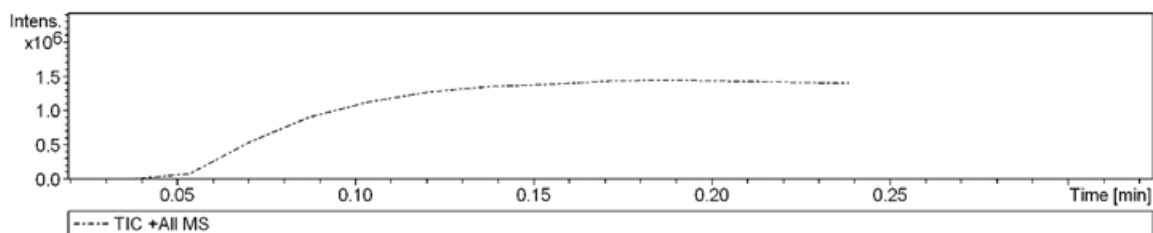
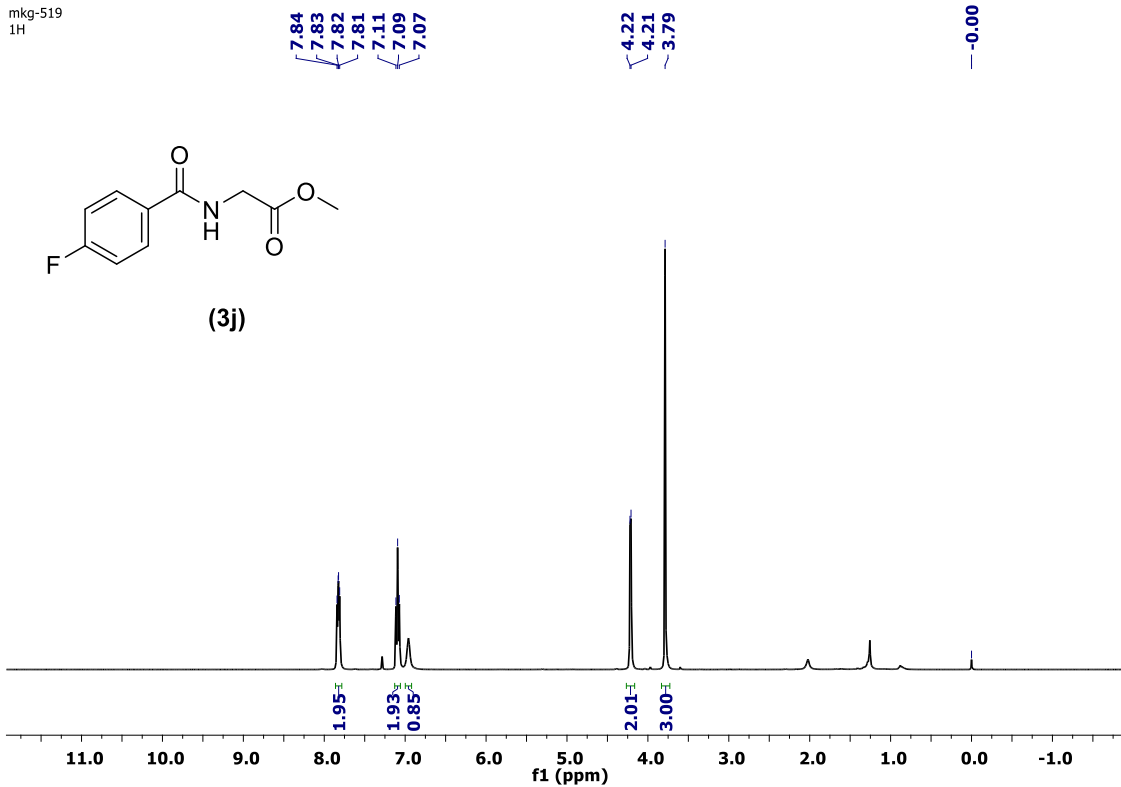


