Supporting Information

Visible Light Photocatalytic Aerobic Oxygenation of Indoles and pH as Chemoselective Switch

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1. General information

Column chromatography was carried out with *silica gel* 60 (particle size 0.040–0.063 mm, 230–400 mesh) or neutral alumina (200–300 mesh) and commercially available solvents. Thin-layer chromatography (TLC) was conducted on aluminum sheets coated with *silica gel* 60 F254 with visualization by a UV lamp (254 or 360 nm). Melting points (m.p.) were measured in open capillaries and were uncorrected.

^{1}H and ^{13}C NMR spectra were recorded at 300 and 75 MHz at 25 °C with a 300 MHz instrument. Chemical shifts are reported in parts per million (ppm), using the residual solvent signal as an internal standard: $CDCl_3$ (^{1}H NMR: δ 7.26, singlet; ^{13}C NMR: δ 77.0, triplet). Multiplicities were given as: *s* (singlet), *d* (doublet), *t* (triplet), *q* (quartet), *quintet*, *m* (multiplets), *dd* (doublet of doublets), *dt* (doublet of triplets), and *br* (broad). Coupling constants (*J*) were recorded in Hertz (Hz). The number of proton atoms (*n*) for a given resonance was indicated by *n*H. The number of carbon atoms (*n*) for a given resonance was indicated by *n*C. HRMS was reported in units of mass of charge ratio (m/z). Mass samples were dissolved in DCM and MeOH (HPLC Grade) unless otherwise stated. Electrochemical measurements were carried out by cyclic voltammetry (CV). The cyclic voltammetry was performed with an Autolab Potentiostat under nitrogen atmosphere in a one-compartment electrolysis cell consisting of a platinum wire working electrode, a platinum wire counter electrode, and a quasi Ag/AgCl reference electrode. Cyclic voltammograms were monitored at scan rates of either 100 $mV \bullet s^{-1}$ or 50 $mV \bullet s^{-1}$ and recorded in distilled acetonitrile. The concentration of the complex was maintained at 0.5 mM or less and each solution contained 0.1 M of tetrabutylammonium hexafluorophosphate (TBAP) as the electrolyte.

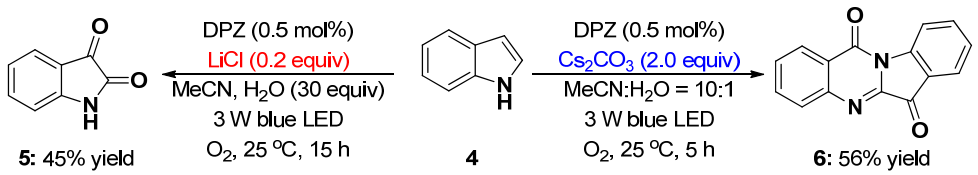
2. General experimental procedure for the synthesis of 2

Conditions I: 28 μL (0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of CH_3CN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Subsequently, **1** (0.1 mmol, 1.0 eq.), LiBr (0.02 mmol, 0.2 eq.) and solvent (2.0 mL CH_3CN and 30 eq. of H_2O) were sequentially added. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25 ^oC under an oxygen atmosphere (The sample vial was fitted with an oxygen balloon and the temperature was maintained in an incubator). The reaction was monitored by TLC. Upon complete consumption of **1**, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short *silica gel* column, followed by gradient elution with petroleum ether/ethyl acetate (10/1–3/1 ratio). Removing the solvent *in vacuo*, afforded products **2a-k**.

3. General experimental procedure for the synthesis of 3

Conditions II: 28 μL (0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of MeCN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Next, **1** (0.1 mmol, 1.0 eq.), K_3PO_4 (0.3 mmol, 3.0 eq.) and solvent (2.0 mL, CH_3CN:H_2O = 10:1, v/v) were added into reaction system successively. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25 °C under an oxygen atmosphere (The sample vial was fitted with an oxygen balloon and the temperature was maintained in an incubator). The reaction was monitored by TLC. Upon complete consumption of **1**, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short *silica gel* column, followed by gradient elution with petroleum ether/ethyl acetate (10/1–2/1 ratio). Removing the solvent *in vacuo*, afforded products **3a-k**. The yields could be improved when 10 mol% TEMPO as an additive was added.

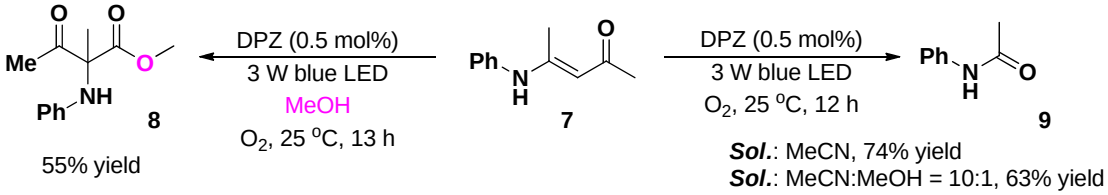
4. Experimental procedure for the synthesis of 5 and 6



The synthesis of 5: 28 μL (0.0005 mmol, 0.005 eq) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of CH_3CN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Subsequently, **4** (0.1 mmol, 1.0 eq.), LiCl (0.2 mmol, 2.0 eq.) and solvent (2.0 mL of CH_3CN and 30.0 eq. of H_2O) were added sequentially. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25 °C under an oxygen atmosphere (The sample vial was fitted with an oxygen balloon and the temperature was maintained in an incubator). After 15 hours, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short *silica gel* column, followed by gradient elution with petroleum ether/ethyl acetate (10/1–3/1 ratio). Removing the solvent *in vacuo*, afforded product **5.**

The synthesis of 6: 28 μL (0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of MeCN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Next, **4** (0.1 mmol, 1.0 eq.), Cs_2CO_3 (0.2 mmol, 2.0 eq.) and solvent (2.0 mL, CH_3CN/H_2O = 10:1, v/v) were added into the reaction system successively. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25 °C under an oxygen atmosphere (The sample vial was fitted with an oxygen balloon and the temperature was maintained in an incubator). After 5 hours, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short *silica gel* column, followed by gradient elution with petroleum ether/ ethyl acetate (10/1–5/1 ratio). Removing the solvent *in vacuo*, afforded product **6**.

5. Experimental procedure for the synthesis of 8 and 9

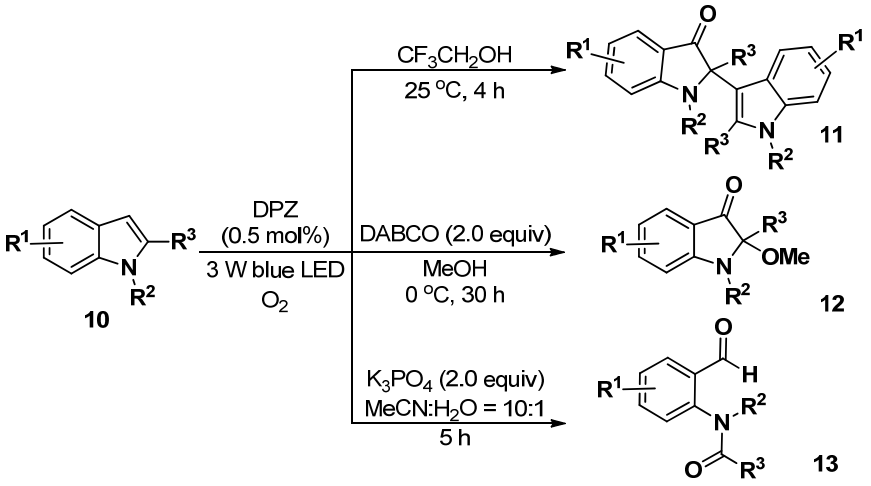


8 **7** **9**

The synthesis of 8: 28 μL **(**0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of MeCN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Next, **7** (0.1 mmol, 1.0 eq.) and solvent (2.0 mL MeOH) were added into the reaction system successively. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25 °C from a 5 cm distance. The reaction was monitored by TLC. Upon complete consumption of **7**, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short *silica gel* column, followed by gradient elution with petroleum ether/ethyl acetate. Removing the solvent *in vacuo*, afforded product **8**.

The synthesis of 9: 28 μL **(**0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of MeCN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Next, **7** (0.1 mmol, 1.0 eq.) and solvent (2.0 mL MeCN or 2.0 mL MeCN/MeOH =10:1) were added into the reaction system successively. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25 °C from a 5 cm distance. The reaction was monitored by TLC. Upon complete consumption of **7**, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short *silica gel* column, followed by gradient elution with petroleum ether/ethyl acetate. Removing the solvent *in vacuo*, afforded product **9**.

6. General experimental procedure for the synthesis of 11, 12 and 13

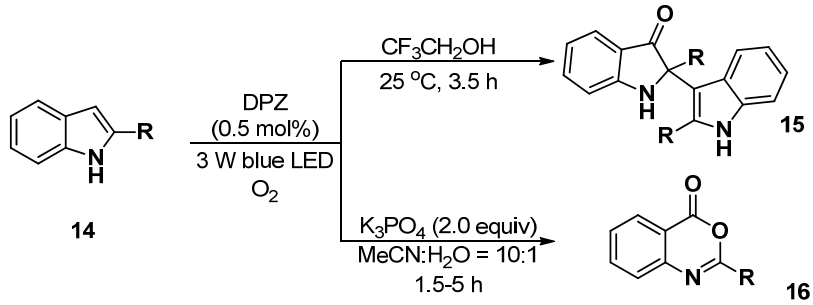


The synthesis of 11: 28 μL (0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of MeCN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Next, **10** (0.1 mmol, 1.0 eq.) and solvent (1.0 mL, CF_3CH_2OH) were added into the reaction system successively. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25°C (The sample vial was fitted with an oxygen balloon and the temperature was maintained in an incubator). The reaction was monitored by TLC. Upon complete consumption of **10**, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short neutral alumina column, followed by gradient elution with petroleum ether/ethyl acetate (10/1–5/1 ratio). Removing the solvent *in vacuo*, afforded products **11a-e**.

The synthesis of 12: 28 μL **(**0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of MeCN) was added into a 10.0 mL sample vial, and then solvent was removed *in vacuo*. Next, **10** (0.1 mmol, 1.0 eq.), DABCO (0.2 mmol, 2.0 equiv) and solvent (2.0 mL, MeOH) were added into the reaction system successively. The reaction mixture was stirred under irradiation by a 3 W blue LED (λ = 450–455 nm) at 0 °C (The sample vial was fitted with an oxygen balloon and the temperature was maintained in an incubator). The reaction was monitored by TLC. Upon complete consumption of **10**, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short neutral alumina column, followed by gradient elution with petroleum ether/ethyl acetate (20/1–5/1 ratio). Removing the solvent *in vacuo*, afforded products **12a-e**.

The synthesis of 13: 28 μL **(**0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of MeCN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Next, **10** (0.1 mmol, 1.0 eq.), K_3PO_4 (0.2 mmol, 2.0 eq.) and solvent (2.0 mL, MeCN/H_2O = 10:1, v/v) were added into the reaction system successively. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25 ^oC (The sample vial was fitted with an oxygen balloon and the temperature was maintained in an incubator). The reaction was monitored by TLC. Upon complete consumption of **10**, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short *silica gel* column, followed by gradient elution with petroleum ether/ethyl acetate (10/1–2/1 ratio). Removing the solvent *in vacuo*, afforded products **13a-e**. The yields could be improved when extra 10 mol% TEMPO was added.

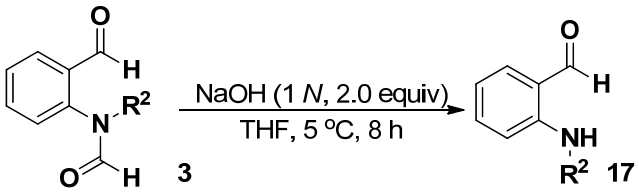
7. General experimental procedure for the synthesis of 15 and 16



The synthesis of 15: 28 μL **(**0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of MeCN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Next, **14** (0.1 mmol, 1.0 eq.) and solvent (1.0 mL CF_3CH_2OH) were added into the reaction system successively. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25 °C (The sample vial was fitted with an oxygen balloon and the temperature was maintained in an incubator). The reaction was monitored by TLC. Upon complete consumption of **14**, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short neutral alumina column, followed by gradient elution with petroleum ether/ethyl acetate (10/1–5/1 ratio). Removing the solvent *in vacuo*, afforded products **15a-b**

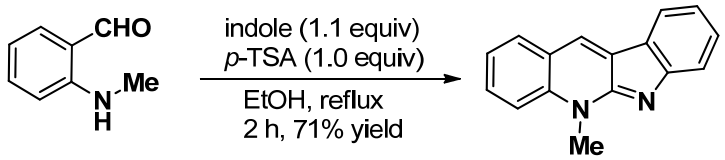
The synthesis of 16: 28 μL **(**0.0005 mmol, 0.005 eq.) of **DPZ** solution (1.0 mg of **DPZ** in 160 μL of MeCN) was added into a 10.0 mL sample vial, and CH_3CN was then removed *in vacuo*. Next, **14** (0.1 mmol, 1.0 eq.), K_3PO_4 (0.2 mmol, 2.0 eq.), AcOH (0.01mmol, 0.1 equiv) and solvent (2.0 mL, MeCN/H_2O = 10:1, v/v) were added into the reaction system successively. The reaction mixture was stirred under the irradiation by a 3 W blue LED (λ = 450–455 nm) at 25 °C from a 5 cm distance. The reaction was monitored by TLC. Upon complete consumption of **14**, the solvent was removed *in vacuo*. The reaction mixture was then loaded onto a short *silica gel* column, followed by gradient elution with petroleum ether/ethyl acetate (10/1–5/1 ratio). Removing the solvent *in vacuo*, afforded products **16a-b**.

8. **Experimental procedure for the synthesis of 18**



3 **17**

An ice-cooled solution of compounds **3** (0.5 mmol, 1.0 eq.) in THF (3.0 mL) was treated with 1 *N* aqueous sodium hydroxide (1.0 mmol, 2.0 eq.) keeping the temperature below 8 °C. After the starting material was consumed, the mixture was diluted with brine and extracted twice with ethyl ether. The organic solution was washed with water until neutral and then with brine, dried over sodium sulfate and concentrated *in vacuo* to afford crude product. The crude product was further purified by SiO_2 column chromatography, and the corresponding aldehyde **17** was obtained.

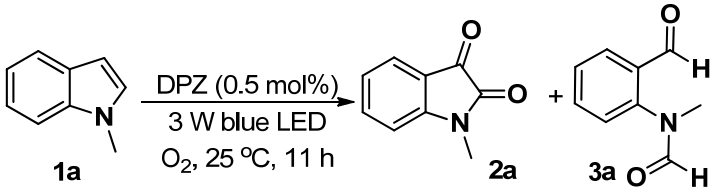


17 **18: Neocryptolepine**

A mixture of compound **17** (0.5 mmol, 1.0 eq.), indole (0.5 mmol, 1.0 eq.) and *p*-TsOH (95 mg, 0.5 mmol, 1.0 eq.) in ethanol (5.0 mL) was stirred open to air in a 25 mL round bottom flask in reflux. After 2 hours, the mixture was cooled to room temperature and washed using 1 *N* NaOH (30 mL). The aqueous layer was extracted with CH_2Cl_2 (3 x 40 mL). The combined organic layers were then dried with anhydrous sodium sulfate. The organic layer was concentrated under reduced pressure. The crude product was subsequently purified by flash chromatography on alumina, affording neocryptolepine **18** in 71% yield.

9. Optimization of the reaction conditions

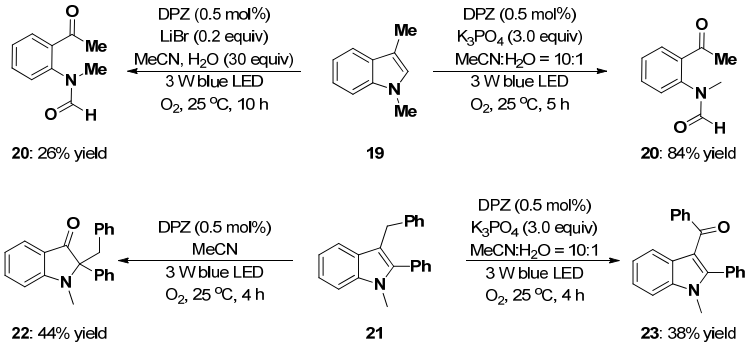
Table S1: Optimization of the reaction conditions[a]



entry	solvent	additive	*t* (h)	conv (%)[b]	**2a**/**3a**[b]
1	CH_2Cl_2	--	9	7	N.D
2	Toluene	--	9	6	N.D
3	CH_3CN	--	9	>99	5:1
4	CH_3CN	NaH_2PO_4 (0.1 eq.)	9	>99	9:1
5	CH_3CN	$LiHPO_4$ (0.1 eq.)	9	>99	10:1
6	CH_3CN	LiCl (1.0 eq.)	9	>99	14:1
7	CH_3CN	LiBr (0.1 eq.)	9	>99[c]	18:1
8	CH_3CN, H_2O (30 eq.)	LiCl (1.0 eq.), Py (1.0 eq.)	9	>99[d]	20:1
9	CH_3CN, H_2O (30 eq.)	LiBr (0.1 eq.)	9	>99[e]	20:1
9	CH_3CN, H_2O (30 eq.)	LiBr (0.2 eq.)	9	>99[f]	20:1
10	CH_3CN	K_2CO_3 (3 eq.)	9	62%[g]	1:4
11	CH_3CN	Cs_2CO_3 (3 eq.)	9	58%[g]	1:6
12	CH_3CN	K_3PO_4 (3 eq.)	9	66%[g]	1:7
13	CH_3CN/H_2O (10:1)	K_3PO_4 (3 eq.)	9	74%[g]	1:20

[a]The reaction was performed on a 0.05 mmol scale, a 3 W blue LED (450−455 nm), 25 °C, ambient atmosphere. [b]Determined by crude ^{1}H NMR. [c]Yield (isol.) of **2a** = 41%. [d]Yield (*isol.*) of **2a** = 53%. [e]Yield (*isol.*) of **2a** = 57%. [f]Yield (*isol.*) of **2a** = 68%. [g]Isolated yield.

10. The preliminary study of photooxygenation of 1,3-disubstituted and 1,2,3-trisubstituted indoles.



In the presence of the reaction conditions A, the oxidation of 1,3-disubstituted indole **19** was messy, and product **20** was obtained in 26% yield. It was found that **20** could be achieved in 84% yield when under the reaction conditions B. While the products were same under two different reaction conditions, it could be deduced that the two reactions would go through SET and ET paths individually.

The reaction of 1,2,3-trisubstituted indole **21** was found to undergo an oxidation/semipinacol rearrangement process,[1] affording to product **22** in 44% yield. On the contrary, an oxygenation product **23** was obtained in 38% yield when in the presence of reaction conditions B. Interestingly, the double bond of **21** could not be oxidized, probably due to the more easily oxidized benzylic sp^3 C−H.

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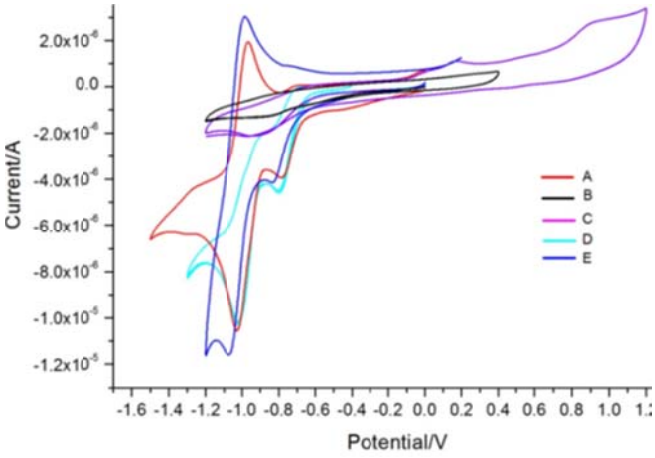
11. . Cyclic voltammetry (CV) measurements



A: DPZ in MeCN;

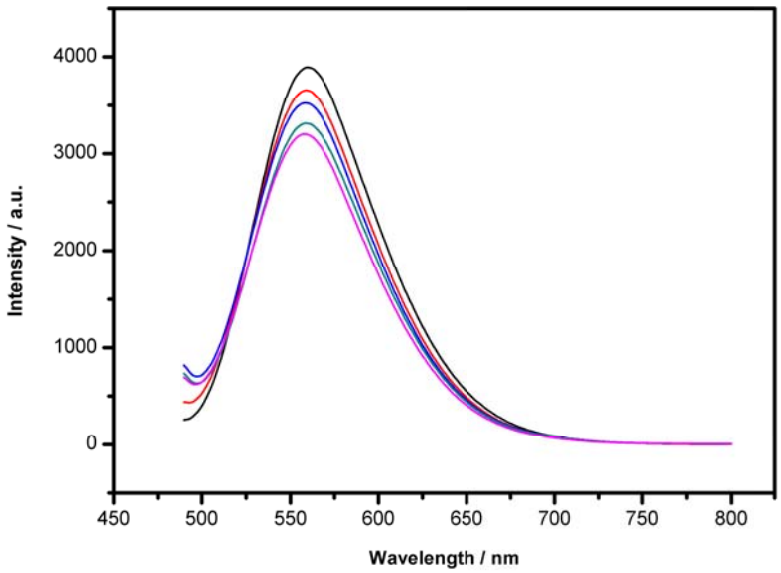
B: MeCN;

C: K_3PO_4 in MeCN;

D: DPZ and K_3PO_4 in MeCN;

E: DPZ and LiBr in MeCN.

12. Phosphorescence quenching experiments



1.8 mg (0.005 mmol, 0.005 equiv) of **DPZ**, 637 mg of K_3PO_4 (3 mmol, 3.0 equiv) and 131.2 mg (1mmol, 1.0 equiv) of 1-methylindole **1a** were added in a 25 mL sample vial. 20.0 mL of CH_3CN (HPLC Grade) were sequentially added, and vigorously shaked for two minutes. Then 4.0 mL of mixture was used for the **p**hosphorescence quenching experiment. The following four experiments were performed through increasing amounts of **1a** 65.6 mg (0.5 mmol, 0.5 equiv) successively. It was found that the phosphorescence intensity decreased in the presence of increasing concentration of **1a**.

13. Computational method

All density functional theory electronic structure calculations were carried out with Gaussian 09[1]. Gas-phase geometry optimization of these structures were done with Truhlar's meta-hyrid functional M06-2X[2] with Pople's 6-31+G(d,p) basis set[3]. Subsequent single point calculations were completed with the same functional and larger triple zeta Wiegend and Aldrich basis set[4] under SMD[5] polarizable continuum model: def2TZVPP/SMD. SMD water parameters were used for fluorescein anion and acetonitrile for DPZ. Zero-point corrections (298K) at M06-2X/6-31+G(d,p) level of theory were used to correct the energies attained from single point M06-2X/def2TZVPP/SMD. Eigenvalues of the minimum structures' Hessian matrix were checked to ensure all are positive. Benchmarking calculations with fluorescein anion to ascertain that the described DFT method is accurate. The calculated singlet (ground state) and triplet energy gap, E_T, for the fluorescein anion is 1.93 eV. This singlet-triplet energy gap is close to the experimental of 1.96 eV.[6]

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Cartesian Coordinates and Energies

All energies in hatrees.

DPZ (S_0)

E = -1781.474323

ZPE = 0.2352600

C 0.9520568085 0.1312772114 -0.7796283197

C -0.0328855802 0.7749976808 -0.0719690765

C -1.2862853225 0.1228366595 -0.2260598011

C -1.2475724915 -0.9626948273 -1.0612167363

H 0.124502481 1.6599998862 0.5291585117

H -2.2005706317 0.4329015041 0.2665173479

S 0.3717517026 -1.2467603686 -1.6544003732

O 2.2572755637 0.4048355482 -0.9002392948

C 2.7132181143 1.5369599539 -0.1718339135

H 3.7800578235 1.6146294196 -0.3731816729

H 2.5425105395 1.3947046318 0.9006878036

H 2.199768348 2.441950405 -0.5142495157

C -2.3508313269 -1.8935786199 -1.2965329747

C -4.1476324959 -3.8800408496 -1.4945875541

C -2.5624841839 -2.5926762932 -2.5259128128

N -3.1710624531 -2.0945851326 -0.264824841

C -4.0920209914 -3.0493608164 -0.3659044105

N -3.4127286415 -3.6185974489 -2.572343173

C -1.919108521 -2.2407769634 -3.7913640286

C -1.1124955658 -1.1266390435 -5.8462374478

C -1.1119934445 -2.4938535854 -5.9726822838

C -1.5979844221 -3.1193189737 -4.7925570328

H -0.7892005184 -3.0264061337 -6.8567338931

H -1.7186806079 -4.1898941839 -4.6742727952

O -0.7465981534 -0.1725624096 -6.7115923626

C -0.2932572804 -0.6370320473 -7.9760715983

H 0.5947234572 -1.2667491891 -7.8542784269

H -0.0435927397 0.2524824385 -8.5515305608

H -1.0842704037 -1.2017049895 -8.481168111

S -1.6732903573 -0.5864221231 -4.2987735572

C -5.0014258875 -3.2274745673 0.7414982353

C -5.0326951566 -5.0199264419 -1.5435732453

N -5.7424151531 -5.9322427055 -1.5692879111

N -5.737782508 -3.3772776253 1.6201458259

DPZ (T_1)

E = -1781.397531

ZPE = 0.2325040

C 1.0291569767 0.1695055152 -0.5750685312

C 0.0086928572 0.8203536923 0.0928199077

C -1.2339129426 0.1968781524 -0.1366502204

C -1.1622076763 -0.8939072939 -0.9869373517

H 0.1502319501 1.6942063317 0.7144068116

H -2.1759116842 0.5153071929 0.293302842

S 0.4851645562 -1.1889718722 -1.4969270844

O 2.335714328 0.43222257 -0.6137060292

C 2.7712190169 1.5416602227 0.1650013418

H 3.8480987269 1.6021728597 0.0213111649

H 2.5405845989 1.3778357777 1.2225748649

H 2.2929967784 2.4619201426 -0.1860196752

C -2.2432948038 -1.7794731746 -1.3247045956

C -3.7980932187 -3.964317938 -1.5076058353

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N -3.2530629182 -1.8410410778 -0.4768287517

C -4.1322129728 -2.8466389931 -0.6349485677

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C -1.792198931 -2.3080875492 -3.7785956763

C -1.1481748647 -1.2133516192 -5.9256294582

C -1.4278834488 -2.5609294019 -6.1029698072

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O -0.7444872549 -0.2913852306 -6.7953599358

C -0.568734132 -0.7282404541 -8.1402818014

H 0.1965776454 -1.509449582 -8.18686777

H -0.2454854168 0.147771509 -8.6985676181

H -1.5151171684 -1.1029310395 -8.5428510177

S -1.3228947037 -0.6767921634 -4.2674146023

C -5.2953940926 -2.879308882 0.1857379911

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N -6.2580512505 -2.9094765996 0.8343254908

Fluorescein anion (S_0)

E = -1145.066646

ZPE = 0.2622870

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C -2.4384877212 0.9627213791 -0.1578932018

C -2.2032738666 -1.821829127 -0.1457534229

C -1.1572119453 0.3908096627 -0.1499500289

C -3.5754848288 0.177104486 -0.1608523945

C -3.4515691565 -1.220073703 -0.1513531211

C -1.0720622688 -1.0080542501 -0.1407114412

C 0.0678364461 1.1670515148 -0.1299773165

O 0.122119884 -1.6502550237 -0.1327229554

C 1.2668519421 0.4947031141 -0.1346455758

C 1.2953478961 -0.9516814232 -0.1308981504

C 2.4464998805 -1.673768933 -0.1540172323

C 3.7416417726 -1.0136451016 -0.1621351723

C 3.7122230329 0.4596243946 -0.1211960008

C 2.5541059879 1.1546509897 -0.1092118949

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C -0.1012183563 5.3703183978 -0.9487092851

C 0.3802545946 3.1071237552 -1.6216776276

C -0.4172425988 3.5474608681 0.6262335696

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C 0.3272900765 4.46540384 -1.9210253156

H 0.7118426803 2.3931585184 -2.3720568003

H -0.8112111676 5.5739623515 1.0975902465

H 0.6224582174 4.812732867 -2.9071833073

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O -1.297470996 3.9319397721 2.7924510381

O -0.5922668083 1.8462167557 2.2450956448

O 4.809520953 -1.6365960714 -0.1994595541

H -2.1025317527 -2.9004814722 -0.1322318801

H 2.4160608417 -2.7572432989 -0.166405612

H 4.6761256947 0.9582498885 -0.0905970408

H -4.5604184457 0.6366970186 -0.152246023

Fluorescein anion (T_1)

E = -1144.992658

ZPE = 0.2590980

H -2.6616731909 1.975849167 -0.6253282506

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C -2.2373838398 -1.8581247808 -0.2540067161

C -1.233212332 0.3871077627 -0.3477354422

C -3.6513797109 0.0743513949 -0.6079092651

C -3.5013651014 -1.3070502434 -0.4663514184

C -1.1359380518 -1.0237910434 -0.18935074

C -0.0725953182 1.188675856 -0.2480187299

O 0.0585929128 -1.6374198979 0.0444717625

C 1.2060549576 0.5093471724 -0.0242075541

C 1.2178596812 -0.898786953 0.1008199309

C 2.3810343049 -1.6093038684 0.2684591032

C 3.6579747559 -0.9339782095 0.310331028

C 3.6346566674 0.5031062144 0.1763245603

C 2.4516083463 1.1800225573 0.0121307017

H 2.4469586944 2.2620202763 -0.0714735528

C -0.1249052025 2.6546642097 -0.4326934284

C -0.191458317 5.4257999752 -0.8611407359

C -0.4121873422 3.1773855648 -1.7035684382

C 0.112499893 3.5371907724 0.633397956

C 0.0819398743 4.9140587643 0.4042219981

C -0.4463768788 4.5523308787 -1.9199708913

H -0.6001503387 2.488470246 -2.524496708

H 0.2677398642 5.5567099283 1.2600307077

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H -0.2132427928 6.5003044691 -1.0234675961

O -4.5629591952 -2.1727735632 -0.5218853872

H -5.3765374684 -1.6662920359 -0.6182026386

C 0.3714724312 3.0165041032 2.0623064065

O 0.5087438726 3.8799780501 2.9457295905

O 0.4085818527 1.7609536949 2.1747414791

O 4.730681952 -1.5809880743 0.4478800711

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H 2.3587118467 -2.6898923358 0.3596962125

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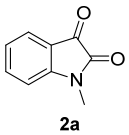
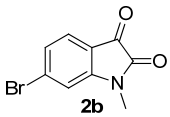
H -4.6405765346 0.5026559194 -0.7542830016

14. Characterization data of products



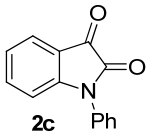
2a

Red solid, Mp 121.7–123.5 °C, 63% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59–7.54 (m, 2H), 7.10 (t, *J* = 7.6, 1H), 6.88 (d, *J* = 7.6, 1H), 3.23 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.3, 158.2, 151.4, 138.4, 125.1, 123.8, 117.3, 109.9, 26.1; HRMS (ESI) m/z 162.0554 ($M+H^+$), calc. for $C_9H_8NO_2$ 162.0555.



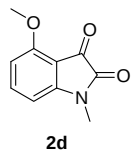
2b

Red solid, Mp 121.1–122.3 °C; 65% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.4, 1H), 7.40 (t, *J* = 7.9, 1H), 7.26 (s, 5H), 7.01 (t, *J* = 7.4, 1H), 6.70 (d, *J* = 7.9, 1H), 4.85 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.2, 158.2, 150.7, 138.30, 134.4, 129.0, 128.1, 127.4, 125.4, 123.8, 117.6, 111.0, 44.0; HRMS (ESI) m/z 260.0690 ($M+Na^+$), calc. for $C_{15}H_{11}NO_2Na$ 260.0687.



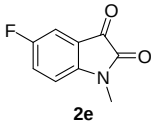
2c

Red solid, Mp 127.3–128.7 °C; 67% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.4, 1H), 7.56–7.54 (m, 3H), 7.43–7.41 (m, 3H), 7.17 (t, *J* = 7.4, 1H), 6.90 (d, *J* = 7.9, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.9, 157.3, 151.6, 138.3, 132.8, 129.9, 128.8, 126.0, 125.6, 124.3, 117.4, 111.3; HRMS (ESI) m/z 224.0715 ($M+H^+$), calc. for $C_{14}H_{10}NO_2$ 224.0712.



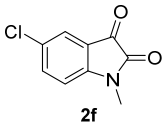
2d

Red solid, Mp 151.6–153.2 °C; 60% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53 (t, *J* = 8.2, 1H), 6.62 (t, *J* = 8.2, 1H), 6.45 (t, *J* = 8.2, 1H), 3.97 (s, 3H), 3.22 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.9, 158.7, 158.6, 152.3, 140.3, 107.6, 105.7, 102.1, 56.3, 26.4; HRMS (ESI) m/z 192.0659 ($M+H^+$), calc. for $C_{10}H_{12}NO_3$ 192.0661.



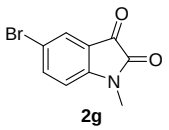
2e

Red solid, Mp 150.7–152.5 °C; 65% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30–7.21 (m, 2H), 6.82 (d, *J* = 7.0, 1H), 3.19 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.7, 160.9, 157.9, 157.7, 147.5, 124.8, 124.5, 117.9, 117.8, 112.4, 112.1, 111.1, 111.0, 26.3; HRMS (ESI) m/z 180.0466 ($M+H^+$), calc. for $C_9H_7FNO_2$ 180.0461.



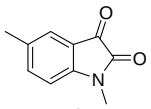
2f

Red solid, Mp 165.1–166.8 °C; 71% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58–7.54 (m, 2H), 6.86 (d, *J* = 8.2, 1H), 3.25 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.3, 157.6, 149.6, 137.7, 129.6, 125.2, 118.1, 111.2, 26.3; HRMS (ESI) m/z 196.0161 ($M+H^+$), calc. for $C_9H_7ClNO_2$ 196.0165.



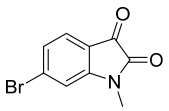
2g

Red solid, Mp 150.0–151.7 °C; 60% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.72–7.60 (m, 2H), 6.81 (d, *J* = 7.9, 1H), 3.24 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.12, 157.4, 150.1, 140.6, 128.00), 118.5, 116.6, 111.61, 26.32; HRMS (ESI) m/z 239.9663 ($M+H^+$), calc. for $C_9H_6BrNO_2$ 239.9660.



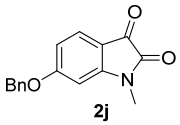
2h

Red solid, Mp 150.5–151.2 °C; 64% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.37–7.30 (m, 2H), 6.75 (d, *J* = 7.9, 1H), 3.17 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.5, 158.2, 149.1, 138.7, 133.5, 125.3, 117.2, 109.7, 26.0, 20.5; HRMS (ESI) m/z 176.0708 ($M+H^+$), calc. for $C_{10}H_{10}NO_2$ 176.0712.



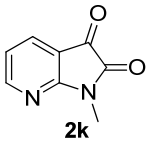
2i

Orange solid, Mp 161.9–163.1 °C; 66% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.9, 1H), 7.23–7.22 (m, 1H), 7.04 (s, 1H), 3.21 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.0, 158.0, 152.1, 133.6, 127.0, 126.3, 116.0, 113.7, 26.4; HRMS (ESI) m/z 239.9653 ($M+H^+$), calc. for $C_9H_7BrNO_2$ 239.9660.



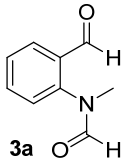
2j

Red solid, Mp 162.2–163.5 °C; 56% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.4, 1H), 7.42 (s, 5H), 6.62 (d, *J* = 8.4, 1H), 6.45 (s, 1H), 5.18 (s, 2H), 3.20 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 180.7, 167.4, 159.6, 154.0 135.2, 128.8, 128.6, 127.9, 127.6, 111.3, 108.5, 98.0, 70.9, 26.1; HRMS (ESI) m/z 268.0972 ($M+H^+$), calc. for $C_{16}H_{14}NO_3$ 268.0974.



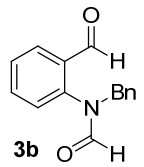
2k

Yellow solid, Mp 133.5–134.8 °C; 66% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.47 (d, *J* = 5.1, 1H), 7.83 (d, *J* = 7.3, 1H), 7.11–7.07 (m, 1H), 3.34 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.9, 163.9, 158.4, 155.9, 132.8, 119.6, 112.1, 25.1; HRMS (ESI) m/z 163.0512 ($M+H^+$), calc. for $C_8H_7N_2O_2$ 163.0508.



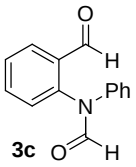
3a

Pale yellow solid , Mp 69.0–70.1 °C; 78% yield (86% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.03 and 9.92 (s, 1H, CHO rotameric), 8.32 and 8.18 (s, 1H, CHO rotameric), 7.91 (t, *J* = 7.3 Hz, 1H), 7.64 (t, *J* = 7.3 Hz, 1H), 7.47 (t, *J* = 7.3 Hz, 1H), 7.23 (dd, *J* = 16.5, 11.7 Hz, 1H), 3.36 and 3.28 (s, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.2, 189.0, 162.9, 162.7, 143.4, 141.2, 135.3, 135.1, 131.7, 130.9, 130.5, 128.4, 127.7, 127.4, 37.8, 34.5; HRMS (ESI) m/z 164.0708 ($M+H^+$), calc. for $C_9H_{10}NO_2$ 164.0712.



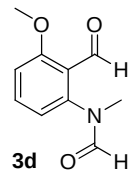
3b

Yellow oil, 76% yield (96% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.64 and 8.27 (s, 1H, CHO rotameric), 7.92–7.87 (m, 1H), 7.64–7.55 (m, 1H), 7.43 – 7.51 (m, *J* = 11.3, 7.5, 1H), 7.31–7.28 (m, 1H), 7.25–7.03 (m, 5H), 4.98 and 4.83 (s , 2H, CH_2 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.2, 189.0, 162.8, 162.3, 142.0, 139.9, 135.3, 135.2, 134.9, 132.8, 132.3, 130.4, 129.2, 129.1, 129.0, 128.8, 128.6, 128.6, 128.5, 128.2, 55.2, 50.8; HRMS (ESI) m/z 240.1022 ($M+H^+$), calc. for $C_{15}H_{14}NO_2$ 240.1025.



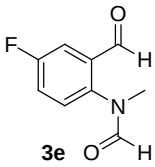
3c

Yellow oil, 71% yield (85% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.14 and 10.07 (s, 1H, CHO rotameric), 8.84 and 8.54 (s, 1H, CHO rotameric), 8.01 (dd, *J* = 7.7, 1.6, 1H), 7.73−7.65 (m, 1H), 7.60–7.48 (m, 1H), 7.43–7.27 (m, 4H), 7.25–7.13 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.0, 188.9, 162.3, 161.6, 141.6, 140.4, 135.4, 135.1, 132.2, 130.6, 130.2, 129.9, 129.7, 129.3, 128.9, 128.7, 126.9, 126.6, 124.5,123.4; HRMS (ESI) m/z 226.0870 ($M+H^+$), calc. for $C_{14}H_{12}NO_2$ 226.0868.



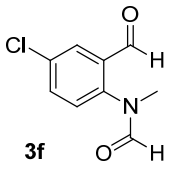
3d

Yellow solid, MP 74.0–75.0 ^{o}C; 58% yield (89% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.45 and 10.42 (s, 1H, CHO rotameric), 8.17 and 8.12 (s, 1H, CHO rotameric), 7.57 (t, *J* = 8.2, 1H), 7.02 (d, *J* = 8.5, 1H), 6.82 (d, *J* = 7.8, 1H), 3.96 and 3.93 (s, 1H, CH_3 rotameric), 3.29 and 3.20 (s, 1 H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.2, 188.7, 163.2, 162.6, 142.6, 135.6, 135.5, 120.5, 120.2, 111.7, 111.1, 56.3, 56.1, 37.5, 33.7; HRMS (ESI) m/z 194.0822 ($M+H^+$), calc. for $C_{10}H_{12}NO_3$ 194.0817.



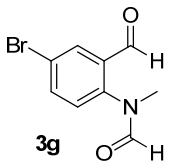
3e

Pale yellow solid, Mp 72.1–73.8 ^{o}C; 75% yield (87% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.04 and 9.91 (s, 1H , CHO rotameric), 8.39 and 8.22 (s, 1H, CHO rotameric), 7.67–7.64(m, 1H), 7.43–7.35 (m, 1H), 7.33–7.28 (m, 1H), 3.41 and 3.33 (s, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.8, 187.7, 163.6, 163.1, 162.6, 160.3, 139.9, 133.8, 133.7, 130.2, 130.1, 129.5, 129.4, 122.7, 122.4, 122.1, 116.6, 116.3, 38.0, 35.0; HRMS (ESI) m/z 204.0438 ($M+Na^+$), calc. for $C_9H_8FNO_2Na$ 204.0437.



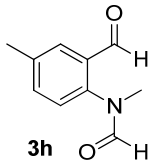
3f

Yellow oil, 86 % yield (92% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.07 and 9.94 (s, 1H, CHO rotameric), 8.42 and 8.26 (s, 1H, CHO rotameric), 7.97(dd, *J* = 2.5, 1H), 7.69 (dd, *J* = 8.5, 2.5, 1H), 7.35–7.27 (m, 1H), 3.46 and 3.38 (s, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.7, 187.6, 162.9, 162.43, 141.9, 139.6, 135.2, 134.9, 134.7, 134.6, 132.9, 132.8, 130.4, 130.0, 129.1, 128.6, 37.7, 34.7; HRMS (ESI) m/z 198.0321 ($M+H^+$), calc. for $C_9H_8ClNO_2$ 198.0322.



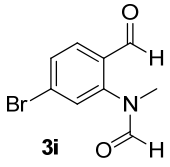
3g

Yellow oil, 72% yield (86% yield, 10 mol% TEMPO used); 1H NMR (300 MHz, $CDCl_3$) δ 10.03 and 9.91 (s, 1H, CHO rotameric), 8.36 and 8.24 (s, 1H, CHO rotameric), 7.83 (dd, *J* = 8.3, 5.4, 1H), 7.68–7.62 (m, 1H), 7.49–7.44 (m, 1.7, 1H), 3.41 and 3.34 (s, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.1, 187.9, 162.9, 162.4, 144.4, 132.0, 131.8, 131.6, 130.7, 130.6, 130.4, 129.9, 129.5, 37.7, 34.5; HRMS (ESI) m/z 263.9635 ($M+Na^+$), calc. for $C_9H_8BrNO_2Na$ 263.9636.



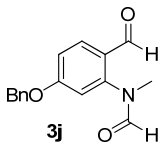
3h

Yellow oil, 66% yield (84% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.06 and 9.96 (s, 1H, CHO rotameric), 8.37 and 8.21 (s, 1H, CHO rotameric), 7.76(d, *J* = 1.5, 1H), 7.49 (dd, *J* = 8.0, 1.5, 1H), 7.18 (d, *J* = 8.0, 1H), 3.38 and 3.31 (s, 3H, CH_3 rotameric), 2.45 and 2.43 (s, 1H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.5, 189.2, 163.0, 162.8, 141.2, 138.8, 136.0, 135.9, 131.6, 131.0, 127.8, 127.5, 38.0, 34.7, 20.9; HRMS (ESI) m/z 178.0866 ($M+H^+$), calc. for $C_{10}H_{12}NO_2$ 178.0868.



