A facile copper(I)-catalyzed homo-coupling of indanone derivatives using diaziridinone under mild conditions

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Supporting Information

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General methods. All commercially available reagents were used without further purification. Column chromatography was performed on silica gel (200-400 mesh). ^{1}H NMR spectra were recorded on a 300 or 400 MHz NMR spectrometer and ^{13}C NMR spectra were recorded on a 75 or 100 MHz NMR spectrometer. IR spectra were recorded on a FT-IR spectrometer. Melting points were uncorrected.

Di-*t*-butyldiaziridinone (**1**) was prepared according to the reported procedures.[1] Methyl 1-oxo-2-indane-carboxylates **2a**-**2i** were prepared from corresponding 1-indanones and dimethyl carbonate based on the reported procedures.[2] 2-Phenyl-1*H*-indene-1,3(2*H*)-dione (**2k**) and ethyl 3-oxo-3-phenylpropanoate (**5**) were purchased from commercial suppliers. Diethyl 2,3-dibenzoylsuccinate (**7**) was prepared according to the reported procedures.[3]

(1) H. Du, B. Zhao and Y. Shi, *Org. Synth.*, 2009, **86**, 315.
(2) K. Van Emelen, T. De Wit, G. J. Hoornaert and F. Compernolle, *Org. Lett.*, 2000, **2**, 3083.
(3) A. Wu, Y. Zhao, N. Chen and X. Pan, *Synth. Commun.*, 1997, **27**, 331.

Representative procedure for the homo-coupling of 1,3-dicarbonyl compounds (Table 2, entry 1). To a 1.5 mL vial equipped with a stir bar were successively added methyl 1-oxo-2-indane-carboxylate (**2a**) (0.0951 g, 0.50 mmol), CuCl (0.0025 g, 0.025 mmol), P(*n*-Bu)$_3$ (0.0051 g, 0.025 mmol), and toluene (0.8 mL). The mixture was stirred at room temperature for 10 minutes, followed by the addition of di-*t*-butyldiaziridinone (**1**) (0.1022 g, 0.60 mmol). The reaction mixture was vigorously stirred at room temperature for 24 h, concentrated, and purified by flash chromatography (silica gel, CH_2Cl_2:MeOH = 400:1) to give coupling products **3a** as white solid (major, 0.0851 g, 90%) and **3a'** as colorless oil (minor, 0.0038 g, 4%).

Procedure for Scheme 3. To a 4 mL vial equipped with a stir bar were successively added methyl 1-oxo-2-indane-carboxylate (**2a**) (0.0951 g, 0.50 mmol), CuCl (0.0025 g, 0.025 mmol), P(*n*-Bu)$_3$ (0.0051 g, 0.025 mmol), and toluene (0.8 mL). After the mixture was stirred at room temperature for 10 minutes, di-*t*-butyldiaziridinone (**1**) (0.1022 g, 0.60 mmol) was added, immediately followed by the addition of TEMPO

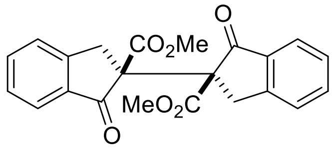
(0.1563 g, 1.0 mmol). The reaction mixture was vigorously stirred at room temperature for 24 h, concentrated, and purified by flash chromatography (silica gel, hexane:EtOAc = 25:1) to give compound **4** as a light yellow oil (0.1283 g, 74%).

Procedure for Scheme 4. To a 4 mL vial equipped with a stir bar were successively added ethyl 3-oxo-3-phenylpropanoate (**5**) (0.0961 g, 0.50 mmol), CuCl (0.0050 g, 0.050 mmol), P(*n*-Bu)$_3$ (0.0202 g, 0.10 mmol), and $CDCl_3$ (1.0 mL). The mixture was stirred at room temperature for 10 minutes, followed by the addition of di-*t*-butyl-diaziridinone (**1**) (0.3405 g, 2.0 mmol). The reaction mixture was vigorously stirred at 65 °C for 24 h, concentrated, and purified by flash chromatography (silica gel, hexane:CH_2Cl_2:EtOAc = 10:2:1) to give tetrasubstituted olefins **6** as a light yellow solid (mixture of ***E*-6** and ***Z*-6**, 0.070 g, 74%).

Procedure for Scheme 5. To a 4 mL vial equipped with a stir bar were successively added diethyl 2,3-dibenzoylsuccinate (**7**) (0.0956 g, 0.25 mmol), CuCl (0.0050 g, 0.050 mmol), P(*n*-Bu)$_3$ (0.0202 g, 0.10 mmol), and $CDCl_3$ (1.0 mL). The mixture was stirred at room temperature for 10 minutes, followed by the addition of di-*t*-butyl-diaziridinone (**1**) (0.3405 g, 2.0 mmol). The reaction mixture was vigorously stirred at 65 °C for 24 h, concentrated, and purified by flash chromatography (silica gel, hexanes:CH_2Cl_2:EtOAc = 10:2:1) to give tetrasubstituted olefins **6** as a light yellow solid (mixture of ***E*-6** and ***Z*-6**, 0.087 g, 91%).

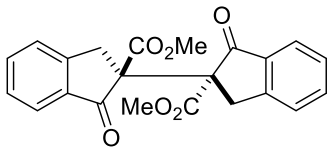
Characterization data

Table 2, entry 1



3a

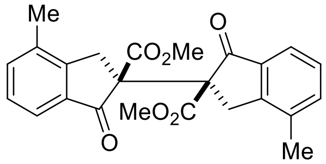
3a (major): white solid; mp 146-147 ºC; IR (neat) 1730, 1703, 1172 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65-7.56 (m, 2H), 7.51 (d, *J* = 7.8 Hz, 2H), 7.43 (d, *J* = 7.8 Hz, 2H), 7.26 (dd, *J* = 7.8 Hz, 6.9 Hz, 2H), 4.53 (d, *J* = 18.3 Hz, 2H), 3.92 (d, *J* = 18.3 Hz, 2H), 3.68 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.2, 171.0, 154.9, 136.1, 135.3, 127.6, 126.4, 124.6, 62.9, 53.2, 37.6; HRMS calcd for $C_{22}H_{19}O_6$ $(M+H)^+$: 379.1176; found: 379.1186.



3a'

3a' (minor): colorless oil; IR (neat) 1760, 1723, 1249 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90-7.83 (m, 2H), 7.75-7.66 (m, 2H), 7.52-7.44 (m, 4H), 4.11 (d, *J* = 17.7 Hz, 2H), 3.82 (s, 6H), 3.57 (d, *J* = 17.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.2, 167.9, 150.7, 136.7, 132.7, 128.8, 126.5, 126.2, 68.1, 54.3, 43.6; HRMS calcd for $C_{22}H_{19}O_6$ $(M+H)^+$: 379.1176; found: 379.1180.

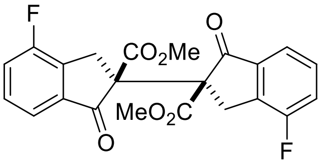
Table 2, entry 2



3b

White solid; mp 165-166 ºC; IR (neat) 1735, 1703, 1265, 1213 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (d, *J* = 6.9 Hz, 2H), 7.22 (d, *J* = 7.5 Hz, 2H), 7.15 (dd, *J* = 7.5, 6.9 Hz, 2H), 4.40 (d, *J* = 18.3 Hz, 2H), 3.79 (d, *J* = 18.3 Hz, 2H), 3.70 (s, 6H), 2.42 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.4, 171.2, 153.9, 136.5, 135.8, 135.0, 127.8, 121.9, 63.1, 53.1, 36.4, 18.0; HRMS calcd for $C_{24}H_{23}O_6$ $(M+H)^+$: 407.1489; found: 407.1491.

