Supporting Information

$AlCl_3$-Promoted Formal [2+3]-Cycloaddition of 1,1-Cyclopropane Diesters with *N*-Benzylic Sulfonamides To Construct Highly Stereoselective Indane Derivatives

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General Information

Solvents were dried and distilled prior to use. NMR spectrum: ^{1}H and ^{13}C spectra were recorded on a Bruker AVANCE 400 spectrometer, operating at 400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR. For ^{1}H NMR, chemical shifts were reported downfield from $CDCl_3$ (δ: 7.26 ppm). For ^{13}C NMR, chemical shifts were reported in the scale relative to the solvent of $CDCl_3$ (δ: 77.0 ppm) used as an internal reference. 2D NOESY spectra were recorded on a Bruker AVANCE 400 spectrometer (400 MHz). High Resolution Mass spectra (HRMS): High Resolution Mass spectra (HRMS) were recorded on Micromass GCT with Electron Ionization (EI) resource or on a Waters LCT Premier XE spectrometer. Chromatography: Column chromatography was performed with silica gel (200-300 mesh ASTM).

Experimental Section

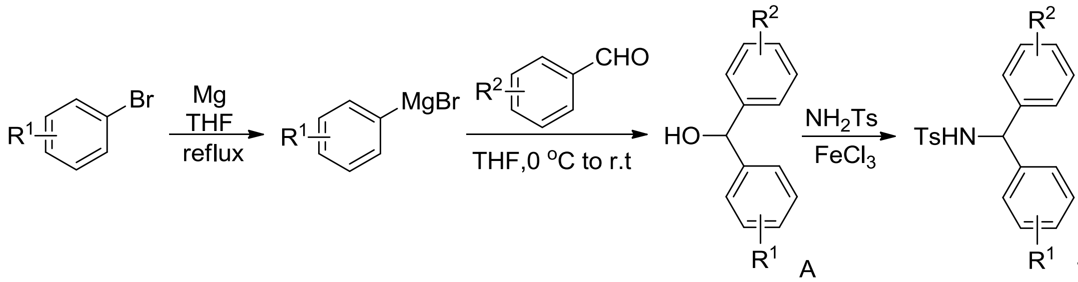
Preparation of 1,1-Cyclopropane Diesters

Donor-acceptor cyclopropanes were prepared according to the method of Stoltz and coworkers.[1a]

Vinylcyclopropane **1m** was prepared according to the method of Johnson and coworkers.[1b]

Preparation of Sulfonamides

Sulfonamides were prepared from the corresponding alcohols and *p*-toluenesulfonamide according to a literature procedure.[2]

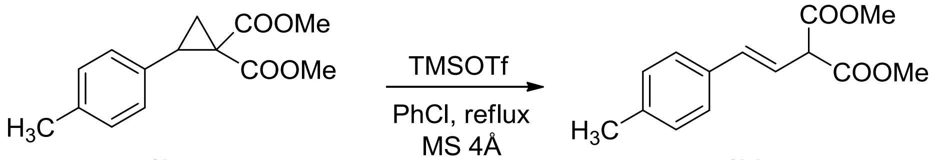


(S 1)

The structures **2a-f** were synthesized from the corresponding alcohols **A** according to a literature procedure.

Preparation of Styrylmalonate 1b'

Styrylmalonate **1b'** was prepared from **1b** according to the published method.[3]



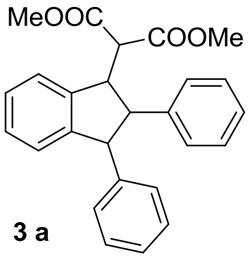
1b **1b'**

A mixture of cyclopropane **1b** (4.0 mmol) and TMSOTf (4.8 mmol) in dry chlorobenzene (50 mL) containing activated molecular sieves 4 at room temperature under an argon atmosphere.The resulting mixture was heated under reflux for 1 h, then poured into 80 mL of saturated aqueous $NaHCO_3$ solution and diluted with CH_2Cl_2 (3×20 mL). The solvent were washed with aqueous $NaHCO_3$ solution (20 mL), water (20 mL) and dried with anhydrous Na_2SO_4. After evaporated under reduced pressure, the crude product was purified by silica gel column chromatography to afford styrylmalonate **1b'** as colorless oil.

General Procedure for $AlCl_3$-Promoted [2+3]-Cycloaddition of 1,1-Cyclopropanes with *N*-Benzylic Sulfonamides

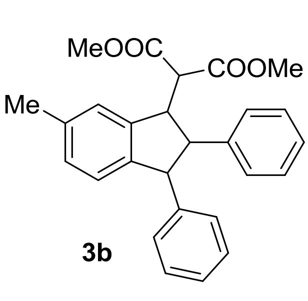
To a solution of *N*-benzylic sulfonamide **2** (0.25 mmol) in dry dichloroethane (1.0 mL) were added cyclopropane **1** (0.25 mmol) and $AlCl_3$ (66.7 mg, 0.5 mmol). The resulting mixture was stirred at 80 ℃ for 1 h. The mixture was cooled to room temperature, and purified by silica gel column chromatography, eluting with petroleum ether/ethyl acetate (80:1 to 50:1), to give indane derivative **3**. NH_2Ts could be detected by TLC. It also has been isolated by silica gel column, eluting with petroleum ether/ethyl acetate (30:1 to 10:1). All the desired products were colorless oil which turned to solid after a period of time. All substrates gave only one product except for **2b** (Table 2, entry 2). The structure of compound **3c** was unambiguously demonstrated by X-ray diffraction analysis and the stereochemistry of compound **3a**, **3c**, **3m** was confirmed by NOESY spectroscopy. The structures of the rest of new products shown in Tables 1 and 2 were determined by analogy with the X-ray structure of **3c**, combining with the acquired ^{1}H, ^{13}C NMR and NOESY spectroscopy.

Characterization Data of Products

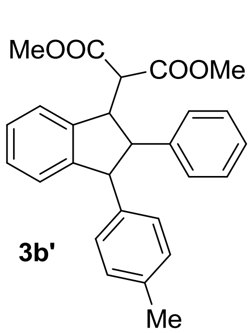


3 a

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26-7.14 (m, 11H), 7.02 (d, J = 7.2 Hz, 2H), 6.89 (d, J = 7.2 Hz, 1H), 4.31 (d, J = 10.0 Hz, 1H), 4.27 (dd, J = 6.4, 10.0 Hz, 1H), 3.86 (d, J = 6.4 Hz, 1H), 3.73 (t, J = 10.0 Hz, 1H), 3.47 (s, 3H), 3.43 (s, 3H); ^{13}C NMR (100 MHz, CDCl3) δ 168.71, 168.38, 145.17, 142.71, 142.15, 140.95, 128.70, 128.47, 128.38, 128.31, 127.73, 127.28, 126.82, 126.71, 125.05, 123.45, 60.27, 59.95, 53.86, 52.29, 52.21, 49.73; HRMS (EI) Calcd for $C_{26}H_{24}O_4$ (M): 400.1675. Found: 400.1678.

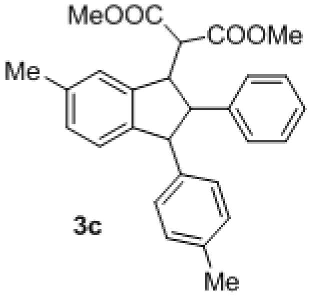


3b

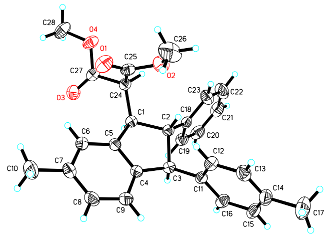


3b'

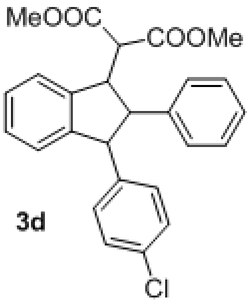
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26-7.13 (m, 8H), 7.05-7.00 (m, 3H), 6.92-6.87 (m, 1H), 7.68 (d, J = 7.6Hz, 1H), 4.30-4.21 (m, 2H), 4.86-4.83 (m, 1H), 3.73-3.67 (m, 1H), 3.47 (s, 3H), 3.42 (s, 3H), 2.35 (s, 2H), 2.30 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.75, 168.71, 168.39, 168.37, 145.39, 143.01, 142.34, 142.25, 142.11, 141.22, 141.15, 139.68, 136.93, 136.14, 129.09, 128.64, 128.59, 128.54, 128.47, 128.44, 128.34, 128.28, 127.69, 127.18, 126.75, 126.63, 125.04, 124.78, 124.02, 123.45, 60.44, 59.70, 53.97, 53.94, 52.21, 52.16, 52.14, 49.67, 21.55, 21.10; HRMS: m/z calcd for ($[C_{27}H_{26}O_4 +H]^+$): 415.1909. Found: 415.1906.



3c

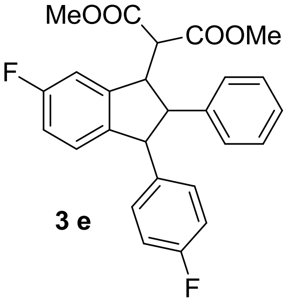


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29-7.24 (m, 2H), 7.22-7.16 (m, 3H), 7.08-7.02 (m, 4H), 6.93 (d, J = 8.0 Hz, 2H), 6.81 (d, J = 8.0 Hz, 1H), 4.22-4.28 (m, 2H) 3.86 (d, J = 6.4 Hz, 1H), 3.70 (t, J = 10.0 Hz, 1H), 3.52 (s, 3H), 3.46 (s, 3H), 2.38 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.77, 168.37, 142.44, 142.27, 141.35, 139.93, 136.83, 136.05, 129.03, 128.54, 128.47, 128.44, 128.24, 126.68, 124.76, 123.99, 60.41, 59.26, 54.03, 52.20, 52.12, 49.66, 21.53, 21.09; HRMS (EI) Calcd for $C_{28}H_{28}O_4$ (M): 428.1988. Found: 428.1992. The X-ray structure of compound **3c** is shown above.



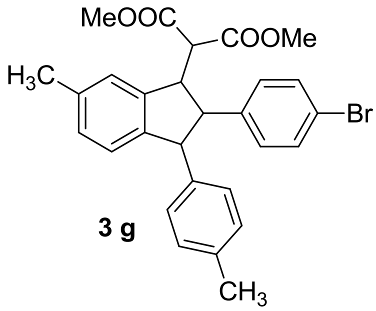
3d

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27-7.19 (m, 8H), 7.16-7.13 (m, 2H), 6.98-6.94 (m, 2H), 6.86 (d, J = 8.0 Hz, 1H), 4.30-4.23 (m, 2H), 3.87 (d, J = 6.0 Hz, 1H), 3.70 (t, J = 10.4 Hz, 1H), 3.44 (s, 3H), 3.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.56, 168.32, 144.61, 142.19, 141.32, 140.61, 132.47, 130.02, 128.57, 128.42, 128.39, 127.82, 127.48, 126.97, 124.87, 123.47, 60.27, 59.41, 53.58, 52.23, 52.18, 49.69; HRMS: m/z calcd for ($[C_{26}H_{23}ClO_4+Na]^+$): 457.1183. Found: 457.1180.



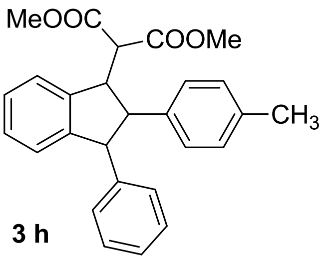
3 e

[1]H NMR (400 MHz, $CDCl_3$): δ 7.31-7.20 (m, 3H), 7.18-7.15 (m, 2H), 7.02-76.91 (m, 6H), 5.85-5.81 (m, 1H), 4.29-4.21 (m, 2H), 3.84 (d, *J* = 6.0 Hz, 1H), 3.73 (t, *J* = 10.4 Hz, 1H), 3.51 (d, *J* = 0.8 Hz, 6H); [13]C NMR (100 MHz, $CDCl_3$) δ 168.32, 168.25, 163.74, 163.06, 161.31, 160.63, 144.14, 144.06, 140.44, 140.41, 140.25, 138.19, 138.16, 130.01, 129.93, 128.43, 128.36, 127.07, 125.98, 125.89, 115.40, 115.19, 114.89, 114.67, 111.00, 110.77, 60.86, 58.42, 53.31, 52.33, 52.30, 49.47, 49.45; HRMS (EI) Calcd for $C_{26}H_{22}F_2O_4$ (M): 436.1486. Found: 436.1483.



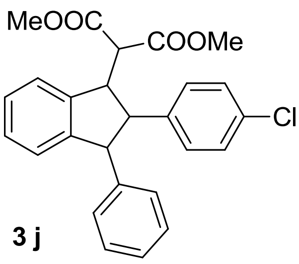
3 g

[1]H NMR (400 MHz, $CDCl_3$): δ 7.42-7.38 (m, 2H), 7.10-7.05 (m, 5H)， 7.02 (s, 1H), 6.93 (d, *J* = 8 Hz, 2H), 6.81 (d, *J* = 8 Hz, 1H), 4.22-4.17 (m, 2H), 3.86 (d, *J* = 6.4 Hz, 1H), 3.70 (t, *J* = 10.0 Hz, 1H), 3.53 (s, 3H), 3.50 (s, 3H), 2.38 (s, 3H), 2.34 (s, 3H); [13]C NMR (100 MHz, $CDCl_3$) δ 168.63, 168.25, 142.13, 141.97, 140.47, 139.52, 136.99, 136.28, 131.34, 130.14, 129.14, 128.68, 128.43, 124.76, 123.91, 120.46, 59.78, 59.26, 53.84, 52.27, 52.19, 49.52, 21.52, 21.09; HRMS (EI) Calcd for $C_{28}H_{27}BrO_4$ (M): 506.1093. Found: 506.1041.



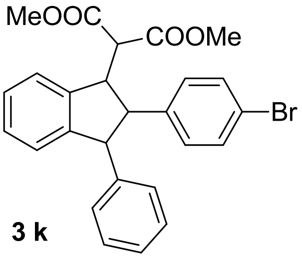
3 h

[1]H NMR (400 MHz, $CDCl_3$): δ 7.29-7.23 (m, 6H), 7.09-7.05 (m, 6H), 6.93-6.90 (m, 1H), 4.33 (d, *J* =10.0 Hz, 1H), 4.26 (dd, *J* = 6.4, 10.0 Hz, 1H), 3.88 (d, *J* = 6.4 Hz, 1H), 3.72 (t, *J* = 10.0 Hz), 3.53 (s, 3H), 3.50 (s, 3H), 2.33 (s, 3H); [13]C NMR (100 MHz, $CDCl_3$) δ 168.68, 168.51, 145.27, 142.85, 142.12, 137.80, 136.24, 128.97, 128.69, 128.33, 128.27, 127.63, 127.17, 126.62, 125.00, 123.54, 59.94, 59.75, 53.80, 52.22, 52.19, 49.87, 21.06; HRMS (EI) Calcd for $C_{27}H_{26}O_4$ (M): 414.1831. Found: 414.1834.