Figure. S52. ESI-HRMS spectra of benzamide **3i**

mkg-519
1H



mkg-519
13C

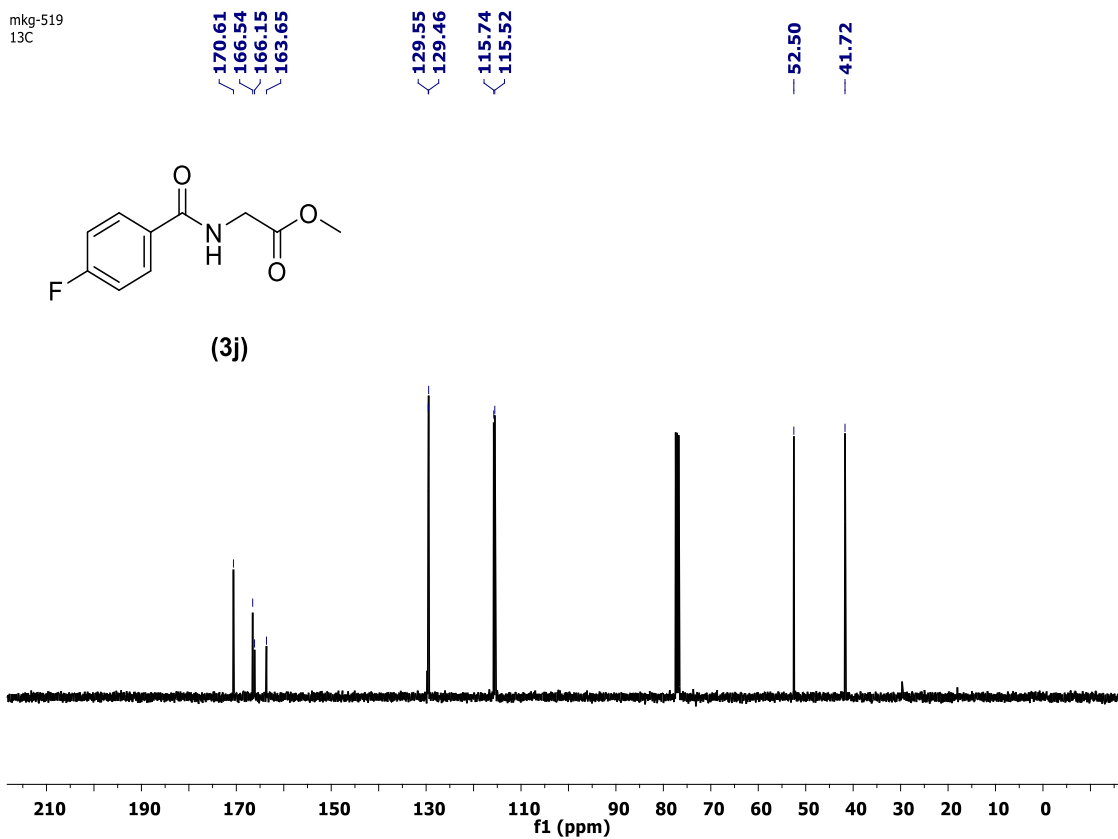


Figure. S53. ¹H, ¹³C NMR spectra of benzamide 3j

Display Report

Analysis Info

Analysis Name D:\Data\JULY-2021\NKS\MKG\13072021_NKS-MKG-519.d
Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/13/2021 5:59:41 PM

Operator PRAKASH BEHERA
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

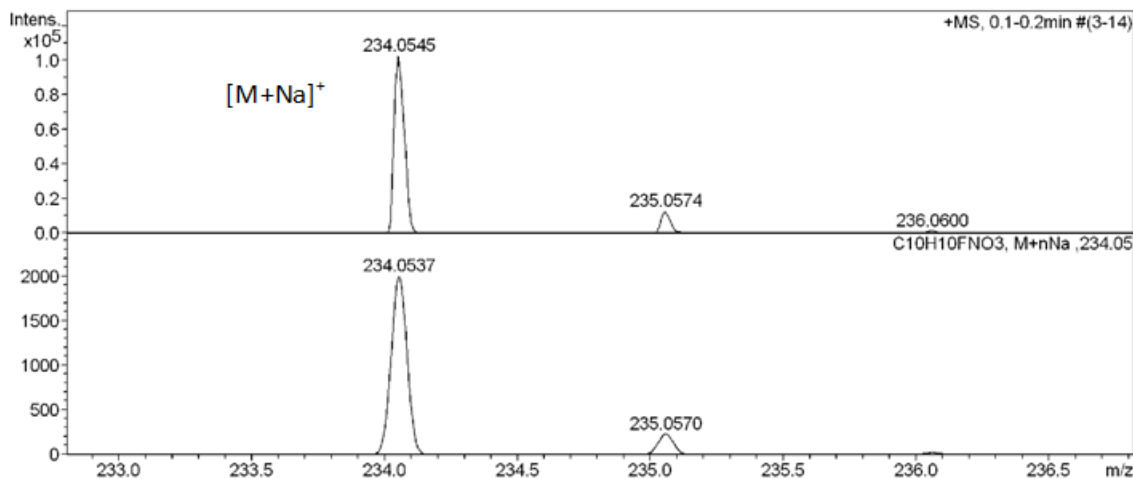
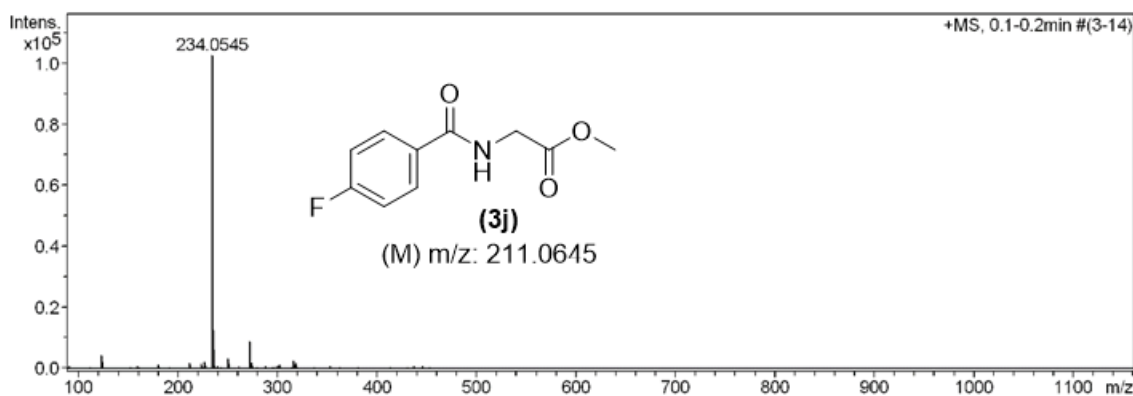
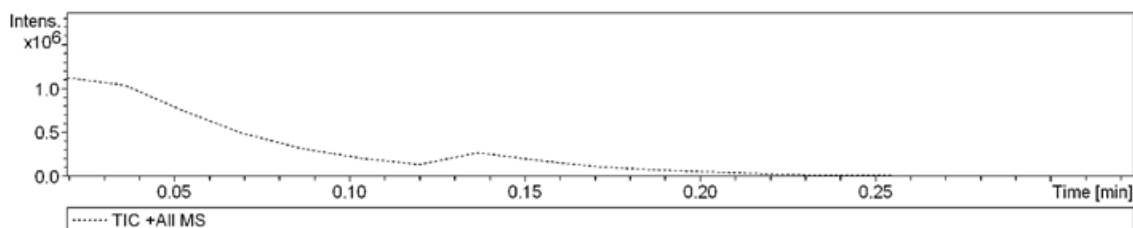


