

Electronic Supporting Information (ESI)

Crystal Structure of 1-(2,4,6-Trichlorobenzoyloxy) Benzotriazole (TCB-OBt): Observation of uncommon intermolecular oxygen-oxygen interaction and synthetic application in amidation

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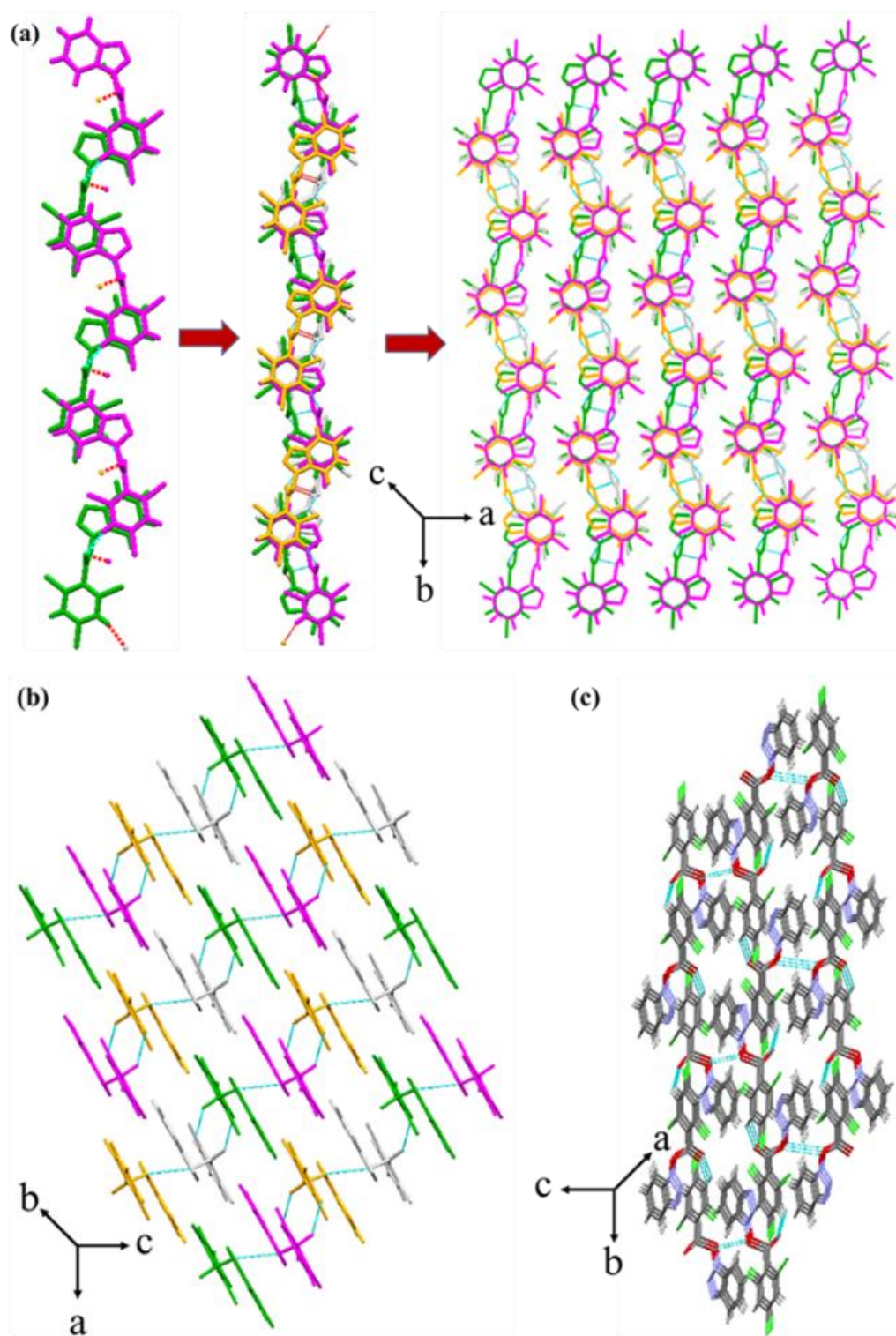


Figure. S1 (a) Supramolecular helical architecture formation through non-covalent π - π stacking interaction, oxygen-oxygen interaction, and aromatic C-H...O interaction, (b) supramolecular packing like zigzag manner along c-axis, (c) grid-like supramolecular architecture along b-axis in higher-order self-assembly.

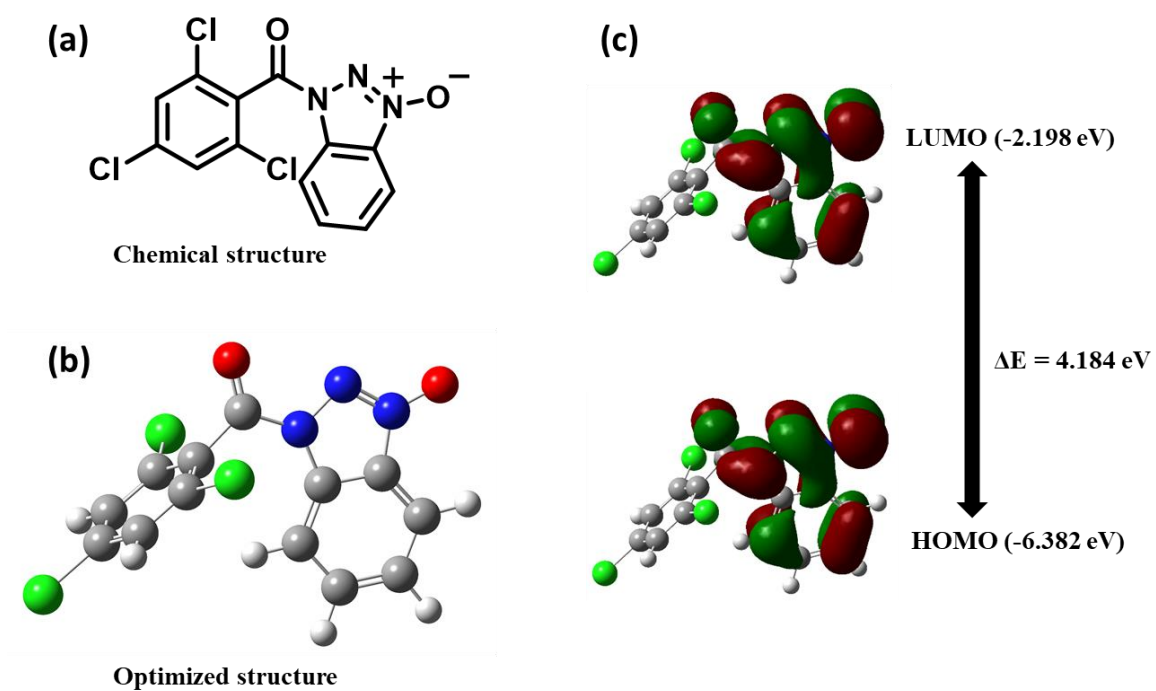


Figure. S2 (a) Chemical structure of TCB-(N)-OBt, (b) Optimized structure TCB-(N)-OBt, (c) HOMO and LUMO molecular orbitals and energy gap of the molecule TCB-OBt, respectively.

Table S1. Crystallographic refinement details for TCB-OBt

Parameters	TCB-OBt
Formula	C ₁₃ H ₆ Cl ₃ N ₃ O ₂
Fw	342.56
Crystal system	Monoclinic
Space group	<i>P2(1)/n</i>
a/Å	10.0723(6)
b/Å	13.1809(9)
c/Å	10.5798(8)
α /°	90.00
β /°	90.957(3)
γ /°	90.00
V/Å ³	1404.40(17)
Z	4
D _c /g cm ⁻³	1.620
μ Mo K α /mm ⁻¹	0.658
F000	688
T/K	296(2)
θ max.	25.00
Total no. of reflections	13230
Independent reflections	2452
Observed reflections	2112
Parameters refined	190
R ₁ , I > 2 σ (I)	0.0368
wR ₂ , I > 2 σ (I)	0.1227
GOF (<i>F</i> ²)	0.968
CCDC No.	2102031

2. NMR (^1H and ^{13}C) and Mass spectra of TCBOXY:

GD-SS-TCB-OBT-1-1H
GD-SS-TCB-OBT-1-1H

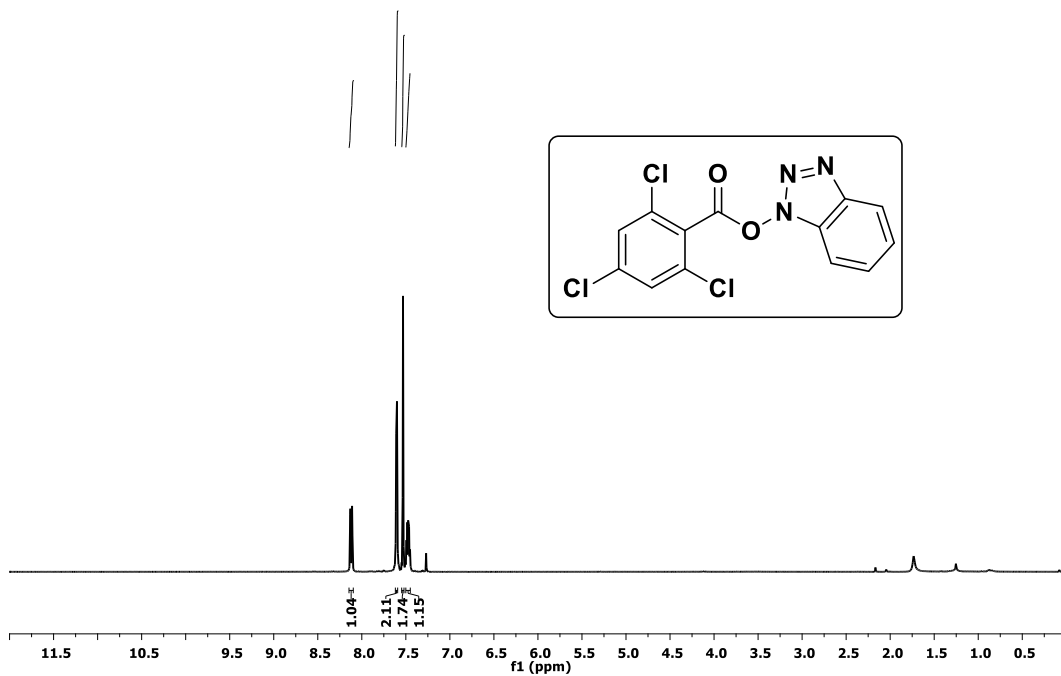


Figure S3: ^1H NMR spectra of TCB-OBt

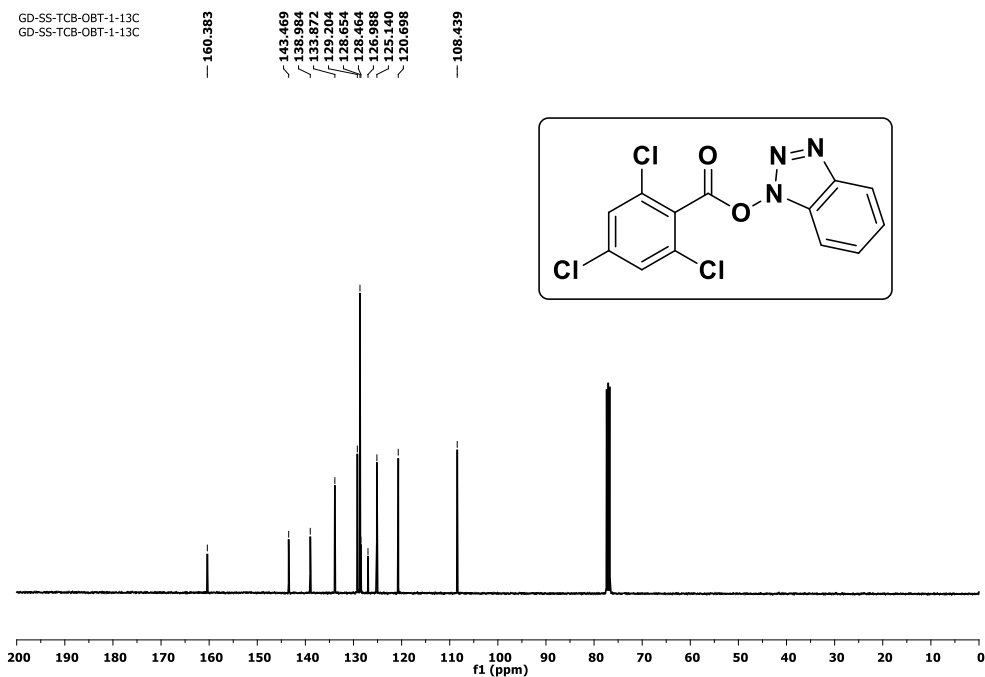


Figure S4: ^{13}C NMR spectra of TCB-OBt

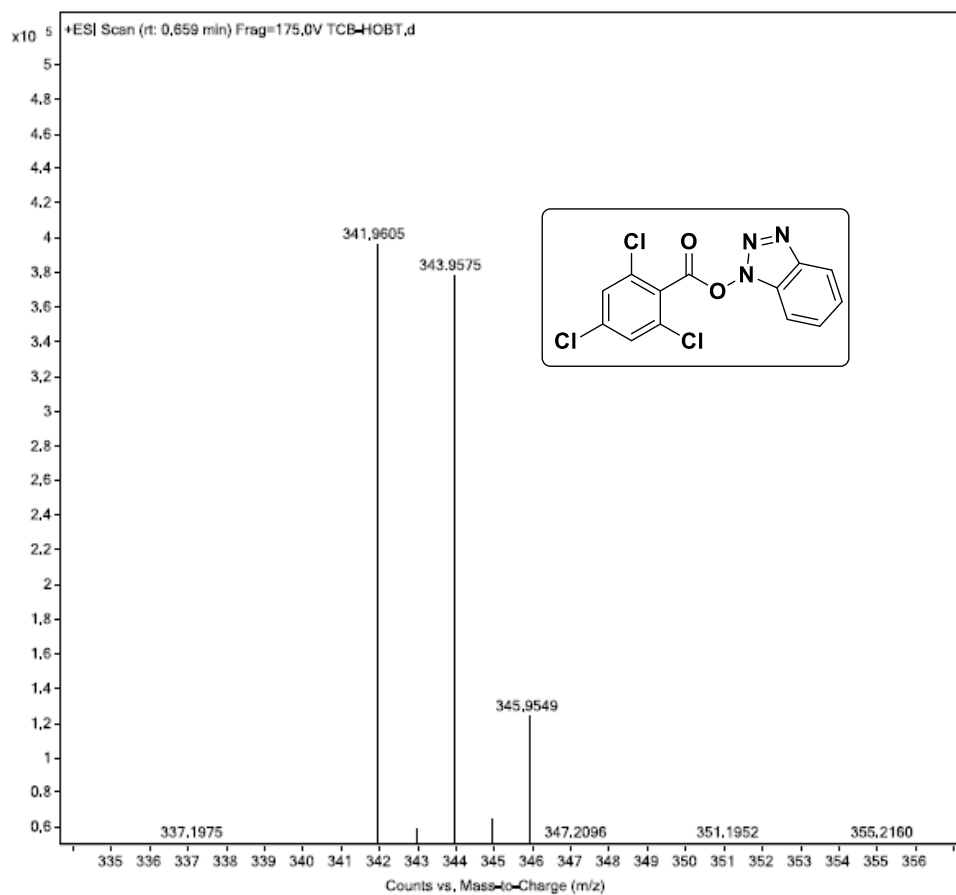


Figure S5: ESI-MASS spectra of spectra of TCB-OBt

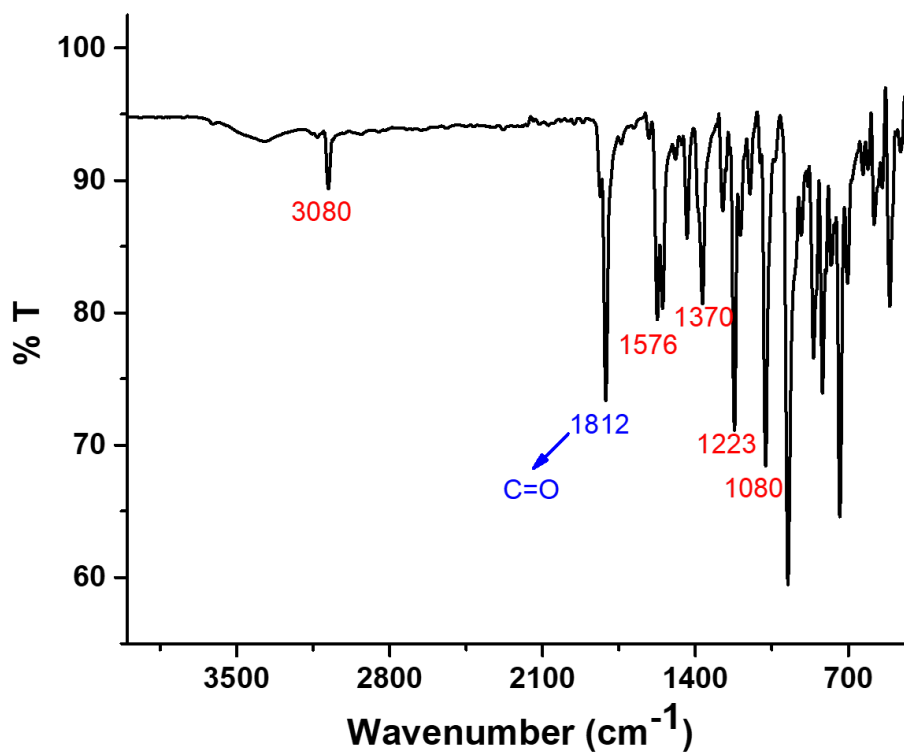


Figure S6: FT-IR spectra of TCB-OBt

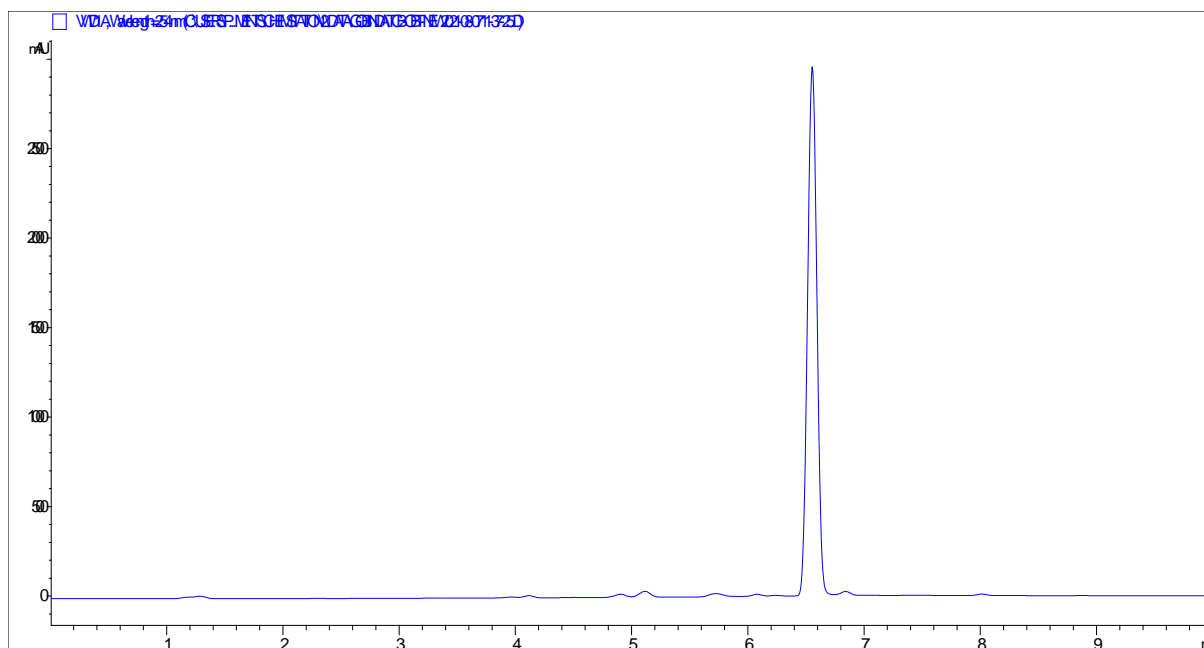


Figure S7: HPLC chromatogram of TCB-OBt, first day of synthesis. (retention time $R_t = 6.554$ min)