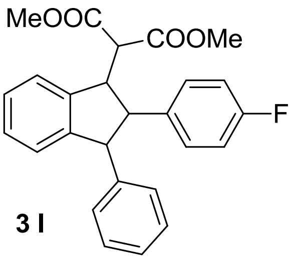
3 j

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.22 (m, 8H), 7.15-7.11 (m, 2H), 7.07-7.03 (m, 2H), 6.93-6.90 (m, 1H), 4.30-4.23 (m, 2H), 3.89 (d, *J* = 6.0 Hz, 1H), 3.77 (t, *J* = 10.4 Hz, 1H), 3.52 (s, 3H), 3.50 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.53, 168.24, 144.86, 142.32, 141.86, 139.58, 132.49, 129.75, 128.63, 128.46, 128.44, 127.83, 127.37, 126.86, 125.03, 123.38, 59.98, 59.57, 53.69, 52.30, 52.22, 49.66; HRMS (EI) Calcd for $C_{26}H_{23}ClO_4$ (M): 434.1285. Found: 434.1288.



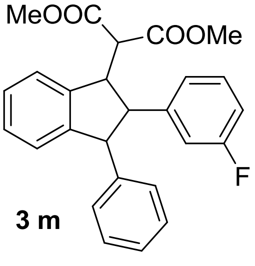
3 k

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39-7.35 (m, 2H), 7.28-7.18 (m, 6H), 7.05-7.00 (m, 4H), 6.90-6.87 (m, 1H), 4.26-4.19 (m, 2H), 3.86 (d, *J* = 6.0 Hz, 1H), 3.72 (t, *J* = 10.0 Hz, 1H), 3.49 (s, 3H), 3.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.71, 168.38, 145.17, 142.71, 142.15, 140.95, 128.70, 128.47, 128.38, 128.31, 127.73, 127.28, 126.82, 126.71, 125.05, 123.45, 60.27, 59.95, 53.86, 52.29, 52.21, 49.73; HRMS: m/z calcd for ($[C_{26}H_{23}BrO_4+H]^+$): 479.0858. Found: 479.0858.



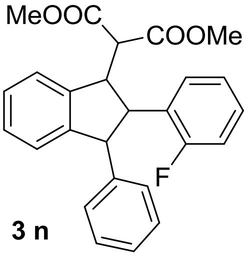
3 l

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28-7.19 (m, 6H), 7.14-7.10 (m, 2H), 7.03-7.00 (m, 2H)，6.96-6.91 (m, 2H), 6.90-6.87 (m, 1H), 4.26-4.19 (m, 2H), 3.86 (d, *J* = 6.0 Hz, 1H), 3.73 (t, *J* = 10.0 Hz, 1H), 3.50 (s, 3H), 3.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.60, 168.27, 162.94, 160.50, 144.92, 142.41, 141.95, 136.67, 129.90, 129.82, 128.64, 128.43, 127.80, 127.34, 126.81, 125.02, 123.37, 115.21, 115.00, 60.07, 59.49, 53.72, 52.29, 52.20, 49.71; HRMS (EI) Calcd for $C_{26}H_{23}FO_4$ (M): 418.1580. Found: 418.1583.



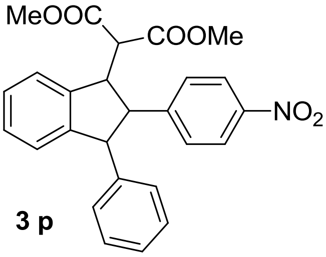
3 m

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.26 (m, 4H), 7.25-7.20 (m, 3H), 7.07-7.05 (m, 2H), 6.97-6.90 (m, 4H), 4.32 (d, *J* = 10.0 Hz, 1H), 4.25 (dd, *J* = 6.0, 10.0 Hz, 1H), 3.90 (d, *J* = 6.0 Hz, 1H), 3.79 (t, *J* = 10.0 Hz, 1H), 3.53 (s, 3H), 3.52 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 168.53, 168.30, 163.94, 161.50, 144.88, 143.95, 143.88, 142.41, 141.79, 129.80, 129.72, 128.62, 128.48, 127.85, 127.39, 126.88, 125.06, 124.23, 124.20, 123.44, 115.19, 114.98, 113.86, 113.65, 59.86, 59.82, 59.81, 53.70, 52.27, 52.23, 49.83; HRMS: m/z calcd for ($[C_{26}H_{23}FO_4+H]^+$): 419.1659. Found: 419.1658.



3 n

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.15 (m, 8H), 7.12-7.09 (m, 2H), 7.07-7.00 (m, 2H), 6.96-6.92 (m, 1H), 4.50 (d, *J* = 10.0 Hz, 1H), 4.44 (dd, *J* = 6.4, 10.0 Hz, 1H), 4.02 (t, *J* = 10.0 Hz, 1H), 3.93 (d, *J* = 6.4 Hz, 1H), 3.52 (s, 3H), 3.50 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.58, 168.30, 162.71, 160.26, 145.22, 142.63, 142.22, 130.87, 130.82, 128.56, 128.45, 128.39, 128.37, 128.00, 127.87, 127.64, 127.20, 126.77, 125.07, 123.99, 123.96, 123.30, 115.81, 115.59, 57.91, 57.89, 54.47, 53.83, 52.22, 52.18, 48.41, 48.38; HRMS (EI) Calcd for $C_{26}H_{23}FO_4$ (M): 418.1580. Found: 418.1581.



3 p

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16-8.13 (m, 2H), 7.37-7.34 (m, 2H), 7.34-7.25 (m, 5H), 7.25-7.23 (m, 1H), 4.34-4.31 (m, 2H), 3.98-3.91 (m, 2H), 3.51 (s, 3H), 3.50 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.33, 168.07, 149.36, 146.91, 144.46, 141.83, 141.46, 129.20, 128.65, 128.52, 128.13, 127.65, 127.17, 125.12, 123.55, 123.37, 60.21, 59.75, 53.62, 52.35, 52.28, 49.67; HRMS (EI) Calcd for $C_{26}H_{23}NO_6$ (M): 445.1525. Found: 445.1528.

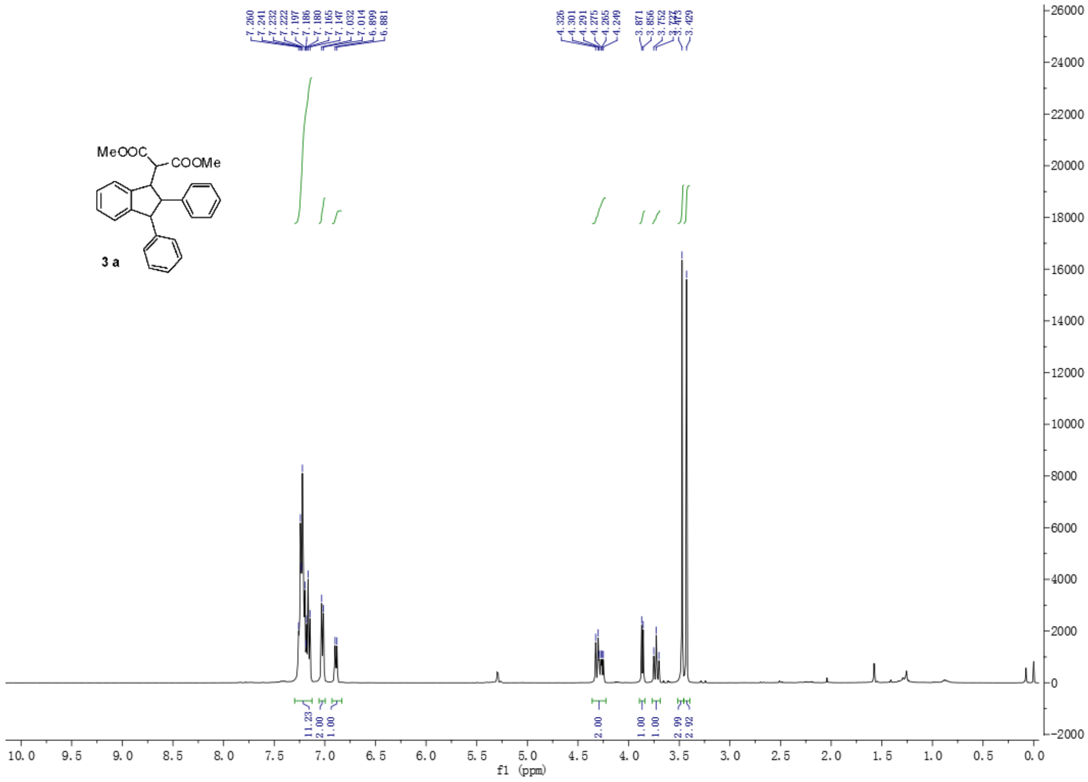
References

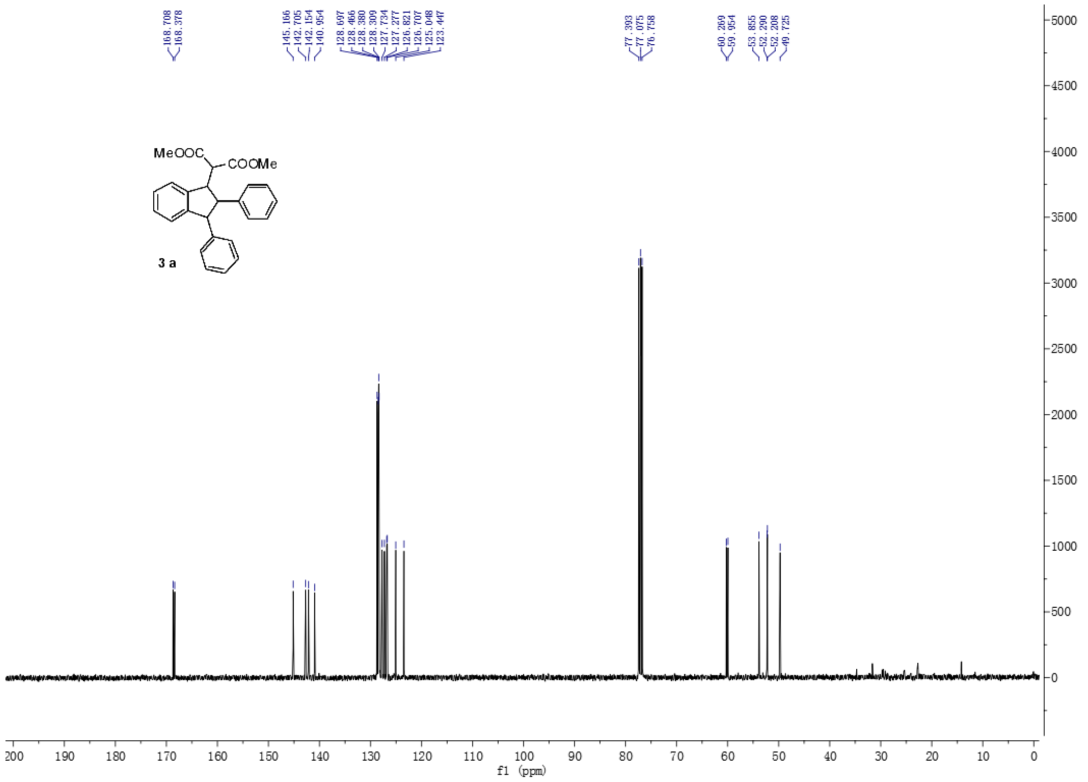
(1) (a) Goldberg, A. F. G.; O’Connor, N. R.; Craig, R. A.; Stoltz, B. M. *Org. Lett.* **2012**, *14*, 5314. (b) Parsons, A. T.; Campbell, M. J.; Johnson, J. S. *Org. Lett.*, **2008**, *10*, 2541.

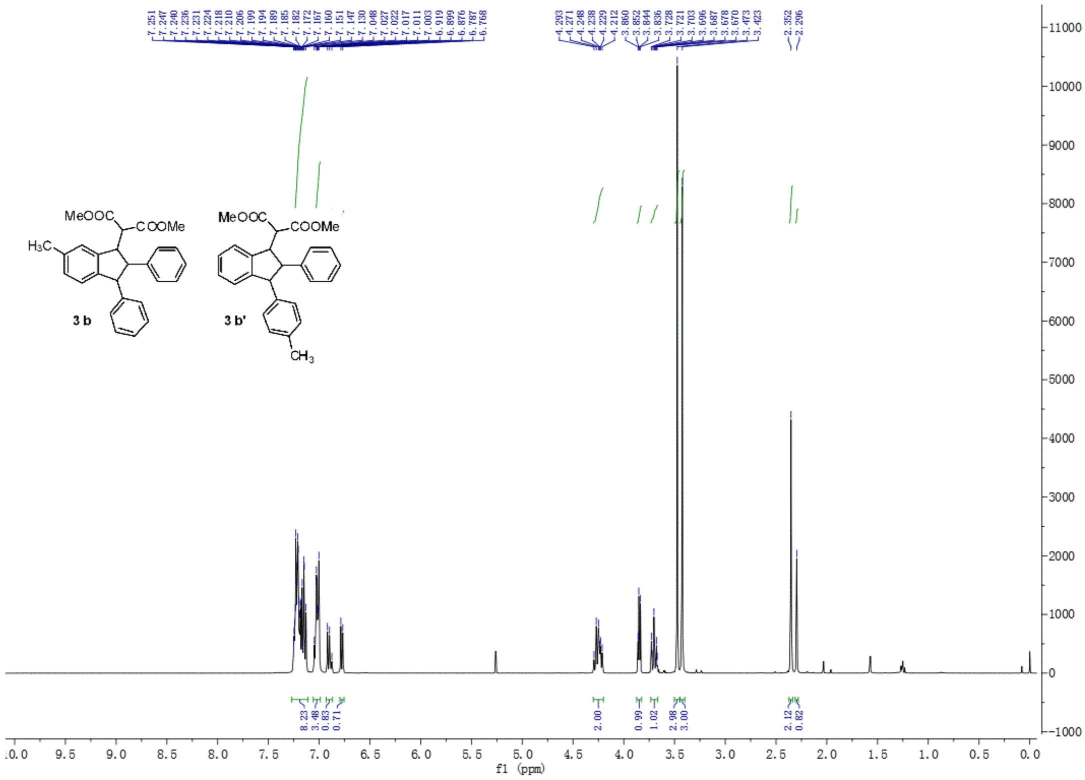
(2) (a) Li, H.; Zhu, R-Y.; Shi, W-J.; He, K-H.; Shi, Z-J. *Org. Lett.* **2012**, *14*, 4850-4853. (b) Jana, U.; Maiti, S.; Biswas, S.; *Tetrahedron Lett.* **2008**, *48*, 858.