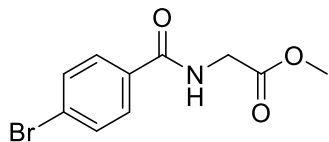
Figure. S54. ESI-HRMS spectra of benzamide 3j

mkg-520
1H

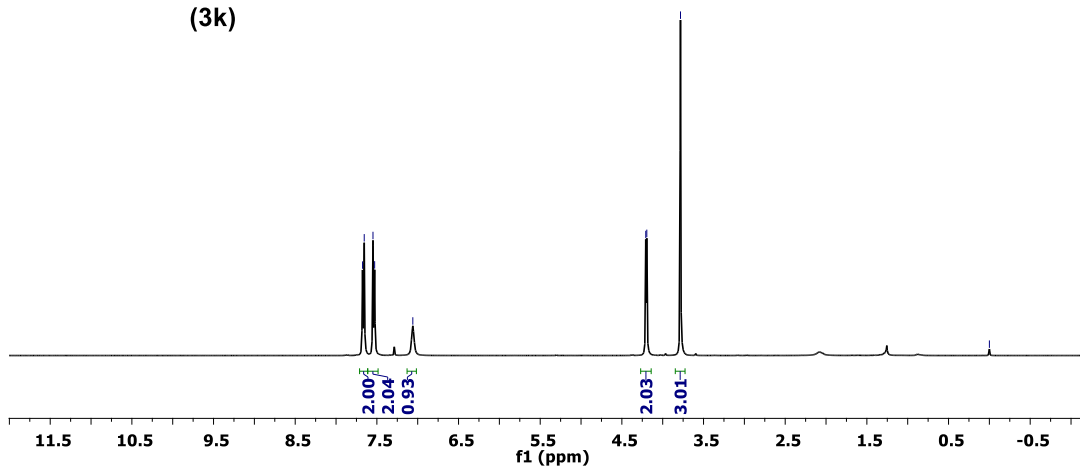
7.68
7.65
7.55
7.53
7.06

4.21
4.19
3.78

-0.00



(3k)

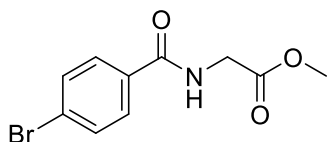


mkg-520
13C

170.56
166.69

132.36
131.80
128.74
126.58

52.55
41.71



(3k)

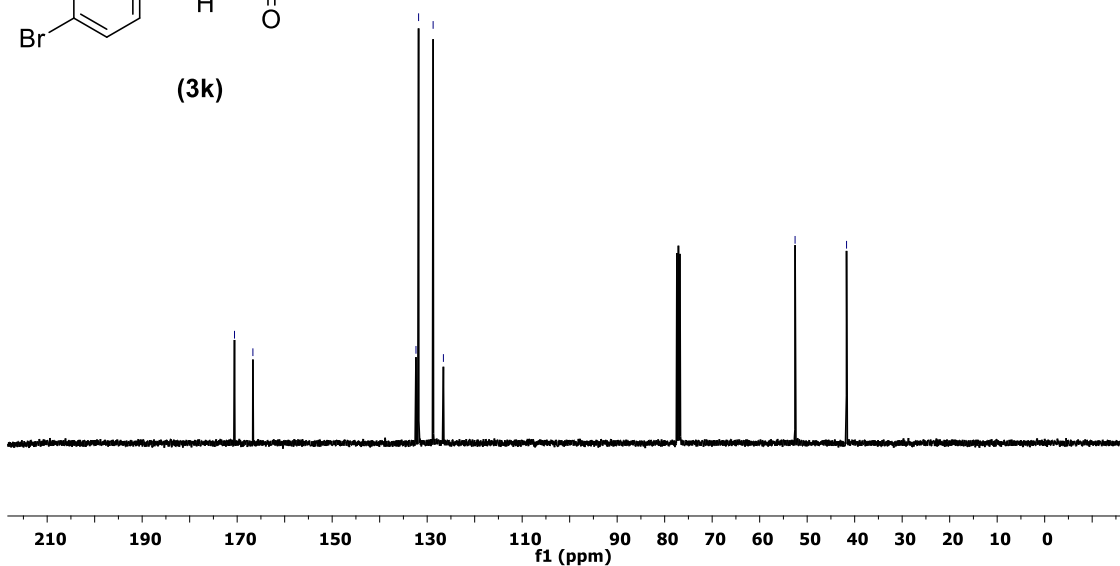


Figure. S55. ¹H, ¹³C NMR spectra of benzamide 3k

Display Report

Analysis Info

Analysis Name D:\Data\JULY-2021\NKS\MKG\13072021_NKS-MKG-520.d
Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/13/2021 6:07:51 PM