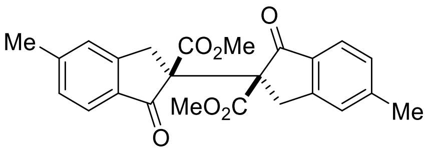
Table 2, entry 3



3c

White solid; mp 151-152 ºC; IR (neat) 1735, 1722, 1481, 1263, 1243 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-7.25 (m, 6H), 4.48 (d, *J* = 18.3 Hz, 2H), 3.93 (d, *J* = 18.3 Hz, 2H), 3.69 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 199.1, 170.2, 159.7 (d, *J* = 250.7 Hz), 140.7 (d, *J* = 20.6 Hz), 138.1 (d, *J* = 4.6 Hz), 129.6 (d, *J* = 5.7 Hz), 122.1 (d, *J* = 20.6 Hz), 120.4 (d, *J* = 3.5 Hz), 62.7, 53.4, 33.6; HRMS calcd for $C_{22}H_{17}F_2O_6$ $(M+H)^+$: 415.0988; found: 415.0990.

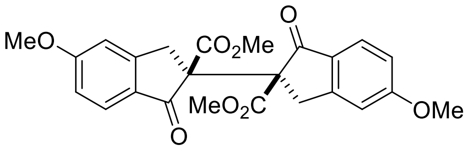
Table 2, entry 4



3d

White solid; mp 218-219 ºC; IR (neat) 1735, 1702, 1606, 1193, 1175 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 (d, *J* = 7.8 Hz, 2H), 7.30 (s, 2H), 7.06 (d, *J* = 7.8 Hz, 2H), 4.50 (d, *J* = 18.3 Hz, 2H), 3.85 (d, *J* = 18.3 Hz, 2H), 3.67 (s, 6H), 2.43 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 199.6, 171.3, 155.6, 147.5, 133.1, 129.0, 126.7, 124.5, 63.0, 53.1, 37.4, 22.5; HRMS calcd for $C_{24}H_{23}O_6$ $(M+H)^+$: 407.1489; found: 407.1498.

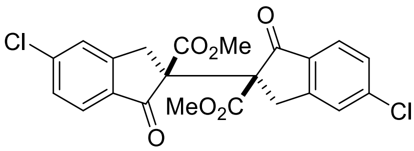
Table 2, entry 5



3e

White solid; mp 189-190 ºC; IR (neat) 1725, 1695, 1595, 1260 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.7 Hz, 2H), 6.91 (d, *J* = 2.1 Hz, 2H), 6.78 (dd, *J* = 8.7, 2.1 Hz, 2H), 4.52 (d, *J* = 18.6 Hz, 2H), 3.88 (s, 6H), 3.84 (d, *J* = 18.6 Hz, 2H), 3.68 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.1, 171.4, 166.4, 158.4, 128.5, 126.4, 116.4, 108.8, 63.1, 55.9, 53.0, 37.5; HRMS calcd for $C_{24}H_{23}O_8$ $(M+H)^+$: 439.1387; found: 439.1390.

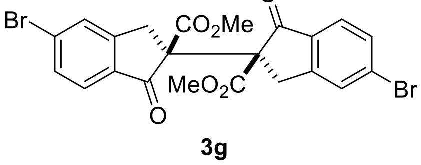
Table 2, entry 6



3f

Yellow solid; mp 175-176 ºC; IR (neat) 1736, 1698, 1598, 1259 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (s, 2H), 7.41 (d, *J* = 8.1 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 2H), 4.49 (d, *J* = 18.3 Hz, 2H), 3.89 (d, *J* = 18.3 Hz, 2H), 3.68 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.9, 170.4, 156.4, 142.9, 133.7, 128.6, 126.6, 125.8, 62.9, 53.3, 37.4; HRMS calcd for $C_{22}H_{16}Cl_2NaO_6$ $(M+Na)^+$: 469.0216; found: 469.0223.

Table 2, entry 7

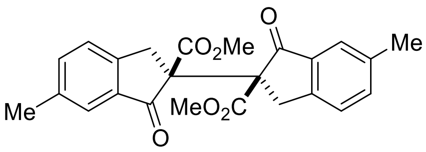


3g

White solid; mp 175-176 ºC; IR (neat) 1731, 1698, 1596, 1169 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 (s, 2H), 7.43 (d, *J* = 8.1 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 4.49 (d, *J* = 18.3 Hz, 2H), 3.89 (d, *J* = 18.3 Hz, 2H), 3.68 (s, 6H); ^{13}C NMR (75

MHz, $CDCl_3$) δ 199.1, 170.4, 156.5, 134.1, 131.9, 131.4, 129.7, 125.9, 62.8, 53.4, 37.3; HRMS calcd for $C_{22}H_{16}Br_2NaO_6$ $(M+Na)^+$: 556.9206; found: 556.9213.

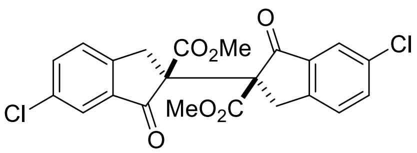
Table 2, entry 8



3h

White solid; mp 184-185 ºC; IR (neat) 1738, 1703, 1168, 1156, 1031 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.46-7.34 (m, 4H), 7.25 (s, 2H), 4.47 (d, *J* = 18.3 Hz, 2H), 3.85 (d, *J* = 18.3 Hz, 2H), 3.66 (s, 6H), 2.31 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.3, 171.1, 152.5, 137.42, 137.39, 135.5, 126.0, 124.5, 63.1, 53.0, 37.3, 21.1; HRMS calcd for $C_{24}H_{23}O_6$ $(M+H)^+$: 407.1489; found: 407.1498.

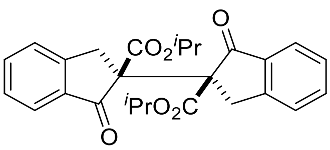
Table 2, entry 9



3i

Light yellow solid; mp 209-210 ºC; IR (neat) 1736, 1702, 1171, 1031 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.58 (dd, *J* = 8.1, 2.1 Hz, 2H), 7.50-7.41 (m, 4H), 4.45 (d, *J* = 18.3 Hz, 2H), 3.87 (d, *J* = 18.3 Hz, 2H), 3.67 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 199.1, 170.3, 153.1, 136.7, 136.2, 134.1, 127.6, 124.4, 63.3, 53.4, 37.4; HRMS calcd for $C_{22}H_{16}Cl_2NaO_6$ $(M+Na)^+$: 469.0216; found: 469.0214.

Table 2, entry 10

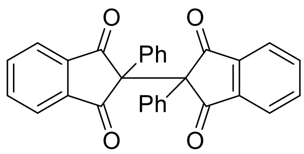


3j

Colorless oil; IR (neat) 1734, 1709, 1607, 1278, 1104 cm^{-1}; ^1H NMR (400 MHz,

$CDCl_3$) δ 7.65-7.55 (m, 2H), 7.50 (d, *J* = 7.7 Hz, 2H), 7.37 (d, *J* = 7.7 Hz, 2H), 7.23 (t, *J* = 7.4 Hz, 2H), 5.04 (septet, *J* = 6.3 Hz, 2H), 4.51 (d, *J* = 18.0 Hz, 2H), 3.89 (d, *J* = 18.0 Hz, 2H), 1.21 (d, *J* = 6.3 Hz, 6H), 1.13 (d, *J* = 6.3 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.5, 169.9, 154.8, 135.7, 135.3, 127.3, 126.2, 124.3, 69.8, 63.4, 37.5, 21.7, 21.4.

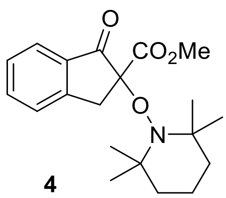
Table 2, entry 11



3k

White solid; mp 211-212 ºC; IR (neat) 1698, 1252 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92-7.85 (m, 4H), 7.78-7.71 (m, 4H), 7.38-7.30 (m, 2H), 7.29-7.22 (m, 4H), 7.22-7.14 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.6, 141.1, 135.8, 130.6, 130.1, 128.9, 127.7, 124.0, 64.6; HRMS calcd for $C_{30}H_{19}O_4$ (M+H)$^+$: 443.1278; found: 443.1283.