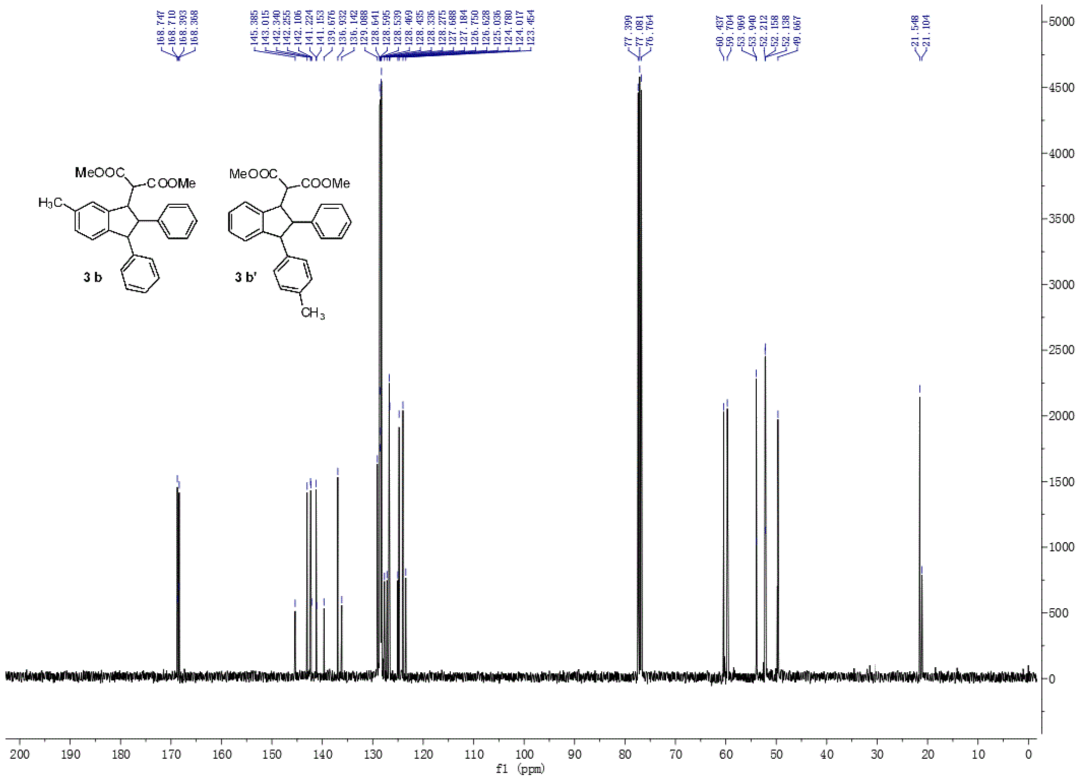
(3) Chagarovskiy, A.O.; Ivanova, O.A.; Rakhmankulov, E. R.; Budynina, E. M.; Trushkov, I. V.; Melnikov, M. Y. *Adv. Synth. Catal.* **2010**, *352*, 3179.

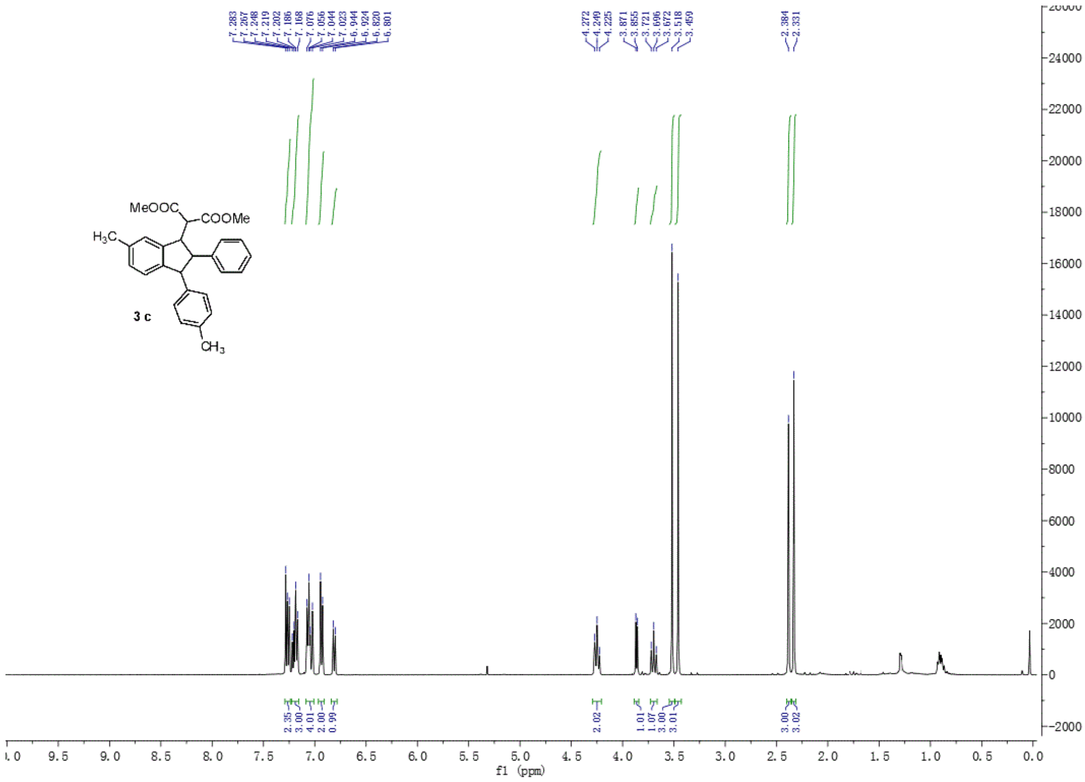
Copies of ^{1}H NMR and ^{13}C NMR







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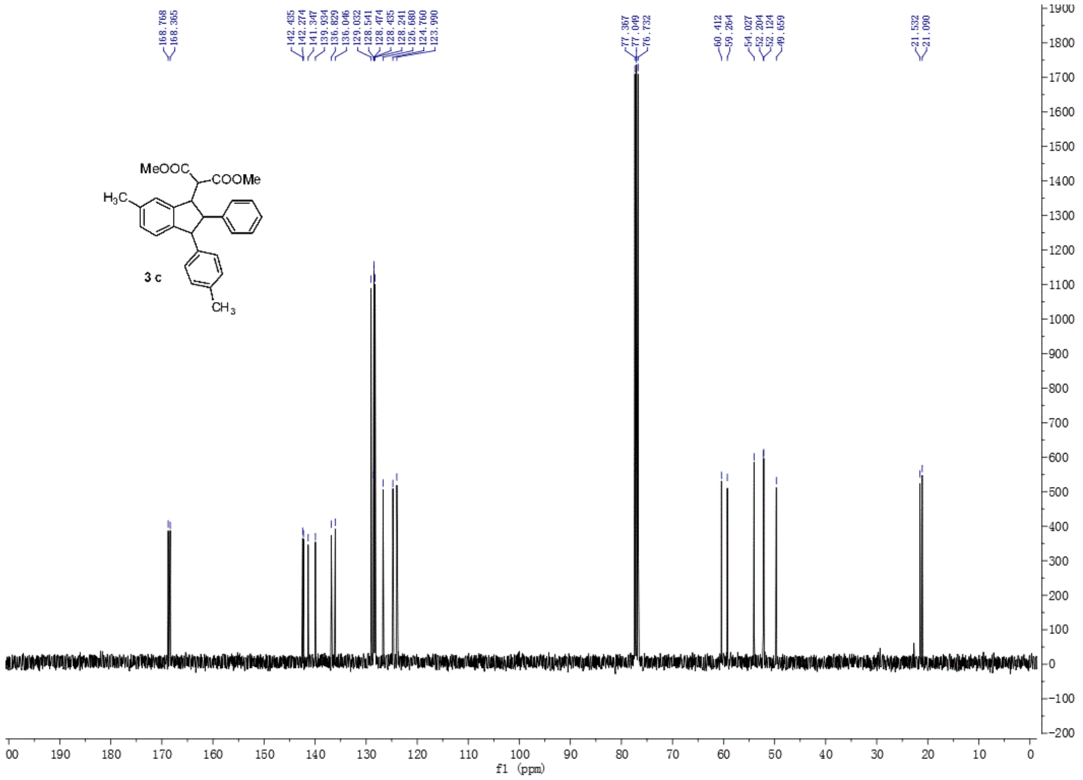
f1 (ppm)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CH3
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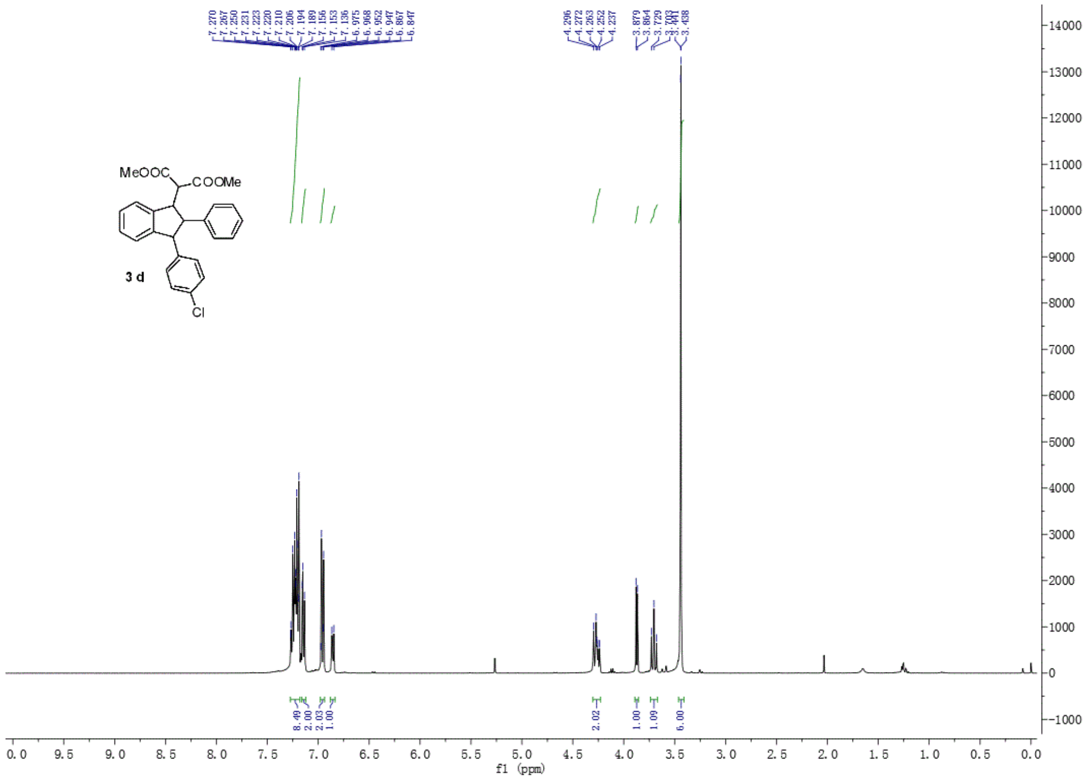
f1 (ppm)



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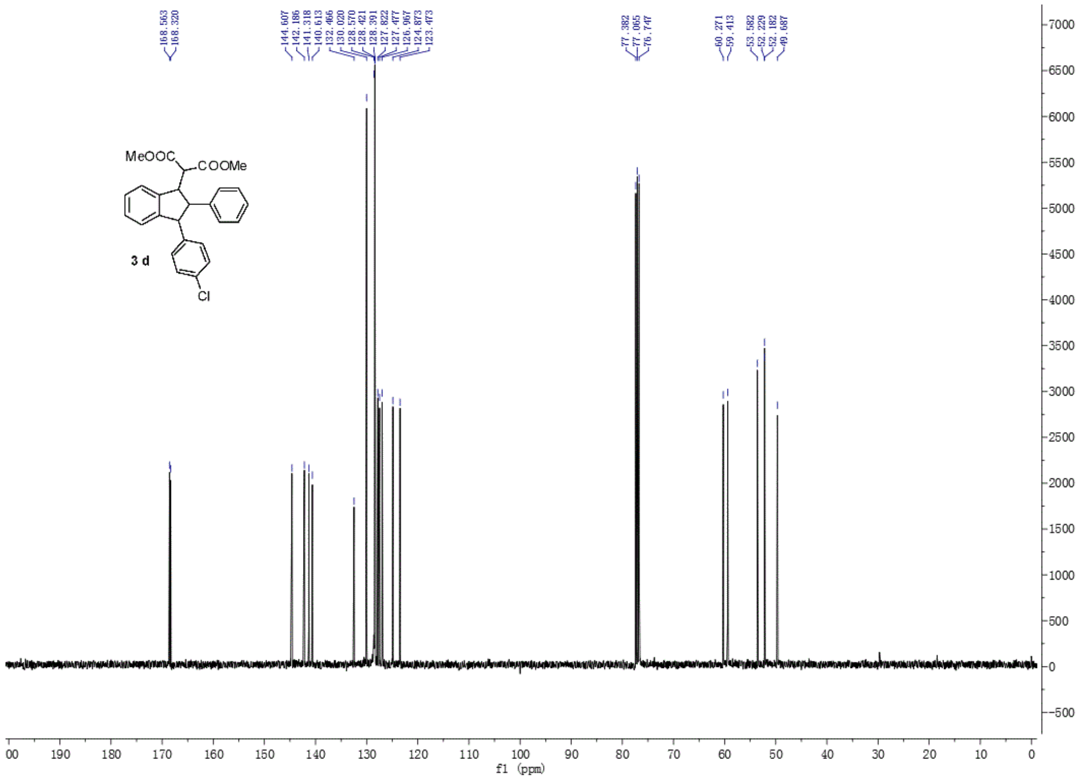