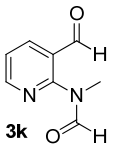
3i

Yellow oil, 78% yield (88% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.01 and 9.88 (s, 1H, CHO rotameric), 8.37 and 8.22 (s, 1H, CHO rotameric), 8.07 (dd, *J* = 6.6, 2.4, 1H), 7.80 (dd, *J* = 8.4, 2.4, 1H), 7.19 (d, *J* = 8.4, 1H), 3.41 and 3.33 (s, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.6, 187.5, 163.0, 162.4, 142.4, 140.1, 138.2, 137.9, 133.5, 133.1, 132.9, 129.3, 128.8, 122.4, 37.7, 34.6; HRMS (ESI) m/z 263.9634($M+Na^+$), calc. for $C_9H_8BrNO_2Na$ 263.9636.



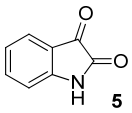
3j

Brown oil, 53% yield (85% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.94 and 9.87 (s, 1H , CHO rotameric), 8.36 and 8.25 (s, 1H, CHO rotameric), 7.99–7.87 (m, 1H), 7.43–7.38 (m, 5H), 7.07 (dt, *J* = 5.8, 3.0, 1H), 6.83 (dd, *J* = 5.8, 2.4, 1H), 5.17 and 5.14 (s, 2H, CH_2 rotameric), 3.36 and 3.32 (s, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.0, 187.7, 164.0, 162.6, 145.5, 135.2, 133.3, 128.8, 128.8 128.7, 128.6, 127.6, 127.5, 125.3, 114.5, 113.9, 70.7, 37.9, 34.5; HRMS (ESI) m/z 270.1133 ($M+H^+$), calc. for $C_{16}H_{16}NO_3$ 270.1130.



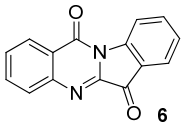
3k

Pale yellow solid, Mp 133.4–134.5 ^{o}C; 62% yield (86% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.03 and 9.84 (s, 1H, CHO rotameric), 8.70 (dd, *J* = 4.7, 1.9, 1H), 8.50 and 8.44 (s, 1H,CHO rotameric), 8.29 (dd, *J* = 7.8, 1.9, 1H), 7.40 (dd, *J* = 7.8, 4.7, 1H), 3.60 and 3.50 (s, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.9, 187.4, 163.5, 163.4, 155.3, 153.7, 152.7, 139.5, 137.0, 124.1, 122.6, 122.0, 34.9, 32.0; HRMS (ESI) m/z 165.0668 ($M+H^+$), calc. for $C_8H_9N_2O_2$ 165.0664.



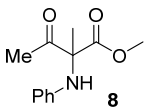
5

Red solid, Mp 199.2–200.1 ^{o}C; 45% yield; ^{1}H NMR (300 MHz, DMSO) δ 11.01 (s, 1H), 7.54 (t, *J* = 7.6, 1H), 7.44 (d, *J* = 7.2, 1H), 7.02 (t, *J* = 7.2, 1H), 6.87 (d, *J* = 7.6, 1H); ^{13}C NMR (75 MHz, DMSO) δ 184.4, 159.4, 150.8, 138.4, 124.7, 122.8, 117.8, 112.3; HRMS (ESI) m/z 148.0395 ($M+H^+$), calc. for $C_8H_6NO_2$ 148.0399.



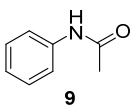
6

Yellow solid, Mp 258.7–260.1 ^{o}C; 56% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.63 (d, *J* = 8.0, 1H), 8.44 (d, *J* = 7.8, 1H), 8.03 (d, *J* = 8.0, 1H), 7.93–7.76 (m, 3H), 7.68 (t, *J* = 7.6, 1H), 7.43 (t, *J* = 7.6, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.6, 158.1, 146.6, 146.3, 144.3, 138.3, 135.1, 130.7, 130.3, 127.5, 127.2, 125.4, 123.7, 121.9, 118.0; HRMS (ESI) m/z 249.0663 ($M+H^+$), calc. For $C_{15}H_9N_2O_2$ 249.0664.



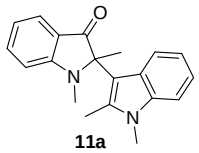
8

Brown oil, 55% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.15 (t, *J* = 7.5, 1H), 6.74 (t, *J* = 7.5, 1H), 6.52 (d, *J* = 7.5, 1H), 3.79 (s, 1H), 2.20 (s, 1H), 1.69 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.5, 171.7, 143.8, 129.4, 118.3, 114.1, 69.5, 53.5, 24.7, 18.6; HRMS (ESI) m/z 222.1135 ($M+H^+$), calc. for $C_{12}H_{16}NO_3$ 222.1130.



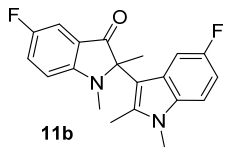
9

Colorless solid, Mp 114.8–116.3 °C; 74% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.8, 1H), 7.32 (t, *J* = 7.8, 1H), 7.11 (t, *J* = 7.2, 1H), 2.19 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.4, 137.8, 129.0, 124.3, 120.0, 24.6; HRMS (ESI) m/z 136.0766 ($M+H^+$), calc. for $C_8H_{10}NO$ 136.0762.



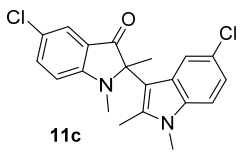
11a

Yellow solid, Mp 153.4–155.2 °C; 82% yield; ^{1}H NMR (300 MHz, DMSO) δ 7.60 (t, *J* = 7.7, 1H), 7.53 (d, *J* = 7.5, 1H), 7.40 (d, *J* = 8.2, 1H), 7.21 (d, *J* = 7.8, 1H), 7.09–6.99 (m, 2H), 6.87 (t, *J* = 7.3, 1H), 6.76 (t, *J* = 7.3, 1H), 3.66 (s, 3H), 2.82 (s, 3H), 2.30 (s, 3H), 1.77 (s, 3H); ^{13}C NMR (75 MHz, DMSO) δ 202.7, 158.7, 138.0, 136.2, 135.9, 126.5, 124.5, 120.3, 119.1, 118.8, 117.5, 116.6, 109.5, 108.8, 106.0, 70.4, 29.3, 27.6, 21.6, 11.4; HRMS (ESI) m/z 305.1650 ($M+H^+$), calc. for $C_{20}H_{25}N_2O$ 305.1654.



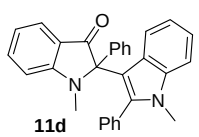
11b

Yellow solid, Mp 121.2–122.6 °C; 82% yield; ^{1}H NMR (300 MHz, DMSO) δ 7.52 (t, *J* = 9.2, 1H), 7.42 (dd, *J* = 9.2, 3.8, 1H), 7.31 (d, *J* = 7.5, 1H), 7.06 (dd, *J* = 8.9, 3.8, 1H), 6.90 (d, *J* = 8.9, 1H), 3.65 (s, 3H), 2.81 (s, 3H), 2.25 (s, 3H), 1.74 (s, 3H); ^{13}C NMR (75 MHz, DMSO) δ 202.3, 158.4, 156.2, 155.9, 155.4, 153.1, 138.0, 133.0, 126.5, 126.4, 126.2, 125.9, 117.2, 117.1, 110.6, 110.5, 10.4, 110.3, 109.3, 109.0, 108.3, 107.9, 106.1, 106.0, 71.1, 29.6, 27.8, 21.4, 11.6; HRMS (ESI) m/z 341.1466 ($M+H^+$), calc. for $C_{20}H_{19}F_2N_2O$ 341.1465.



11c

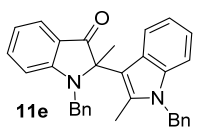
Yellow soild, Mp 134.7–136.2 °C; 88% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 (s, 1H), 7.47 (d, *J* = 8.8, 2H), 7.15 (d, *J* = 8.8, 1H), 7.08 (d, *J* = 8.7, 1H), 6.74 (d, *J* = 8.7, 1H), 3.60 (s, 3H), 2.85 (s, 3H), 2.19 (s, 3H), 1.88 (s, 3H);^{13}C NMR (75 MHz, $CDCl_3$) δ 202.2, 157.2, 137.7, 137.1, 135.2, 128.1, 125.4, 124.7, 122.1, 120.9, 119.1, 118.8, 110.0, 109.4, 106.2, 29.7, 27.9 , 21.9, 11.9; HRMS (ESI) m/z 373.0877 ($M+H^+$), calc. for $C_{20}H_{19}Cl_2N_2O$ 373.0874



11d

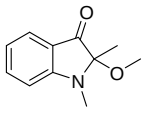
Yellow solid, Mp 230.8–232.5 °C; 56% yield; ^{1}H NMR (300 MHz, DMSO) δ 7.49 (d, *J* = 7.9, 2H), 7.27–7.21 (m, 7H), 7.14 (d, *J* = 7.8, 2H), 6.98–6.79 (m, 5H), 6.56 (t, *J* = 7.4, 1H), 6.40 (d, *J* = 8.1, 1H), 3.43 (s, 3H), 2.80 (s, 3H); ^{13}C NMR (75 MHz, DMSO) δ 199.1, 159.0, 139.9, 138.2, 137.6, 136.4,

130.9, 130.8, 130.6, 128.2, 127.7, 127.4, 126.9, 124.1, 121.2, 120.6, 119.2, 118.5, 116.4, 110.3, 108.9, 108.5, 75.5, 30.5, 29.4; HRMS (ESI) m/z 429.1970 ($M+H^+$), calc. for $C_{30}H_{25}N_2O$ 429.1967.



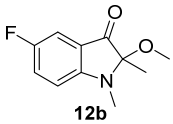
11e

Yellow solid, Mp 156.3−157.7 °C; 91% yield; ^{1}H NMR (300 MHz, DMSO) δ 7.60 (d, *J* = 7.6, 1H), 7.51 (t, *J* = 7.6, 1H), 7.39 (d, *J* = 8.2, 1H), 7.25–7.17 (m, 7H), 7.07–6.90 (m, 6H), 6.80 (t, *J* = 7.4, 1H), 6.74 (d, *J* = 8.2, 1H), 5.43 (s, 2H), 4.58 (d, *J* = 16.5, 1H), 4.33 (d, *J* = 16.5, 1H), 2.18 (s, 3H), 1.76 (s, 3H); ^{13}C NMR (75 MHz, DMSO) δ 202.6, 158.5, 138.4, 138.1, 137.9, 136.2, 128.6, 128.3, 27.1, 126.9, 125.9, 124.6, 120.7, 119.6, 118.9, 117.9, 117.3, 110.0, 109.5, 107.2, 71.1, 46.5, 45.4, 22.6, 11.8; HRMS (ESI) m/z 457.2278 ($M+H^+$), calc. for $C_{32}H_{29}N_2O$ 457.2280.



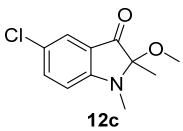
12a

Yellow oil, 66% yield; ^{1}H NMR (300 MHz, MeOD) δ 7.56 (t, *J* = 7.7, 1H), 7.48 (d, *J* = 7.7, 1H), 6.88 (d, *J* = 7.9, 1H), 6.75 (t, *J* = 7.9, 1H), 2.98 (s, 3H), 2.93 (s, 3H), 1.31 (s, 3H); ^{13}C NMR (75 MHz, MeOD) δ 202.8, 162.1, 140.4, 125.2, 119.4, 118.8, 109.8, 93.7, 51.6, 26.6, 19.9; HRMS (ESI) m/z 192.1026 ($M+H^+$), calc. for $C_{11}H_{14}NO_2$ 192.1025.



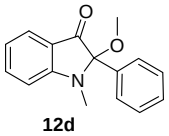
12b

Yellow oil, 67% yield; ^{1}H NMR (300 MHz, DMSO) δ 7.50 (t, *J* = 9.1, 1H), 7.26 (d, *J* = 7.4, 1H), 7.00 (d, *J* = 5.6, 1H), 2.88 (d, *J* = 1.8, 6H), 1.24 (s, 3H); ^{13}C NMR (75 MHz, DMSO) δ 199.2, 156.6, 156.0, 152.8, 126.0, 125.7, 117.2, 117.1, 110.0, 109.9, 108.6, 108.3, 92.0, 50.3, 26.0, 18.8; HRMS (ESI) m/z 210.0928 ($M+H^+$), calc. For $C_{11}H_{13}FNO_2$ 210.0930.



12c

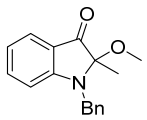
Yellow oil, 76% yield; ^{1}H NMR (300 MHz, DMSO) δ 7.59 (d, *J* = 8.8, 1H), 7.45 (s, 1H), 7.01 (d, *J* = 8.8, 1H), 2.88 (s, 6H), 1.25 (s, 3H); ^{13}C NMR (75 MHz, DMSO) δ 198.5, 158.1, 137.7, 122.5, 120.8, 118.2, 110.3, 91.8, 50.3, 25.8, 18.7; HRMS (ESI) m/z 226.0639 ($M+H^+$), calc. for $C_{11}H_{13}ClNO_2$ 226.0635.



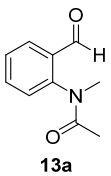
12d

Yellow solid, Mp 73.4–75.1 °C; 85% yield; ^{1}H NMR (300 MHz, MeOD) δ 7.62 (t, *J* = 7.7, 1H), 7.47 (d, *J* = 7.7, 1H), 7.32 (s, 5H), 6.96 (d, *J* = 7.8, 1H), 6.78 (t, *J* = 7.8, 1H), 3.21 (s, 3H), 2.81 (s, 3H); ^{13}C NMR (75 MHz, MeOD) δ

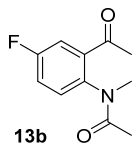
201.4, 163.5, 140.7, 136.8, 130.0, 129.9, 127.4, 126.0, 119.5, 119.2, 109.2, 96.9, 51.9, 27.6; HRMS (ESI) m/z 254.1184 ($M+H^+$), calc. for $C_{16}H_{16}NO_2$ 254.1181.


12e

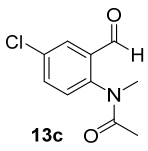
Yellow solid, Mp 79.2–80.8 °C; 64% yield. ^{1}H NMR (300 MHz, MeOD) δ 7.53 (d, *J* = 7.8, 1H), 7.47 (t, *J* = 7.8, 1H), 7.35–7.27 (m, 5H), 6.77 (t, *J* = 7.8, 1H), 6.70 (d, *J* = 7.8, 1H), 4.59 (q, *J* = 16.7, 2H), 2.99 (s, 3H), 1.34 (s, 3H); ^{13}C NMR (75 MHz, MeOD) δ 202.5, 162.2, 140.3, 139.6, 129.9, 128.4, 128.2, 125.5, 119.8, 119.4, 110.7, 94.2, 52.1, 46.0, 21.4; HRMS (ESI) m/z 268.1340 ($M+H^+$), calc. for $C_{17}H_{18}NO_2$ 268.1338.


13a

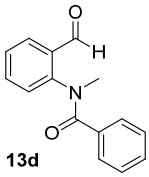
Pale yellow solid, Mp 52.6–54.3 °C; 50% yield (71% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.15 and 9.99 (s, 1H, CHO rotameric), 7.99 (d, *J* = 7.7, 1H), 7.71 (t, *J* = 7.7, 1H), 7.55 (t, *J* = 7.7, 1H), 7.30 (d, *J* = 7.7, 1H), 3.40 and 3.30 (s, 3H, CH_3 rotameric), 2.32 and 1.81 (s, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.4, 169.4, 145.2, 134.8, 131.4, 129.2, 128.1, 128.0, 36.9, 21.5; HRMS (ESI) m/z 178.0873 ($M+H^+$), calc. for $C_{10}H_{12}NO_2$ 178.0868.


13b

Pale yellow oil, 66% yield (74% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.08 and 9.91 (s, 1H, CHO rotameric), 7.66–7.57 (m, 1H), 7.40 (t, *J* = 8.1, 1H), 7.39–7.28 (m, 1H), 3.41 and 3.29 (s, 1H, CH_3 rotameric), 2.31 and 1.81 (s, 1H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.0, 170.4, 163.7, 160.4, 142.5, 142.4, 134.2, 134.1, 131.2, 131.1, 123.0, 122.7, 116.2, 115.9, 38.1, 22.5; HRMS (ESI) m/z 196.0773 ($M+H^+$), calc. for $C_{10}H_{11}FNO_2$ 196.0774.

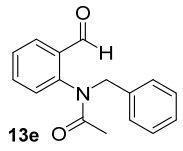

13c

Pale yellow oil, 73% yield (80% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.02 and 9.84 (s, 1H, CHO rotameric), 7.88 and 7.80 (s, 1H, CH rotameric), 7.62–7.52 (m, 1H), 7.21–7.12 (m, 1H), 3.34 and 3.22 (s, 3H, CH_3 rotameric), 2.25 and 1.75 (s, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.3, 188.0, 170.1, 144.6, 135.7, 135.3, 134.9, 133.6, 130.6, 130.1, 129.9, 129.2, 39.7, 38.0, 22.4, 22.0; HRMS (ESI) m/z 212.0477 ($M+H^+$), calc. for $C_{10}H_{11}ClNO_2$ 212.0478.

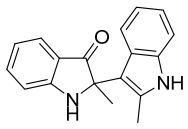

13d

Pale yellow Solid, Mp 106.5–107.5 °C; 60% yield (80% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.05 (s, 1H), 7.69 (d, *J* = 6.1,

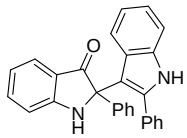
1H), 7.47 (t, 1H), 7.29 (t, 1H), 7.29–7.05 (m, 6H), 3.45 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.2, 170.9, 146.4, 135.0, 131.8, 130.6, 129.9, 129.3, 128.4, 127.9, 127.9, 39.2; HRMS (ESI) m/z 240.1023 ($M+H^+$), calc. for $C_{15}H_{14}NO_2$ 240.1025.


13e

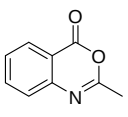
Pale yellow oil, 52% yield (67% yield, 10 mol% TEMPO used); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.63 (s, 1H), 7.84 (d, J = 7.8, 1H), 7.55 (t, J = 7.4, 1H), 7.43 (t, J = 7.4, 1H), 7.18 (s, 3H), 7.08 (s, 2H), 7.00 (d, J = 7.8, 1H), 4.85 (s, 2H), 1.74 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.1, 169.9, 144.3, 135.9, 135.4, 133.0, 123.0, 129.6, 129.5, 128.9, 128.6, 128.0, 53.5, 22.7; HRMS (ESI) m/z 254.1186 ($M+H^+$), calc. for $C_{16}H_{16}NO_2$ 254.1181.


15a

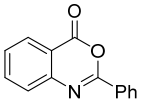
Yellow solid, Mp 194.8–196.5 °C; 87% yield; ^{1}H NMR (300 MHz, DMSO) δ 10.90 (s, 1H), 7.74 (s, 1H), 7.52–7.44 (m, 2H), 7.25–7.21(m, 2H), 6.96–6.86 (m, 2H), 6.81–6.69 (m, 2H), 2.40 (s, 3H), 1.74 (s, 3H); ^{13}C NMR (75 MHz, DMSO) δ 203.9, 159.9, 137.5, 134.7, 133.0, 127.2, 124.4, 120.0, 119.4, 118.4, 117.7, 117.0, 111.8, 110.5, 108.47, 66.2, 24.4, 14.0; HRMS (ESI) m/z 227.1345 ($M+H^+$), calc. for $C_9H_{10}NO_2$ 227.1341.


15b

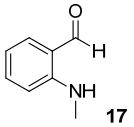
Yellow solid, Mp 227.2−228.8 °C; 95% yield; ^{1}H NMR (300 MHz, DMSO) δ 11.36 (s, 1H), 8.36 (s, 1H), 7.52 (t, J = 7.6, 1H), 7.38–7.34 (m, 3H), 7.26 (d, J = 7.6, 1H), 7.15 (t, J = 8.3, 3H), 7.06–6.98 (m, 7H), 6.79–6.70 (m, 2H), 6.62 (d, J = 8.3, 1H); ^{13}C NMR (75 MHz, DMSO) δ 200.5, 160.1, 139.8, 138.0, 137.5, 135.8, 133.2, 129.5, 127.6, 127.4, 127.3,27.1, 127.0, 124.4, 121.2, 120.3, 118.7, 118.5, 117.5, 111.9, 111.3, 111.0, 71.2; HRMS (ESI) m/z 401.1652 ($M+H^+$), calc. for $C_{28}H_{21}N_2O$ 401.1654.


16a

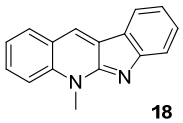
Pale yellow solid, Mp 71.3–72.5 °C; 58% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 (d, J = 7.8, 1H), 7.79 (t, J = 7.8, 1H), 7.55–7.50 (m, 2H), 2.47 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.2, 159.7, 146.4, 136.5, 128.4, 128.2, 126.4, 116.6, 21.4; HRMS (ESI) m/z 162.0559 ($M+H^+$), calc. for $C_9H_8NO_2$ 162.0555.


16b

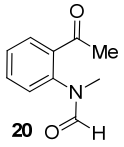
Red solid, Mp 108.9–110.1 °C; 50% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32 (d, *J* = 7.7, 2H), 8.25 (d, *J* = 8.0, 1H), 7.83 (t, *J* = 7.7, 1H), 7.70 (d, *J* = 8.0, 1H), 7.58–7.50 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.6, 157.1, 147.0, 136.6, 132.6, 130.2, 128.7, 128.6, 128.3, 128.3, 127.2, 117.0; HRMS (ESI) m/z 224.0714 ($M+H^+$), calc. for $C_{14}H_{10}NO_2$ 224.0712.


17

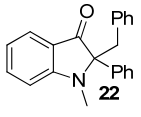
Yellow oil, 81% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.82 (s, 1H), 8.26 (s, 1H), 7.44 (dd, *J* = 17.8, 8.1, 2H), 6.69 (t, *J* = 8.1, 2H), 2.94 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 193.9, 151.6, 136.6 135.9, 118.4, 114.7, 110.4, 29.1; HRMS (ESI) m/z 136.0765 ($M+H^+$), calc. for $C_8H_{10}NO$ 136.0762.


18

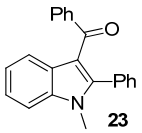
Red solid, Mp 105.8–106.1 °C; 71% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.94 (d, *J* = 7.6, 1H), 7.86 (d, *J* = 7.6, 1H), 7.64 (q, *J* = 8.1, 1H), 7.46 (t, *J* = 7.3, 1H), 7.34 (t, *J* = 7.3, 1H), 7.17–7.12 (m, 1H), 4.23 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.11, 155.3, 136.8, 130.3, 129.9, 129.2, 128.0, 127.9, 123.9, 121.8, 124.0, 120.7, 119.8, 117.6, 114.1, 33.0; HRMS (ESI) m/z 233.1075 ($M+H^+$), calc. for $C_{12}H_{16}NO_3$ 233.1079.


20

Yellow solid, Mp 135.7–136.1 °C; 26% yield (Conditions A), 84% (Conditions B); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 and 8.17 (d, *J* = 7.4 Hz, 1H, CHO rotameric), 7.74 (dd, *J* = 17.1, 7.6 Hz, 1H), 7.67–7.56 (m, 1H), 7.55–7.41 (m, 1H), 7.31 (d, *J* = 8.3 Hz, 1H), 3.30 and 3.42 (d, *J* = 35.9 Hz, 3H, CH_3 rotameric), 2.60 and 2.59 (d, *J* = 13.5 Hz, 3H, CH_3 rotameric); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.1, 199.6, 162.6, 162.4, 139.9, 137.4, 137.2, 136.6, 132.7, 132.3, 129.8, 128.4, 128.0, 127.7, 127.4, 37.8, 33.8, 29.5, 28.6; HRMS (ESI) m/z 178.0870 ($M+H^+$), calc. for $C_{10}H_{12}NO_2$ 178.0868.


22

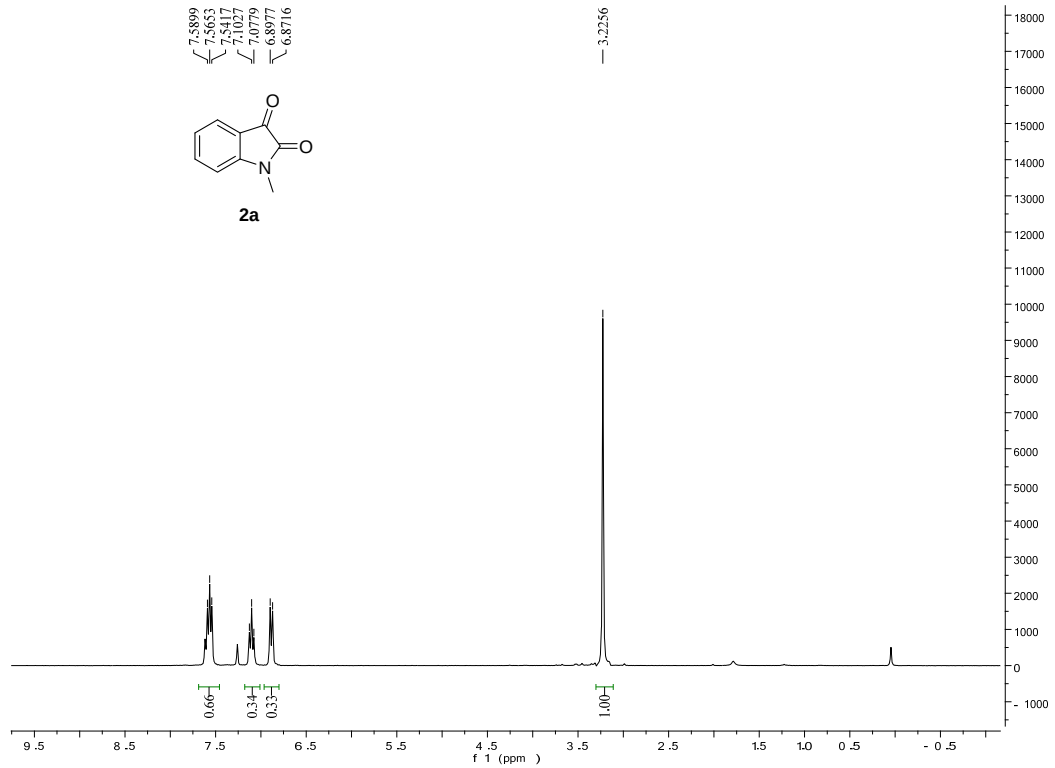
Yellow solid, Mp 187.3–190.1 °C; 44% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65–7.04 (m, 14H), 4.08 (d, *J* = 16.0 Hz, 1H), 3.72 (d, *J* = 16.1 Hz, 1H), 3.34 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.4, 170.0, 143.6, 135.6, 133.6, 132.6, 130.2, 129.6, 129.6, 128.7, 128.6, 127.6, 127.3, 127.1, 126.6, 47.4, 38.4. HRMS (ESI) m/z 314.1549 ($M+H^+$), calc. for $C_{22}H_{20}NO$ 314.1545.

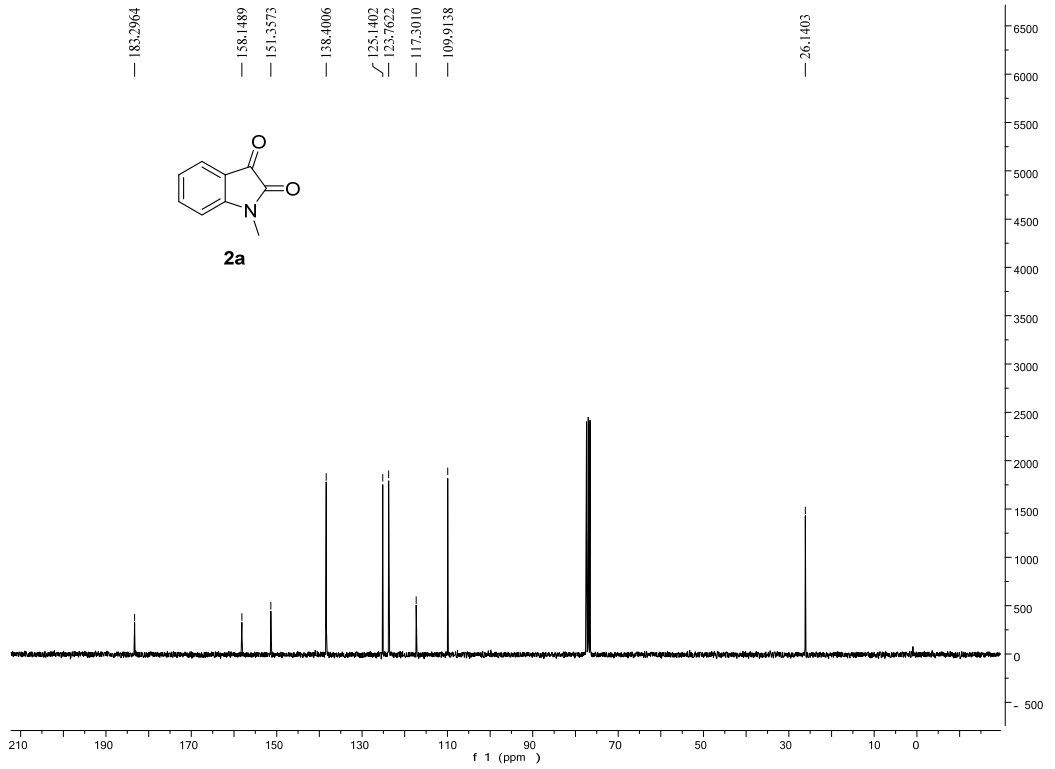


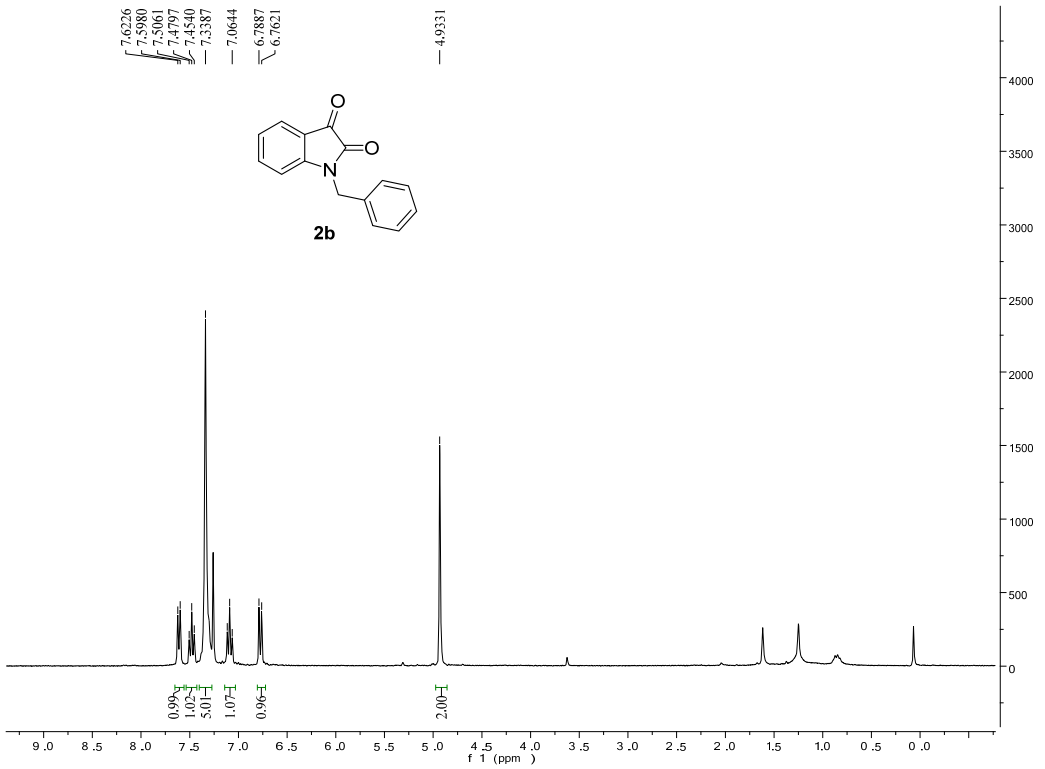
23

Yellow solid, Mp 119.8–120.5 ^{o}C; 38% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.7 Hz, 1H), 7.54 (d, *J* = 7.6 Hz, 2H), 7.50–7.21 (m, 9H), 7.14 (t, *J* = 7.3 Hz, 2H), 3.71 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 192.9, 146.4, 140.1, 137.2, 130.9, 130.9, 130.7, 129.2, 128.7, 128.0, 127.6, 127.5, 114.7, 109.8, 31.2. HRMS (ESI) m/z 314.1387 ($M+H^+$), calc. for $C_{22}H_{18}NO$ 314.1388.

15. ^{1}H and ^{13}C NMR spectra



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7.4540
7.3387
7.0644
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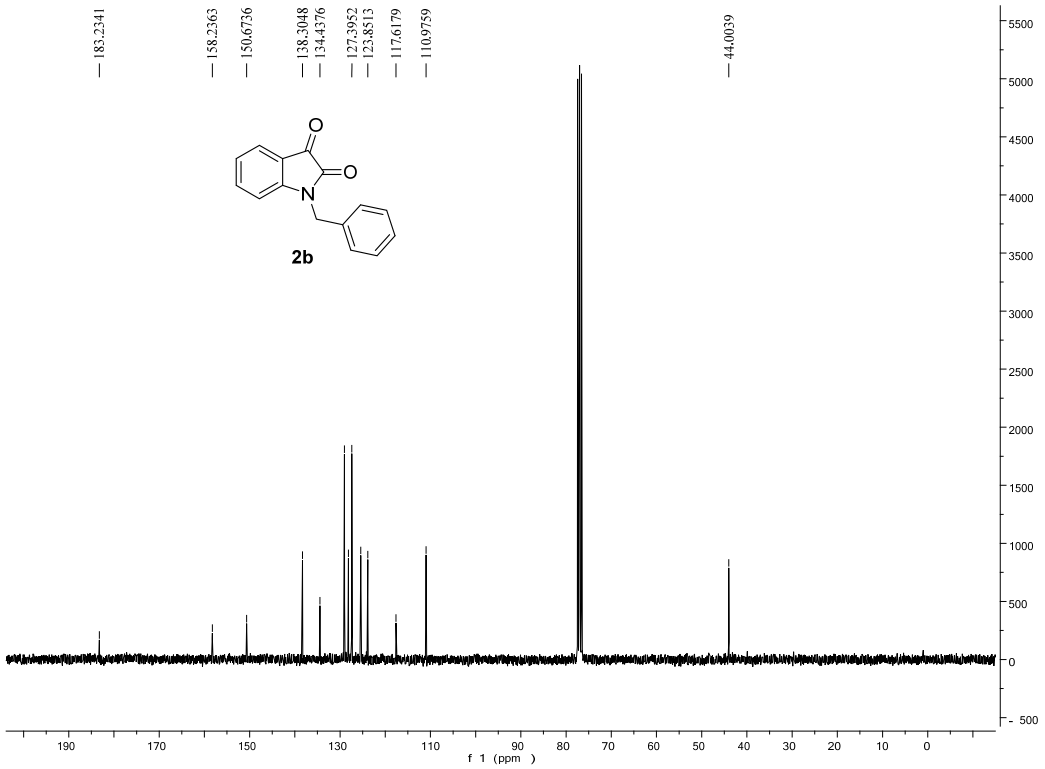
4.9331
2b
0.99
1.02
5.01
1.07
0.96
2.00
f 1 (ppm)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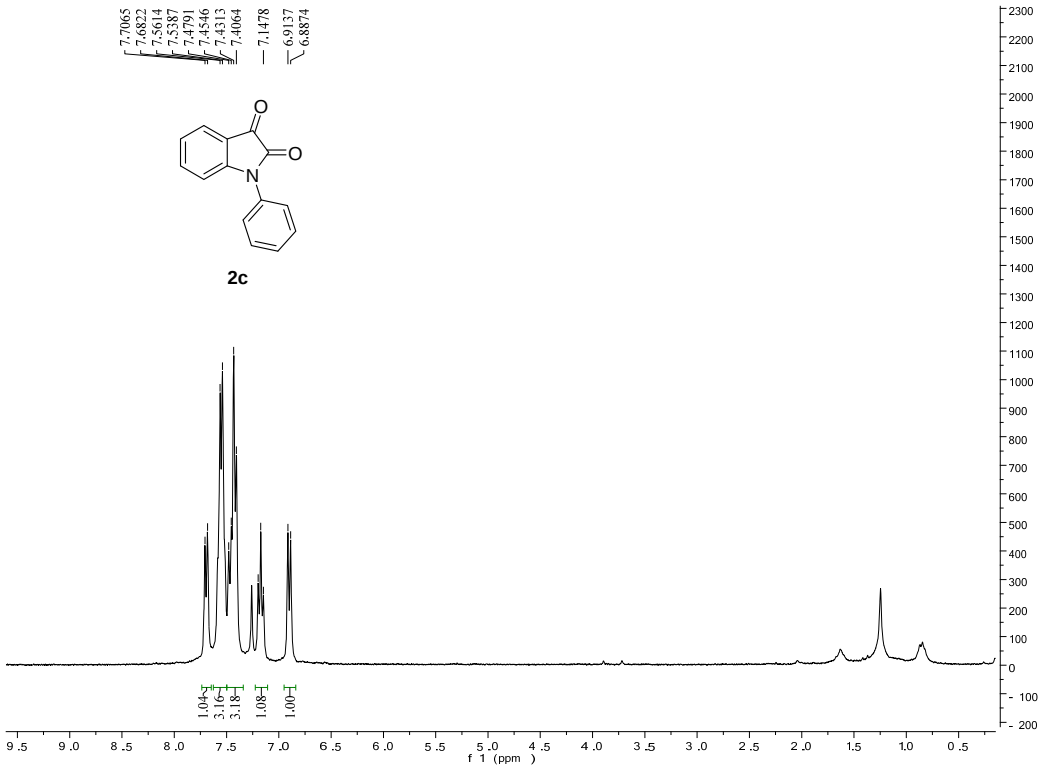
2b
f 1 (ppm)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7.4546
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O
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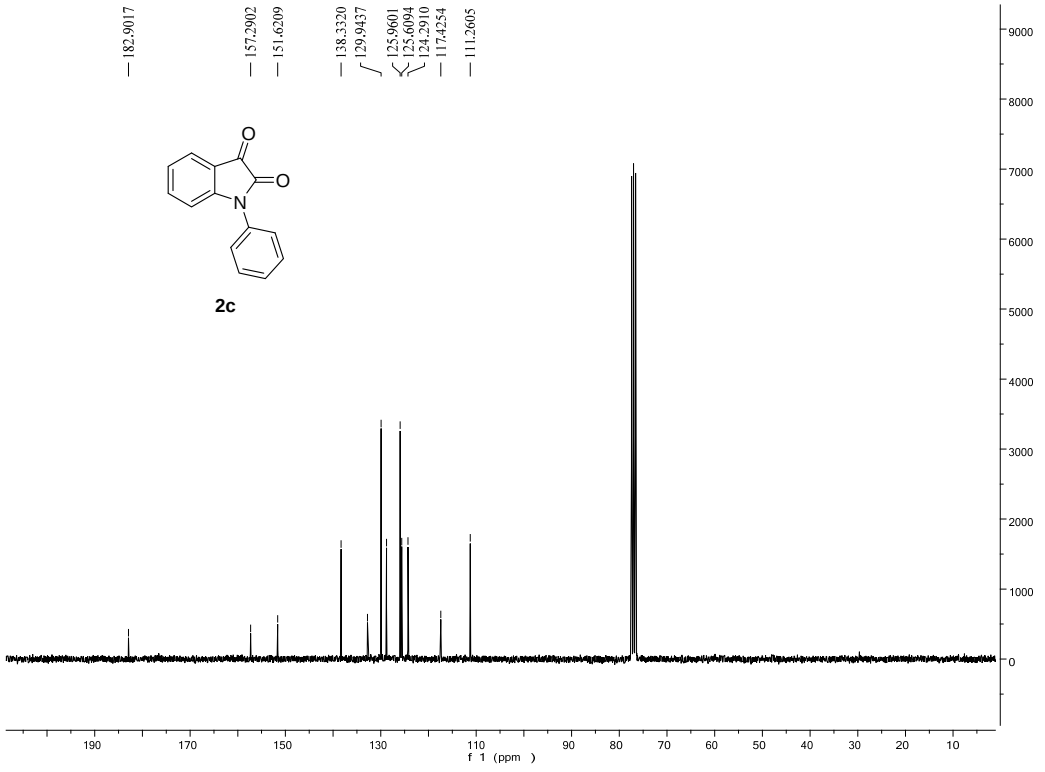
3.18
1.08
1.00
f 1 (ppm)

2c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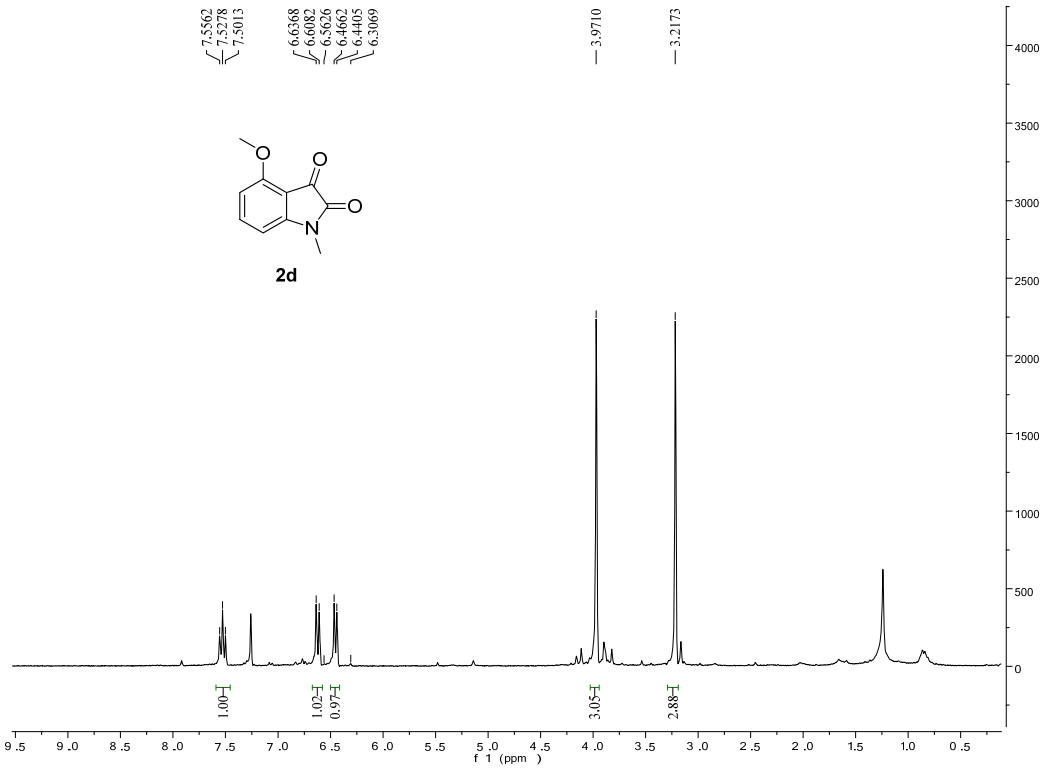
N
f 1 (ppm)