Operator PRAKASH BEHERA
Instrument micrOTOF-Q II 10337

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

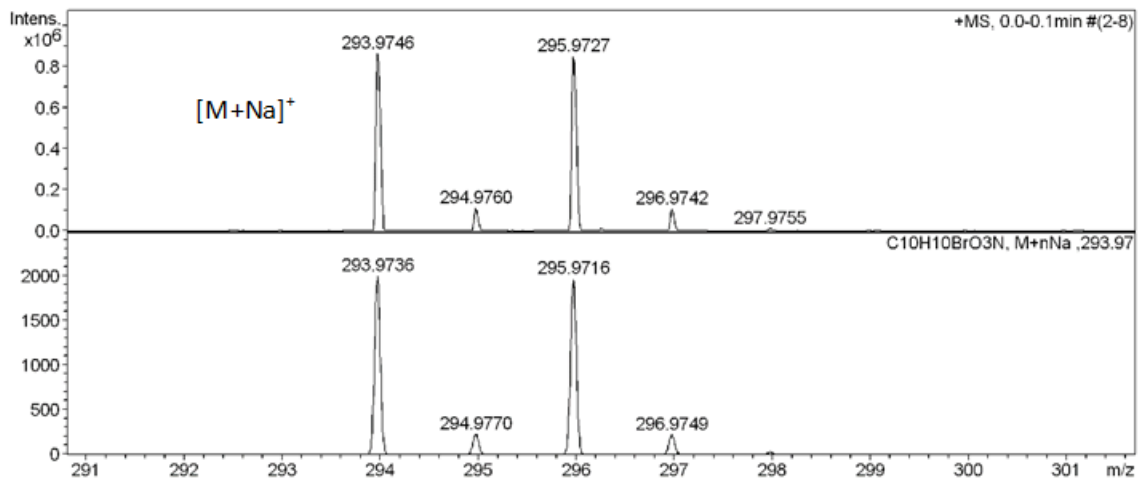
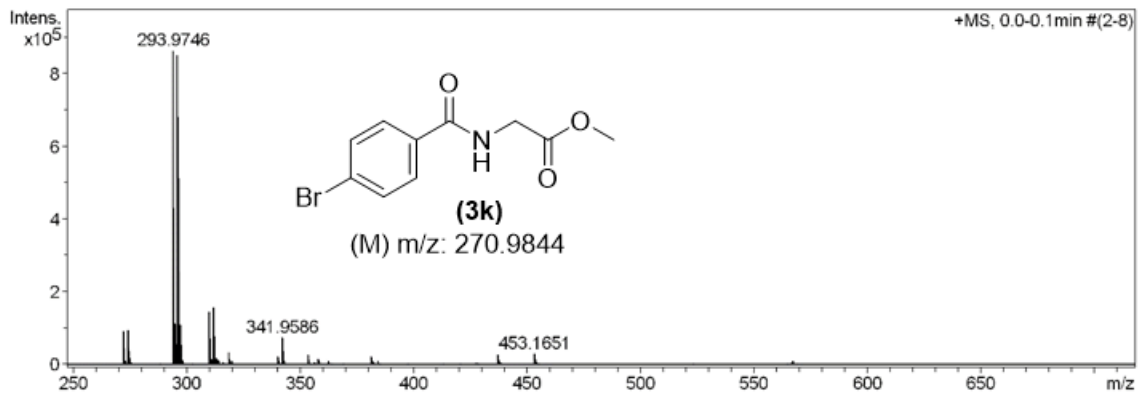
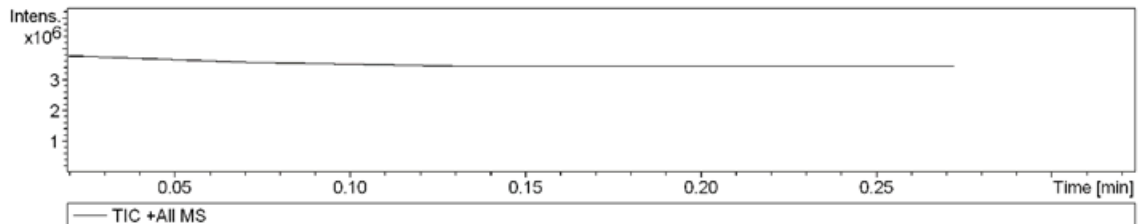
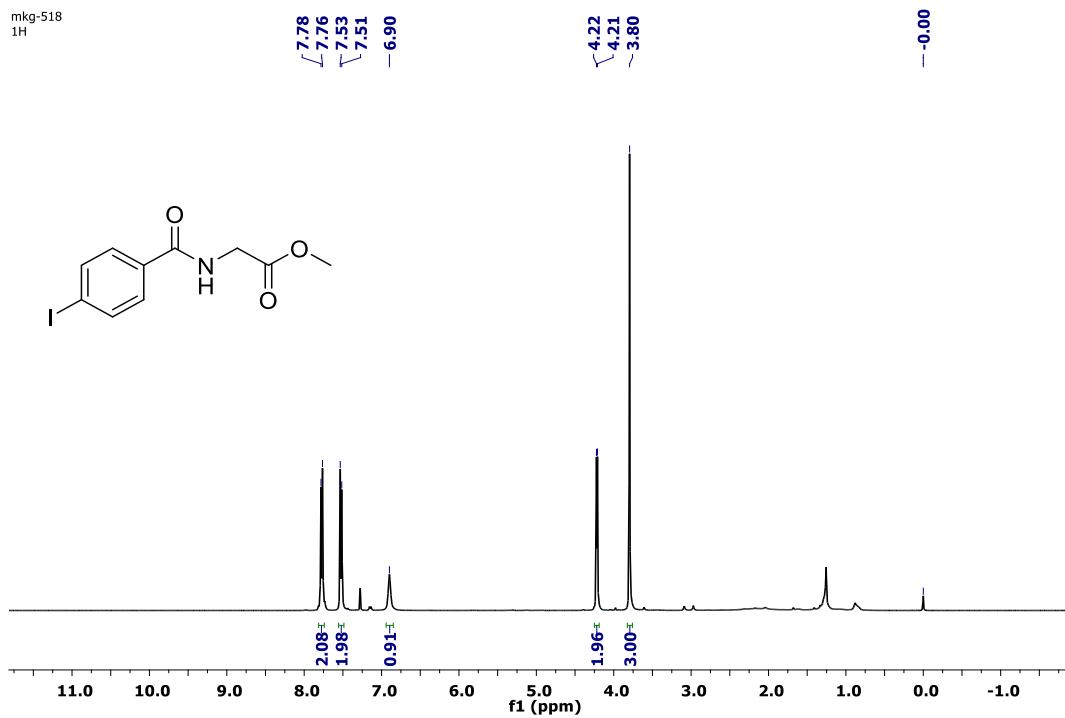