MeOOC
COOMe
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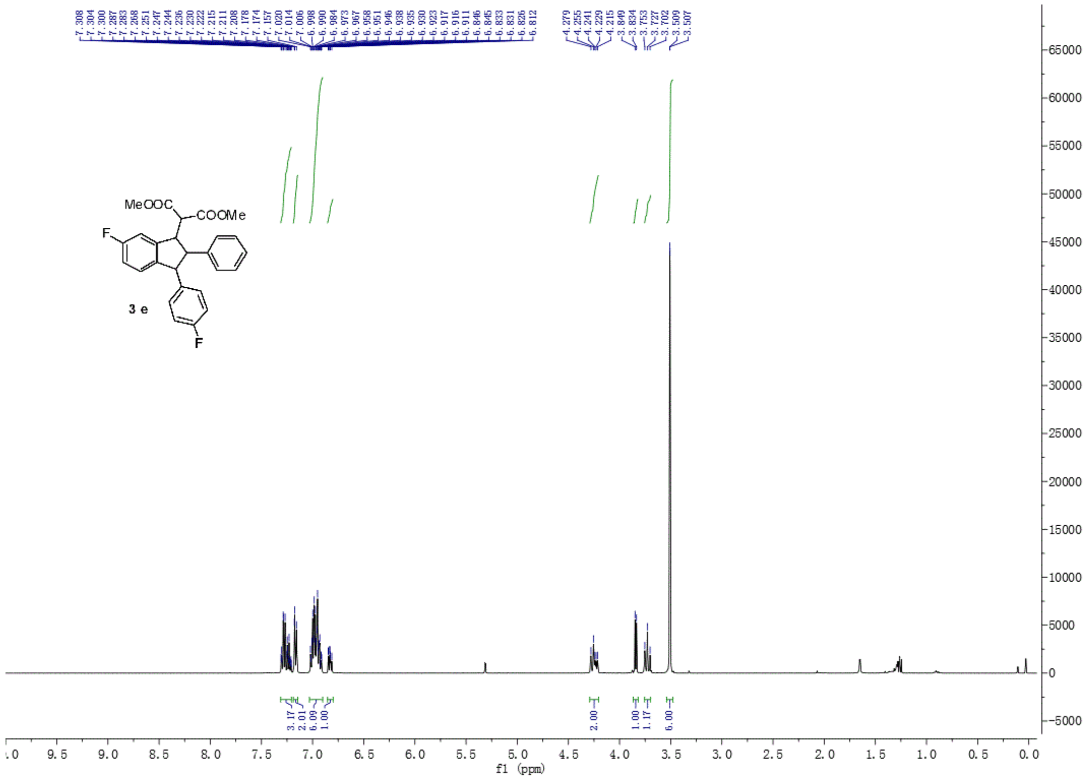
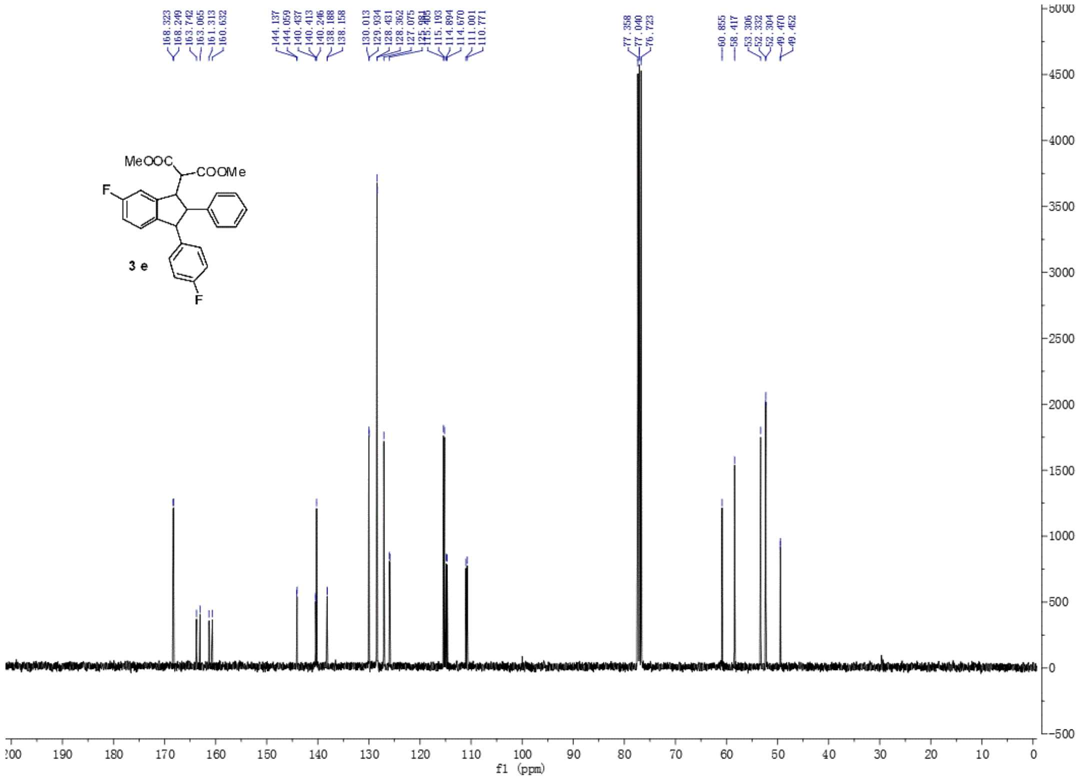
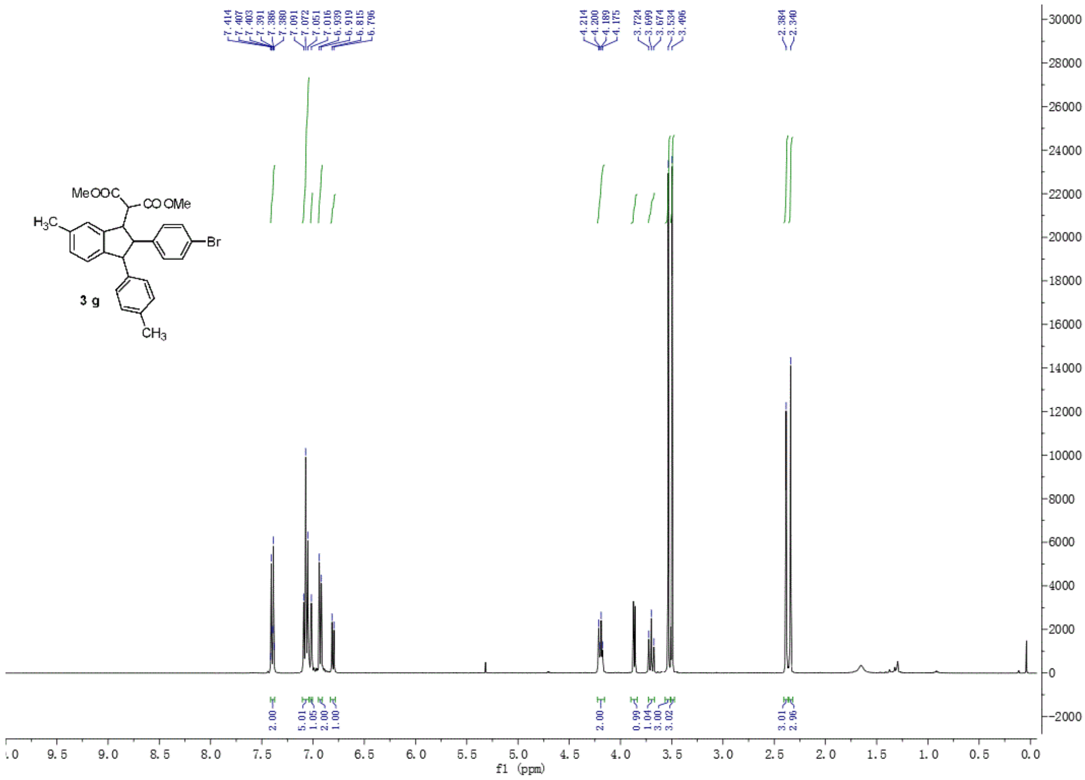
Cl
f1 (ppm)



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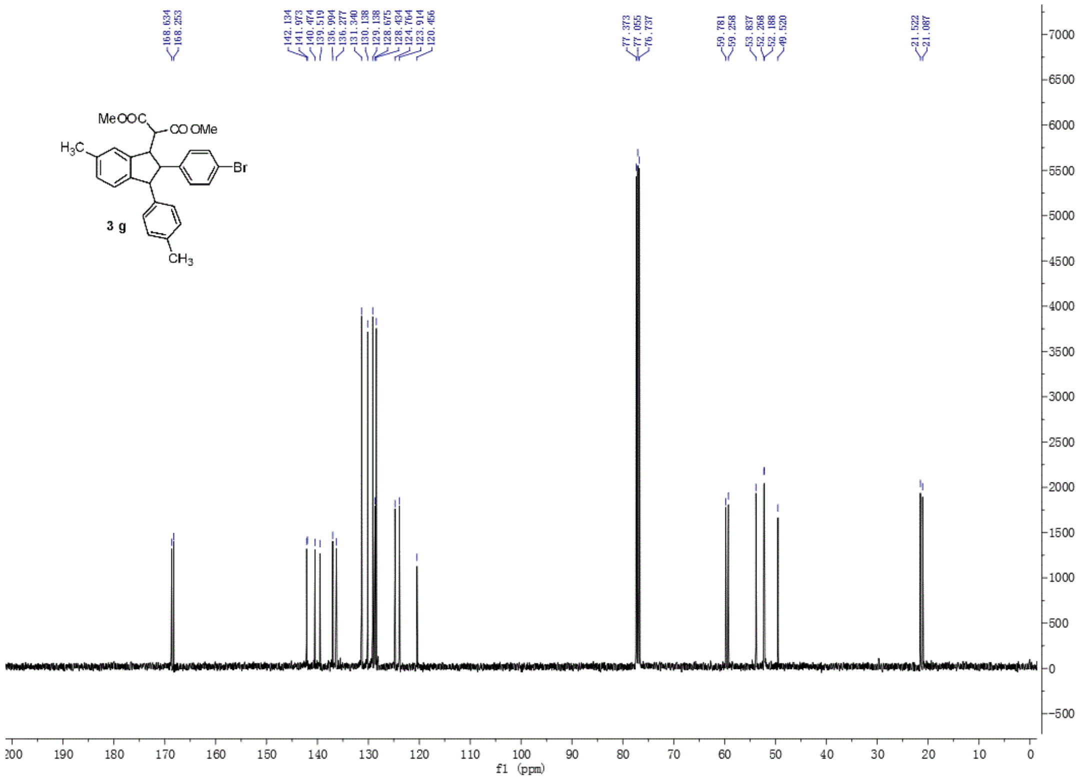
COOMe
H3C
Br
3 g
CH3
f1 (ppm)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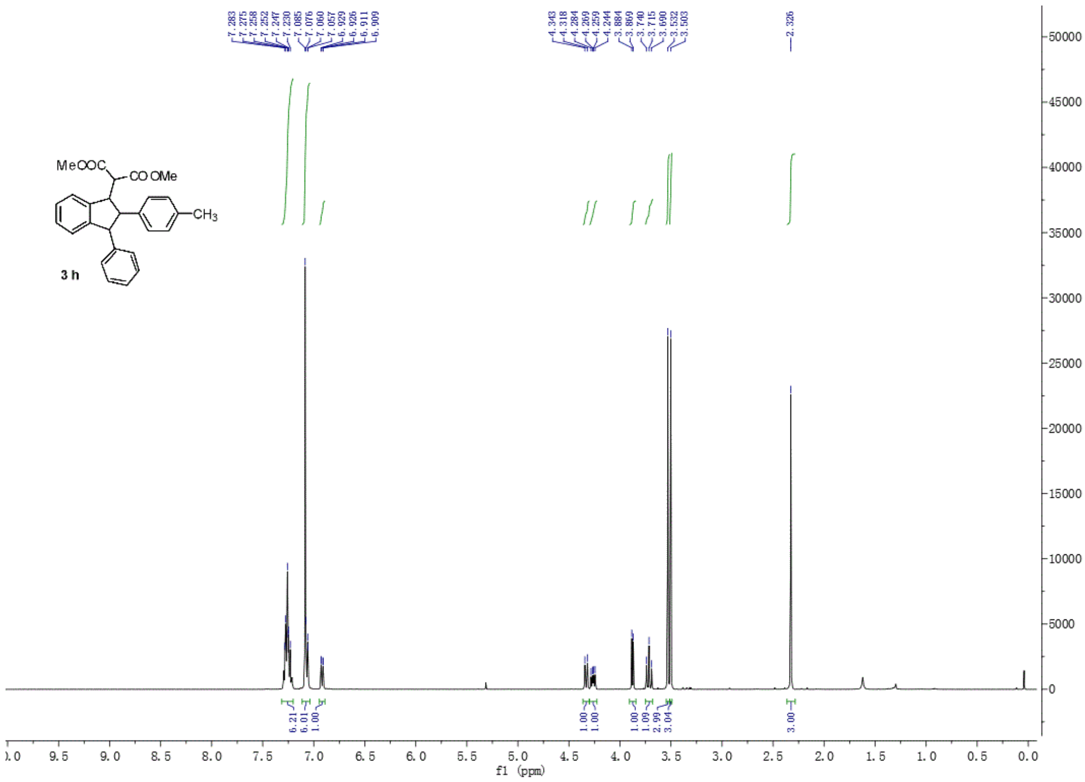
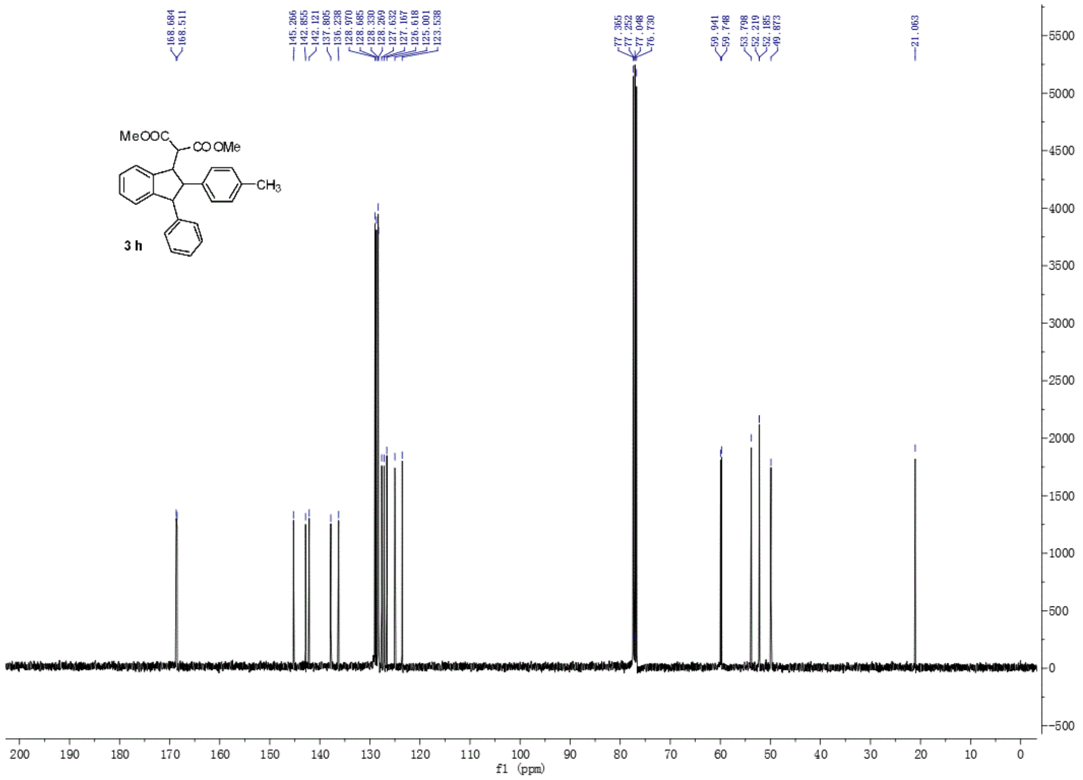
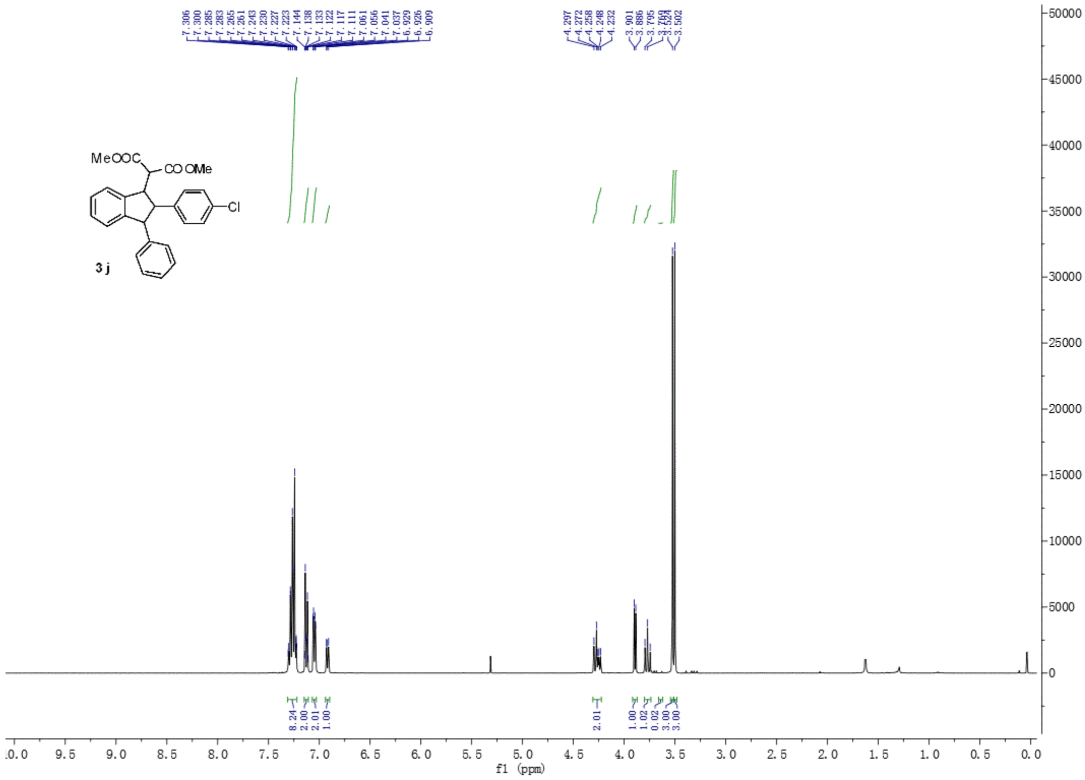
COOMe
H3C
Br
3 g
CH3
f1 (ppm)