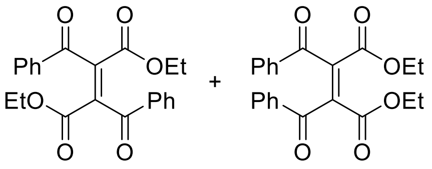
Scheme 3



4

Light yellow oil; IR (neat) 1759, 1717, 1166 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.5 Hz, 1H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.48 (d, *J* = 7.5 Hz, 1H), 7.35 (t, *J* = 7.5 Hz, 1H), 4.59 (d, *J* = 17.7 Hz, 1H), 3.73 (s, 3H), 3.45 (d, *J* = 17.7 Hz, 1H), 1.63-1.24 (m, 6H), 1.32 (s, 3H), 1.18 (s, 3H), 1.04 (s, 3H), 0.56 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.7, 170.3, 153.0, 135.6, 133.7, 127.4, 126.0, 124.6, 89.9, 60.2, 59.2, 52.7, 40.2, 40.0, 33.4, 32.1, 31.9, 20.7, 20.3, 16.8; HRMS calcd for $C_{20}H_{28}NO_4$ (M+H)$^+$: 346.2013; found: 346.2015.

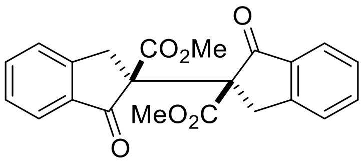
Scheme 4



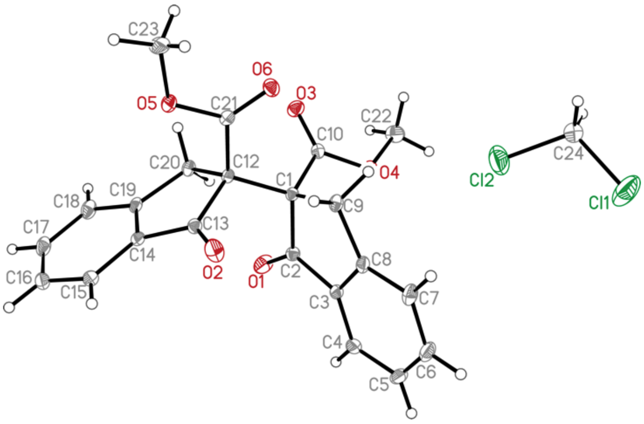
***E*-6** ***Z*-6**

Light yellow solid; IR (neat) 1722, 1680, 1236 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) **Major isomer**: δ 8.04-7.94 (m, 4H), 7.66-7.56 (m, 2H), 7.56-7.46 (m, 4H), 4.02 (q, *J* = 7.2 Hz, 4H), 0.93 (t, *J* = 7.2 Hz, 6H); **Minor isomer**: 7.85-7.78 (m, 4H), 7.66-7.56 (m, 2H), 7.44-7.36 (m, 4H), 4.26 (q, *J* = 7.2 Hz, 4H), 1.20 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) **Major and Minor isomers**: δ 191.2, 190.7, 163.5, 162.7, 142.3, 142.2, 135.8, 135.6, 134.4, 134.1, 129.5, 129.1, 129.0, 128.8, 62.9, 62.8, 13.9, 13.5; HRMS calcd for $C_{22}H_{21}O_6$ $(M+H)^+$: 381.1333; found: 381.1336.

X-ray structure of 3a



3a



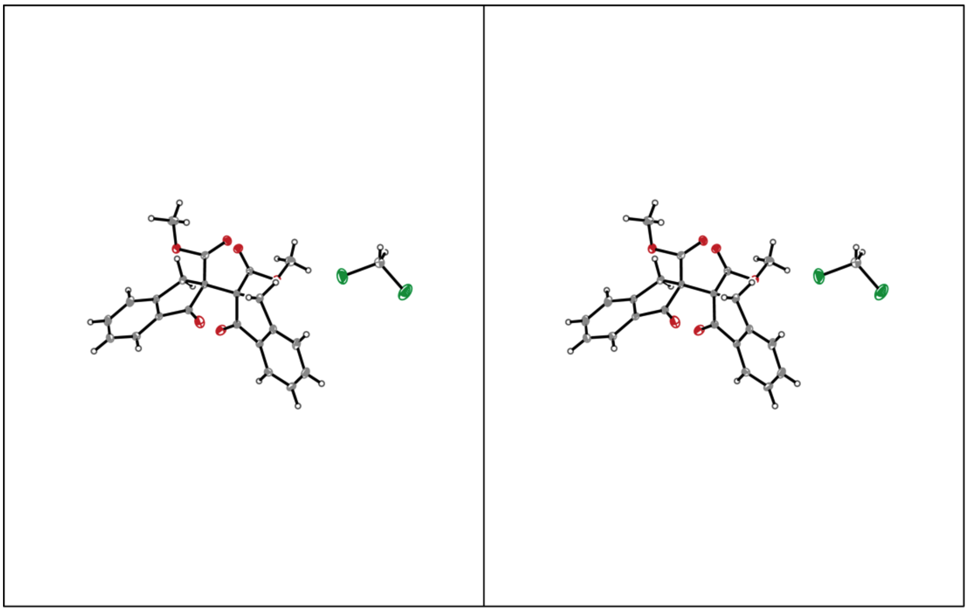


Table 1. Crystal data and structure refinement for **3a**.

Identification code	shi06_new	
Empirical formula	C23 H20 Cl2 O6	
Formula weight	463.29	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 12.9425(9) Å	α= 90°.
	b = 15.6165(11) Å	β= 101.551(3)°.
	c = 10.6618(7) Å	γ = 90°.
Volume	2111.3(3) Å^3	
Z	4	
Density (calculated)	1.458 Mg/m^3	
Absorption coefficient	0.346 mm^{-1}	
F(000)	960	
Crystal size	0.187 x 0.123 x 0.087 mm^3	
Theta range for data collection	2.069 to 27.951°.	
Index ranges	-17<=h<=17, -20<=k<=20, -14<=l<=14	
Reflections collected	18659	
Independent reflections	2541 [R(int) = 0.0368]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2541 / 0 / 142	
Goodness-of-fit on F^2	1.042	
Final R indices [I>2sigma(I)]	R1 = 0.0387, wR2 = 0.0956	
R indices (all data)	R1 = 0.0493, wR2 = 0.1028	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.423 and -0.525 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	9480(1)	2202(1)	3669(1)	13(1)
C(2)	9486(1)	2957(1)	2764(1)	12(1)
C(3)	9402(1)	3788(1)	3557(1)	14(1)
C(4)	8358(1)	4166(1)	3098(1)	14(1)
C(5)	7920(1)	4879(1)	3587(1)	19(1)
C(6)	6901(1)	5112(1)	3013(2)	24(1)
C(7)	6349(1)	4644(1)	1976(2)	24(1)
C(8)	6790(1)	3936(1)	1494(1)	21(1)
C(9)	7811(1)	3696(1)	2072(1)	15(1)
C(10)	8444(1)	2958(1)	1726(1)	15(1)
C(11)	8533(1)	1565(1)	5094(2)	28(1)
O(1)	10140(1)	1652(1)	3901(1)	20(1)
O(2)	8627(1)	2241(1)	4194(1)	22(1)
O(3)	10093(1)	4050(1)	4403(1)	26(1)
C(12)	5000	1100(2)	2500	28(1)
Cl(1)	6135(1)	1732(1)	2697(1)	70(1)

Table 3. Bond lengths [Å] and angles [°] for **3a**.