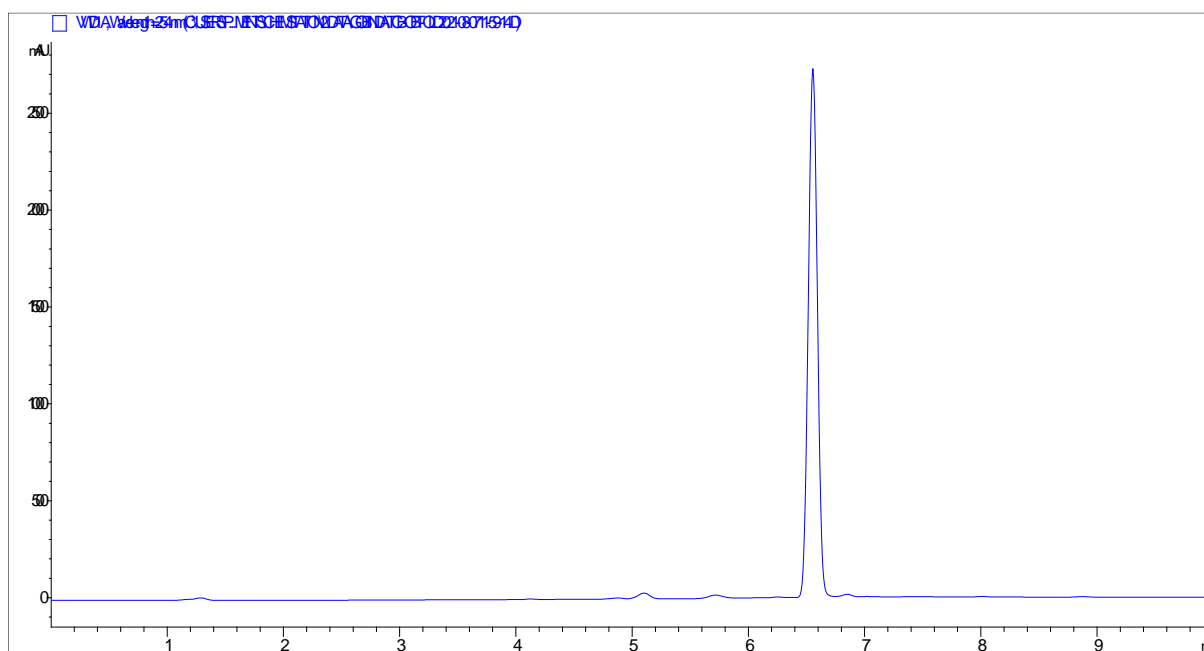


Figure S8: HPLC chromatogram of TCB-OBt, after 6 months of the synthesis. (retention time $R_t = 6.555$ min)