2c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0.97
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2.88
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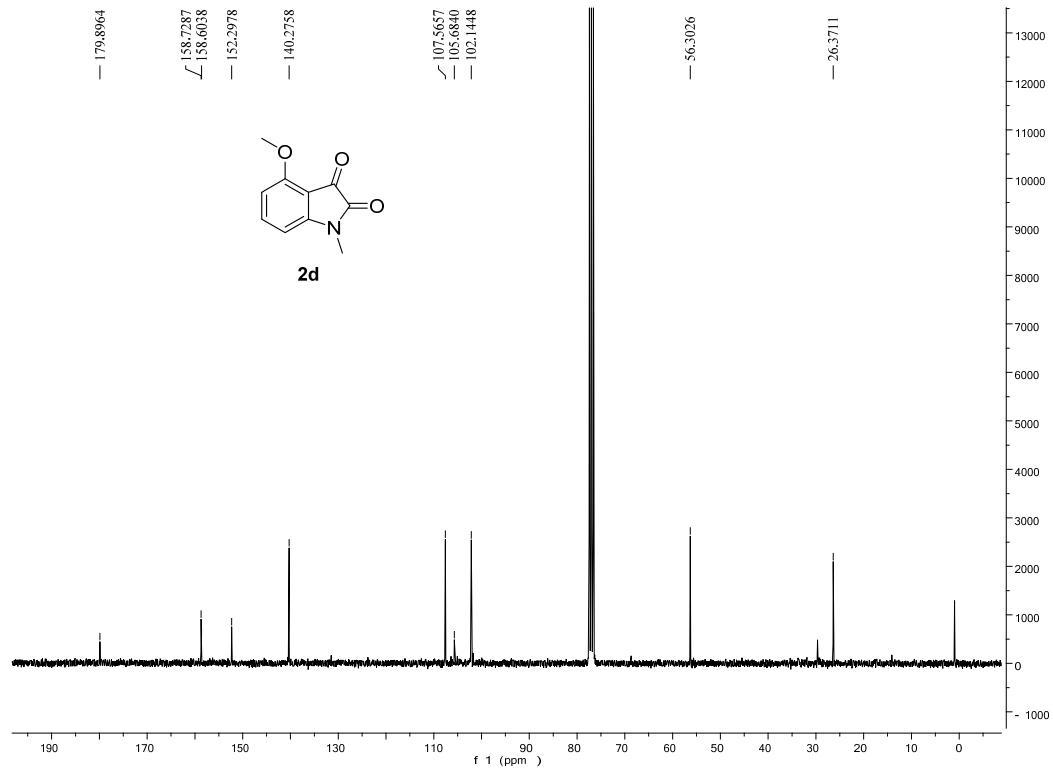
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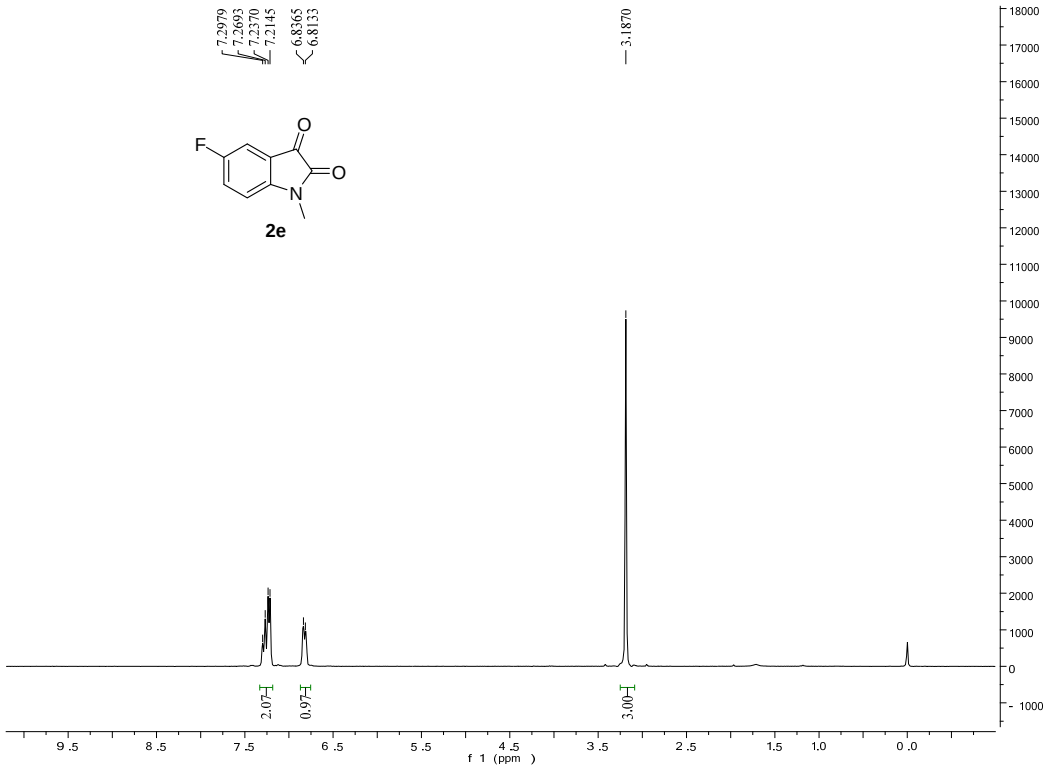
2d
f 1 (ppm)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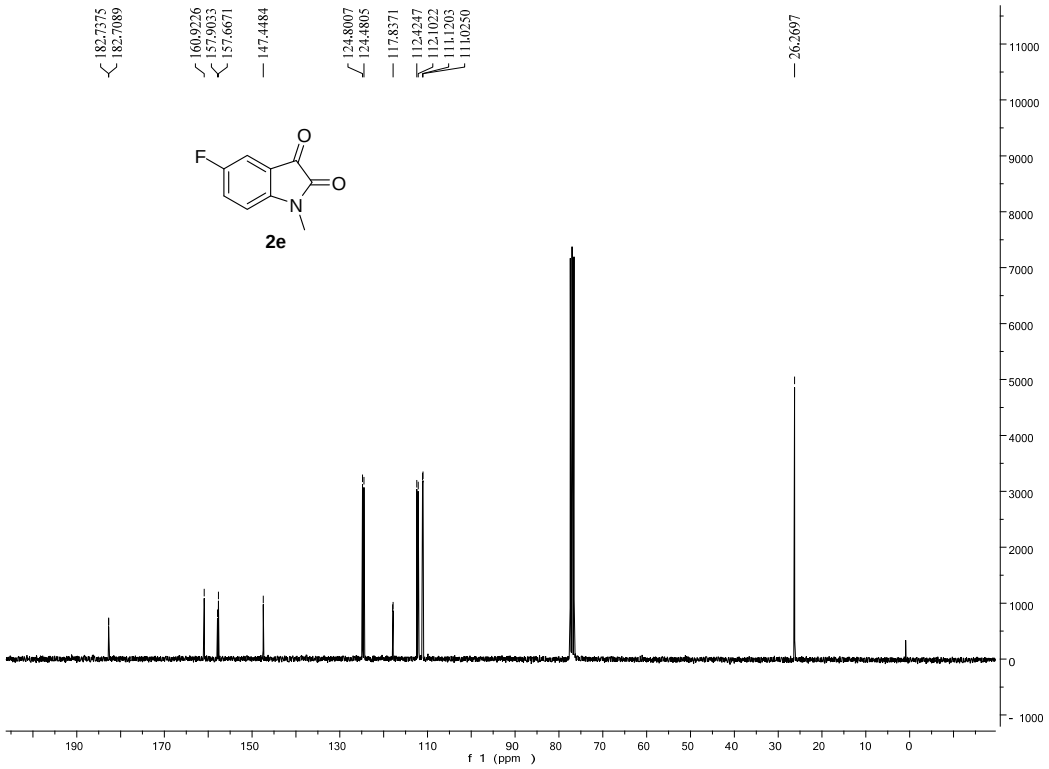
3.00
f 1 (ppm)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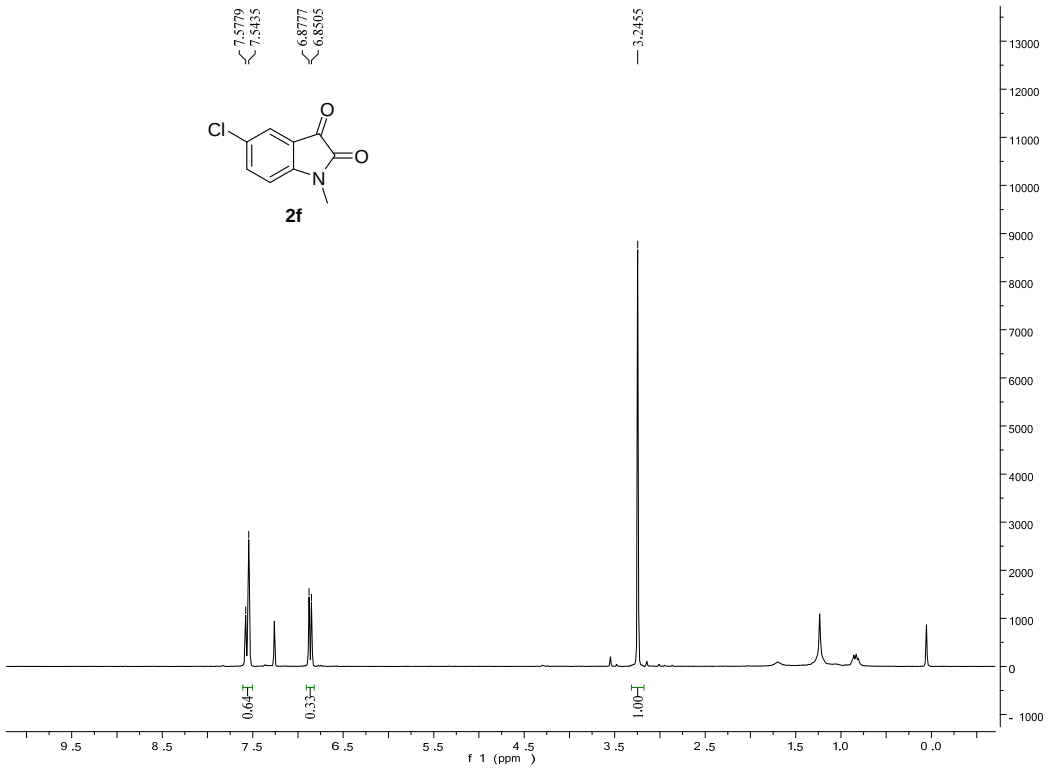
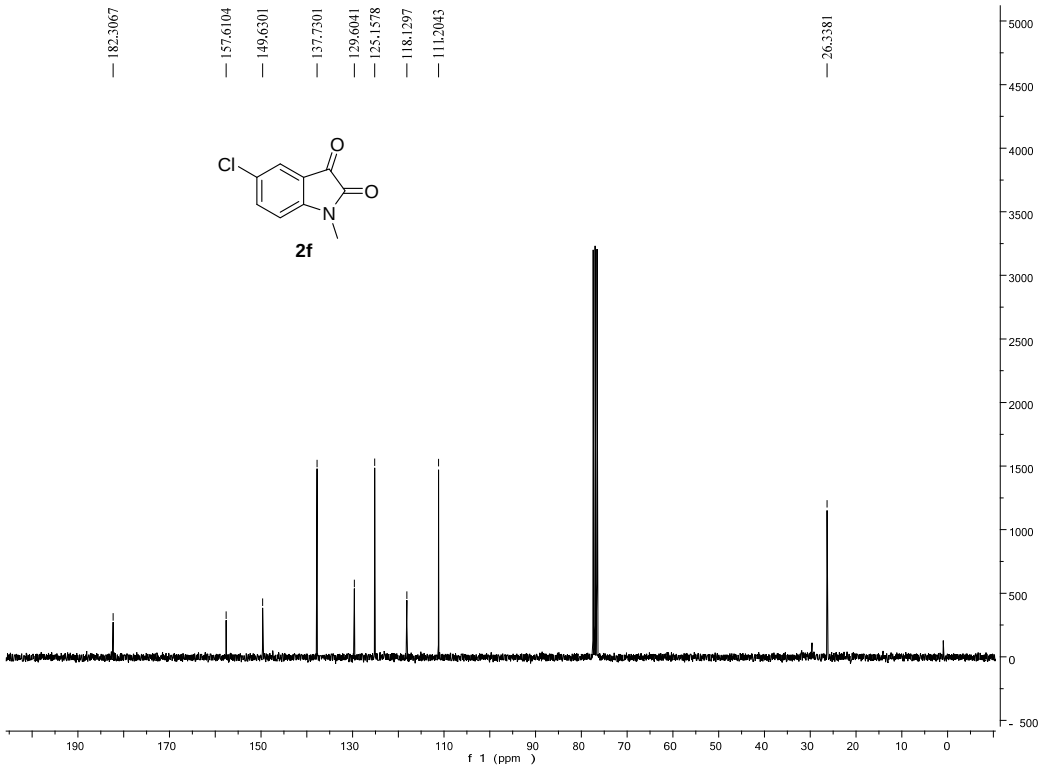
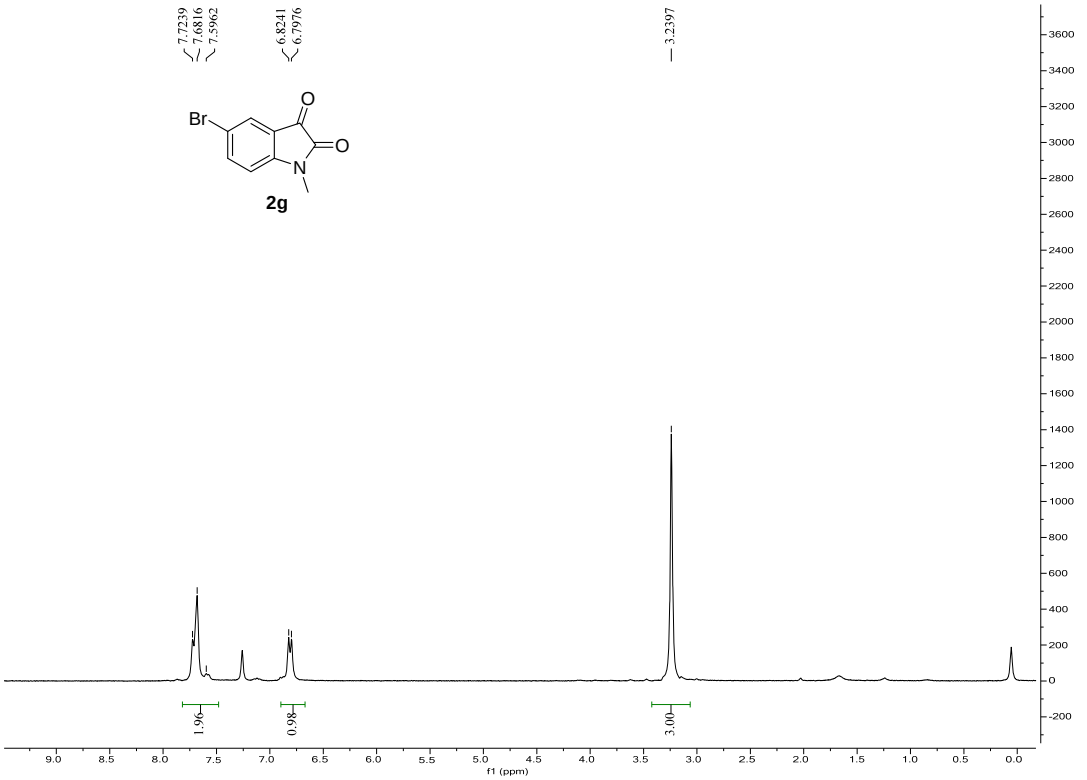
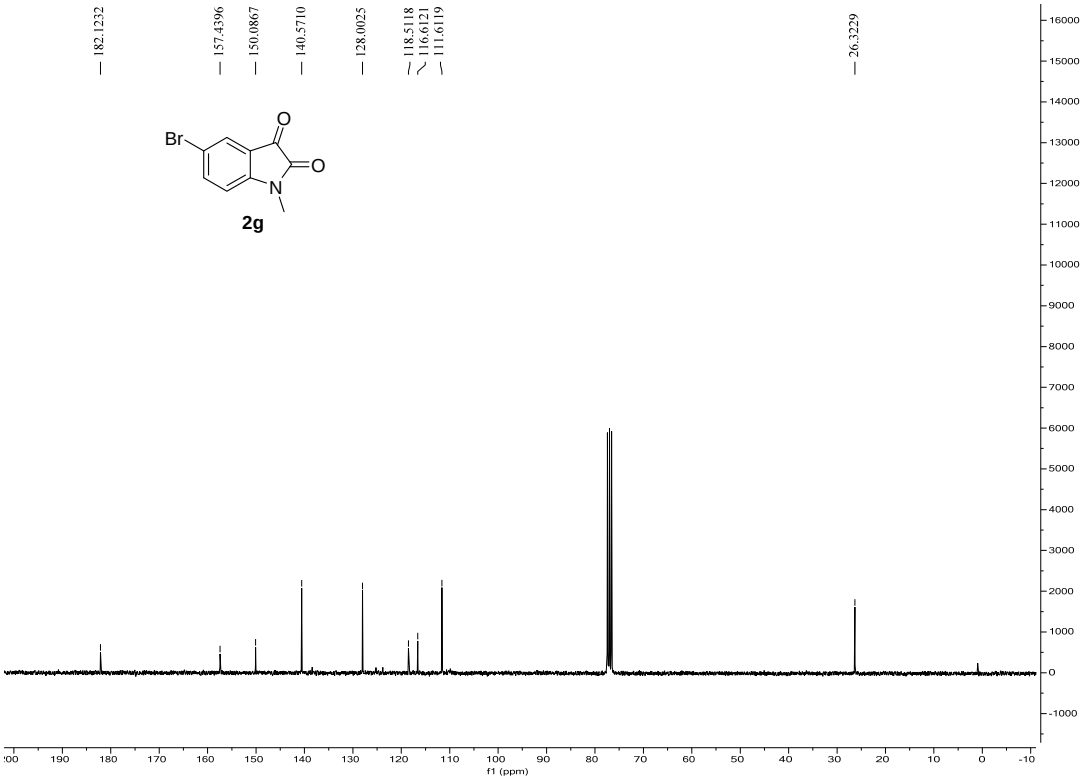
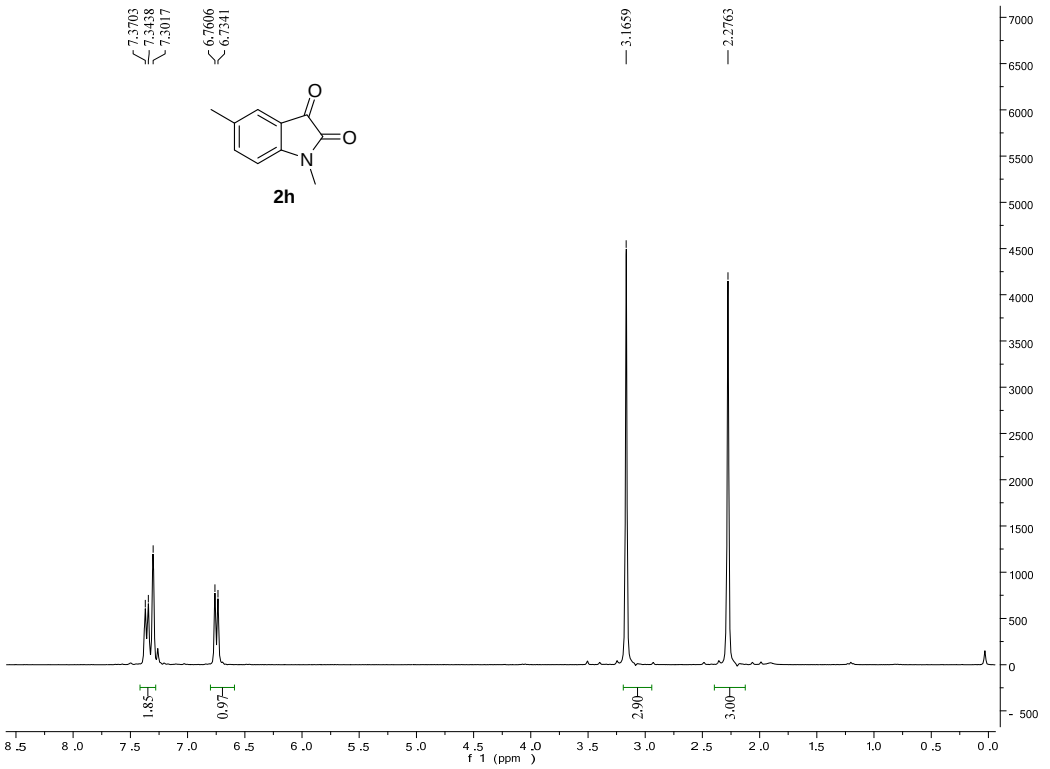
26.2697
F
O
O
N
2e
f 1 (ppm)

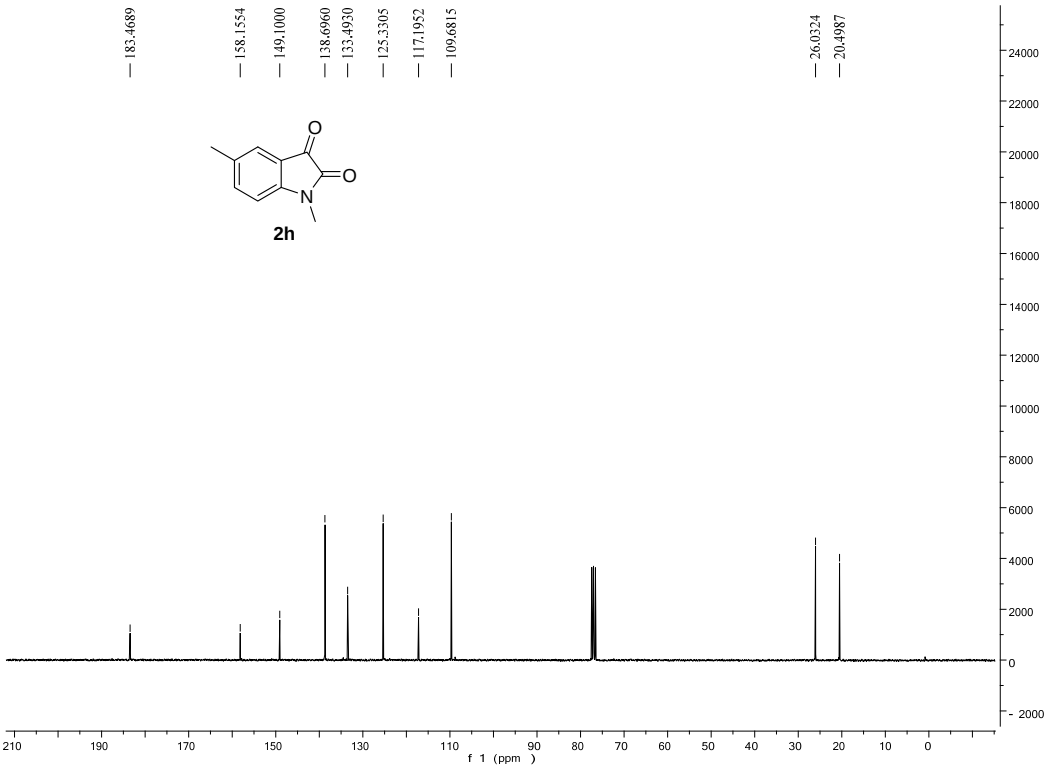

7.5779
7.5435
6.8777
6.8505
3.2455
Cl
O
O
N
2f
0.64
0.33
1.00
f 1 (ppm)

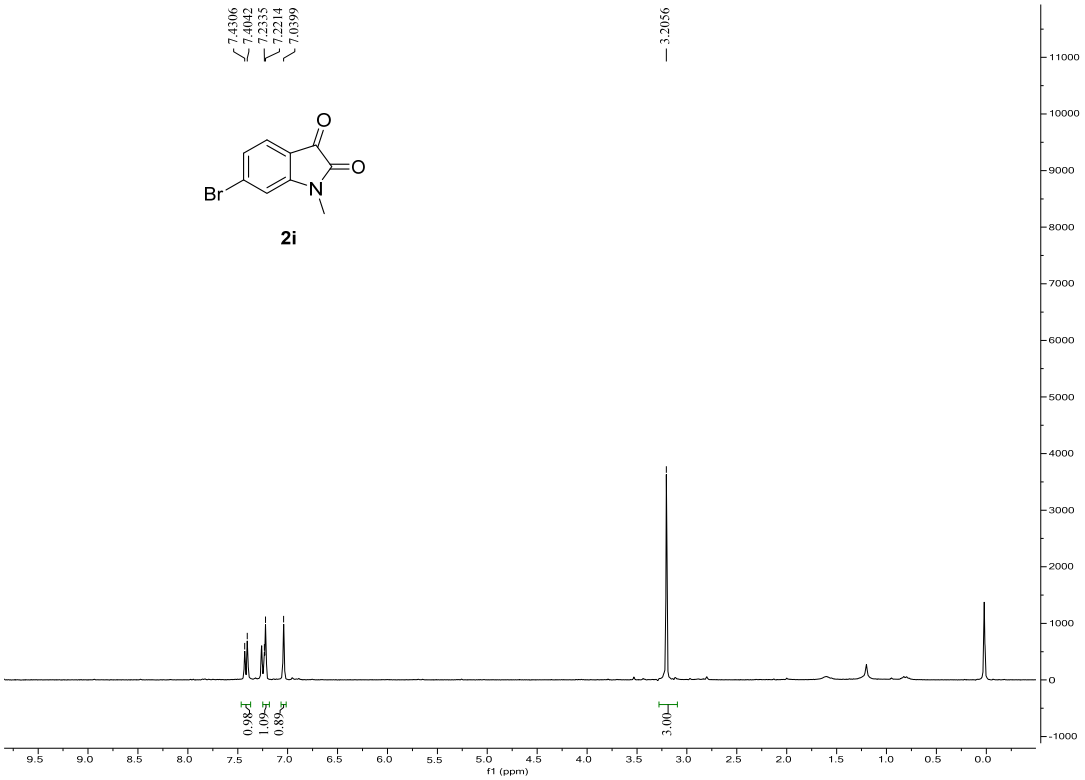

182.3067
157.6104
149.6301
137.7301
129.6041
125.1578
118.1297
111.2043
26.3381
Cl
O
O
N
2f
f 1 (ppm)

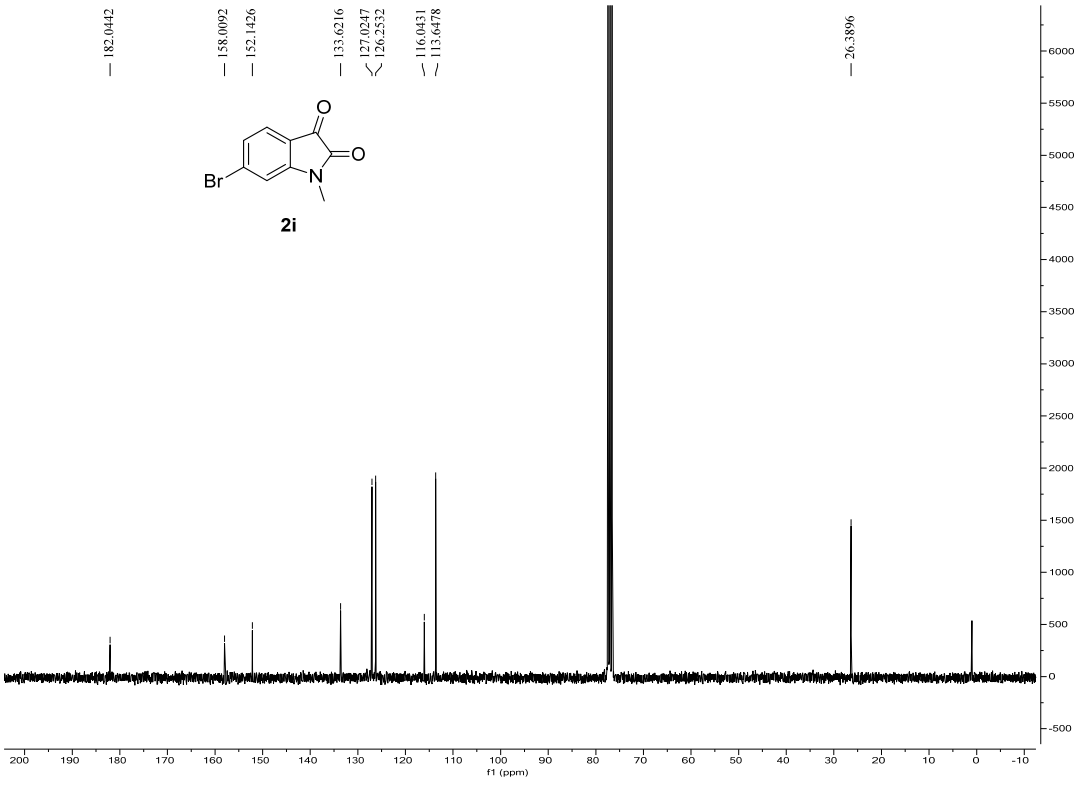

7.7239
7.6816
7.5962
6.8241
6.7976
3.2397
Br
O
O
N
2g
1.96
0.98
3.00
f1 (ppm)

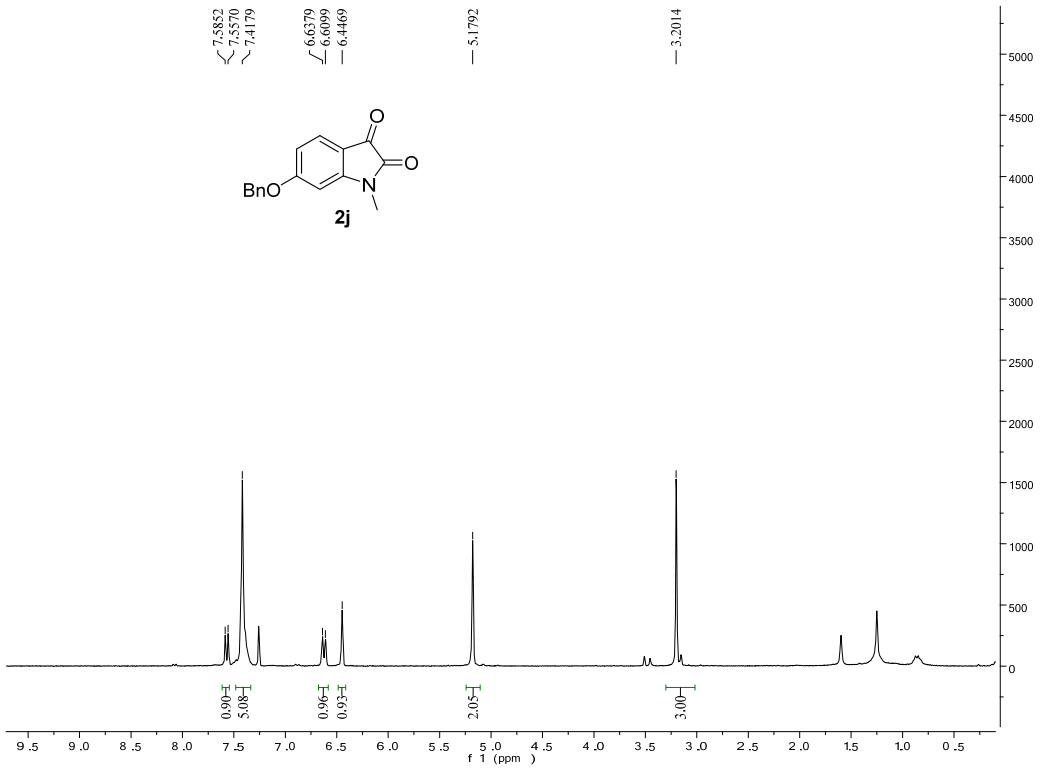

182.1232
157.4396
150.0867
140.5710
128.0025
118.5118
116.6121
111.6119
26.3229
Br
O
O
N
2g
f1 (ppm)

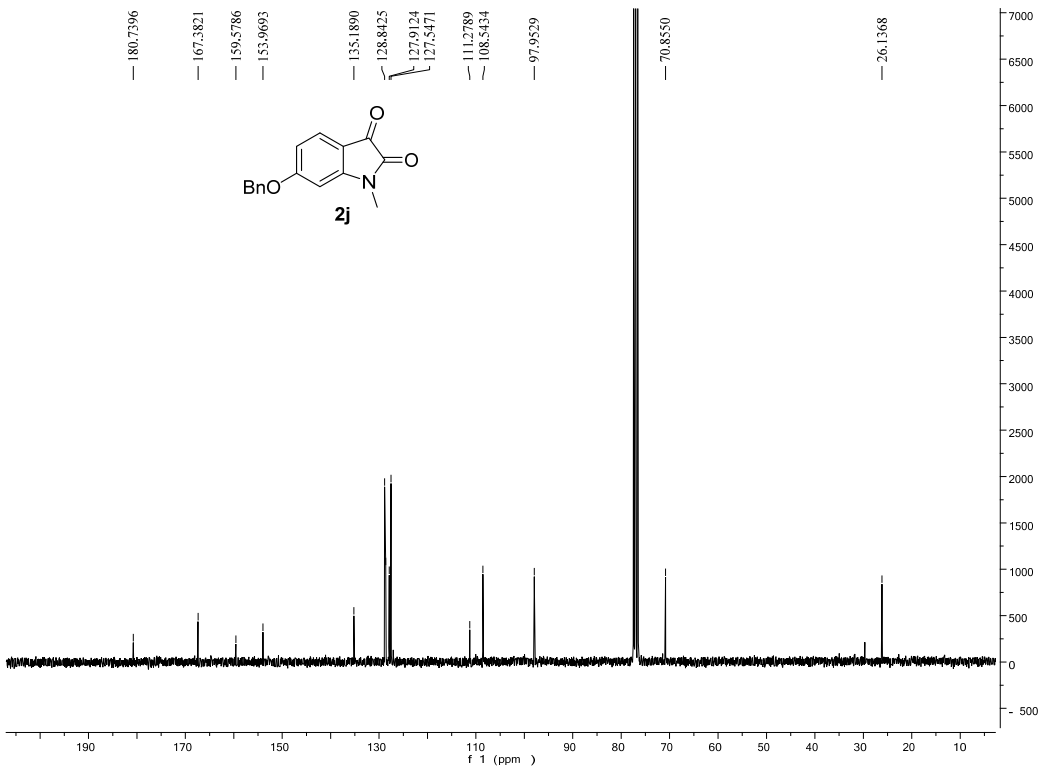

7.3703
7.3438
7.3017
6.7606
6.7341
3.1659
2.2763
O
O
N
2h
1.85
0.97
2.90
3.00
f 1 (ppm)

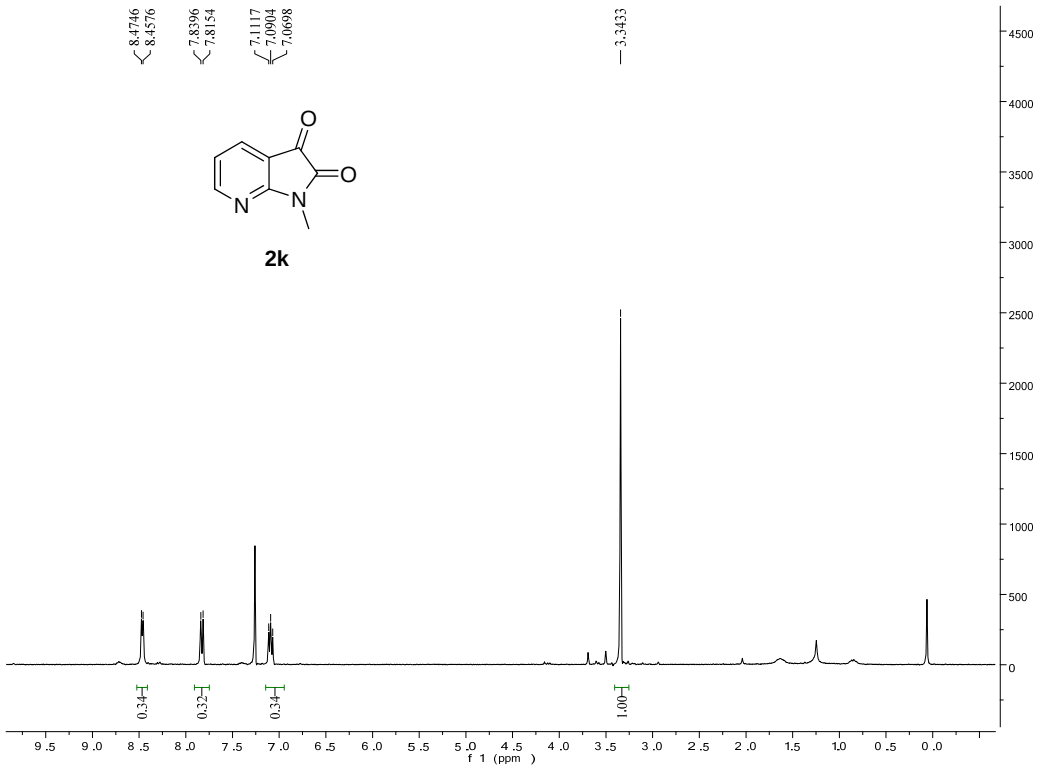

183.4689
158.1554
149.1000
138.6960
133.4930
125.3305
117.1952
109.6815
26.0324
20.4987
O
O
N
2h
f 1 (ppm)

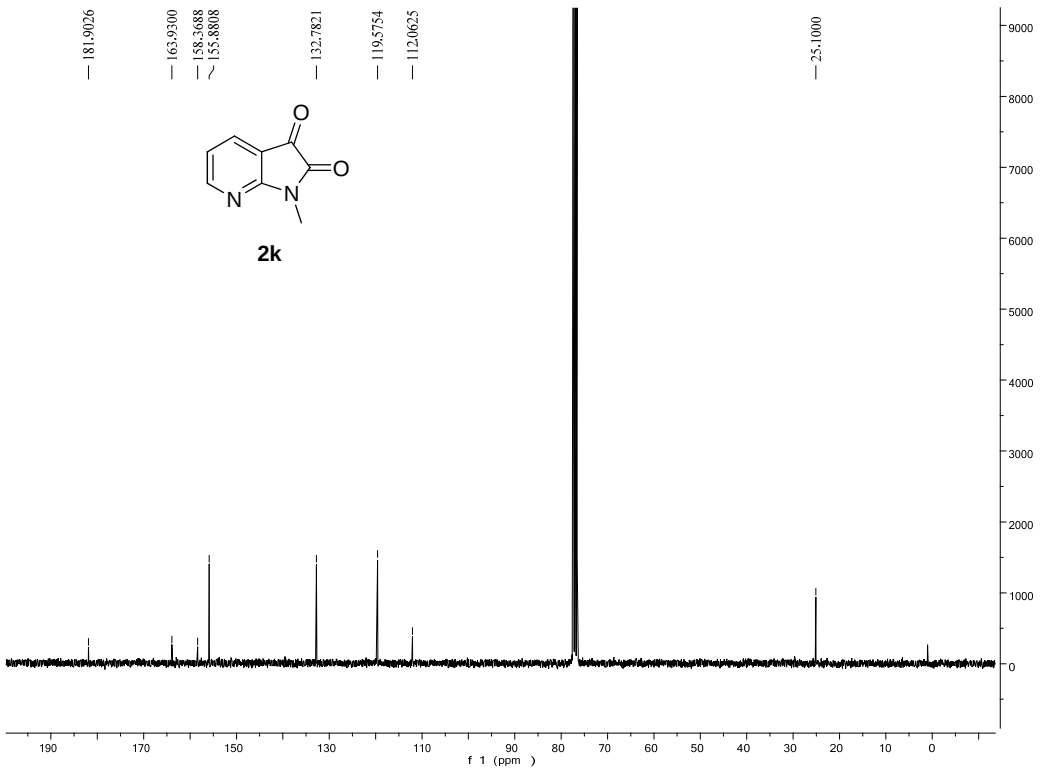

7.4306
7.4042
7.2335
7.2214
7.0399
3.2056
Br
2i
0.98
1.09
0.89
3.00
f1 (ppm)

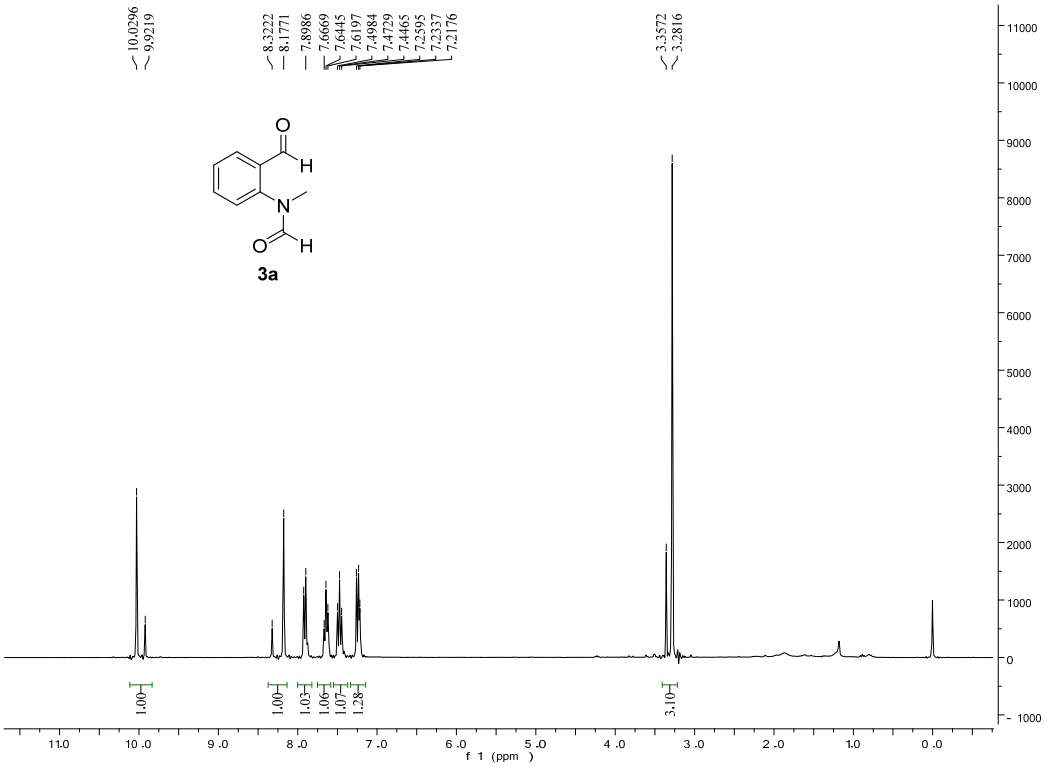

182.0442
158.0092
152.1426
133.6216
127.0247
126.2532
116.0431
113.6478
26.3896
Br
2i
f1 (ppm)