C(1)-O(1)	1.2010(17)
C(1)-O(2)	1.3352(16)
C(1)-C(2)	1.5249(18)
C(2)-C(2)#1	1.546(2)
C(2)-C(10)	1.5635(18)
C(2)-C(3)	1.5640(18)
C(3)-O(3)	1.2066(17)
C(3)-C(4)	1.4671(18)
C(4)-C(9)	1.3887(19)
C(4)-C(5)	1.3956(19)
C(5)-C(6)	1.387(2)
C(6)-C(7)	1.396(2)
C(7)-C(8)	1.390(2)
C(8)-C(9)	1.3926(19)
C(9)-C(10)	1.5019(19)
C(11)-O(2)	1.4483(18)
C(12)-Cl(1)	1.7471(14)
C(12)-Cl(1)#2	1.7471(14)
O(1)-C(1)-O(2)	124.00(12)
O(1)-C(1)-C(2)	126.20(12)
O(2)-C(1)-C(2)	109.80(11)
C(1)-C(2)-C(2)#1	110.11(9)
C(1)-C(2)-C(10)	109.62(10)
C(2)#1-C(2)-C(10)	115.18(13)
C(1)-C(2)-C(3)	106.87(10)
C(2)#1-C(2)-C(3)	110.76(8)
C(10)-C(2)-C(3)	103.84(10)
O(3)-C(3)-C(4)	127.43(13)
O(3)-C(3)-C(2)	124.37(12)
C(4)-C(3)-C(2)	108.20(11)
C(9)-C(4)-C(5)	122.37(12)
C(9)-C(4)-C(3)	109.88(12)
C(5)-C(4)-C(3)	127.74(13)
C(6)-C(5)-C(4)	117.75(14)
C(5)-C(6)-C(7)	120.17(14)
C(8)-C(7)-C(6)	121.75(13)

C(7)-C(8)-C(9)	118.31(14)
C(4)-C(9)-C(8)	119.66(13)
C(4)-C(9)-C(10)	112.47(11)
C(8)-C(9)-C(10)	127.87(13)
C(9)-C(10)-C(2)	105.21(11)
C(1)-O(2)-C(11)	115.52(11)
Cl(1)-C(12)-Cl(1)#2	111.26(13)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,y,-z+1/2 #2 -x+1,y,-z+1/2

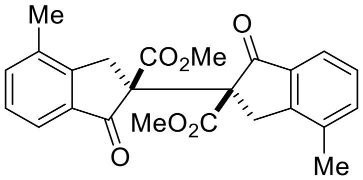
Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for **3a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	14(1)	16(1)	11(1)	-2(1)	3(1)	-2(1)
C(2)	10(1)	13(1)	12(1)	-1(1)	2(1)	0(1)
C(3)	14(1)	14(1)	13(1)	-1(1)	4(1)	0(1)
C(4)	13(1)	14(1)	18(1)	3(1)	5(1)	0(1)
C(5)	20(1)	15(1)	25(1)	2(1)	10(1)	0(1)
C(6)	21(1)	18(1)	34(1)	6(1)	14(1)	6(1)
C(7)	14(1)	29(1)	31(1)	12(1)	7(1)	7(1)
C(8)	13(1)	29(1)	19(1)	5(1)	2(1)	1(1)
C(9)	12(1)	18(1)	16(1)	3(1)	5(1)	0(1)
C(10)	10(1)	19(1)	13(1)	-1(1)	0(1)	-1(1)
C(11)	39(1)	21(1)	31(1)	6(1)	24(1)	1(1)
O(1)	20(1)	20(1)	21(1)	5(1)	7(1)	5(1)
O(2)	23(1)	20(1)	28(1)	6(1)	17(1)	3(1)
O(3)	18(1)	29(1)	27(1)	-13(1)	-3(1)	2(1)
C(12)	24(1)	25(1)	37(1)	0	7(1)	0
Cl(1)	42(1)	61(1)	116(1)	-52(1)	37(1)	-26(1)

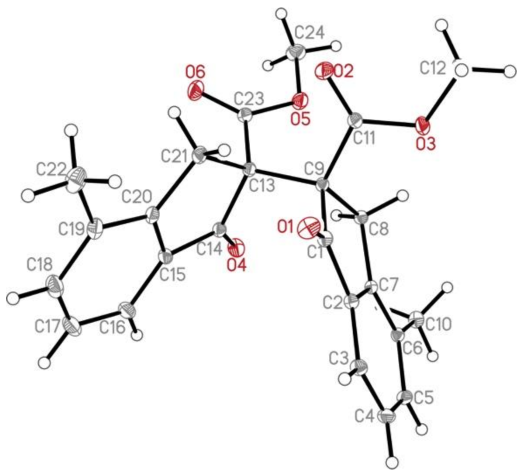
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **3a**.

	x	y	z	U(eq)
H(5)	8308	5193	4289	23
H(6)	6578	5591	3327	28
H(7)	5653	4815	1590	29
H(8)	6405	3623	789	25
H(10A)	8598	3038	861	18
H(10B)	8058	2412	1743	18
H(11A)	8458	1012	4648	42
H(11B)	7912	1669	5467	42
H(11C)	9166	1555	5775	42
H(12A)	5037	727	3259	34
H(12B)	4963	727	1741	34

X-ray structure of 3b



3b



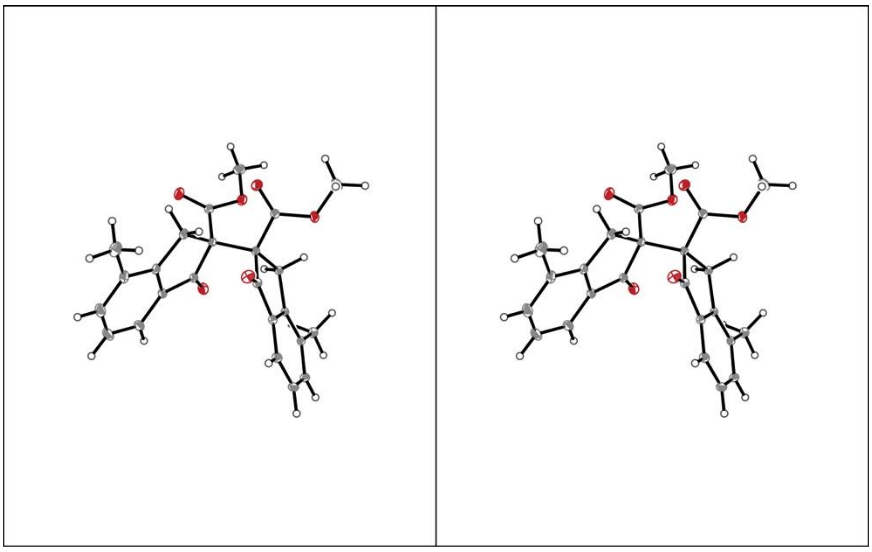


Table 1. Crystal data and structure refinement for **3b**.