3. NMR (^1H and ^{13}C) and Mass spectra of side product:

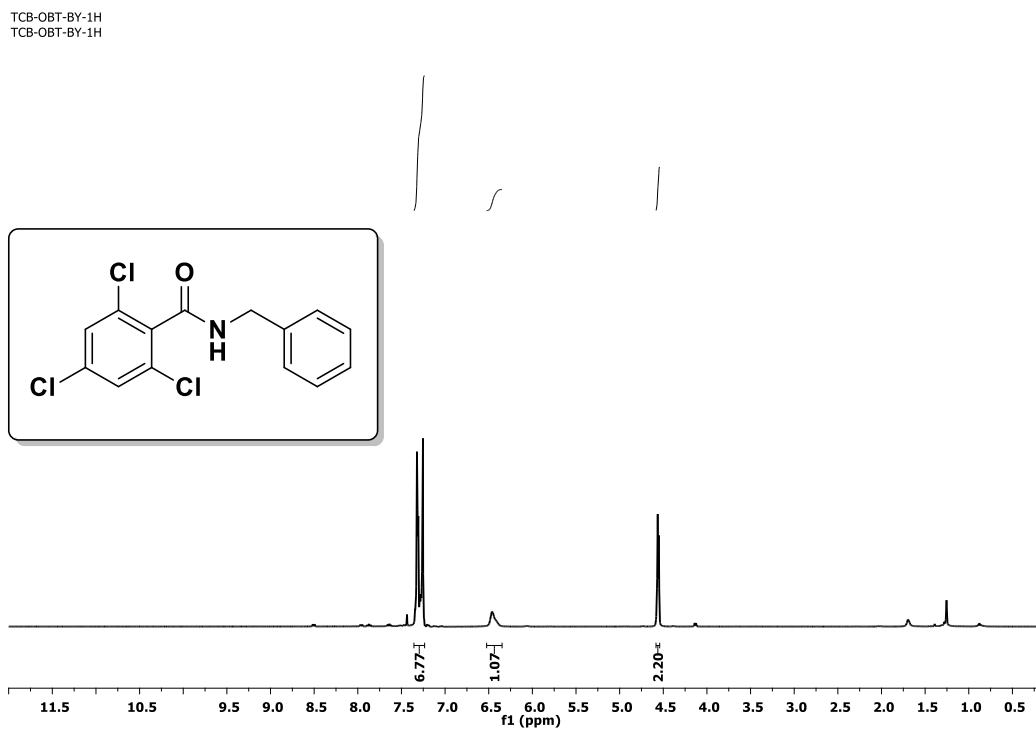


Figure S9: ^1H NMR spectrum of side product

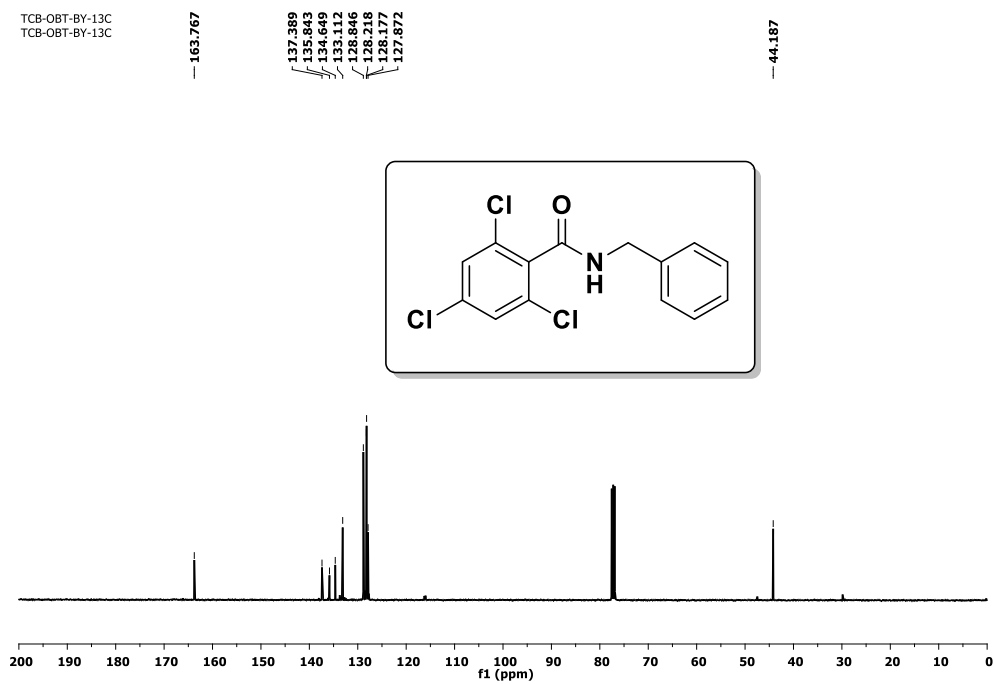


Figure S10: ^{13}C NMR spectrum of side product

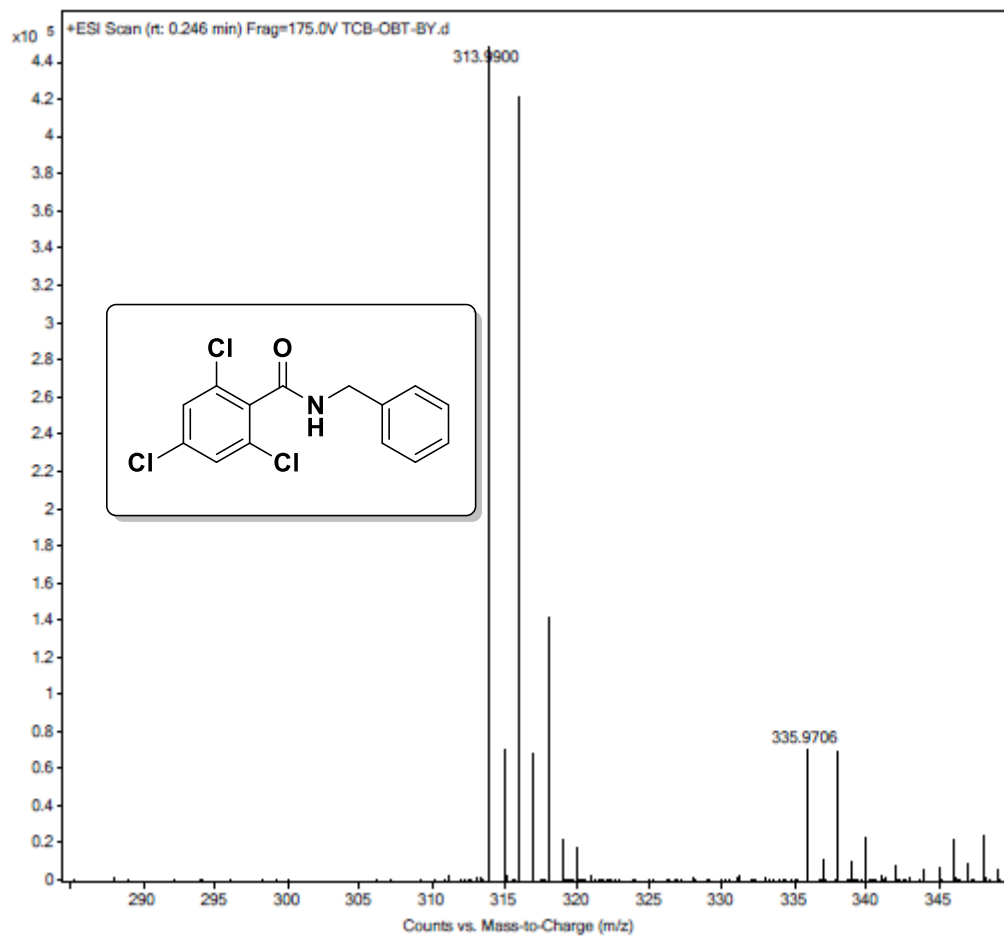


Figure S11: ESI-MASS spectrum of side product

4. NMR (^1H and ^{13}C) and Mass spectra of isolated intermediate:

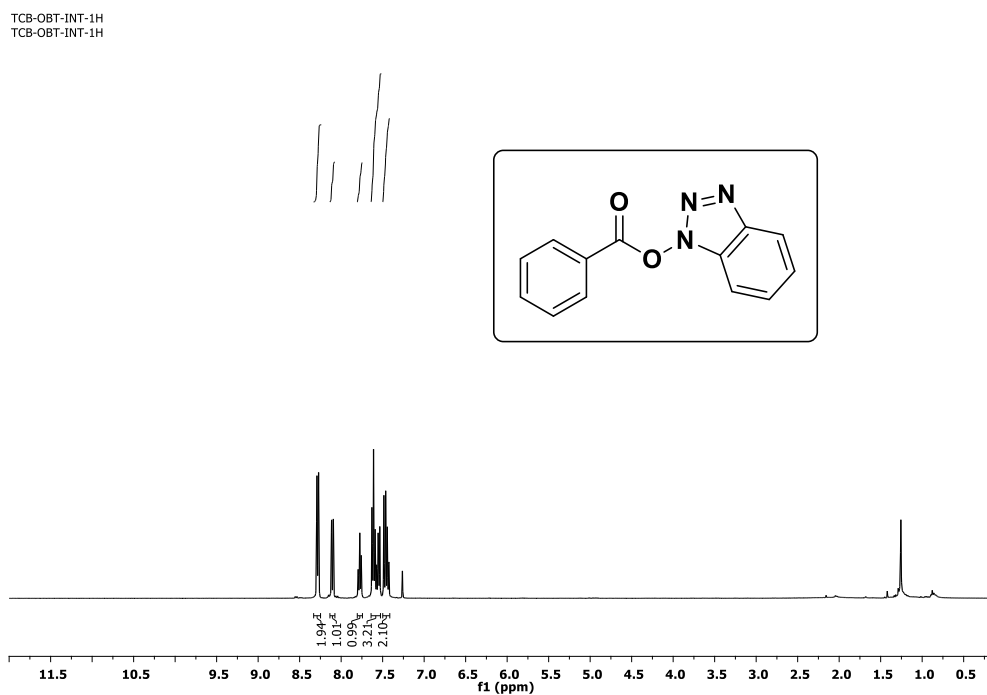


Figure S12: ^1H NMR spectrum of isolated intermediate

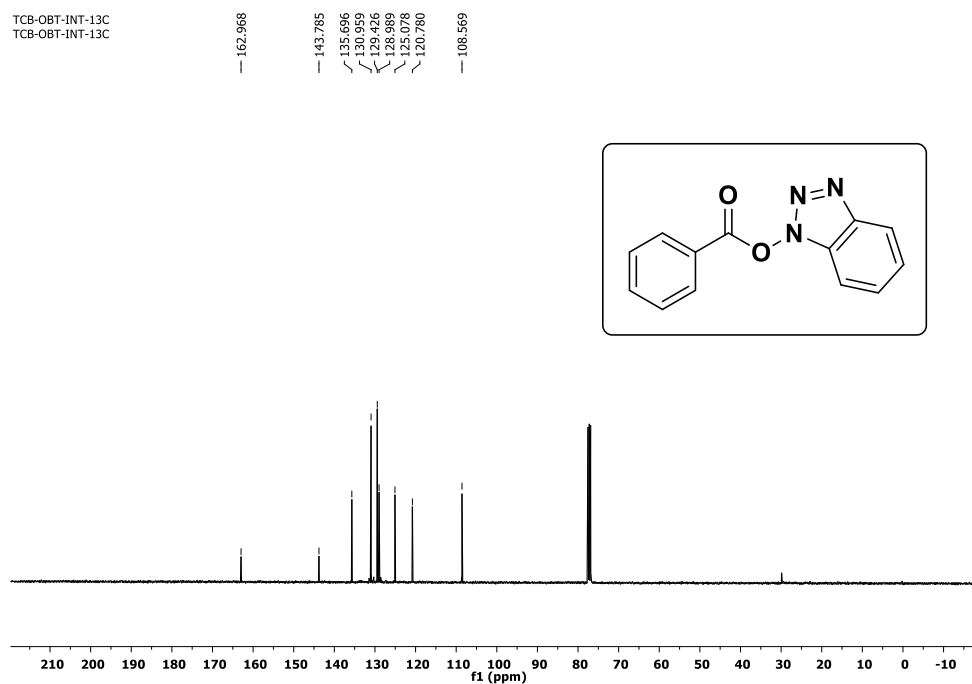


Figure S13: ^{13}C NMR spectrum of isolated intermediate

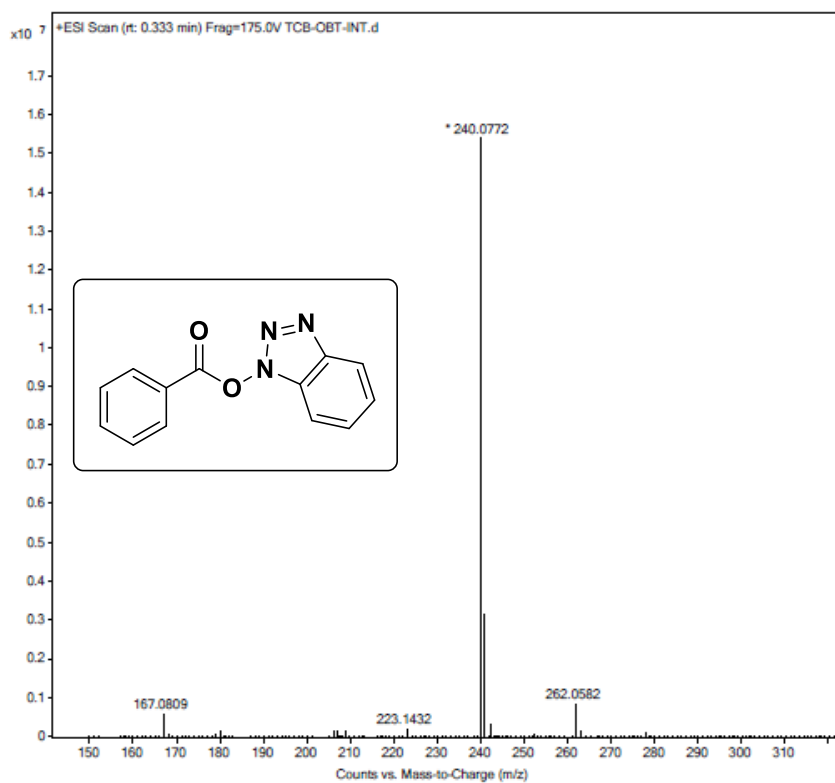


Figure S14: ESI-MASS spectrum of isolated intermediate

4. NMR (¹H and ¹³C) and Mass spectra of amides:

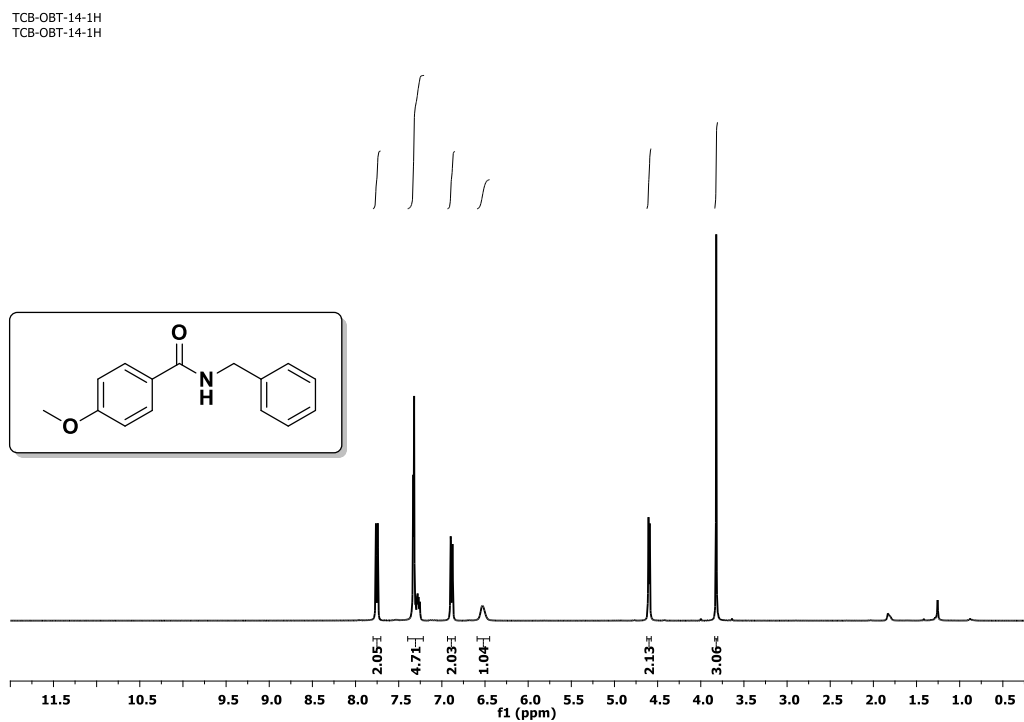


Figure S15: ¹H NMR spectrum of compound 2a

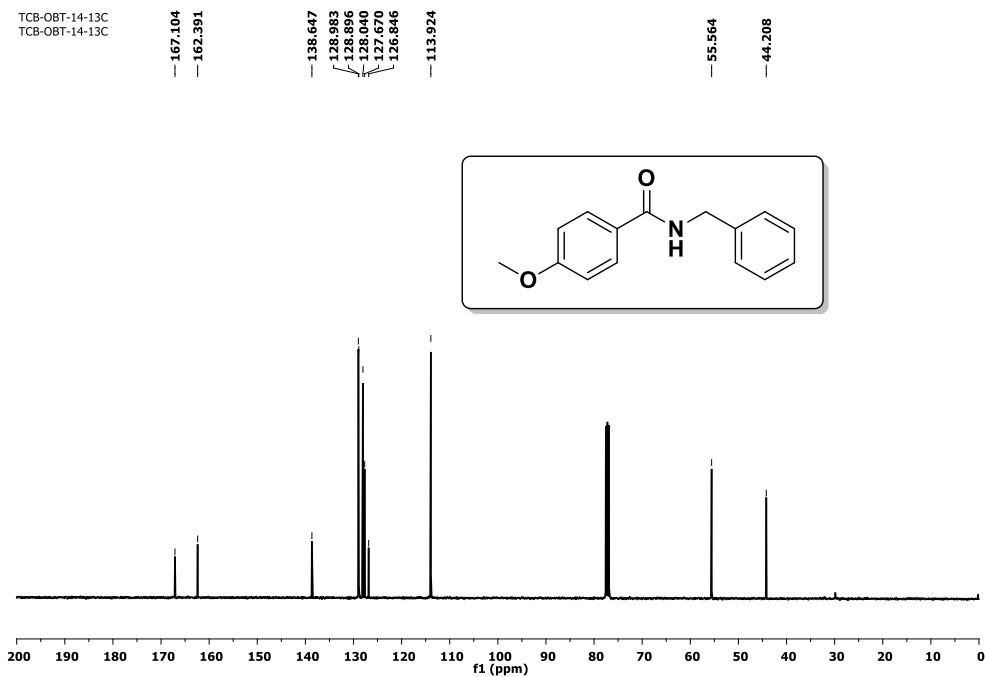


Figure S16: ^{13}C NMR spectrum of compound 2a

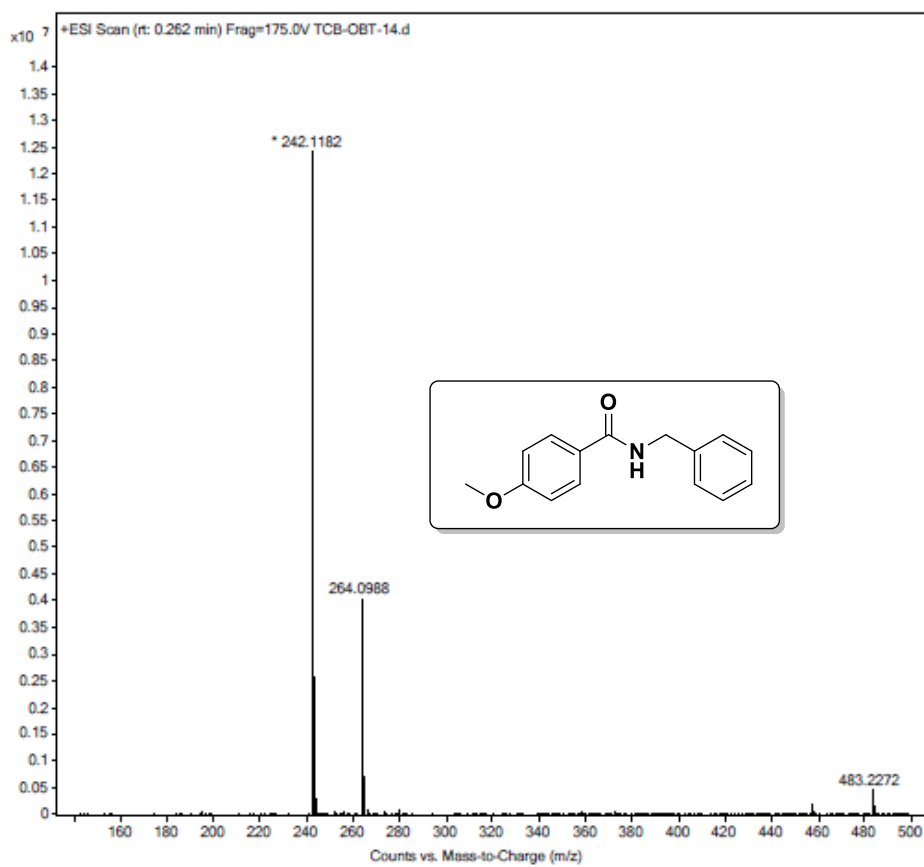


Figure S17: ESI-MASS spectrum of compound 2a

GD-TCB-OBT-3-1H
1H

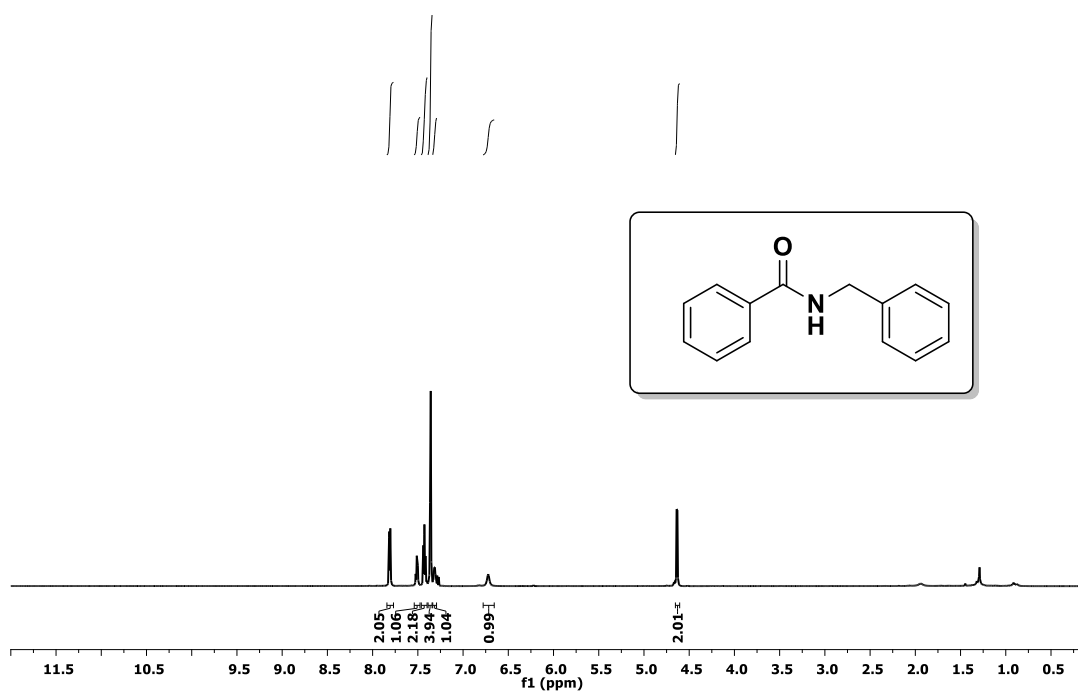


Figure S18: ¹H NMR spectrum of compound 2b

GD-TCB-OBT-3-13C
13C

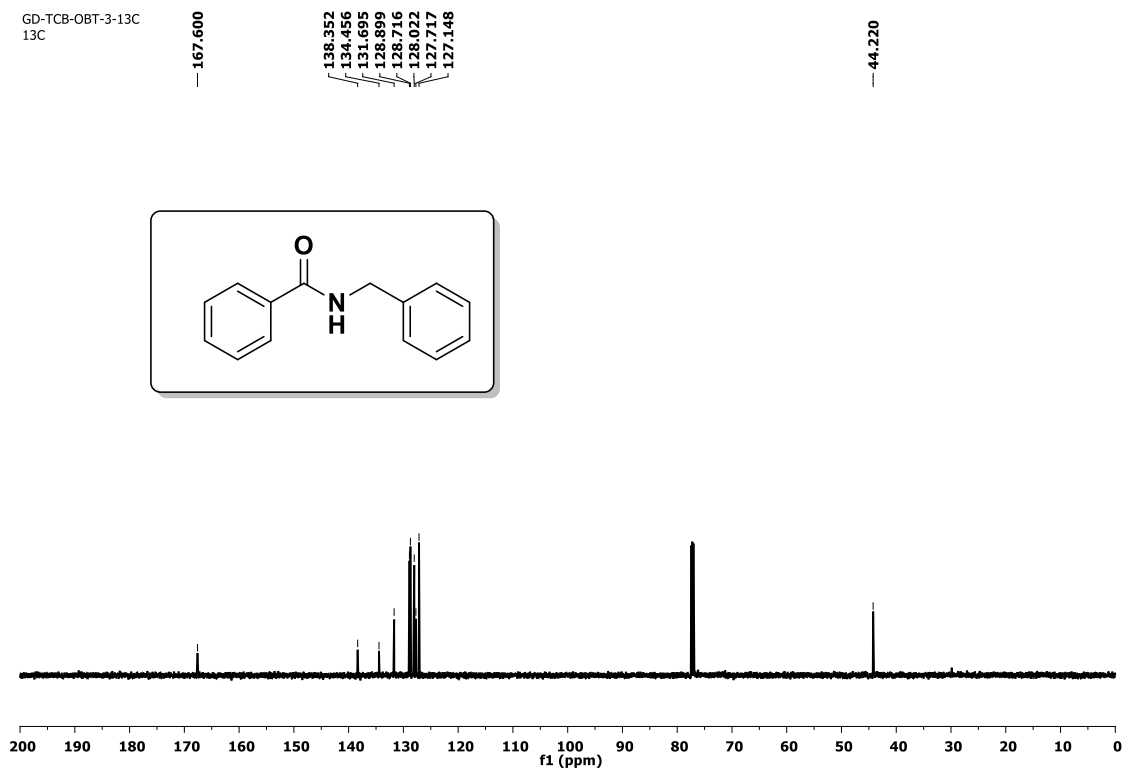


Figure S19: ¹³C NMR spectrum of compound 2b

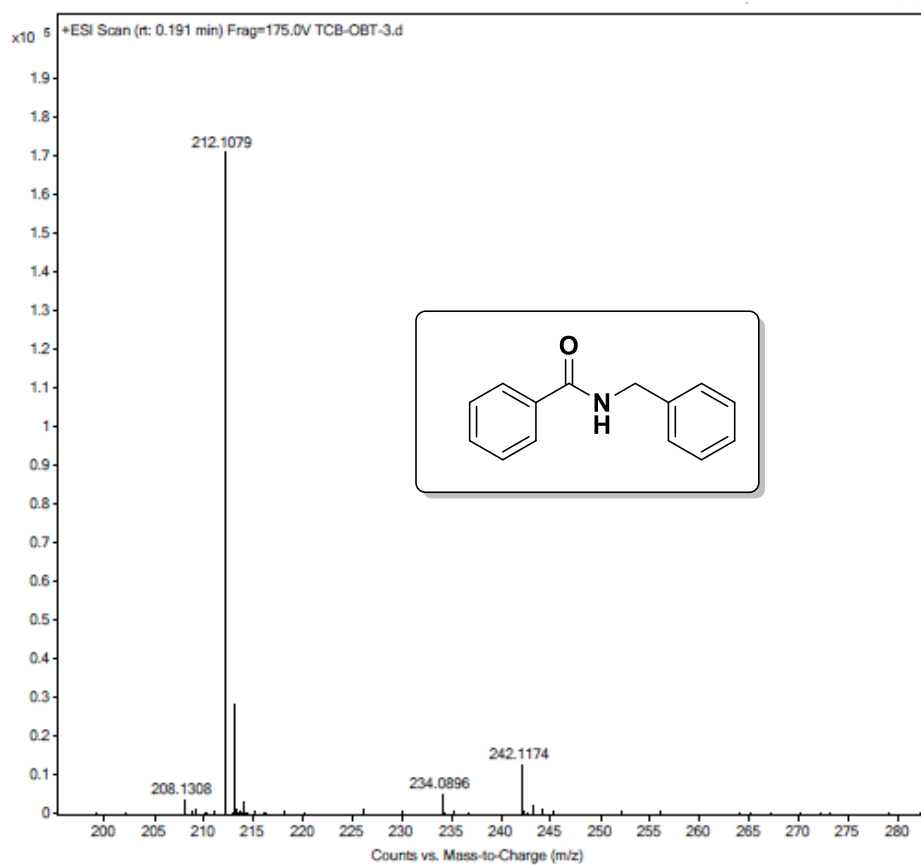


Figure S20: ESI-MASS spectrum of compound **2b**

TCB-OBT-11-1H
TCB-OBT-11-1H

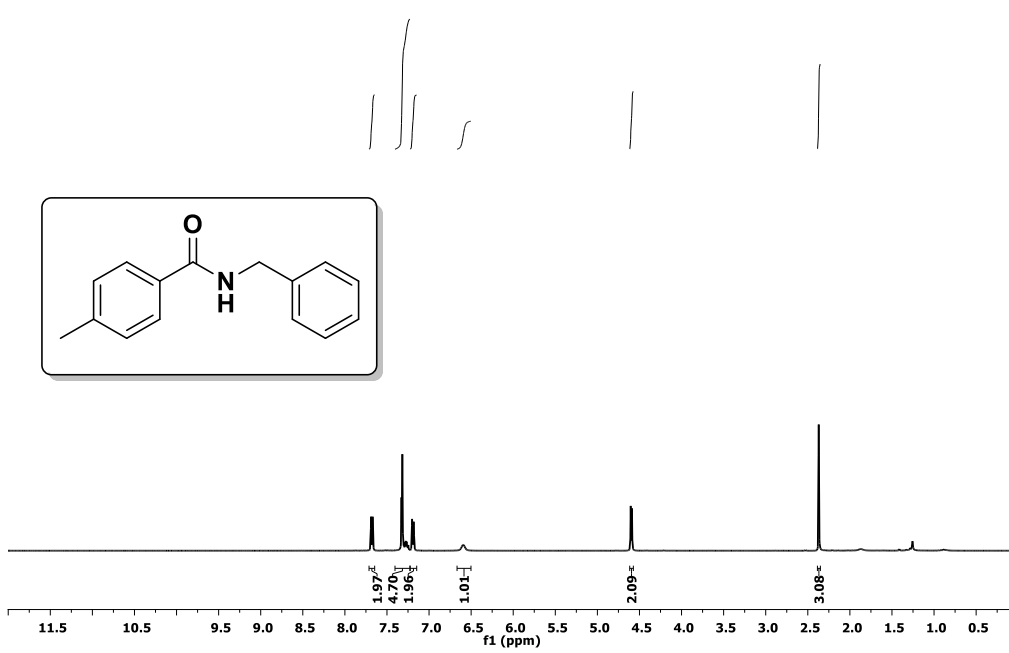


Figure S21: ¹H NMR spectrum of compound **2c**

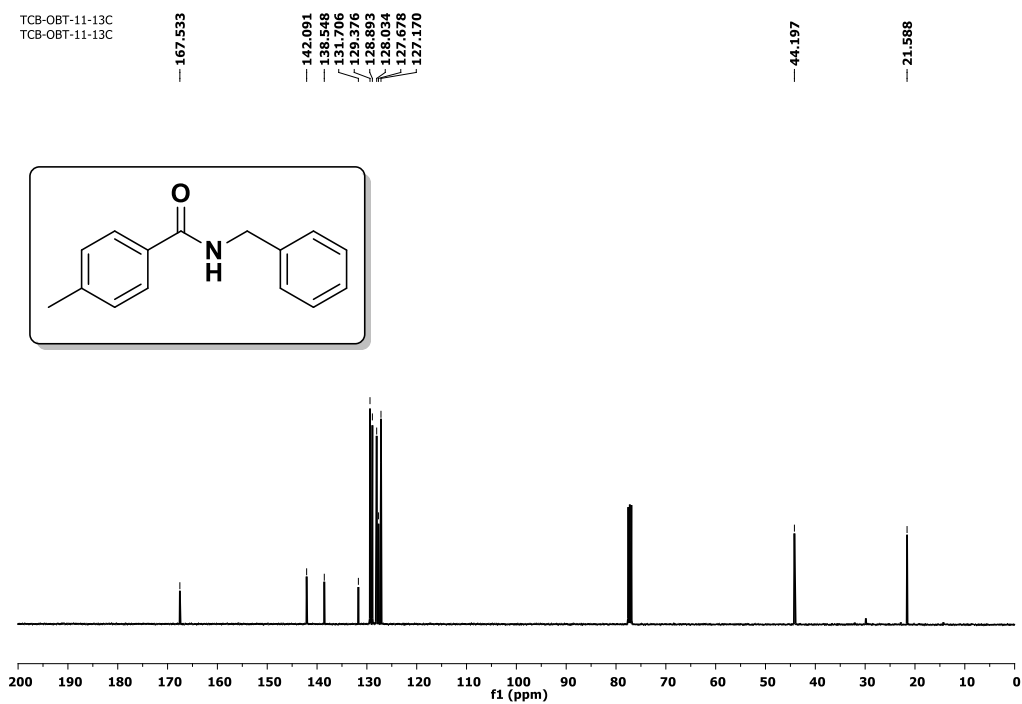


Figure S22: ^{13}C NMR spectrum of compound **2c**

