Electronic Supplementary Material (ESI) for RSC Advances.

Mild Deprotection of *N*-*tert*-butyloxycarbonyl (*N*-Boc) Group Using Oxalyl Chloride

Nathaniel George[‡], Samuel Ofori[‡] and Samuel G. Awuah*
Department of Chemistry, University of Kentucky, Lexington Kentucky 40506, United States
Correspondence to: awuah@uky.edu

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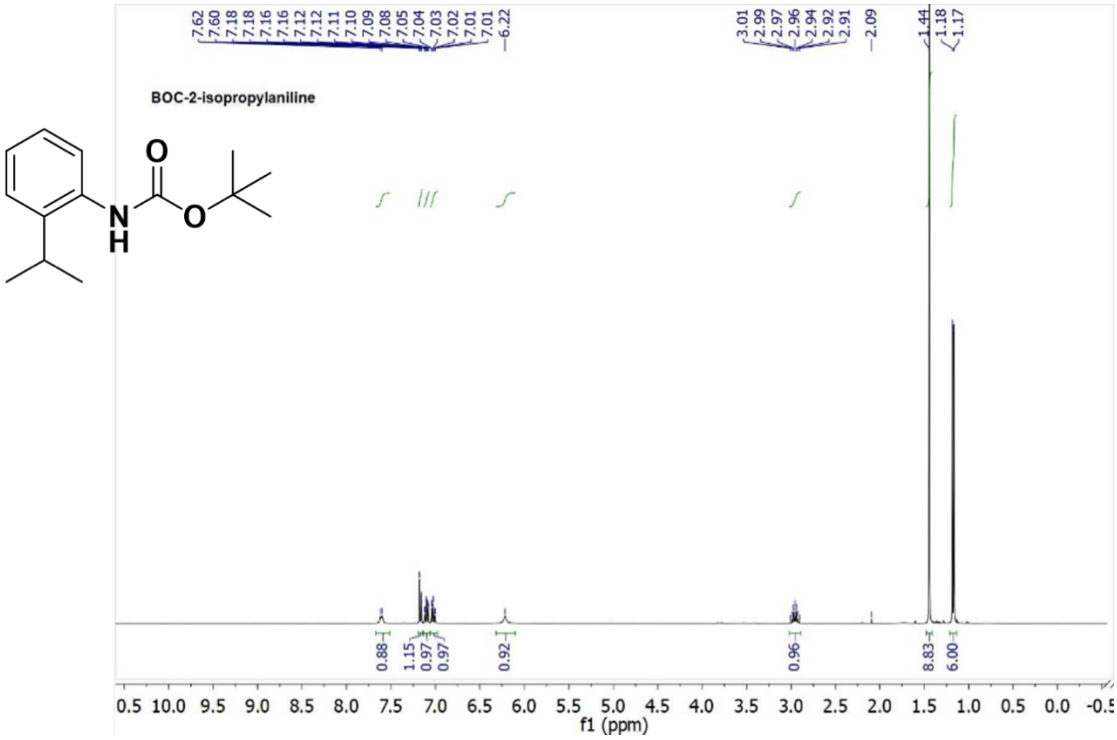


Figure S1: ^{1}H NMR spectrum of *tert*-butyl N-(2-isopropylphenyl)carbamate (Entry 1a) in $CDCl_3$

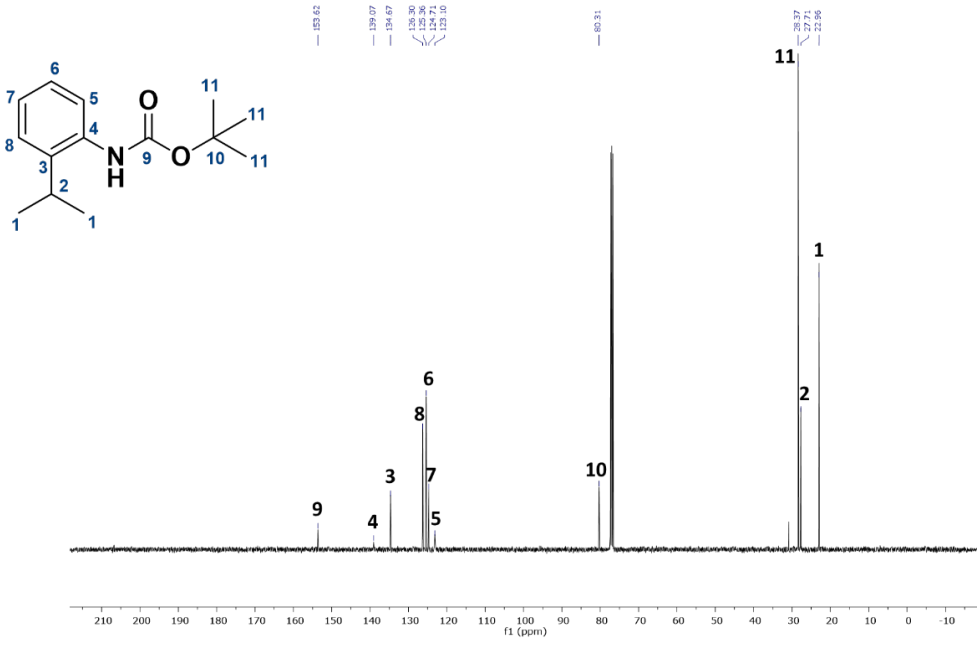


Figure S2: ^{13}C NMR spectrum of *tert*-butyl *N*-(2-isopropylphenyl)carbamate (Entry 1a) in $CDCl_3$

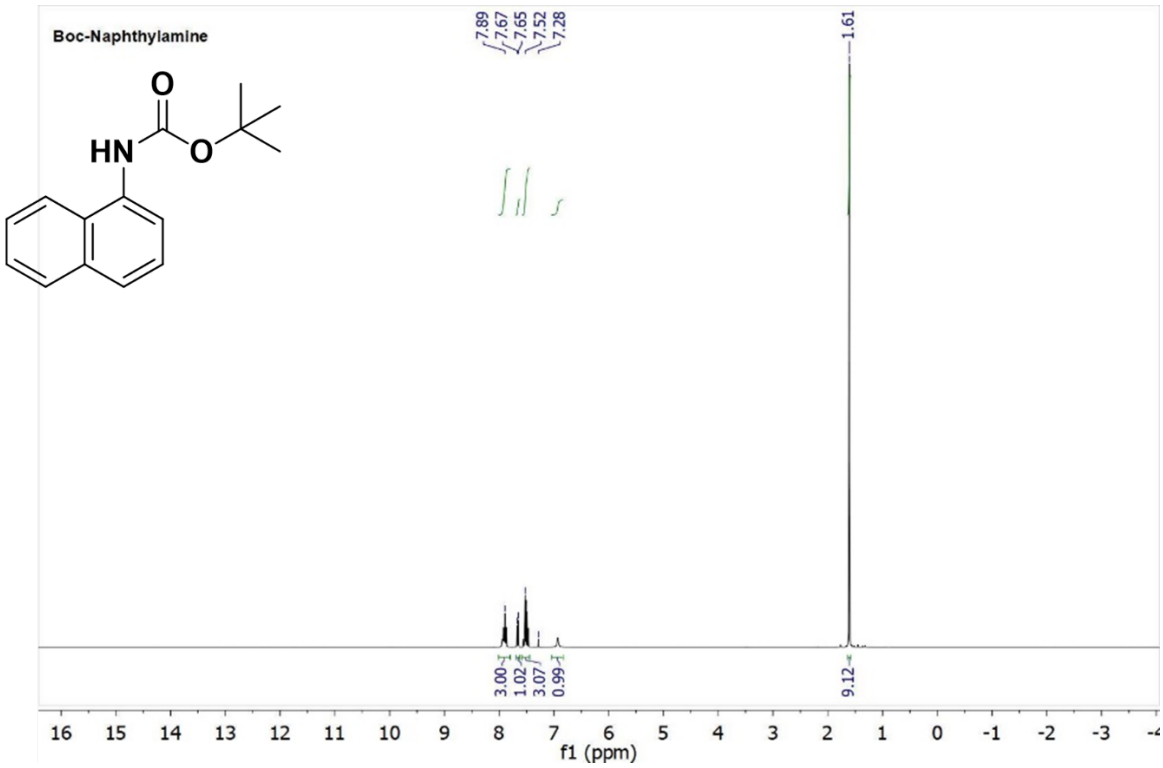


Figure S3: ^{1}H NMR spectrum of *tert*-butyl *N*-(1-naphthyl)carbamate (Entry 2a) in $CDCl_3$

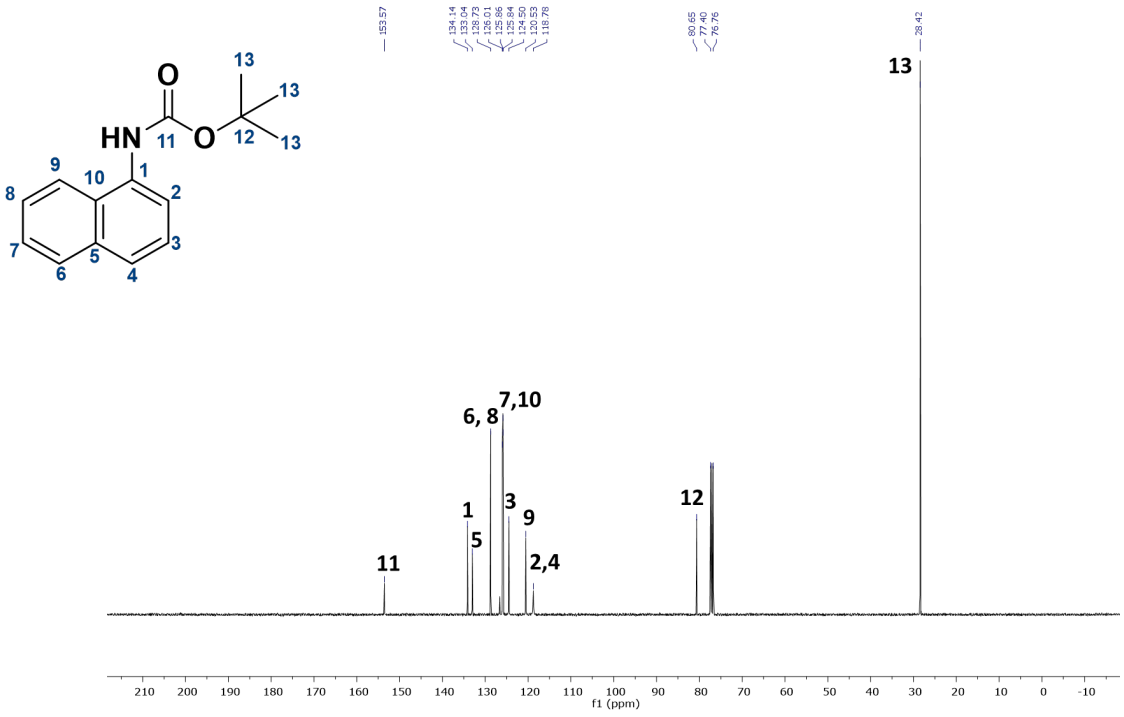


Figure S4: ^{13}C NMR spectrum of *tert*-butyl *N*-(1-naphthyl)carbamate (Entry 2a) in $CDCl_3$

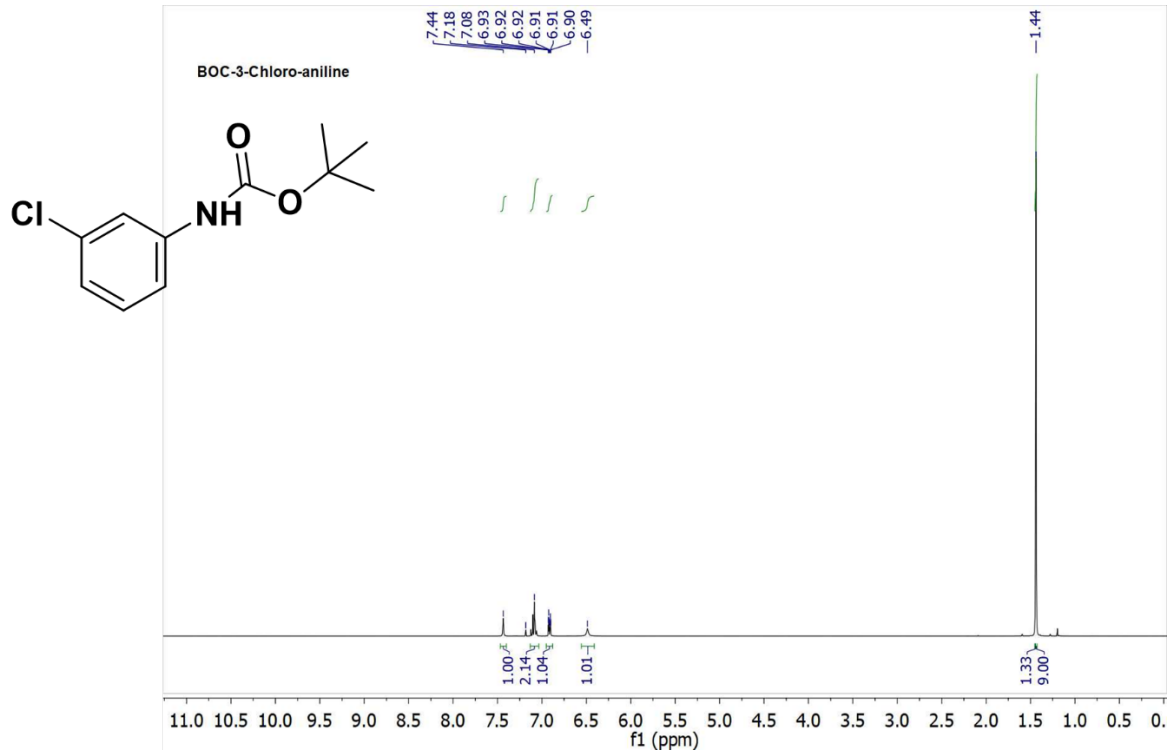


Figure S5: ^{1}H NMR spectrum of *tert*-butyl *N*-(3-chlorophenyl)carbamate (Entry 3a) in $CDCl_3$

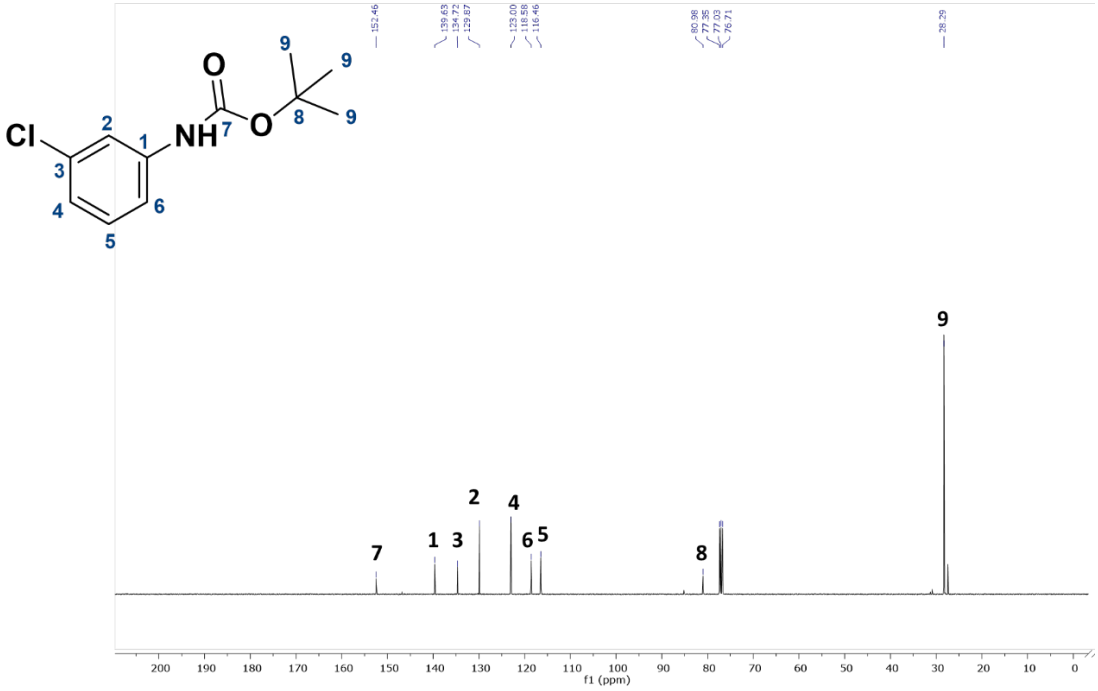


Figure S6: ^{13}C NMR spectrum of *tert*-butyl *N*-(3-chlorophenyl)carbamate (Entry 3a) in $CDCl_3$

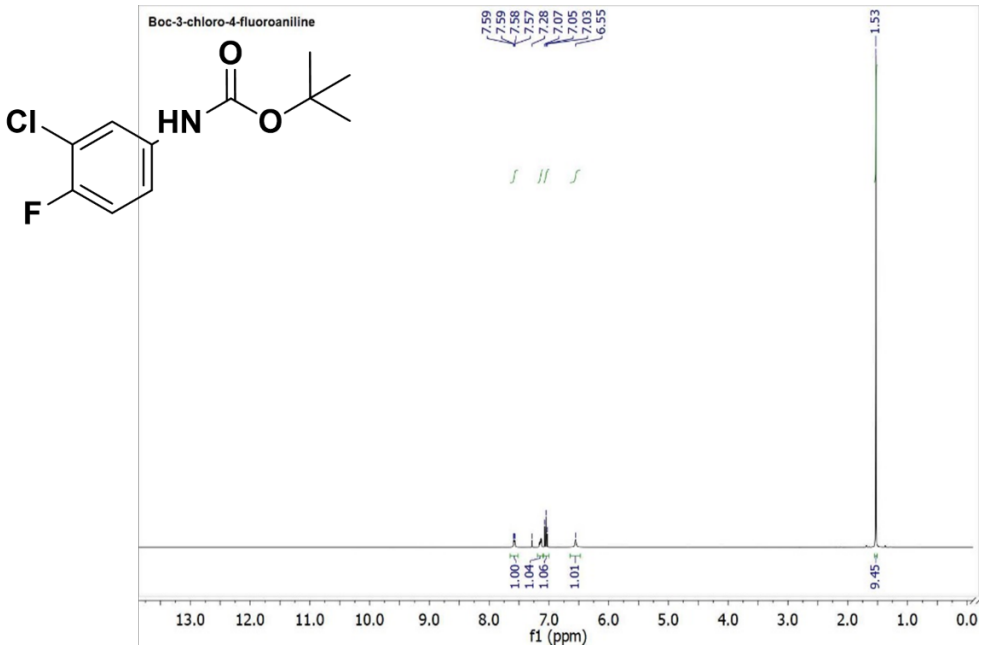


Figure S7: ^{1}H NMR spectrum of *tert*-butyl *N*-(3-chloro-4-fluorophenyl)carbamate (Entry 4a) in $CDCl_3$

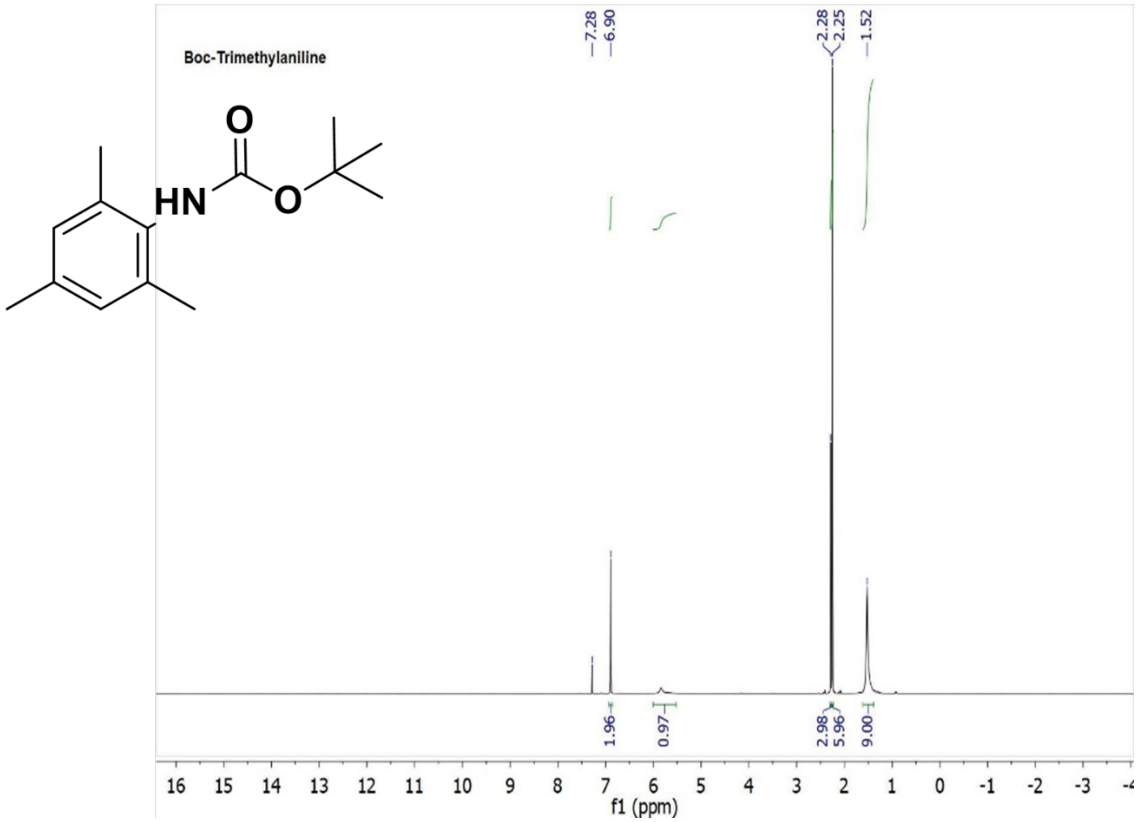


Figure S8: ^{1}H NMR spectrum of *tert*-butyl *N*-(2,4,6-trimethylphenyl)carbamate (Entry 5a) in $CDCl_3$

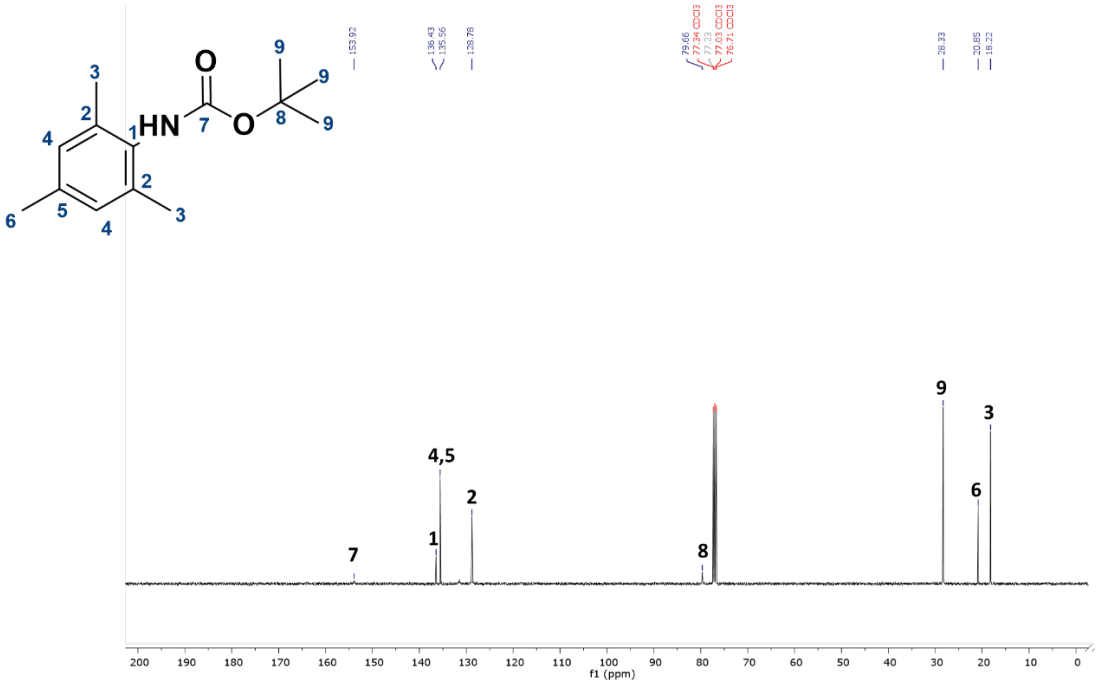


Figure S9: ^{13}C NMR spectrum of *tert*-butyl *N*-(2,4,6-trimethylphenyl)carbamate (Entry 5a) in $CDCl_3$