Identification code	ys217	
Empirical formula	C24 H22 O6	
Formula weight	406.41	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 8.8734(3) Å	α= 90°.
	b = 16.0071(5) Å	β= 100.720(2)°.
	c = 14.1890(5) Å	γ = 90°.
Volume	1980.20(12) $Å^3$	
Z	4	
Density (calculated)	1.363 Mg/m^3	
Absorption coefficient	0.098 mm^{-1}	
F(000)	856	
Crystal size	0.716 x 0.696 x 0.328 mm^3	
Theta range for data collection	1.937 to 36.702°.	
Index ranges	-14<=h<=14, -26<=k<=26, -23<=l<=23	
Reflections collected	173511	
Independent reflections	9831 [R(int) = 0.0428]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9831 / 0 / 275	
Goodness-of-fit on F^2	1.067	
Final R indices [I>2sigma(I)]	R1 = 0.0395, wR2 = 0.1048	
R indices (all data)	R1 = 0.0499, wR2 = 0.1138	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.609 and -0.274 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **3b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	11527(1)	2998(1)	4953(1)	19(1)
O(2)	13084(1)	2623(1)	3046(1)	23(1)
O(3)	13113(1)	1407(1)	3832(1)	17(1)
O(4)	7251(1)	2213(1)	2514(1)	18(1)
O(5)	10507(1)	1815(1)	1604(1)	18(1)
O(6)	9668(1)	3089(1)	1098(1)	23(1)
C(1)	10762(1)	2421(1)	4565(1)	12(1)
C(2)	9753(1)	1864(1)	4985(1)	11(1)
C(3)	9335(1)	1929(1)	5887(1)	14(1)
C(4)	8395(1)	1308(1)	6140(1)	15(1)
C(5)	7912(1)	642(1)	5511(1)	14(1)
C(6)	8343(1)	569(1)	4616(1)	11(1)
C(7)	9282(1)	1203(1)	4361(1)	10(1)
C(8)	9937(1)	1258(1)	3456(1)	11(1)
C(9)	10746(1)	2123(1)	3506(1)	10(1)
C(10)	7825(1)	-147(1)	3951(1)	15(1)
C(11)	12430(1)	2086(1)	3411(1)	13(1)
C(12)	14756(1)	1355(1)	3888(1)	20(1)
C(13)	9891(1)	2779(1)	2777(1)	12(1)
C(14)	8129(1)	2778(1)	2803(1)	12(1)
C(15)	7752(1)	3586(1)	3191(1)	13(1)
C(16)	6323(1)	3848(1)	3363(1)	18(1)
C(17)	6216(1)	4664(1)	3671(1)	22(1)
C(18)	7495(1)	5196(1)	3803(1)	22(1)
C(19)	8919(1)	4934(1)	3632(1)	17(1)
C(20)	9023(1)	4109(1)	3317(1)	13(1)
C(21)	10385(1)	3699(1)	3018(1)	15(1)
C(22)	10280(1)	5510(1)	3763(1)	24(1)
C(23)	10015(1)	2588(1)	1736(1)	14(1)
C(24)	10665(1)	1630(1)	628(1)	22(1)

Table 3. Bond lengths [Å] and angles [°] for **3b**.

O(1)-C(1)	1.2156(8)
O(2)-C(11)	1.2069(8)
O(3)-C(11)	1.3315(8)
O(3)-C(12)	1.4478(9)
O(4)-C(14)	1.2148(8)
O(5)-C(23)	1.3365(9)
O(5)-C(24)	1.4489(9)
O(6)-C(23)	1.2063(9)
C(1)-C(2)	1.4665(9)
C(1)-C(9)	1.5733(9)
C(2)-C(7)	1.3926(9)
C(2)-C(3)	1.4015(9)
C(3)-C(4)	1.3872(10)
C(3)-H(3)	0.9500
C(4)-C(5)	1.4045(10)
C(4)-H(4)	0.9500
C(5)-C(6)	1.3963(9)
C(5)-H(5)	0.9500
C(6)-C(7)	1.4033(9)
C(6)-C(10)	1.5009(9)
C(7)-C(8)	1.5071(9)
C(8)-C(9)	1.5549(9)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(11)	1.5256(9)
C(9)-C(13)	1.5674(9)
C(10)-H(10A)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-C(23)	1.5316(9)
C(13)-C(21)	1.5564(9)
C(13)-C(14)	1.5710(9)
C(14)-C(15)	1.4680(9)

C(15)-C(20)	1.3903(10)
C(15)-C(16)	1.3993(10)
C(16)-C(17)	1.3873(11)
C(16)-H(16)	0.9500
C(17)-C(18)	1.4024(13)
C(17)-H(17)	0.9500
C(18)-C(19)	1.3952(11)
C(18)-H(18)	0.9500
C(19)-C(20)	1.4023(10)
C(19)-C(22)	1.5038(12)
C(20)-C(21)	1.5039(10)
C(21)-H(21A)	0.9900
C(21)-H(21B)	0.9900
C(22)-H(22A)	0.9800
C(22)-H(22B)	0.9800
C(22)-H(22C)	0.9800
C(24)-H(24A)	0.9800
C(24)-H(24B)	0.9800
C(24)-H(24C)	0.9800
C(11)-O(3)-C(12)	116.16(6)
C(23)-O(5)-C(24)	114.57(6)
O(1)-C(1)-C(2)	127.60(6)
O(1)-C(1)-C(9)	124.54(6)
C(2)-C(1)-C(9)	107.82(5)
C(7)-C(2)-C(3)	122.56(6)
C(7)-C(2)-C(1)	109.72(5)
C(3)-C(2)-C(1)	127.67(6)
C(4)-C(3)-C(2)	117.17(6)
C(4)-C(3)-H(3)	121.4
C(2)-C(3)-H(3)	121.4
C(3)-C(4)-C(5)	120.42(6)
C(3)-C(4)-H(4)	119.8
C(5)-C(4)-H(4)	119.8
C(6)-C(5)-C(4)	122.60(6)
C(6)-C(5)-H(5)	118.7
C(4)-C(5)-H(5)	118.7
C(5)-C(6)-C(7)	116.73(6)
C(5)-C(6)-C(10)	122.19(6)

C(7)-C(6)-C(10)	121.08(6)
C(2)-C(7)-C(6)	120.51(6)
C(2)-C(7)-C(8)	112.27(5)
C(6)-C(7)-C(8)	127.19(5)
C(7)-C(8)-C(9)	105.17(5)
C(7)-C(8)-H(8A)	110.7
C(9)-C(8)-H(8A)	110.7
C(7)-C(8)-H(8B)	110.7
C(9)-C(8)-H(8B)	110.7
H(8A)-C(8)-H(8B)	108.8
C(11)-C(9)-C(8)	114.40(5)
C(11)-C(9)-C(13)	109.21(5)
C(8)-C(9)-C(13)	113.79(5)
C(11)-C(9)-C(1)	105.14(5)
C(8)-C(9)-C(1)	103.61(5)
C(13)-C(9)-C(1)	110.17(5)
C(6)-C(10)-H(10A)	109.5
C(6)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(6)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
O(2)-C(11)-O(3)	123.98(6)
O(2)-C(11)-C(9)	124.60(6)
O(3)-C(11)-C(9)	111.32(5)
O(3)-C(12)-H(12A)	109.5
O(3)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
O(3)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(23)-C(13)-C(21)	109.45(5)
C(23)-C(13)-C(9)	112.97(5)
C(21)-C(13)-C(9)	114.03(5)
C(23)-C(13)-C(14)	105.78(5)
C(21)-C(13)-C(14)	103.67(5)
C(9)-C(13)-C(14)	110.23(5)
O(4)-C(14)-C(15)	127.29(6)

O(4)-C(14)-C(13)	124.87(6)
C(15)-C(14)-C(13)	107.79(5)
C(20)-C(15)-C(16)	122.50(6)
C(20)-C(15)-C(14)	110.03(6)
C(16)-C(15)-C(14)	127.35(6)
C(17)-C(16)-C(15)	117.03(7)
C(17)-C(16)-H(16)	121.5
C(15)-C(16)-H(16)	121.5
C(16)-C(17)-C(18)	120.85(7)
C(16)-C(17)-H(17)	119.6
C(18)-C(17)-H(17)	119.6
C(19)-C(18)-C(17)	122.08(7)
C(19)-C(18)-H(18)	119.0
C(17)-C(18)-H(18)	119.0
C(18)-C(19)-C(20)	117.00(7)
C(18)-C(19)-C(22)	121.79(7)
C(20)-C(19)-C(22)	121.20(7)
C(15)-C(20)-C(19)	120.55(6)
C(15)-C(20)-C(21)	112.10(6)
C(19)-C(20)-C(21)	127.24(6)
C(20)-C(21)-C(13)	105.48(5)
C(20)-C(21)-H(21A)	110.6
C(13)-C(21)-H(21A)	110.6
C(20)-C(21)-H(21B)	110.6
C(13)-C(21)-H(21B)	110.6
H(21A)-C(21)-H(21B)	108.8
C(19)-C(22)-H(22A)	109.5
C(19)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(19)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
O(6)-C(23)-O(5)	123.64(7)
O(6)-C(23)-C(13)	122.53(6)
O(5)-C(23)-C(13)	113.81(6)
O(5)-C(24)-H(24A)	109.5
O(5)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5