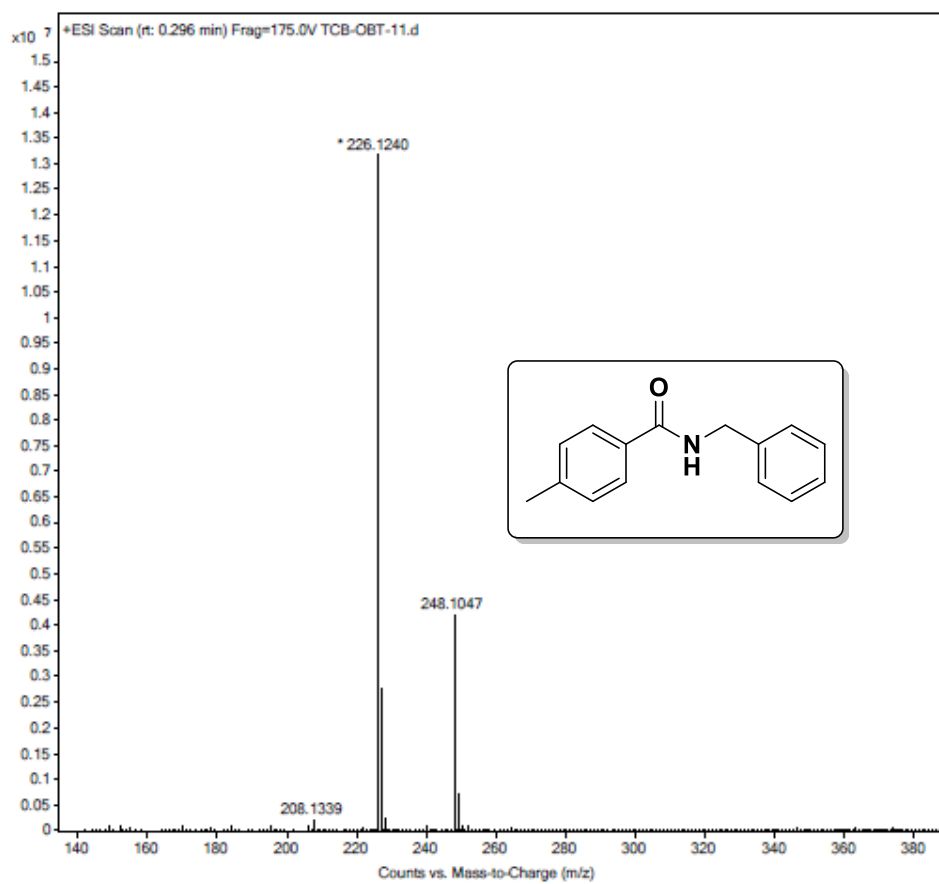


Figure S23: ESI-MASS spectrum of compound **2c**

TCB-OBT-15-H
TCB-OBT-15-H

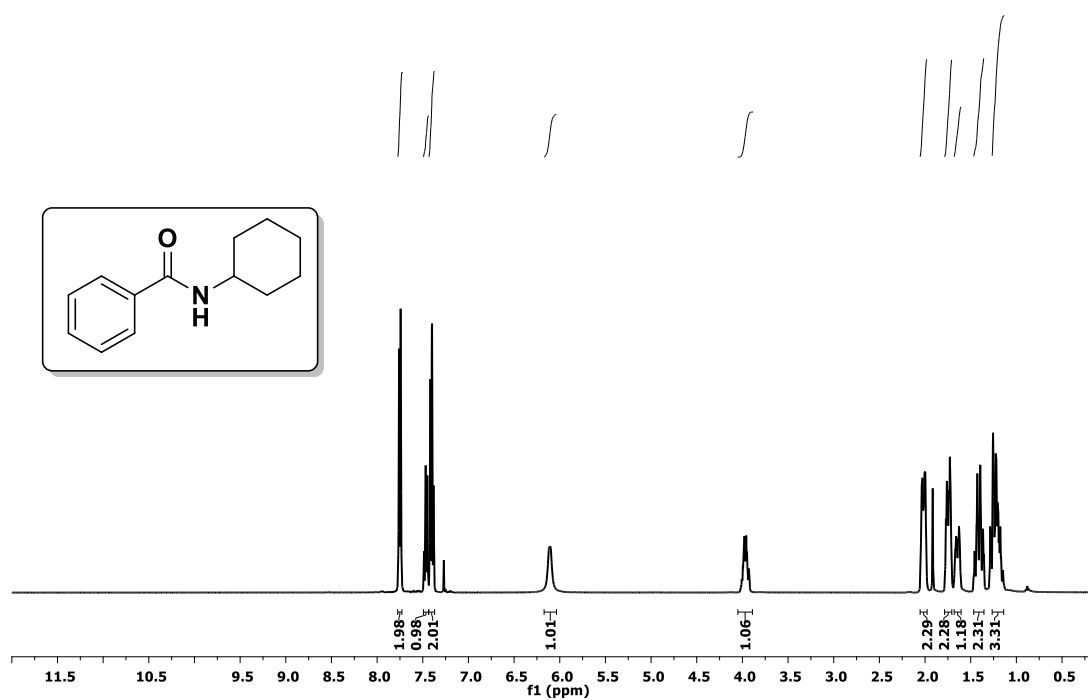


Figure S24: ¹H NMR spectrum of compound 2d

TCB-OBT-15-13C
TCB-OBT-15-13C

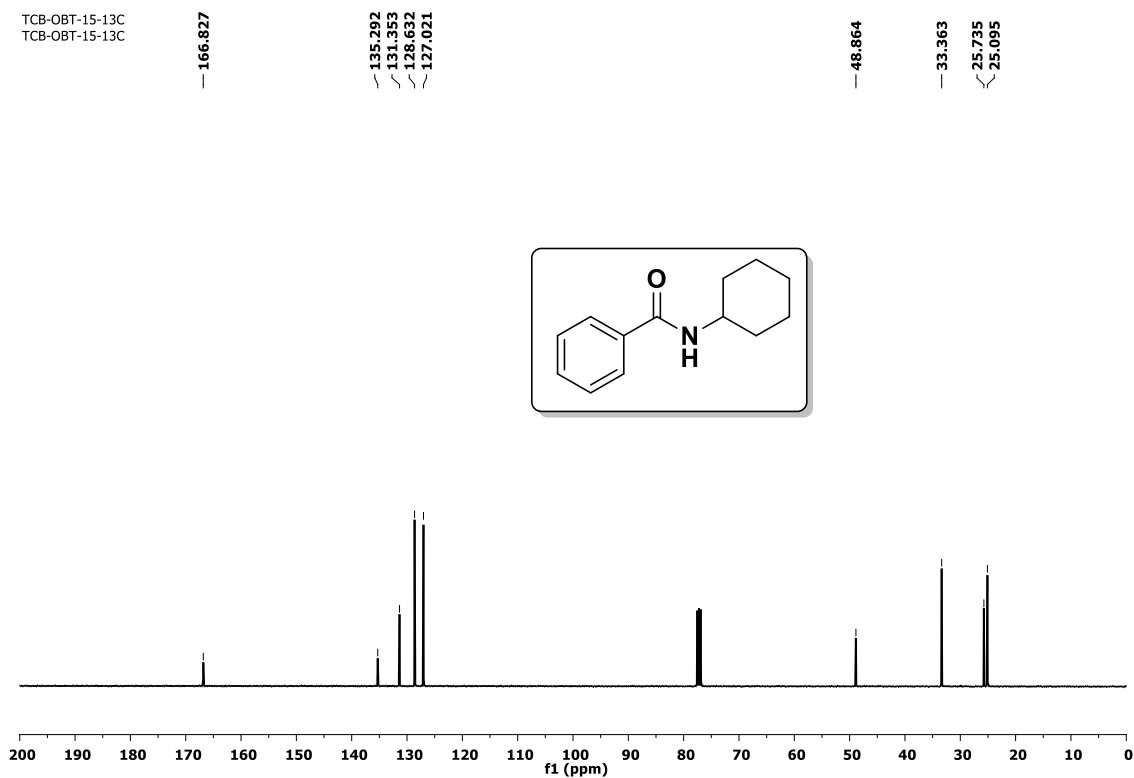


Figure S25: ¹³C NMR spectrum of compound 2d

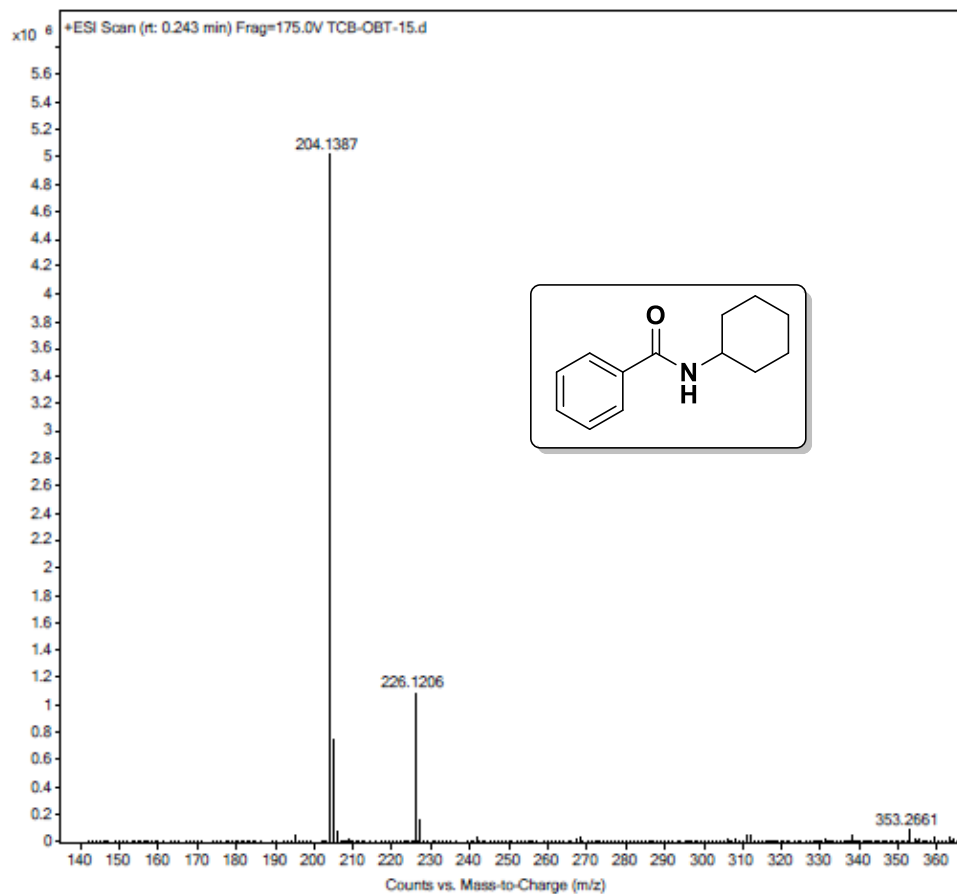


Figure S26: ESI-MASS spectrum of compound 2d

TCB-OBT-12-1H
TCB-OBT-12-1H

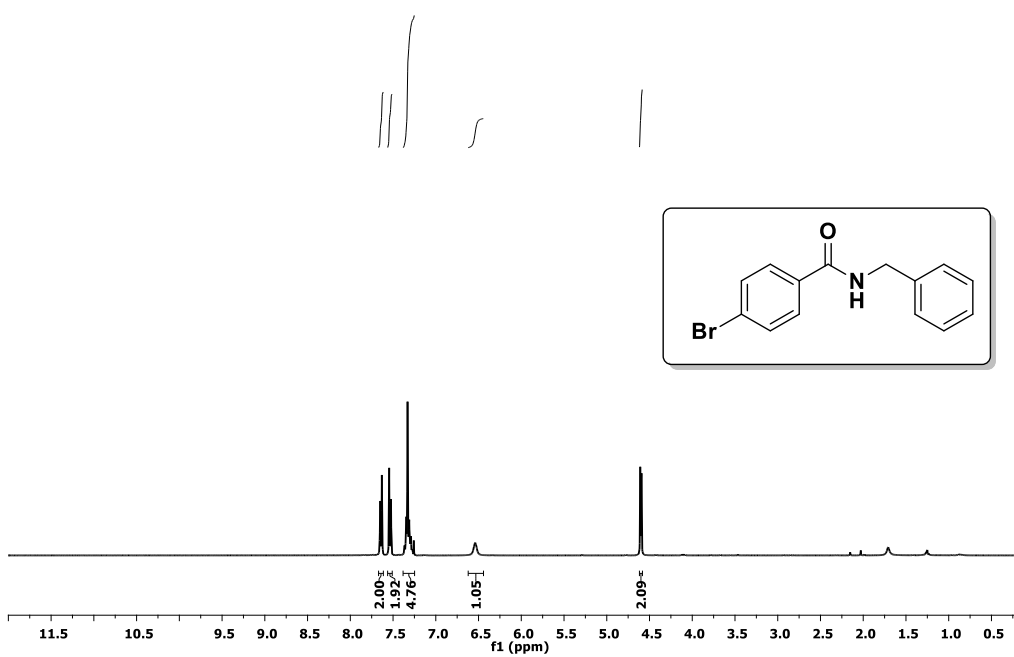


Figure S27: ¹H NMR spectrum of compound 2e

TCB-OBT-12-13C
TCB-OBT-12-13C

— 166.630

138.130
133.394
132.005
129.026
128.810
128.114
127.970
126.432

— 44.418

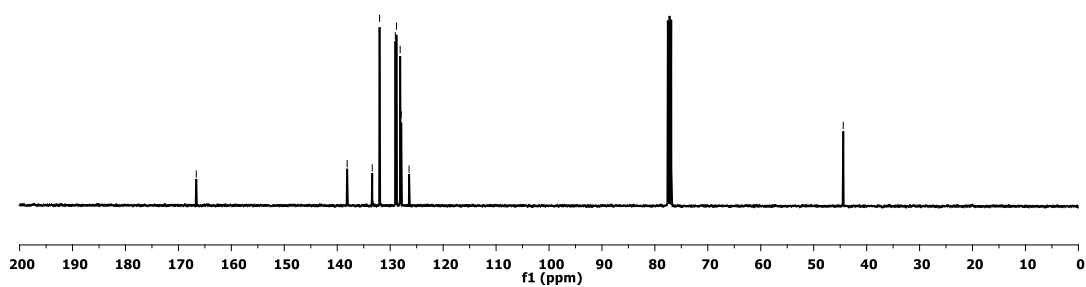
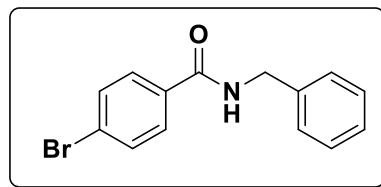