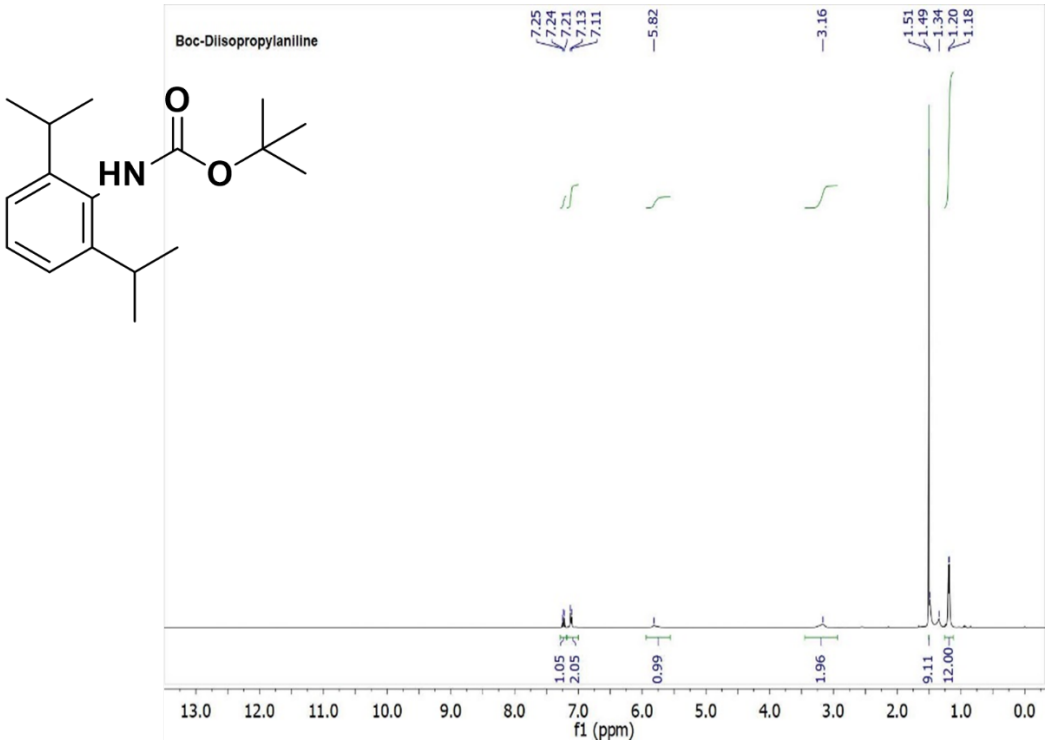


Figure S10: ^{1}H NMR spectrum of *tert*-butyl *N*-(2,6-diisopropylphenyl)carbamate (Entry 6a) in $CDCl_3$

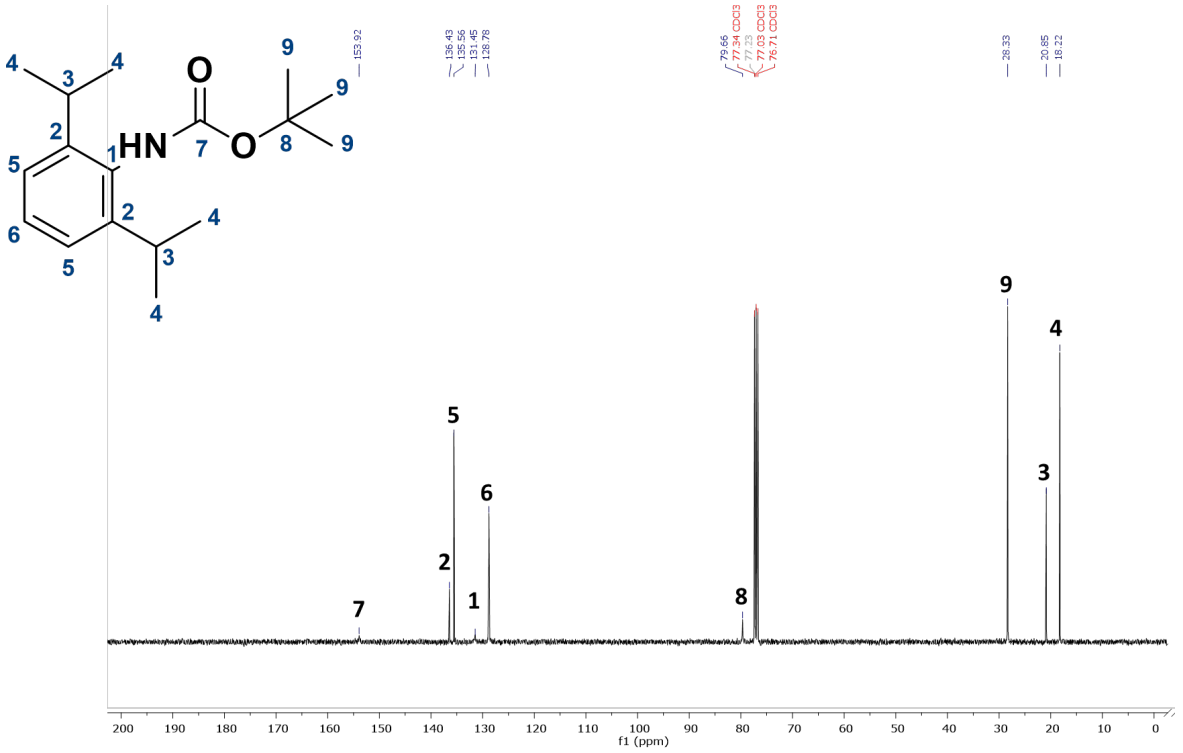


Figure S11: ^{13}C NMR spectrum of *tert*-butyl *N*-(2,6-diisopropylphenyl)carbamate (Entry 6a) in $CDCl_3$

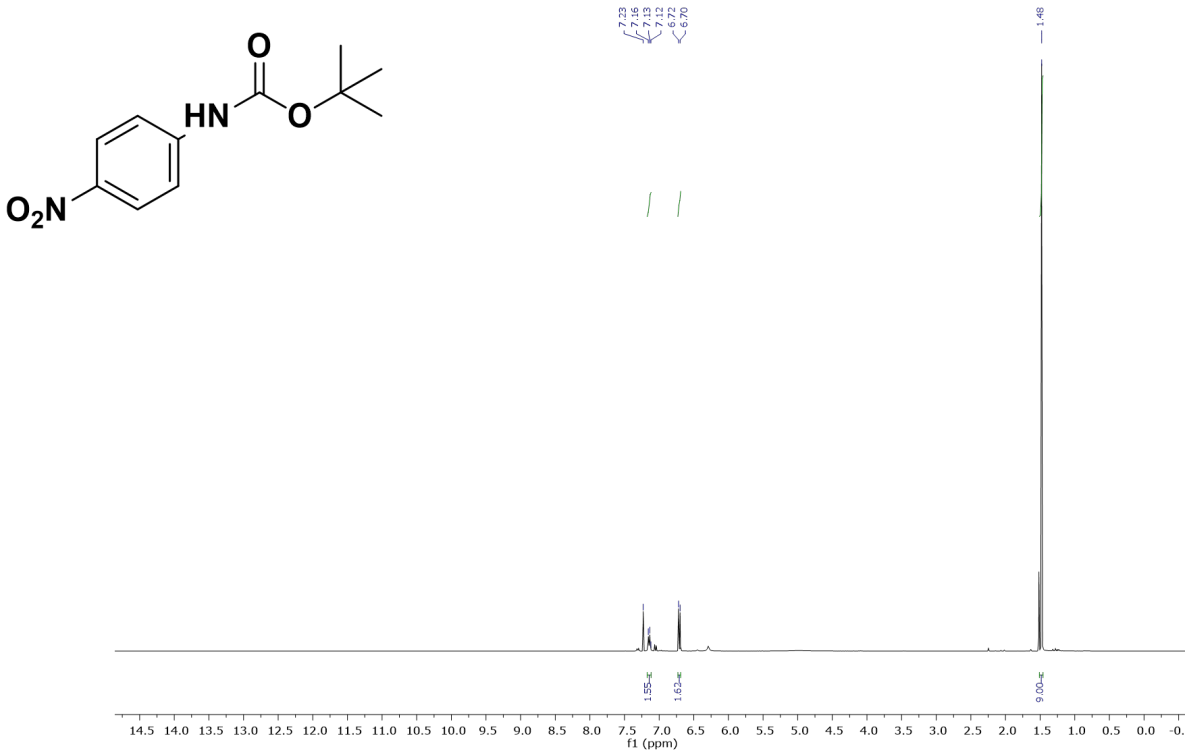


Figure S12: ^{1}H NMR spectrum of *tert*-butyl *N*-(4-nitrophenyl)carbamate (Entry 7a) in $CDCl_3$

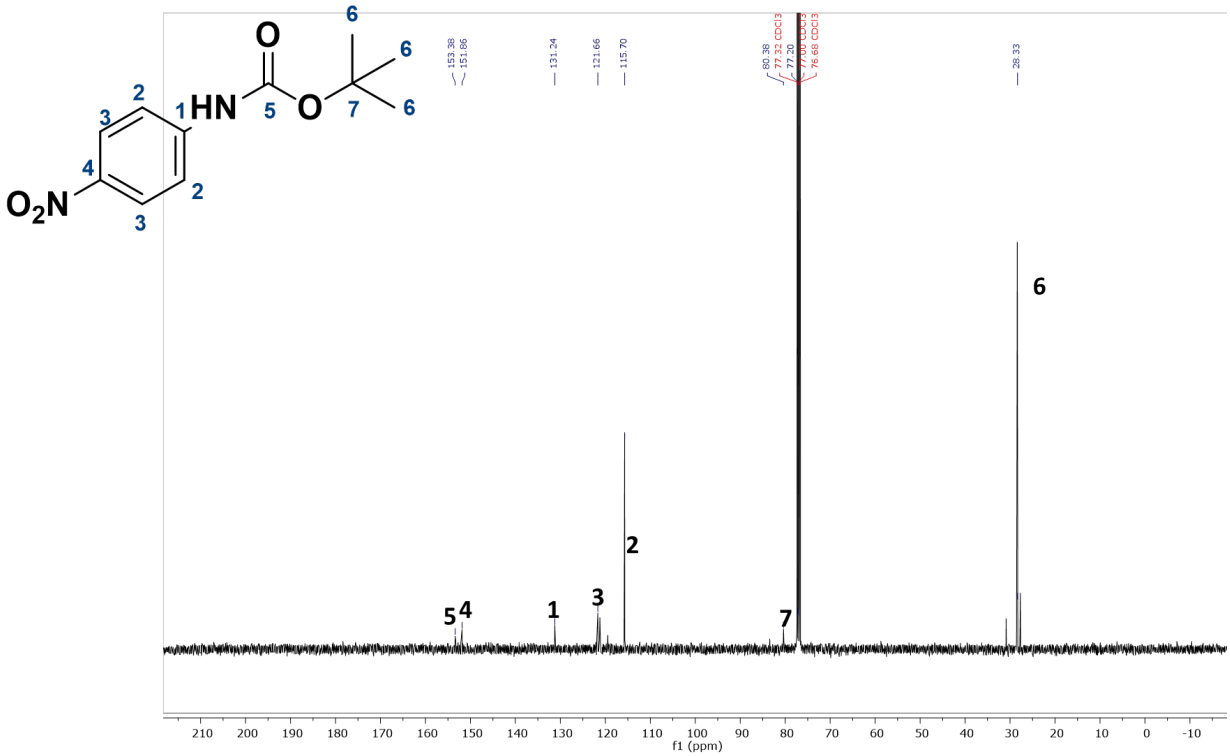


Figure S13: ^{13}C NMR spectrum of *tert*-butyl *N*-(4-nitrophenyl)carbamate (Entry 7a) in $CDCl_3$

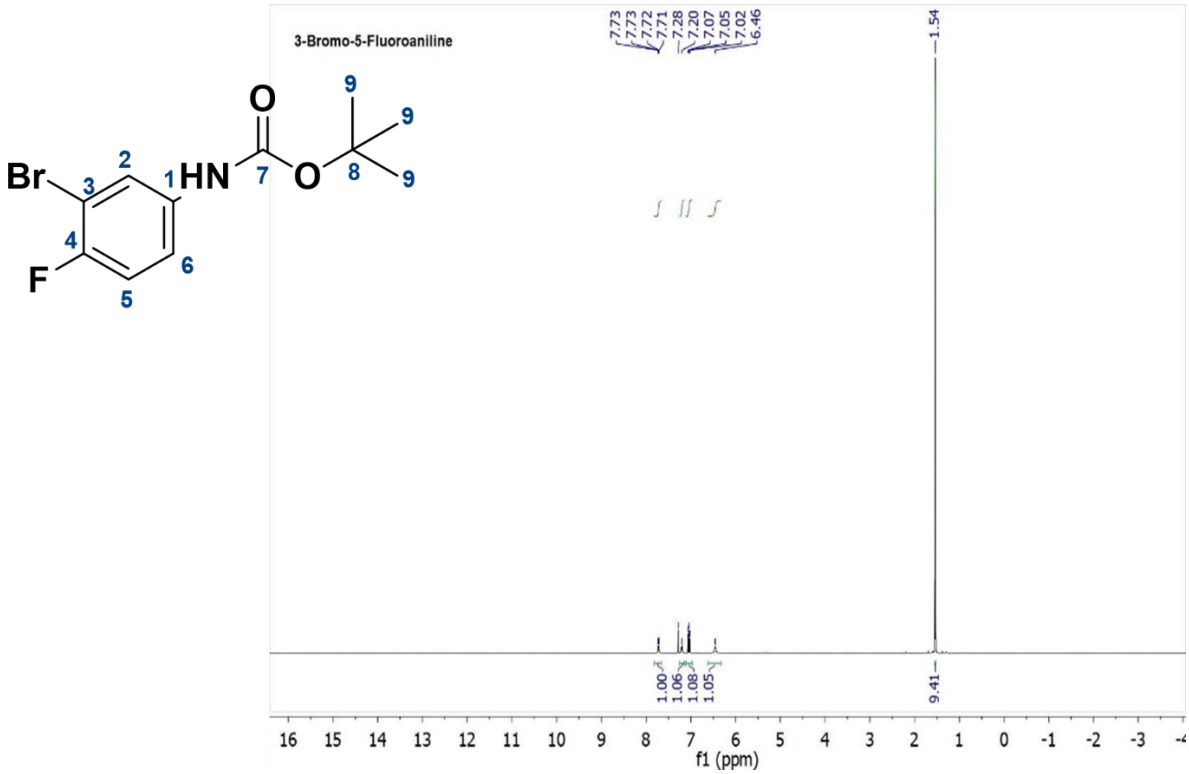


Figure S14: ^{1}H NMR spectrum of *tert*-butyl *N*-(3-bromo-4-fluorophenyl)carbamate (Entry 8a) in $CDCl_3$

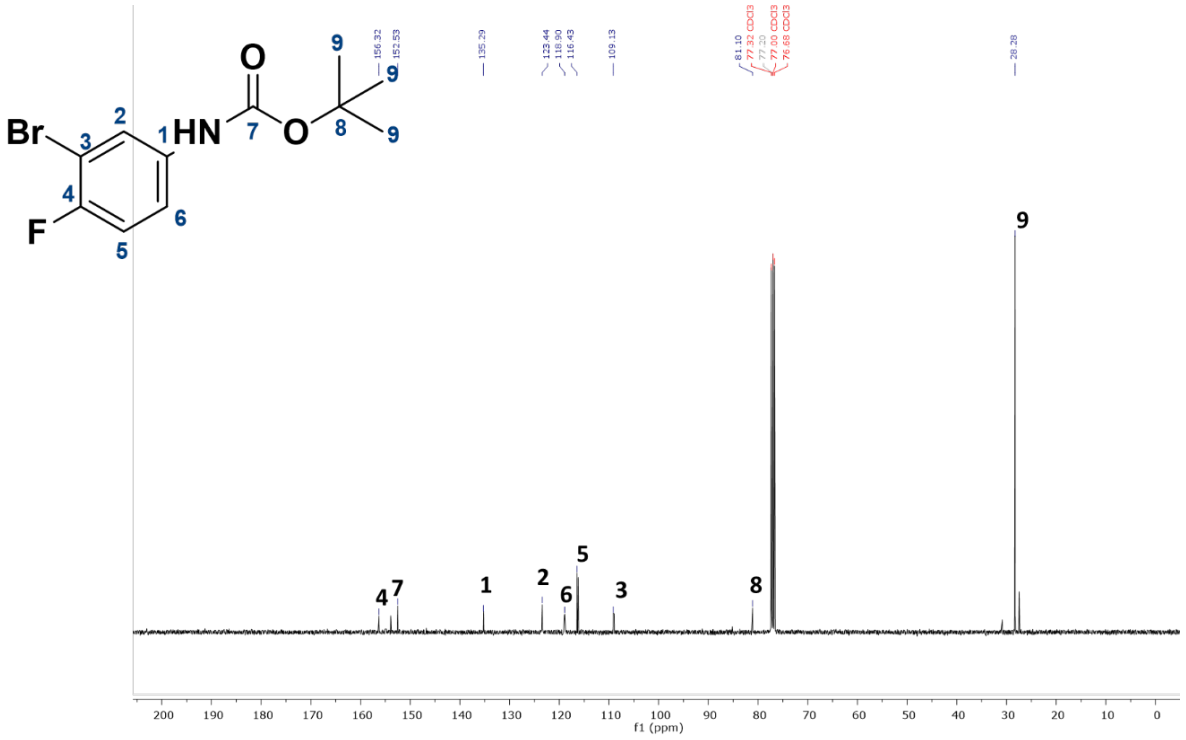


Figure S15: ^{13}C NMR spectrum of *tert*-butyl *N*-(3-bromo-4-fluorophenyl)carbamate (Entry 8a) in $CDCl_3$

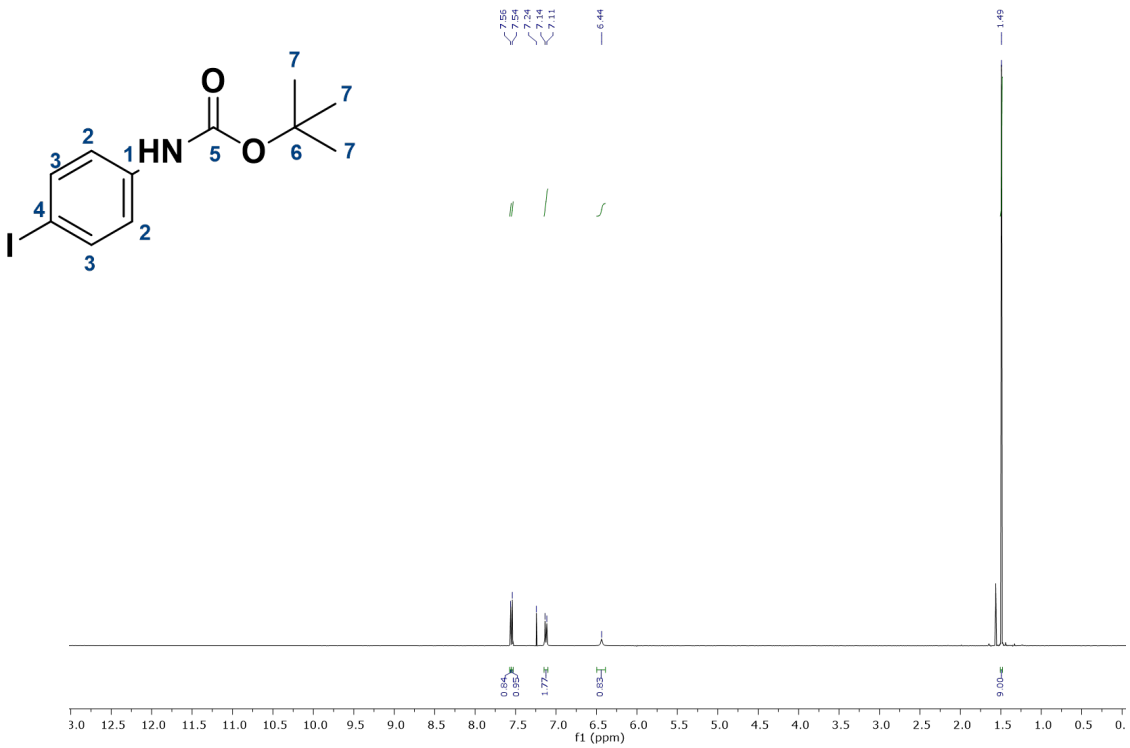


Figure S16: ^{1}H NMR spectrum of *tert*-Butyl *N*-(4-iodophenyl)carbamate (Entry 9a) in $CDCl_3$

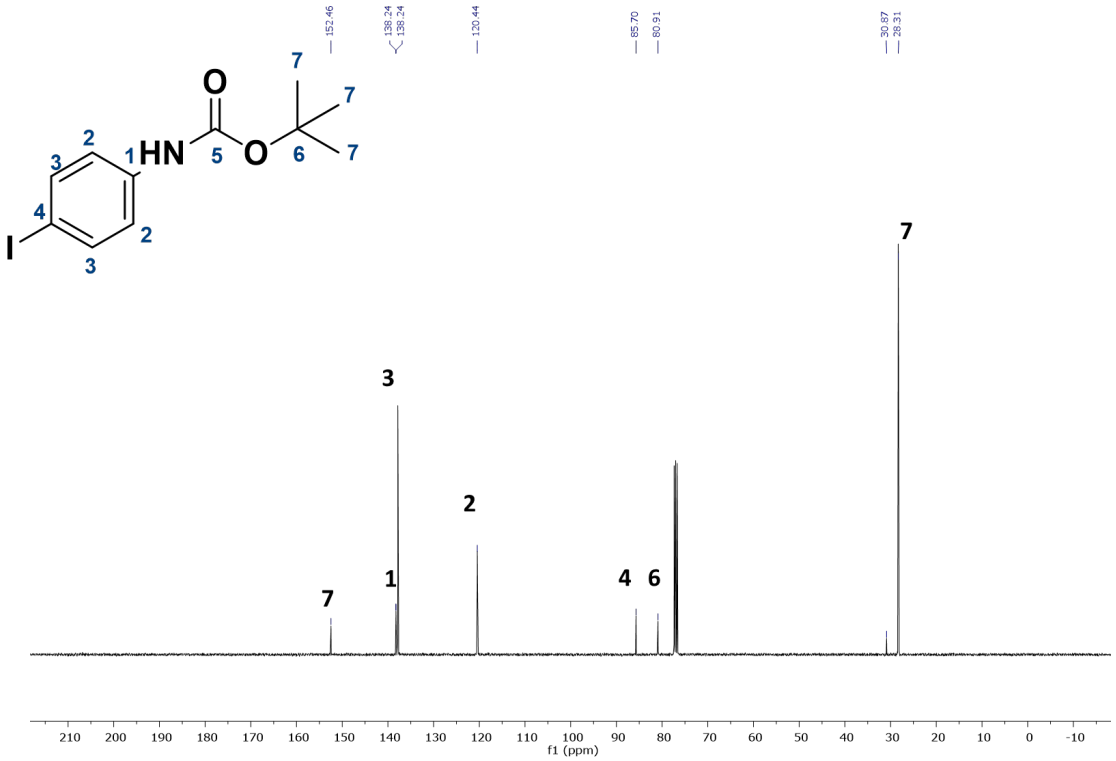


Figure S17: ^{13}C NMR spectrum of *tert*-Butyl *N*- (4-iodophenyl)carbamate (Entry 9a) in $CDCl_3$