O(5)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **3b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2hk a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	22(1)	16(1)	18(1)	-4(1)	2(1)	-8(1)
O(2)	14(1)	22(1)	35(1)	13(1)	9(1)	0(1)
O(3)	11(1)	17(1)	24(1)	6(1)	2(1)	3(1)
O(4)	13(1)	16(1)	24(1)	-1(1)	1(1)	-2(1)
O(5)	26(1)	16(1)	14(1)	1(1)	7(1)	3(1)
O(6)	30(1)	22(1)	17(1)	8(1)	5(1)	7(1)
C(1)	12(1)	11(1)	13(1)	-1(1)	1(1)	-1(1)
C(2)	12(1)	11(1)	11(1)	-1(1)	2(1)	0(1)
C(3)	15(1)	14(1)	12(1)	-2(1)	3(1)	1(1)
C(4)	15(1)	18(1)	13(1)	0(1)	5(1)	1(1)
C(5)	12(1)	15(1)	14(1)	2(1)	3(1)	0(1)
C(6)	10(1)	11(1)	12(1)	1(1)	1(1)	0(1)
C(7)	10(1)	10(1)	10(1)	0(1)	2(1)	0(1)
C(8)	12(1)	10(1)	11(1)	-1(1)	3(1)	-1(1)
C(9)	10(1)	10(1)	12(1)	1(1)	2(1)	0(1)
C(10)	15(1)	12(1)	17(1)	-2(1)	1(1)	-2(1)
C(11)	11(1)	13(1)	14(1)	1(1)	2(1)	1(1)
C(12)	11(1)	24(1)	25(1)	0(1)	2(1)	4(1)
C(13)	11(1)	10(1)	14(1)	2(1)	2(1)	0(1)
C(14)	11(1)	12(1)	13(1)	2(1)	2(1)	1(1)
C(15)	14(1)	13(1)	13(1)	1(1)	2(1)	2(1)
C(16)	16(1)	20(1)	18(1)	0(1)	4(1)	4(1)
C(17)	23(1)	23(1)	22(1)	-2(1)	6(1)	8(1)
C(18)	29(1)	17(1)	19(1)	-3(1)	3(1)	7(1)
C(19)	24(1)	12(1)	14(1)	0(1)	1(1)	2(1)
C(20)	16(1)	11(1)	12(1)	2(1)	1(1)	2(1)
C(21)	14(1)	10(1)	19(1)	2(1)	4(1)	-1(1)
C(22)	32(1)	15(1)	24(1)	-1(1)	2(1)	-4(1)
C(23)	13(1)	15(1)	15(1)	2(1)	3(1)	1(1)
C(24)	29(1)	22(1)	16(1)	-2(1)	10(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **3b**.

	x	y	z	U(eq)
H(3)	9681	2379	6308	17
H(4)	8076	1332	6742	18
H(5)	7268	225	5701	16
H(8A)	9112	1223	2883	13
H(8B)	10680	802	3430	13
H(10A)	7192	-528	4252	22
H(10B)	8723	-447	3815	22
H(10C)	7222	68	3350	22
H(12A)	15011	1518	3270	31
H(12B)	15099	781	4040	31
H(12C)	15271	1732	4391	31
H(16)	5464	3482	3273	21
H(17)	5265	4866	3795	27
H(18)	7387	5752	4015	26
H(21A)	11289	3720	3545	18
H(21B)	10646	3982	2448	18
H(22A)	9967	6070	3931	36
H(22B)	10680	5538	3165	36
H(22C)	11080	5299	4279	36
H(24A)	9668	1693	201	32
H(24B)	11030	1055	592	32
H(24C)	11404	2017	430	32

Table 6. Torsion angles [°] for **3b**.

O(1)-C(1)-C(2)-C(7)	-170.26(7)
C(9)-C(1)-C(2)-C(7)	7.61(7)
O(1)-C(1)-C(2)-C(3)	7.02(11)
C(9)-C(1)-C(2)-C(3)	-175.11(6)
C(7)-C(2)-C(3)-C(4)	-1.11(10)
C(1)-C(2)-C(3)-C(4)	-178.07(6)
C(2)-C(3)-C(4)-C(5)	0.77(10)
C(3)-C(4)-C(5)-C(6)	0.10(10)
C(4)-C(5)-C(6)-C(7)	-0.66(9)
C(4)-C(5)-C(6)-C(10)	179.80(6)
C(3)-C(2)-C(7)-C(6)	0.56(9)
C(1)-C(2)-C(7)-C(6)	178.00(5)
C(3)-C(2)-C(7)-C(8)	-177.60(6)
C(1)-C(2)-C(7)-C(8)	-0.16(7)
C(5)-C(6)-C(7)-C(2)	0.33(9)
C(10)-C(6)-C(7)-C(2)	179.88(6)
C(5)-C(6)-C(7)-C(8)	178.19(6)
C(10)-C(6)-C(7)-C(8)	-2.26(10)
C(2)-C(7)-C(8)-C(9)	-7.35(7)
C(6)-C(7)-C(8)-C(9)	174.64(6)
C(7)-C(8)-C(9)-C(11)	124.97(5)
C(7)-C(8)-C(9)-C(13)	-108.54(6)
C(7)-C(8)-C(9)-C(1)	11.09(6)
O(1)-C(1)-C(9)-C(11)	45.99(8)
C(2)-C(1)-C(9)-C(11)	-131.96(5)
O(1)-C(1)-C(9)-C(8)	166.37(6)
C(2)-C(1)-C(9)-C(8)	-11.58(6)
O(1)-C(1)-C(9)-C(13)	-71.55(8)
C(2)-C(1)-C(9)-C(13)	110.49(6)
C(12)-O(3)-C(11)-O(2)	3.66(11)
C(12)-O(3)-C(11)-C(9)	-172.86(6)
C(8)-C(9)-C(11)-O(2)	147.81(7)
C(13)-C(9)-C(11)-O(2)	18.98(9)
C(1)-C(9)-C(11)-O(2)	-99.21(8)
C(8)-C(9)-C(11)-O(3)	-35.70(8)
C(13)-C(9)-C(11)-O(3)	-164.53(5)