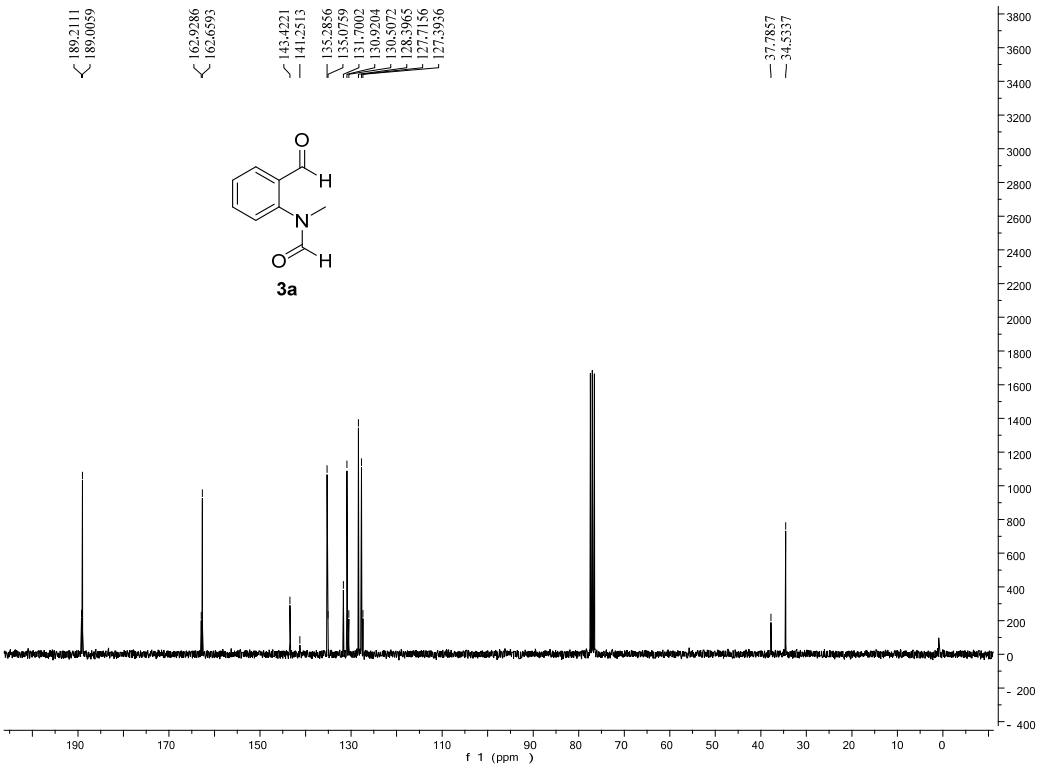

7.5852
7.5570
7.4179
6.6379
6.6099
6.4469
5.1792
3.2014
BnO
2j
0.90
5.08
0.96
0.93
2.05
3.00
f 1 (ppm)


180.7396
167.3821
159.5786
153.9693
135.1890
128.8425
127.9124
127.5471
111.2789
108.5434
97.9529
70.8550
26.1368
BnO
2j
f 1 (ppm)


8.4746
8.4576
7.8396
7.8154
7.1117
7.0904
7.0698
3.3433
O
O
N
N
2k
0.34
0.32
0.34
1.00
f 1 (ppm)


181.9026
163.9300
158.3688
155.8808
132.7821
119.5754
112.0625
25.1000
O
O
N
N
2k
f 1 (ppm)


10.0296
9.9219
8.3222
8.1771
7.8986
7.6669
7.6445
7.6197
7.4984
7.4729
7.4465
7.2595
7.2337
7.2176
3.3572
3.2816
3a
1.00
1.00
1.03
1.06
1.07
1.28
3.10
f 1 (ppm)


189.2111
189.0059
162.9286
162.6593
143.4221
141.2513
135.2856
135.0759
131.7002
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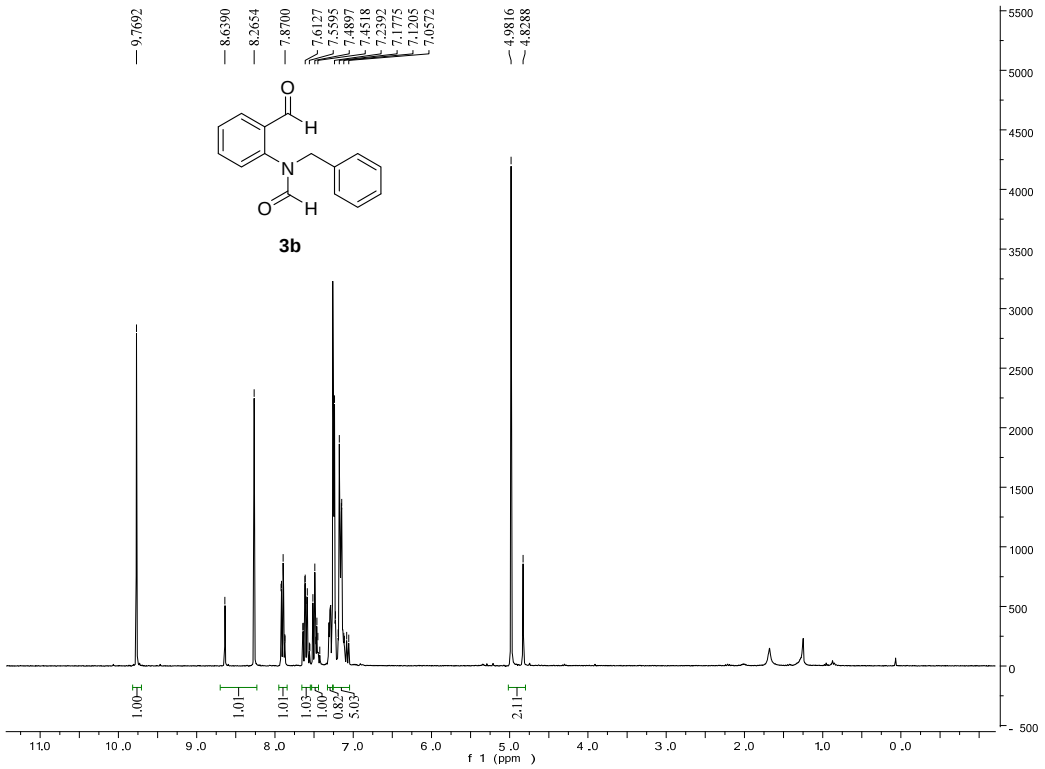
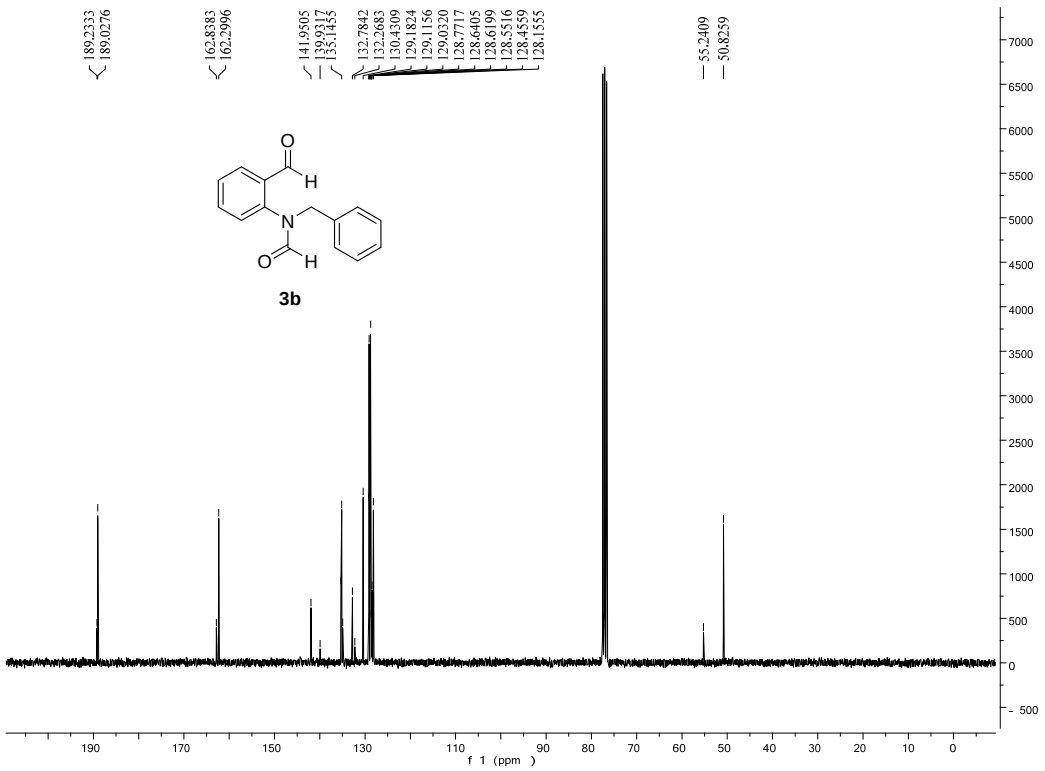
130.5072
128.3965
127.7156
127.3936
37.7857
34.5337
3a
f 1 (ppm)


9.7692
8.6390
8.2654
7.8700
7.6127
7.5595
7.4897
7.4518
7.2392
7.1775
7.1205
7.0572
4.9816
4.8288
3b
1.00
1.01
1.01
1.03
1.00
0.82
5.03
2.11
f 1 (ppm)


189.2333
189.0276
162.8383
162.2996
141.9505
139.9317
135.1455
132.7842
132.2683
130.4309
129.1824
129.1156
129.0320
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55.2409
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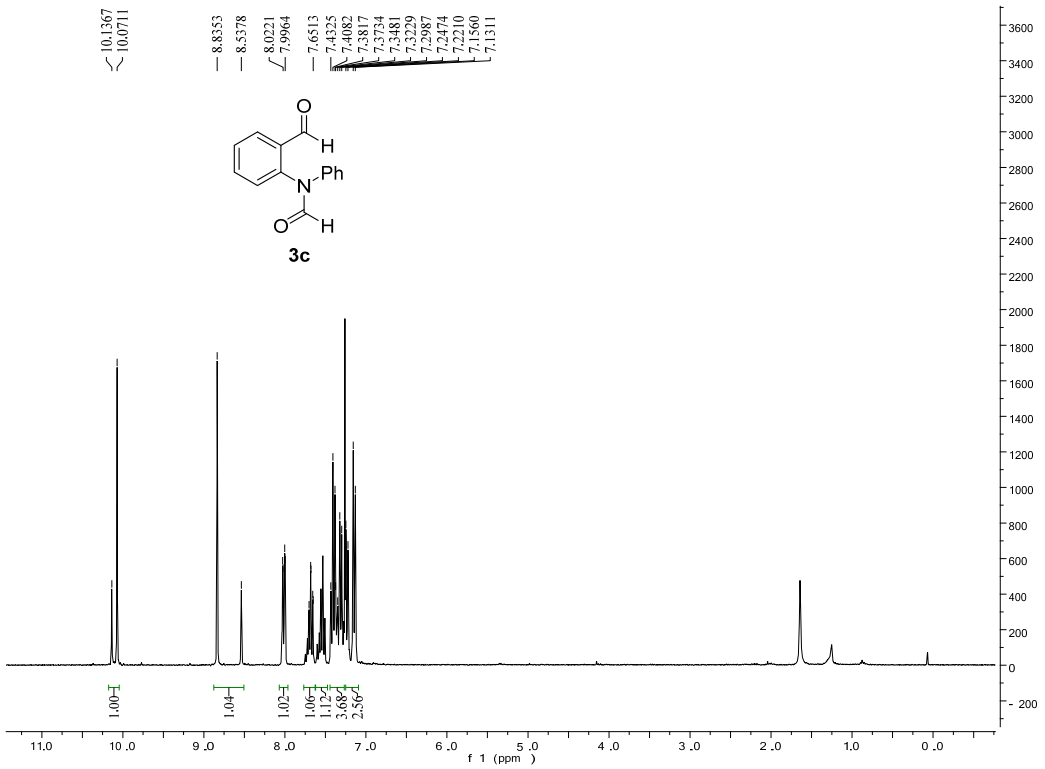
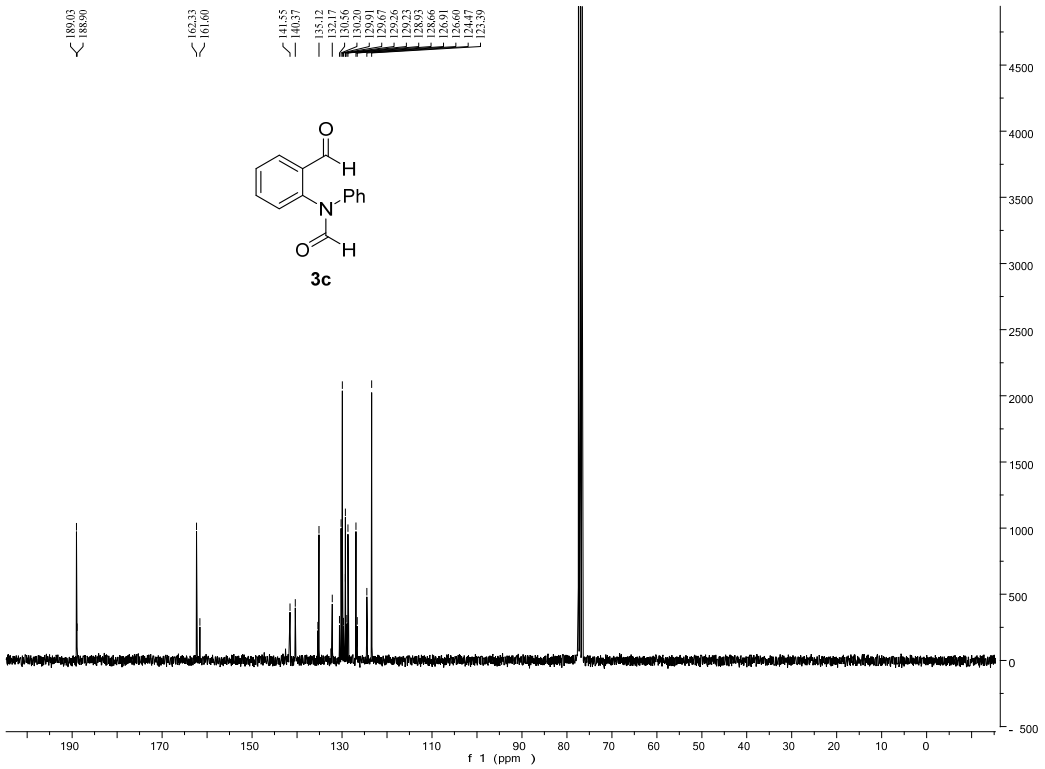
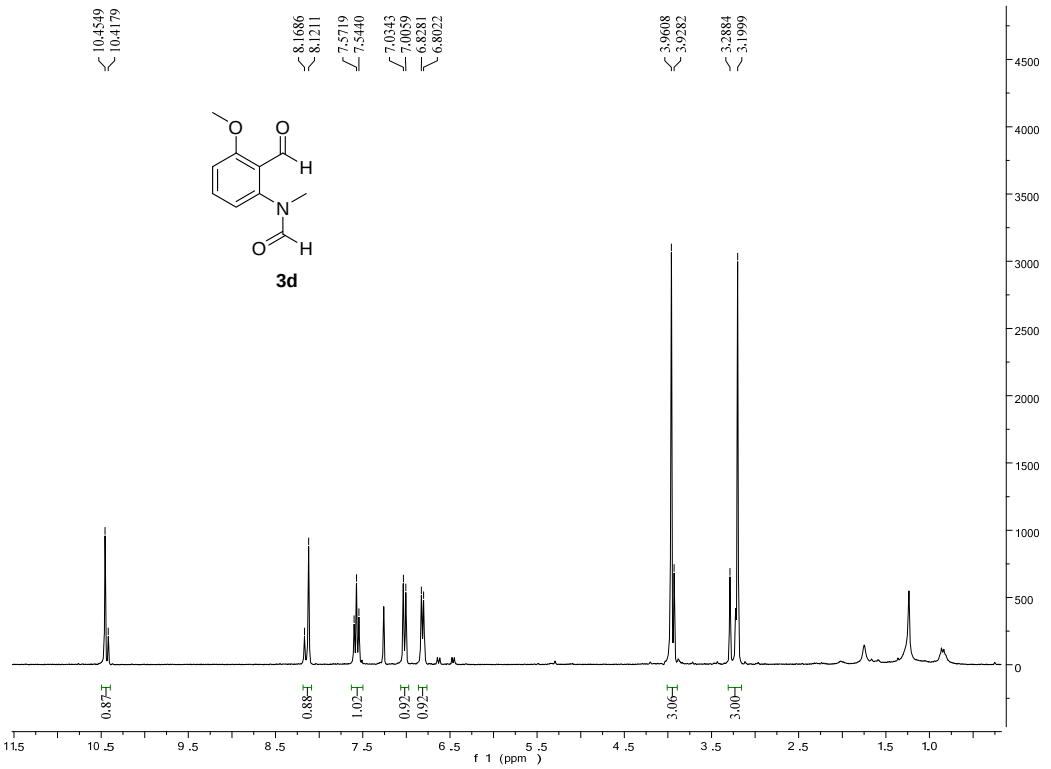
3b
f 1 (ppm)


10.1367
10.0711
8.8353
8.5378
8.0221
7.9964
7.6513
7.4325
7.4082
7.3817
7.3734
7.3481
7.3229
7.2987
7.2474
7.2210
7.1560
7.1311
O
H
Ph
N
O
H
3c
1.00
1.04
1.02
1.06
1.12
3.68
2.56
f 1 (ppm)


189.03
188.90
162.33
161.60
141.55
140.37
135.12
132.17
130.56
130.30
129.91
129.67
129.26
129.23
128.93
128.66
126.91
126.60
124.47
123.39
O
H
Ph
N
O
H
3c
f 1 (ppm)


10.4549
10.4179
8.1686
8.1211
7.5719
7.5440
7.0343
7.0059
6.8281
6.8022
3.9608
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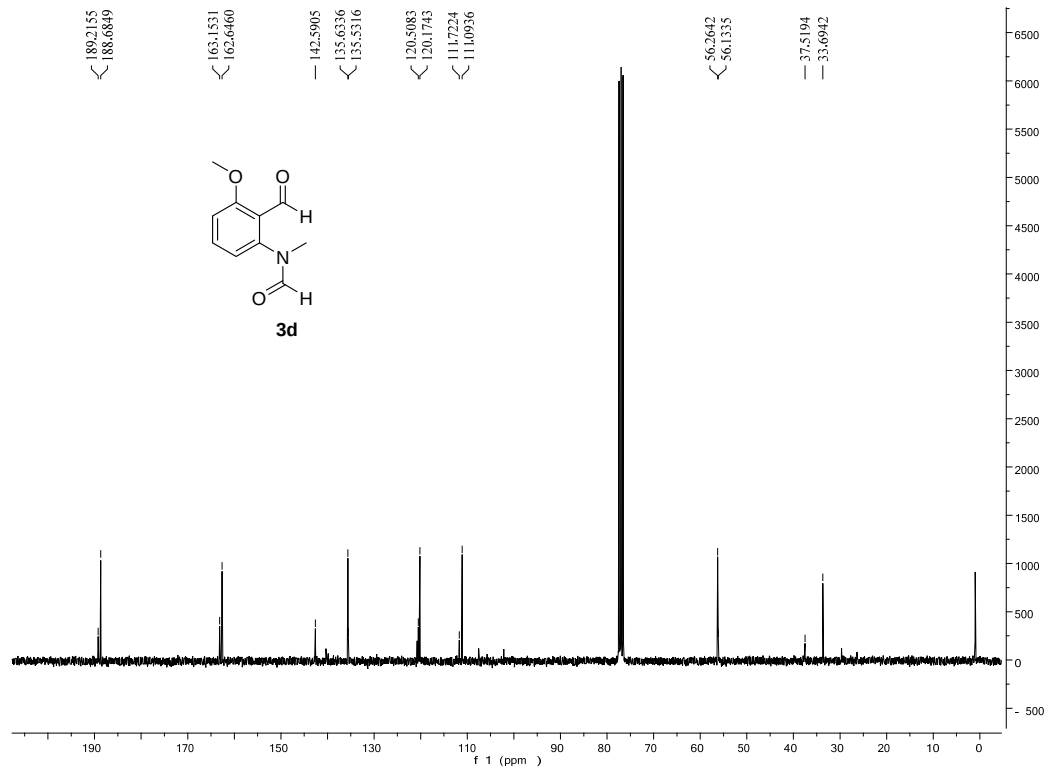
3d
0.87
0.88
1.02
0.92
0.92
3.06
3.00
f 1 (ppm)


189.2155
188.6849
163.1531
162.6460
142.5905
135.6336
135.5316
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111.7224
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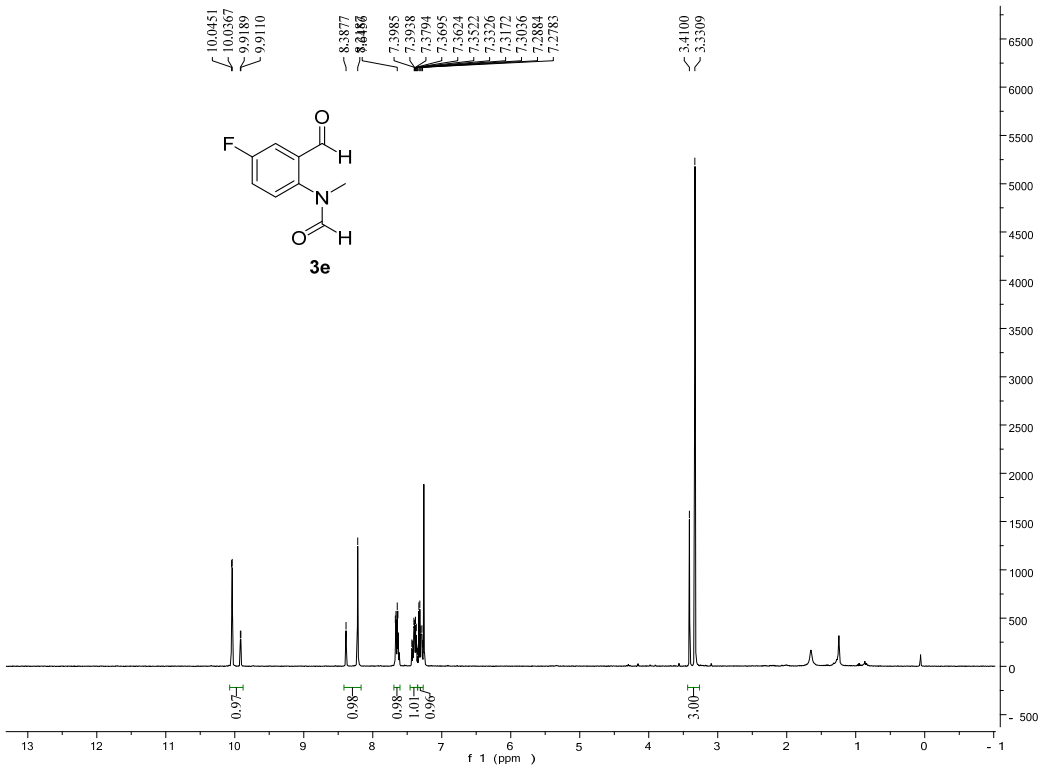
56.2642
56.1335
37.5194
33.6942
3d
f 1 (ppm)


10.0451
10.0367
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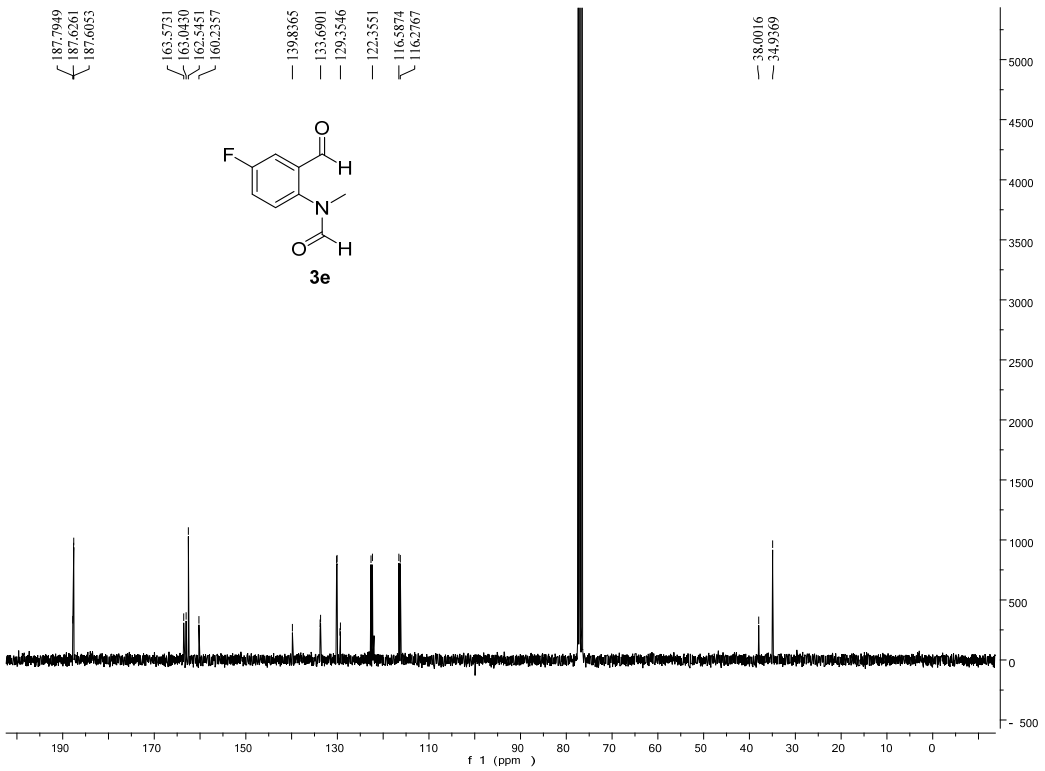
9.9110
8.3877
7.3985
7.3938
7.3794
7.3695
7.3624
7.3522
7.3326
7.3172
7.3036
7.2884
7.2783
3.4100
3.3309
F
O
H
N
O
H
3e
0.97
0.98
0.98
1.01
0.96
3.00
f 1 (ppm)


187.7949
187.6261
187.6053
163.5731
163.0430
162.5451
160.2357
139.8365
133.6901
129.3546
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116.2767
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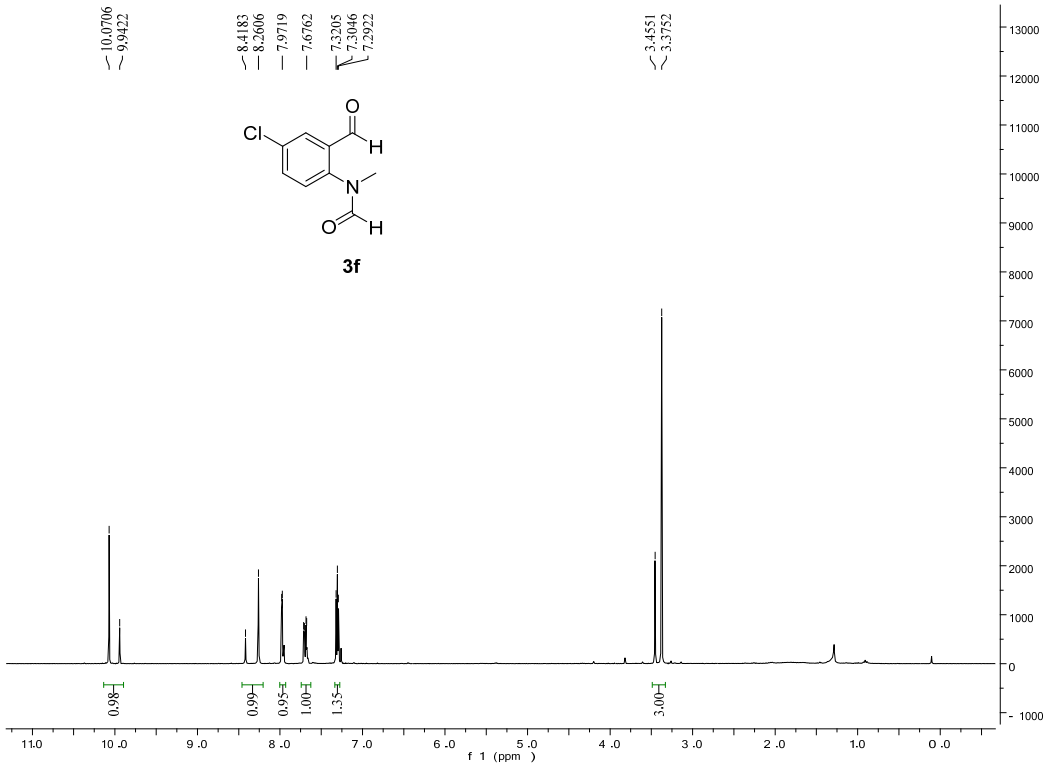
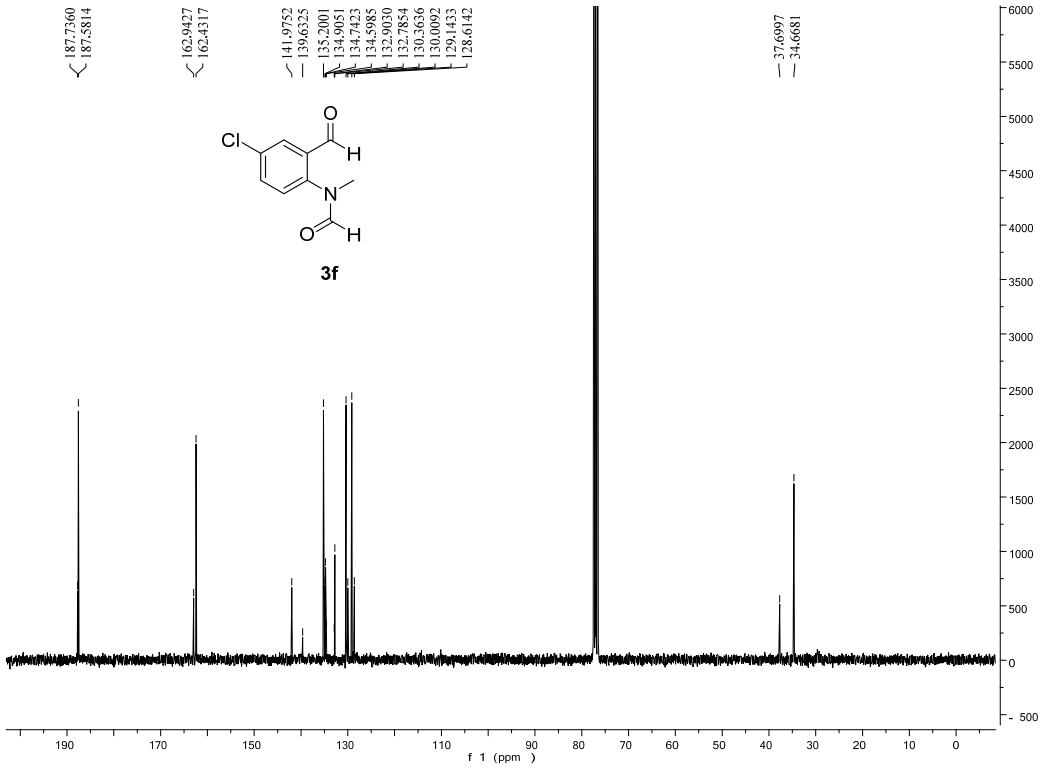
F
O
H
N
O
H
3e
f 1 (ppm)


10.0706
9.9422
8.4183
8.2606
7.9719
7.6762
7.3205
7.3046
7.2922
3.4551
3.3752
Cl
O
H
N
O
H
3f
0.98
0.99
0.95
1.00
1.35
3.00
f 1 (ppm)


187.7360
187.5814
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162.4317
141.9752
139.6325
135.2001
134.9051
134.7423
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132.9030
132.7854
130.3636
130.0092
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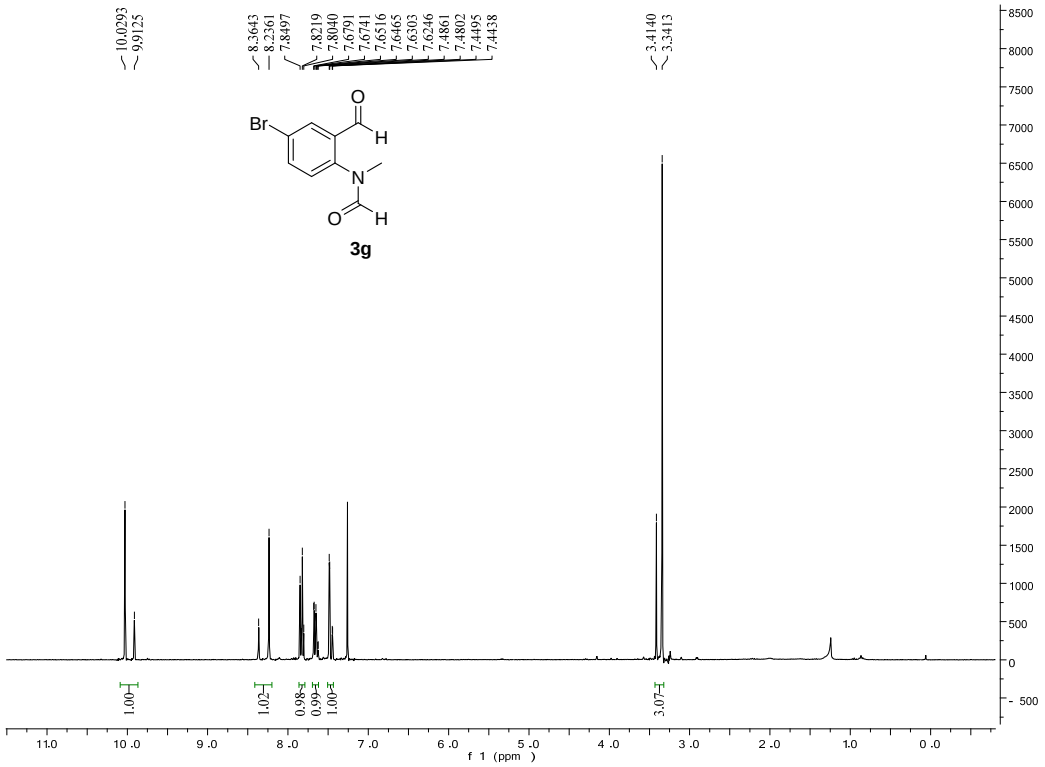
128.6142
37.6997
34.6681
Cl
O
H
N
O
H
3f
f 1 (ppm)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7.6741
7.6516
7.6465
7.6303
7.6246
7.4861
7.4802
7.4495
7.4438
3.4140
3.3413
Br
O
H
N
O
H
3g
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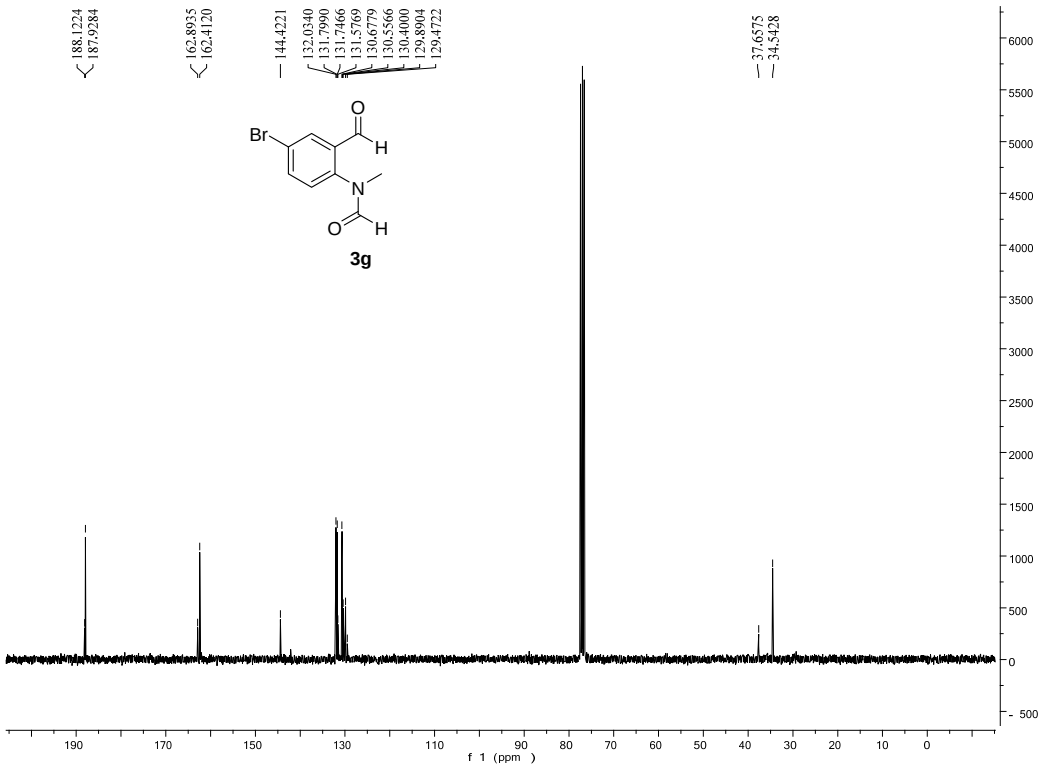
1.02
0.98
0.99
1.00
3.07
f 1 (ppm)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131.7990
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131.5769
130.6779
130.5566
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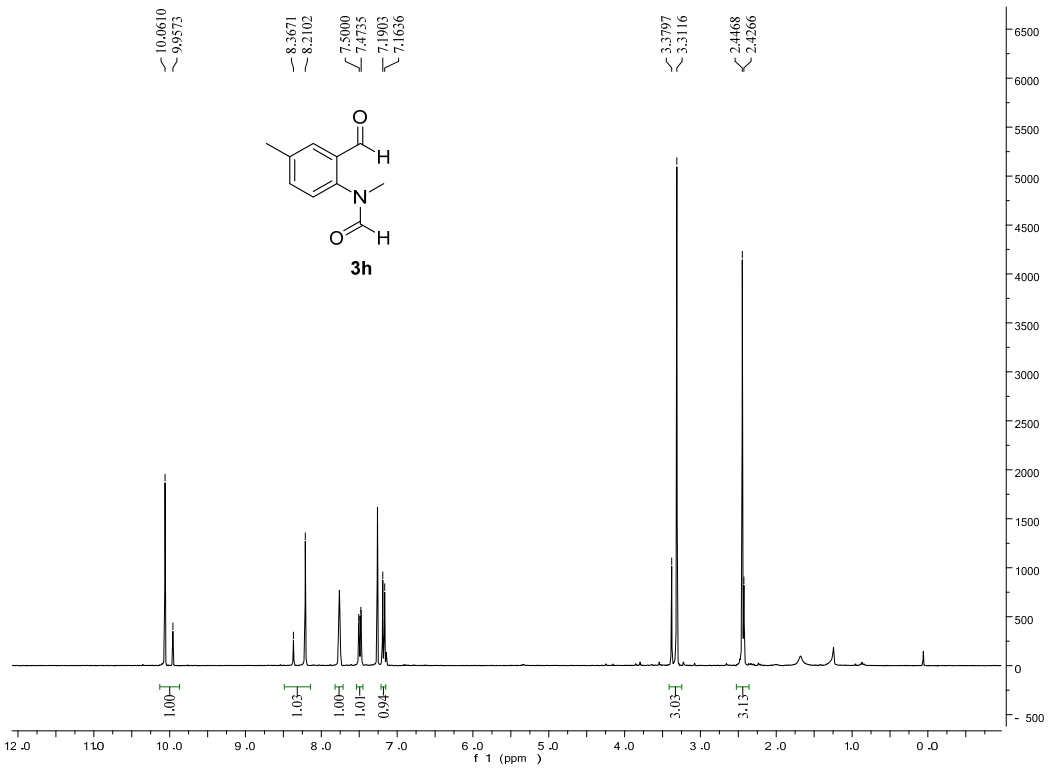
129.8904
129.4722
37.6575
34.5428
Br
O
H
N
O
H
3g
f 1 (ppm)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8.2102
7.5000
7.4735
7.1903
7.1636
3.3797
3.3116
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2.4266
3h
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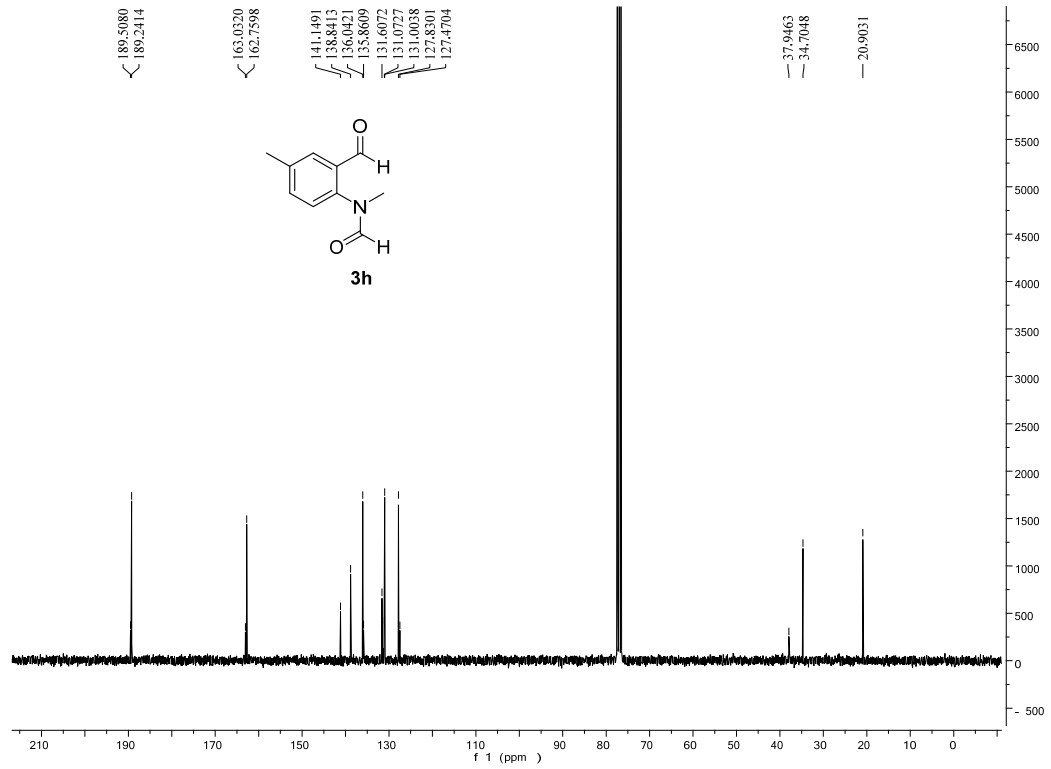
1.03
1.00
1.01
0.94
3.03
3.13
f 1 (ppm)