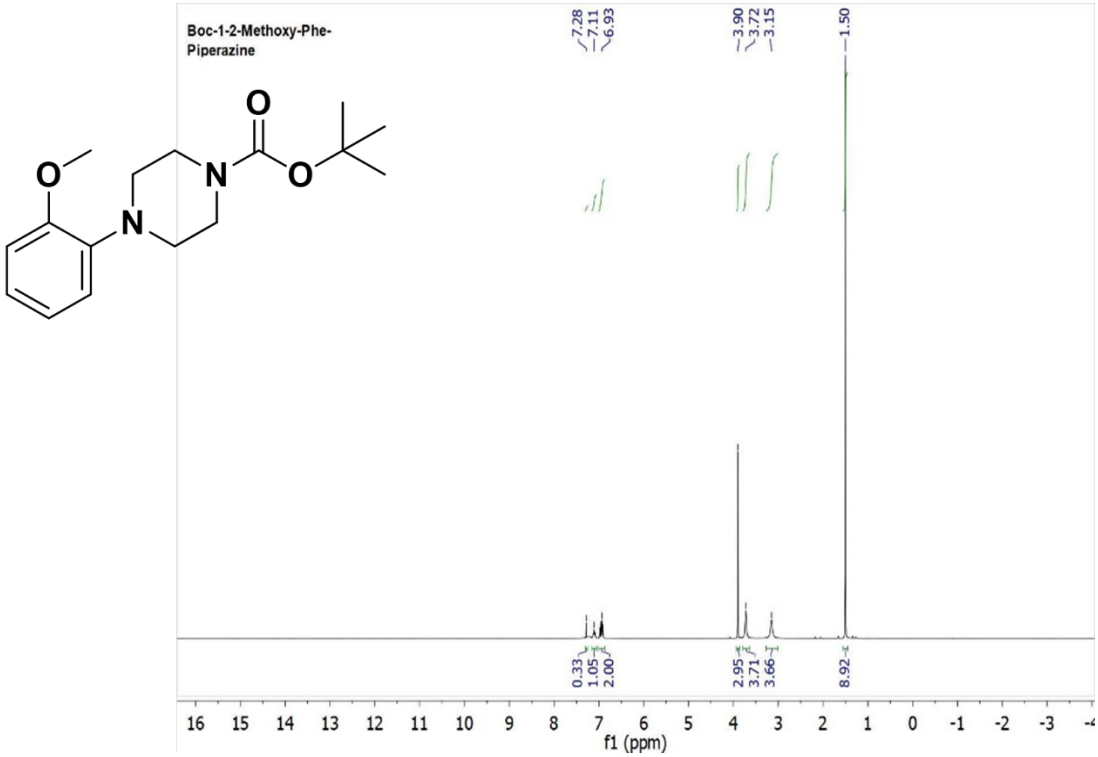


Figure S18: ^{1}H NMR spectrum of *tert*-butyl *N*-(N-(2-Methoxyphenyl)piperazine)carbamate(Entry 10a) in $CDCl_3$

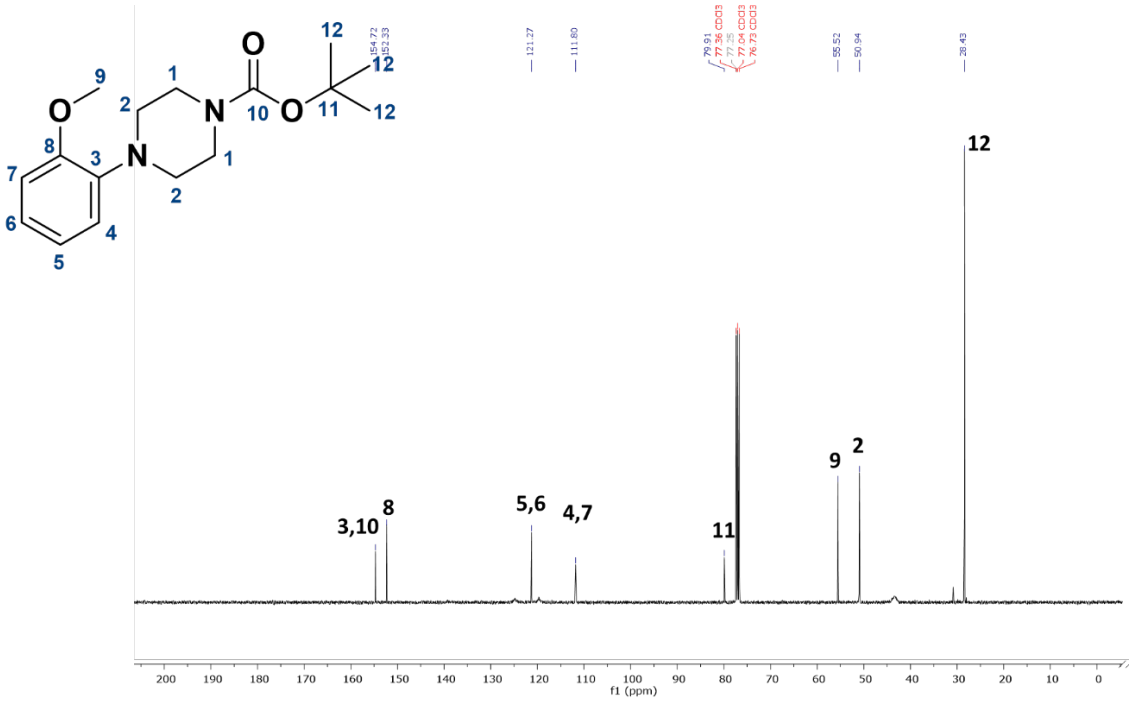


Figure S19: ^{13}C NMR spectrum of *tert*-butyl *N*-(N-(2-Methoxyphenyl)piperazine)carbamate (Entry 10a) in $CDCl_3$

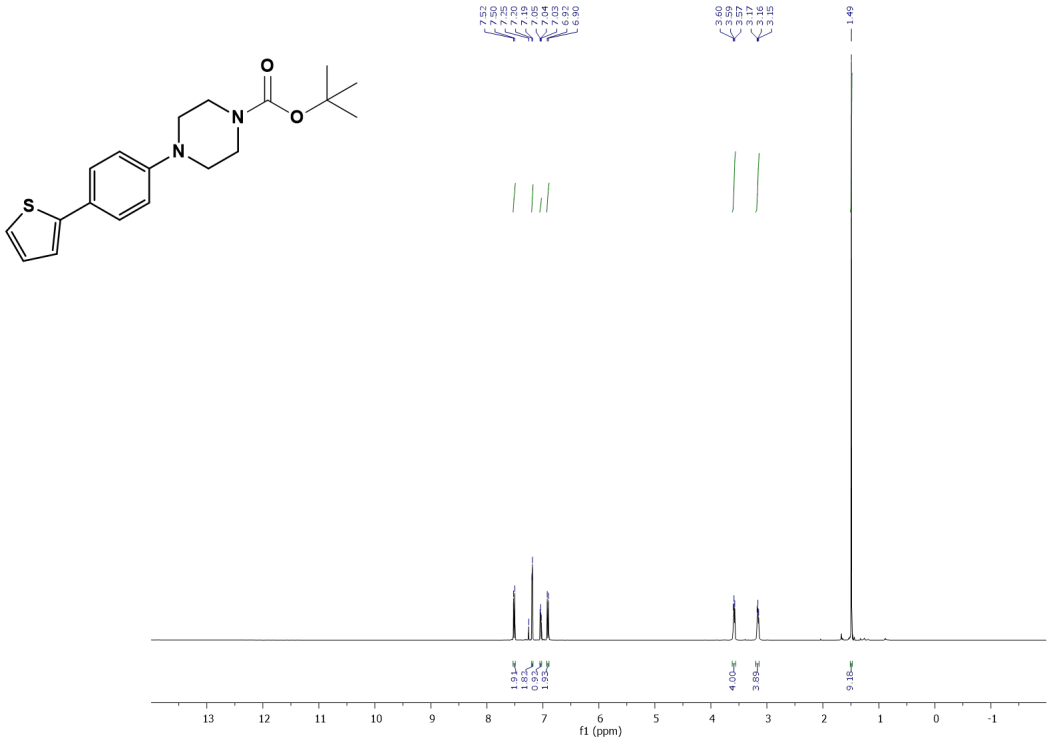


Figure S20: ^{1}H NMR spectrum of *tert*-butyl *N*-(N-(4-thiophene-phenyl)piperazine)carbamate (Entry 11a) in $CDCl_3$

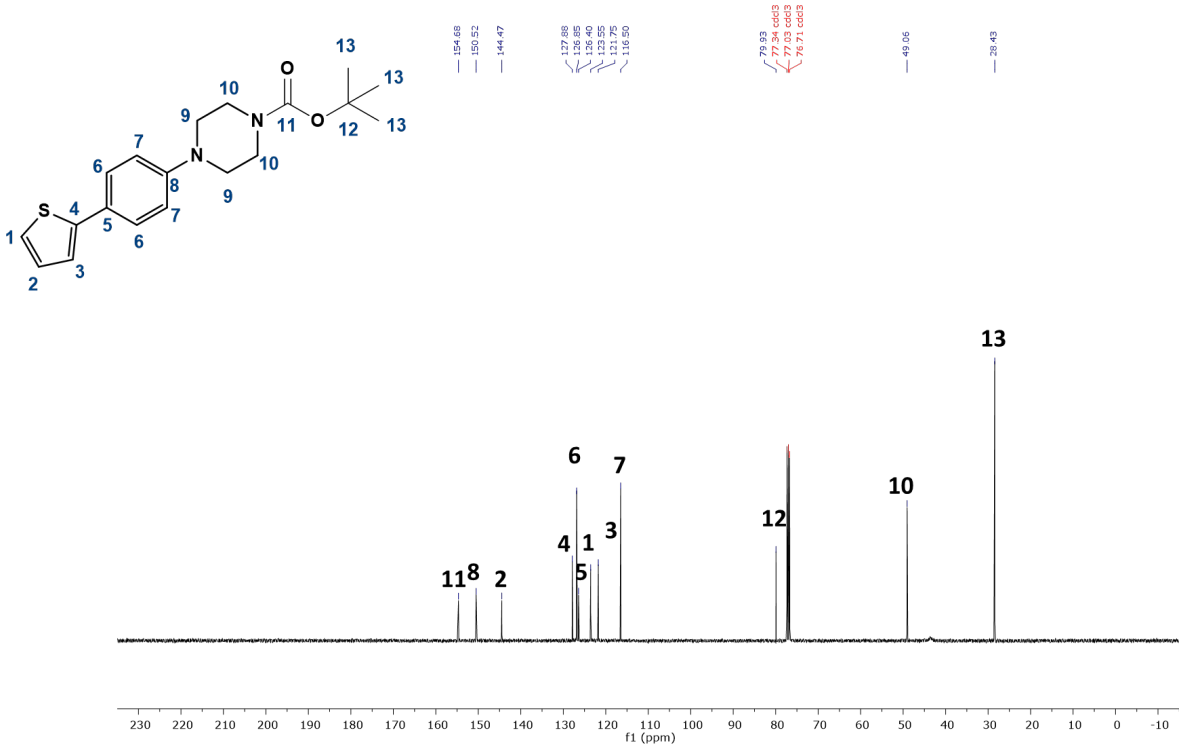


Figure S21: ^{13}C NMR spectrum of *tert*-butyl *N*-(N-(4-thiophene-phenyl)piperazine)carbamate (Entry 11a) in $CDCl_3$

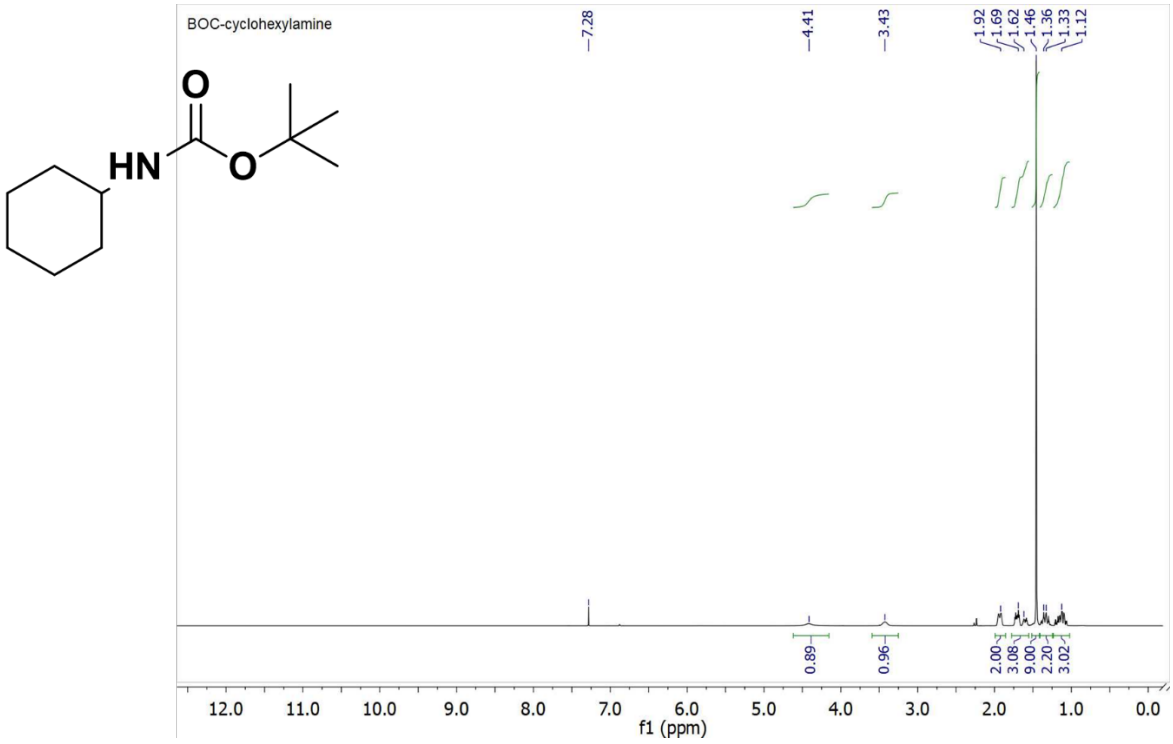


Figure S22: ^{1}H NMR spectrum of *tert*-butyl *N*-(cyclohexyl)carbamate (Entry 12a) in $CDCl_3$

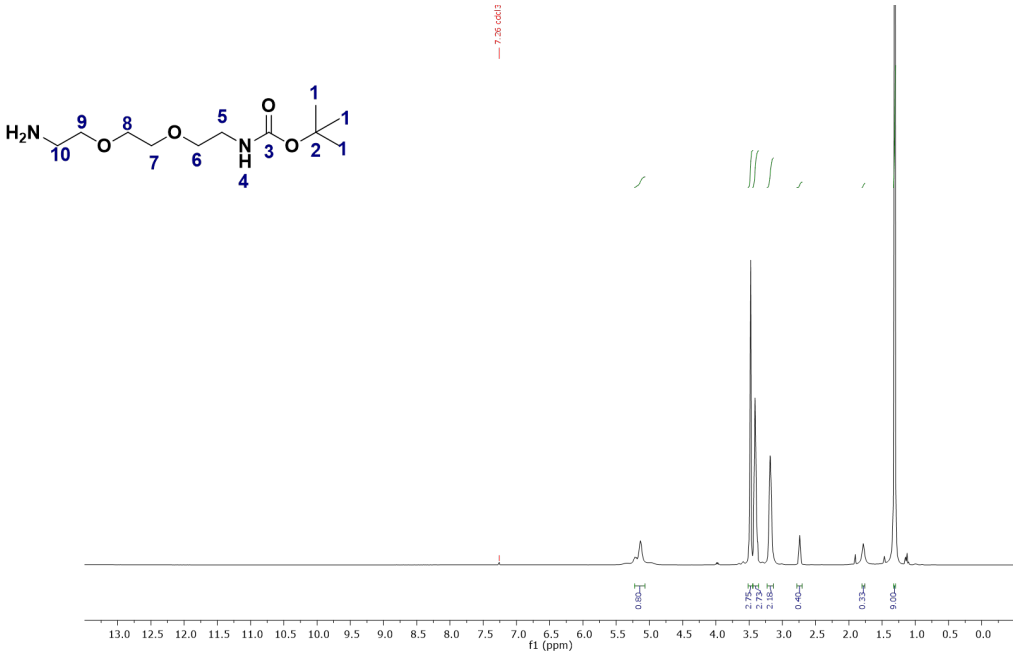


Figure S23: ^{1}H NMR spectrum of *tert*-butyl *N*-(2-[2-(2-aminoethoxy)ethoxy]ethanamine)carbamate (Entry 13a) in $CDCl_3$

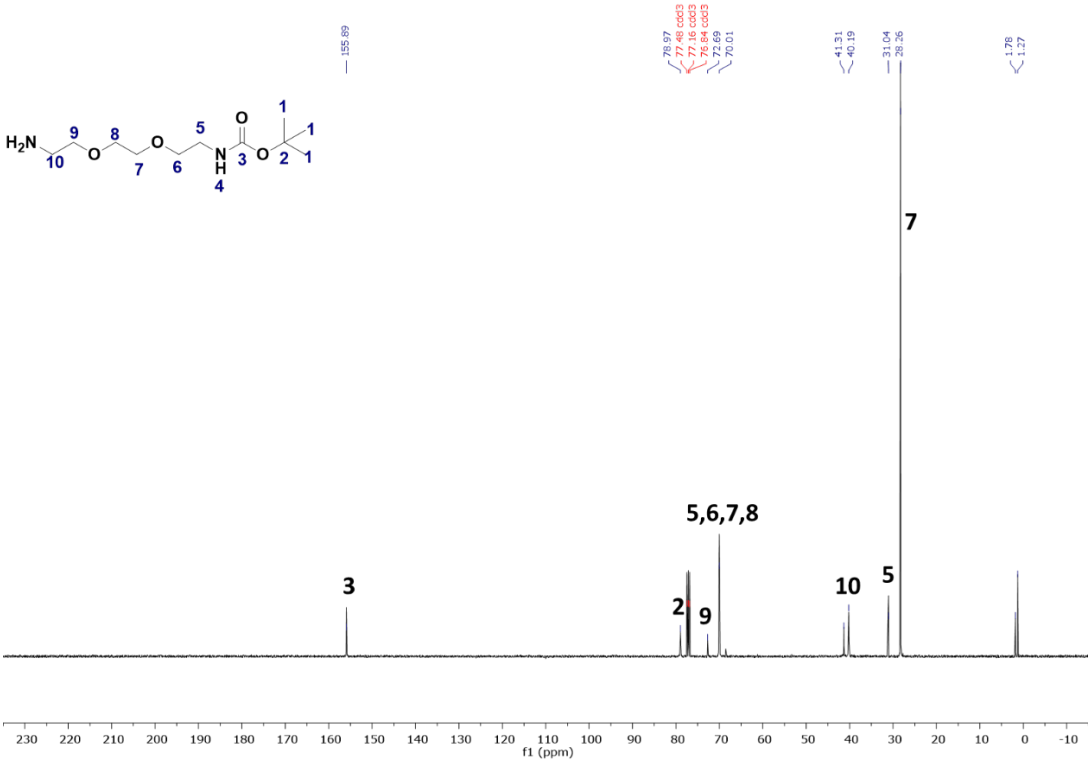


Figure S24: ^{13}C NMR spectrum of *tert*-butyl *N*-(2-[2-(2-aminoethoxy)ethoxy]ethanamine)carbamate (Entry 13a) in $CDCl_3$