Figure S28: ^{13}C NMR spectrum of compound 2e

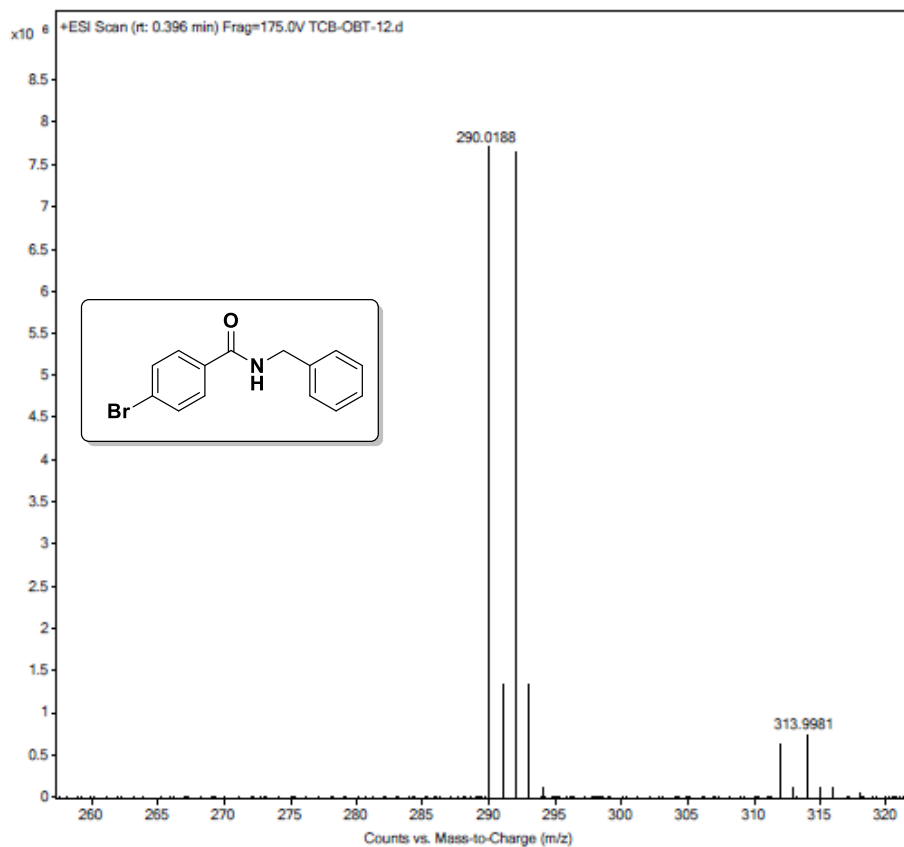


Figure S29: ESI-MASS spectrum of compound 2e

TCB-OBT-16-1H
TCB-OBT-16-1H

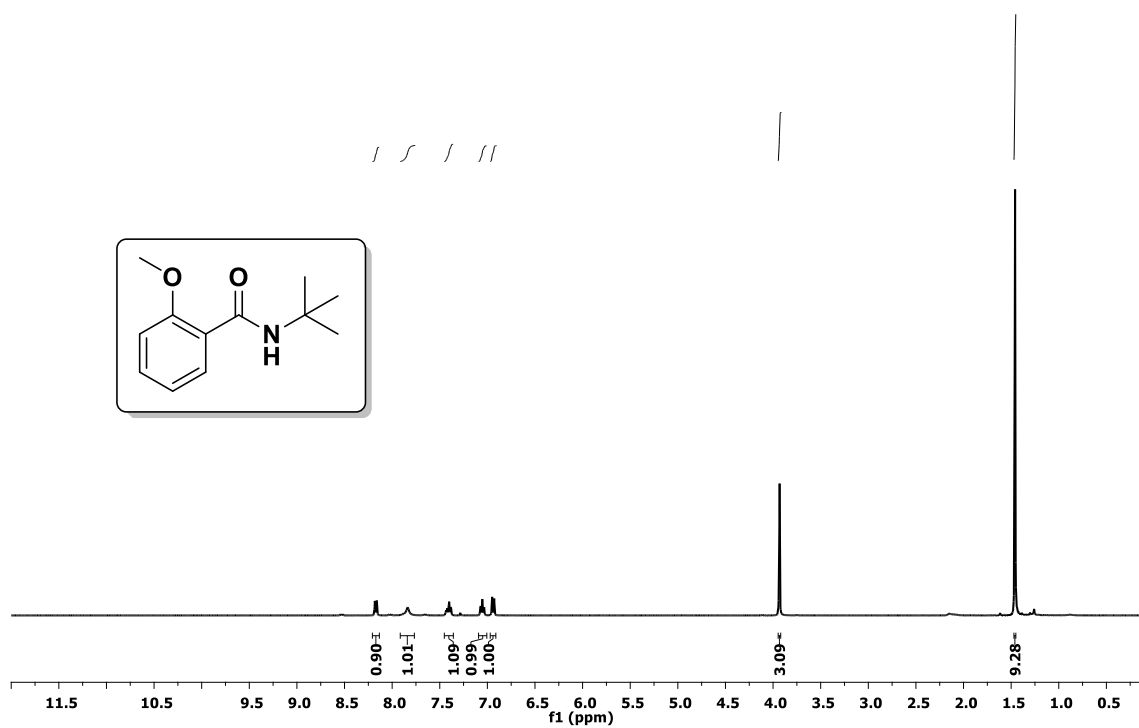


Figure S30: ¹H NMR spectrum of compound 2f

TCB-OBT-16-13C
TCB-OBT-16-13C

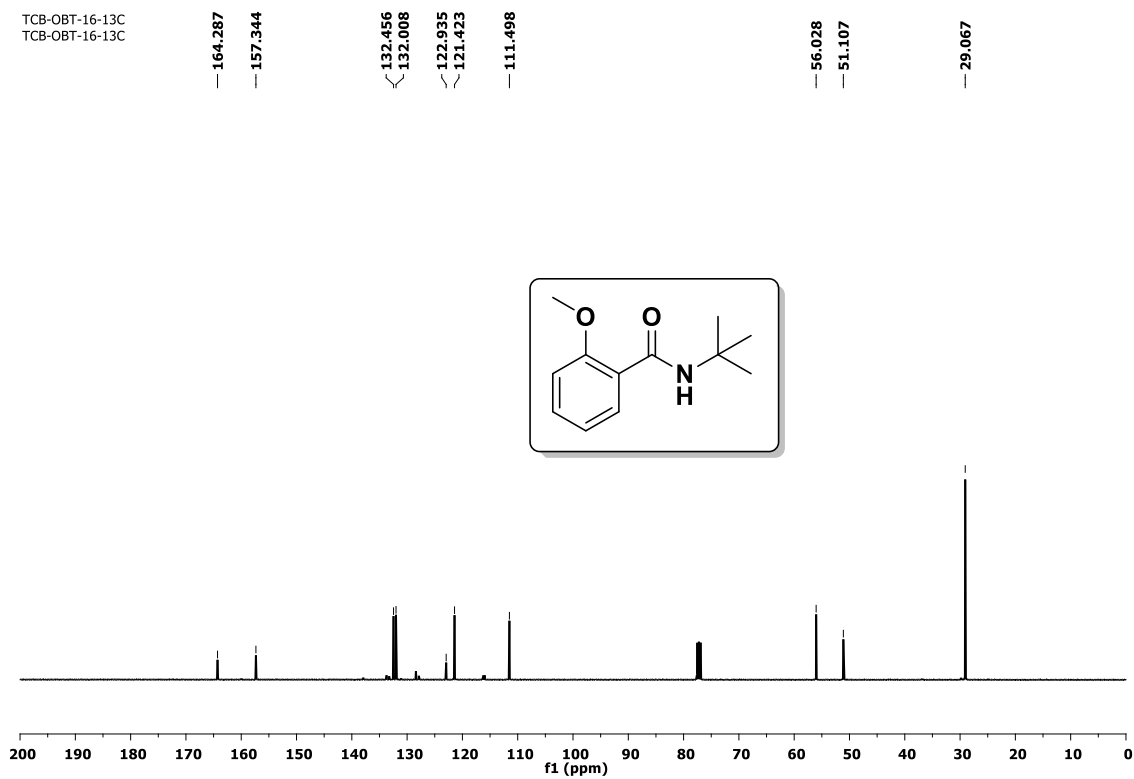


Figure S31: ¹³C NMR spectrum of compound 2f

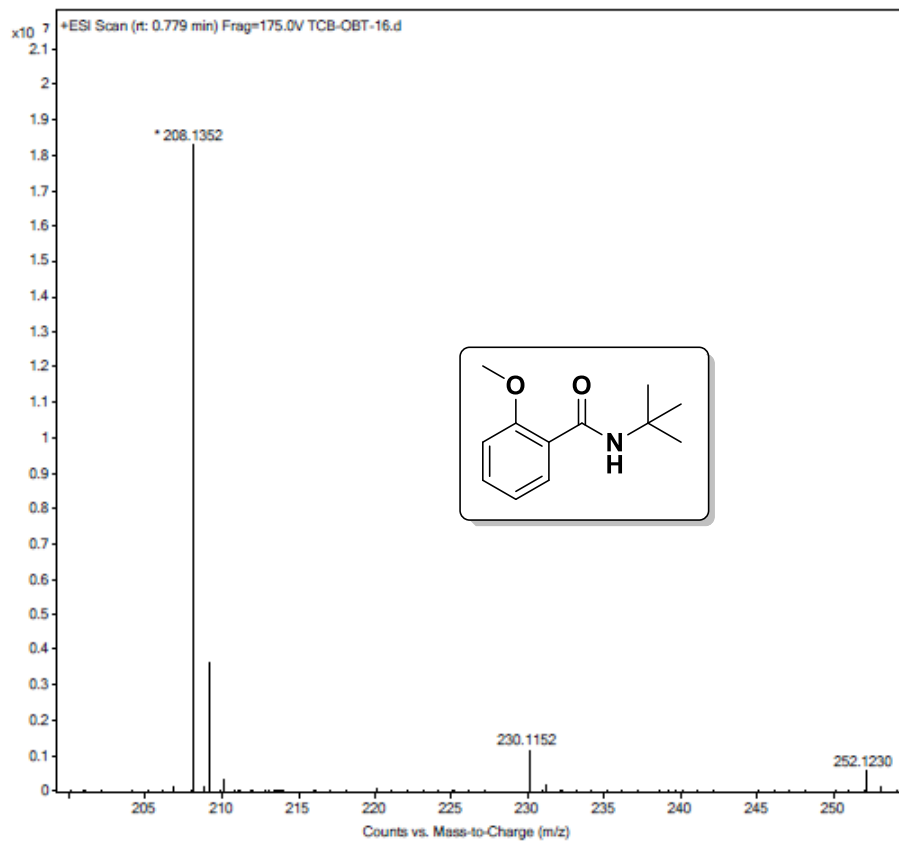


Figure S32: ESI-MASS spectrum of compound 2f

TCB-OBT-17-1H
TCB-OBT-17-1H

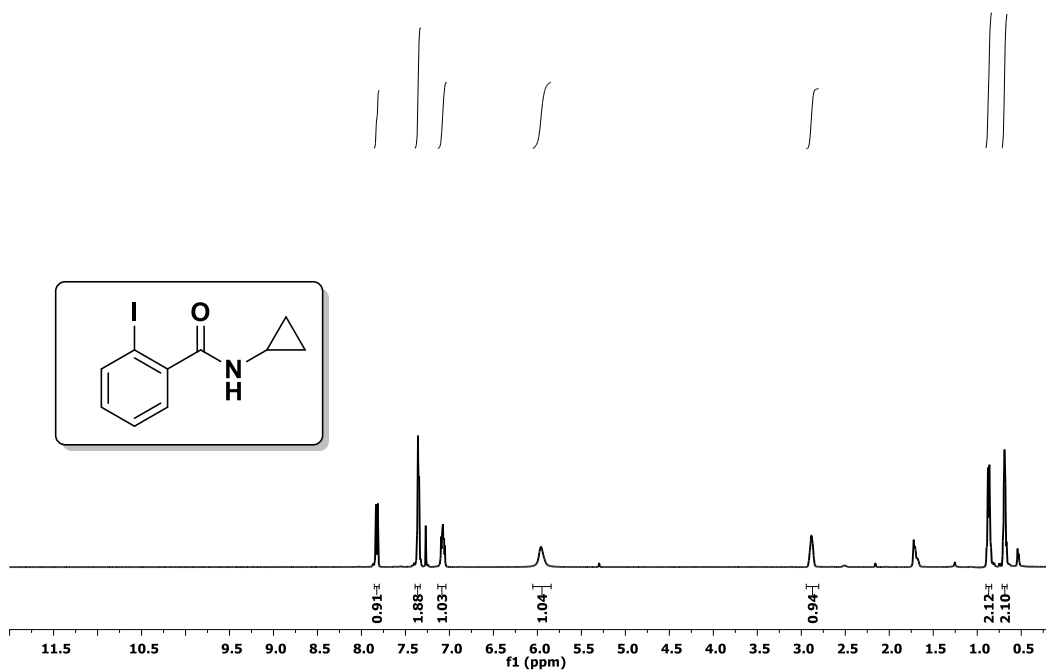
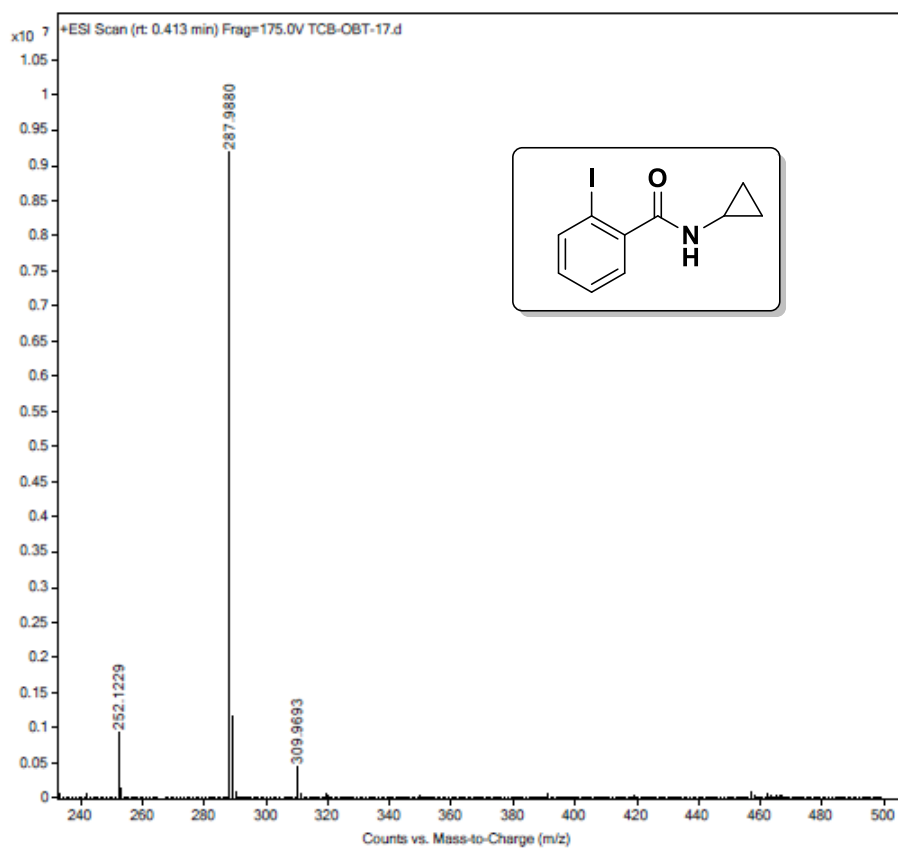
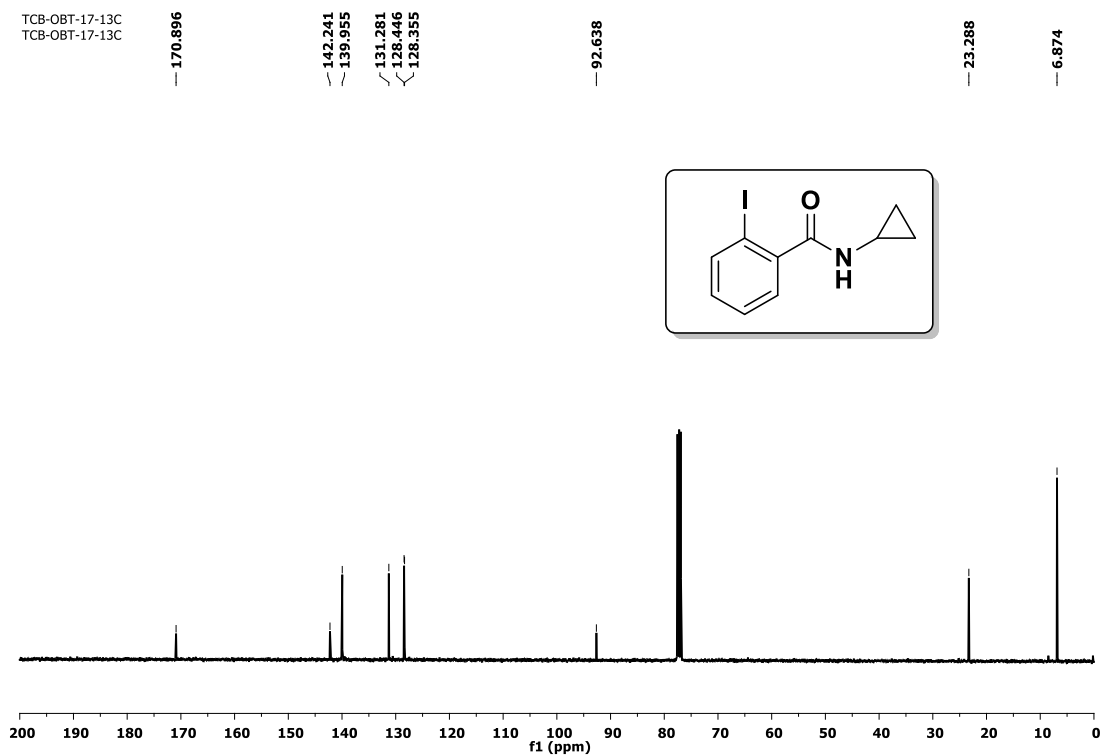