189.5080
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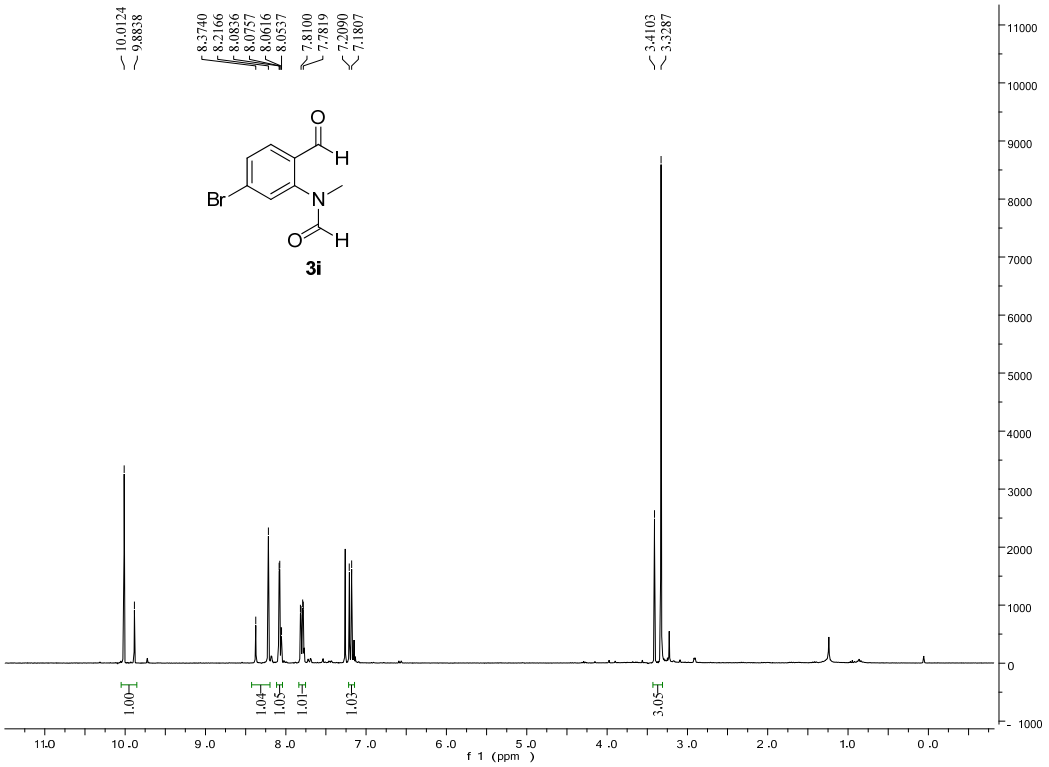
3h
f 1 (ppm)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8.0616
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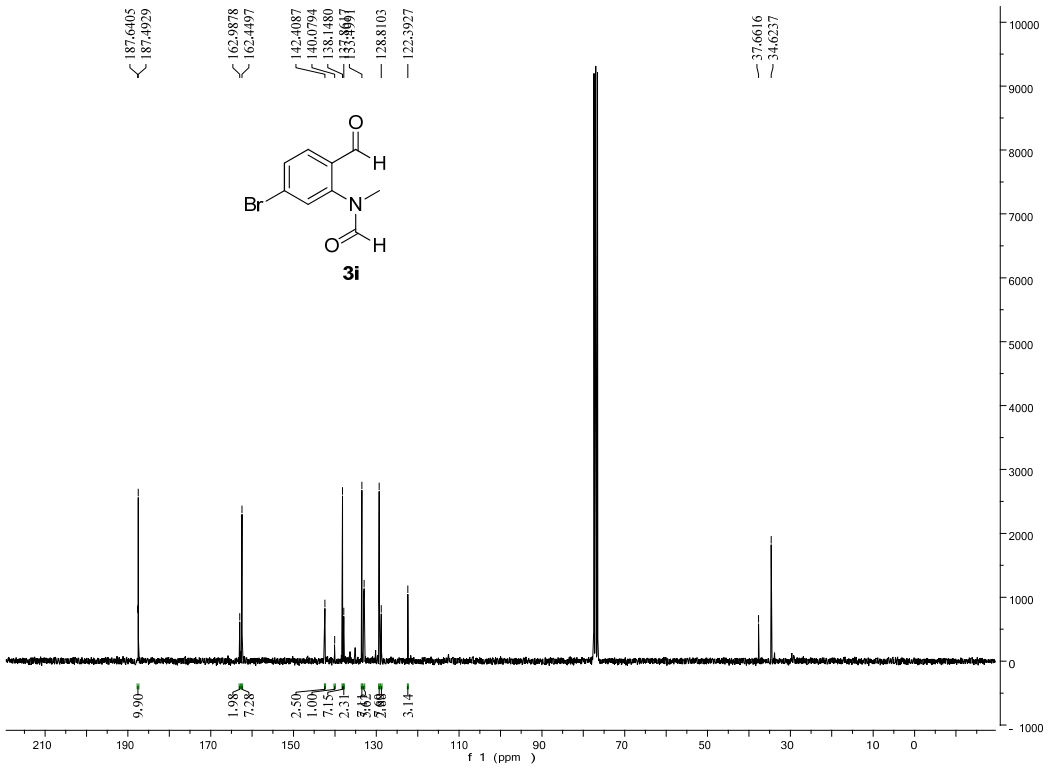
7.8100
7.7819
7.2090
7.1807
3.4103
3.3287
Br
O
H
N
O
H
3i
1.00
1.04
1.05
1.01
1.03
3.05
f 1 (ppm)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140.0794
138.1480
128.8103
122.3927
37.6616
34.6237
Br
O
H
N
O
H
3i
9.90
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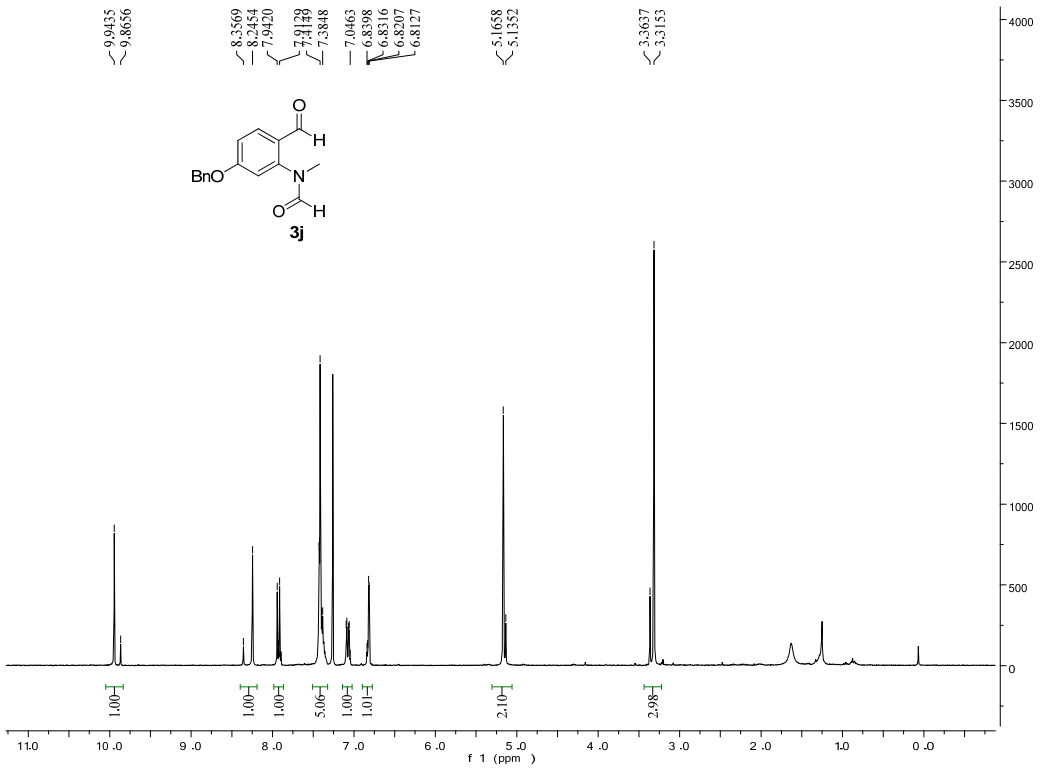
7.28
2.50
1.00
7.15
2.31
3.62
7.69
3.14
f 1 (ppm)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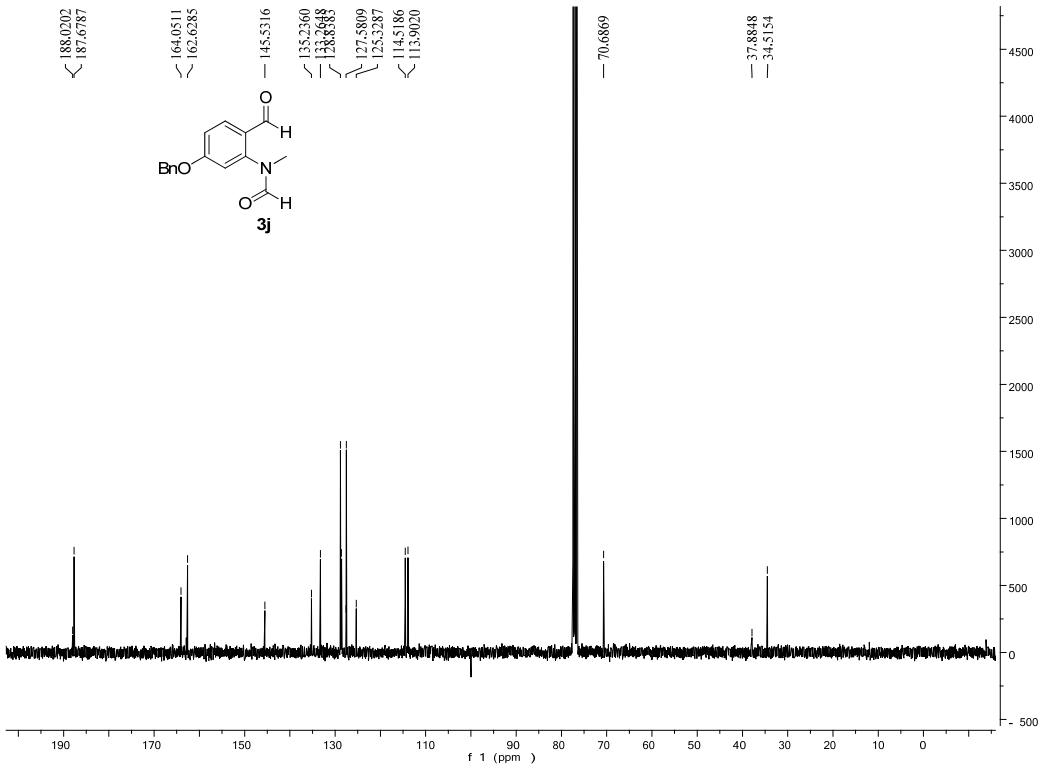
8.3569
8.2454
7.9420
7.9139
7.3848
7.0463
6.8398
6.8316
6.8207
6.8127
5.1658
5.1352
3.3637
3.3153
BnO
O
H
N
O
H
3j
1.00
1.00
1.00
5.06
1.00
1.01
2.10
2.98
f 1 (ppm)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133.7648
128.8583
127.5809
125.3287
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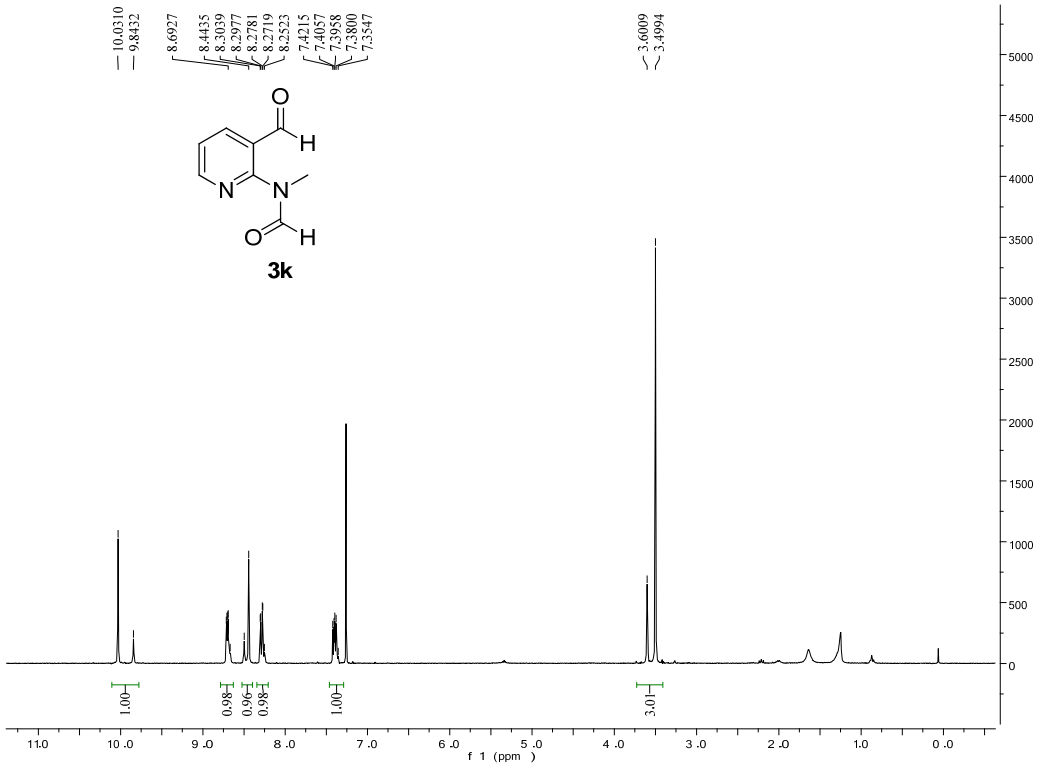
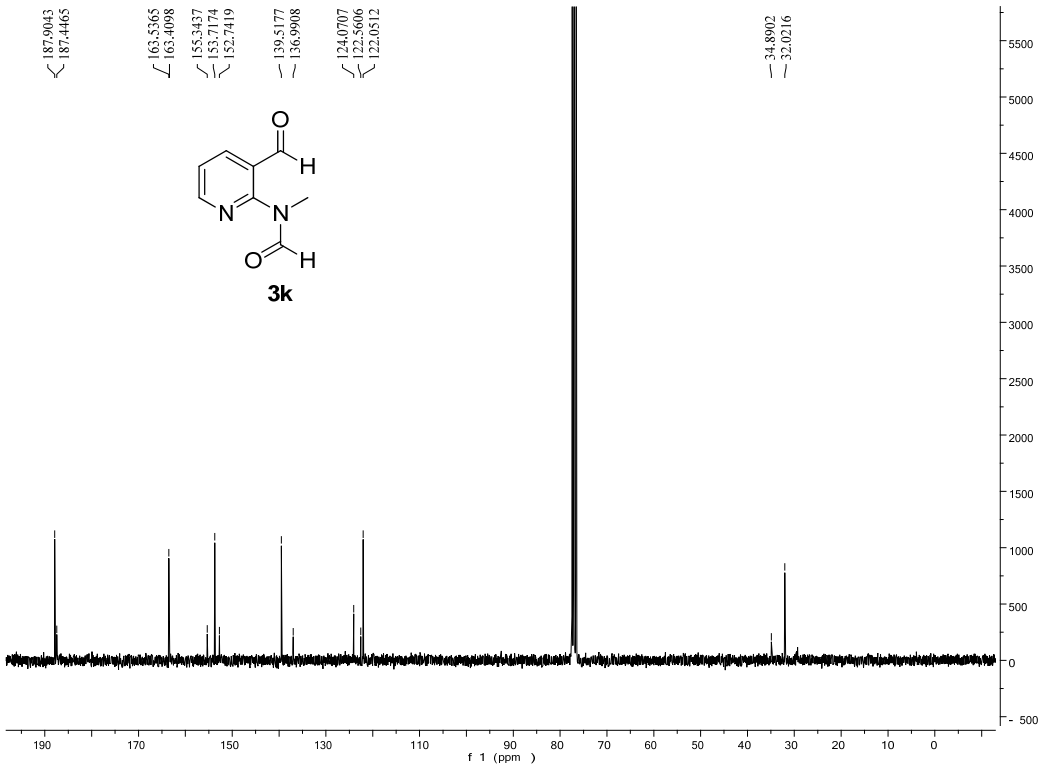
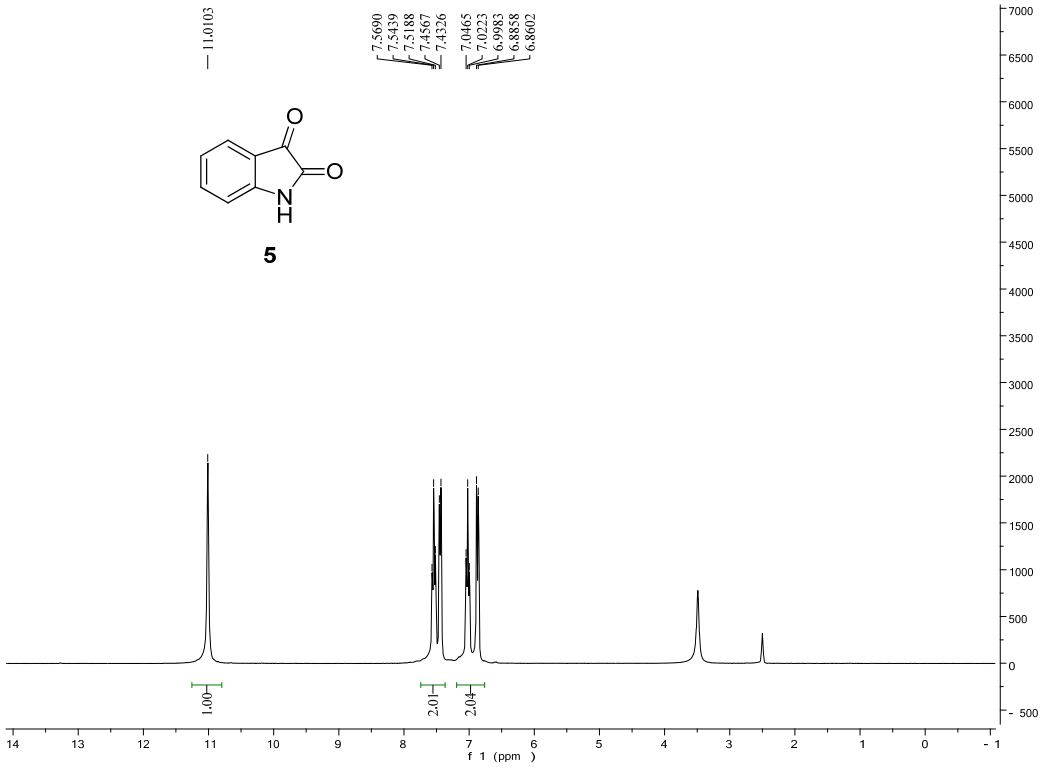
70.6869
37.8848
34.5154
BnO
O
H
N
O
H
3j
f 1 (ppm)


10.0310
9.8432
8.6927
8.4435
8.3039
8.2977
8.2781
8.2719
8.2523
7.4215
7.4057
7.3958
7.3800
7.3547
3.6009
3.4994
3k
1.00
0.98
0.96
0.98
1.00
3.01
f 1 (ppm)


187.9043
187.4465
163.5365
163.4098
155.3437
153.7174
152.7419
139.5177
136.9908
124.0707
122.5606
122.0512
34.8902
32.0216
3k
f 1 (ppm)


11.0103
7.5690
7.5439
7.5188
7.4567
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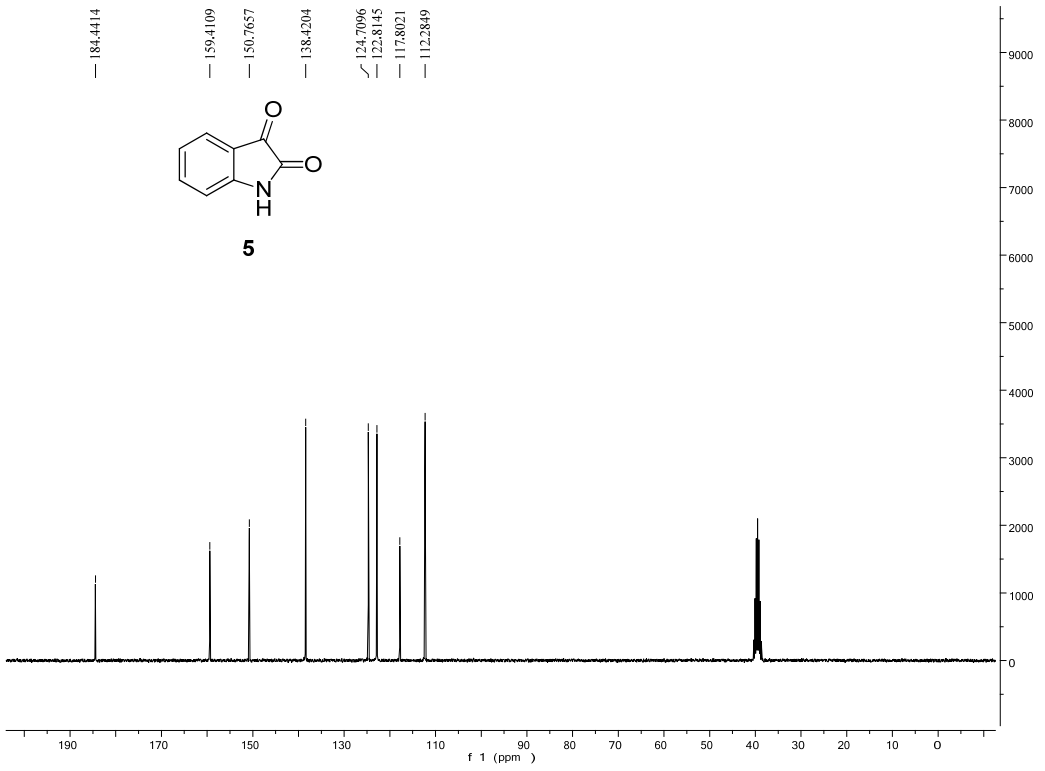
7.0465
7.0223
6.9983
6.8858
6.8602
1.00
2.01
2.04
f 1 (ppm)

5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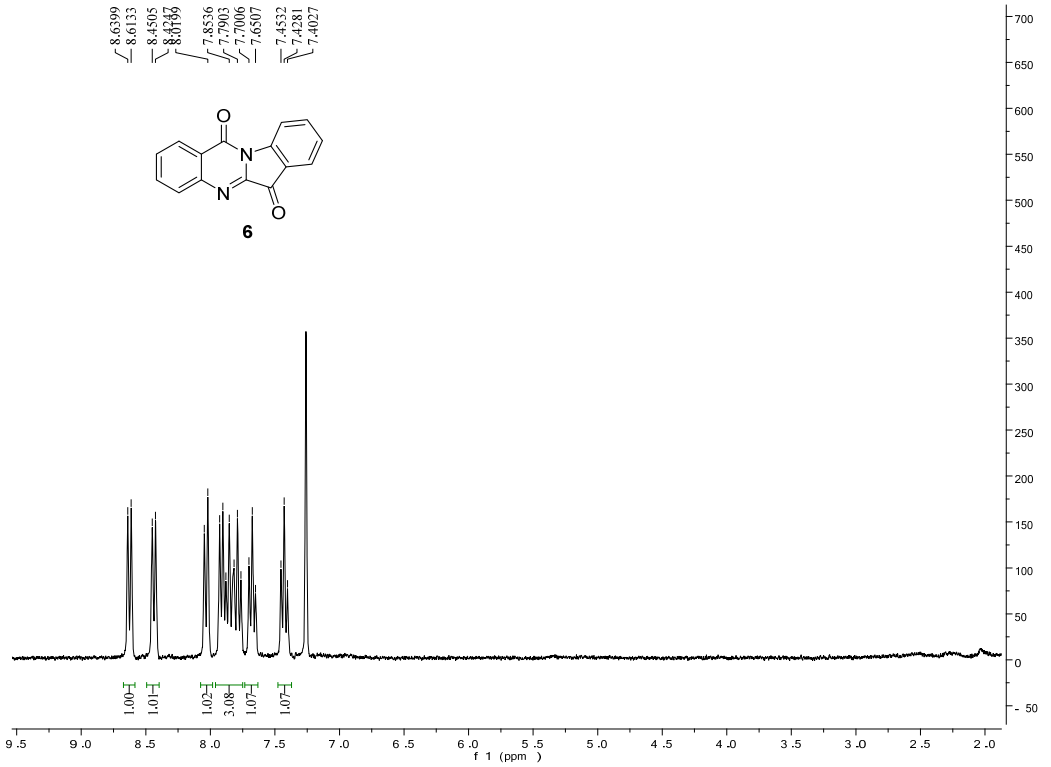
159.4109
150.7657
138.4204
124.7096
122.8145
117.8021
112.2849
f 1 (ppm)

5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8.6133
8.4505
8.4247
8.0196
7.8536
7.7903
7.7006
7.6507
7.4532
7.4281
7.4027
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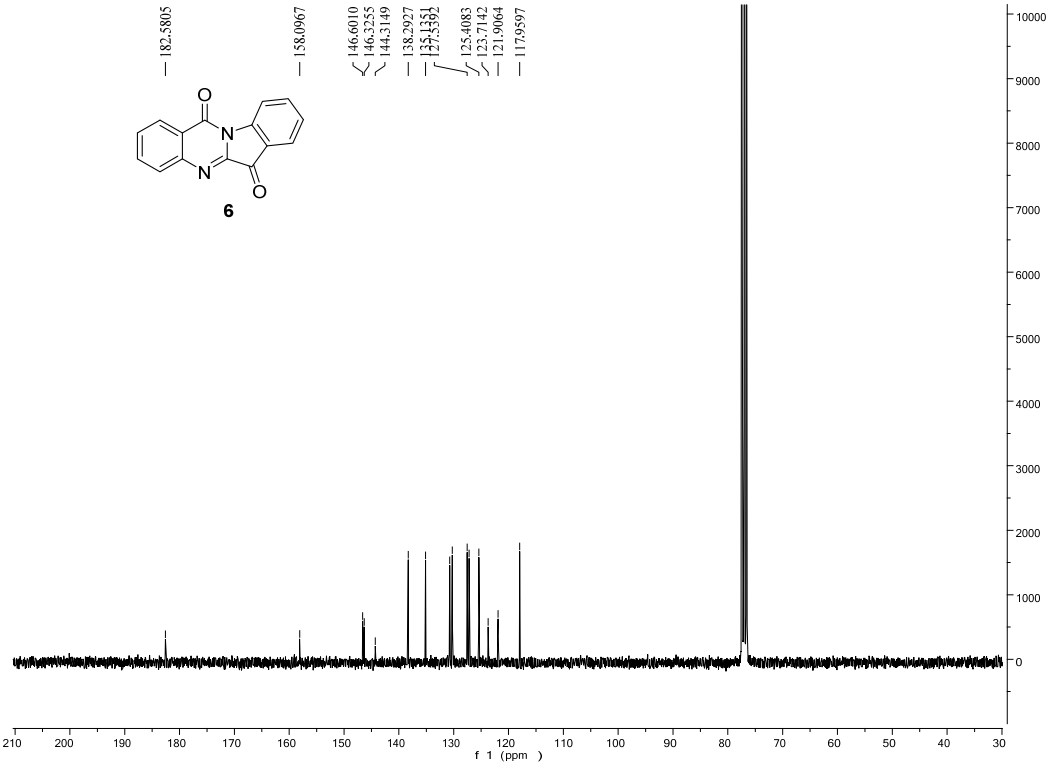
N
N
O
6
1.00
1.01
1.02
3.08
1.07
1.07
f 1 (ppm)


182.5805
158.0967
146.6010
146.3255
144.3149
138.2927
135.1331
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125.4083
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121.9064
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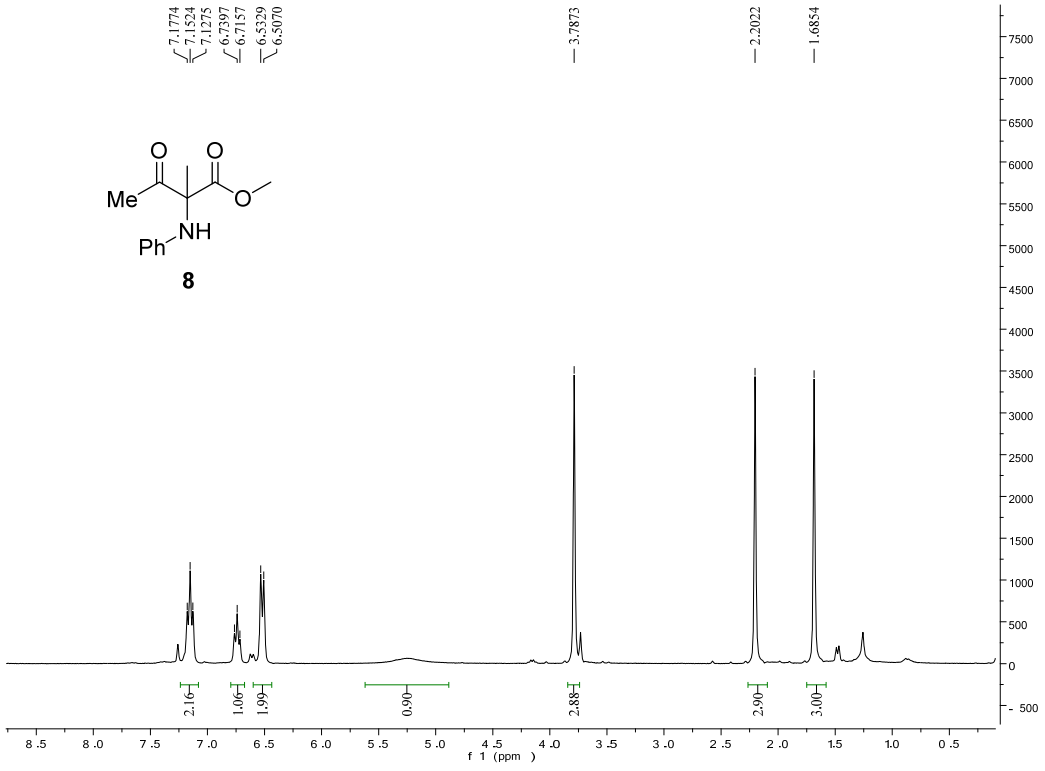
O
N
N
O
6
f 1 (ppm)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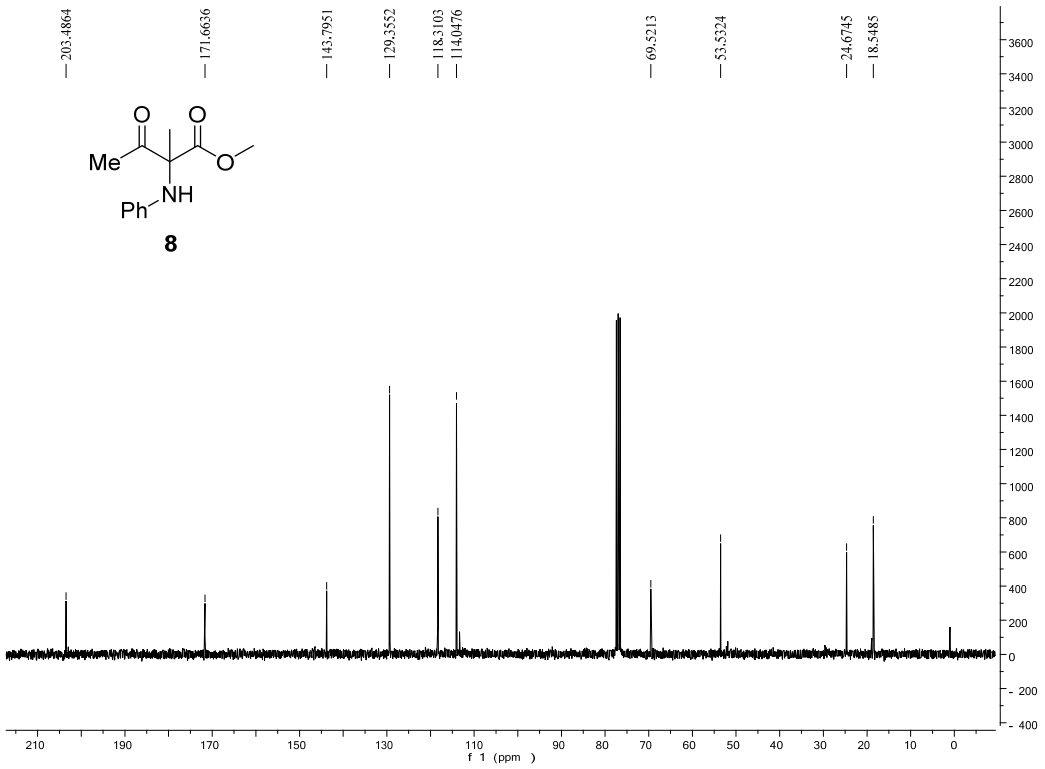
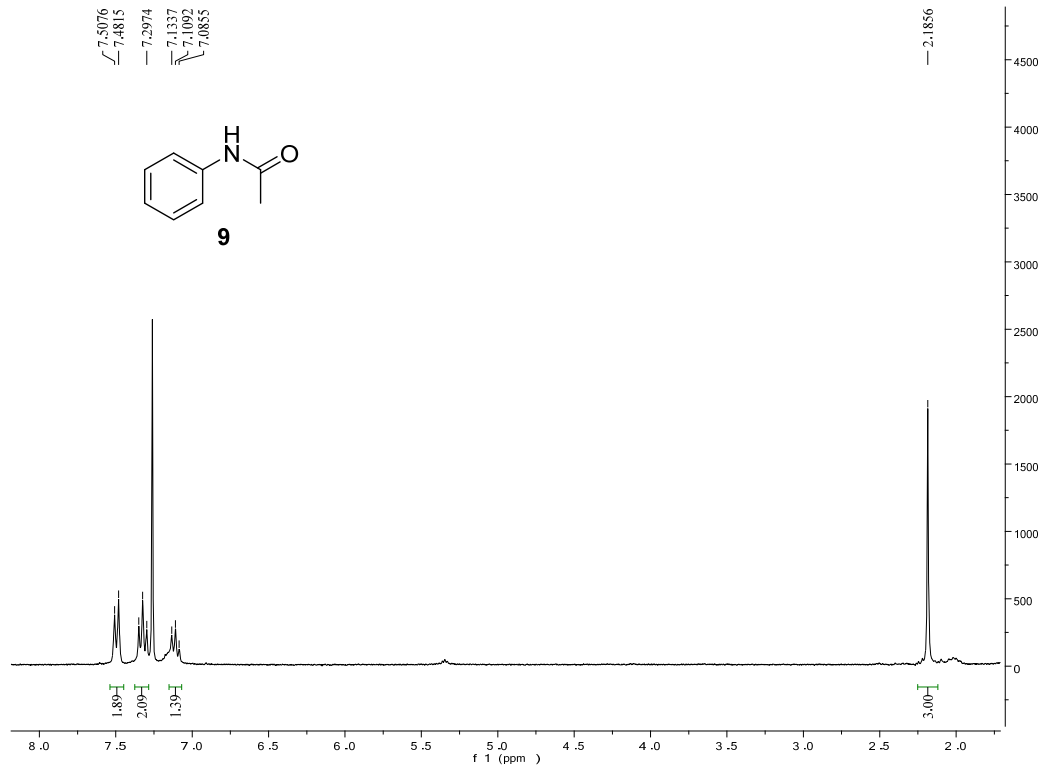
7.1524
7.1275
6.7397
6.7157
6.5329
6.5070
3.7873
2.2022
1.6854
Me
O
O
O
NH
Ph
8
2.16
1.06
1.99
0.90
2.88
2.90
3.00
f 1 (ppm)


203.4864
171.6636
143.7951
129.3552
118.3103
114.0476
69.5213
53.5324
24.6745
18.5485
Me
O
O
O
NH
Ph
8
f 1 (ppm)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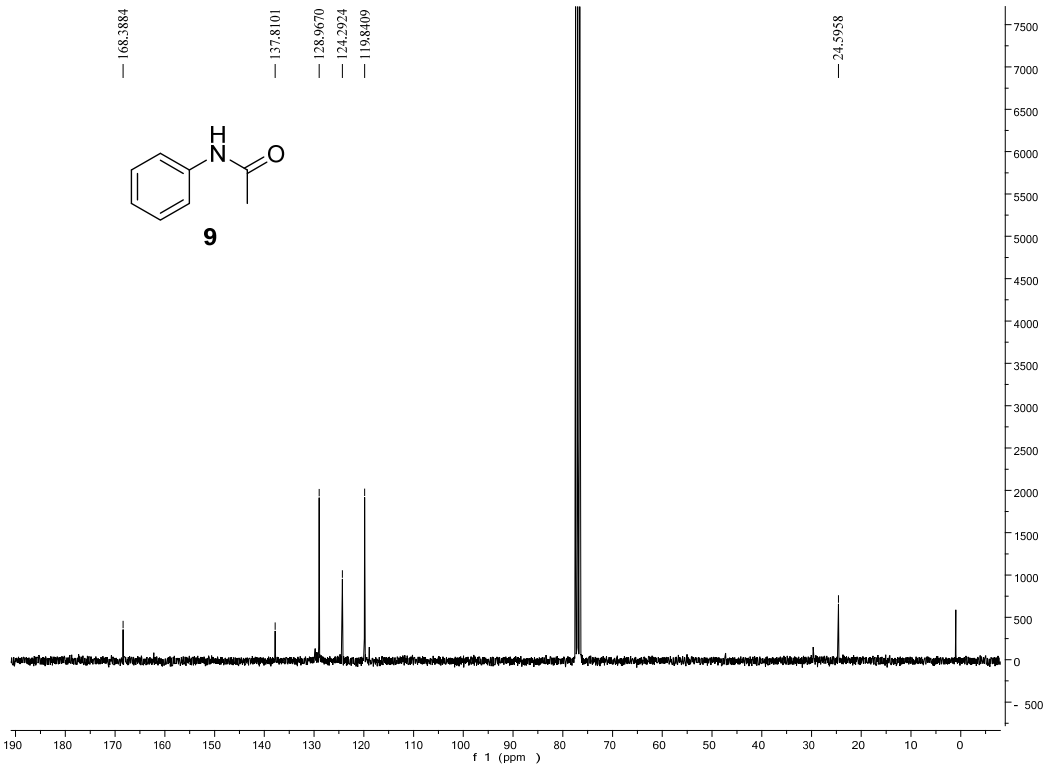
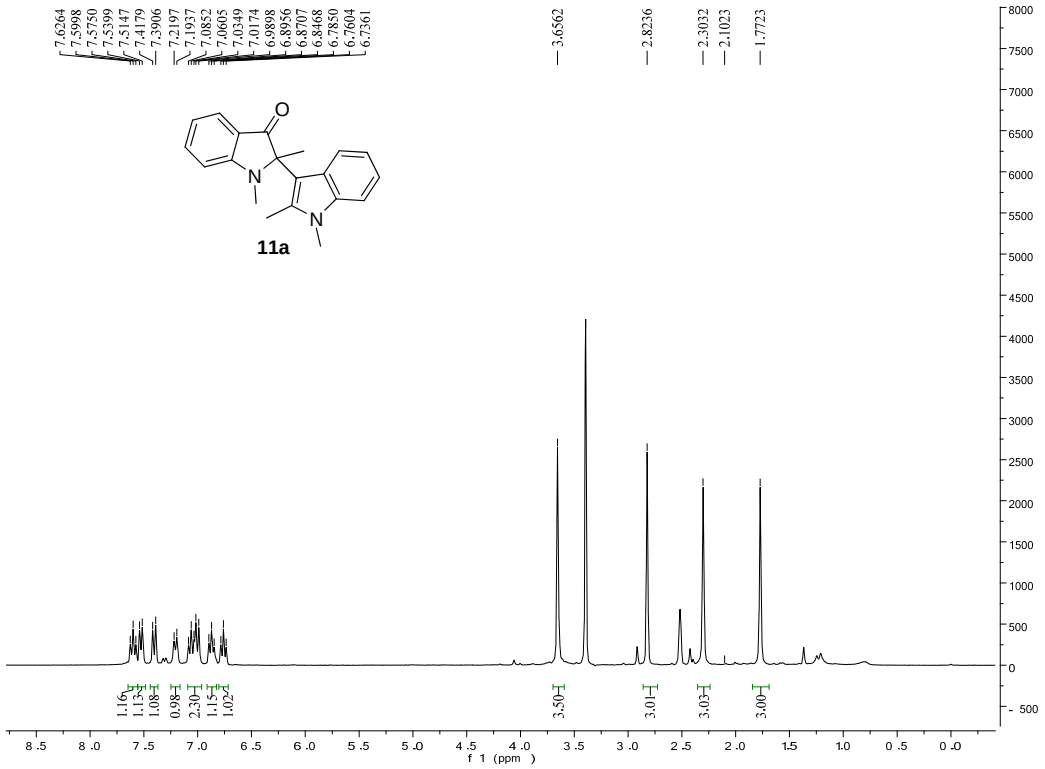
7.4815
7.2974
7.1337
7.1092
7.0855
2.1856
9
1.89
2.09
1.39
3.00
f 1 (ppm)