Figure. S56. ESI-HRMS spectra of benzamide **3k**

mkg-518
1H



mkg-518
13C

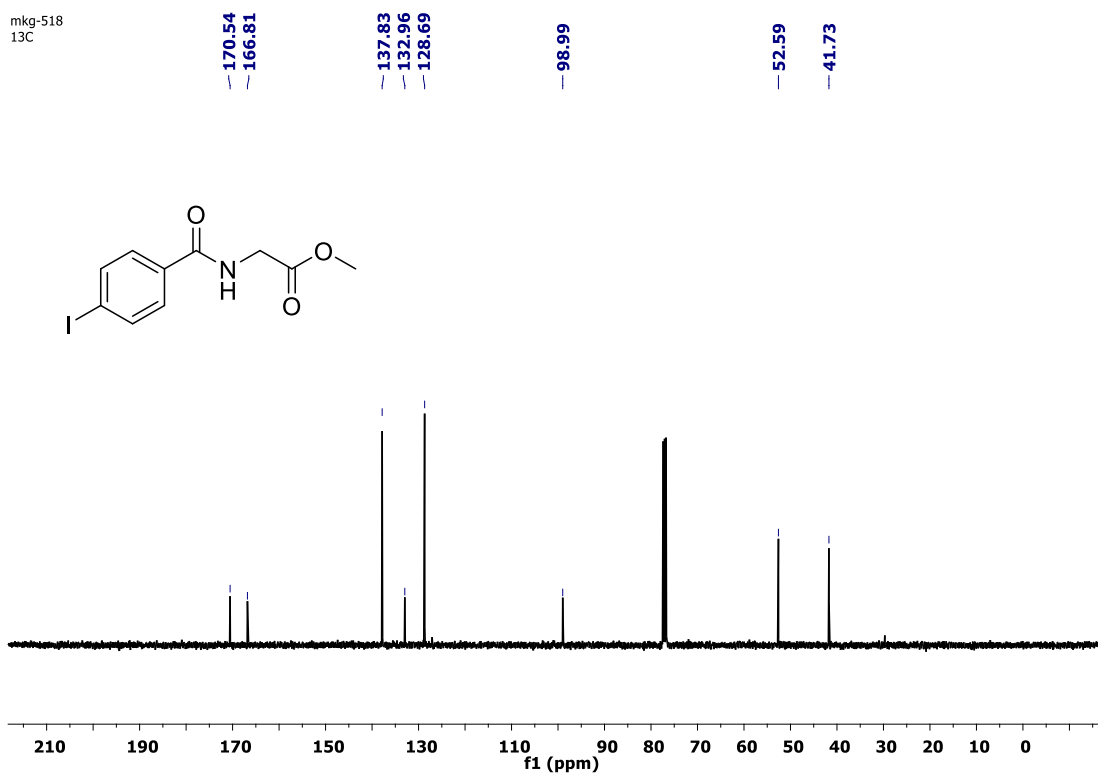


Figure. S57. ¹H, ¹³C NMR spectra of benzamide 31

Display Report

Analysis Info

Analysis Name D:\Data\JULY-2021\NKS\MKG\13072021_NKS-MKG-518.d
Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/13/2021 6:03:15 PM