C(1)-C(9)-C(11)-O(3)	77.28(6)
C(11)-C(9)-C(13)-C(23)	57.44(7)
C(8)-C(9)-C(13)-C(23)	-71.73(7)
C(1)-C(9)-C(13)-C(23)	172.44(5)
C(11)-C(9)-C(13)-C(21)	-68.35(7)
C(8)-C(9)-C(13)-C(21)	162.48(5)
C(1)-C(9)-C(13)-C(21)	46.65(7)
C(11)-C(9)-C(13)-C(14)	175.54(5)
C(8)-C(9)-C(13)-C(14)	46.37(7)
C(1)-C(9)-C(13)-C(14)	-69.46(6)
C(23)-C(13)-C(14)-O(4)	53.23(8)
C(21)-C(13)-C(14)-O(4)	168.37(7)
C(9)-C(13)-C(14)-O(4)	-69.20(8)
C(23)-C(13)-C(14)-C(15)	-124.36(6)
C(21)-C(13)-C(14)-C(15)	-9.22(7)
C(9)-C(13)-C(14)-C(15)	113.22(6)
O(4)-C(14)-C(15)-C(20)	-171.63(7)
C(13)-C(14)-C(15)-C(20)	5.88(7)
O(4)-C(14)-C(15)-C(16)	4.24(12)
C(13)-C(14)-C(15)-C(16)	-178.24(6)
C(20)-C(15)-C(16)-C(17)	-0.12(11)
C(14)-C(15)-C(16)-C(17)	-175.53(7)
C(15)-C(16)-C(17)-C(18)	-0.11(12)
C(16)-C(17)-C(18)-C(19)	0.04(13)
C(17)-C(18)-C(19)-C(20)	0.26(11)
C(17)-C(18)-C(19)-C(22)	179.20(8)
C(16)-C(15)-C(20)-C(19)	0.43(10)
C(14)-C(15)-C(20)-C(19)	176.54(6)
C(16)-C(15)-C(20)-C(21)	-175.90(6)
C(14)-C(15)-C(20)-C(21)	0.21(8)
C(18)-C(19)-C(20)-C(15)	-0.48(10)
C(22)-C(19)-C(20)-C(15)	-179.43(7)
C(18)-C(19)-C(20)-C(21)	175.25(7)
C(22)-C(19)-C(20)-C(21)	-3.69(11)
C(15)-C(20)-C(21)-C(13)	-6.19(7)
C(19)-C(20)-C(21)-C(13)	177.77(6)
C(23)-C(13)-C(21)-C(20)	121.55(6)
C(9)-C(13)-C(21)-C(20)	-110.82(6)

C(14)-C(13)-C(21)-C(20)	9.05(6)
C(24)-O(5)-C(23)-O(6)	2.70(11)
C(24)-O(5)-C(23)-C(13)	-178.61(6)
C(21)-C(13)-C(23)-O(6)	-38.61(9)
C(9)-C(13)-C(23)-O(6)	-166.83(7)
C(14)-C(13)-C(23)-O(6)	72.51(8)
C(21)-C(13)-C(23)-O(5)	142.68(6)
C(9)-C(13)-C(23)-O(5)	14.46(8)
C(14)-C(13)-C(23)-O(5)	-106.20(6)

Symmetry transformations used to generate equivalent atoms:

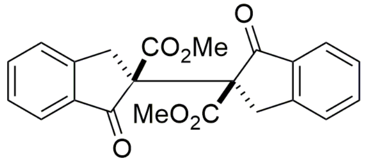
Table 7. Hydrogen bonds for **3b** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(12)-H(12C)...O(6)#1	0.98	2.59	3.2739(10)	126.8
C(12)-H(12C)...O(6)#1	0.98	2.59	3.2739(10)	126.8
C(12)-H(12C)...O(6)#1	0.98	2.59	3.2739(10)	126.8
C(12)-H(12C)...O(6)#1	0.98	2.59	3.2739(10)	126.8
C(12)-H(12C)...O(6)#1	0.98	2.59	3.2739(10)	126.8

Symmetry transformations used to generate equivalent atoms:

#1 x+1/2,-y+1/2,z+1/2

Table 2, entry 1



3a

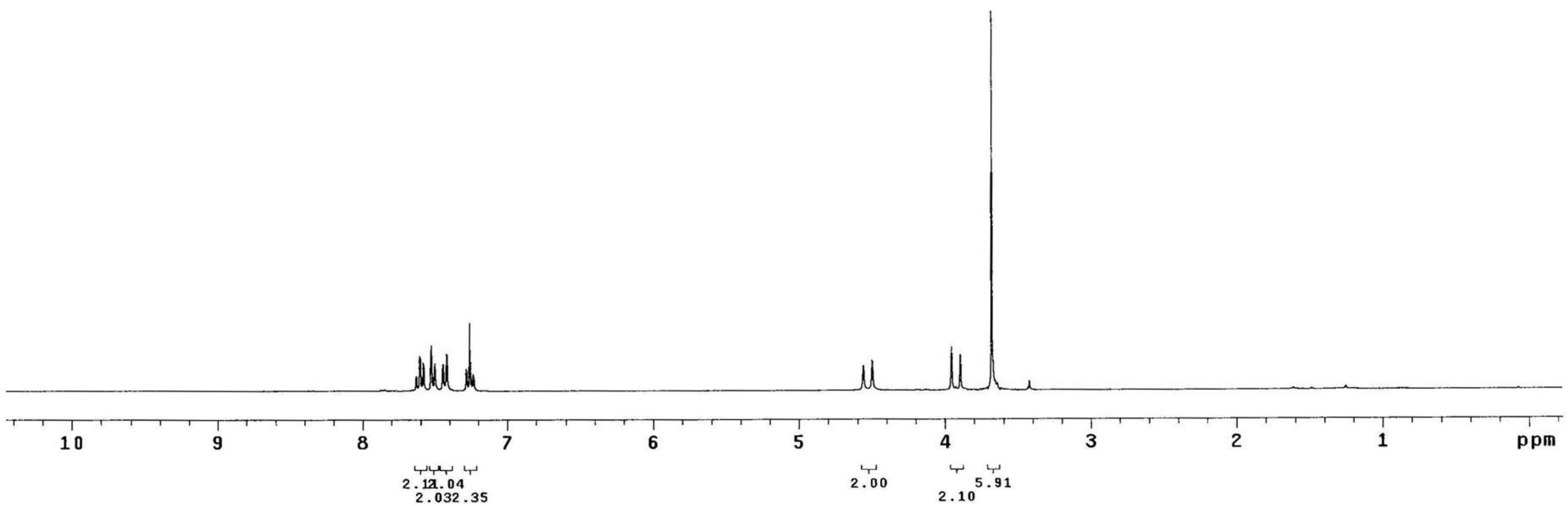
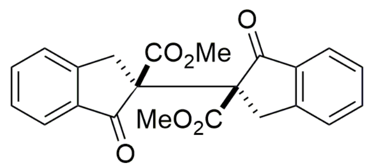


Table 2, entry 1



3a

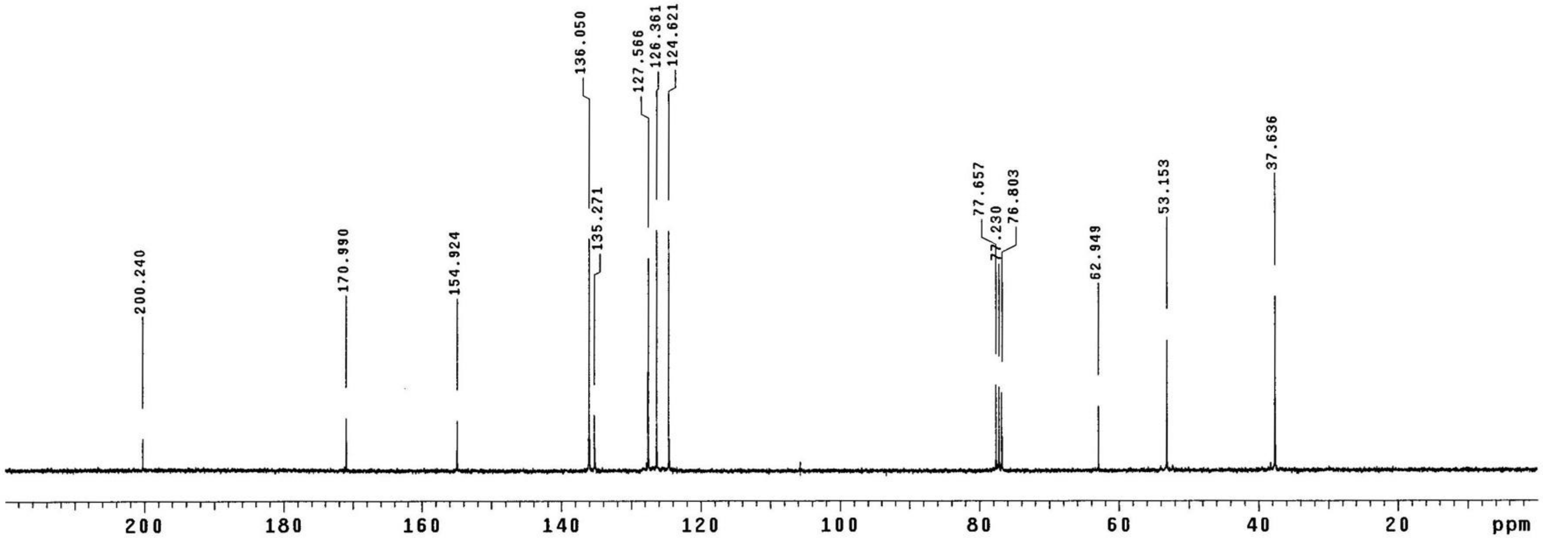
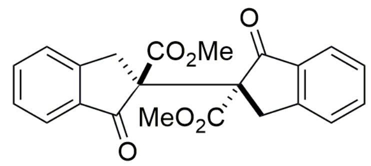