168.3884
137.8101
128.9670
124.2924
119.8409
24.5958
9
f 1 (ppm)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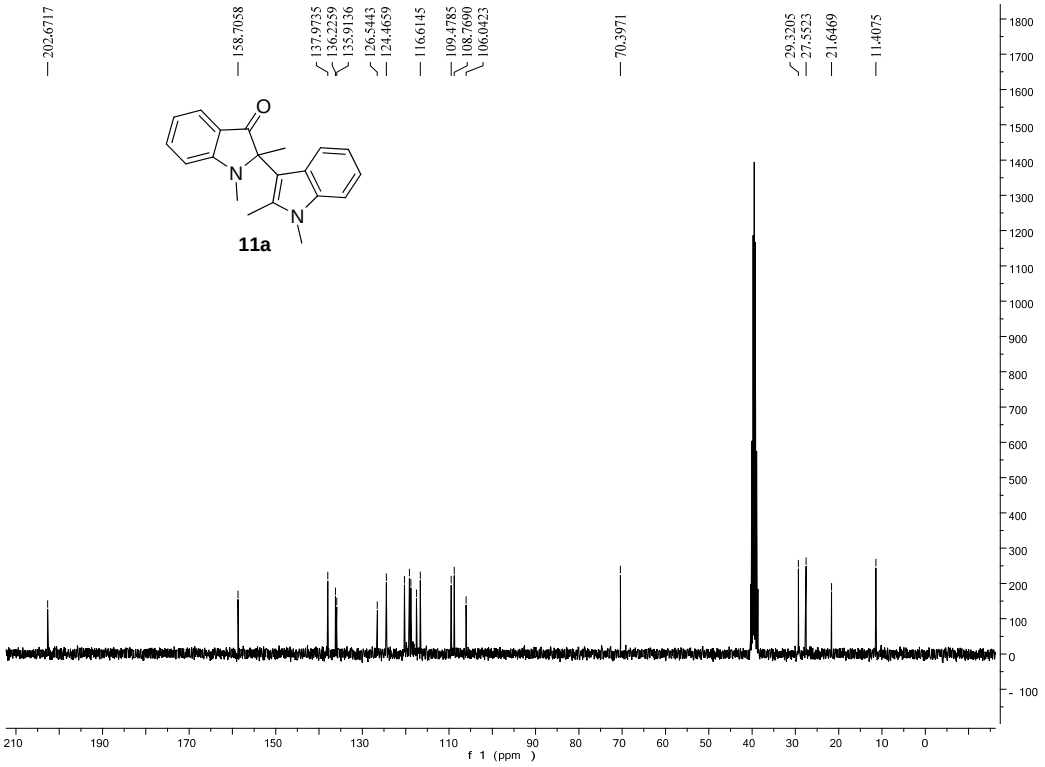
7.5998
7.5750
7.5399
7.5147
7.4179
7.3906
7.2197
7.1937
7.0852
7.0605
7.0349
7.0174
6.9898
6.8956
6.8707
6.8468
6.7850
6.7604
6.7361
3.6562
2.8236
2.3032
2.1023
1.7723
O
N
N
11a
1.16
1.13
1.08
0.98
2.30
1.15
1.02
3.50
3.01
3.03
3.00
f 1 (ppm)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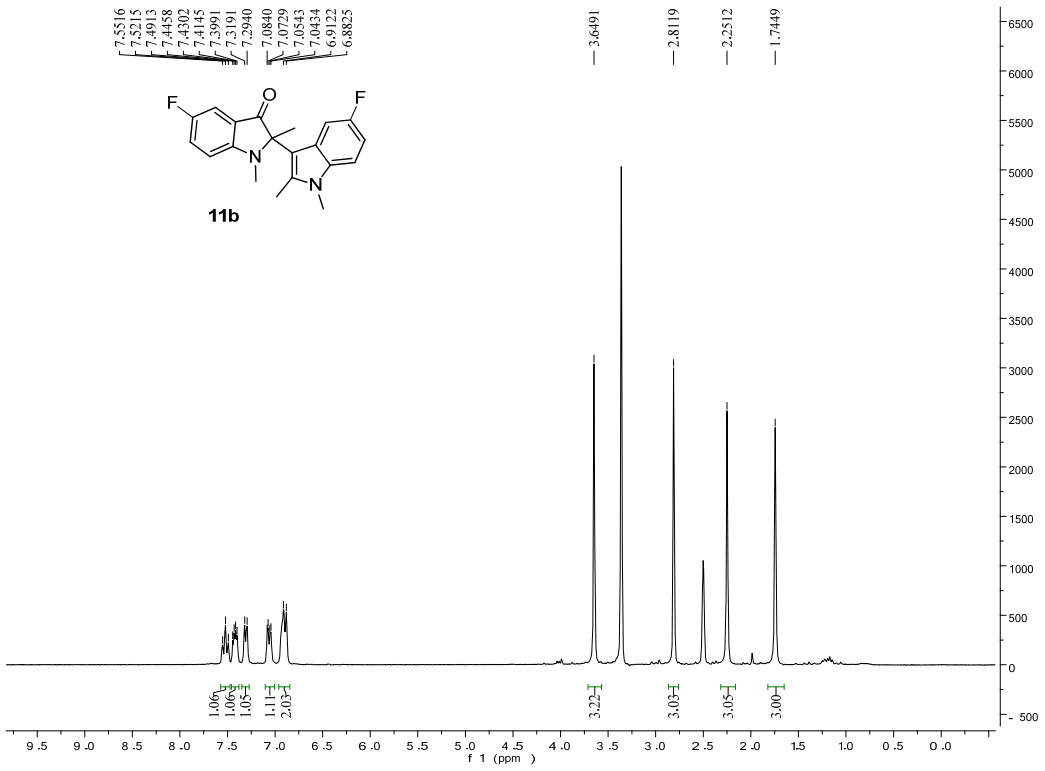
137.9735
136.2259
135.9136
126.5443
124.4659
116.6145
109.4785
108.7690
106.0423
70.3971
29.3205
27.5523
21.6469
11.4075
O
N
N
11a
f 1 (ppm)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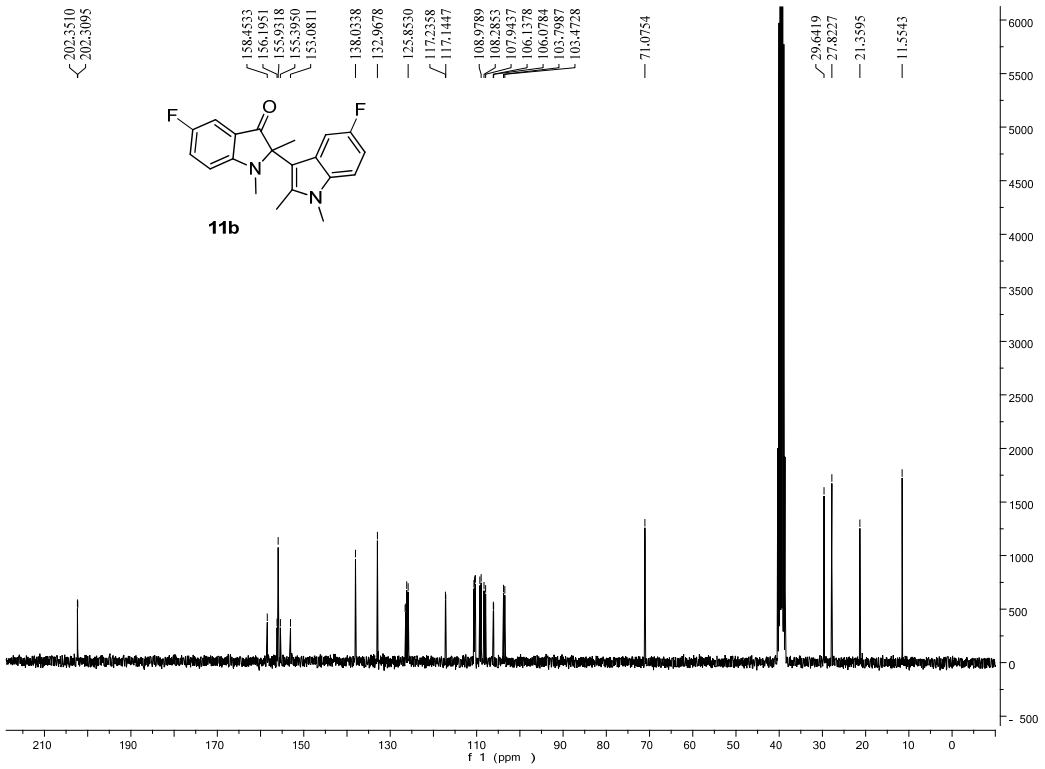
7.5215
7.4913
7.4458
7.4302
7.4145
7.3991
7.3191
7.2940
7.0840
7.0729
7.0543
7.0434
6.9122
6.8825
3.6491
2.8119
2.2512
1.7449
F
O
N
N
F
11b
1.06
1.06
1.05
1.11
2.03
3.22
3.03
3.05
3.00
f 1 (ppm)


202.3510
202.3095
158.4533
156.1951
155.9318
155.3950
153.0811
138.0338
132.9678
125.8530
117.2358
117.1447
108.9789
108.2853
107.9437
106.1378
106.0784
103.7987
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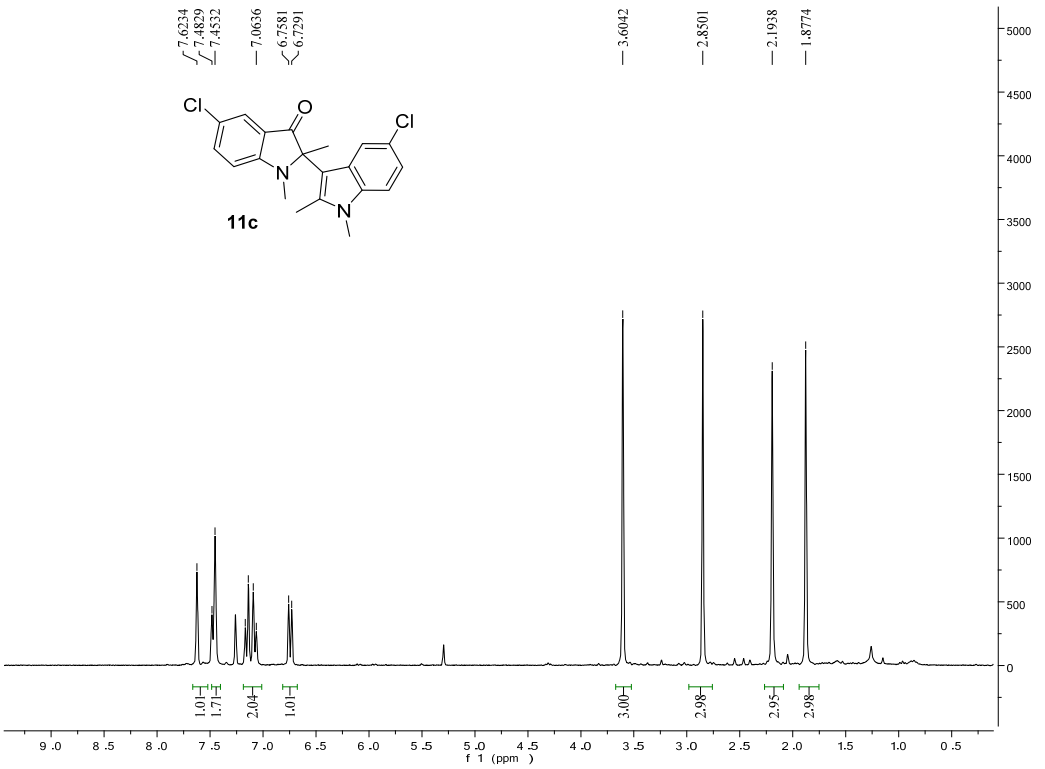
29.6419
27.8227
21.3595
11.5543
F
O
N
N
F
11b
f 1 (ppm)


7.6234
7.4829
7.4532
7.0636
6.7581
6.7291
3.6042
2.8501
2.1938
1.8774
Cl
O
Cl
N
N
11c
1.01
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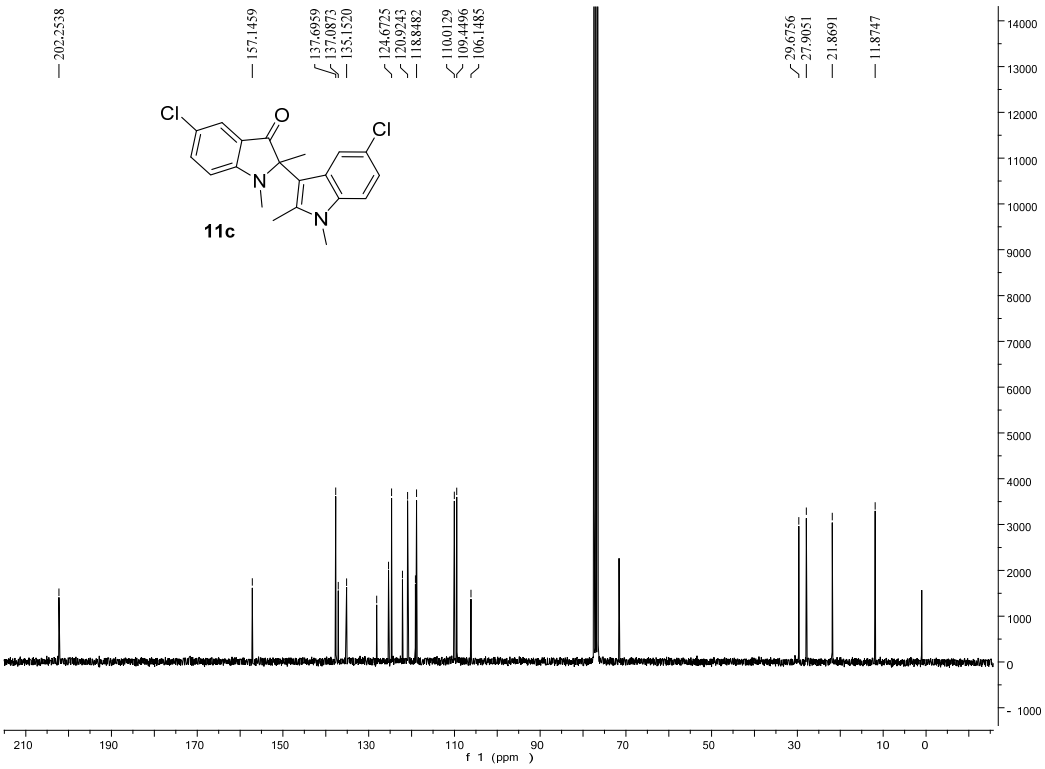
1.01
3.00
2.98
2.95
2.98
f 1 (ppm)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137.6959
137.0873
135.1520
124.6725
120.9243
118.8482
110.0129
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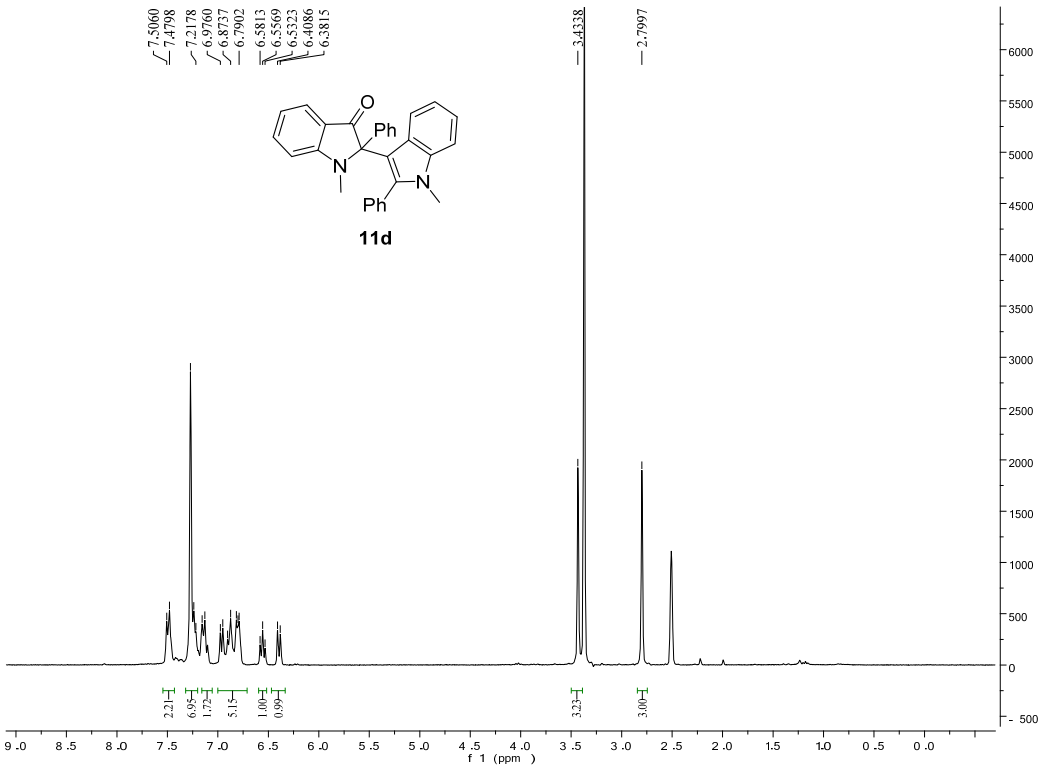
29.6756
27.9051
21.8691
11.8747
Cl
O
Cl
N
N
11c
f 1 (ppm)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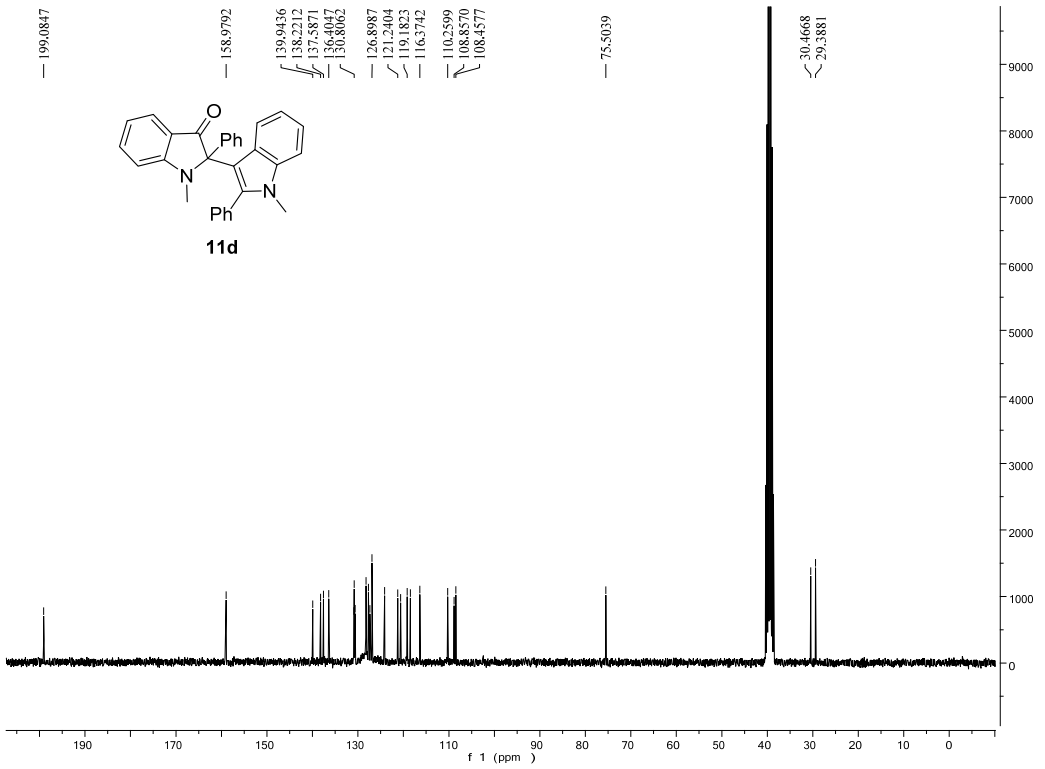
7.4798
7.2178
6.9760
6.8737
6.7902
6.5813
6.5569
6.5323
6.4086
6.3815
3.4338
2.7997
O
Ph
N
N
Ph
11d
2.21
6.95
1.72
5.15
1.00
0.99
3.23
3.00
f 1 (ppm)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139.9436
138.2212
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136.4047
130.8062
126.8987
121.2404
119.1823
116.3742
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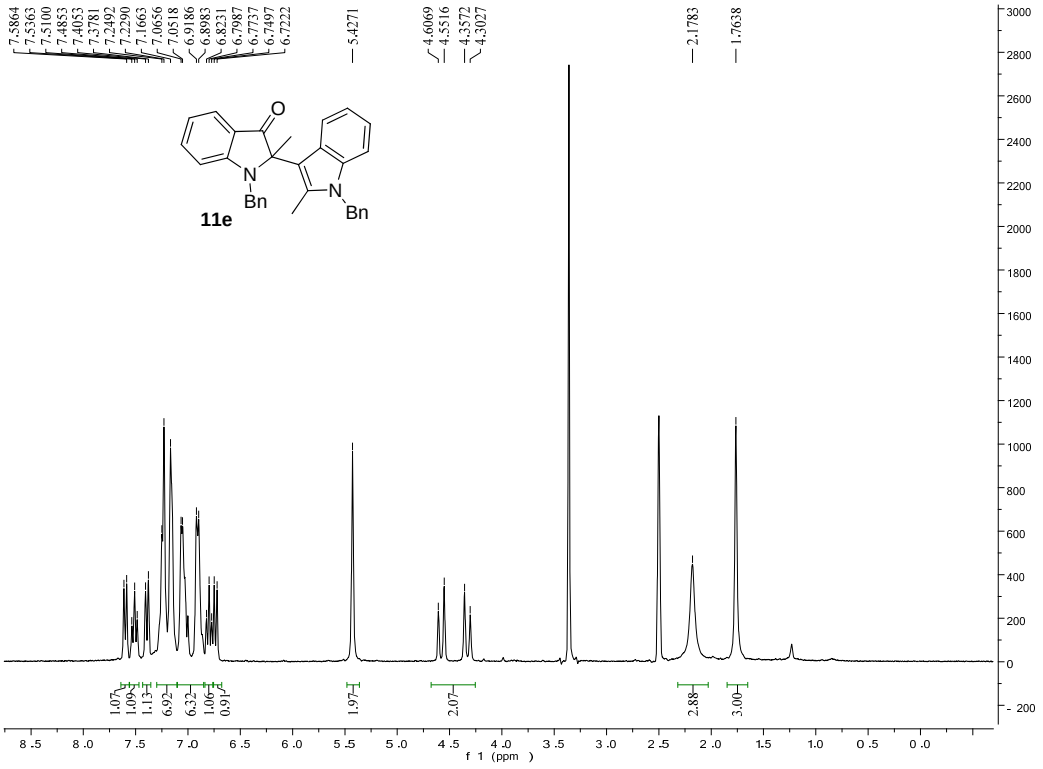
108.8570
108.4577
75.5039
30.4668
29.3881
O
Ph
N
N
Ph
11d
f 1 (ppm)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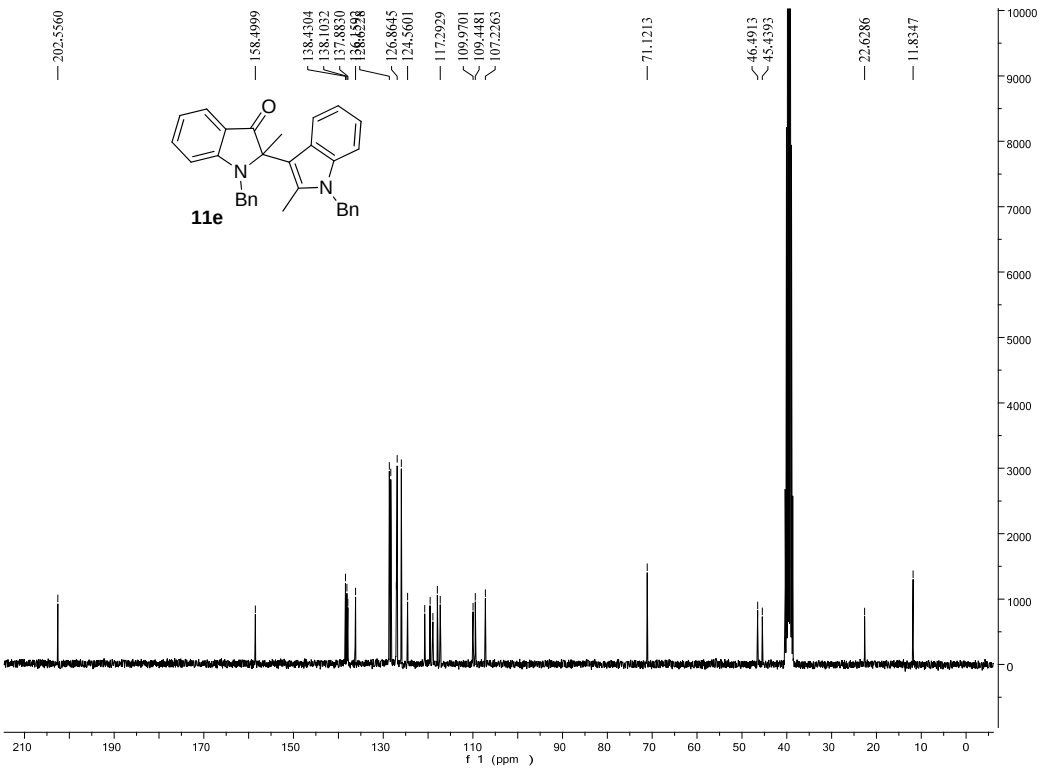
7.5363
7.5100
7.4853
7.4053
7.3781
7.2492
7.2290
7.1663
7.0656
7.0518
6.9186
6.8983
6.8231
6.7987
6.7737
6.7497
6.7222
5.4271
4.6069
4.5516
4.3572
4.3027
2.1783
1.7638
O
N
Bn
N
Bn
11e
1.07
1.09
1.13
6.92
6.32
1.06
0.91
1.97
2.07
2.88
3.00
f 1 (ppm)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126.8645
124.5601
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109.9701
109.4481
107.2263
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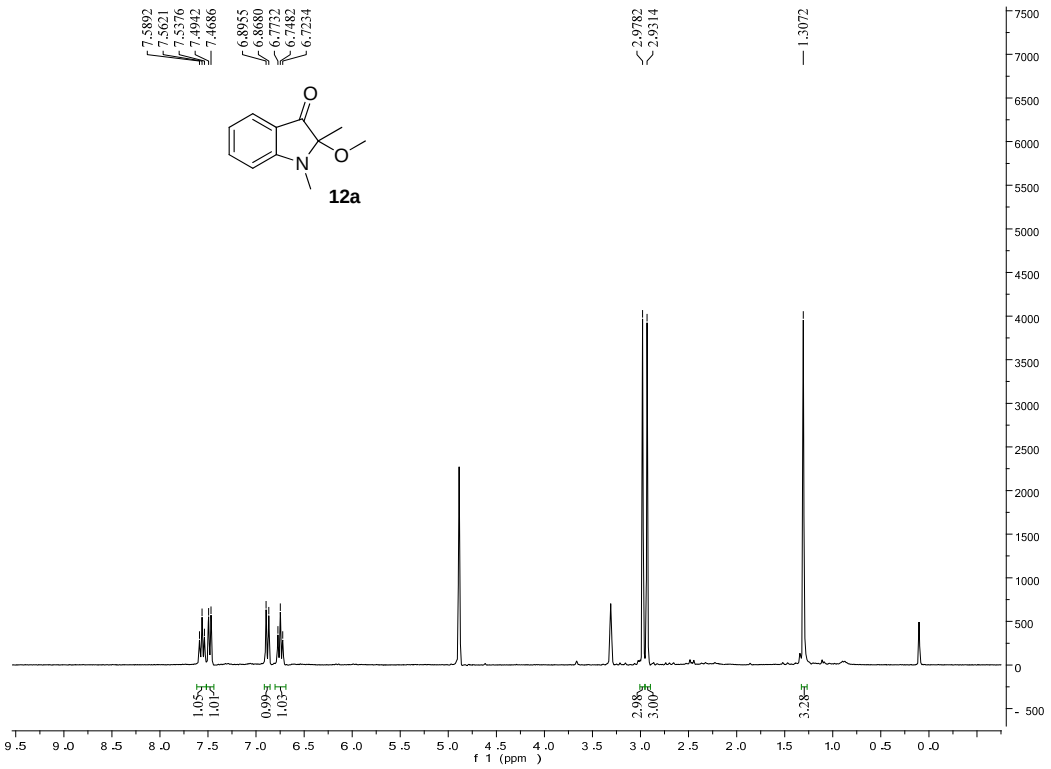
46.4913
45.4393
22.6286
11.8347
O
N
Bn
N
Bn
11e
f 1 (ppm)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7.5376
7.4942
7.4686
6.8955
6.8680
6.7732
6.7482
6.7234
2.9782
2.9314
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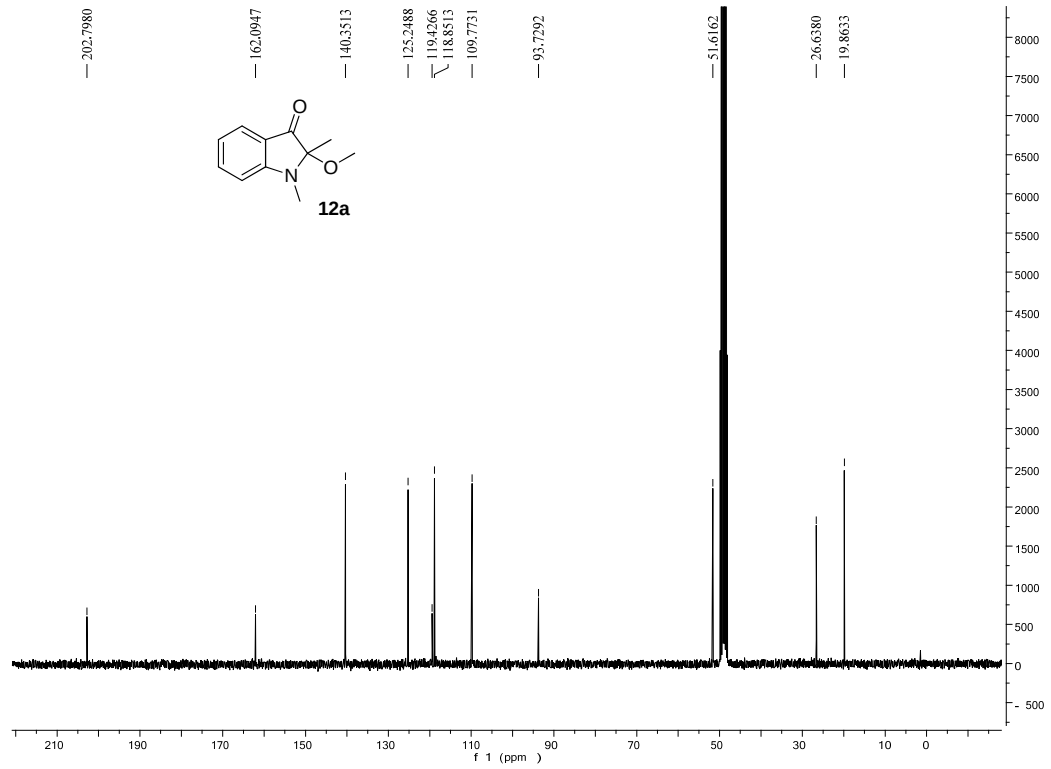
12a
1.05
1.01
0.99
1.03
2.98
3.00
3.28
f 1 (ppm)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140.3513
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109.7731
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51.6162
26.6380
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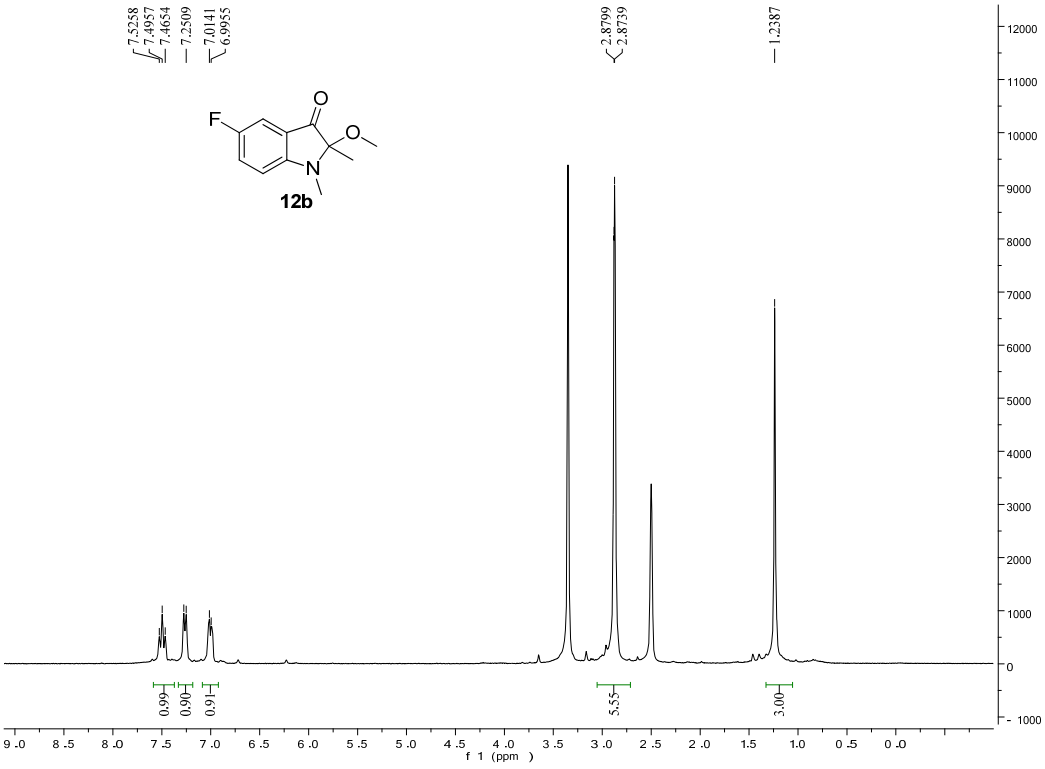
12a
f 1 (ppm)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7.4957
7.4654
7.2509
7.0141
6.9955
2.8799
2.8739
1.2387
F
O
O
N
12b
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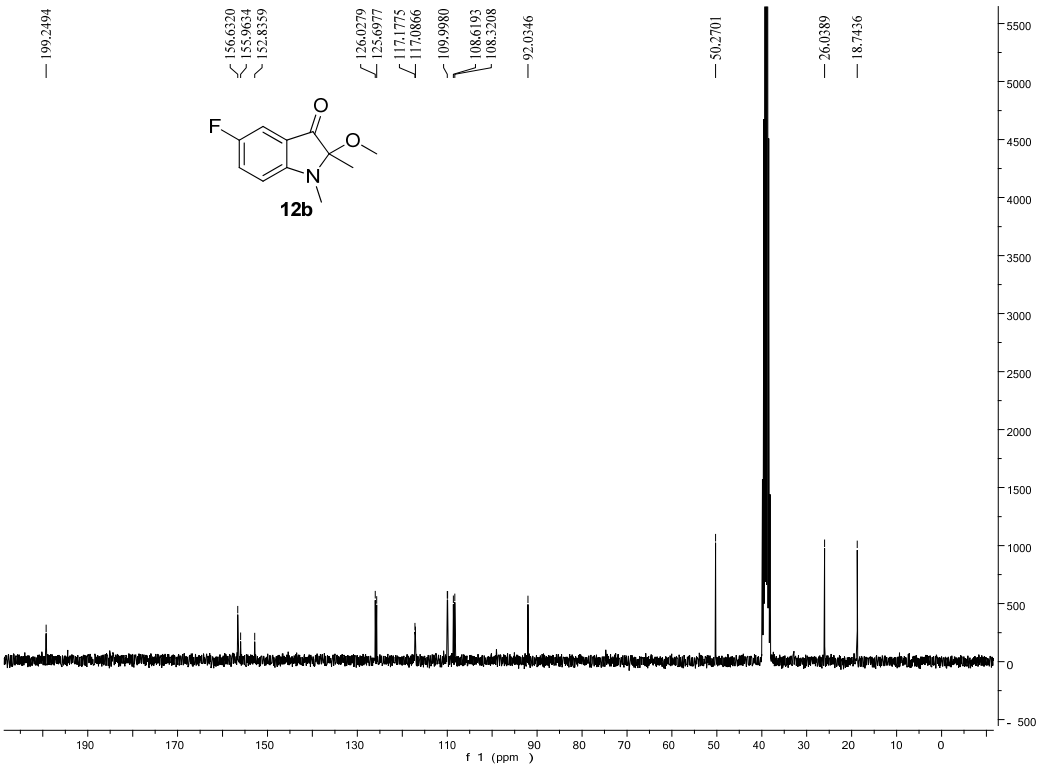
0.90
0.91
5.55
3.00
f 1 (ppm)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152.8359
126.0279
125.6977
117.1775
117.0866
109.9980
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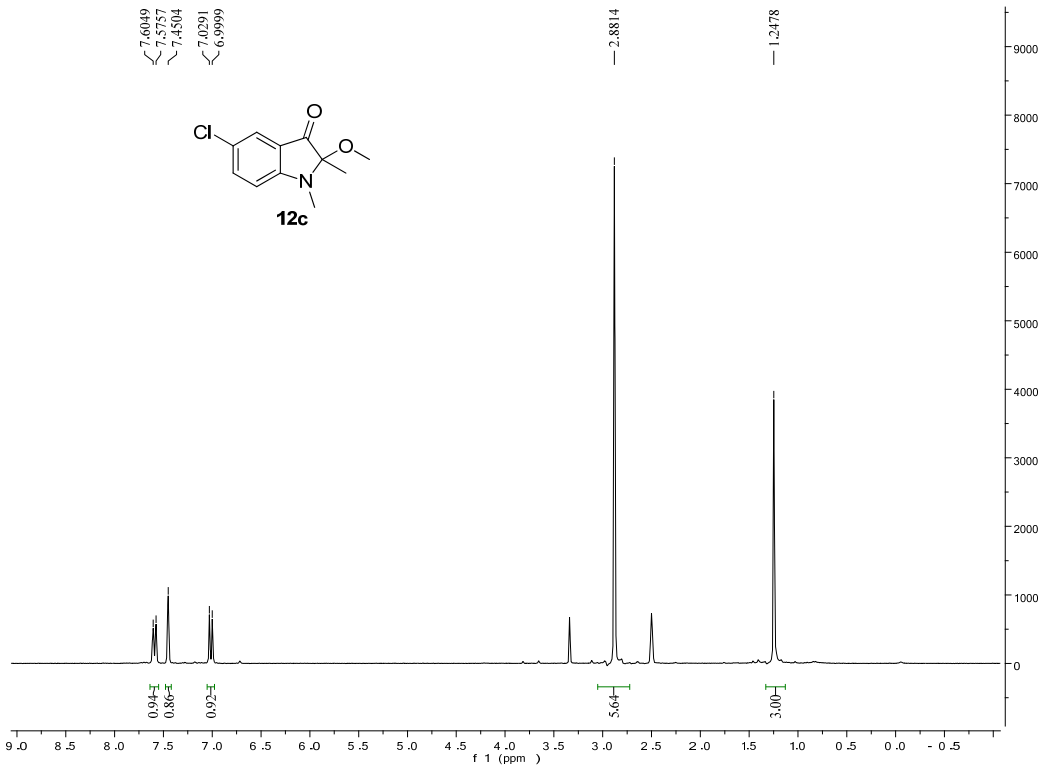
92.0346
50.2701
26.0389
18.7436
F
O
O
N
12b
f 1 (ppm)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7.4504
7.0291
6.9999
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N
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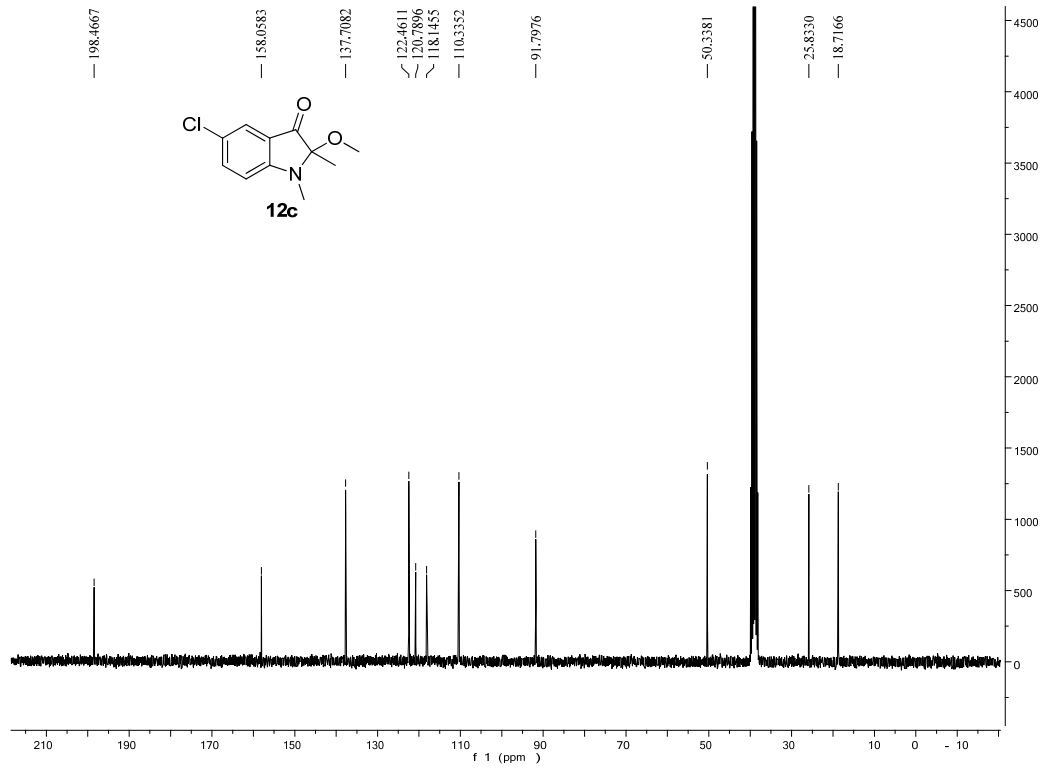
3.00
f 1 (ppm)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120.7896
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25.8330
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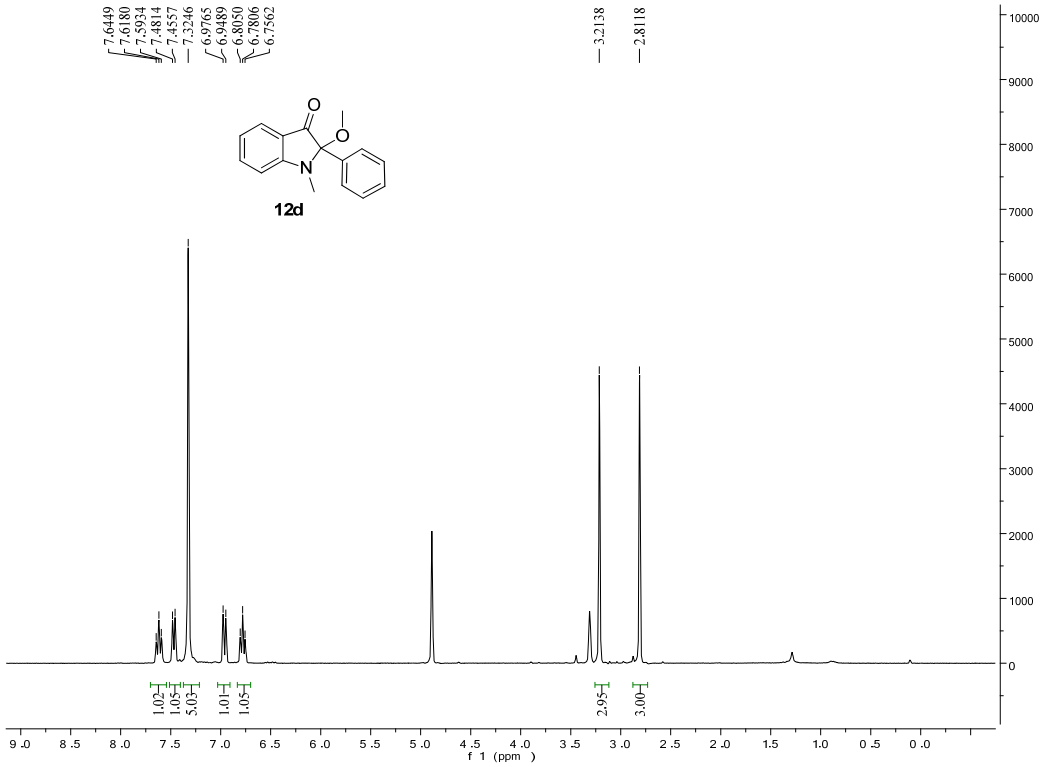
Cl
O
O
N
12c
f 1 (ppm)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7.4814
7.4557
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6.9765
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6.8050
6.7806
6.7562
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12d
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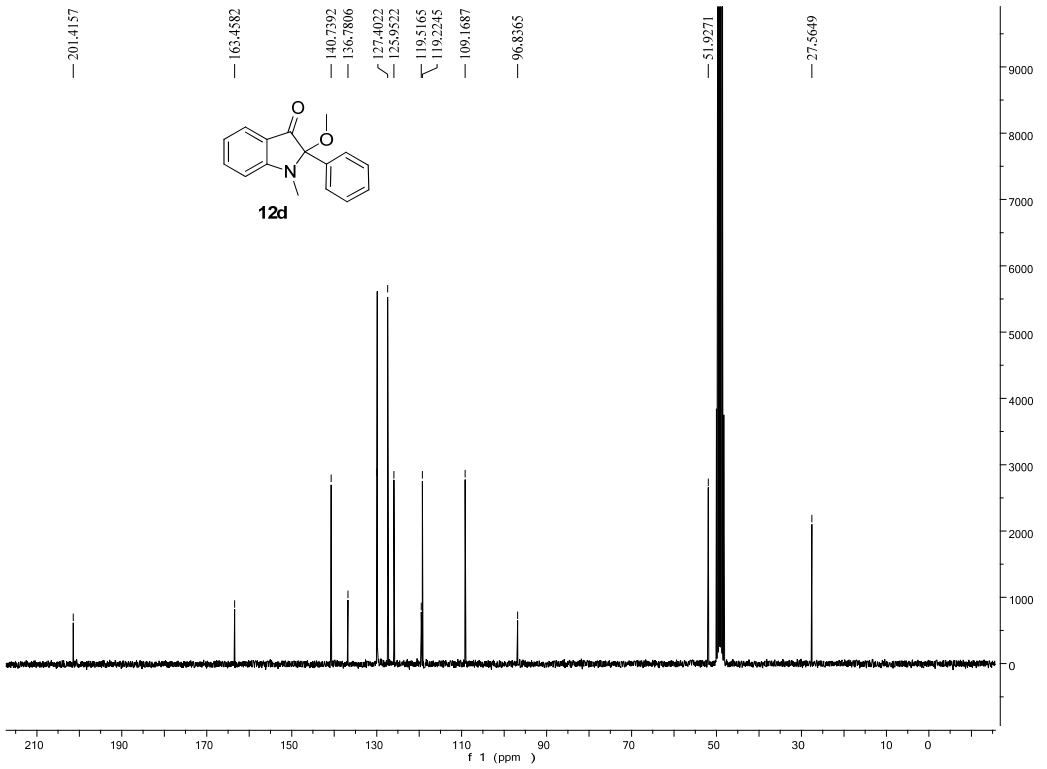
5.03
1.01
1.05
2.95
3.00
f 1 (ppm)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136.7806
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119.5165
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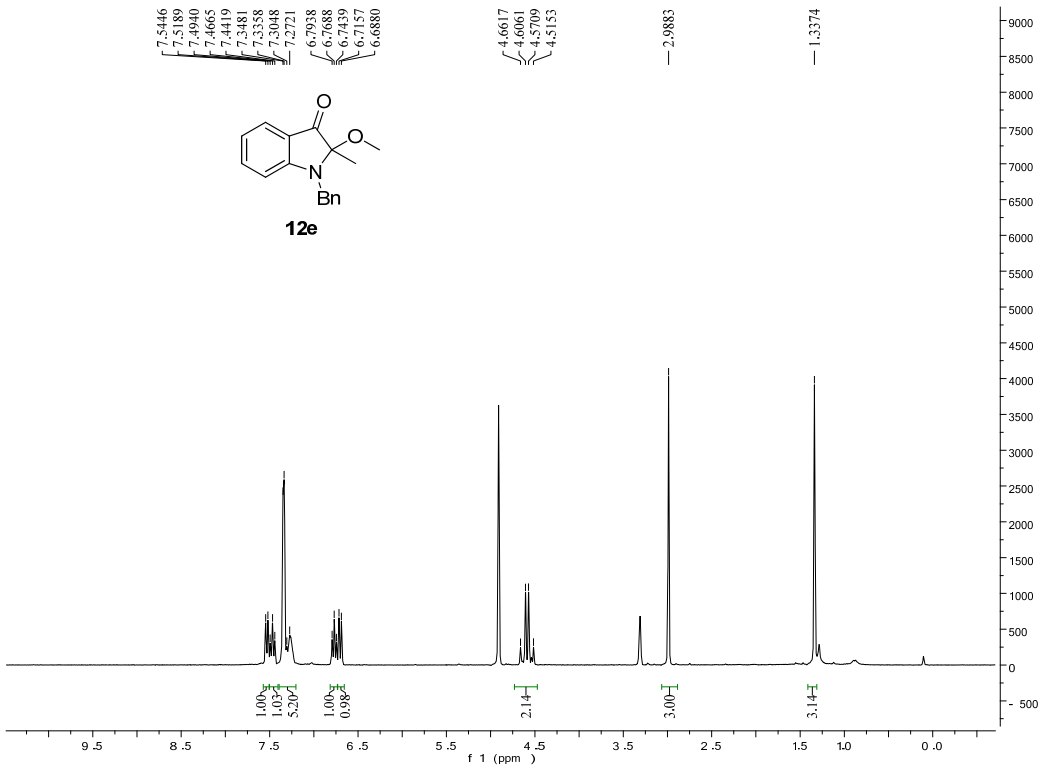
12d
f 1 (ppm)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7.5189
7.4940
7.4665
7.4419
7.3481
7.3358
7.3048
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6.7688
6.7439
6.7157
6.6880
4.6617
4.6061
4.5709
4.5153
2.9883
1.3374
O
O
N
Bn
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1.03
5.20
1.00
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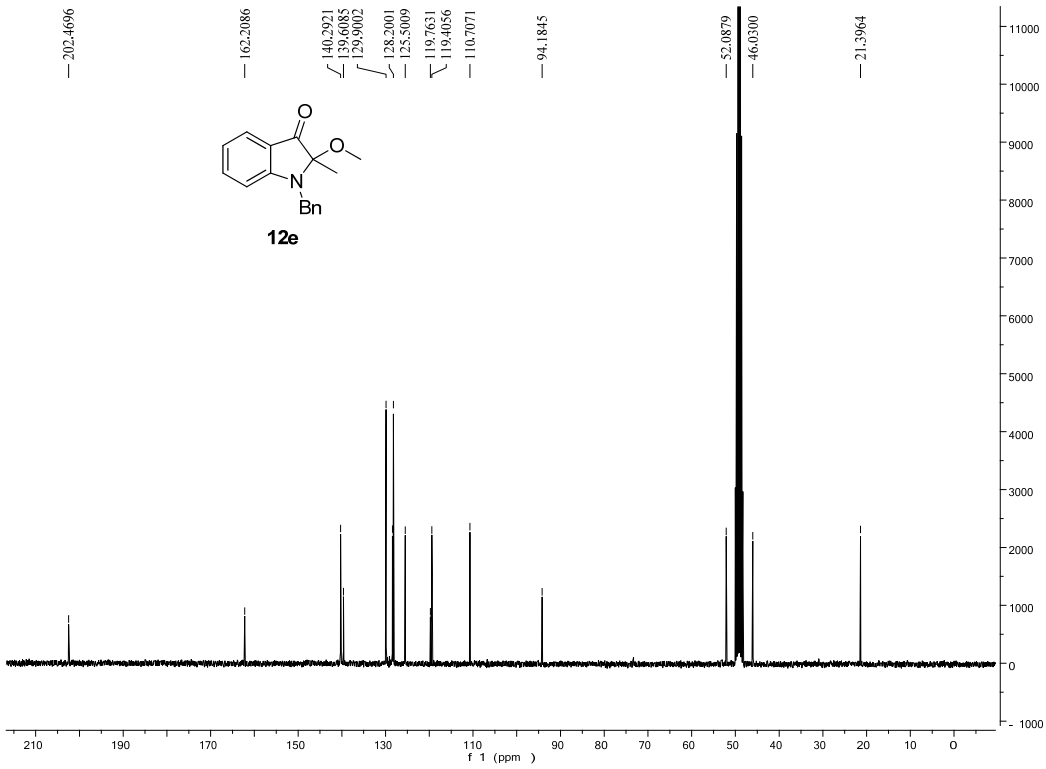
3.00
3.14
f 1 (ppm)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46.0300
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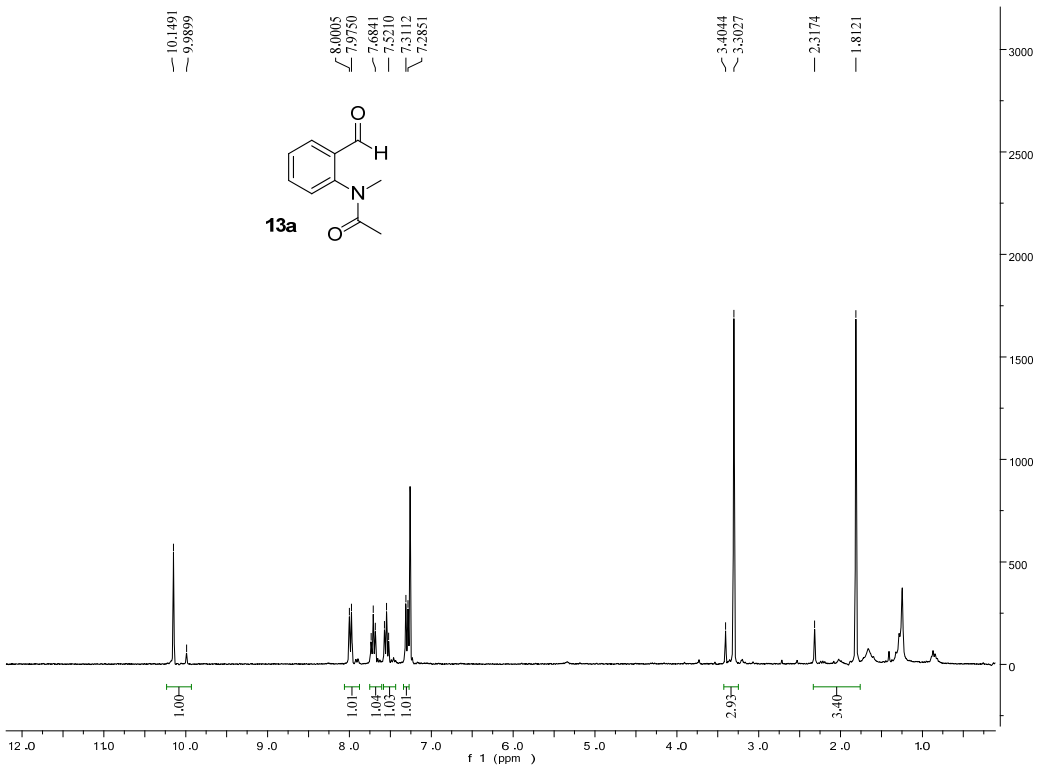
O
O
N
Bn
12e
f 1 (ppm)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7.5210
7.3112
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3.3027
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13a
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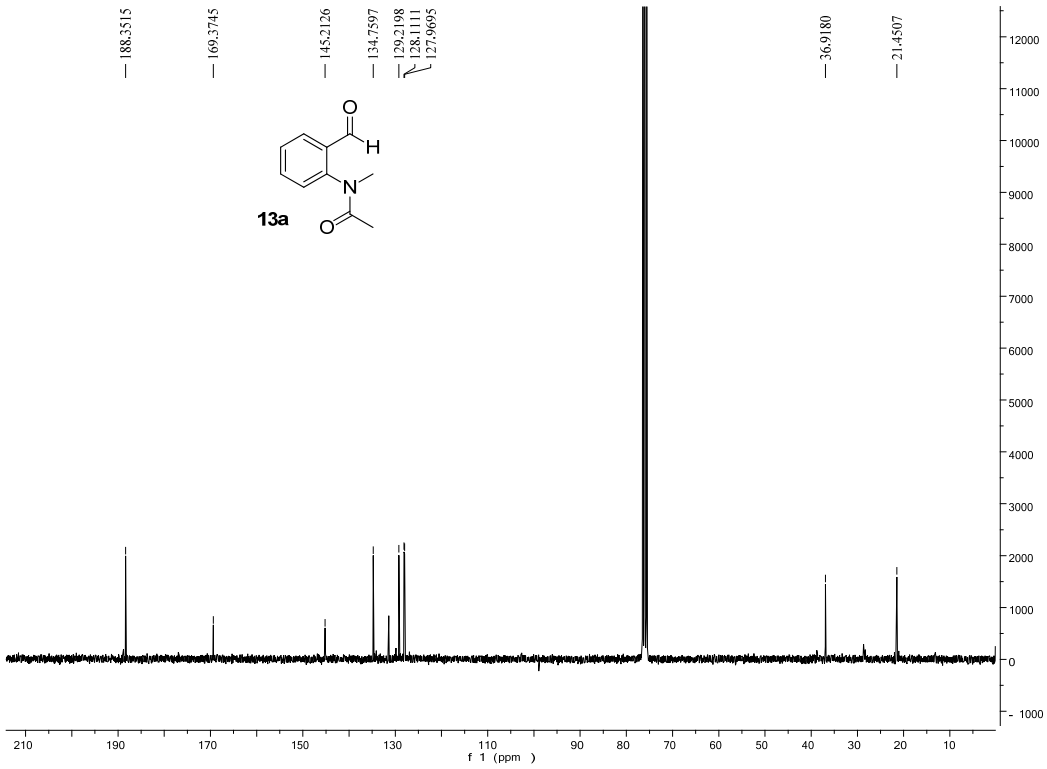
1.01
1.04
1.03
1.01
2.93
3.40
f 1 (ppm)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134.7597
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128.1111
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36.9180
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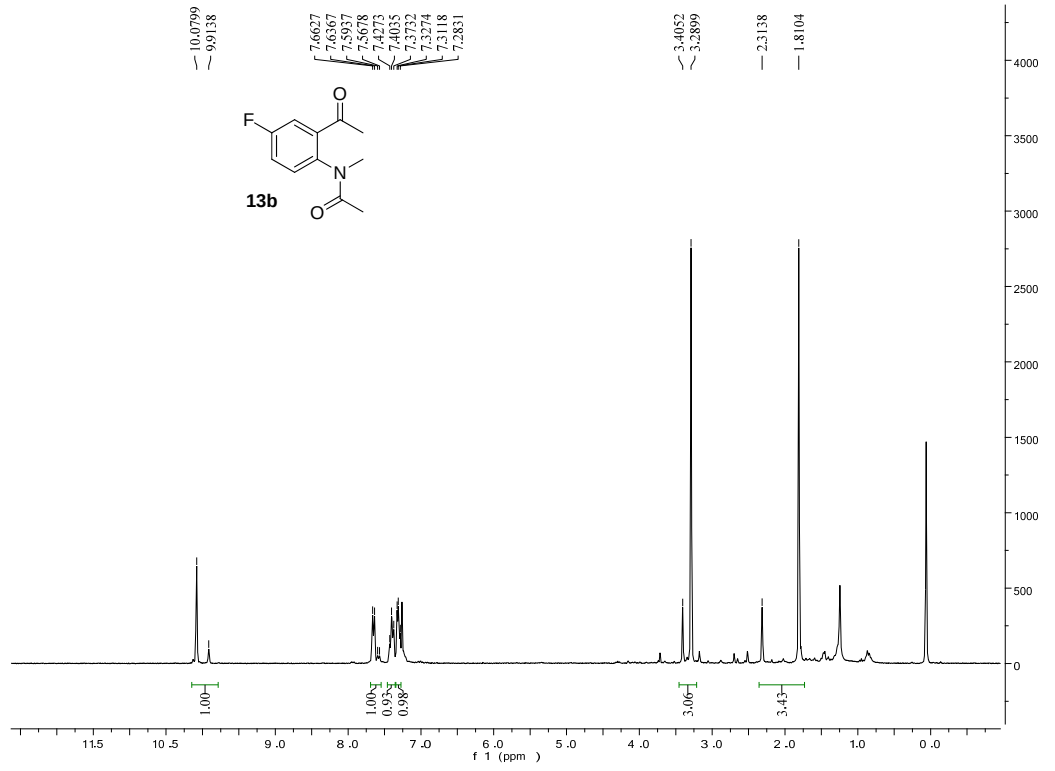
13a
f 1 (ppm)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7.5937
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7.4273
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7.3732
7.3274
7.3118
7.2831
3.4052
3.2899
2.3138
1.8104
F
O
N
O
13b
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1.00
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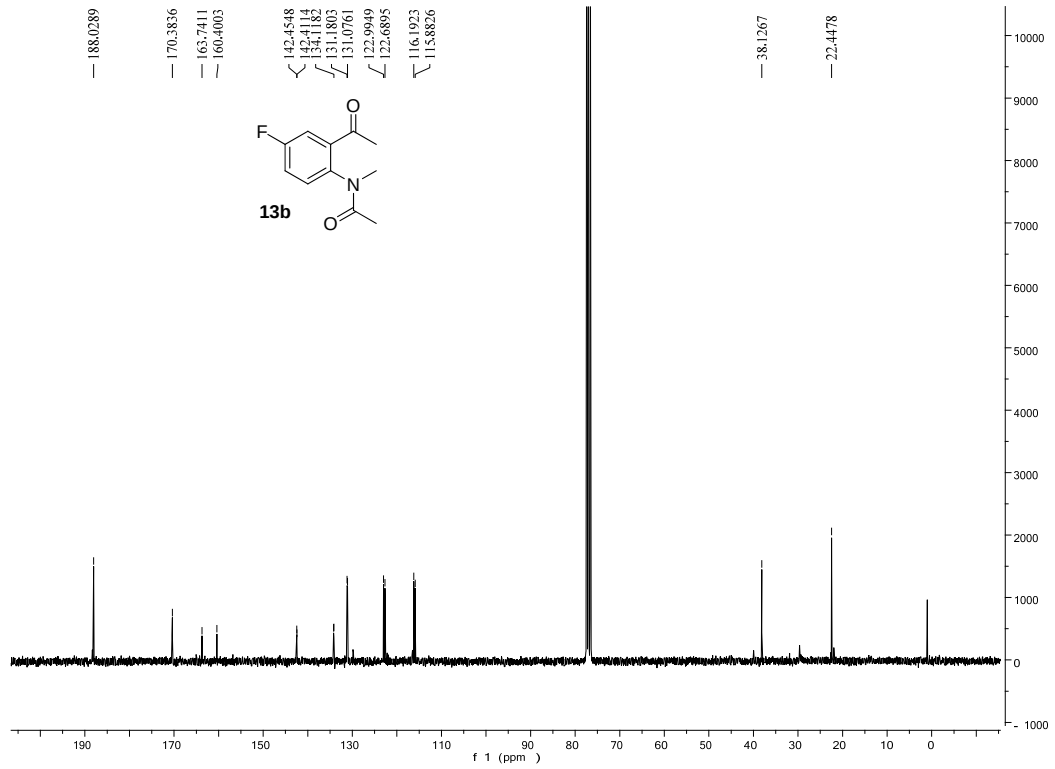
0.98
3.06
3.43
f 1 (ppm)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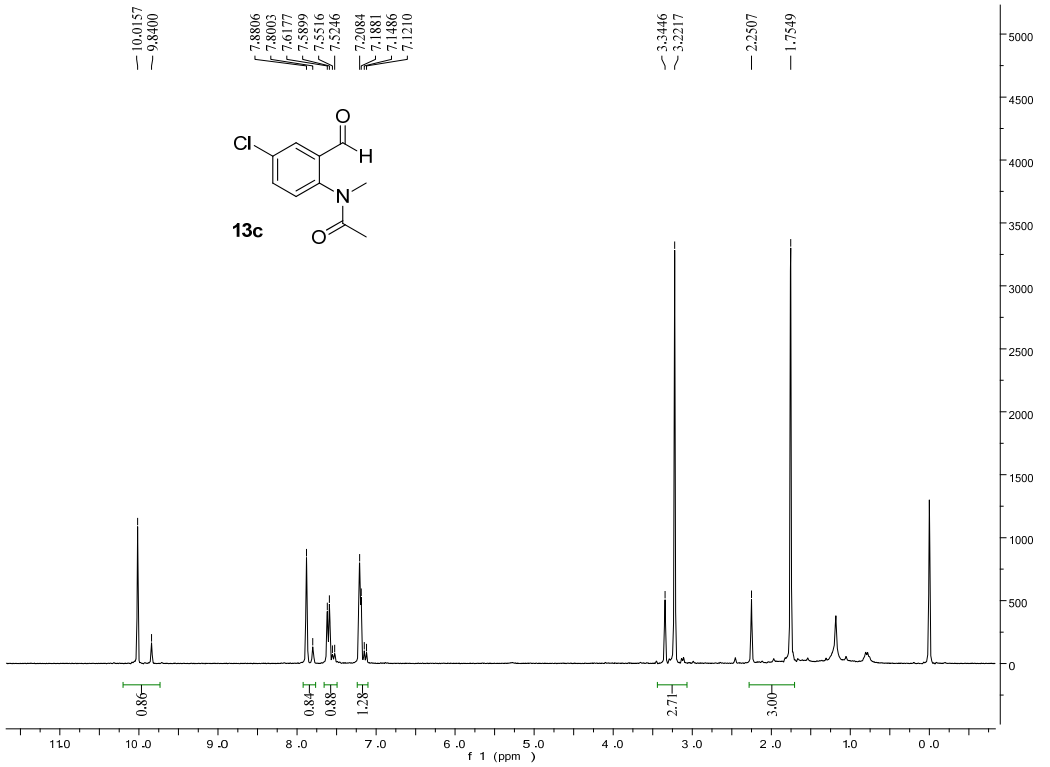
163.7411
160.4003
142.4548
142.4114
134.1182
131.1803
131.0761
122.9949
122.6895
116.1923
115.8826
38.1267
22.4478
F
O
N
O
13b
f 1 (ppm)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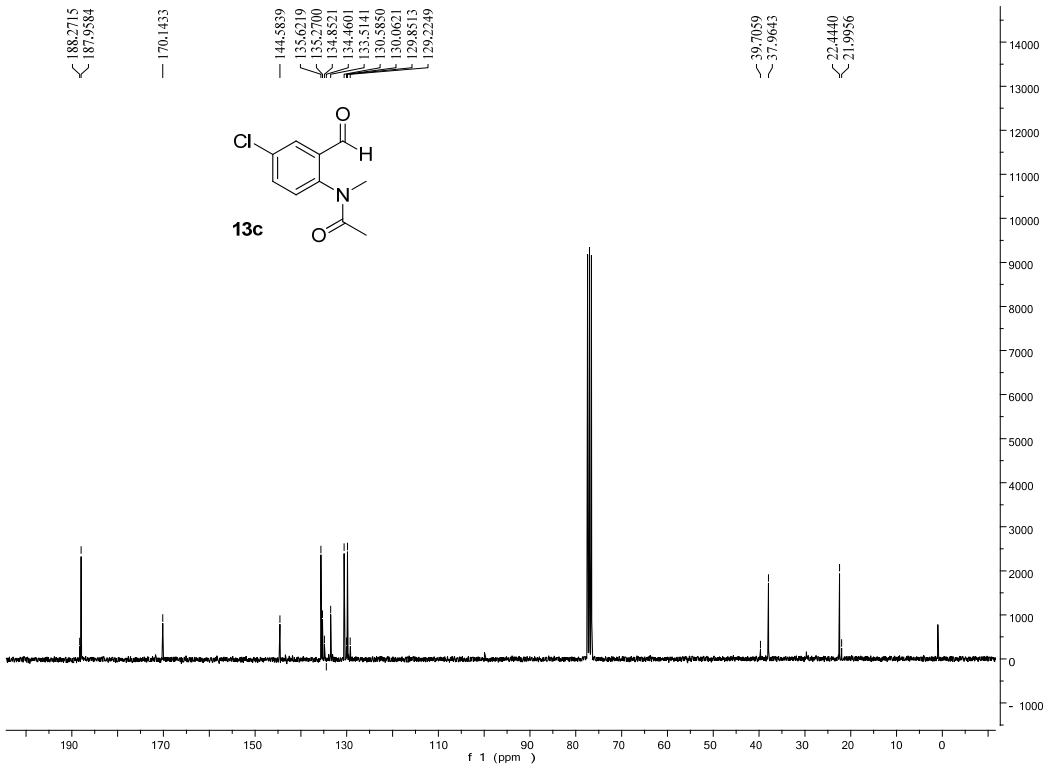
9.8400
7.8806
7.8003
7.6177
7.5899
7.5516
7.5246
7.2084
7.1881
7.1486
7.1210
3.3446
3.2217
2.2507
1.7549
Cl
O
H
N
O
13c
0.86
0.84
0.88
1.28
2.71
3.00
f 1 (ppm)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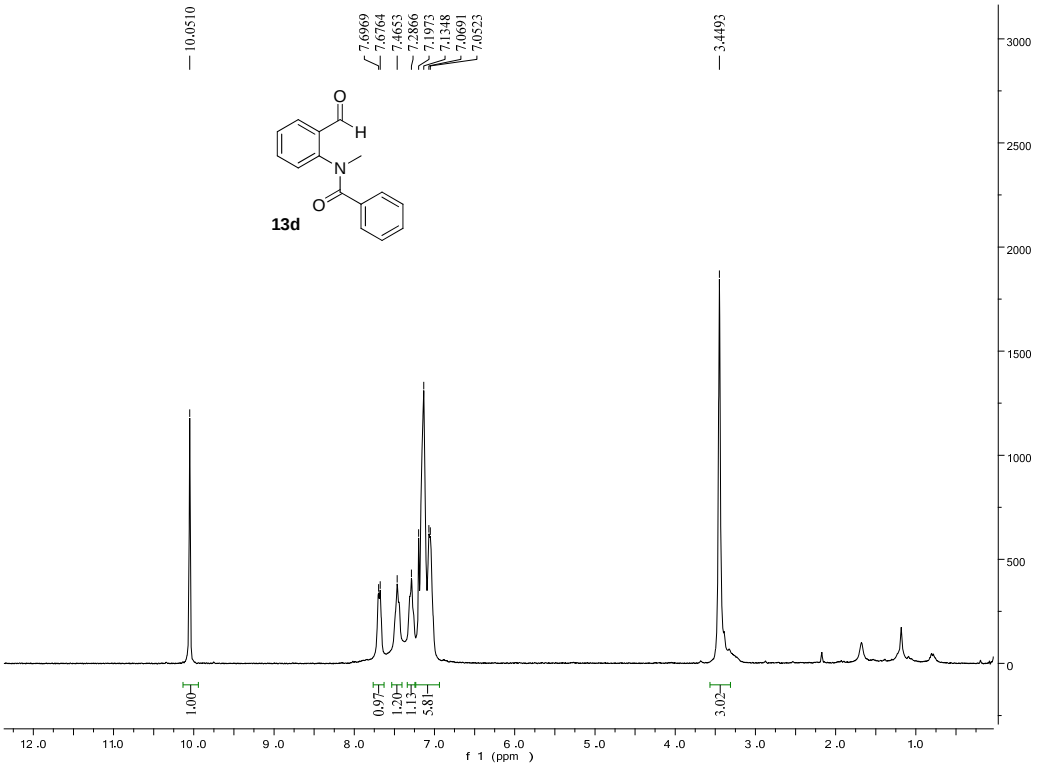
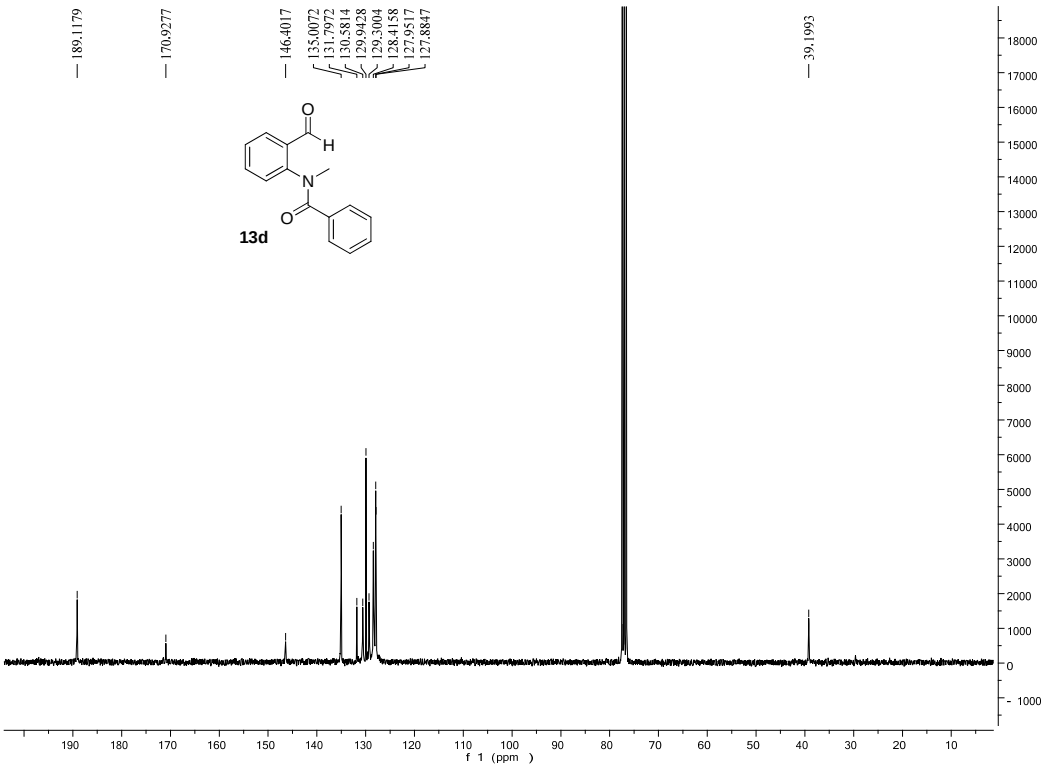
187.9584
170.1433
144.5839
135.6219
135.2700
134.8521
134.4601
133.5141
130.5850
130.0621
129.8513
129.2249
39.7059
37.9643
22.4440
21.9956
Cl
O
H
N
O
13c
f 1 (ppm)