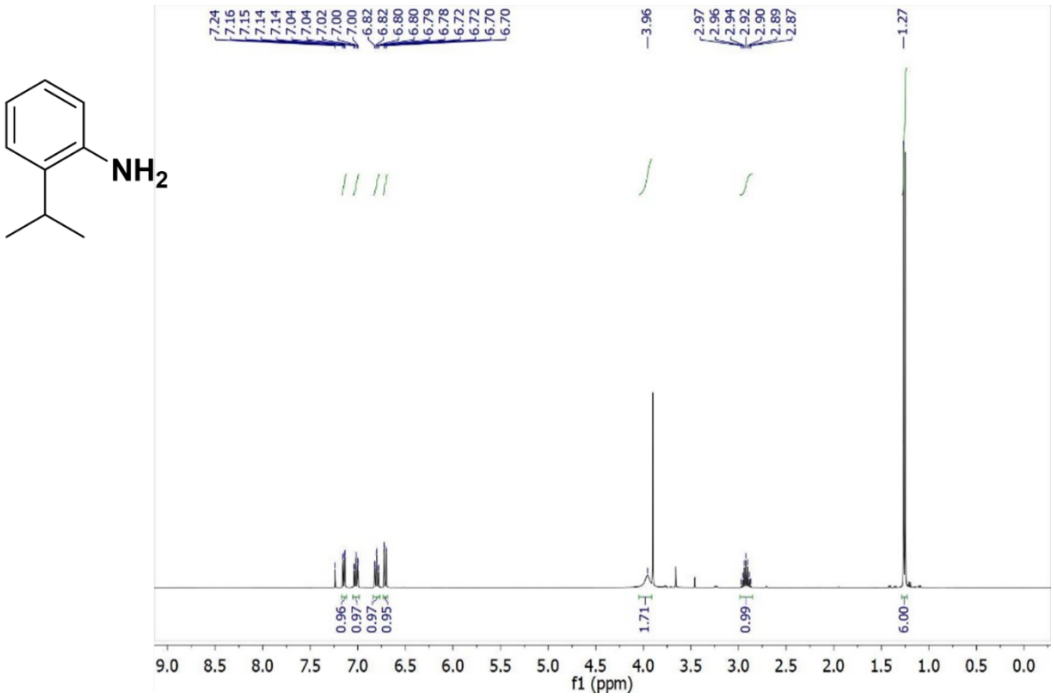


Figure S25: ^{1}H NMR spectrum of 2-isopropylaniline (Entry 1b) in $CDCl_3$

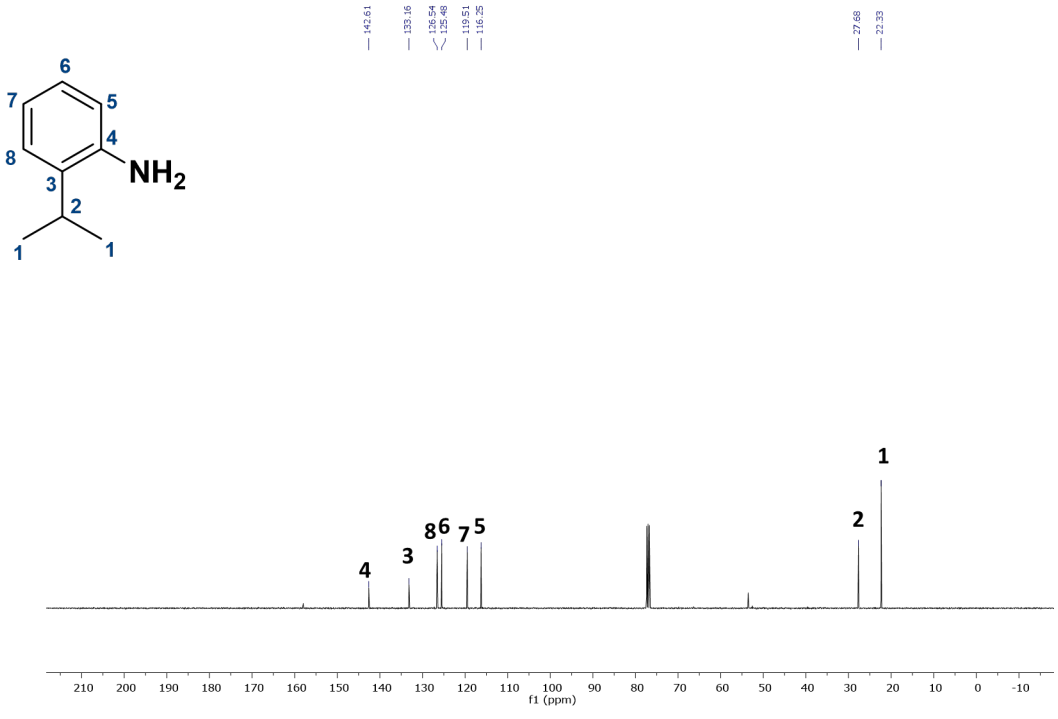


Figure S26: ^{13}C NMR spectrum of 2-isopropylaniline (Entry 1b) in $CDCl_3$

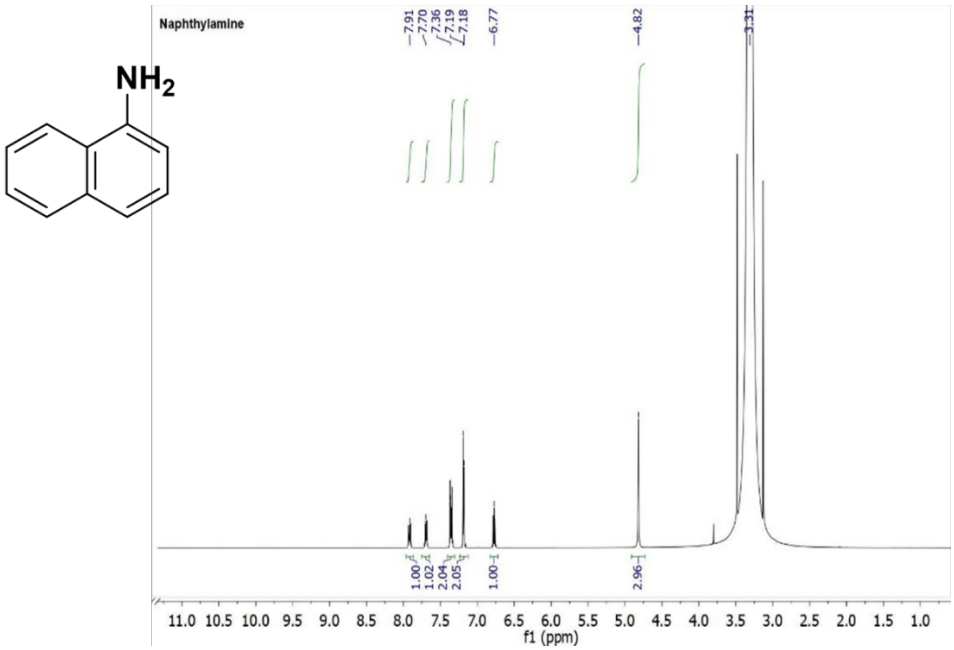


Figure S27: ^{1}H NMR spectrum of Naphthylamine (Entry 2b) in MeOD

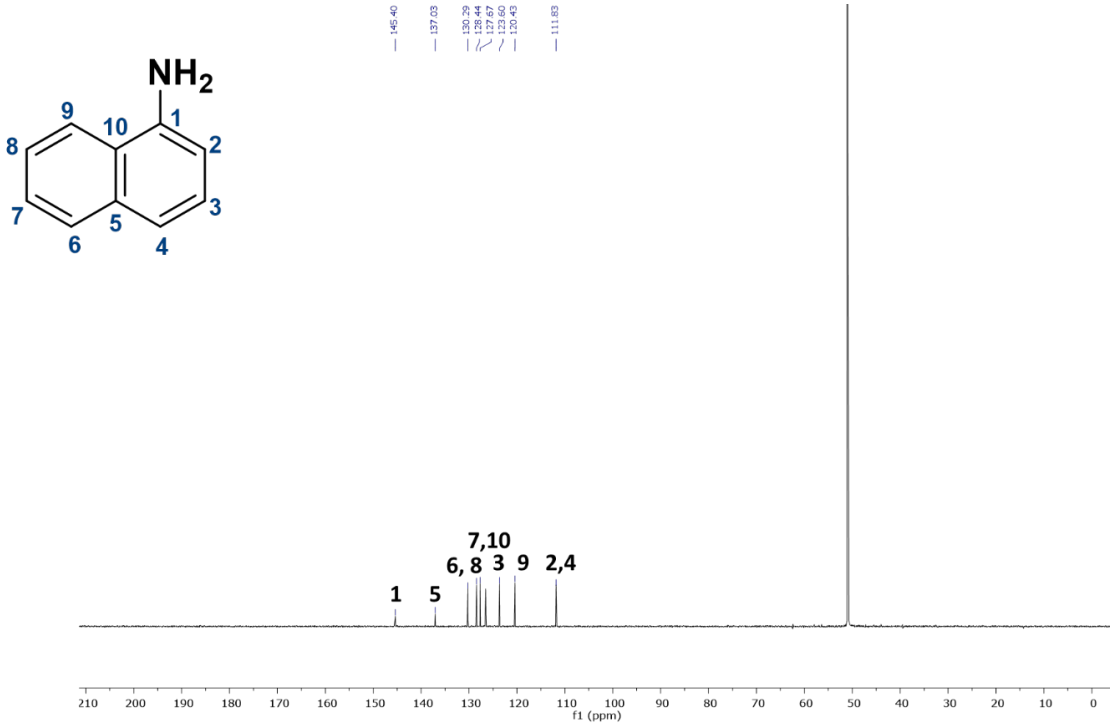


Figure S28: ^{13}C NMR spectrum of Naphthylamine (Entry 2b) in MeOD

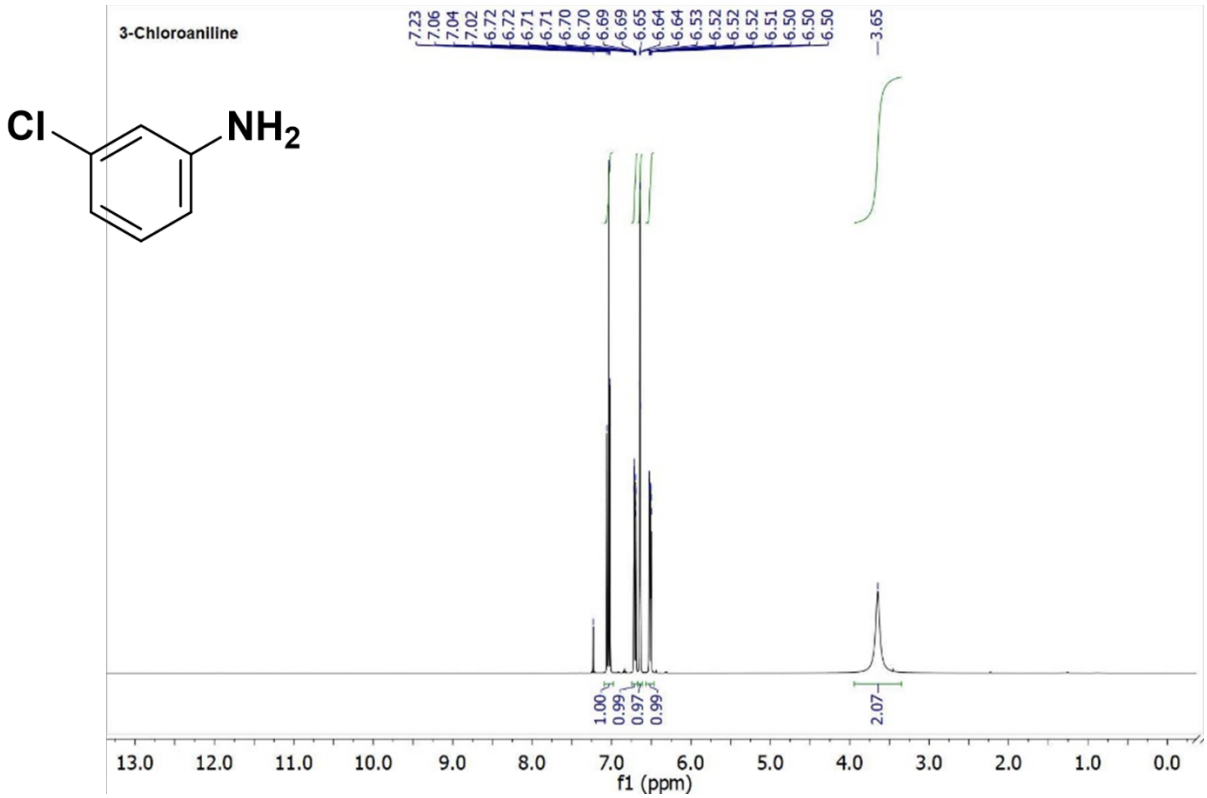


Figure S29: ^{1}H NMR spectrum of 3-chloroaniline (Entry 3b) in $CDCl_3$

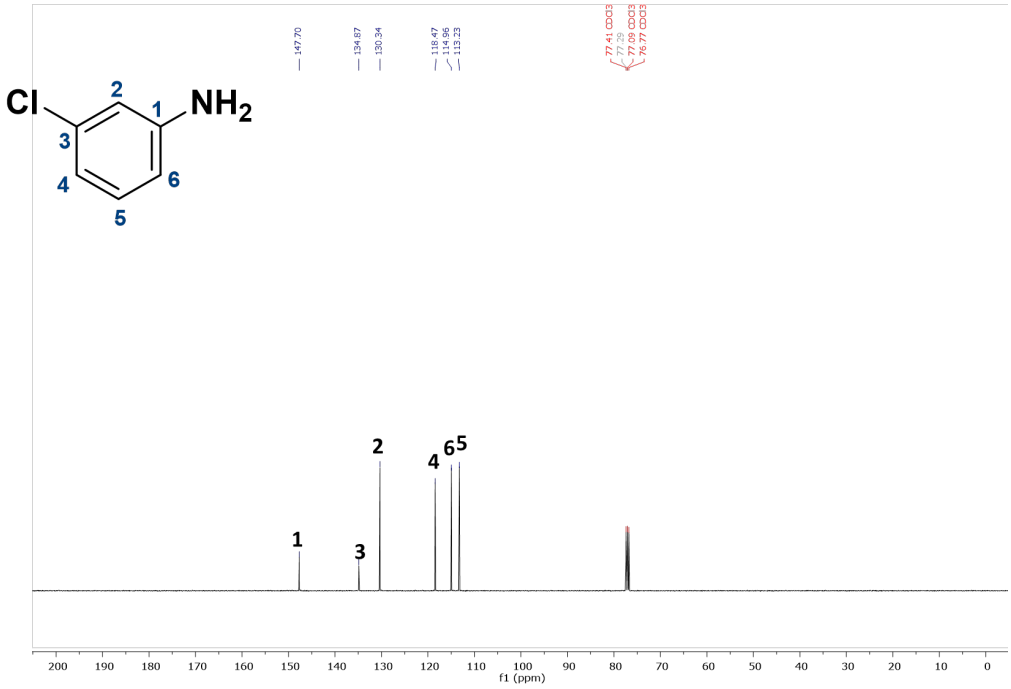


Figure S30: ^{13}C NMR spectrum of 3-chloroaniline (Entry 3b) in $CDCl_3$

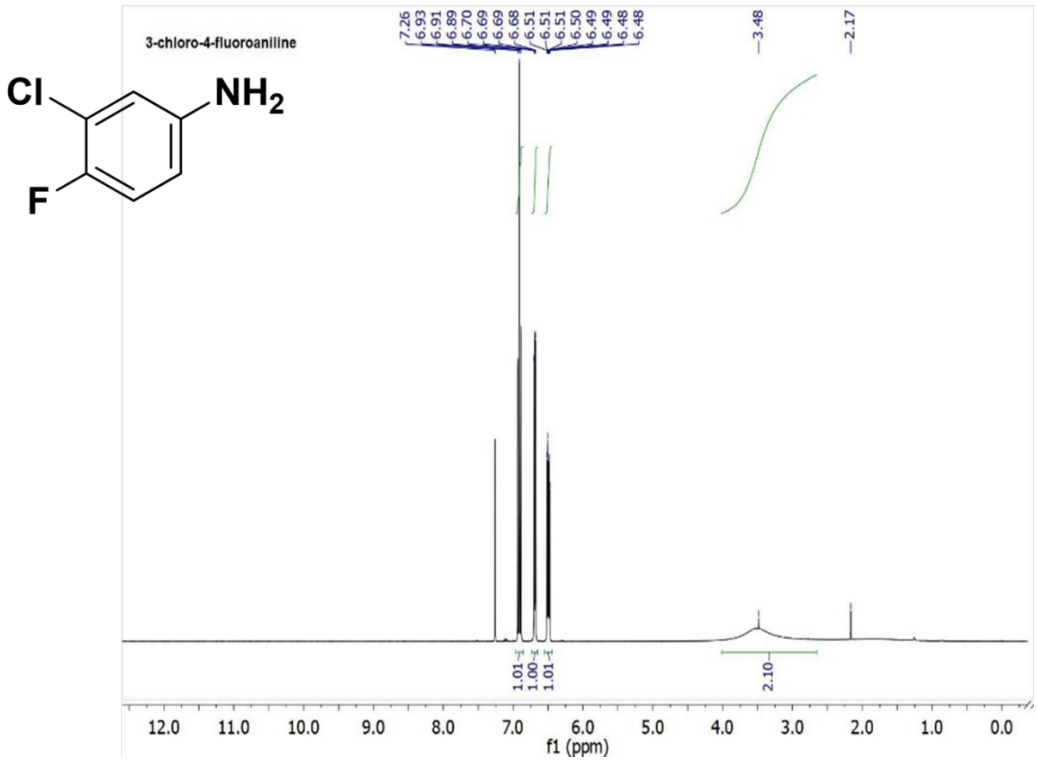


Figure S31: ^{1}H NMR spectrum of 3-chloro-4-flouroaniline (Entry 4b) in $CDCl_3$

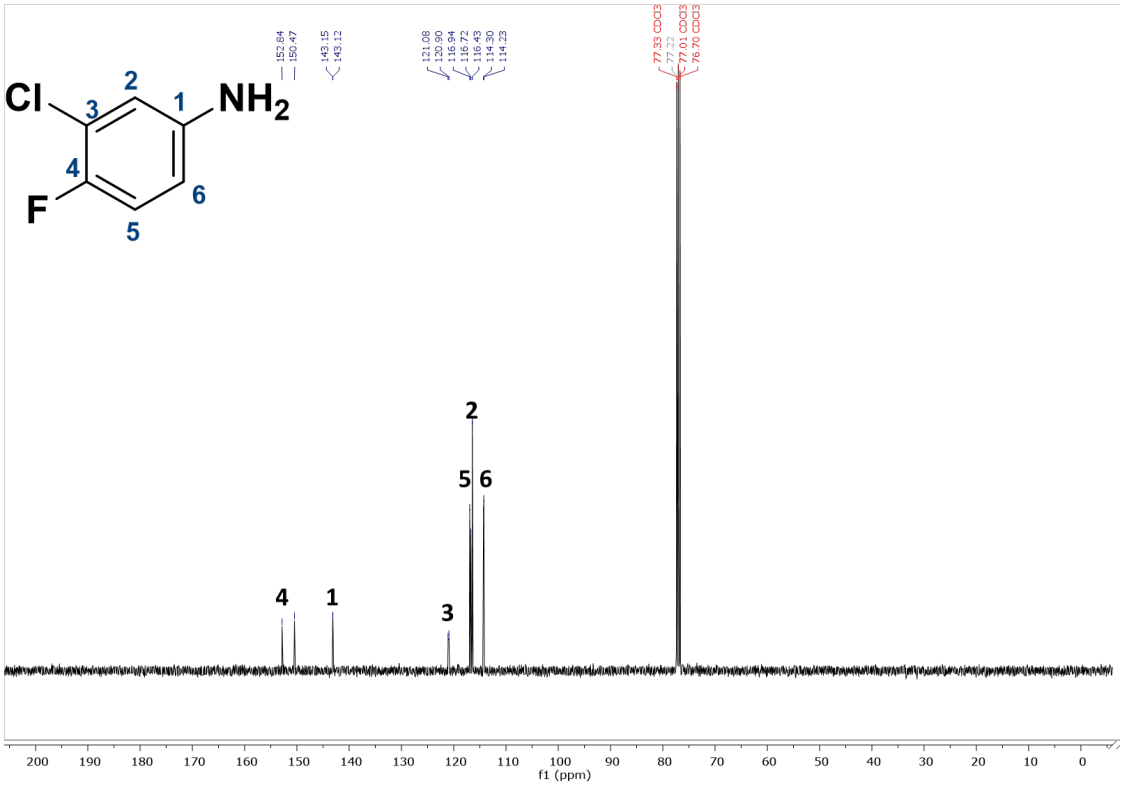


Figure S32: ^{13}C NMR spectrum of 3-chloro-4-flouroaniline (Entry 4b) in $CDCl_3$