Operator PRAKASH BEHERA
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

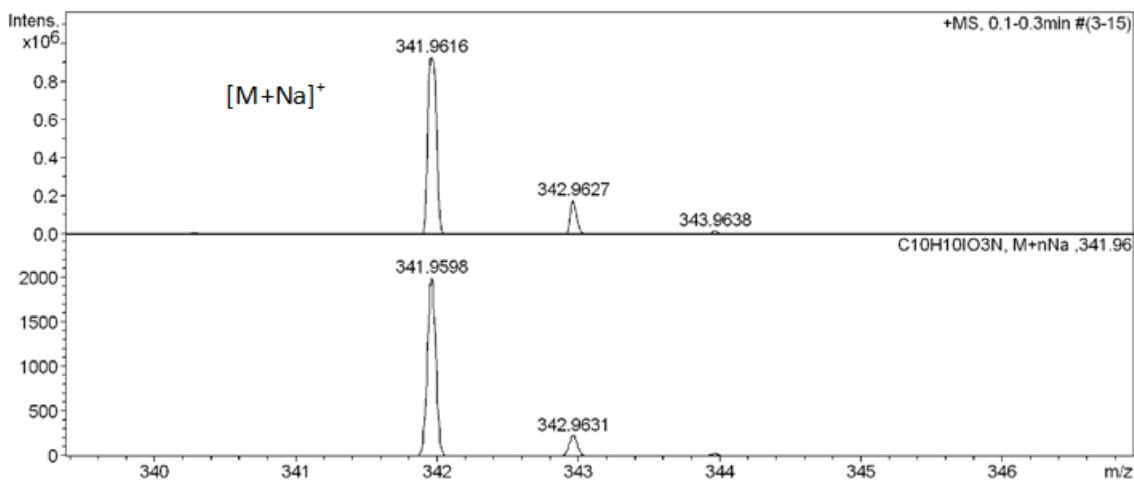
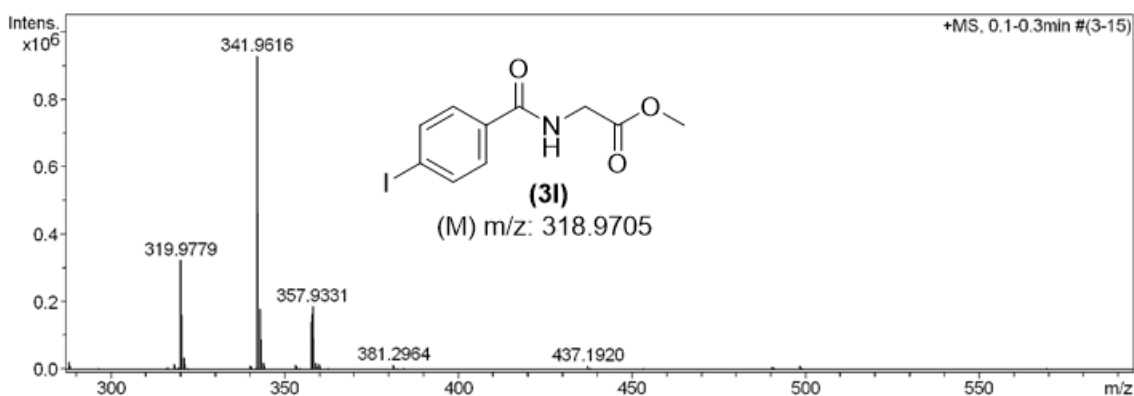
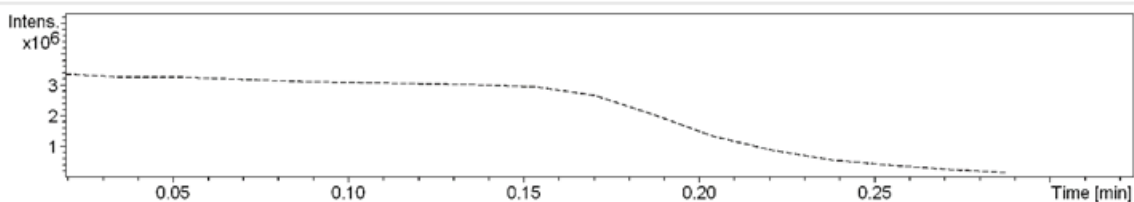
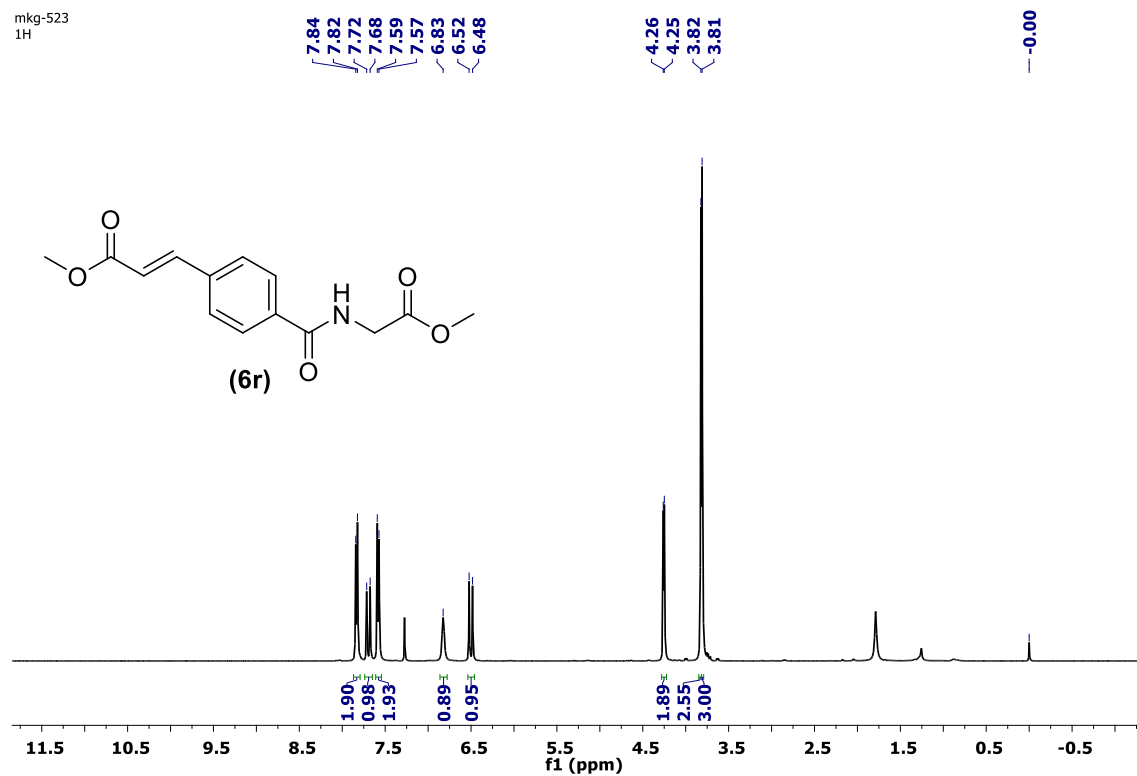