10.0510
7.6969
7.6764
7.4653
7.2866
7.1973
7.1348
7.0691
7.0523
3.4493
13d
1.00
0.97
1.20
1.13
5.81
3.02
f 1 (ppm)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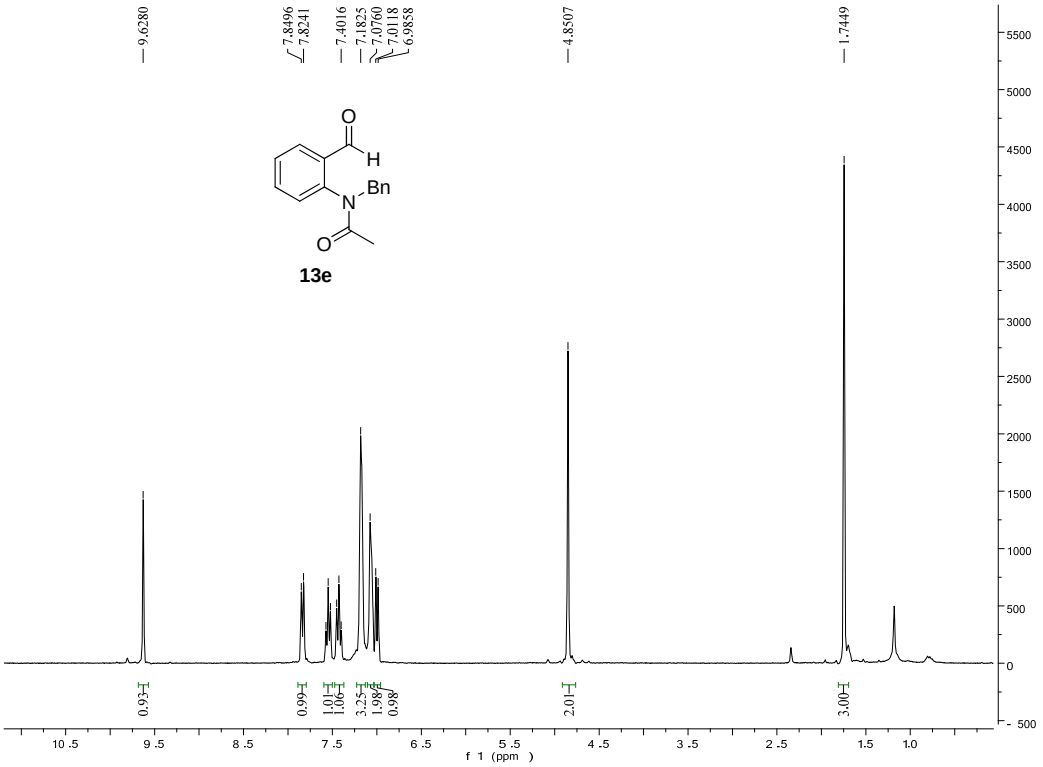
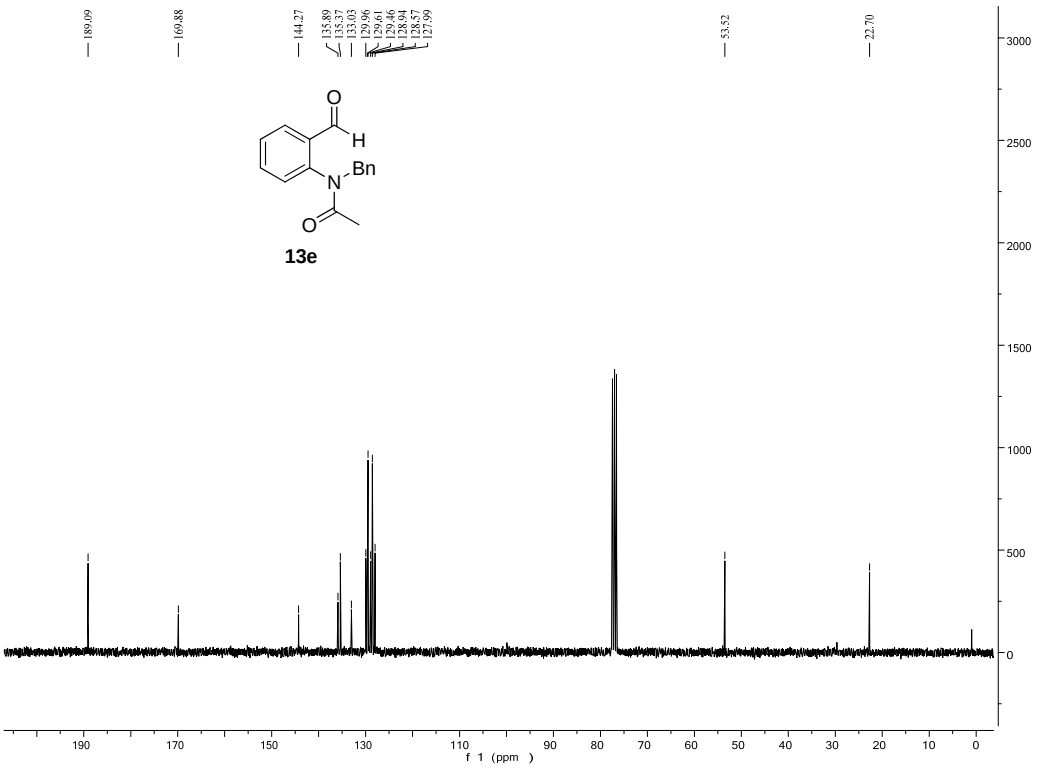
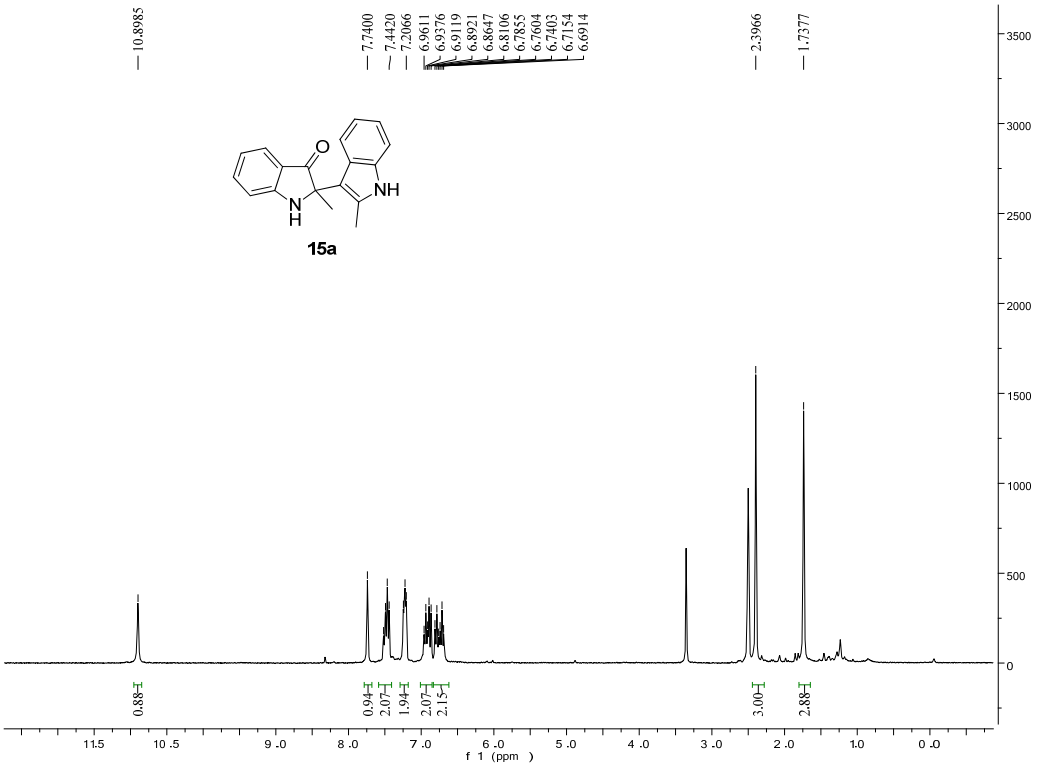
170.9277
146.4017
135.0072
131.7972
130.5814
129.9428
129.3004
128.4158
127.9517
127.8847
39.1993
13d
f 1 (ppm)


9.6280
7.8496
7.8241
7.4016
7.1825
7.0760
7.0118
6.9858
4.8507
1.7449
O
H
Bn
N
O
0.93
0.99
1.01
1.06
3.25
1.98
0.98
2.01
3.00
f 1 (ppm)

13e


189.09
169.88
144.27
135.89
135.37
133.03
129.96
129.61
129.46
128.94
128.57
127.99
53.52
22.70
O
H
Bn
N
O
f 1 (ppm)

13e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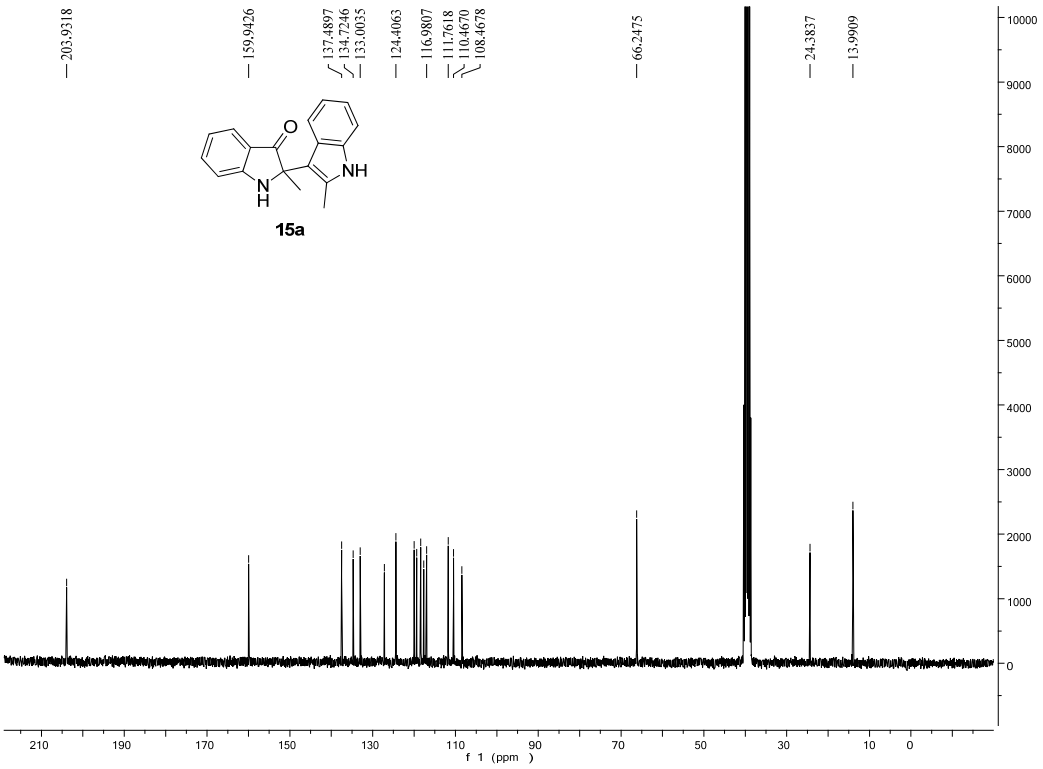
7.7400
7.4420
7.2066
6.9611
6.9376
6.9119
6.8921
6.8647
6.8106
6.7855
6.7604
6.7403
6.7154
6.6914
2.3966
1.7377
O
NH
N
H
15a
0.88
0.94
2.07
1.94
2.07
2.15
3.00
2.88
f 1 (ppm)