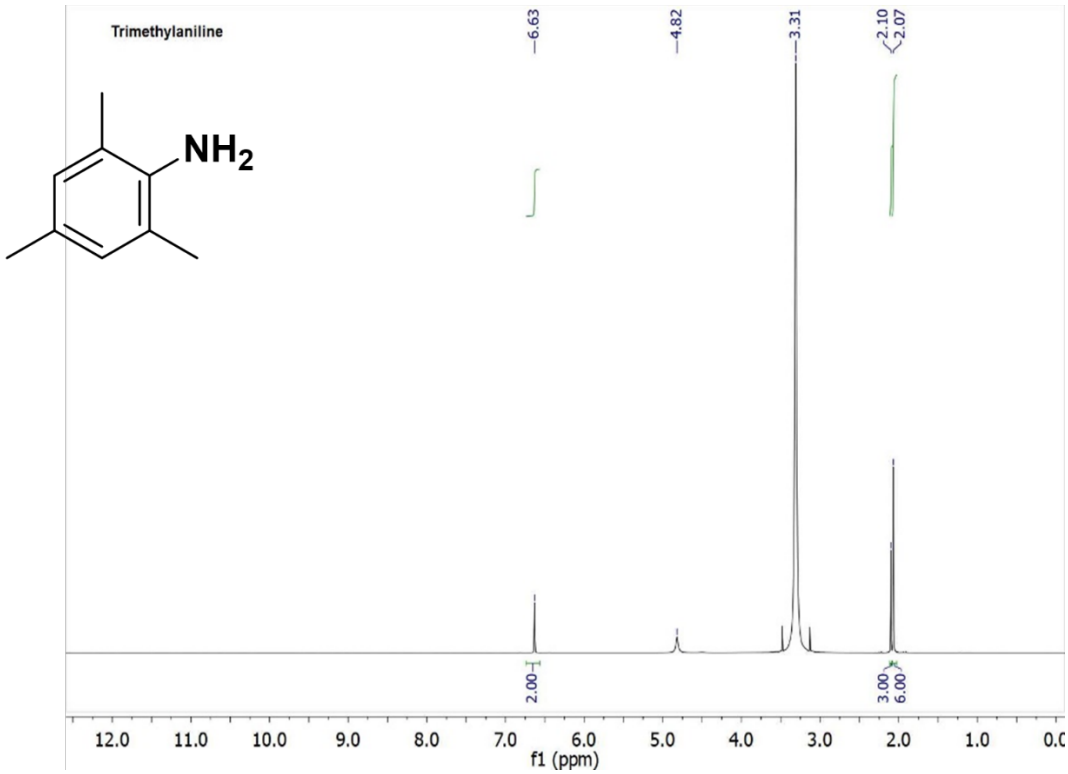


Figure S33: ^{1}H NMR spectrum of 2,4,6-trimethylaniline (Entry 5b) in $CDCl_3$

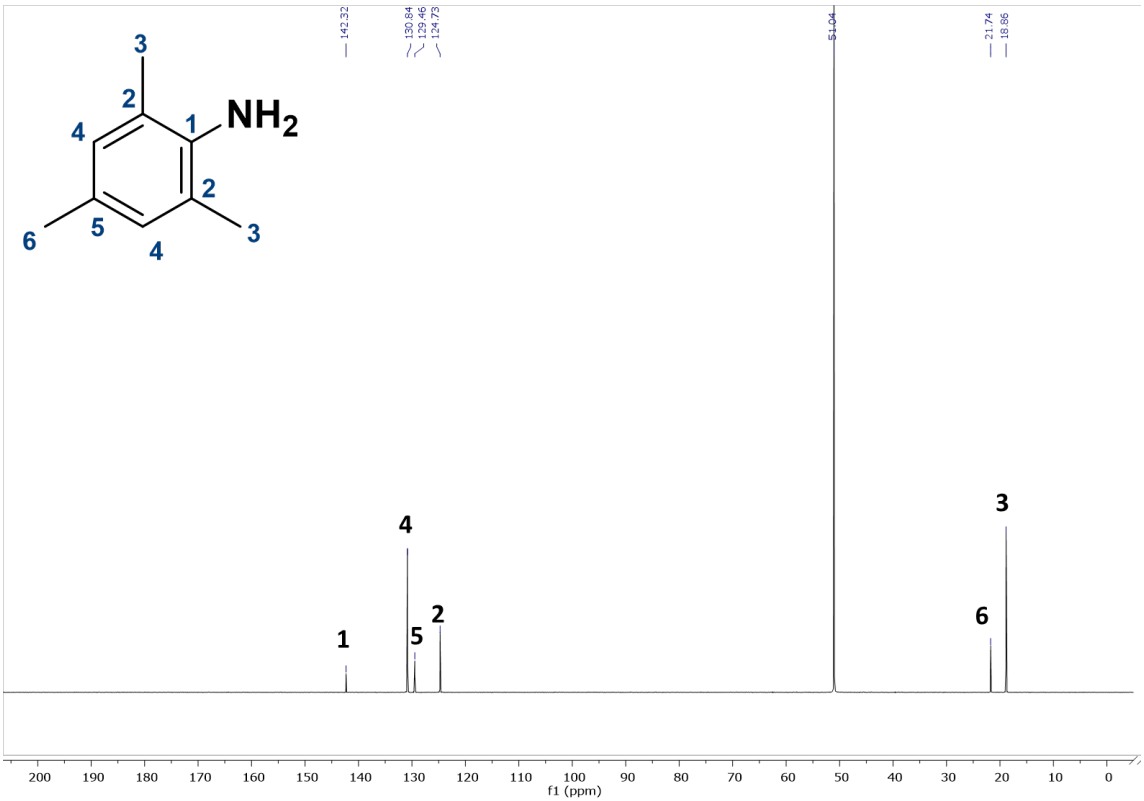


Figure S34: ^{13}C NMR spectrum of 2,4,6-trimethylaniline (Entry 5b) in $CDCl_3$

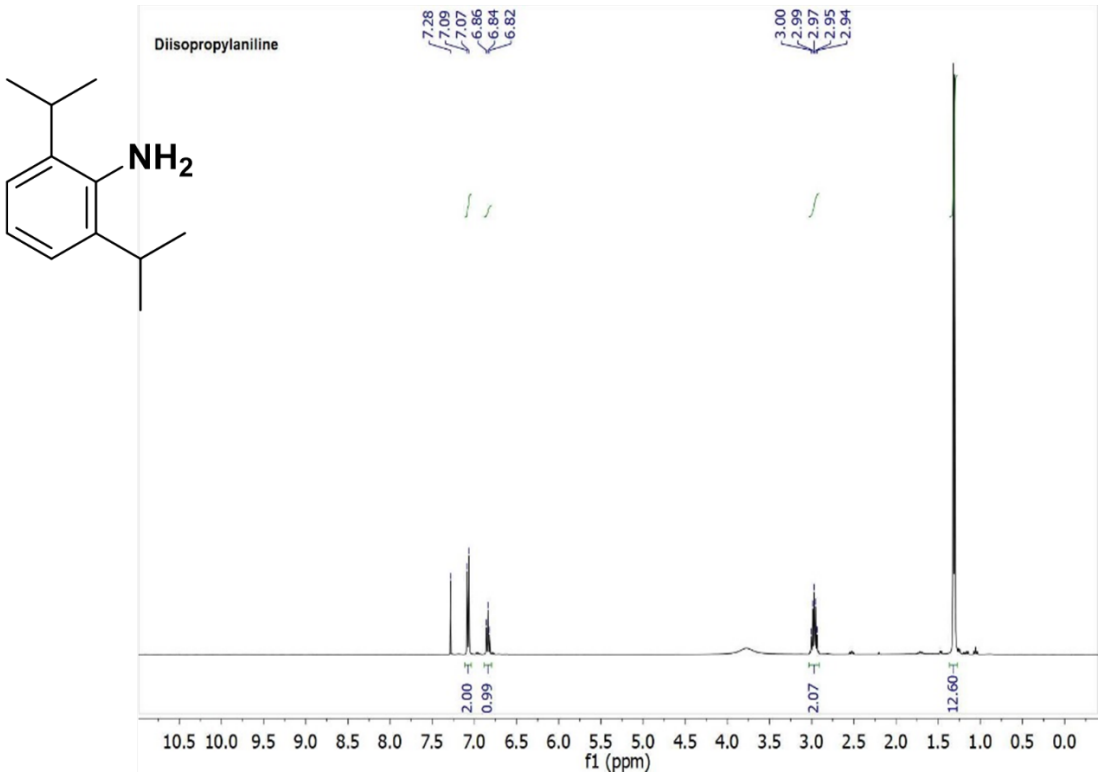


Figure S35: ^{1}H NMR spectrum of 2,6-diisopropylaniline (Entry 6b) in $CDCl_3$

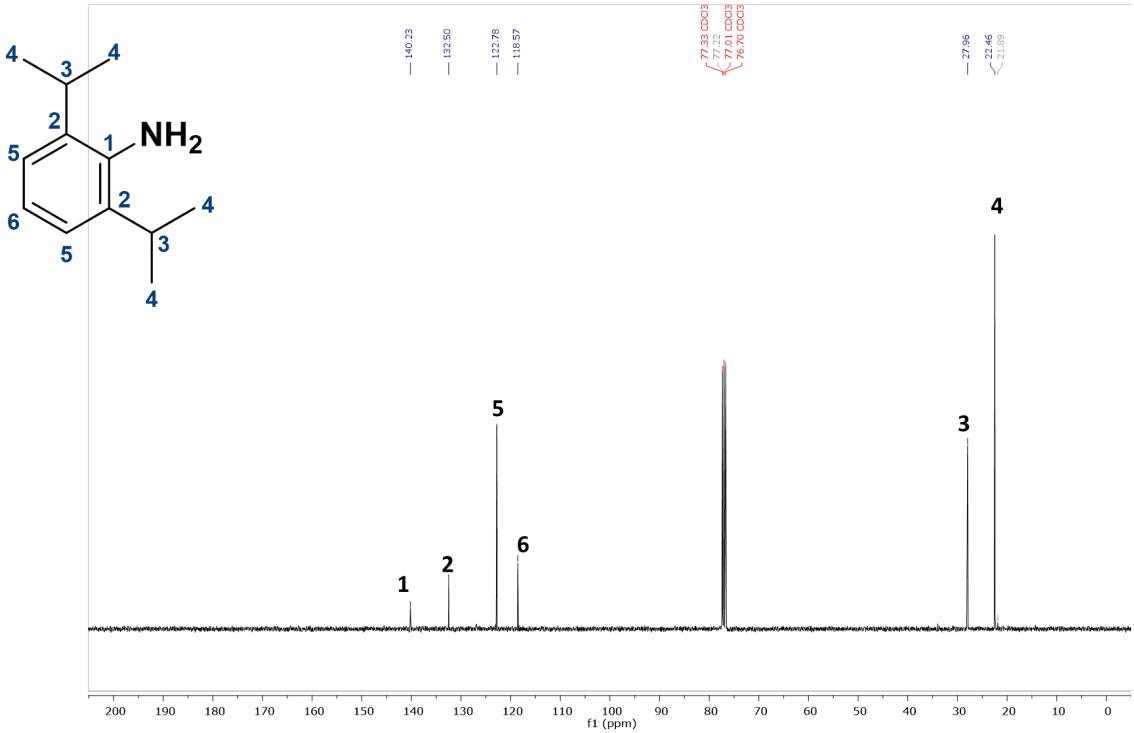


Figure S36: ^{13}C NMR spectrum of 2,6-diisopropylaniline (Entry 6b) in $CDCl_3$

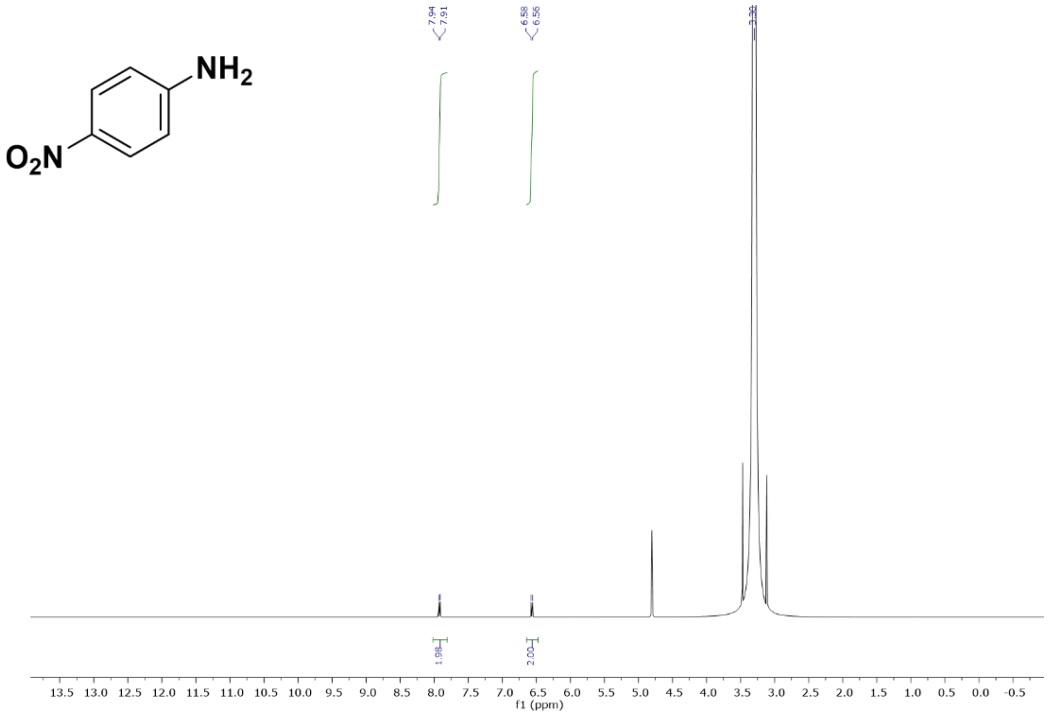


Figure S37: ^{1}H NMR spectrum of 4-Nitroaniline in $CDCl_3$ (Entry 7b)

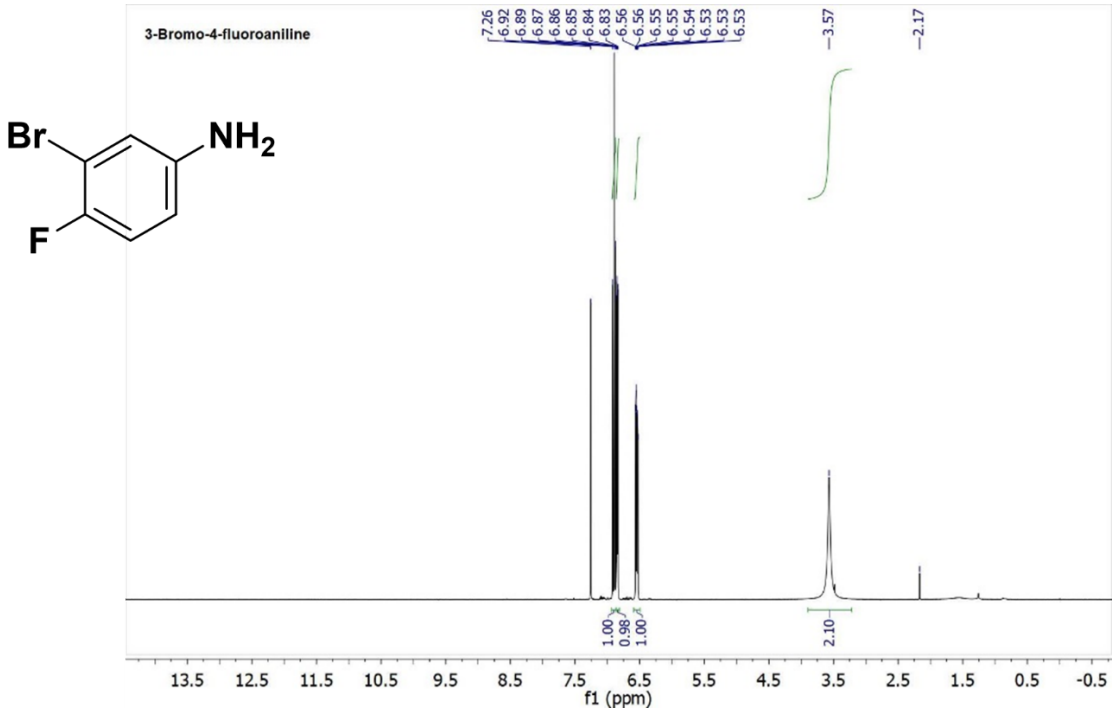


Figure S38: ^{1}H NMR spectrum of 3-bromo-4-fluoroaniline (Entry 8b) in $CDCl_3$

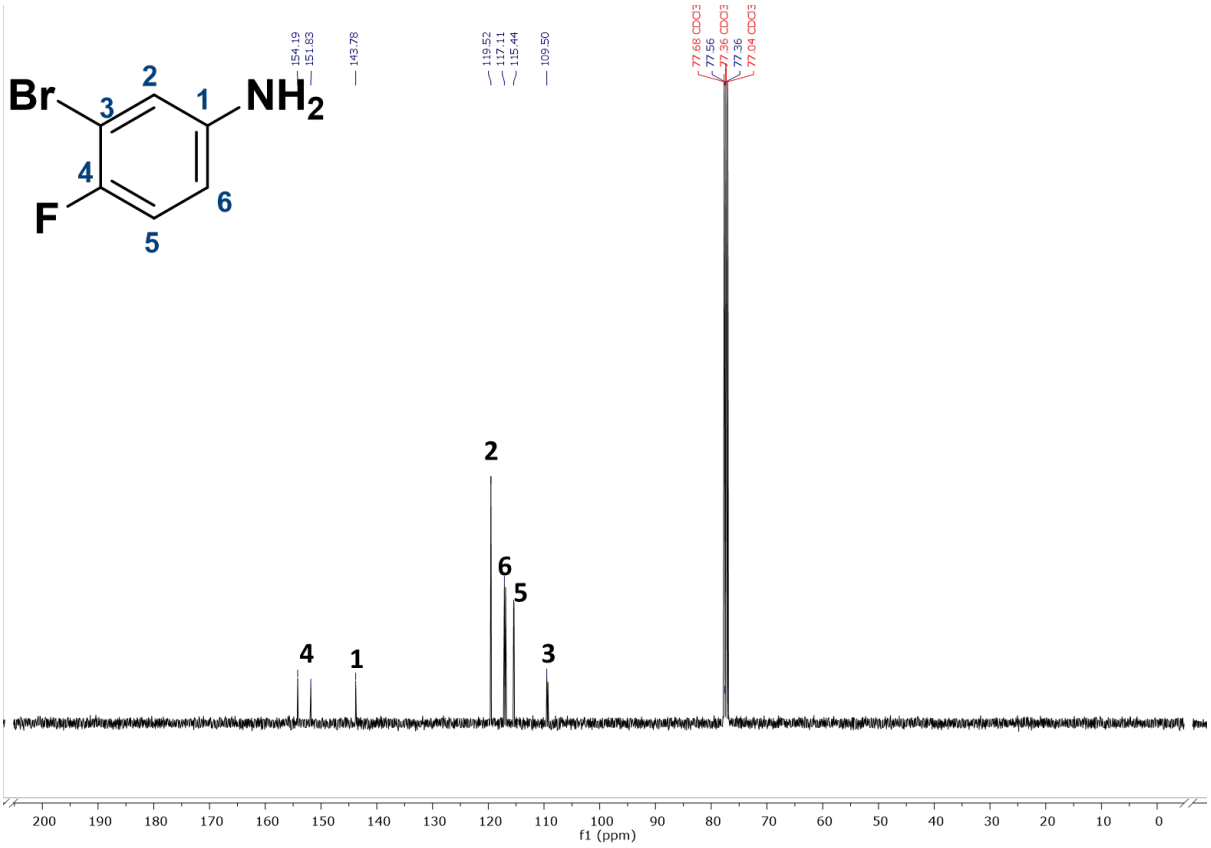


Figure S39: ^{13}C NMR spectrum of 3-bromo-4-fluoroaniline (Entry 8b) in $CDCl_3$

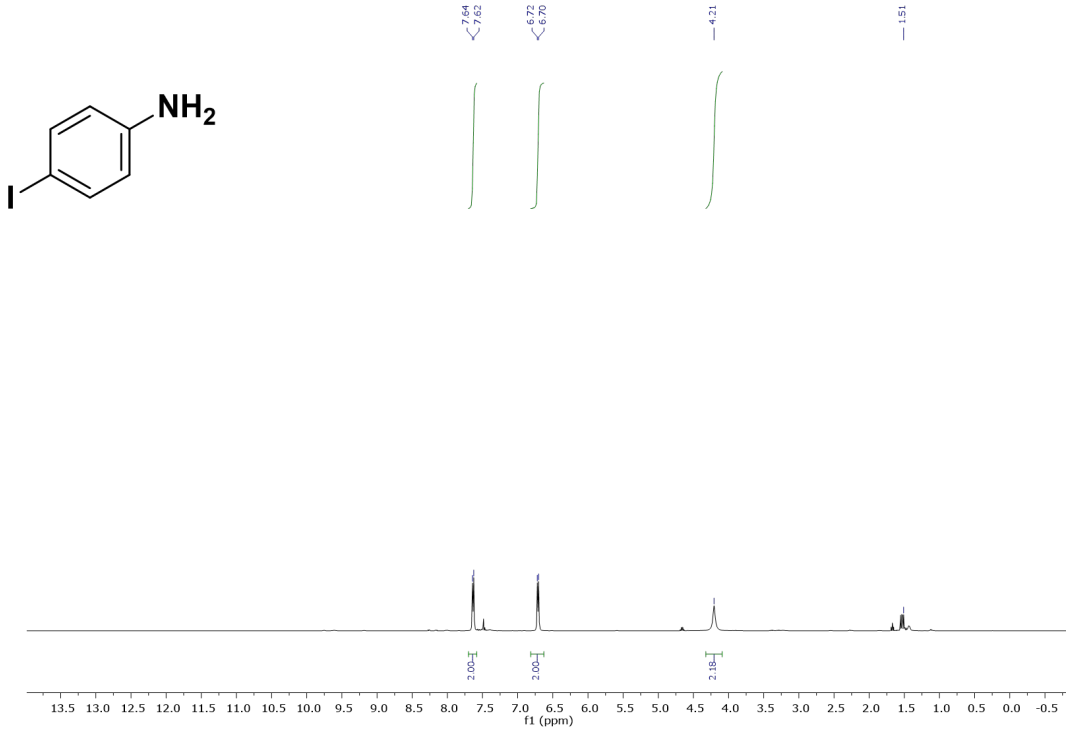


Figure S40: ^{1}H NMR spectrum of 4-iodoaniline (Entry 9b) in $CDCl_3$

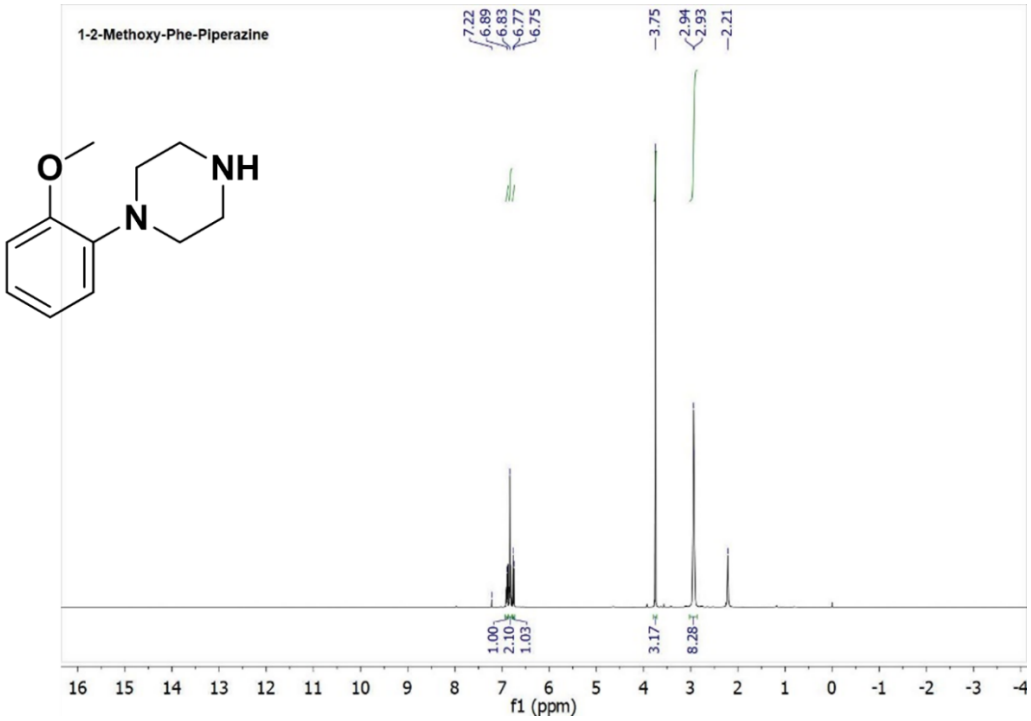


Figure S41: ^{1}H NMR spectrum of N-(2-Methoxyphenyl)piperazine (Entry 10b) in $CDCl_3$

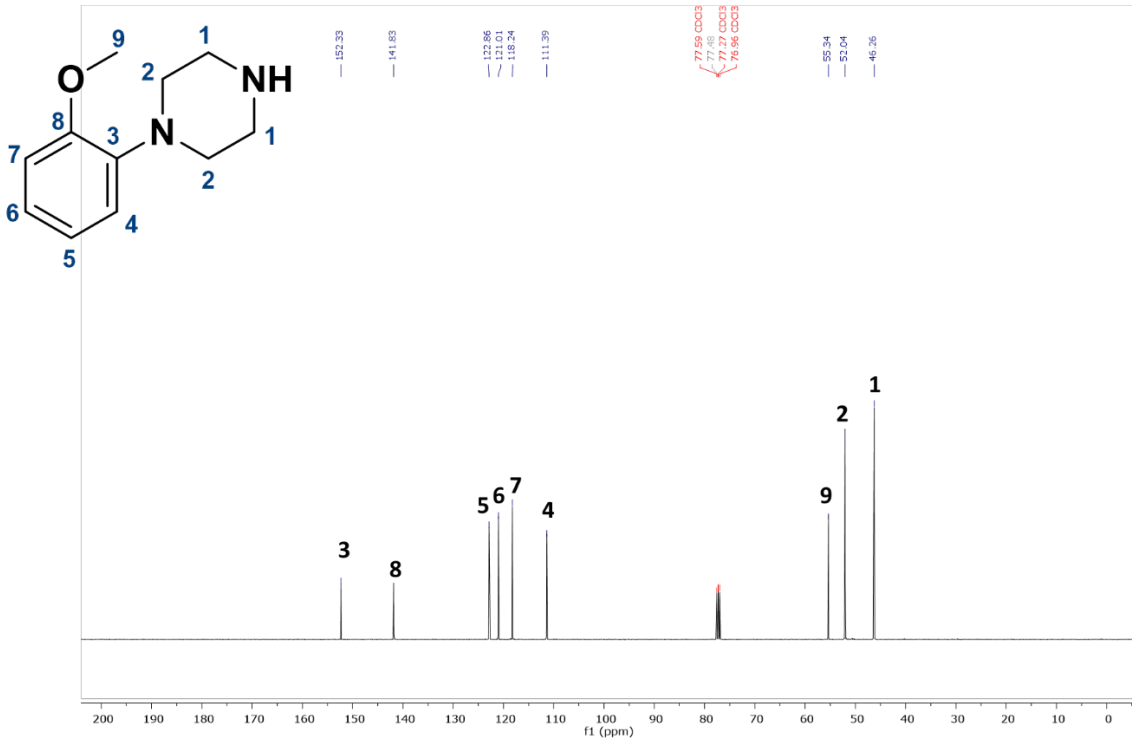


Figure S42: ^{13}C NMR spectrum of N-(2-Methoxyphenyl)piperazine (Entry 10b) in $CDCl_3$

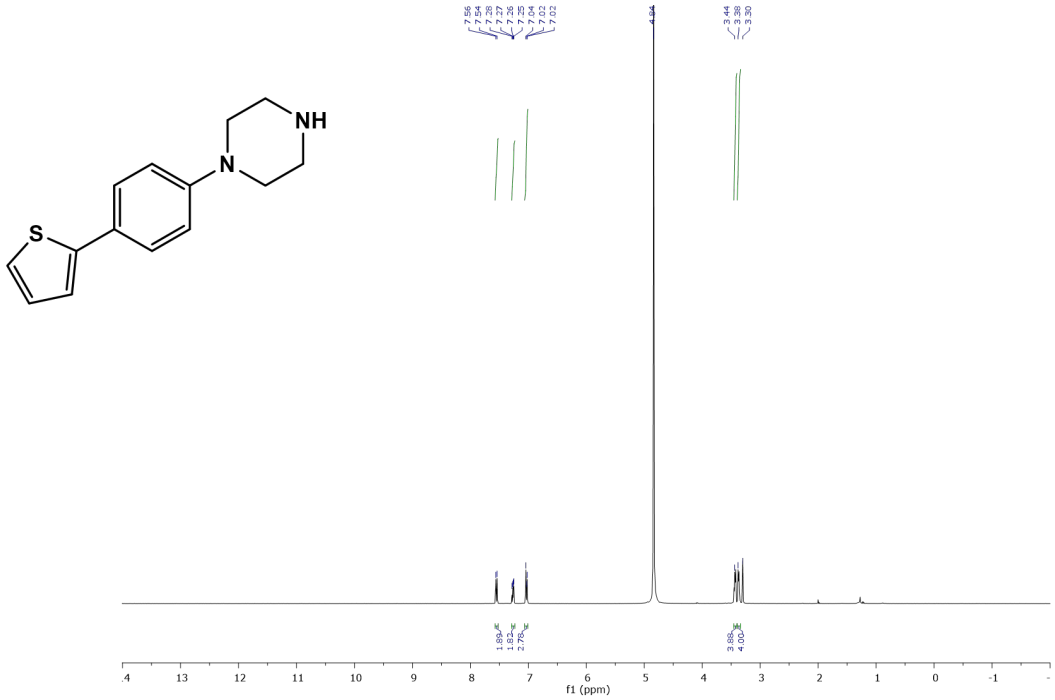


Figure S43: ^{1}H NMR spectrum of N-(4-thiophene-phenyl)piperazine (Entry 11b) in $CDCl_3$

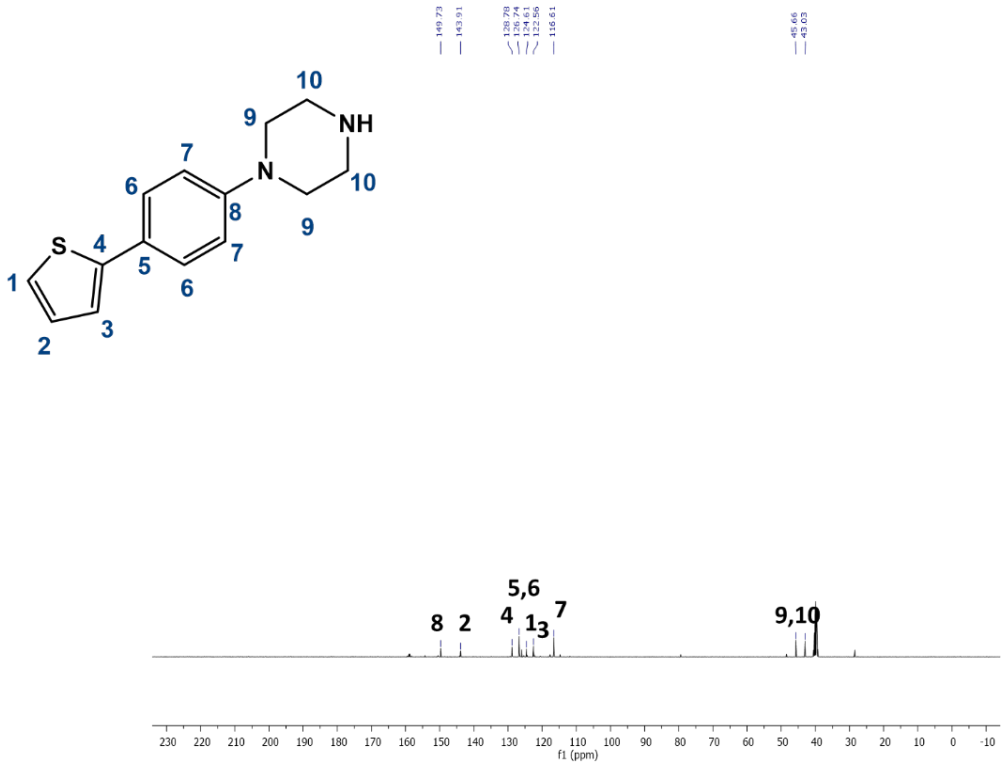


Figure S44: ^{13}C NMR spectrum of N-(4-thiophene-phenyl)piperazine (Entry 11b) in $CDCl_3$