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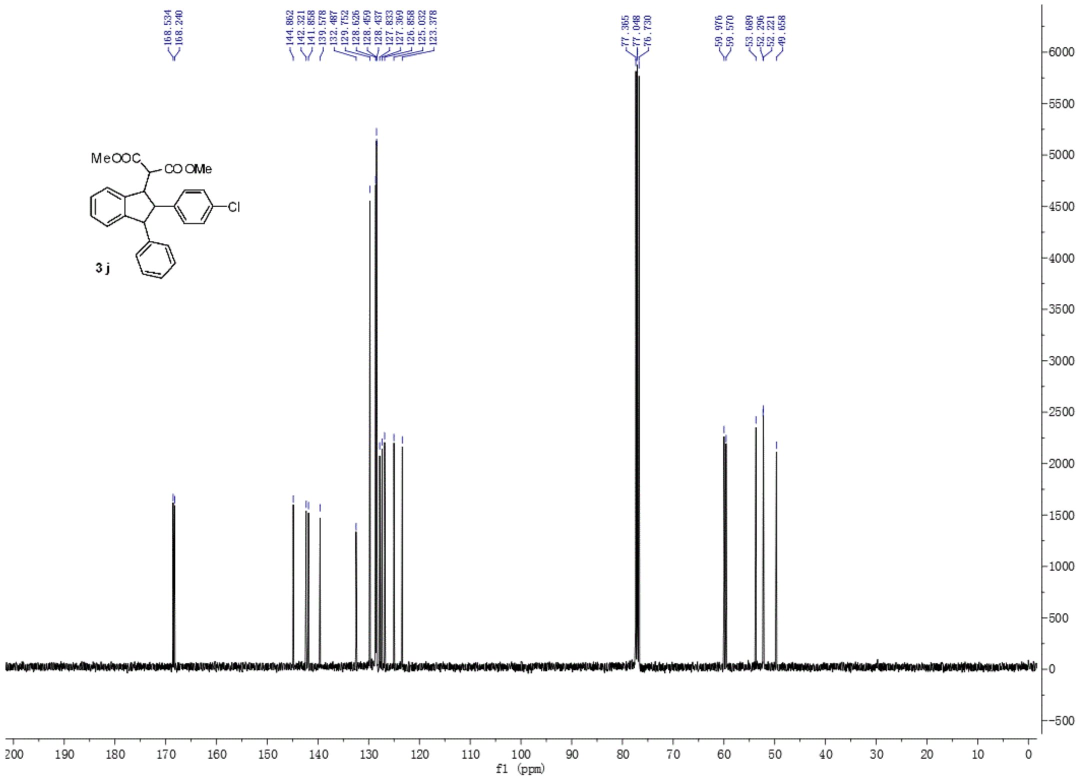
COOMe
Cl
3j
f1 (ppm)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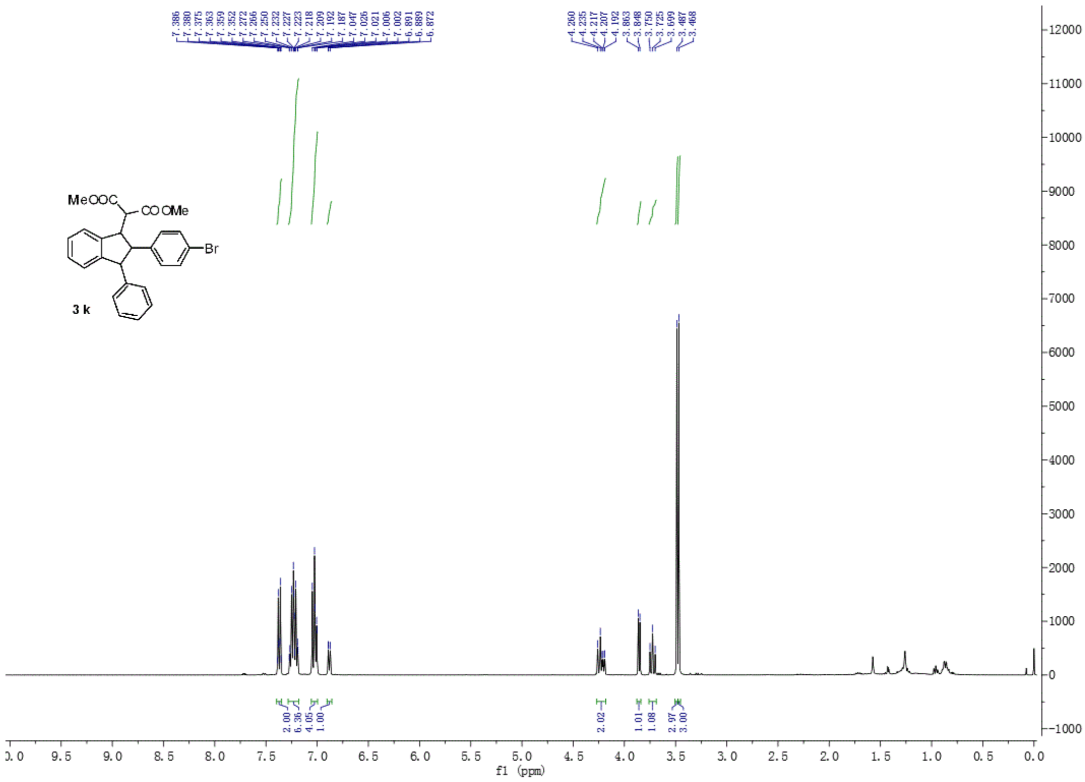
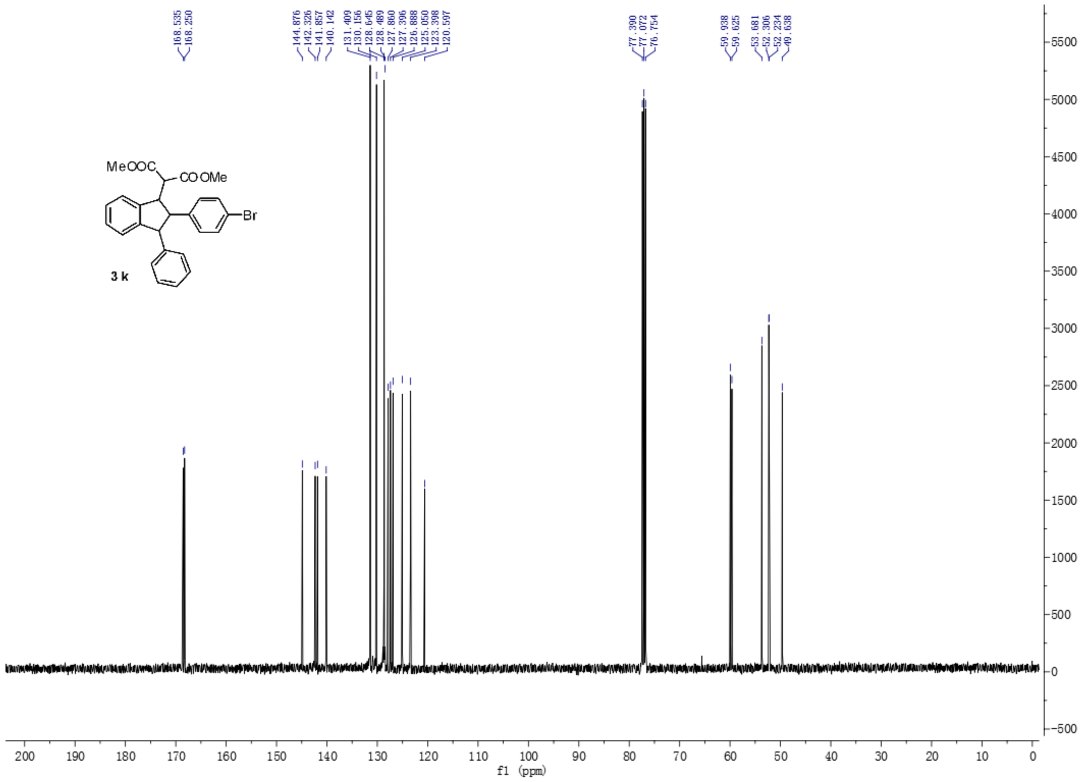
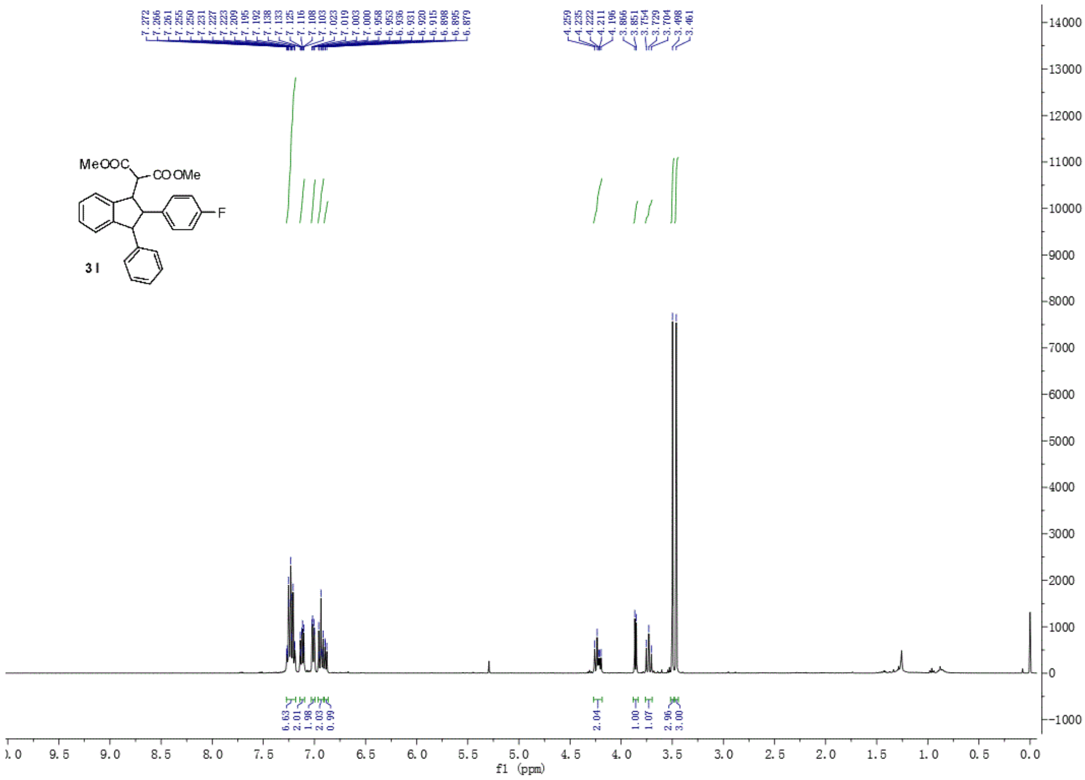
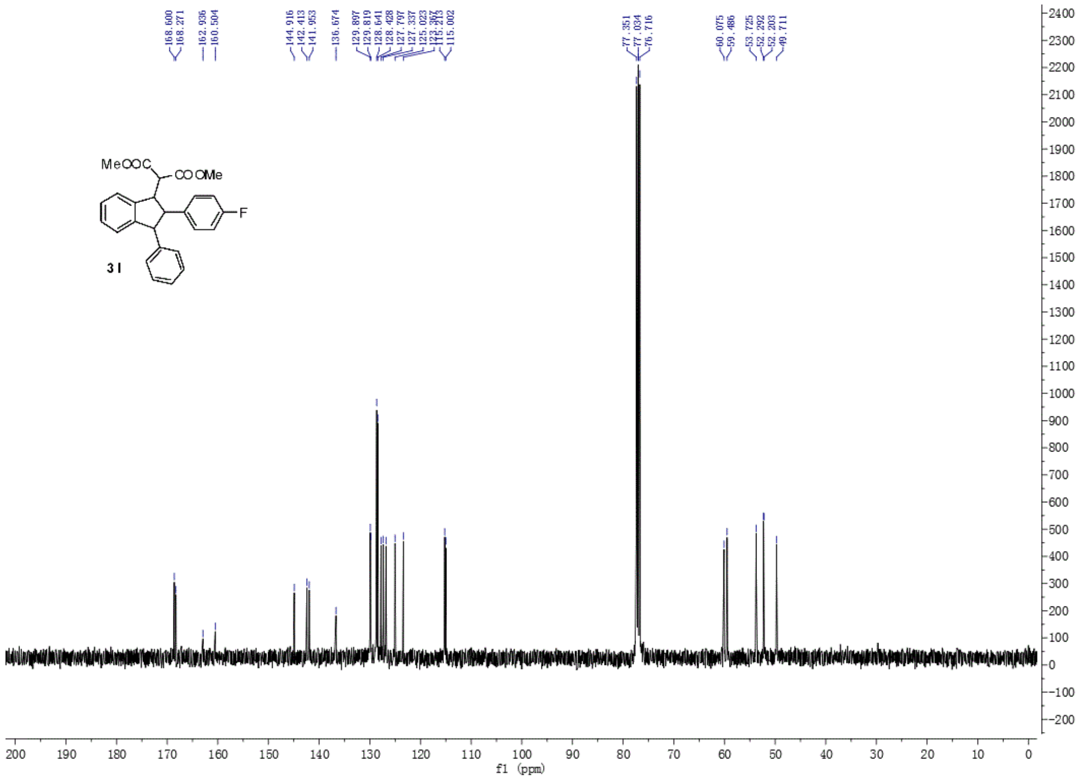
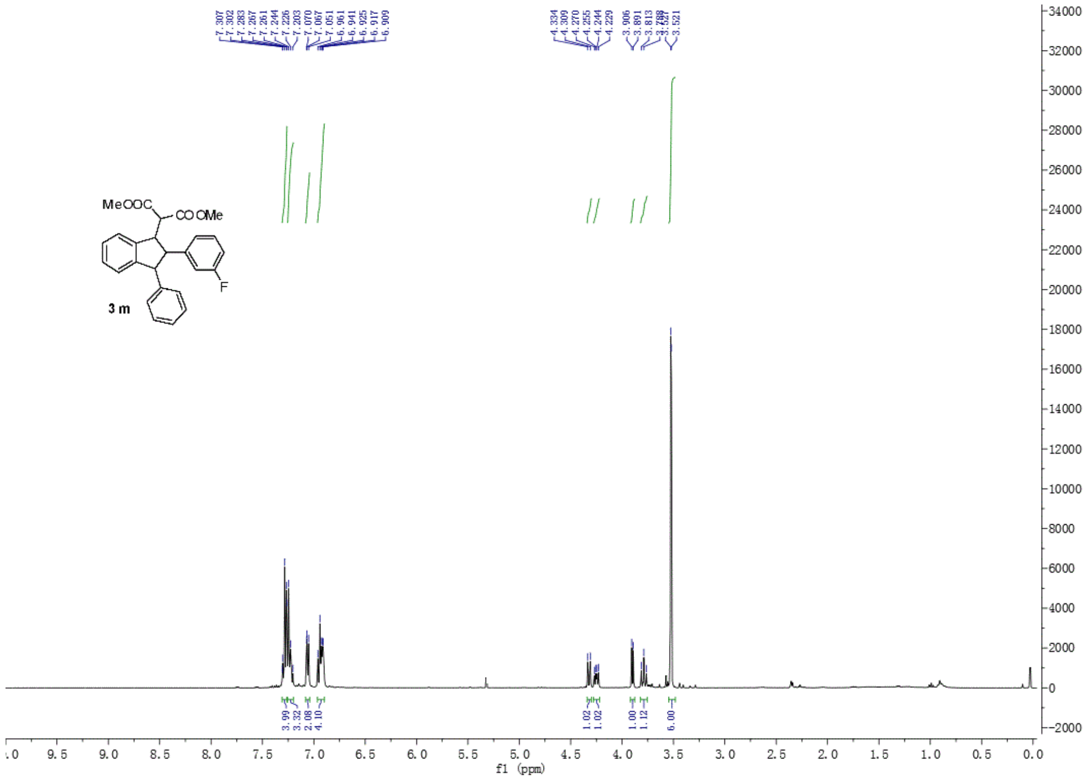
COOMe
Cl
3j
f1 (ppm)