203.9318
159.9426
137.4897
134.7246
133.0035
124.4063
116.9807
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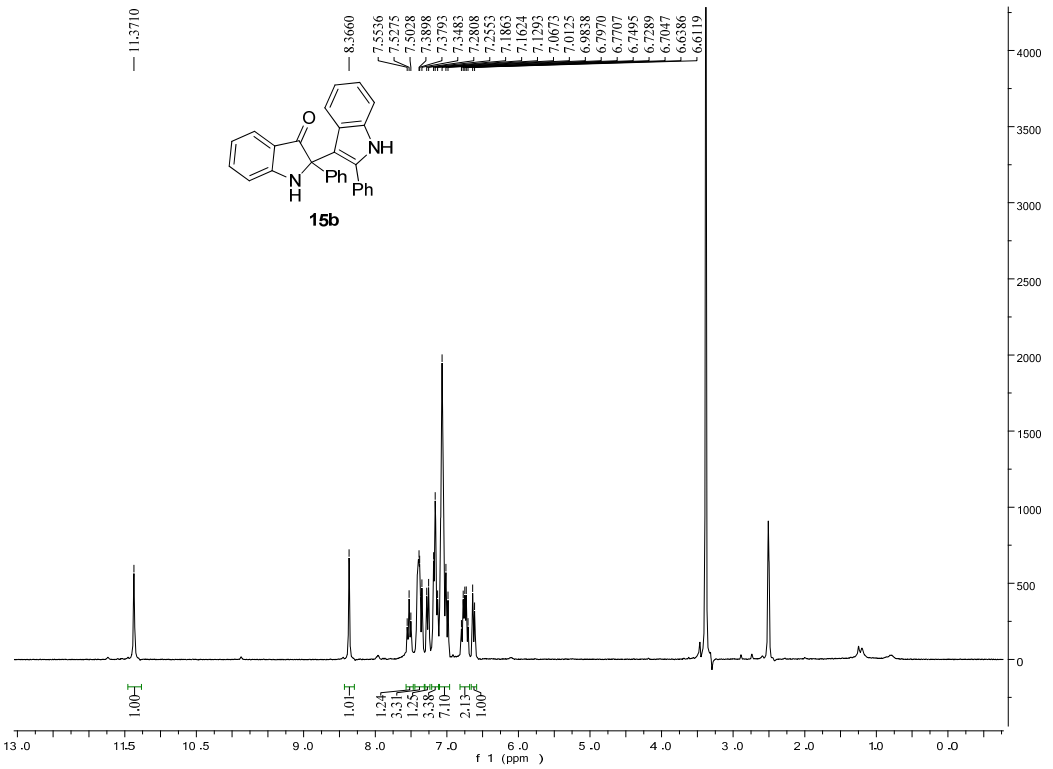
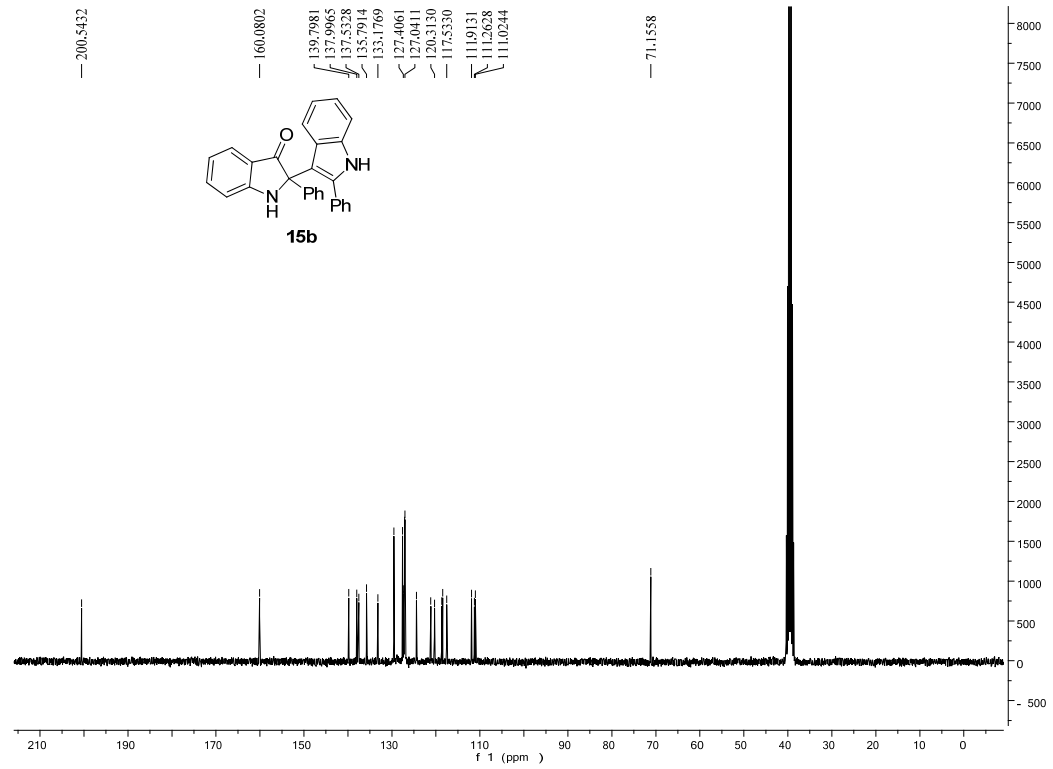
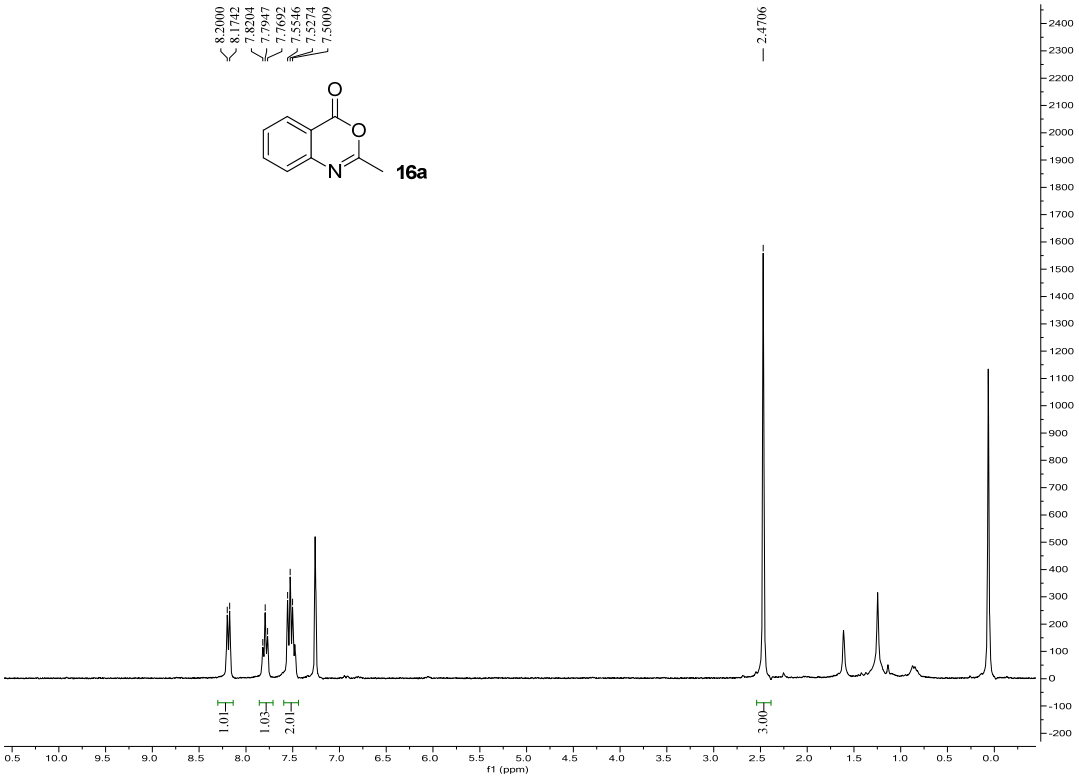
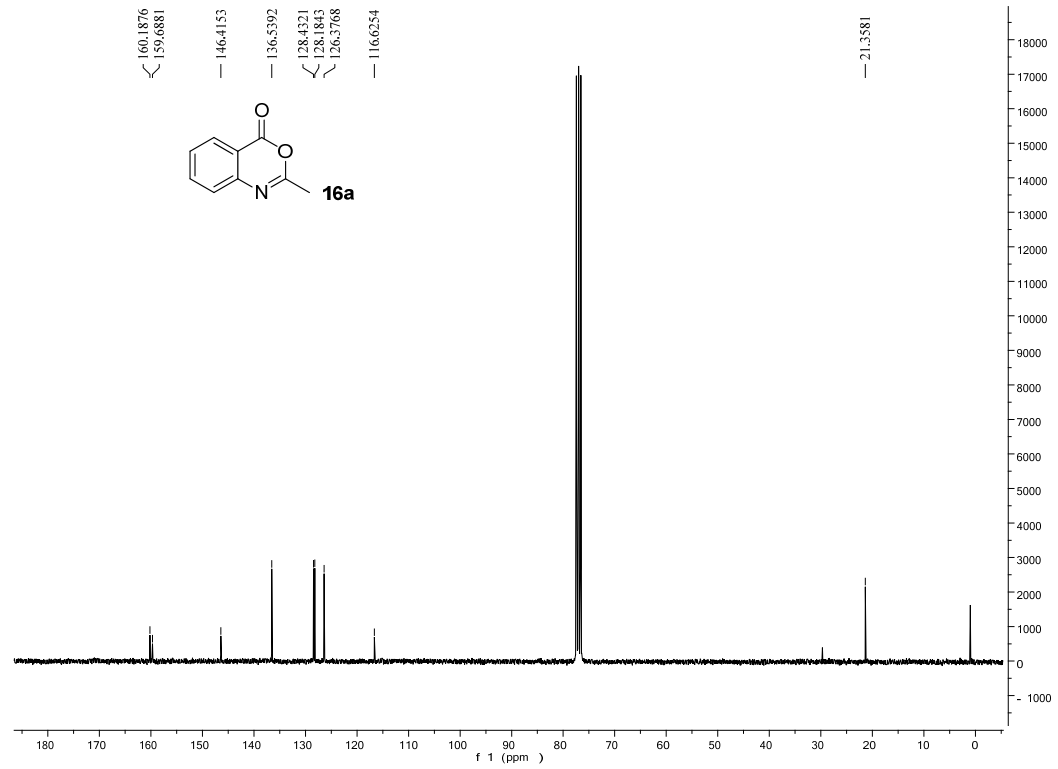
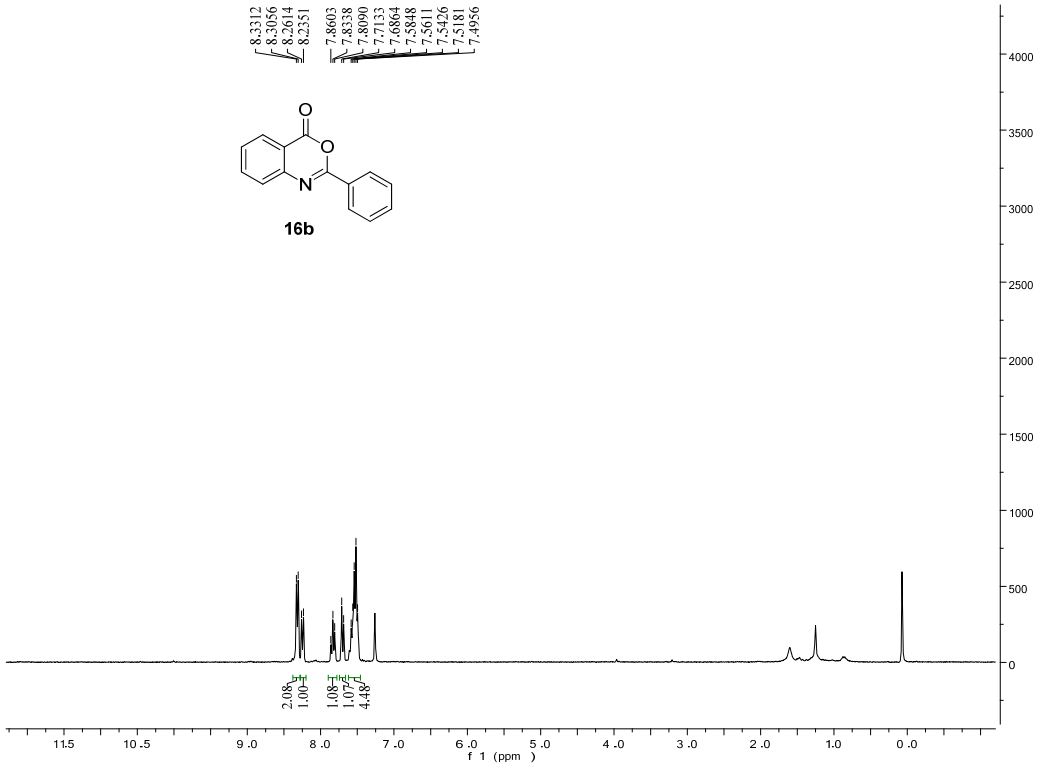
108.4678
66.2475
24.3837
13.9909
O
NH
N
H
15a
f 1 (ppm)


11.3710
8.3660
7.5536
7.5275
7.5028
7.3898
7.3793
7.3483
7.2808
7.2553
7.1863
7.1624
7.1293
7.0673
7.0125
6.9838
6.7970
6.7707
6.7495
6.7289
6.7047
6.6386
6.6119
O
NH
N
H
Ph
Ph
15b
1.00
1.01
1.24
3.31
1.25
3.38
7.10
2.13
1.00
f 1 (ppm)


200.5432
160.0802
139.7981
137.9965
137.5328
135.7914
133.1769
127.4061
127.0411
120.3130
117.5330
111.9131
111.2628
111.0244
71.1558
O
NH
N
H
Ph
Ph
15b
f 1 (ppm)


8.2000
8.1742
7.8204
7.7947
7.7692
7.5546
7.5274
7.5009
2.4706
16a
1.01
1.03
2.01
3.00
f1 (ppm)


160.1876
159.6881
146.4153
136.5392
128.4321
128.1843
126.3768
116.6254
21.3581
16a
f 1 (ppm)


8.3312
8.3056
8.2614
8.2351
7.8603
7.8338
7.8090
7.7133
7.6864
7.5848
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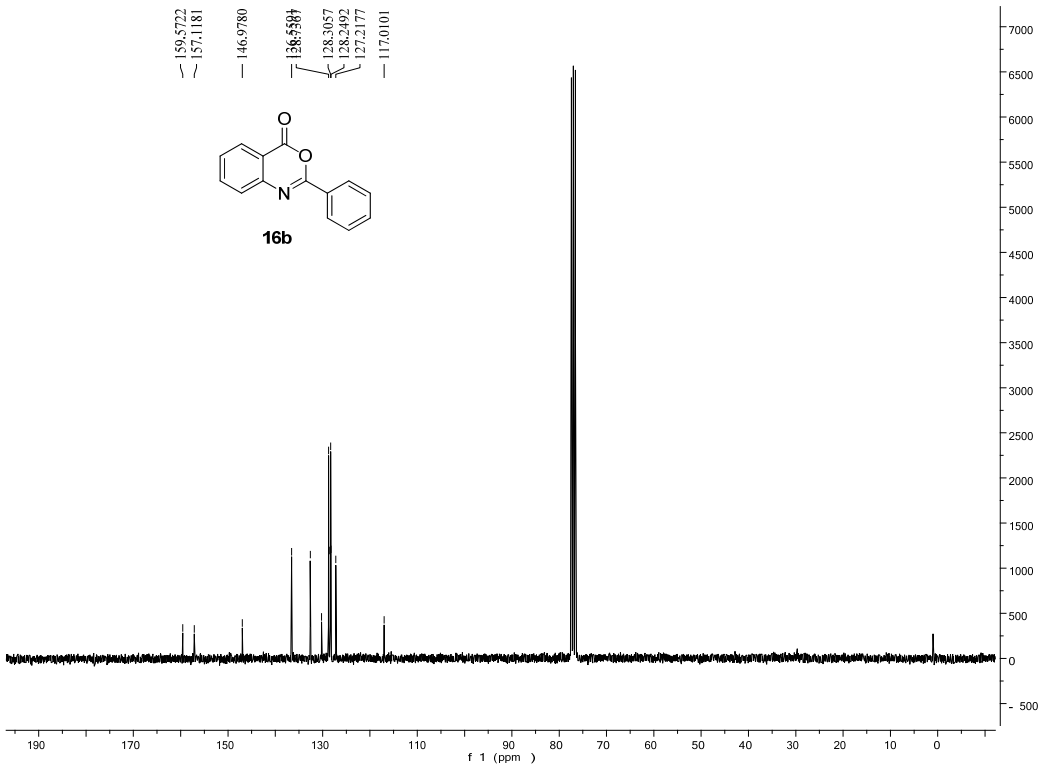
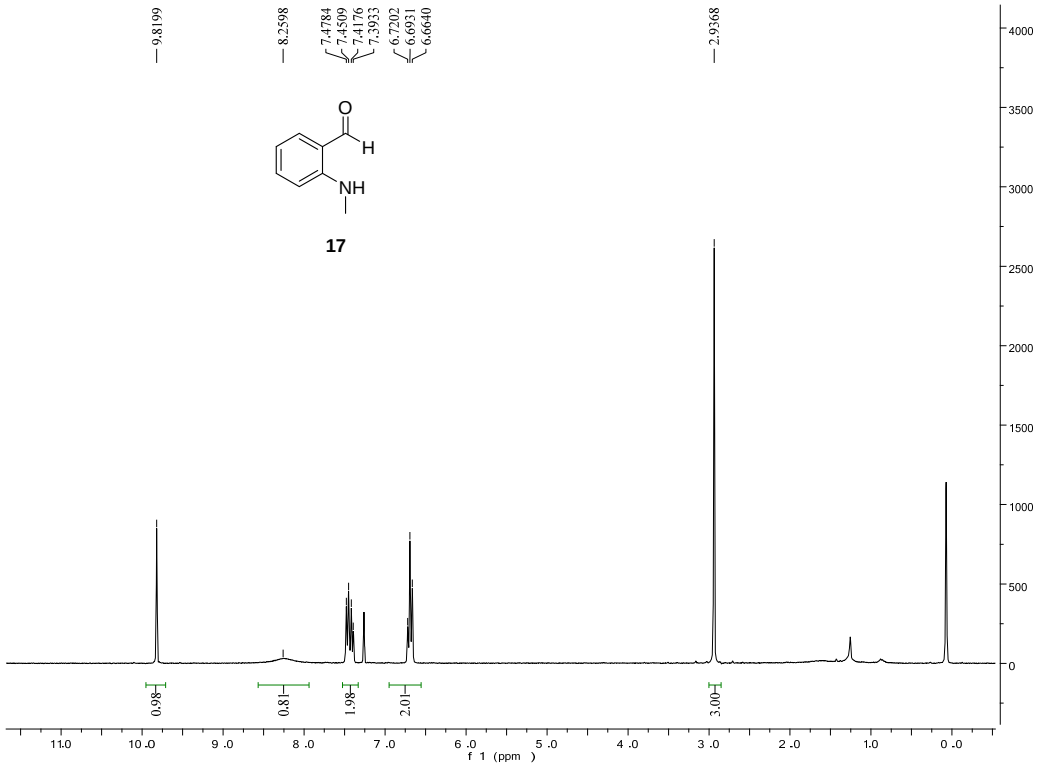
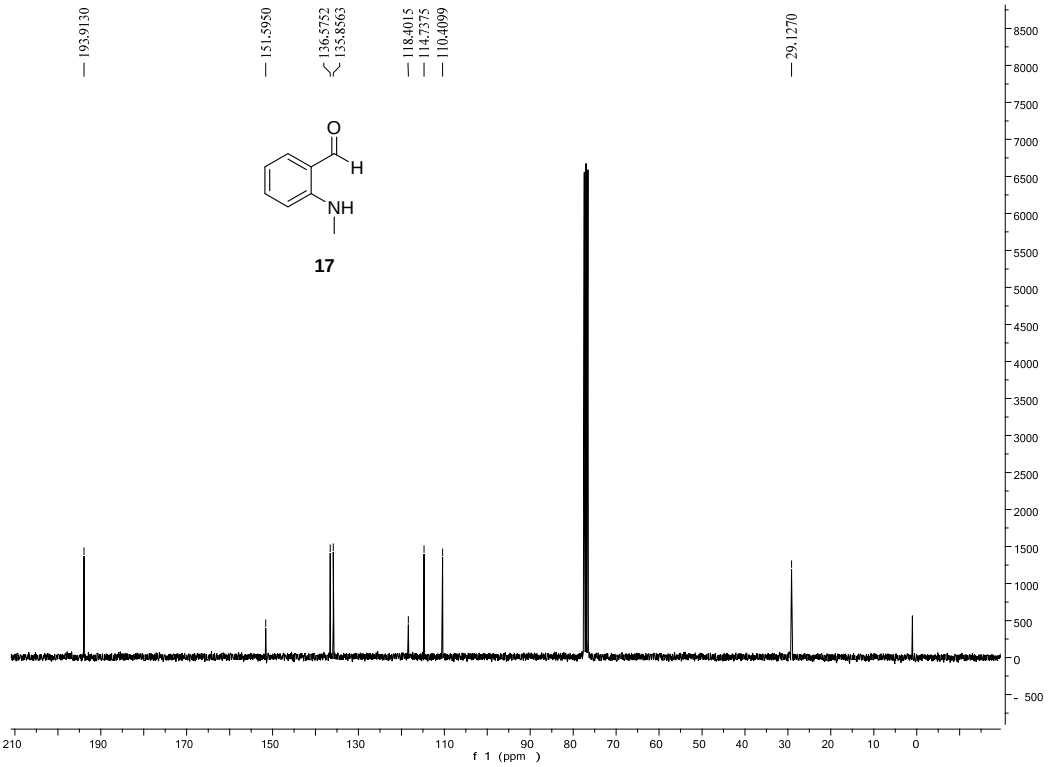
7.5181
7.4956
16b
2.08
1.00
1.08
1.07
4.48
f 1 (ppm)

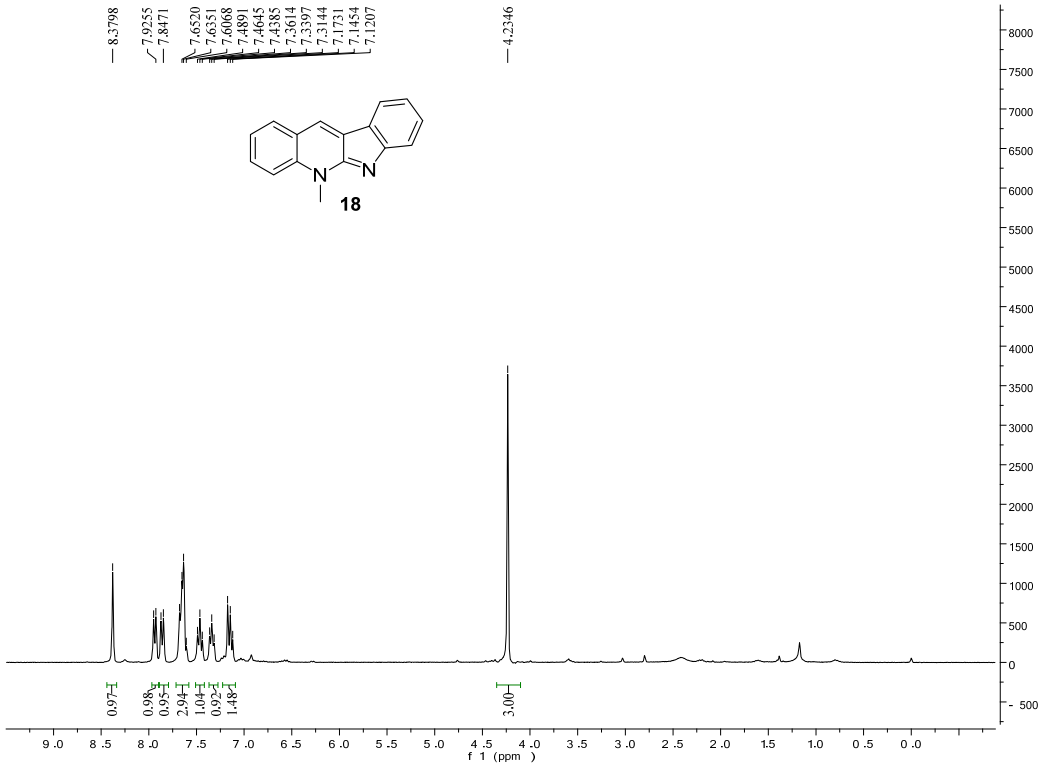

159.5722
157.1181
146.9780
136.5381
128.3057
128.2492
127.2177
117.0101
16b
f 1 (ppm)


9.8199
8.2598
7.4784
7.4509
7.4176
7.3933
6.7202
6.6931
6.6640
2.9368
O
H
NH
0.98
0.81
1.98
2.01
3.00
f 1 (ppm)

17


193.9130
151.5950
136.5752
135.8563
118.4015
114.7375
110.4099
29.1270
O
H
NH
f 1 (ppm)

17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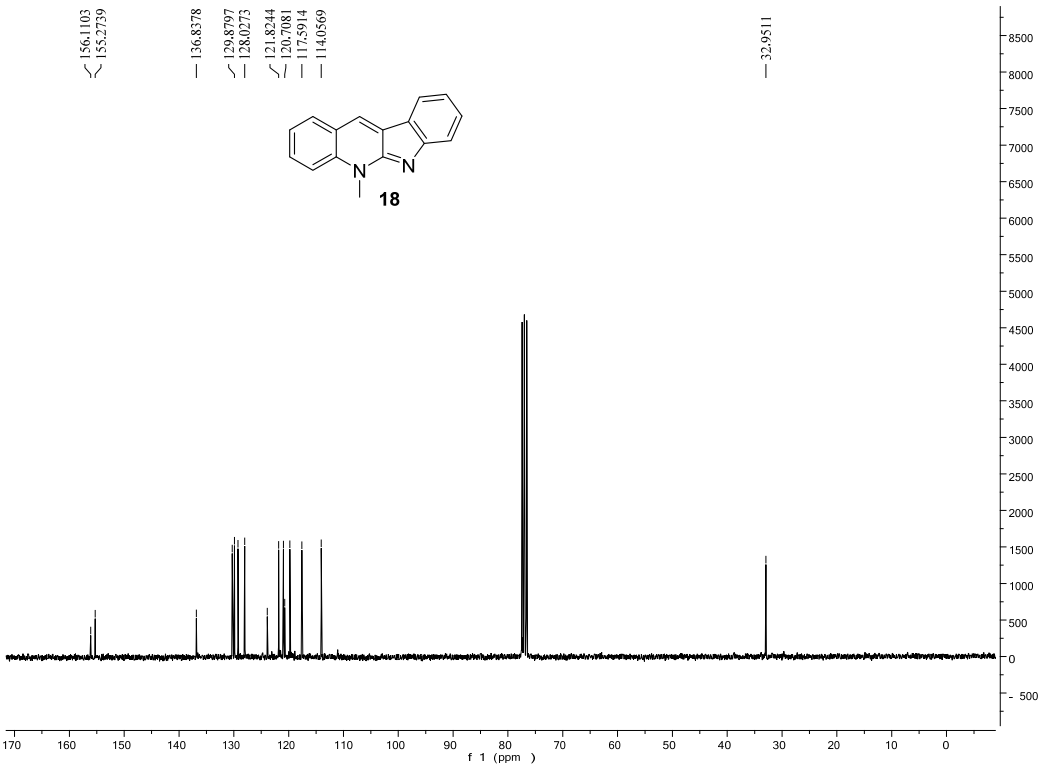
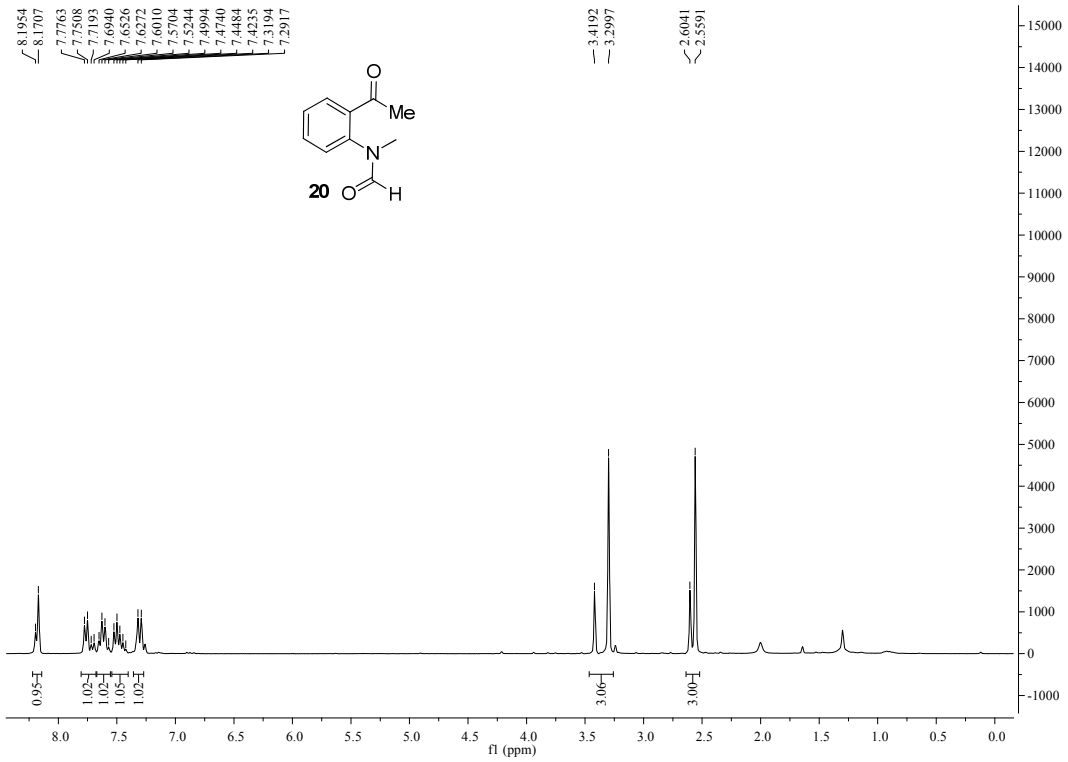
7.9255
7.8471
7.6520
7.6351
7.6068
7.4891
7.4645
7.4385
7.3614
7.3397
7.3144
7.1731
7.1454
7.1207
4.2346
18
0.97
0.98
0.95
2.94
1.04
0.92
1.48
3.00
f 1 (ppm)


156.1103
155.2739
136.8378
129.8797
128.0273
121.8244
120.7081
117.5914
114.0569
32.9511
18
f 1 (ppm)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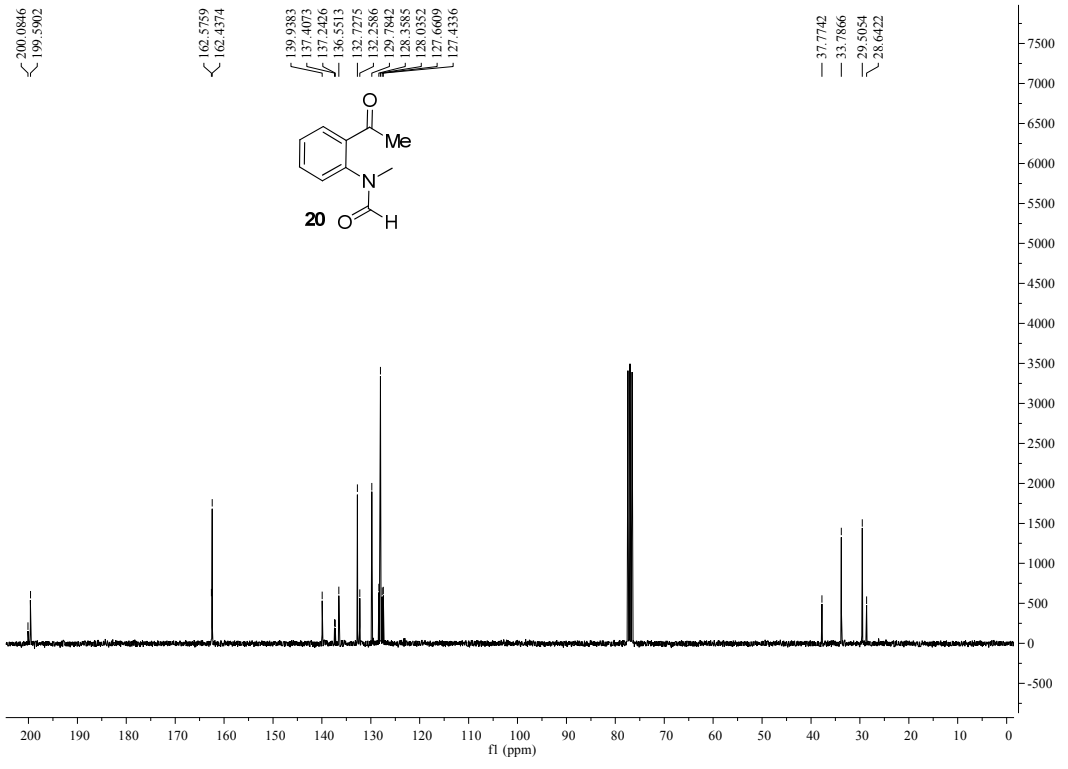
8.1707
7.7763
7.7508
7.7193
7.6940
7.6526
7.6272
7.6010
7.5704
7.5244
7.4994
7.4740
7.4484
7.4235
7.3194
7.2917
3.4192
3.2997
2.6041
2.5591
Me
N
O
H
20
0.95
1.02
1.02
1.05
1.02
3.06
3.00
f1 (ppm)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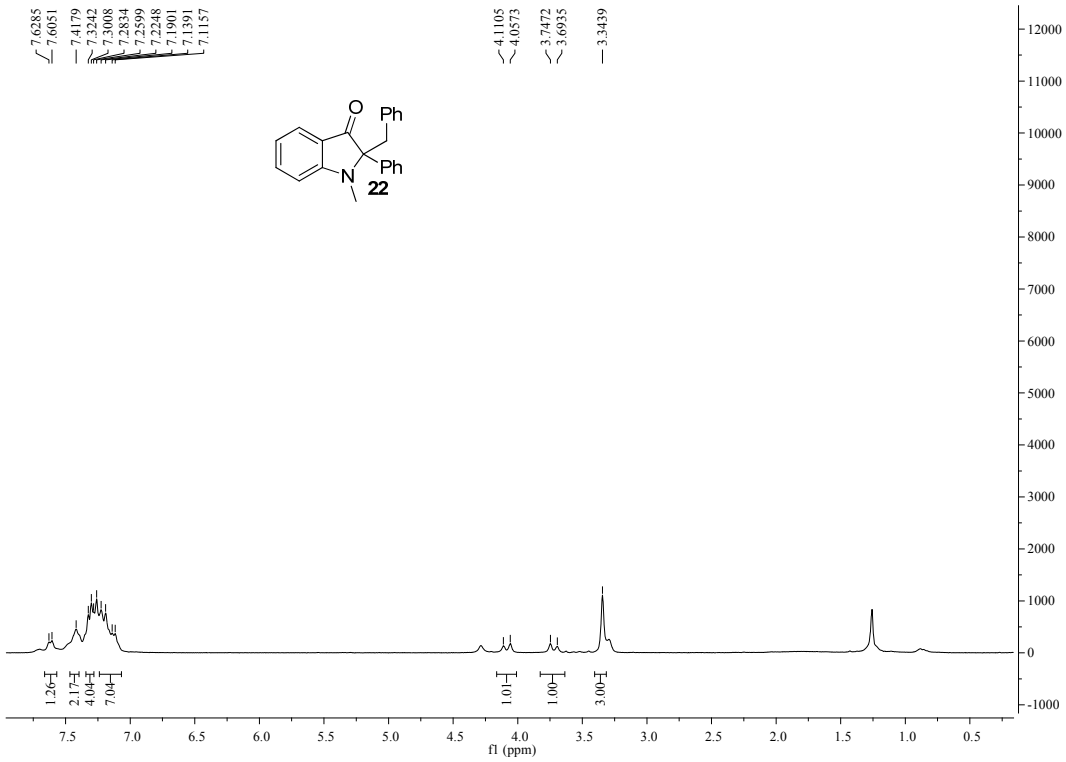
136.5513
132.7275
132.2586
129.7842
128.3585
128.0352
127.6609
127.4336
37.7742
33.7866
29.5054
28.6422
Me
N
O
H
20
f1 (ppm)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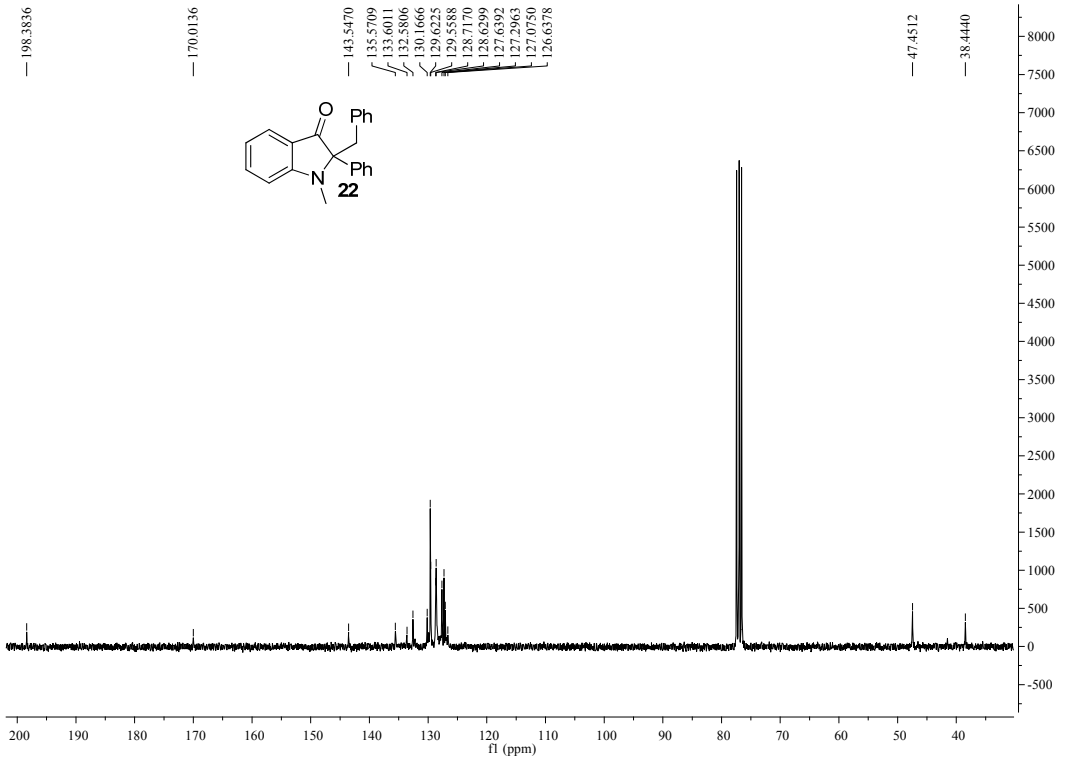
7.4179
7.3242
7.3008
7.2834
7.2599
7.2248
7.1901
7.1391
7.1157
4.1105
4.0573
3.7472
3.6935
3.3439
O
Ph
Ph
N
22
1.26
2.17
4.04
7.04
1.01
1.00
3.00
f1 (ppm)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130.1666
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128.7170
128.6299
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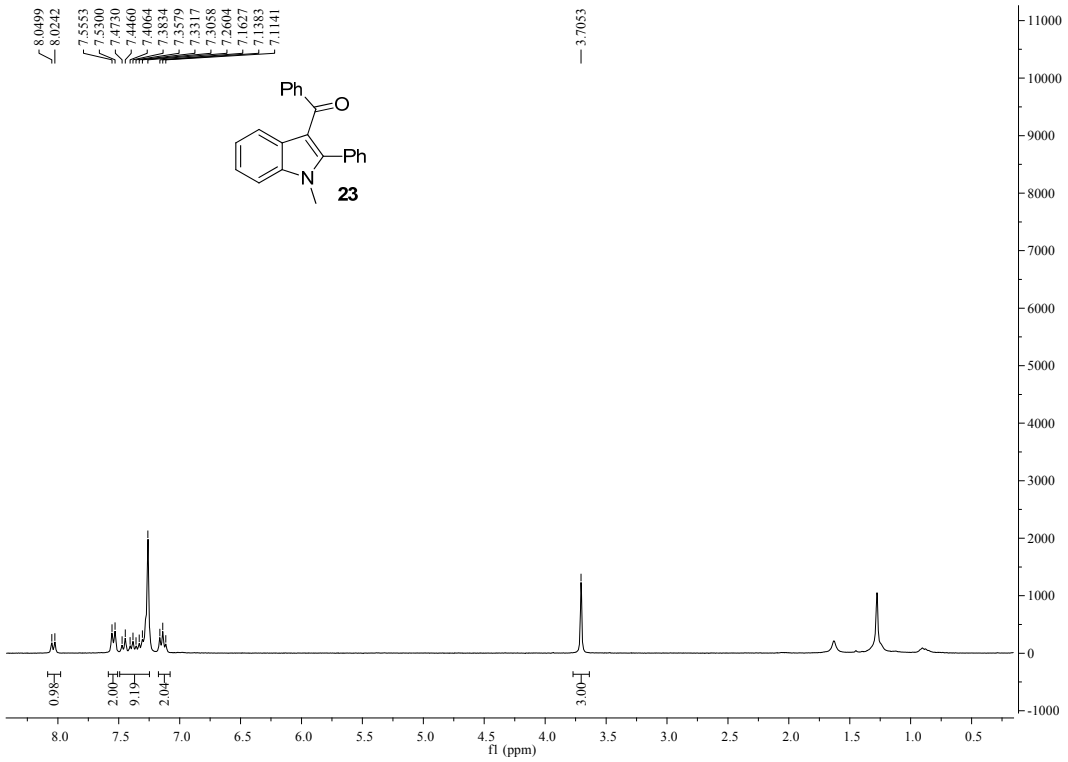
127.2963
127.0750
126.6378
47.4512
38.4440
O
Ph
Ph
N
22
f1 (ppm)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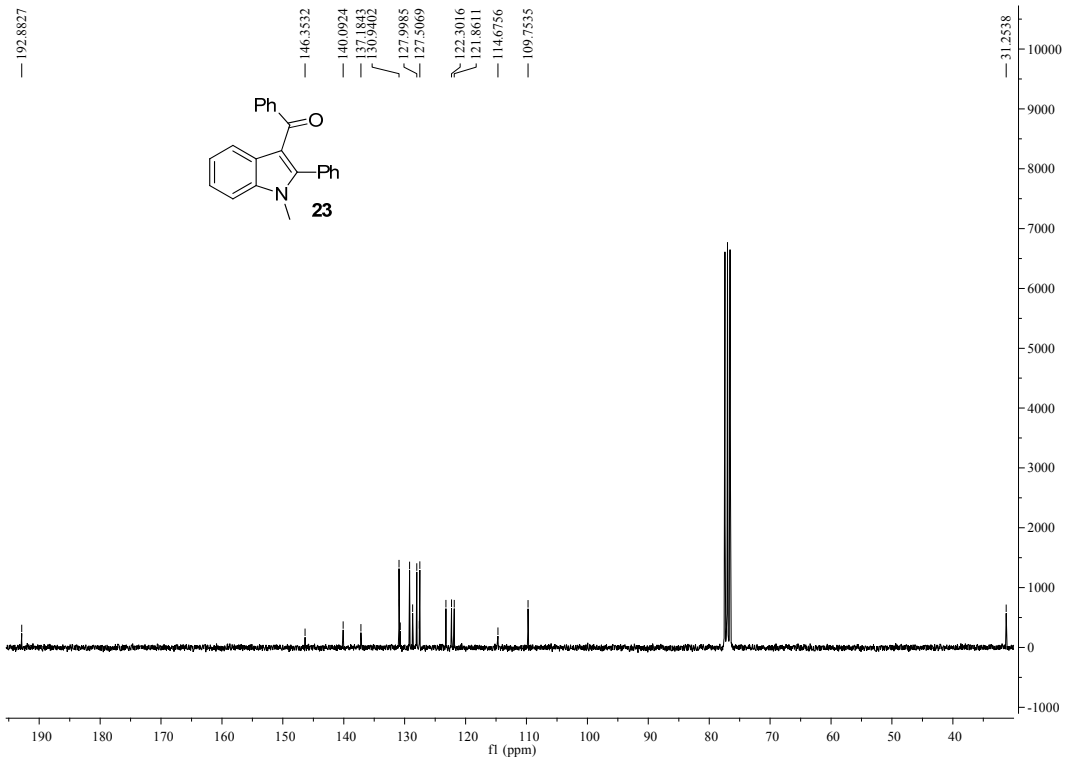
8.0242
7.5553
7.5300
7.4730
7.4460
7.4064
7.3834
7.3579
7.3317
7.3058
7.2604
7.1627
7.1383
7.1141
3.7053
Ph
O
Ph
N
23
0.98
2.00
9.19
2.04
3.00
f1 (ppm)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146.3532
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137.1843
130.9402
127.9985
127.5069
122.3016
121.8611
114.6756
109.7535
31.2538
Ph
O
Ph
N
23
f1 (ppm)