Figure. S58. ESI-HRMS spectra of benzamide 31

mkg-523
1H



mkg-523
13C

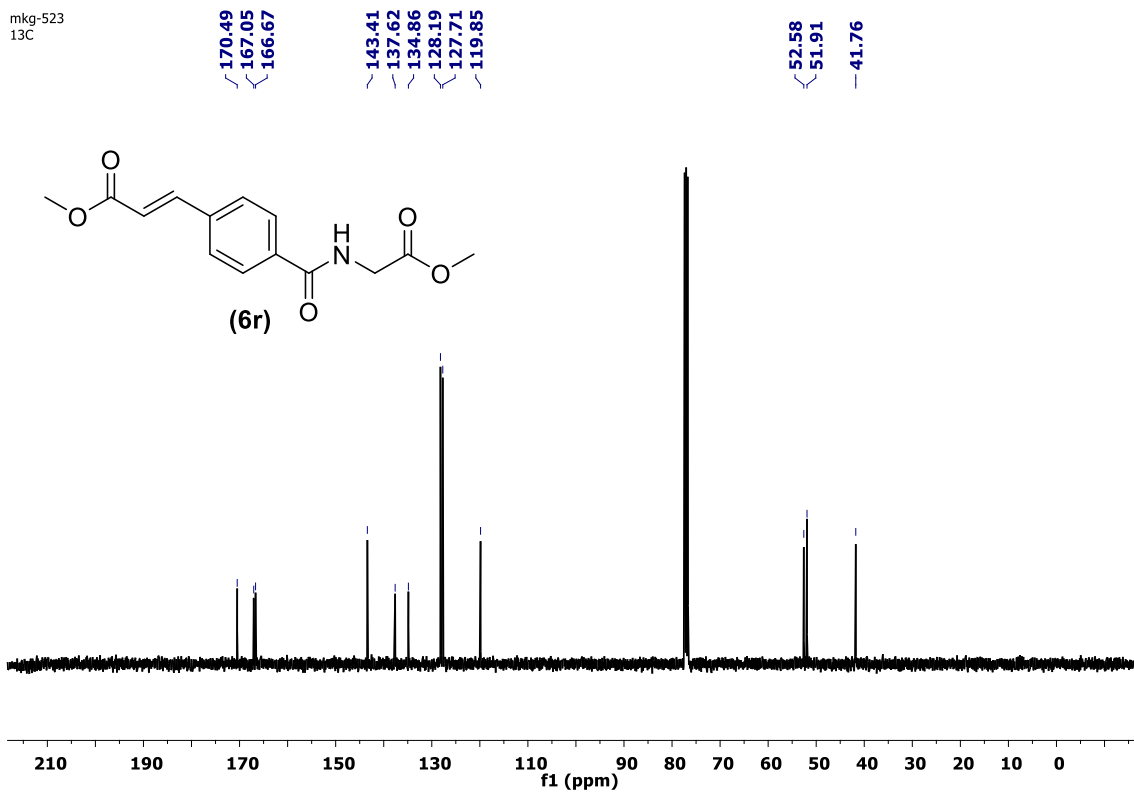


Figure. S59. ¹H, ¹³C NMR spectra of benzamide **6r**

Display Report

Analysis Info

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Method Pos_tune_low.m
Sample Name Tmix-131118
Comment