Figure S33: ¹H NMR spectrum of compound 2g



TCB-OBT-13-1H
TCB-OBT-13-1H

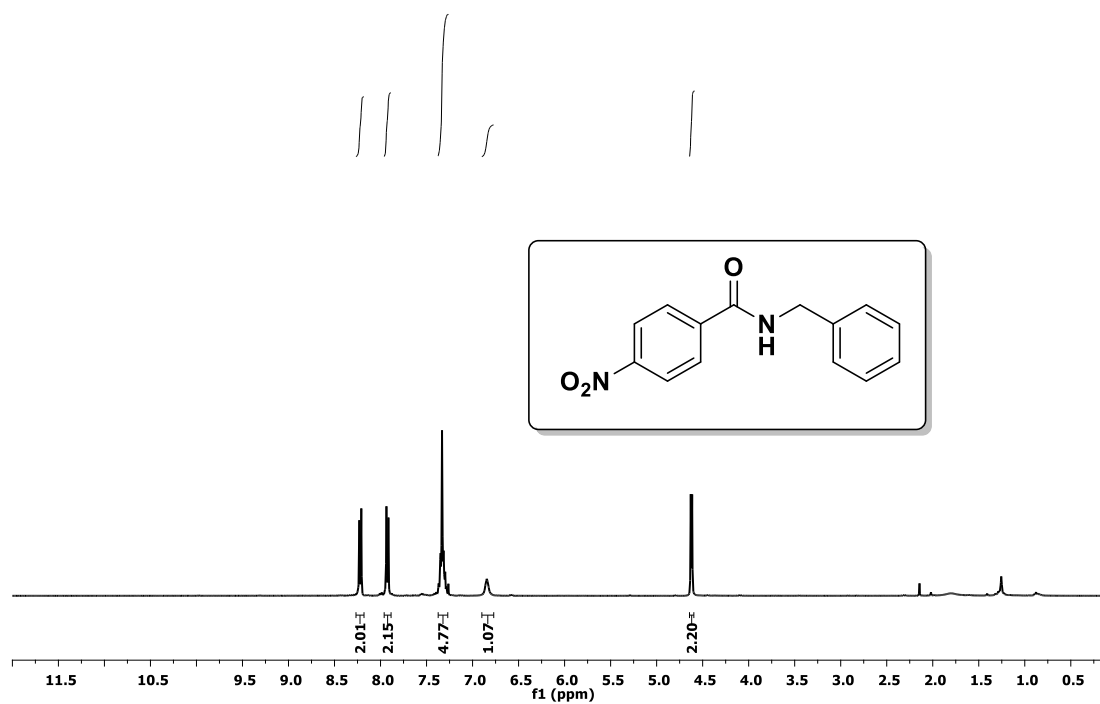


Figure S36: ¹H NMR spectrum of compound 2h

TCB-OBT-13-13C
TCB-OBT-13-13C

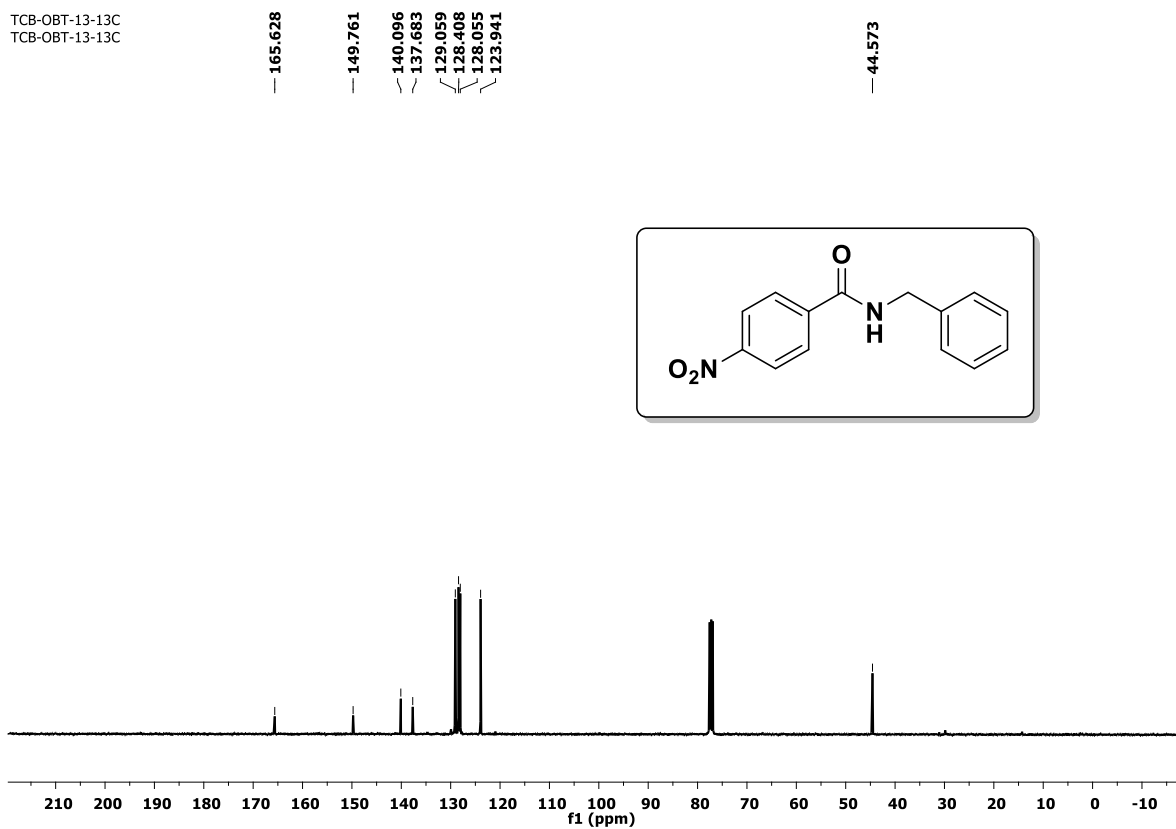


Figure S37: ¹³C NMR spectrum of compound 2h

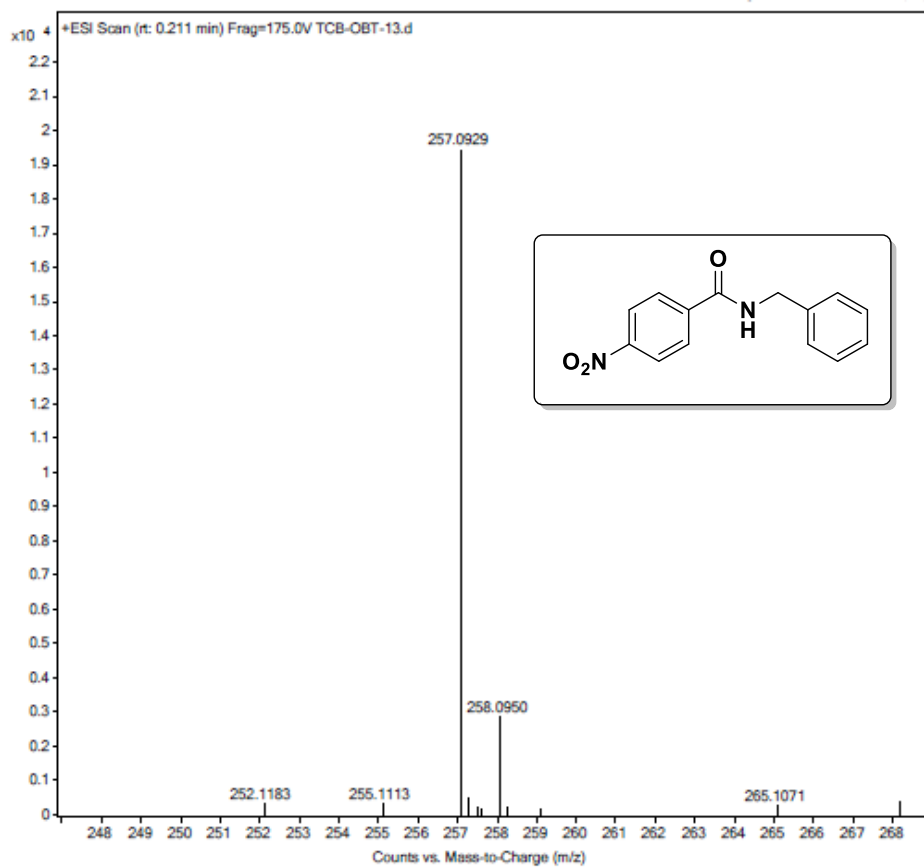


Figure S38: ESI-MASS spectrum of compound 2h

TCB-OBT-26-1H.1.fid
TCB-OBT-26-1H

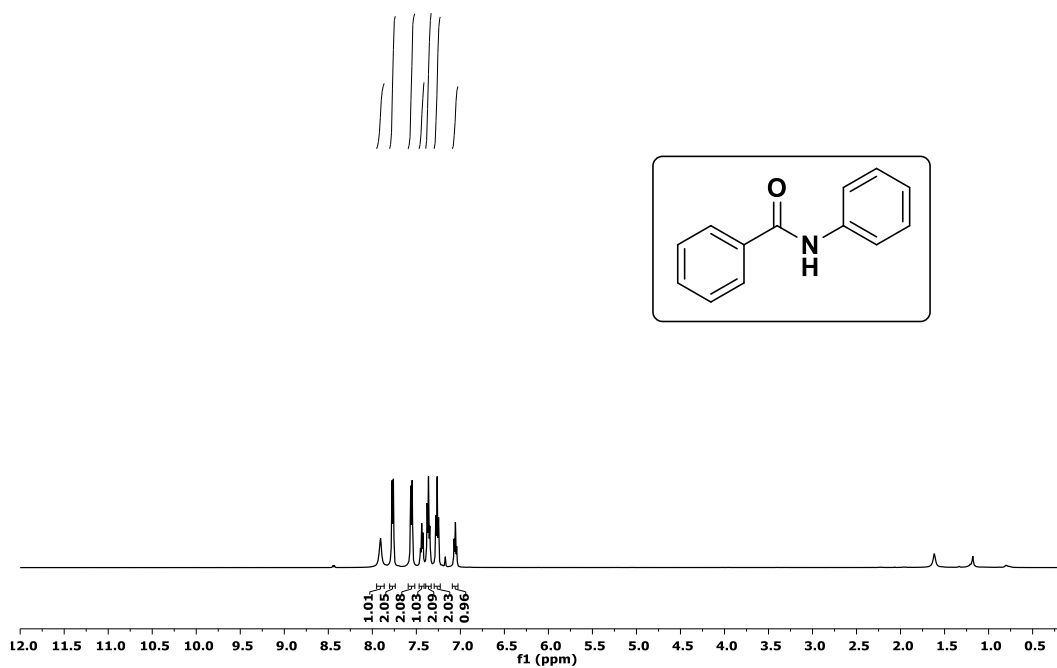


Figure S39: ¹H NMR spectrum of compound 2i

TCB-OBT-26-13C.3.fid
TCB-OBT-26-13C

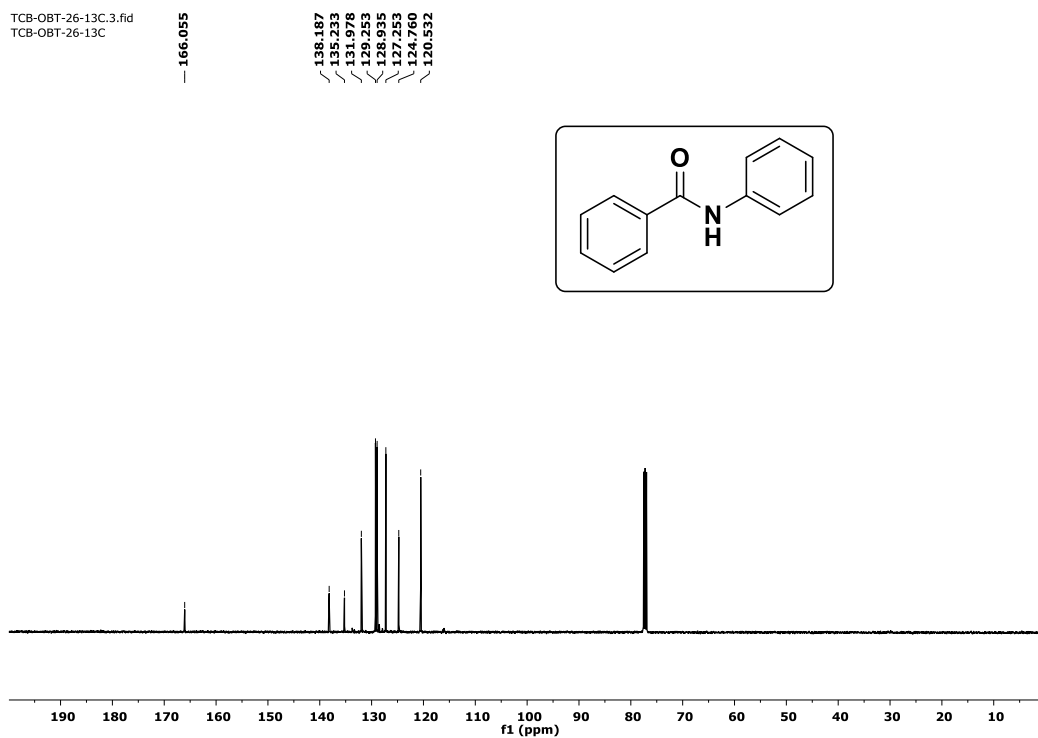


Figure S40: ^{13}C NMR spectrum of compound **2i**

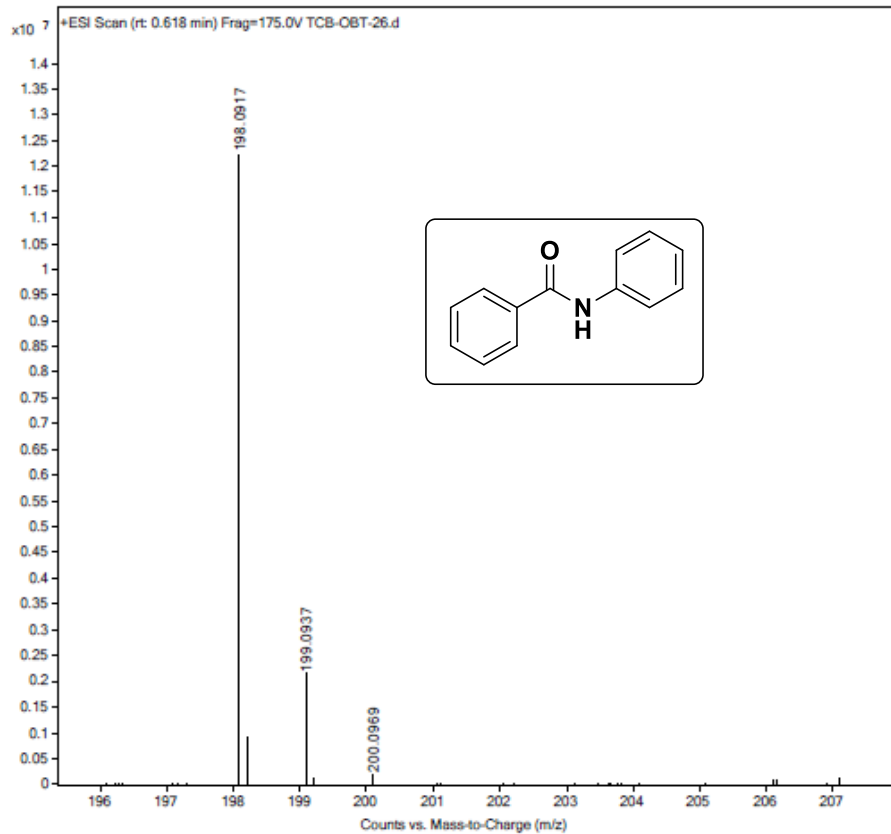


Figure S41: ESI-MASS spectrum of compound **2i**

TCB-OBT-29-1H.4.fid
1H

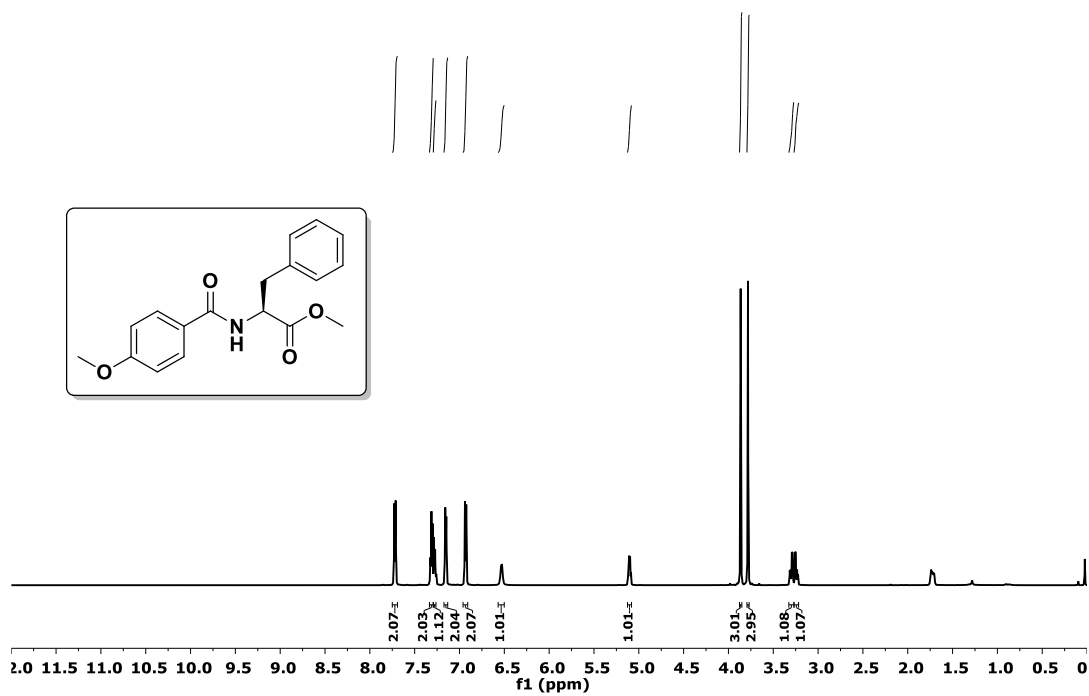


Figure S42: ^1H NMR spectrum of compound 2j

TCB-OBT-29-1H.5.fid
13C

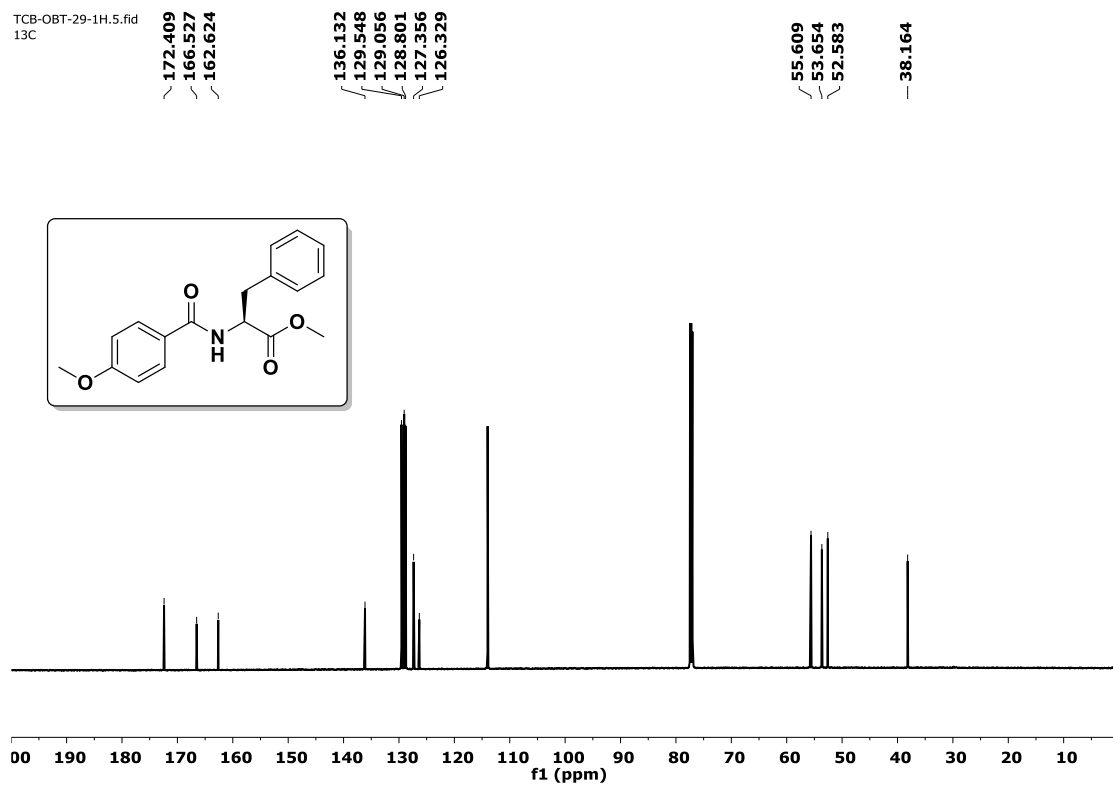


Figure S43: ^{13}C NMR spectrum of compound 2j

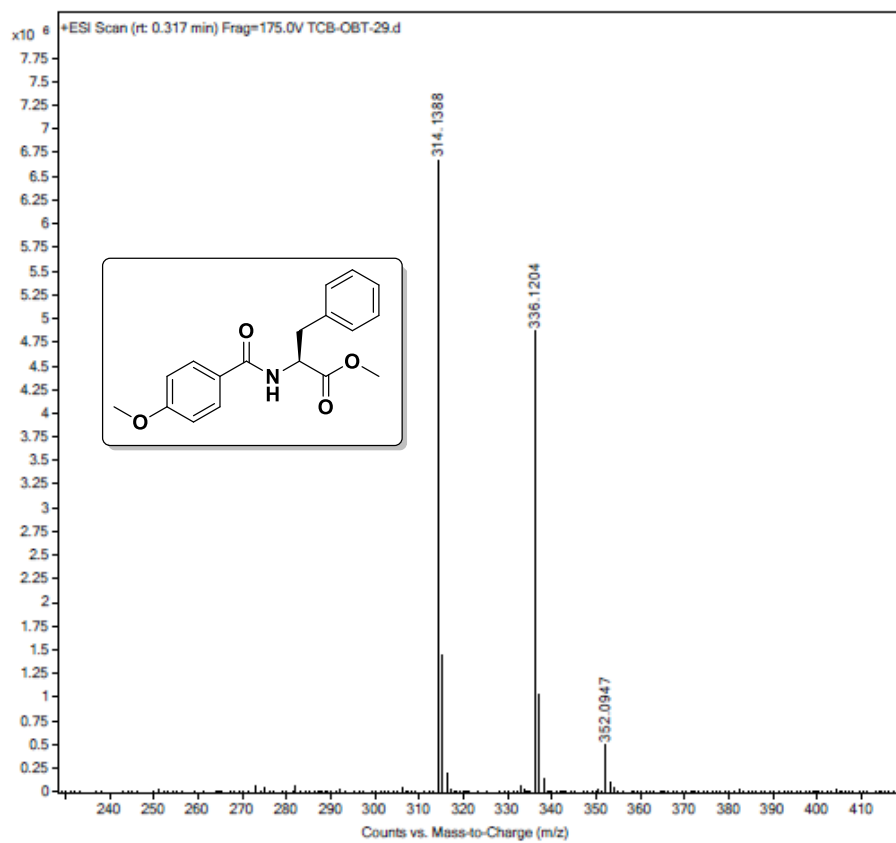


Figure S44: ESI-MASS spectrum of compound 2j

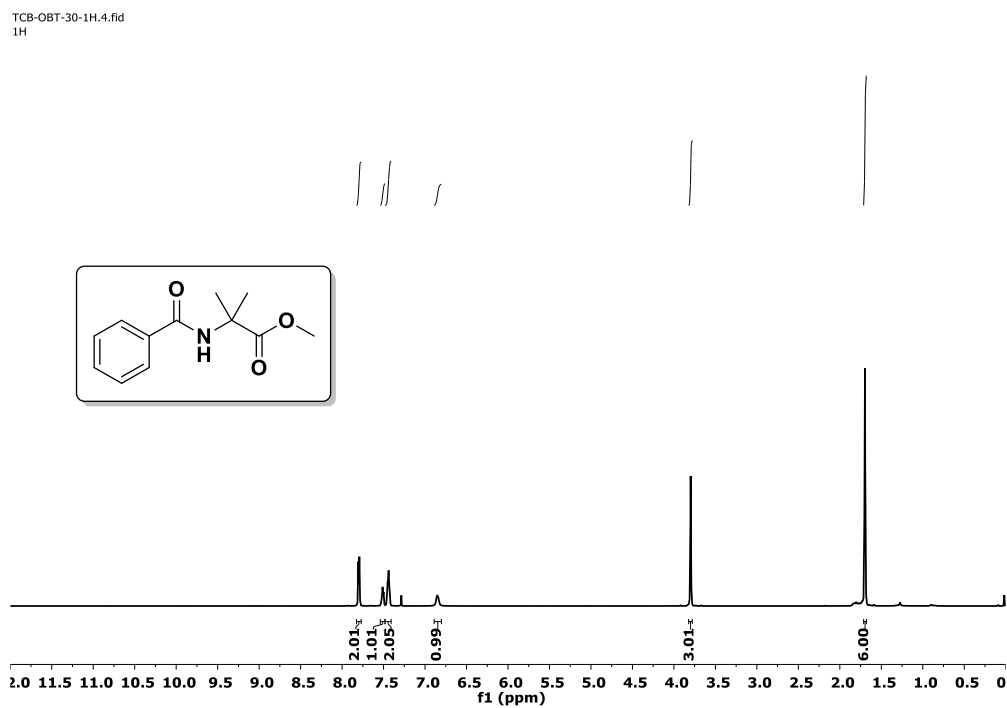


Figure S45: ¹H NMR spectrum of compound 2k

TCB-OBT-30-13C.6.f1
13C

— 175.490

— 166.806

✓ 134.665

✓ 131.751

✓ 128.722

✓ 127.140

— 57.077

— 52.965

— 24.947

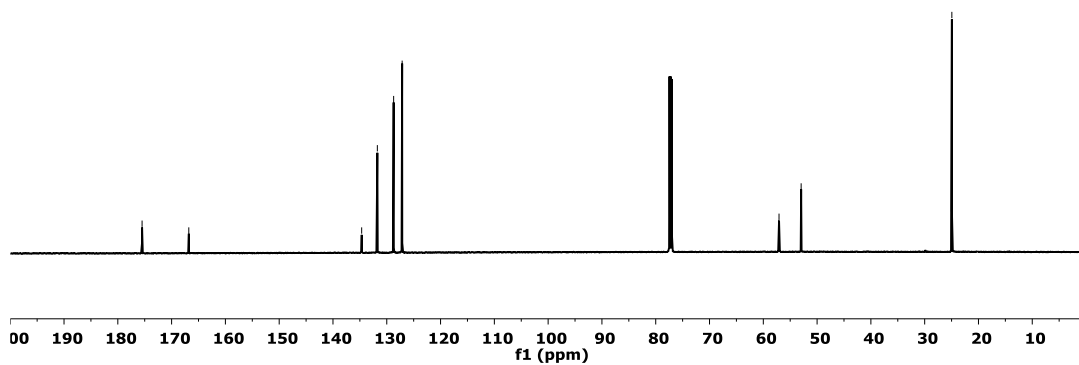
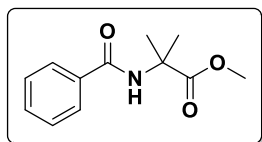