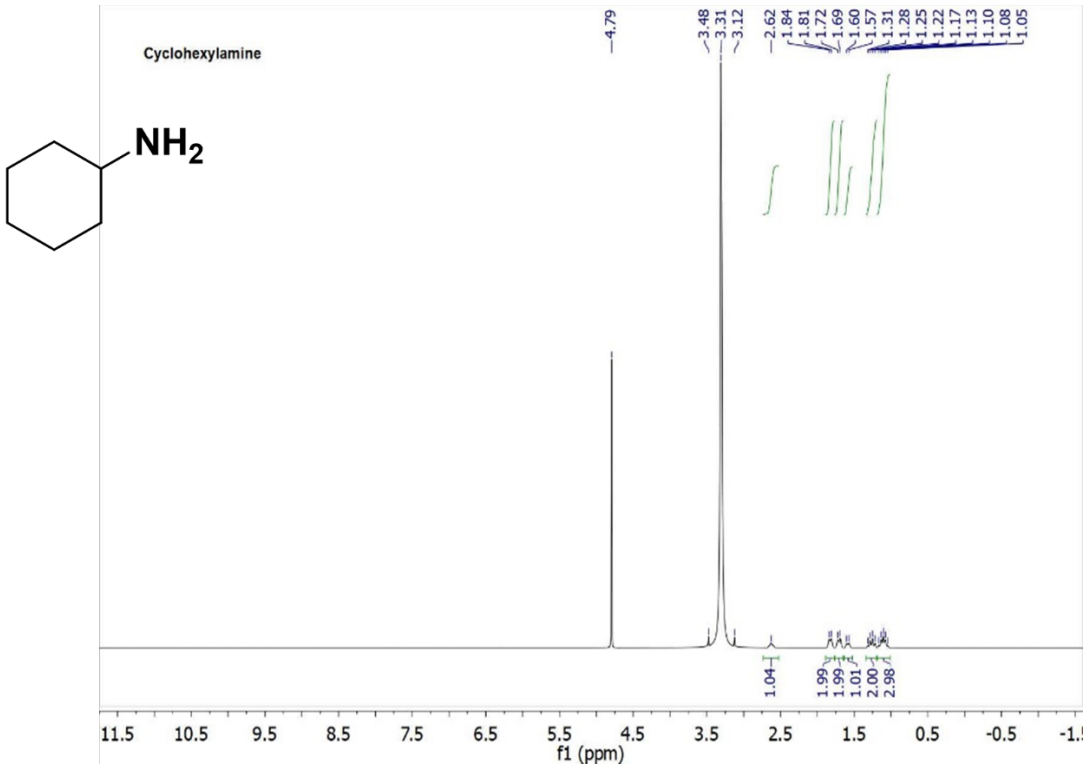


Figure S45: ^{1}H NMR spectrum of cyclohexylamine (Entry 12b) in $CDCl_3$

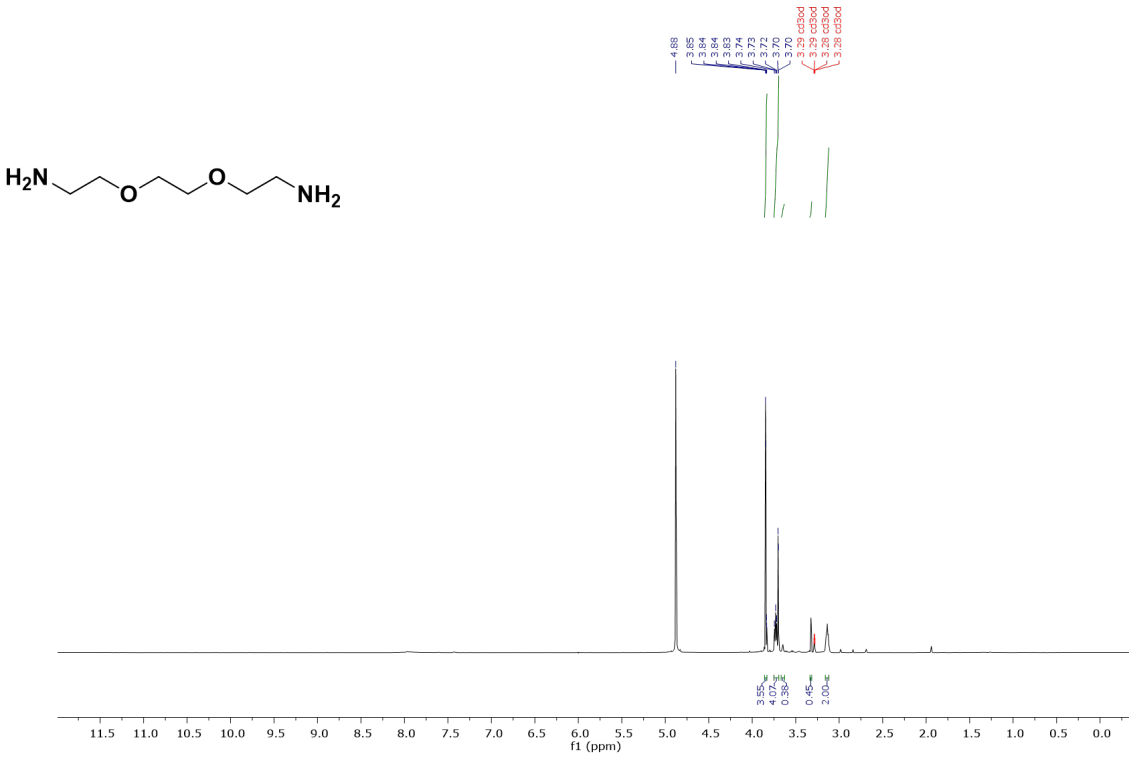


Figure S46: ^{1}H NMR spectrum of 2-[2-(2-aminoethoxy)ethoxy]ethanamine (Entry 13b) in $CDCl_3$

EC1 Deprotection Utilizing Oxalyl Chloride

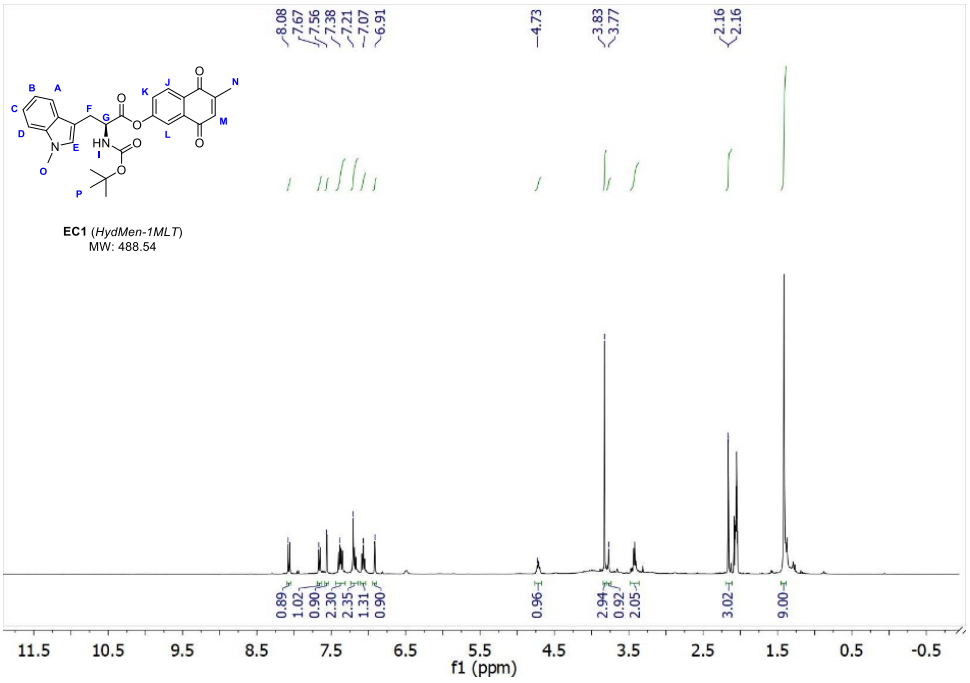


Figure S47: ^{1}H NMR spectrum of compound **4**

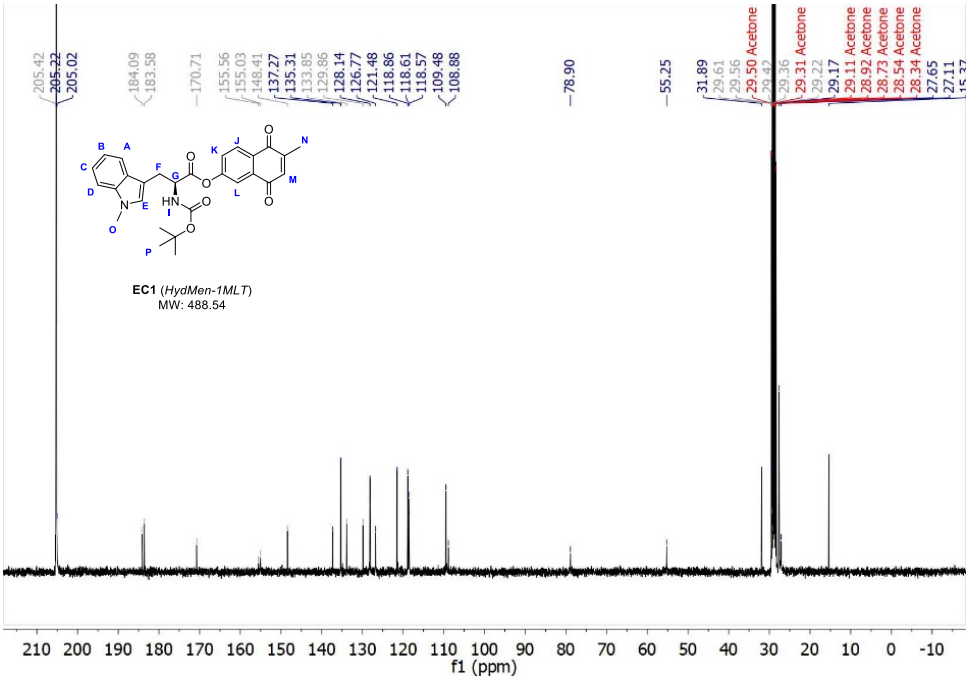


Figure S48: ^{13}C NMR spectrum of compound **4**

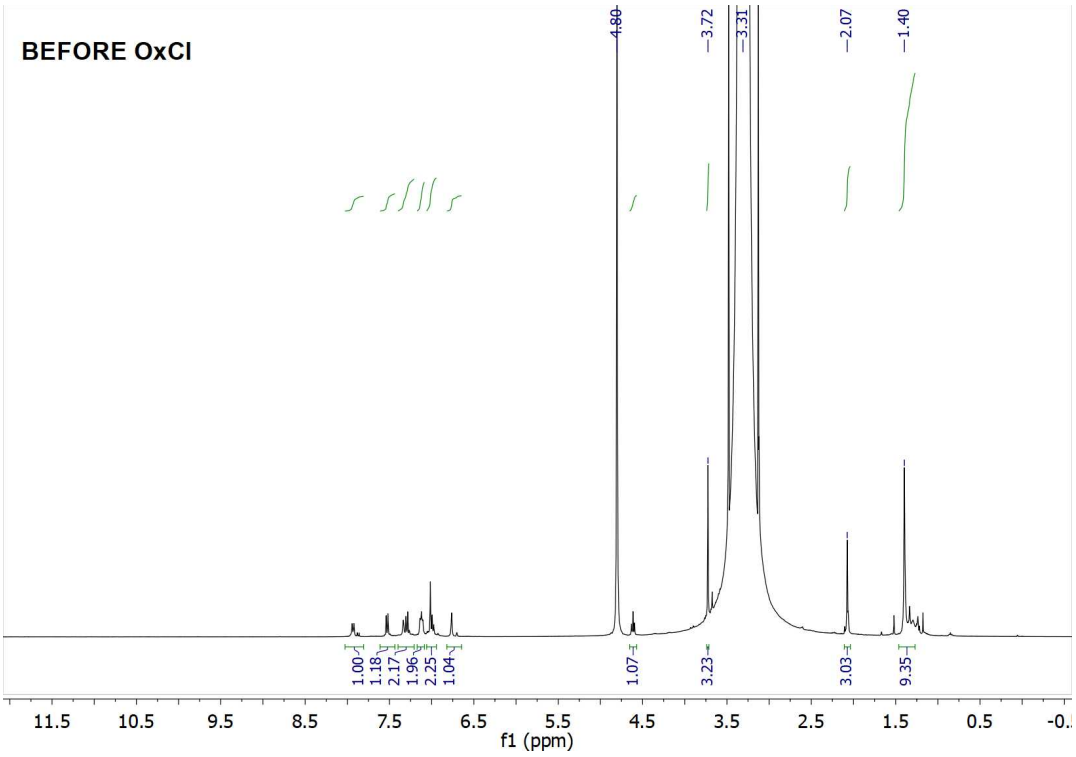


Figure S48: Reaction Monitoring via NMR (Before OxCl)

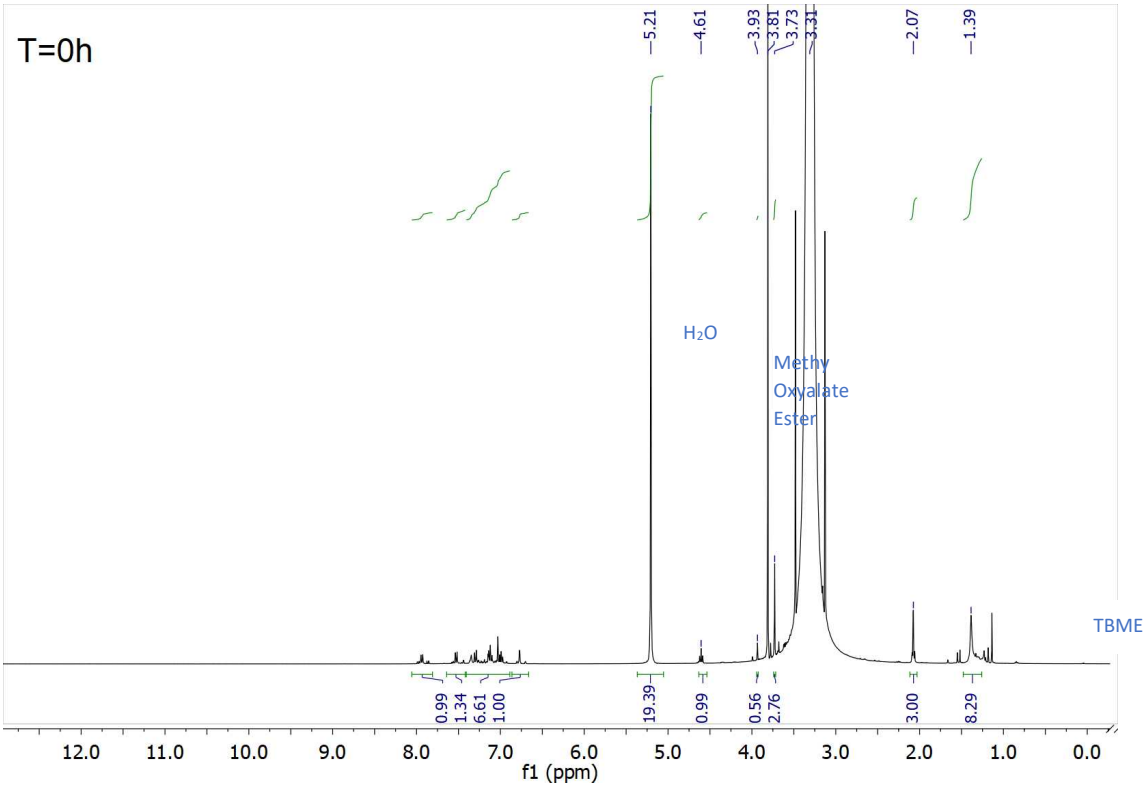


Figure S49: Reaction Monitoring via NMR (T = 0)

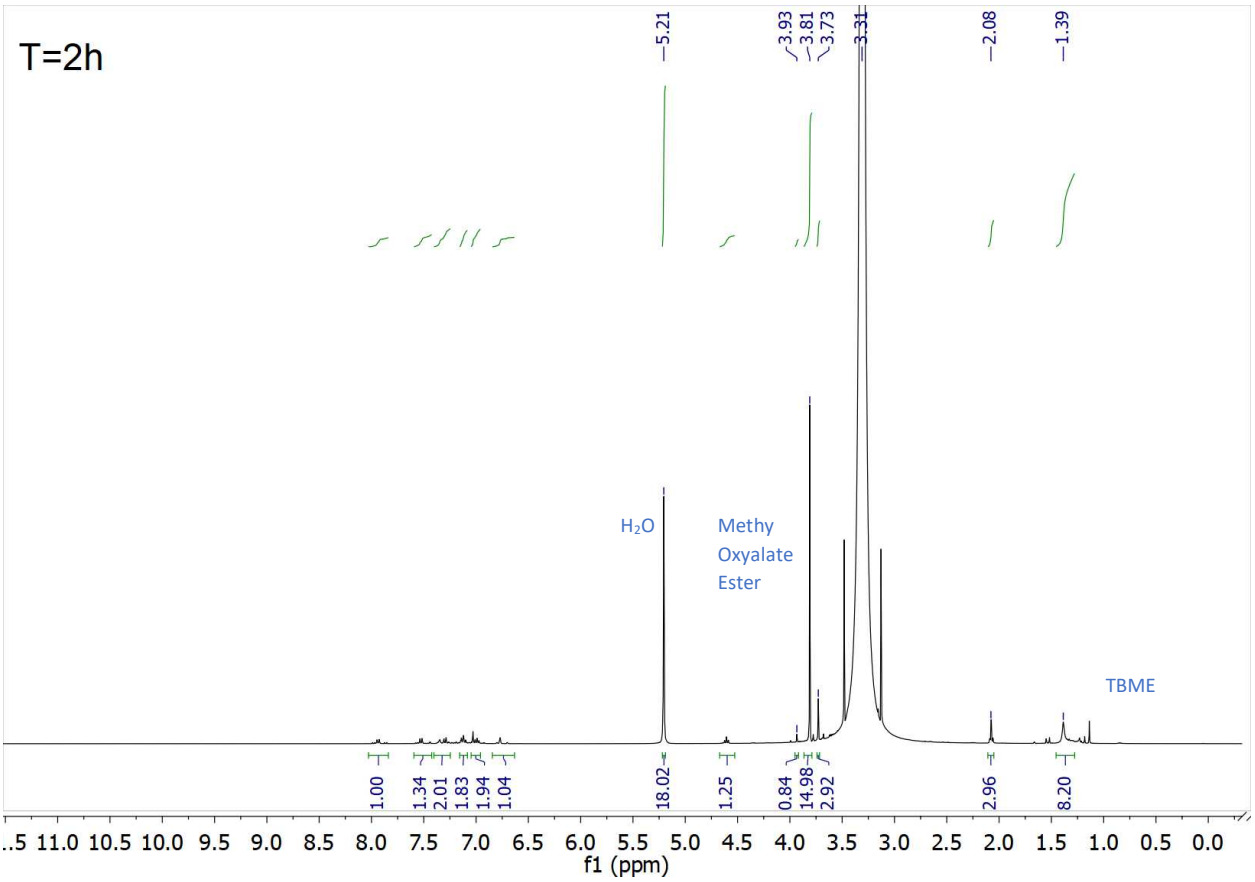


Figure S50: Reaction Monitoring via NMR (T = 2h)

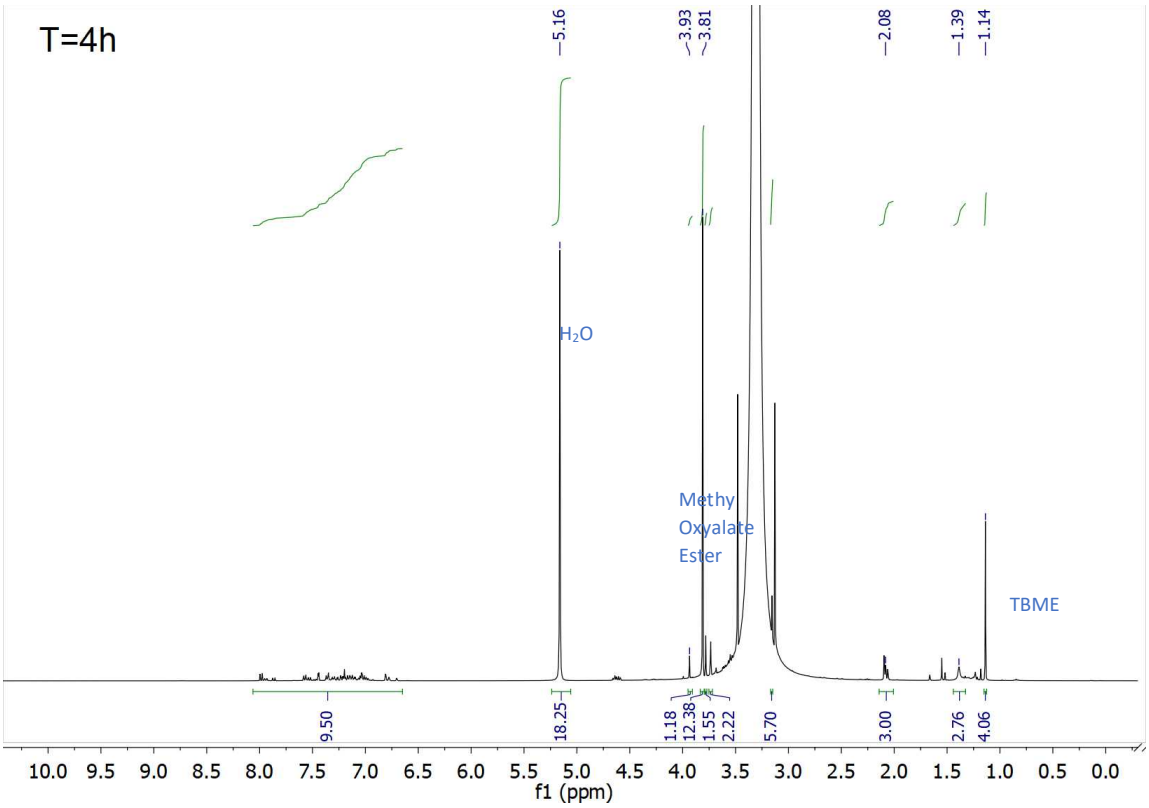


Figure S51: Reaction Monitoring via NMR (T = 4h)

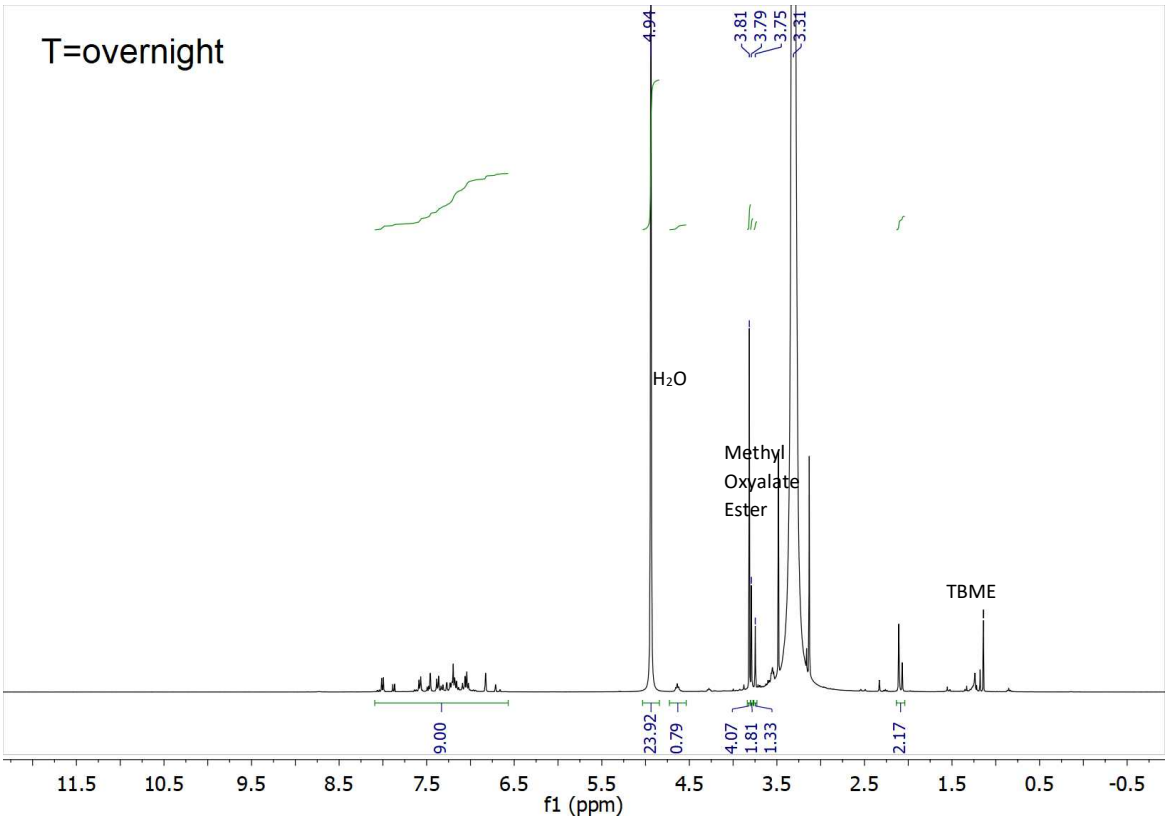
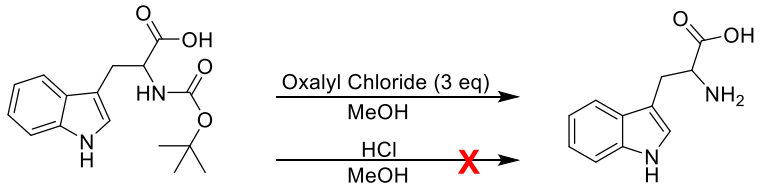