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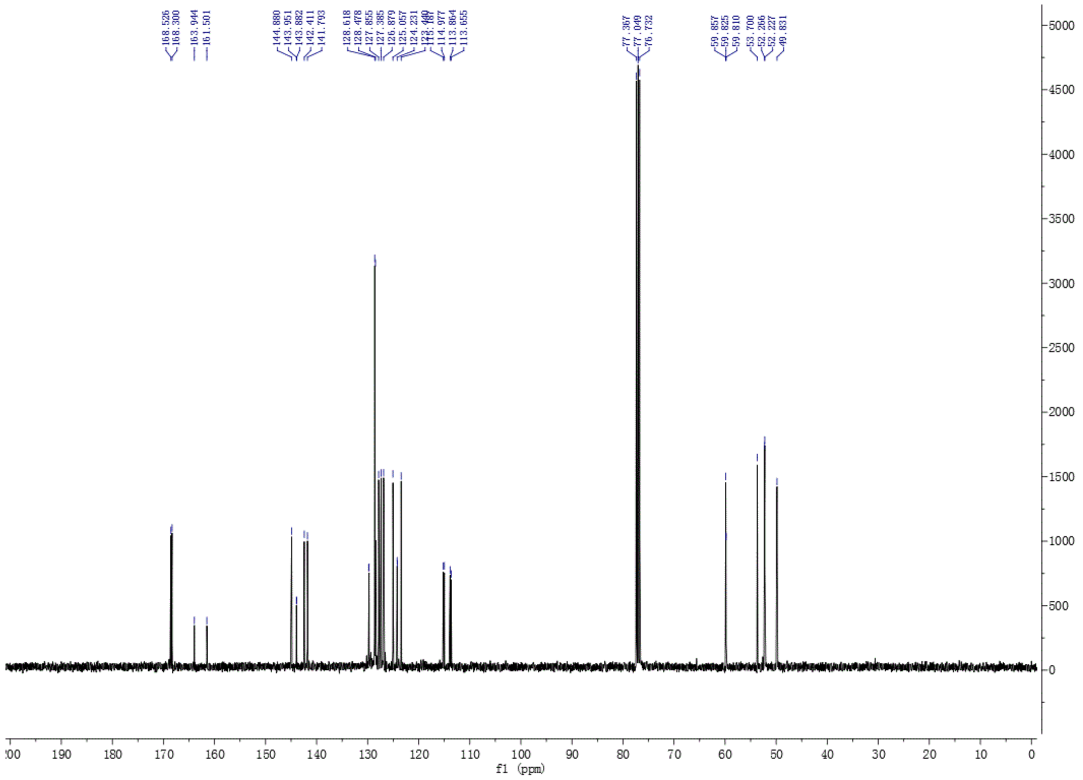
COOMe
F
3m
f1 (ppm)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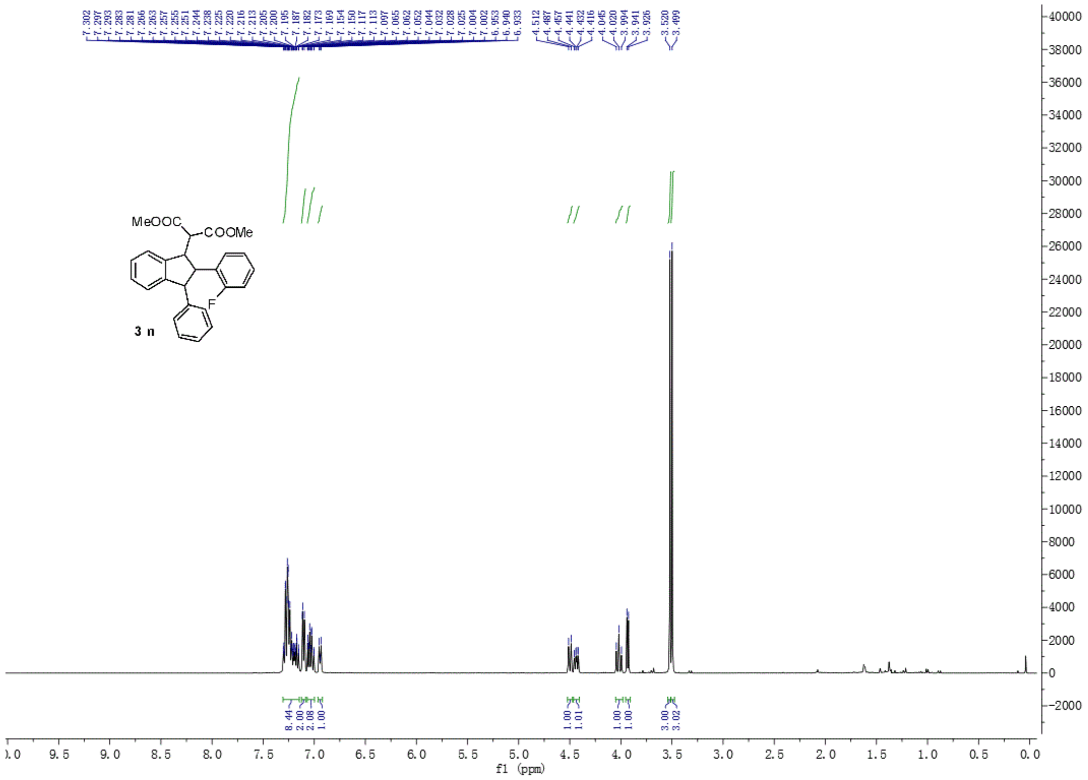

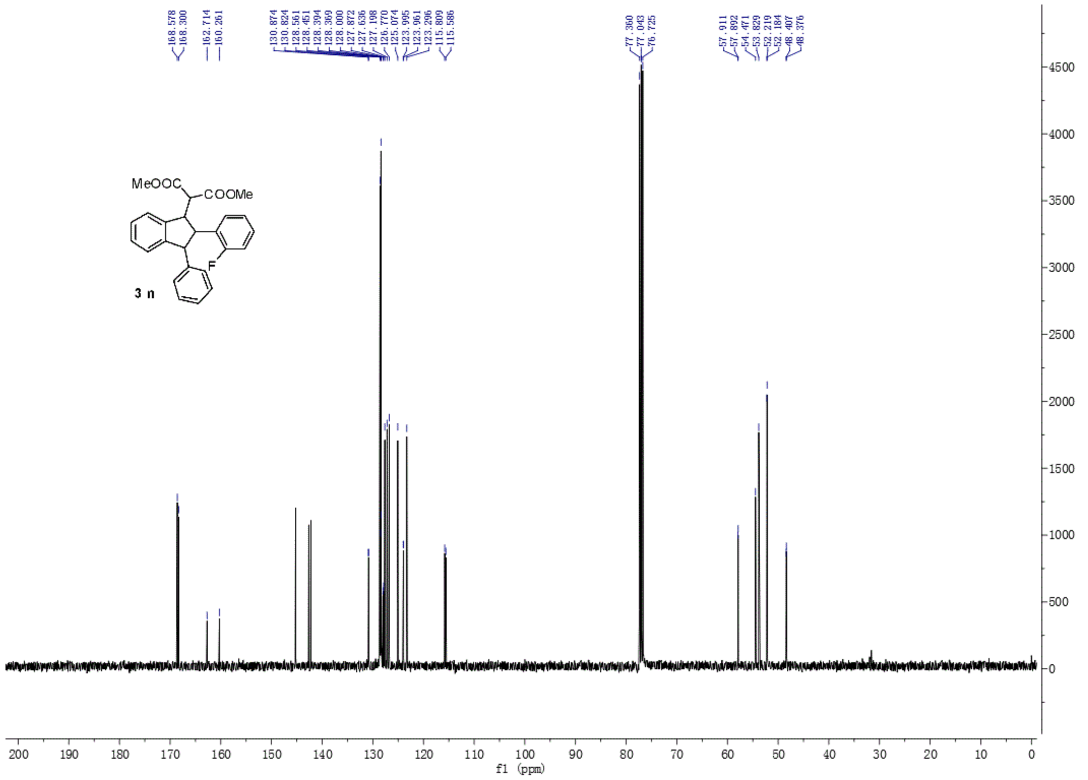

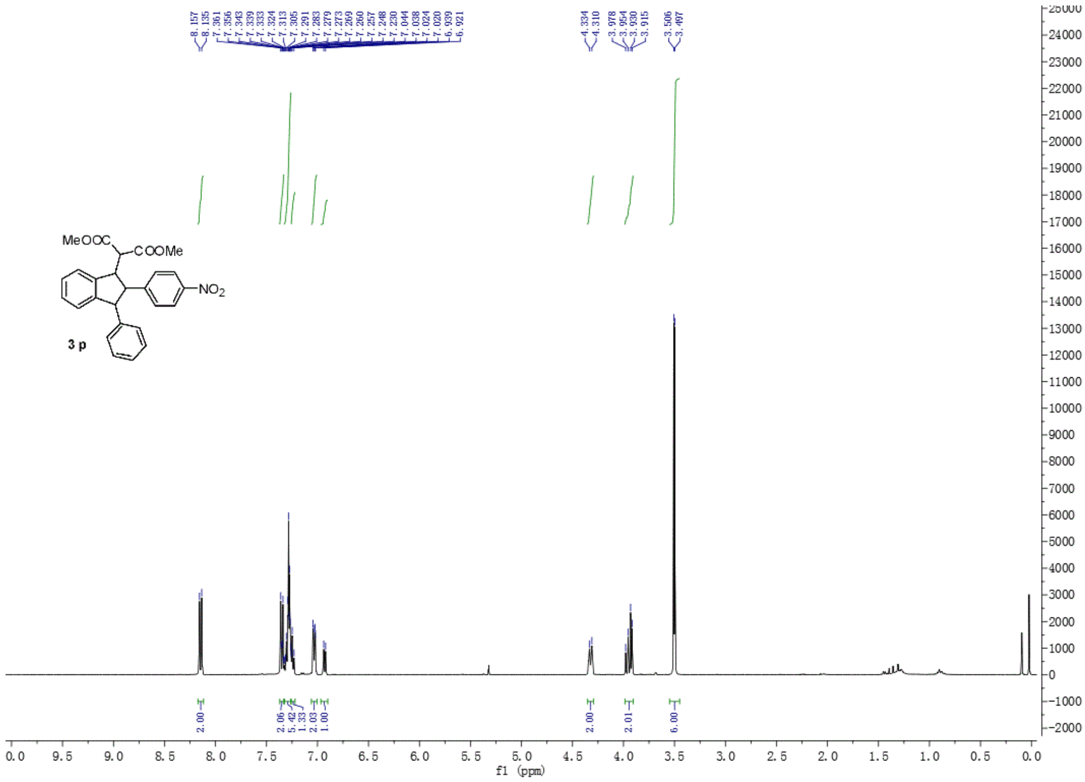
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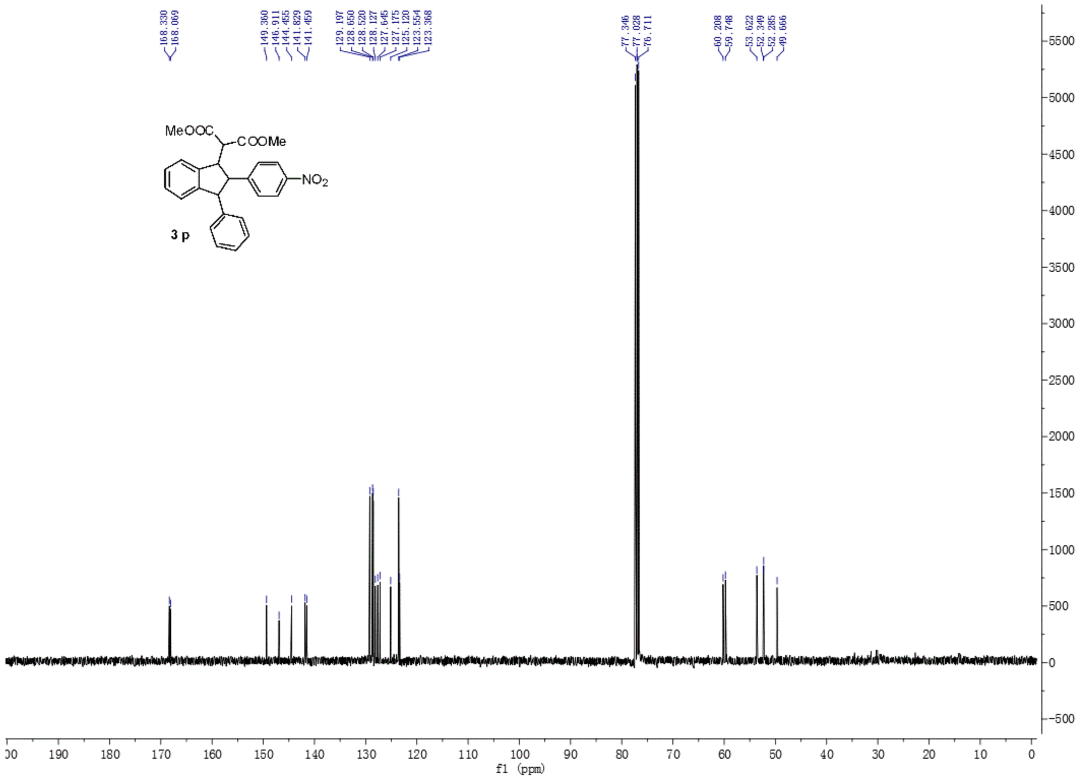
7.361
7.356
7.343
7.339
7.333
7.324
7.313
7.305
7.291
7.283
7.279
7.273
7.269
7.260
7.257
7.248
7.230
7.044
7.038
7.024
7.020
6.939
6.921
4.334
4.310
3.978
3.954
3.939
3.915
3.506
3.497
MeOOC
COOMe
NO2
3 p
2.00
2.06
5.42
1.33
2.03
1.00
2.00
2.01
6.00
f1 (ppm)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149.360
146.911
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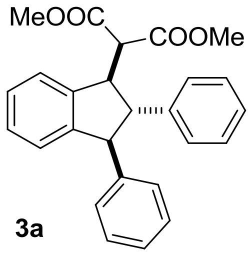
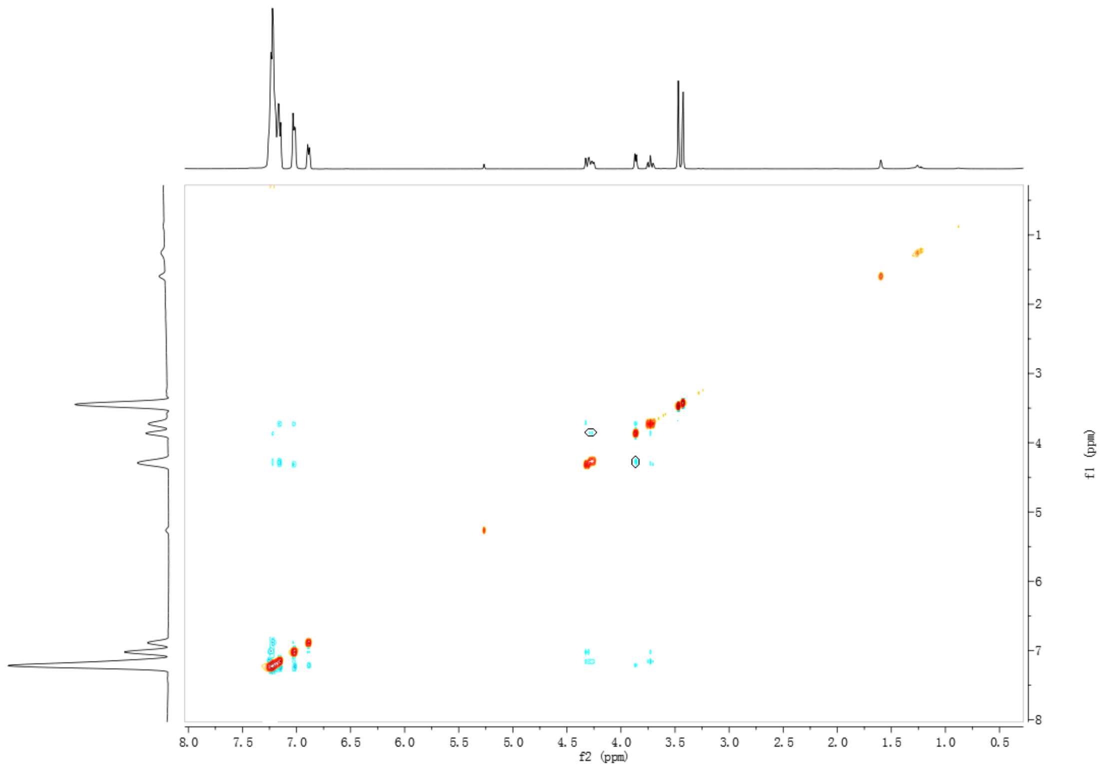
129.197
128.650
128.520
128.127
127.645
127.175
125.120
123.554
123.368
77.346
77.028
76.711
60.208
59.748
53.622
52.390
52.285
49.666
MeOOC
COOMe
NO2
3 p
f1 (ppm)