Acquisition Date 7/13/2021 6:19:38 PM

Operator PRAKASH BEHERA
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

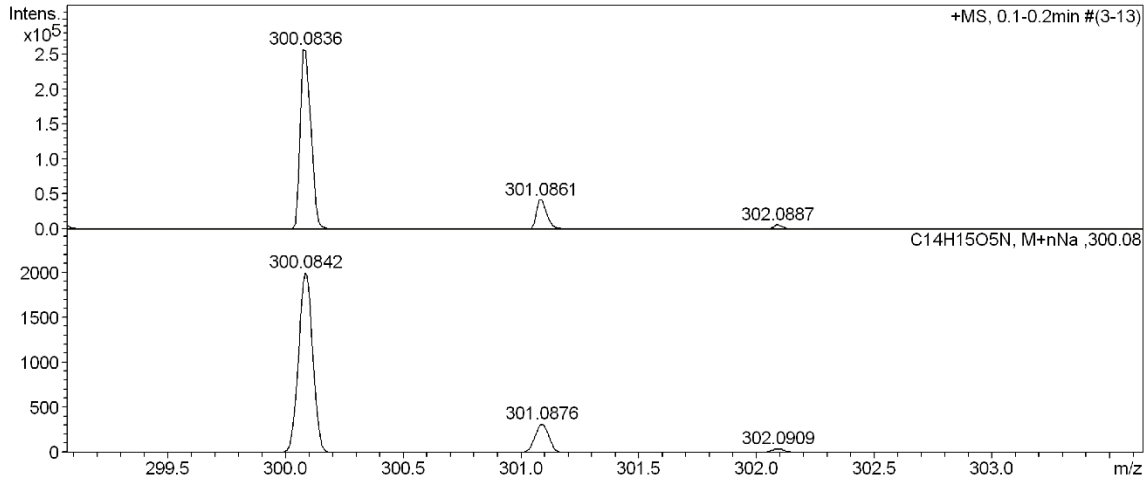
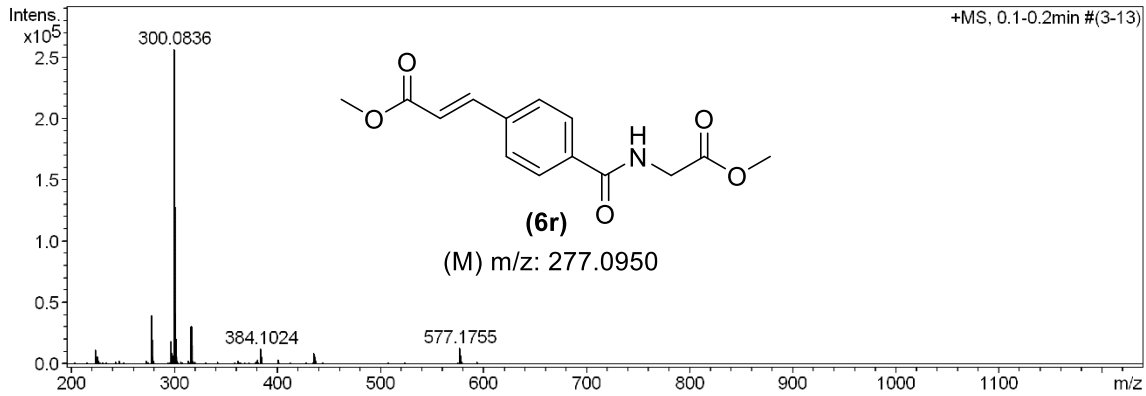
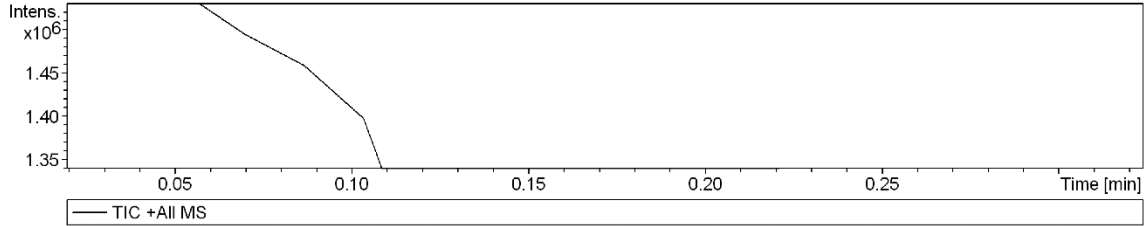


Figure. S60. ESI-HRMS spectra of benzamide 6r

2. X-ray studies of single crystal of indolinone (6a)

Crystal of indolinone(6a) was obtained in solvent mixture ethylacetate and hexane by slow evaporation method. The crystal data was collected on a Rigaku Oxford diffractometer at 293 K. Selected data collection parameters and other crystallographic results are summarized below. The program package SHELXTL1 and Olex2 was used for structure solution.

Table S1. Crystal data and structure refinement for Isoindolinone **6a** (CCDC with reference number 2098113).

Identification code	NKS_MKG_454B
Empirical formula	C ₁₄ H ₁₃ NO ₅
Formula weight	275.25
Temperature/K	301(3)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	11.9576(3)
b/Å	13.9570(3)
c/Å	8.15414(18)
α/°	90
β/°	105.587(2)
γ/°	90
Volume/Å ³	1310.81(5)
Z	4
ρ _{calc} /g/cm ³	1.395
μ/mm ⁻¹	0.903
F(000)	576.0
Crystal size/mm ³	0.02 × 0.02 × 0.001
Radiation	CuKα (λ = 1.54184)
2θ range for data collection/°	7.676 to 150.506
Index ranges	-15 ≤ h ≤ 15, -17 ≤ k ≤ 17, -6 ≤ l ≤ 9
Reflections collected	10426
Independent reflections	2602 [R _{int} = 0.0328, R _{sigma} = 0.0268]
Data/restraints/parameters	2602/0/184
Goodness-of-fit on F ²	1.080
Final R indexes [I >= 2σ (I)]	R ₁ = 0.0386, wR ₂ = 0.1123
Final R indexes [all data]	R ₁ = 0.0420, wR ₂ = 0.1155
Largest diff. peak/hole / e Å ⁻³	0.23/-0.15

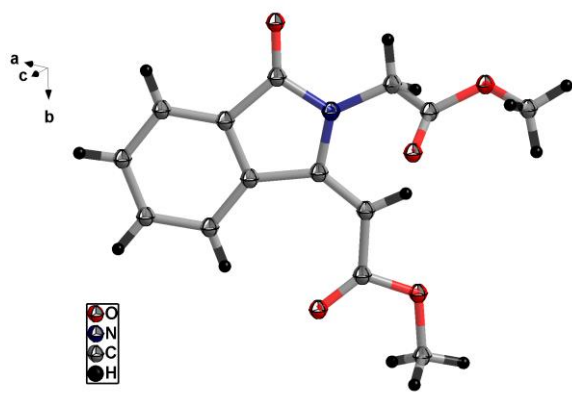


Figure. S61. ORTEP Diagram of indolinone(**6a**) [ellipsoid contour probability: 50%].