Figure S52: Reaction Monitoring via NMR (T = Overnight)



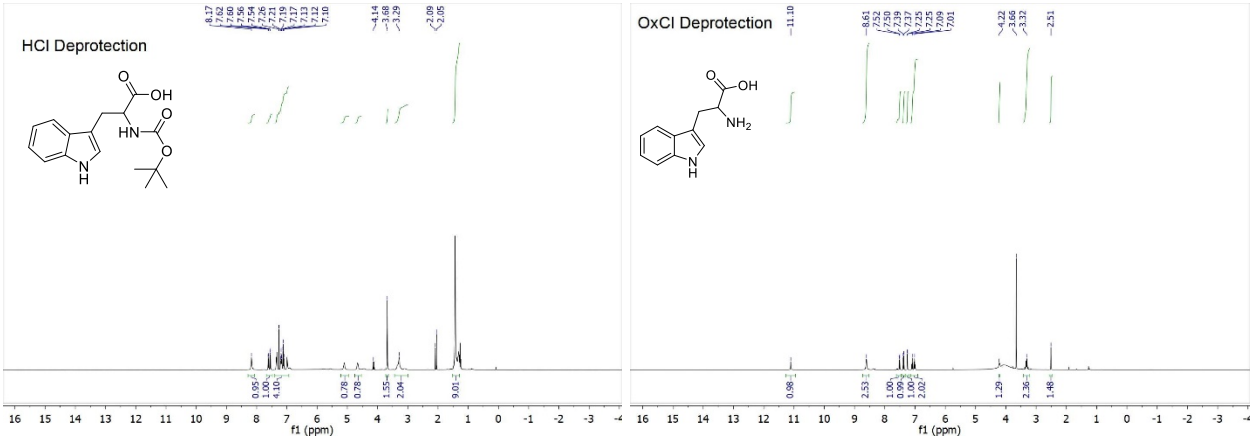


Figure S53: Determination of HCl effectiveness in Deprotection

X-ray Crystal Structure Data

Table S1: X-ray Parameters

X-ray Structural Data and Crystal Refinement

	EC1
Empirical Formula	$C_{28}H_{28}N_2O_6$
Molecular Weight (g/mol)	458.54
Temperature (K)	90.0(2)
X-ray Radiation (Å)	CuKα (1.54178 Å)
Crystal System, Space Group	Monoclinic, C2
Unit Cell Dimensions (Å, °)	a = 23.791(2) Å alpha = 90 b = 6.7169 (6) Å beta =103.374(4) c = 17.0032(16) Å gamma = 90
Volume	2643.5(4) $Å^3$
Z	2
Absorption Coefficient	1.663 mm^{-1}
F(000)	1116.0
Crystal Size (mm)	0.300 x 0.030 x 0.020
Theta Range	2.671 to 74.520

Completeness to Theta = 67.679	97.6 %
F^2	1.033
Final R indices [I>2sigma(I)]	R1 = 0.0380, wR2 = 0.0975

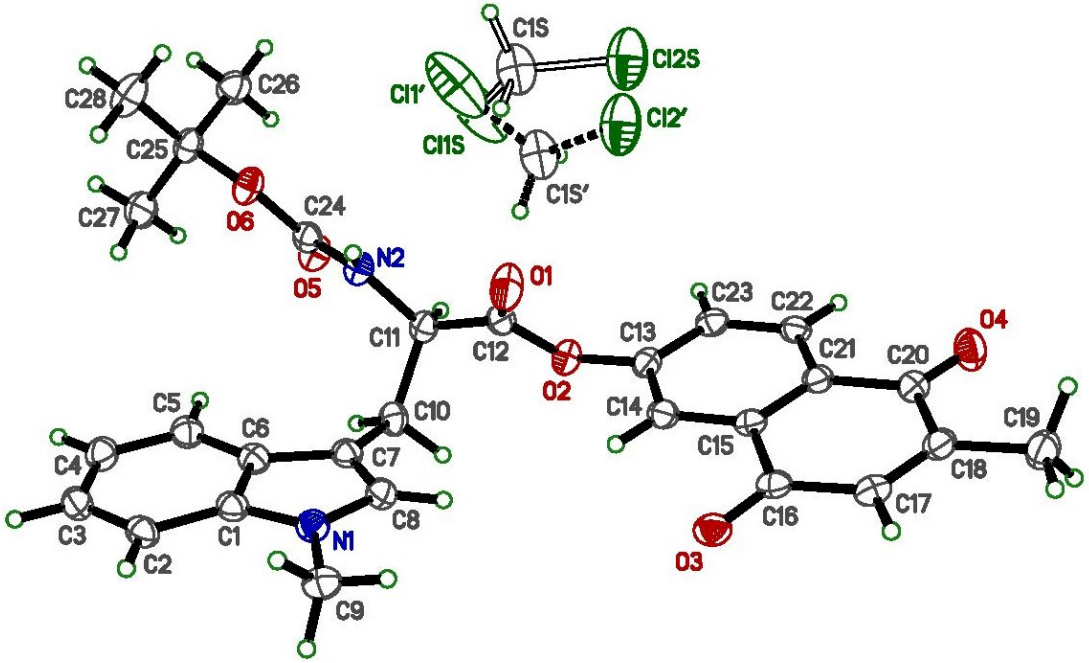


Figure S54: X-ray crystal structure of Compound **4** (EC1). X-ray structure crystalized with 2 molecules of dichloromethane per unit cell

Real time GC-MS spectra of oxalyl chloride deprotection

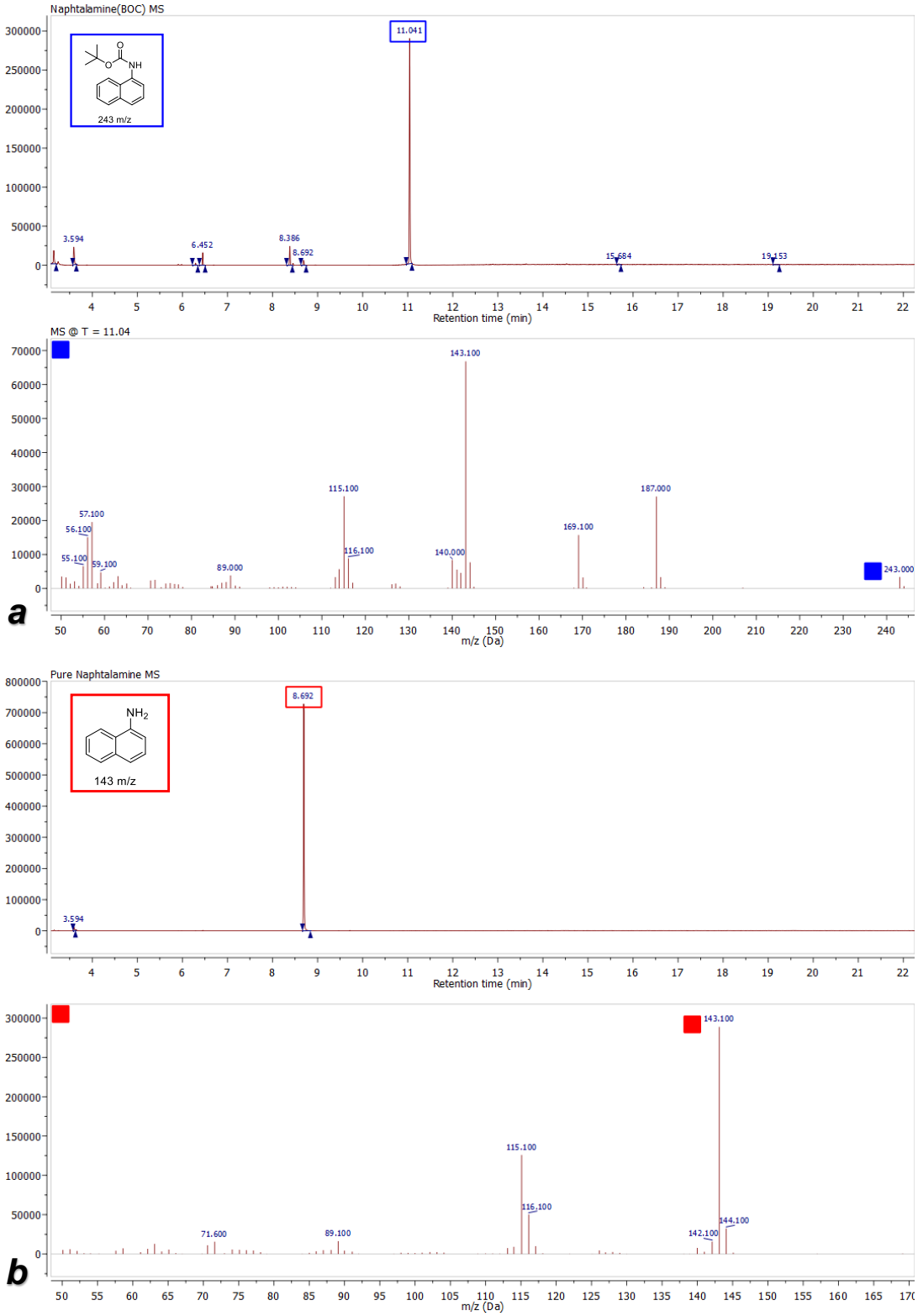


Figure S55*:* (***a***) GCMS of (N-BOC) Naphtalamine with rt = 11.041. (***b***) GCMS of Naphtalamine with rt = 8.69

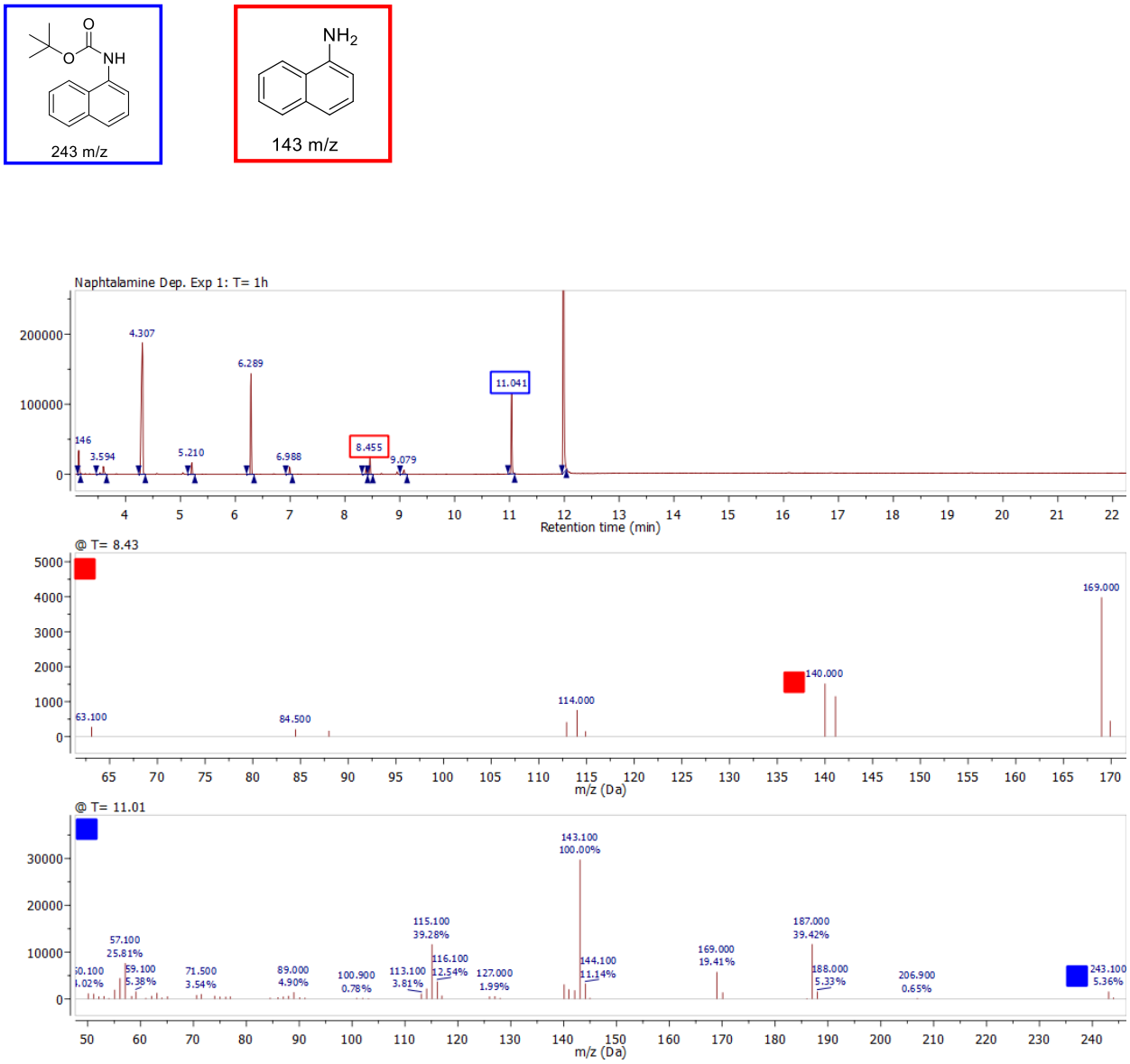


Figure S56*:* GCMS of (N-BOC) Naphtalamine deprotection reaction at time = 1 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455

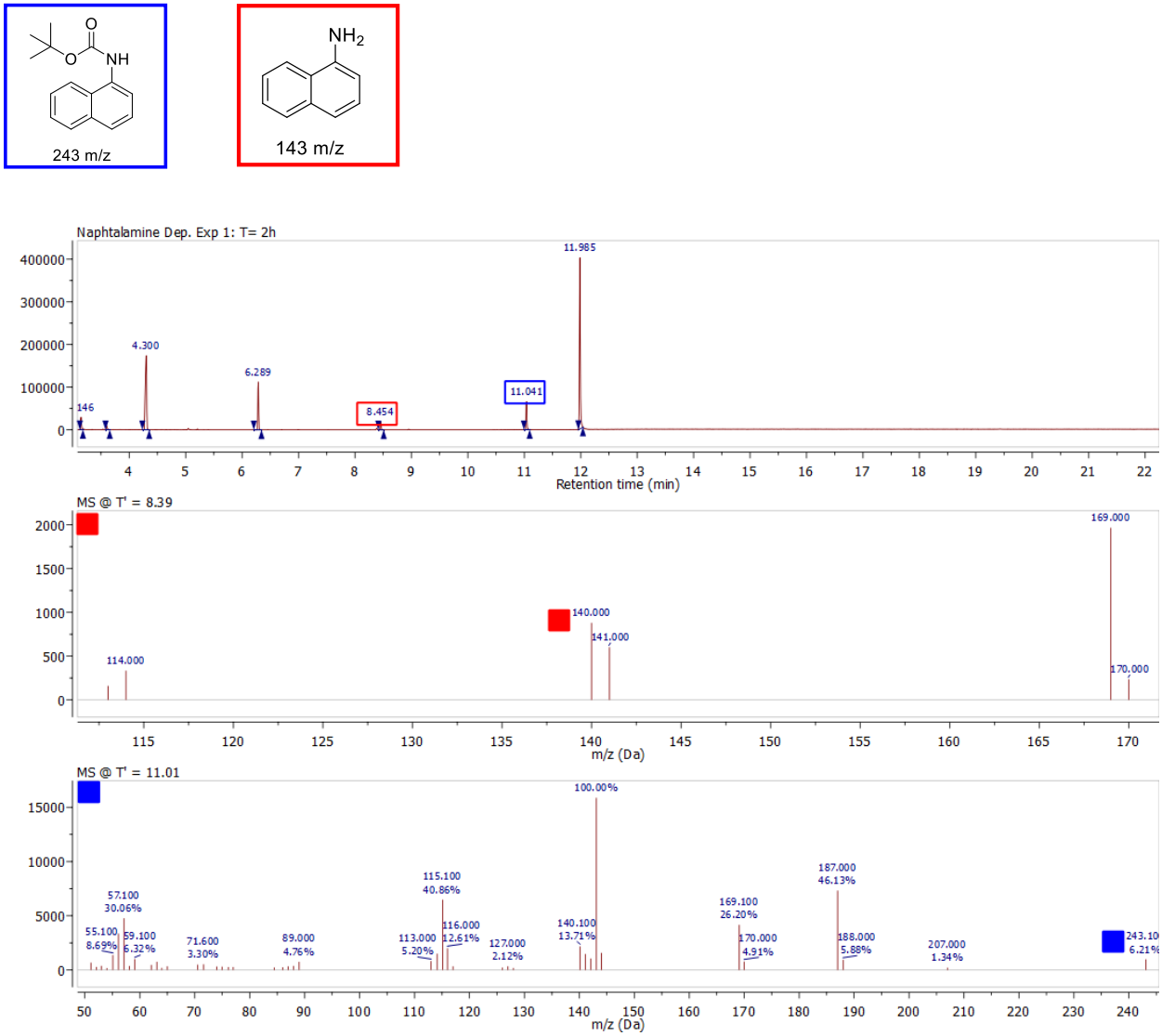


Figure S57: GCMS of (N-BOC) Naphtalamine deprotection reaction at time = 2 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455

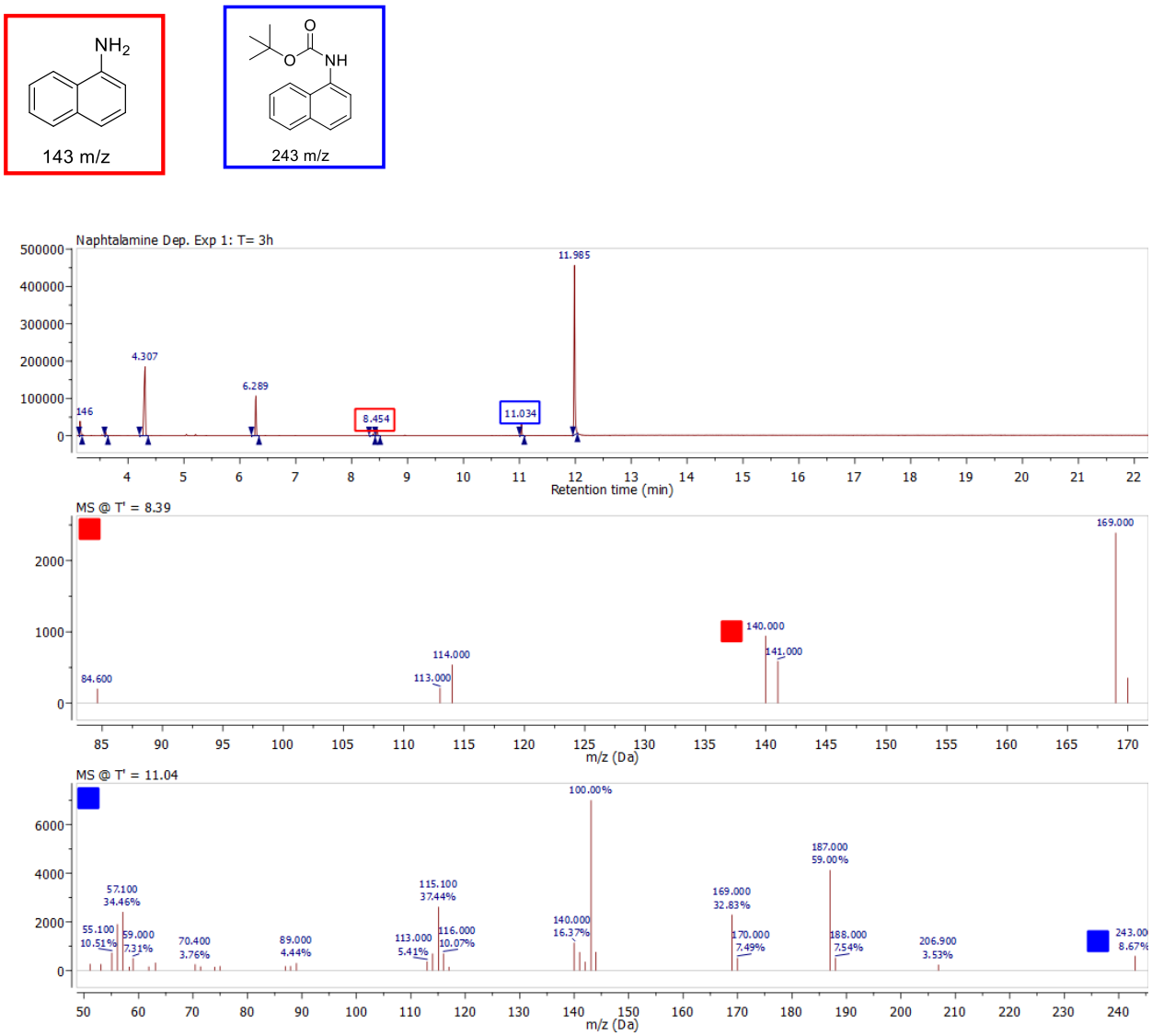


Figure S58: GCMS of (N-BOC) Naphtalamine deprotection reaction at time = 3 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455

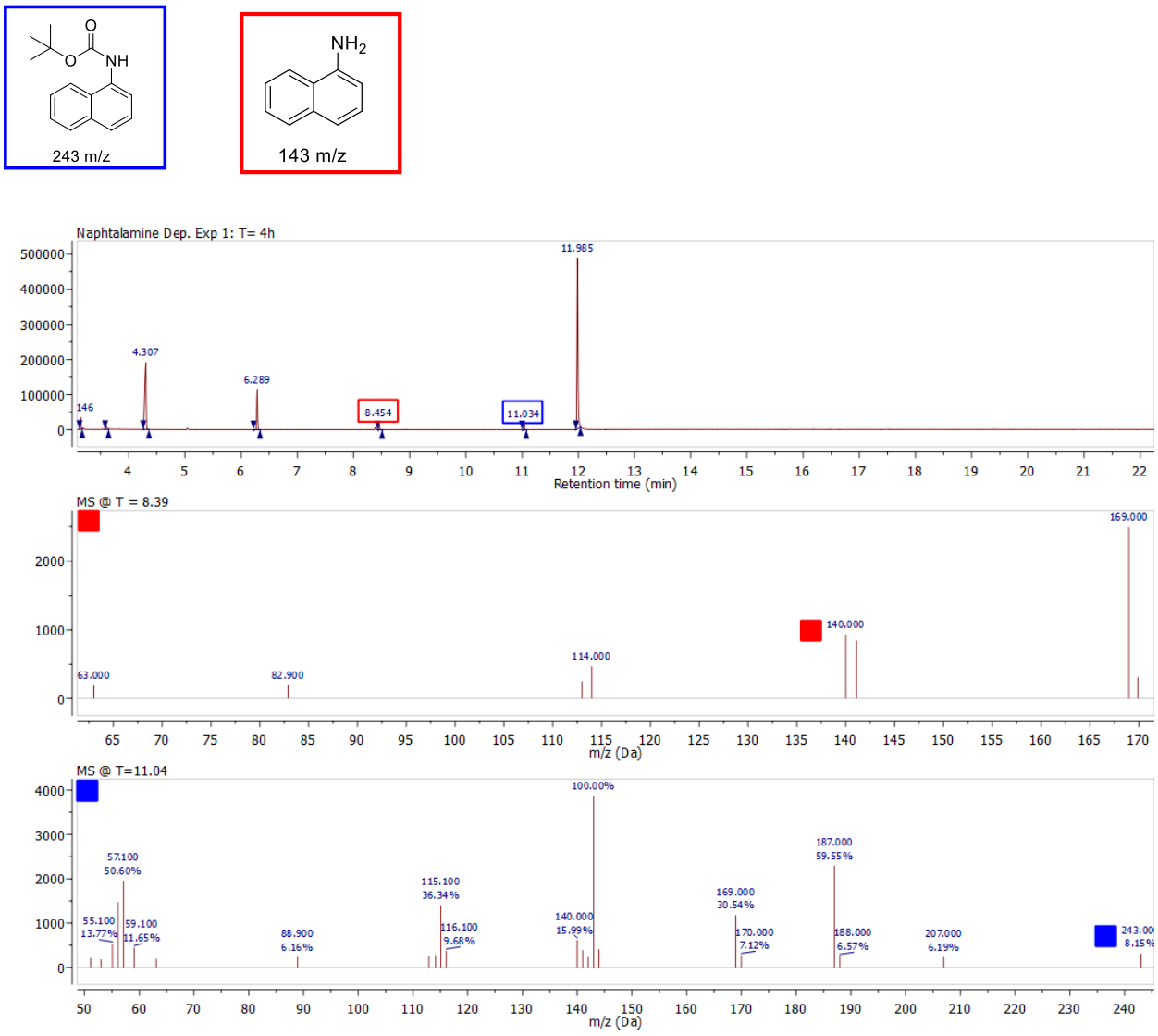
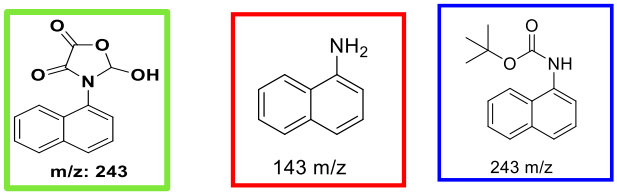


Figure S59: GCMS of (N-BOC) Naphtalamine deprotection reaction at time = 4 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455.