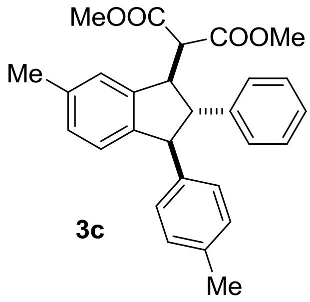
Copies of 2D NOESY Spectra



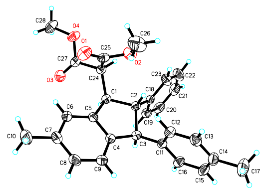
3a

2D NOESY (400 MHz, $CDCl_3$)

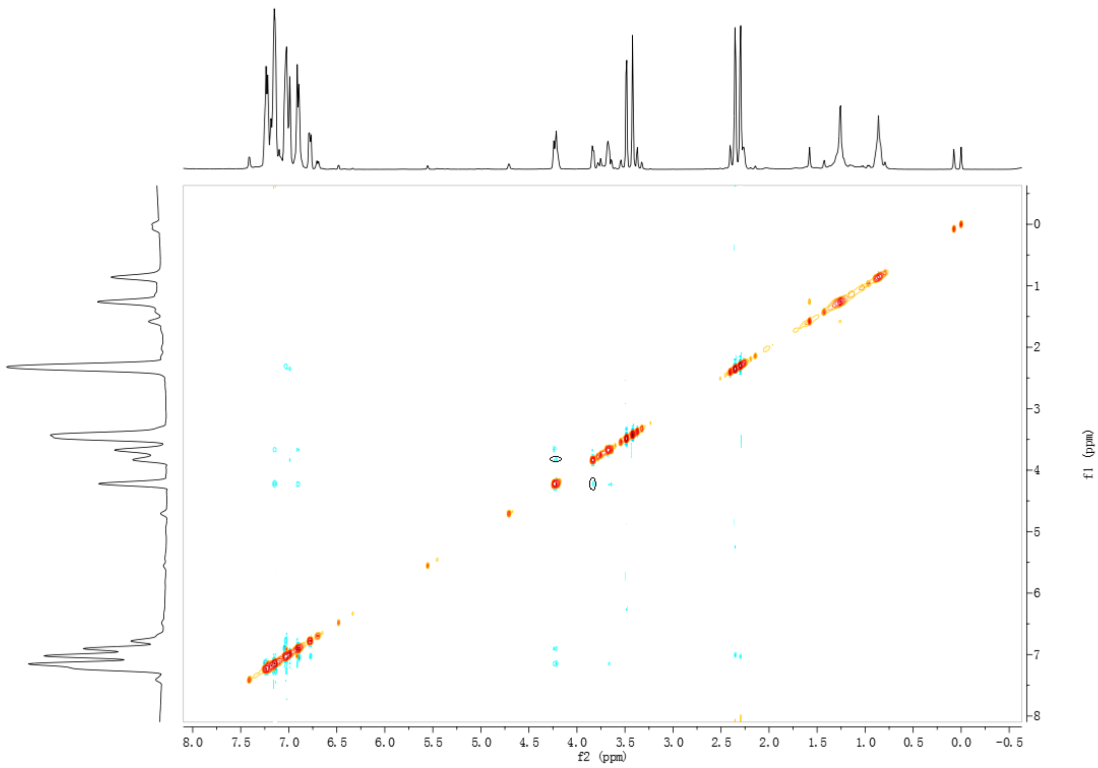


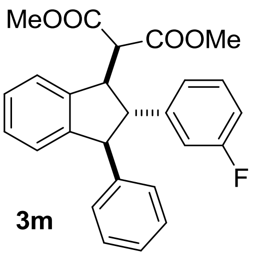


3c



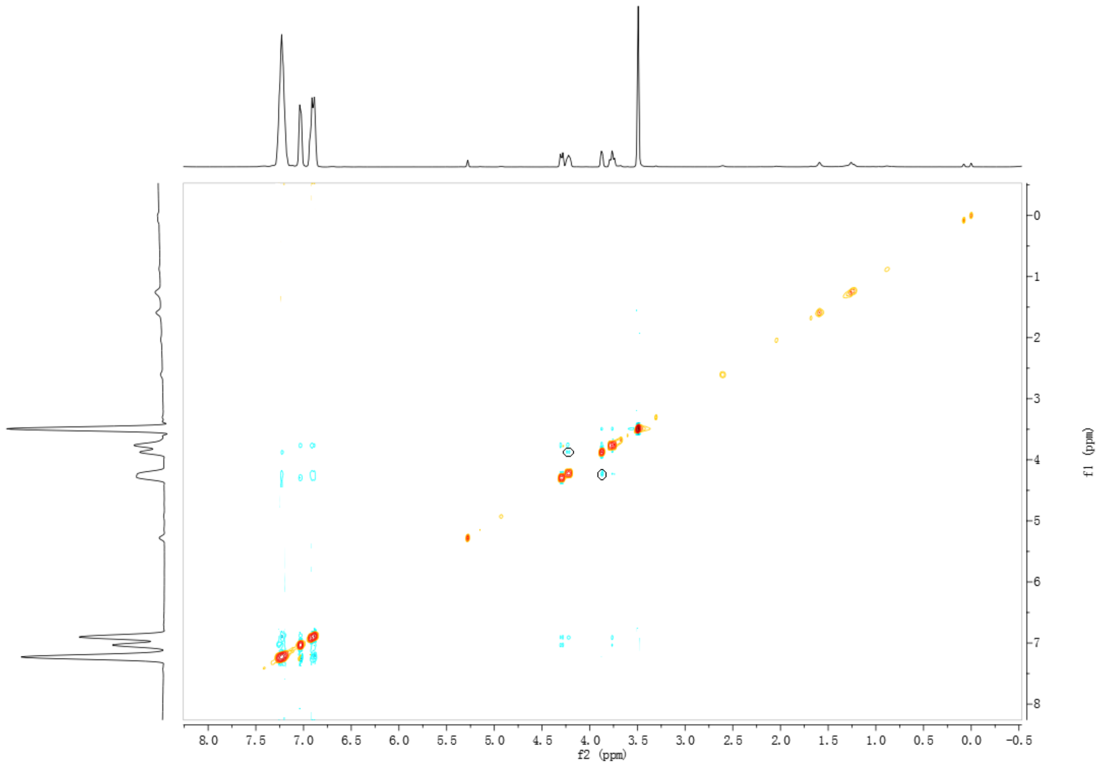
2D NOESY (400 MHz, $CDCl_3$)





3m

2D NOESY (400 MHz, $CDCl_3$)



Crystallographic data

The crystal structure has been deposited at the Cambridge Crystallographic Data Centre and allocated the deposition number : CCDC 855246.

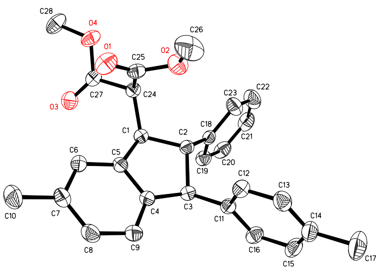


Table 1. Crystal data and structure refinement for 3c.

Identification code	3c	
Empirical formula	C28 H28 O4	
Formula weight	428.50	
Temperature	293(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Orthorhombic, Pbca	
Unit cell dimensions	a = 16.8408(10) A	alpha = 90 deg.
	b = 14.2476(8) A	beta = 90 deg.
	c = 19.6198(11) A	gamma = 90 deg.
Volume	4707.6(5) A^3	
Z, Calculated density	8, 1.209 Mg/m^3	
Absorption coefficient	0.080 mm^-1	
F(000)	1824	
Crystal size	0.256 x 0.201 x 0.164 mm	
Theta range for data collection	2.08 to 26.00 deg.	
Limiting indices	-20<=h<=20, -17<=k<=10, -24<=l<=24	
Reflections collected / unique	26921 / 4630 [R(int) = 0.0395]	
Completeness to theta = 26.00	100.0 %	
Absorption correction	Empirical	
Max. and min. transmission	1.00000 and 0.52456	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4630 / 0 / 293	
Goodness-of-fit on F^2	1.021	
Final R indices [I>2sigma(I)]	R1 = 0.0428, wR2 = 0.1118	
R indices (all data)	R1 = 0.0614, wR2 = 0.1248	
Largest diff. peak and hole	0.172 and -0.144 e.A^-3	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 3c. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	1933(1)	9099(1)	4384(1)	68(1)
O(2)	951(1)	9290(1)	3634(1)	67(1)
O(3)	3002(1)	7131(1)	3653(1)	66(1)
O(4)	1862(1)	6999(1)	4230(1)	59(1)
C(1)	2365(1)	8543(1)	2774(1)	38(1)
C(2)	1829(1)	9040(1)	2242(1)	39(1)
C(3)	2387(1)	9744(1)	1871(1)	41(1)
C(4)	3005(1)	9948(1)	2413(1)	42(1)
C(5)	2991(1)	9278(1)	2927(1)	41(1)
C(6)	3525(1)	9334(1)	3463(1)	49(1)
C(7)	4082(1)	10056(1)	3489(1)	55(1)
C(8)	4091(1)	10709(1)	2964(1)	59(1)
C(9)	3559(1)	10666(1)	2429(1)	54(1)
C(10)	4657(1)	10131(2)	4076(1)	77(1)
C(11)	1960(1)	10593(1)	1591(1)	41(1)
C(12)	1569(1)	11211(1)	2019(1)	49(1)
C(13)	1170(1)	11983(1)	1763(1)	58(1)
C(14)	1145(1)	12165(1)	1075(1)	63(1)
C(15)	1541(1)	11559(1)	648(1)	64(1)
C(16)	1945(1)	10783(1)	899(1)	52(1)
C(17)	698(2)	13009(2)	801(2)	109(1)
C(18)	1363(1)	8381(1)	1792(1)	41(1)
C(19)	1726(1)	7857(1)	1286(1)	51(1)
C(20)	1294(1)	7269(1)	867(1)	63(1)
C(21)	491(1)	7184(1)	957(1)	71(1)
C(22)	122(1)	7664(2)	1473(1)	77(1)
C(23)	554(1)	8268(1)	1883(1)	61(1)
C(24)	1889(1)	8130(1)	3376(1)	40(1)
C(25)	1608(1)	8881(1)	3869(1)	46(1)
C(26)	654(2)	10083(2)	4017(1)	100(1)
C(27)	2334(1)	7375(1)	3761(1)	45(1)
C(28)	2197(1)	6245(1)	4627(1)	73(1)

Table 3. Bond lengths [A] and angles [deg] for 3c.