Figure S46: ^{13}C NMR spectrum of compound **2k**

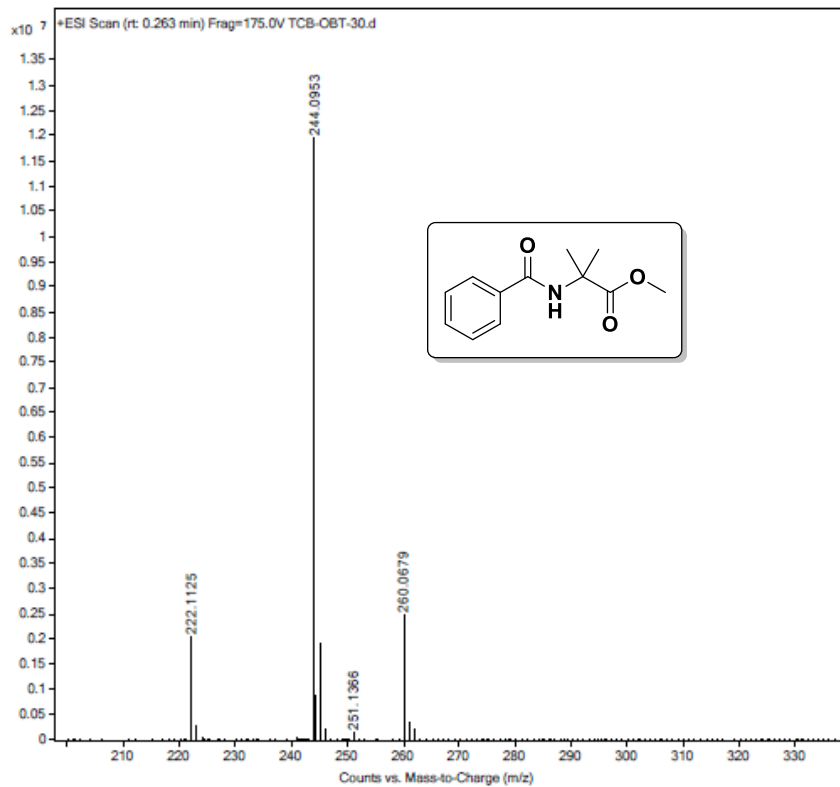


Figure S47: ESI-MASS spectrum of compound **2k**

TCB-OBT-31-1H.4.fid
1H

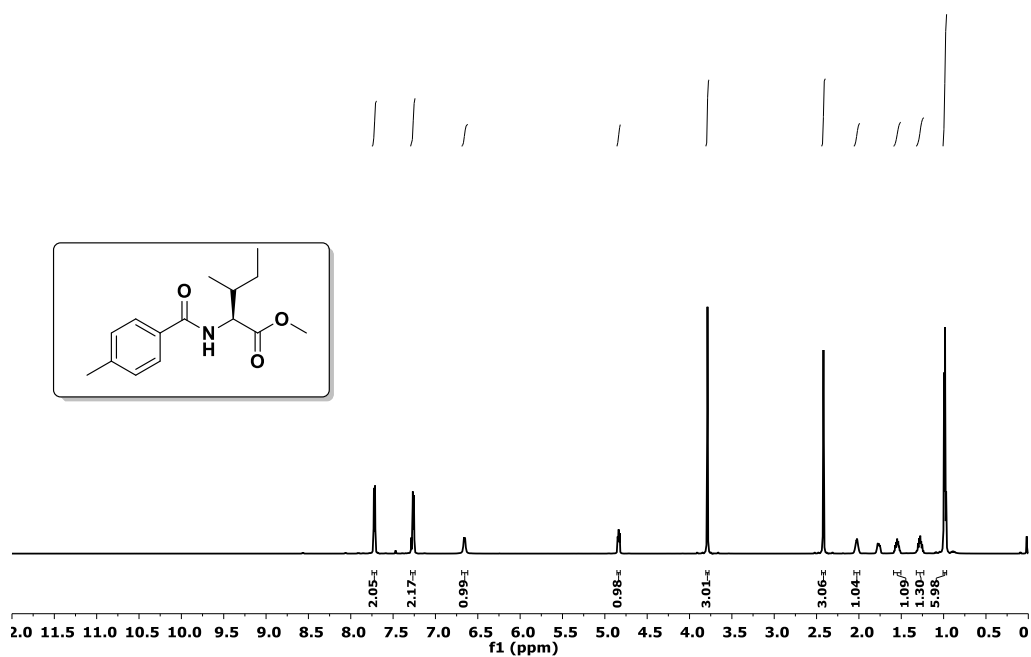


Figure S48: ¹H NMR spectrum of compound 21

TCB-OBT-31-13C.6.fid
13C

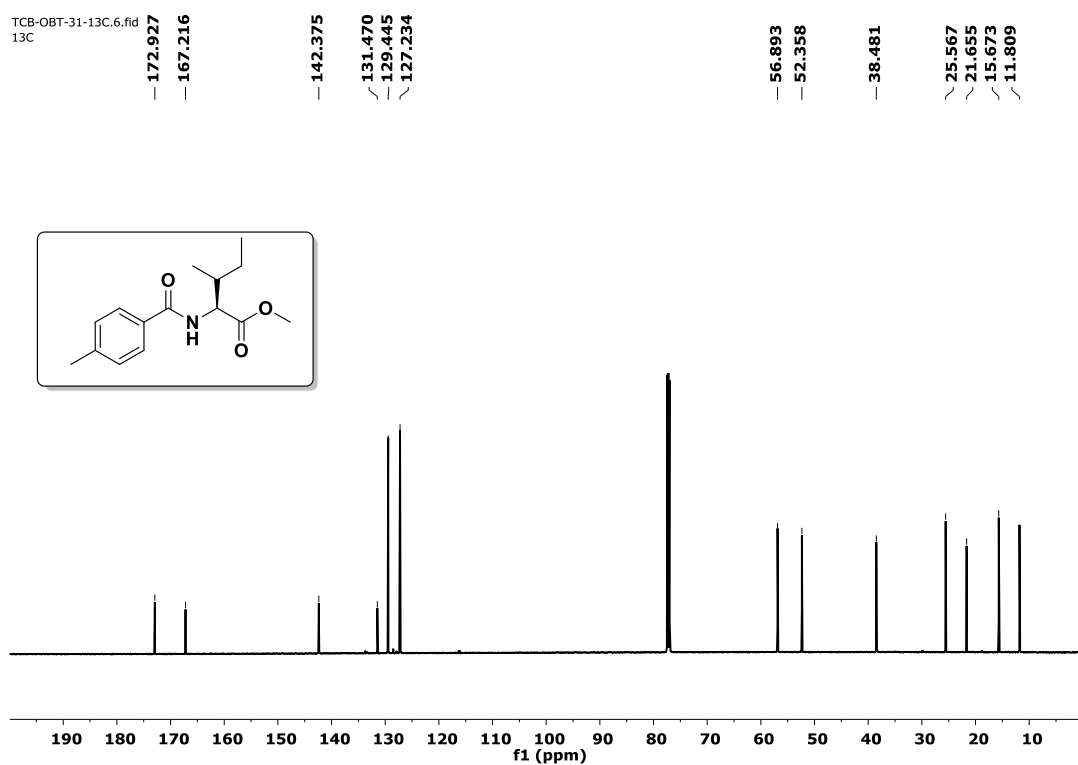


Figure S49: ¹³C NMR spectrum of compound 21

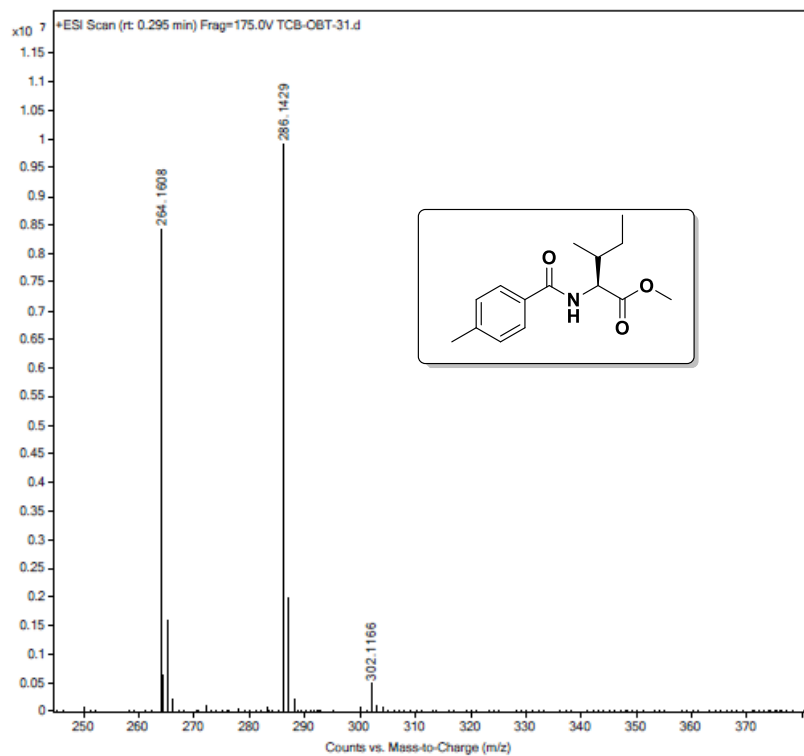


Figure S50: ESI-MASS spectrum of compound 2l

TCB-OBT-32-1H.1.fid
1H

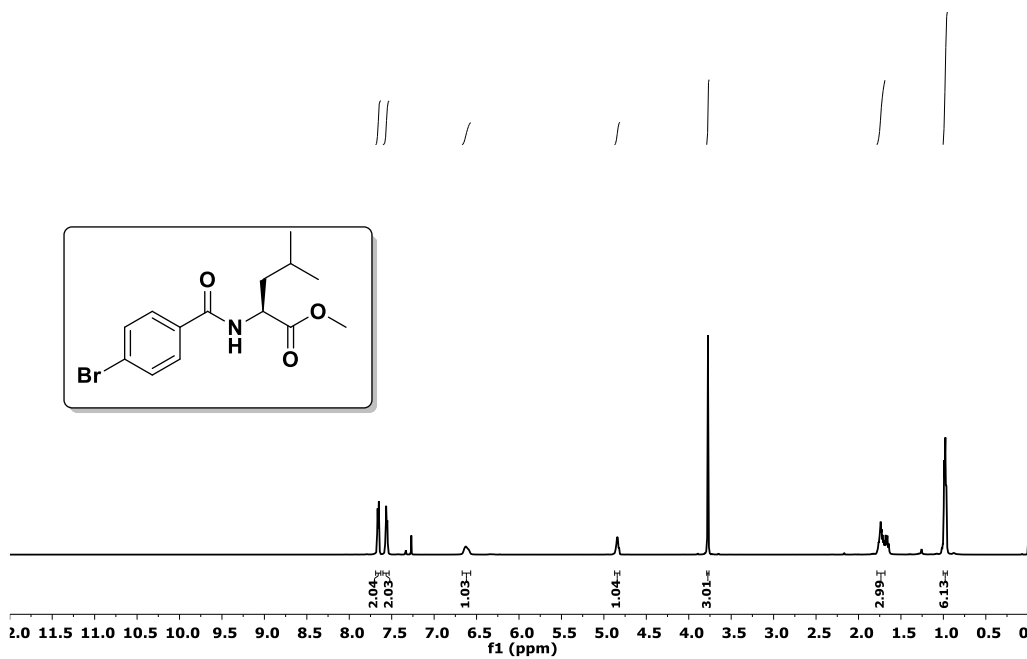


Figure S51: ^1H NMR spectrum of compound 2m

TCB-OBT-32-13C.3.fid
13C

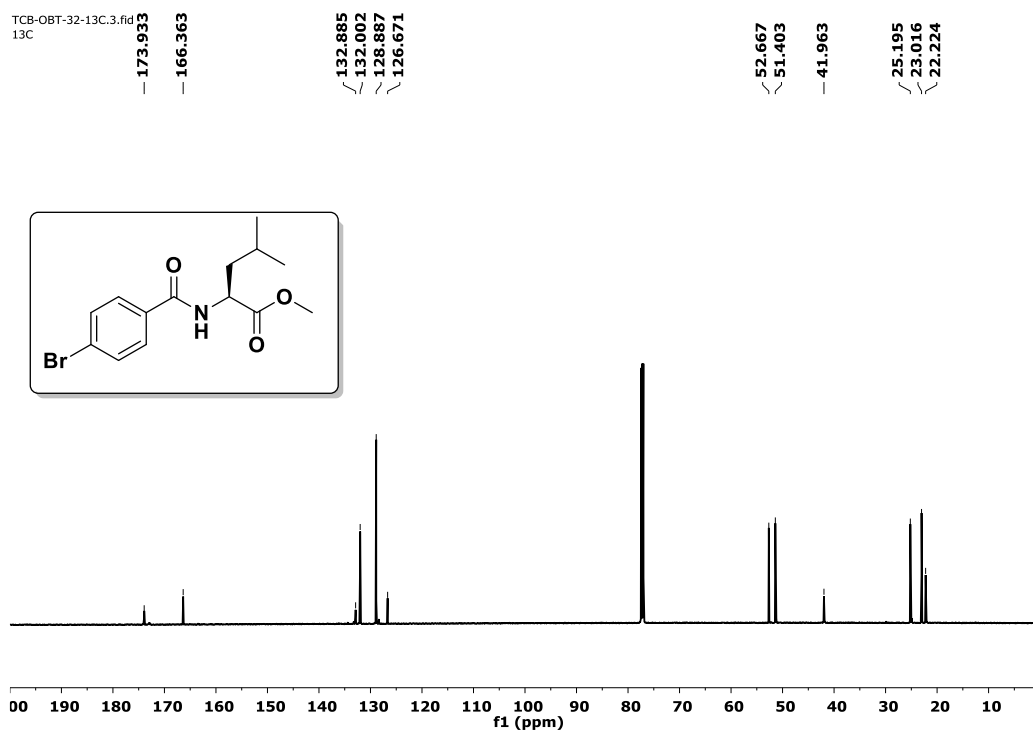


Figure S52: ^{13}C NMR spectrum of compound **2m**

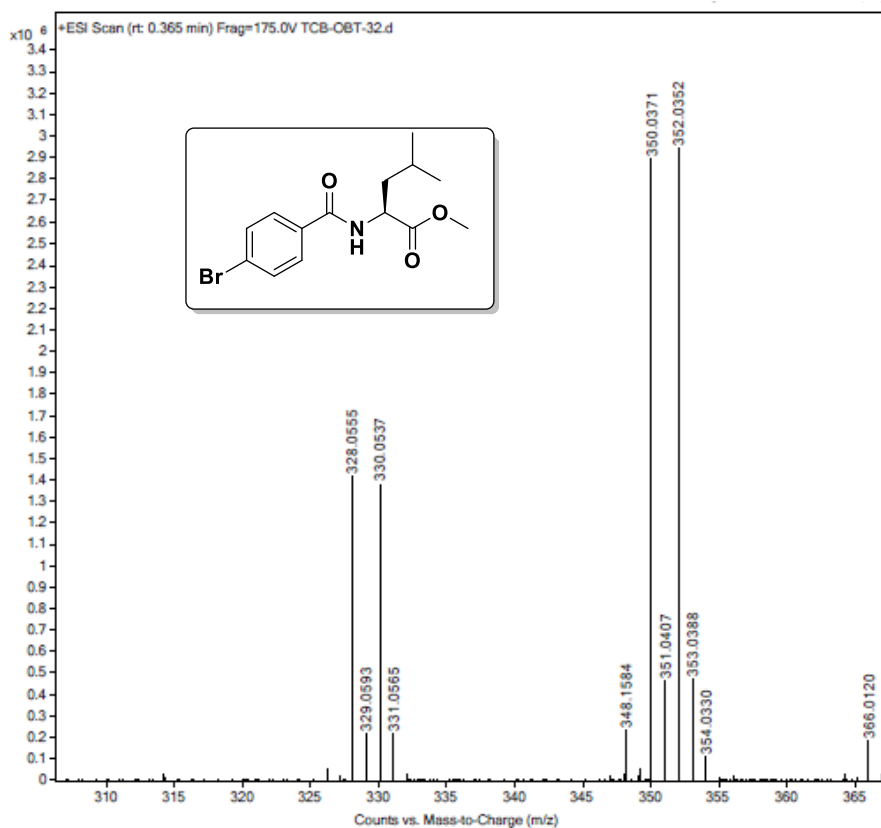


Figure S53: ESI-MASS spectrum of compound **2m**

TCB-OBT-33-1H.4.fid
1H

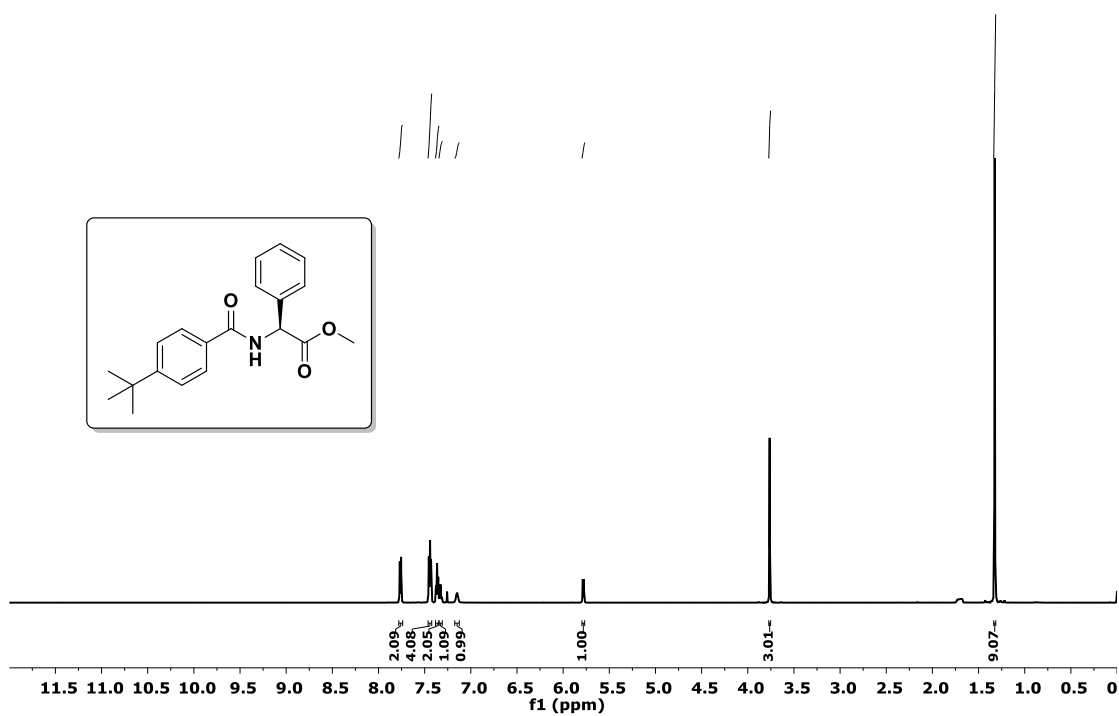


Figure S54: ^1H NMR spectrum of compound 2n

TCB-OBT-33-13C.5.fid
13C

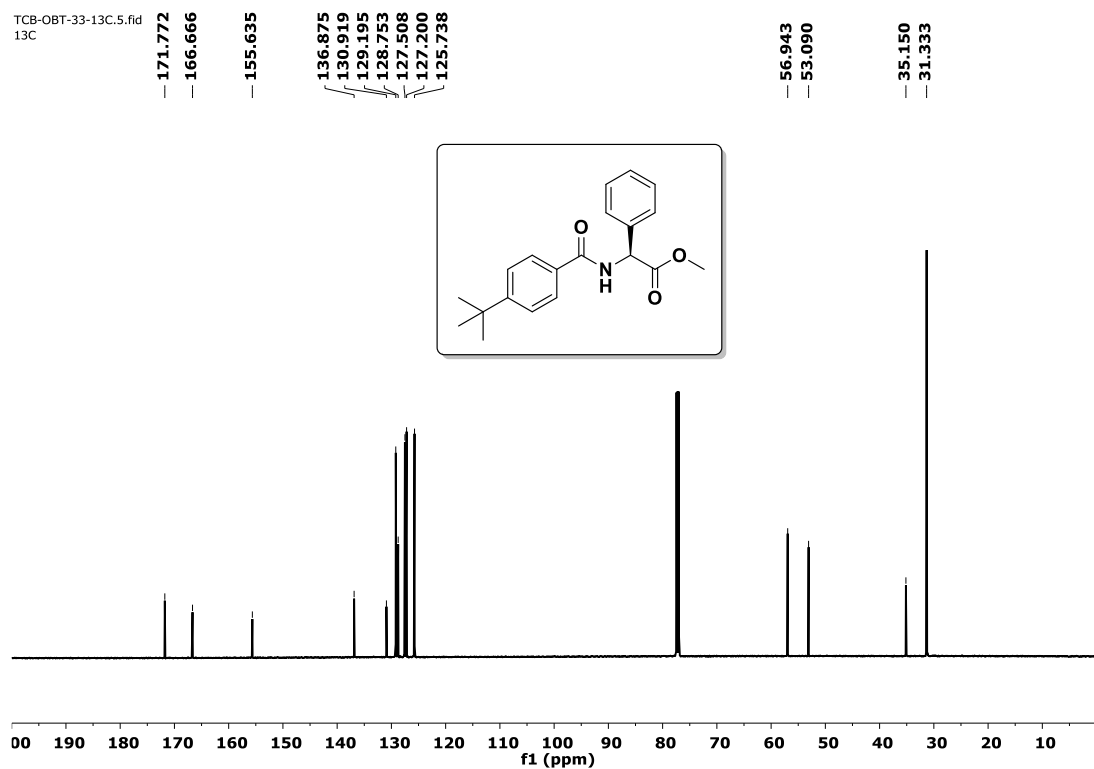


Figure S55: ^{13}C NMR spectrum of compound 2n

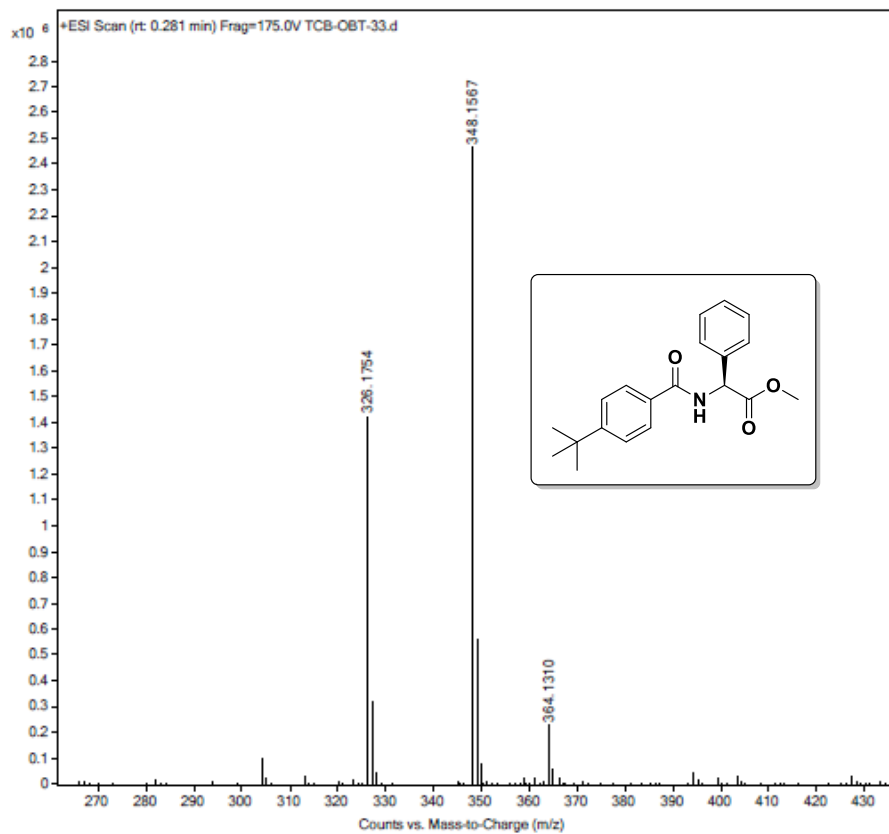


Figure S56: ESI-MASS spectrum of compound **2n**