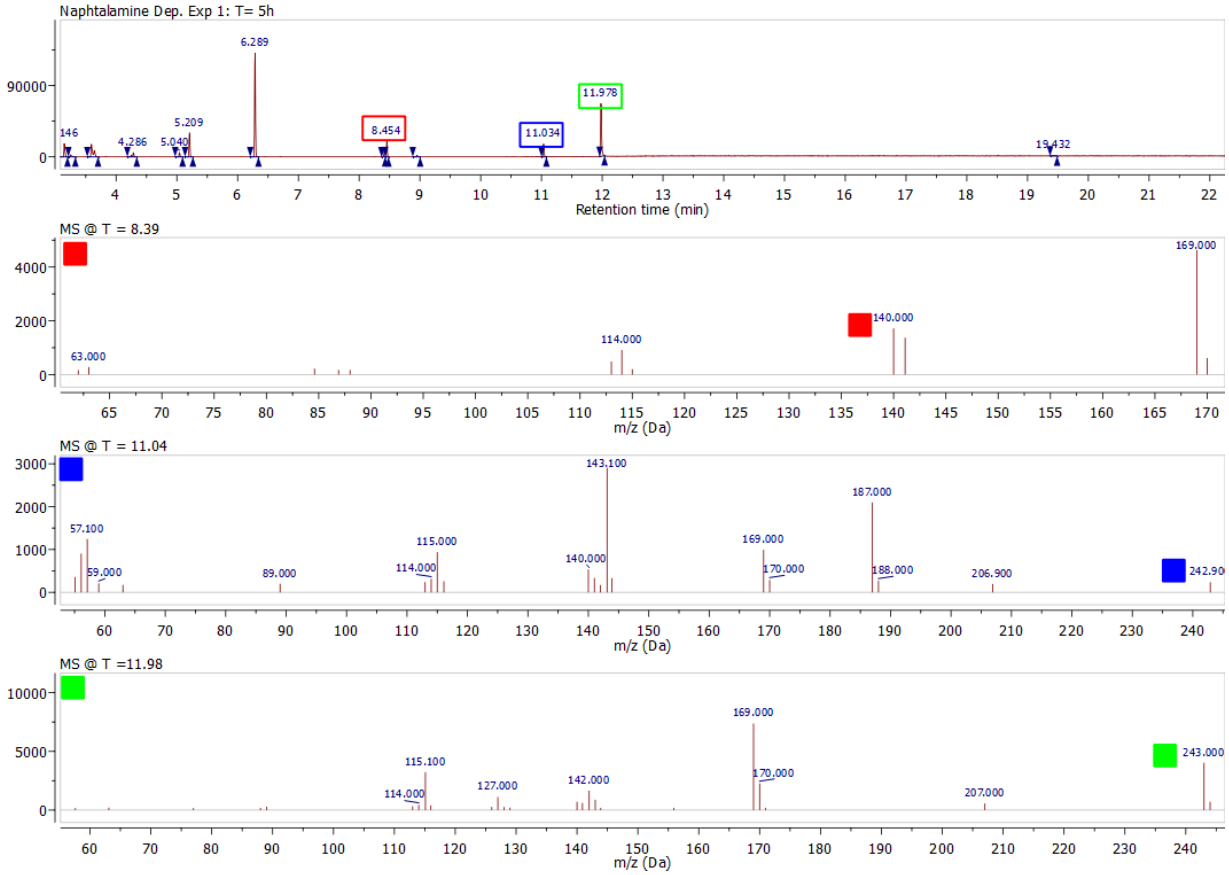


Figure S60: GCMS of (N-BOC) Naphtalamine deprotection reaction at time = 5 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455

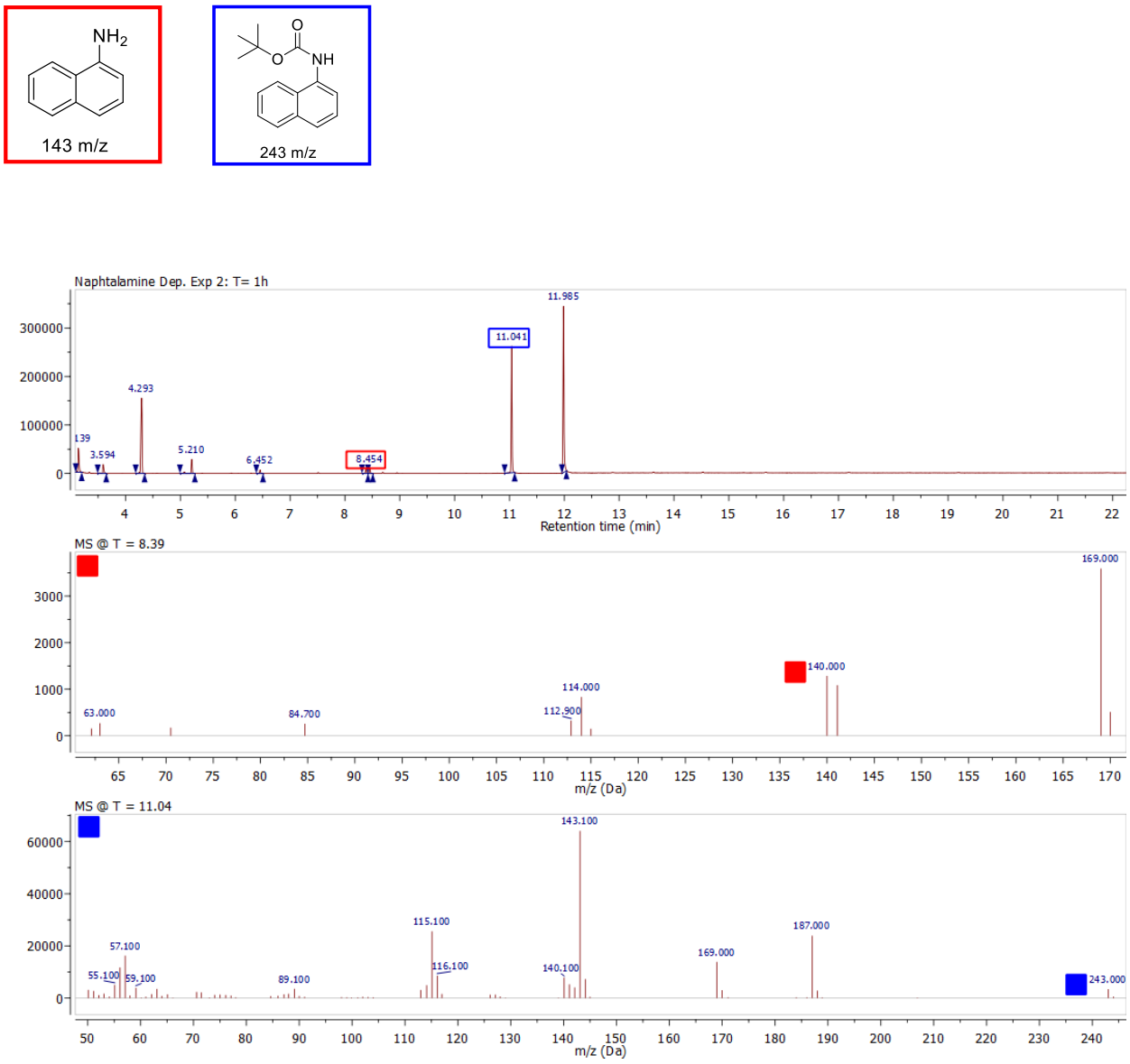


Figure S61: GCMS of (N-BOC) Naphtalamine deprotection reaction at time = 6 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455

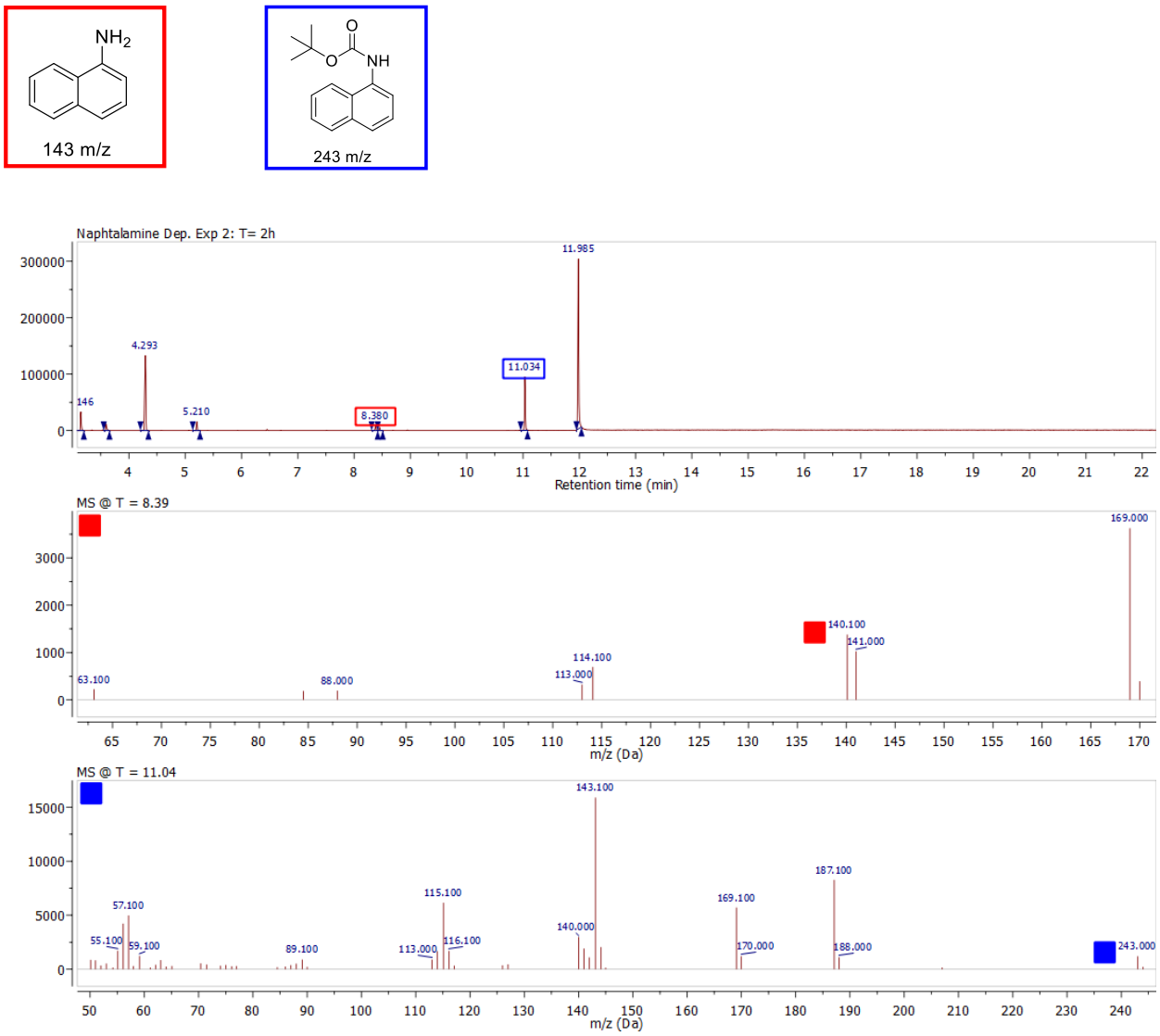


Figure S62: GCMS of (N-BOC) Naphtalamine deprotection reaction at time = 2 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455

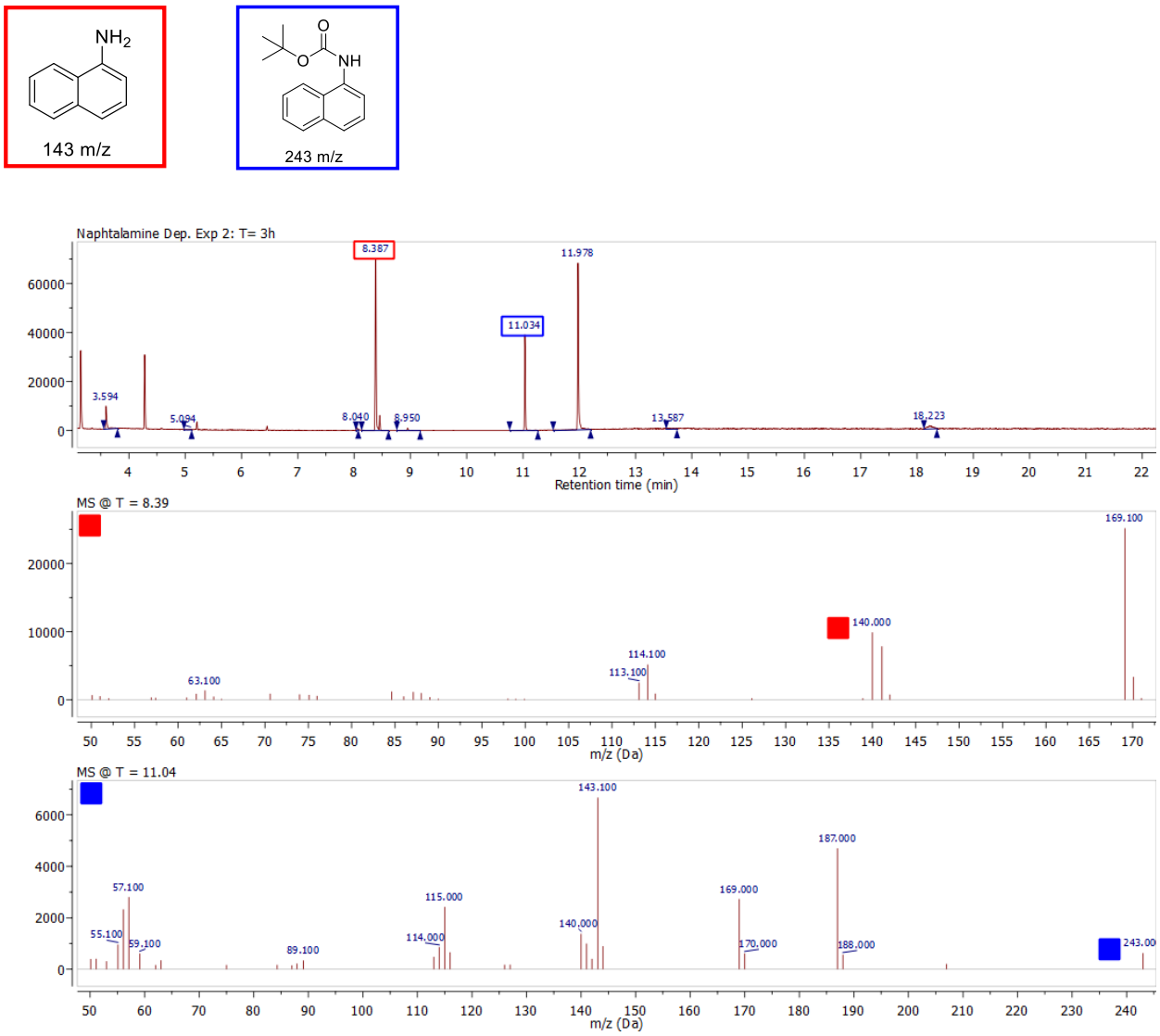


Figure S63: GCMS of (BOC) Naphtalamine deprotection reaction at time = 3 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455

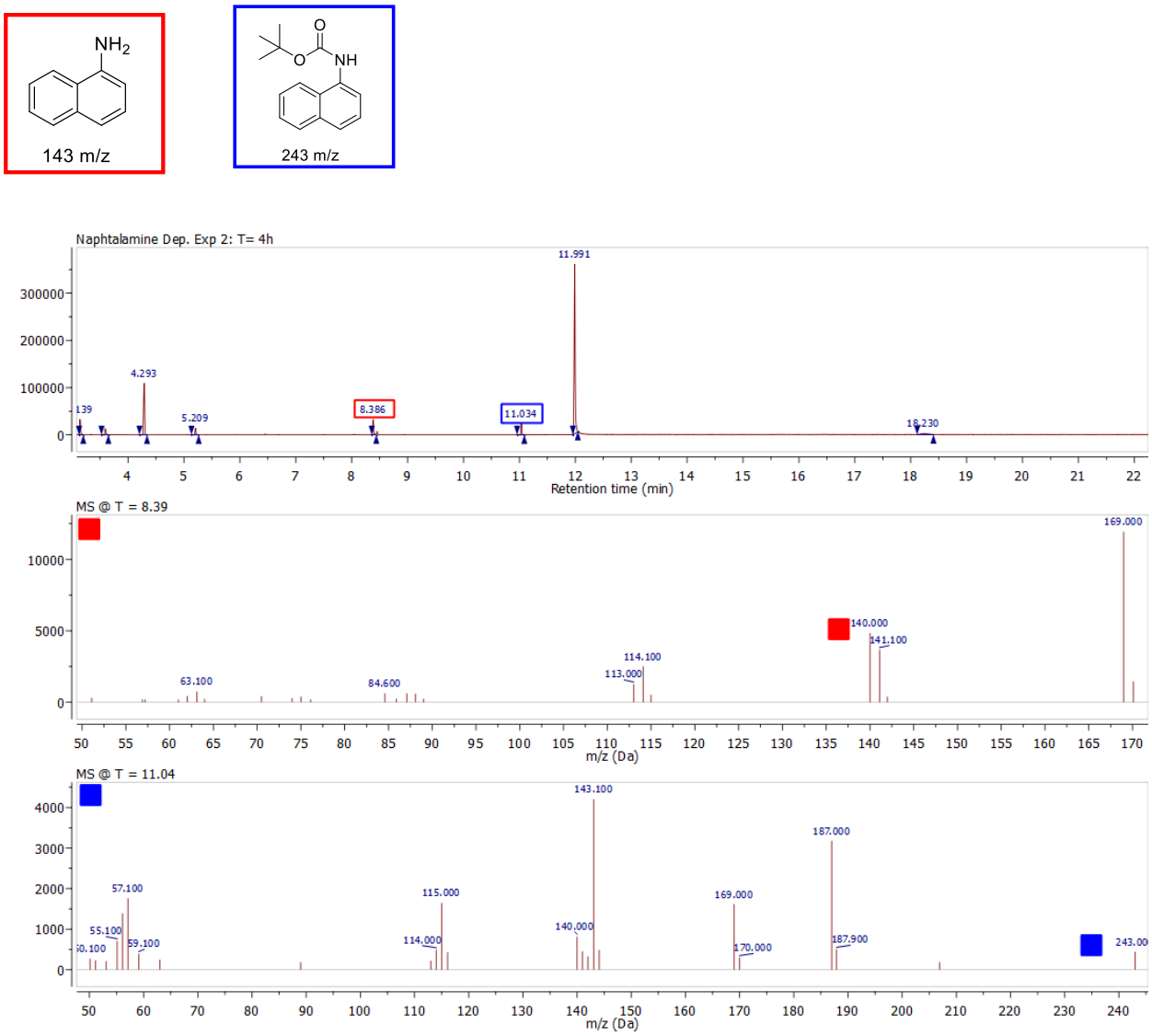
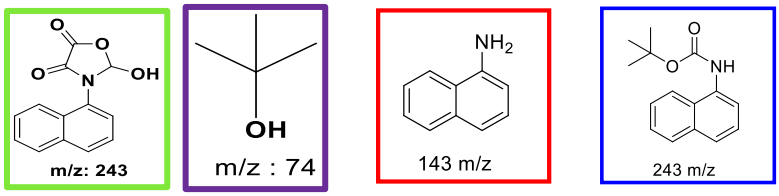


Figure S64: GCMS of (BOC) Naphtalamine deprotection reaction at time = 4 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455



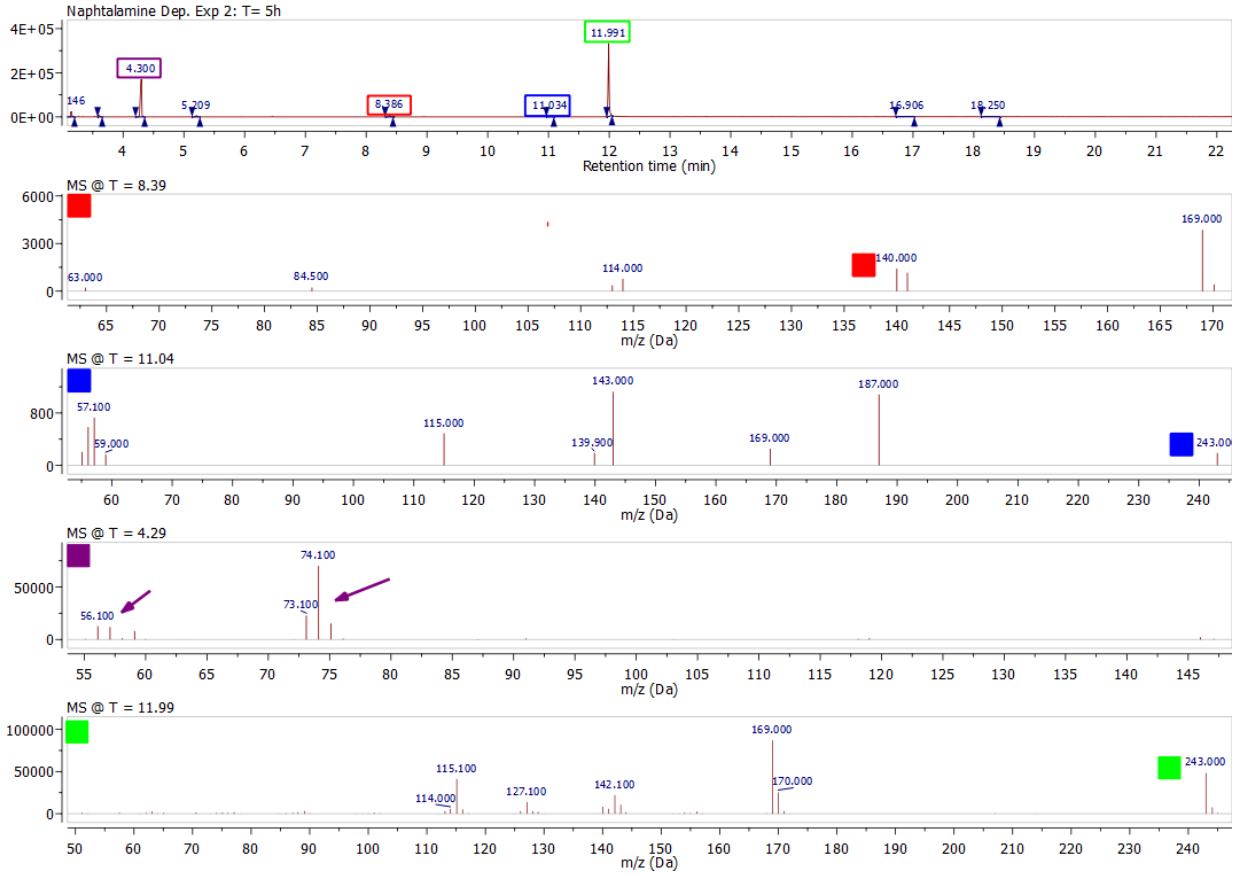


Figure S65: GCMS of (BOC) Naphtalamine deprotection reaction at time = 5 h. Boc protected starting material rt = 11.04. Intermediate rt = 8.455. Tert-butyl and t-butyl oxide ions rt = 4.3. Dichloro-intermediate located at rt= 11.991