O(1)-C(25)	1.1916(19)
O(2)-C(25)	1.333(2)
O(2)-C(26)	1.447(2)
O(3)-C(27)	1.1955(19)
O(4)-C(27)	1.3293(18)
O(4)-C(28)	1.442(2)
C(1)-C(5)	1.516(2)
C(1)-C(24)	1.5425(19)
C(1)-C(2)	1.5509(19)
C(1)-H(1)	0.9800
C(2)-C(18)	1.509(2)
C(2)-C(3)	1.5553(19)
C(2)-H(2)	0.9800
C(3)-C(11)	1.511(2)
C(3)-C(4)	1.515(2)
C(3)-H(3)	0.9800
C(4)-C(9)	1.385(2)
C(4)-C(5)	1.388(2)
C(5)-C(6)	1.388(2)
C(6)-C(7)	1.392(2)
C(6)-H(6)	0.9300
C(7)-C(8)	1.387(2)
C(7)-C(10)	1.508(2)
C(8)-C(9)	1.382(2)
C(8)-H(8)	0.9300
C(9)-H(9)	0.9300
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
C(11)-C(12)	1.384(2)
C(11)-C(16)	1.383(2)
C(12)-C(13)	1.384(2)
C(12)-H(12)	0.9300
C(13)-C(14)	1.374(3)
C(13)-H(13)	0.9300
C(14)-C(15)	1.376(3)
C(14)-C(17)	1.517(3)
C(15)-C(16)	1.388(2)
C(15)-H(15)	0.9300
C(16)-H(16)	0.9300
C(17)-H(17A)	0.9600
C(17)-H(17B)	0.9600

C(17)-H(17C)	0.9600
C(18)-C(23)	1.384(2)
C(18)-C(19)	1.384(2)
C(19)-C(20)	1.379(2)
C(19)-H(19)	0.9300
C(20)-C(21)	1.369(3)
C(20)-H(20)	0.9300
C(21)-C(22)	1.370(3)
C(21)-H(21)	0.9300
C(22)-C(23)	1.385(3)
C(22)-H(22)	0.9300
C(23)-H(23)	0.9300
C(24)-C(27)	1.513(2)
C(24)-C(25)	1.518(2)
C(24)-H(24)	0.9800
C(26)-H(26A)	0.9600
C(26)-H(26B)	0.9600
C(26)-H(26C)	0.9600
C(28)-H(28A)	0.9600
C(28)-H(28B)	0.9600
C(28)-H(28C)	0.9600
C(25)-O(2)-C(26)	116.66(17)
C(27)-O(4)-C(28)	116.13(14)
C(5)-C(1)-C(24)	118.28(11)
C(5)-C(1)-C(2)	102.81(11)
C(24)-C(1)-C(2)	112.78(11)
C(5)-C(1)-H(1)	107.5
C(24)-C(1)-H(1)	107.5
C(2)-C(1)-H(1)	107.5
C(18)-C(2)-C(1)	114.30(11)
C(18)-C(2)-C(3)	116.23(11)
C(1)-C(2)-C(3)	104.94(11)
C(18)-C(2)-H(2)	106.9
C(1)-C(2)-H(2)	106.9
C(3)-C(2)-H(2)	106.9
C(11)-C(3)-C(4)	115.43(12)
C(11)-C(3)-C(2)	113.48(11)
C(4)-C(3)-C(2)	102.15(11)
C(11)-C(3)-H(3)	108.5
C(4)-C(3)-H(3)	108.5
C(2)-C(3)-H(3)	108.5
C(9)-C(4)-C(5)	120.20(14)
C(9)-C(4)-C(3)	128.30(13)

C(5)-C(4)-C(3)	111.47(12)
C(6)-C(5)-C(4)	120.02(14)
C(6)-C(5)-C(1)	129.84(13)
C(4)-C(5)-C(1)	110.10(12)
C(5)-C(6)-C(7)	120.42(15)
C(5)-C(6)-H(6)	119.8
C(7)-C(6)-H(6)	119.8
C(8)-C(7)-C(6)	118.44(15)
C(8)-C(7)-C(10)	120.77(16)
C(6)-C(7)-C(10)	120.78(16)
C(9)-C(8)-C(7)	121.80(15)
C(9)-C(8)-H(8)	119.1
C(7)-C(8)-H(8)	119.1
C(8)-C(9)-C(4)	119.10(15)
C(8)-C(9)-H(9)	120.4
C(4)-C(9)-H(9)	120.4
C(7)-C(10)-H(10A)	109.5
C(7)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(7)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(12)-C(11)-C(16)	117.55(14)
C(12)-C(11)-C(3)	120.93(13)
C(16)-C(11)-C(3)	121.52(14)
C(11)-C(12)-C(13)	121.01(15)
C(11)-C(12)-H(12)	119.5
C(13)-C(12)-H(12)	119.5
C(14)-C(13)-C(12)	121.46(17)
C(14)-C(13)-H(13)	119.3
C(12)-C(13)-H(13)	119.3
C(13)-C(14)-C(15)	117.73(16)
C(13)-C(14)-C(17)	120.8(2)
C(15)-C(14)-C(17)	121.45(19)
C(14)-C(15)-C(16)	121.31(16)
C(14)-C(15)-H(15)	119.3
C(16)-C(15)-H(15)	119.3
C(11)-C(16)-C(15)	120.93(16)
C(11)-C(16)-H(16)	119.5
C(15)-C(16)-H(16)	119.5
C(14)-C(17)-H(17A)	109.5
C(14)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(14)-C(17)-H(17C)	109.5

H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(23)-C(18)-C(19)	117.61(15)
C(23)-C(18)-C(2)	120.60(14)
C(19)-C(18)-C(2)	121.78(13)
C(20)-C(19)-C(18)	121.47(17)
C(20)-C(19)-H(19)	119.3
C(18)-C(19)-H(19)	119.3
C(21)-C(20)-C(19)	119.86(18)
C(21)-C(20)-H(20)	120.1
C(19)-C(20)-H(20)	120.1
C(20)-C(21)-C(22)	119.92(17)
C(20)-C(21)-H(21)	120.0
C(22)-C(21)-H(21)	120.0
C(21)-C(22)-C(23)	120.03(18)
C(21)-C(22)-H(22)	120.0
C(23)-C(22)-H(22)	120.0
C(18)-C(23)-C(22)	121.02(18)
C(18)-C(23)-H(23)	119.5
C(22)-C(23)-H(23)	119.5
C(27)-C(24)-C(25)	109.75(12)
C(27)-C(24)-C(1)	113.32(12)
C(25)-C(24)-C(1)	112.35(11)
C(27)-C(24)-H(24)	107.0
C(25)-C(24)-H(24)	107.0
C(1)-C(24)-H(24)	107.0
O(1)-C(25)-O(2)	124.14(15)
O(1)-C(25)-C(24)	125.53(15)
O(2)-C(25)-C(24)	110.30(13)
O(2)-C(26)-H(26A)	109.5
O(2)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
O(2)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
O(3)-C(27)-O(4)	124.51(15)
O(3)-C(27)-C(24)	125.83(14)
O(4)-C(27)-C(24)	109.64(14)
O(4)-C(28)-H(28A)	109.5
O(4)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
O(4)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 3c. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	96(1)	57(1)	51(1)	-13(1)	-2(1)	-3(1)
O(2)	65(1)	63(1)	74(1)	-9(1)	11(1)	20(1)
O(3)	59(1)	63(1)	76(1)	17(1)	0(1)	14(1)
O(4)	81(1)	43(1)	53(1)	13(1)	10(1)	5(1)
C(1)	40(1)	37(1)	38(1)	0(1)	1(1)	1(1)
C(2)	40(1)	38(1)	39(1)	2(1)	1(1)	0(1)
C(3)	44(1)	40(1)	39(1)	1(1)	2(1)	-2(1)
C(4)	38(1)	44(1)	45(1)	4(1)	2(1)	-1(1)
C(5)	38(1)	43(1)	44(1)	1(1)	1(1)	0(1)
C(6)	46(1)	52(1)	49(1)	6(1)	-5(1)	-3(1)
C(7)	43(1)	59(1)	62(1)	-3(1)	-7(1)	-4(1)
C(8)	47(1)	55(1)	74(1)	4(1)	-5(1)	-13(1)
C(9)	48(1)	50(1)	62(1)	11(1)	0(1)	-8(1)
C(10)	64(1)	86(2)	81(1)	-2(1)	-24(1)	-17(1)
C(11)	43(1)	39(1)	40(1)	2(1)	-4(1)	-6(1)
C(12)	54(1)	50(1)	44(1)	2(1)	-1(1)	1(1)
C(13)	50(1)	50(1)	75(1)	1(1)	-1(1)	4(1)
C(14)	56(1)	56(1)	78(1)	17(1)	-16(1)	-1(1)
C(15)	76(1)	66(1)	49(1)	17(1)	-19(1)	-10(1)
C(16)	64(1)	51(1)	41(1)	1(1)	-4(1)	-6(1)
C(17)	102(2)	91(2)	133(2)	39(2)	-26(2)	27(2)
C(18)	44(1)	39(1)	41(1)	8(1)	-6(1)	-1(1)
C(19)	57(1)	52(1)	44(1)	0(1)	-3(1)	-6(1)
C(20)	89(1)	53(1)	47(1)	0(1)	-9(1)	-12(1)
C(21)	87(2)	58(1)	67(1)	8(1)	-32(1)	-24(1)
C(22)	53(1)	76(1)	104(2)	5(1)	-18(1)	-16(1)
C(23)	46(1)	61(1)	77(1)	-2(1)	-5(1)	-3(1)
C(24)	45(1)	35(1)	40(1)	1(1)	-1(1)	-2(1)
C(25)	55(1)	37(1)	46(1)	3(1)	10(1)	-3(1)
C(26)	99(2)	71(1)	132(2)	-23(1)	37(2)	27(1)
C(27)	58(1)	37(1)	41(1)	-1(1)	-2(1)	-2(1)
C(28)	114(2)	47(1)	59(1)	16(1)	-3(1)	9(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 3c.

	x	y	z	U(eq)
H(1)	2629	8020	2543	46
H(2)	1441	9414	2498	47
H(3)	2646	9417	1493	49
H(6)	3512	8887	3808	59
H(8)	4465	11189	2973	70
H(9)	3572	11114	2084	64
H(10A)	5185	10005	3917	116
H(10B)	4516	9683	4421	116
H(10C)	4634	10753	4263	116
H(12)	1575	11106	2487	59
H(13)	913	12387	2062	70
H(15)	1539	11671	181	76
H(16)	2208	10386	599	62
H(17A)	797	13542	1088	163
H(17B)	139	12875	796	163
H(17C)	875	13143	346	163
H(19)	2272	7901	1227	61
H(20)	1548	6932	525	75
H(21)	197	6802	668	85
H(22)	-419	7583	1548	93
H(23)	296	8604	2224	73
H(24)	1414	7835	3183	48
H(26A)	544	9892	4477	151
H(26B)	175	10311	3809	151
H(26C)	1045	10573	4020	151
H(28A)	2327	5729	4332	110
H(28B)	1819	6043	4962	110
H(28C)	2670	6461	4851	110

Table 6. Torsion angles [deg] for 3c.

C(5)-C(1)-C(2)-C(18)	156.62(12)
C(24)-C(1)-C(2)-C(18)	-74.89(15)
C(5)-C(1)-C(2)-C(3)	28.13(14)
C(24)-C(1)-C(2)-C(3)	156.62(11)
C(18)-C(2)-C(3)-C(11)	80.31(15)
C(1)-C(2)-C(3)-C(11)	-152.37(11)
C(18)-C(2)-C(3)-C(4)	-154.76(12)
C(1)-C(2)-C(3)-C(4)	-27.44(14)
C(11)-C(3)-C(4)-C(9)	-41.8(2)
C(2)-C(3)-C(4)-C(9)	-165.42(15)
C(11)-C(3)-C(4)-C(5)	140.58(13)
C(2)-C(3)-C(4)-C(5)	16.96(15)
C(9)-C(4)-C(5)-C(6)	0.9(2)
C(3)-C(4)-C(5)-C(6)	178.72(13)
C(9)-C(4)-C(5)-C(1)	-176.98(14)
C(3)-C(4)-C(5)-C(1)	0.87(16)
C(24)-C(1)-C(5)-C(6)	39.1(2)
C(2)-C(1)-C(5)-C(6)	164.02(15)
C(24)-C(1)-C(5)-C(4)	-143.37(13)
C(2)-C(1)-C(5)-C(4)	-18.40(15)
C(4)-C(5)-C(6)-C(7)	-0.5(2)
C(1)-C(5)-C(6)-C(7)	176.92(15)
C(5)-C(6)-C(7)-C(8)	-0.4(2)
C(5)-C(6)-C(7)-C(10)	179.00(17)
C(6)-C(7)-C(8)-C(9)	0.9(3)
C(10)-C(7)-C(8)-C(9)	-178.55(18)
C(7)-C(8)-C(9)-C(4)	-0.4(3)
C(5)-C(4)-C(9)-C(8)	-0.4(2)
C(3)-C(4)-C(9)-C(8)	-177.88(15)
C(4)-C(3)-C(11)-C(12)	-55.17(18)
C(2)-C(3)-C(11)-C(12)	62.26(17)
C(4)-C(3)-C(11)-C(16)	124.70(15)
C(2)-C(3)-C(11)-C(16)	-117.87(15)
C(16)-C(11)-C(12)-C(13)	0.8(2)
C(3)-C(11)-C(12)-C(13)	-179.28(14)
C(11)-C(12)-C(13)-C(14)	0.1(2)
C(12)-C(13)-C(14)-C(15)	-0.9(3)
C(12)-C(13)-C(14)-C(17)	179.41(19)
C(13)-C(14)-C(15)-C(16)	0.8(3)
C(17)-C(14)-C(15)-C(16)	-179.51(19)
C(12)-C(11)-C(16)-C(15)	-0.9(2)
C(3)-C(11)-C(16)-C(15)	179.19(14)

C(14)-C(15)-C(16)-C(11)	0.1(3)
C(1)-C(2)-C(18)-C(23)	106.62(16)
C(3)-C(2)-C(18)-C(23)	-130.84(15)
C(1)-C(2)-C(18)-C(19)	-72.22(17)
C(3)-C(2)-C(18)-C(19)	50.32(18)
C(23)-C(18)-C(19)-C(20)	2.5(2)
C(2)-C(18)-C(19)-C(20)	-178.65(14)
C(18)-C(19)-C(20)-C(21)	-1.2(3)
C(19)-C(20)-C(21)-C(22)	-1.7(3)
C(20)-C(21)-C(22)-C(23)	3.1(3)
C(19)-C(18)-C(23)-C(22)	-1.0(2)
C(2)-C(18)-C(23)-C(22)	-179.91(16)
C(21)-C(22)-C(23)-C(18)	-1.7(3)
C(5)-C(1)-C(24)-C(27)	-80.09(16)
C(2)-C(1)-C(24)-C(27)	159.99(12)
C(5)-C(1)-C(24)-C(25)	45.02(17)
C(2)-C(1)-C(24)-C(25)	-74.91(15)
C(26)-O(2)-C(25)-O(1)	4.0(2)
C(26)-O(2)-C(25)-C(24)	-174.29(15)
C(27)-C(24)-C(25)-O(1)	30.1(2)
C(1)-C(24)-C(25)-O(1)	-96.96(17)
C(27)-C(24)-C(25)-O(2)	-151.70(13)
C(1)-C(24)-C(25)-O(2)	81.26(15)
C(28)-O(4)-C(27)-O(3)	-0.1(2)
C(28)-O(4)-C(27)-C(24)	178.21(13)
C(25)-C(24)-C(27)-O(3)	-122.77(17)
C(1)-C(24)-C(27)-O(3)	3.7(2)
C(25)-C(24)-C(27)-O(4)	58.96(16)
C(1)-C(24)-C(27)-O(4)	-174.55(12)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for 3c [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)