Table 2, entry 1



3a'

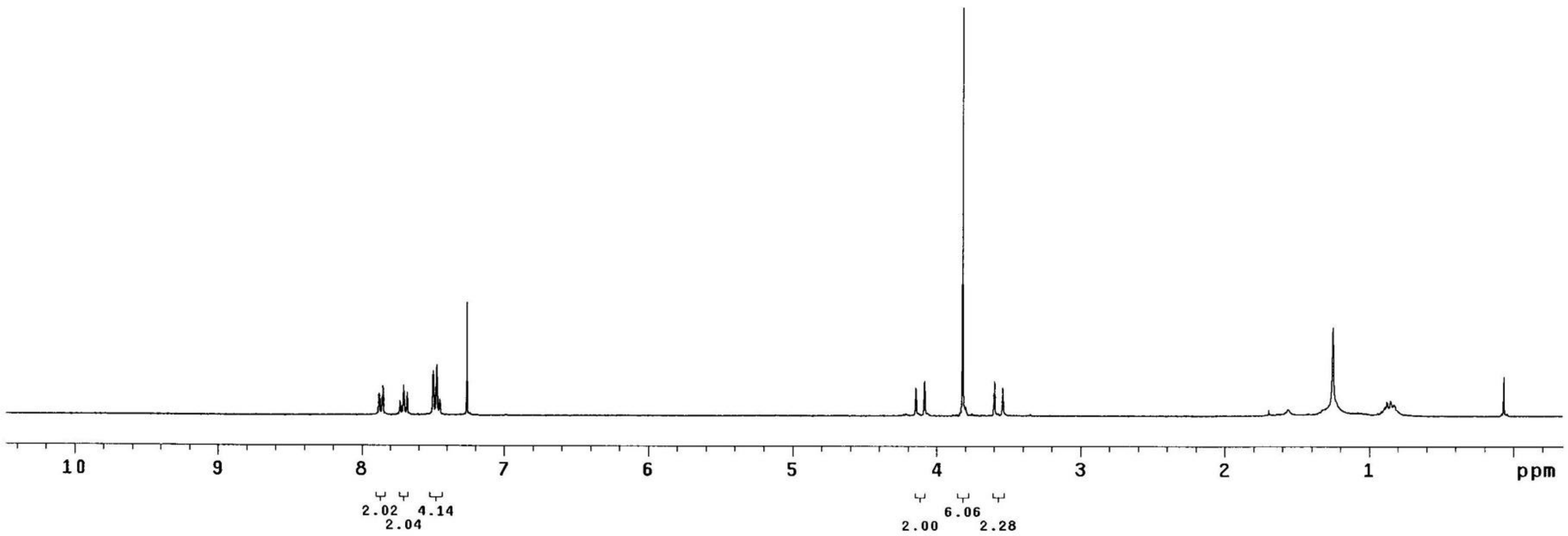
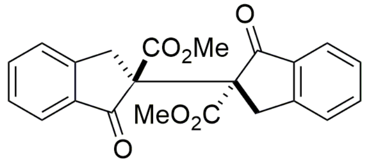


Table 2, entry 1



3a'

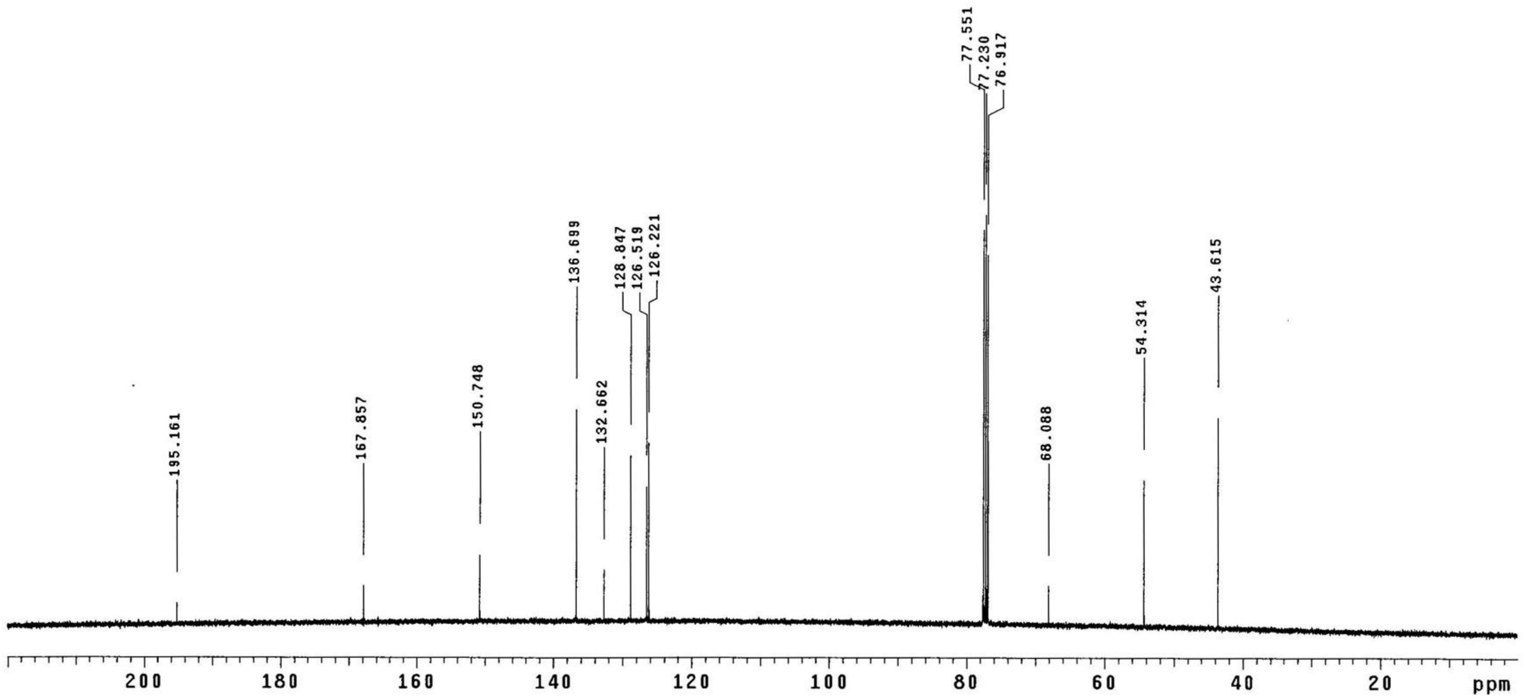
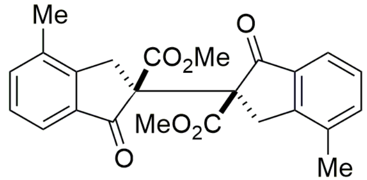
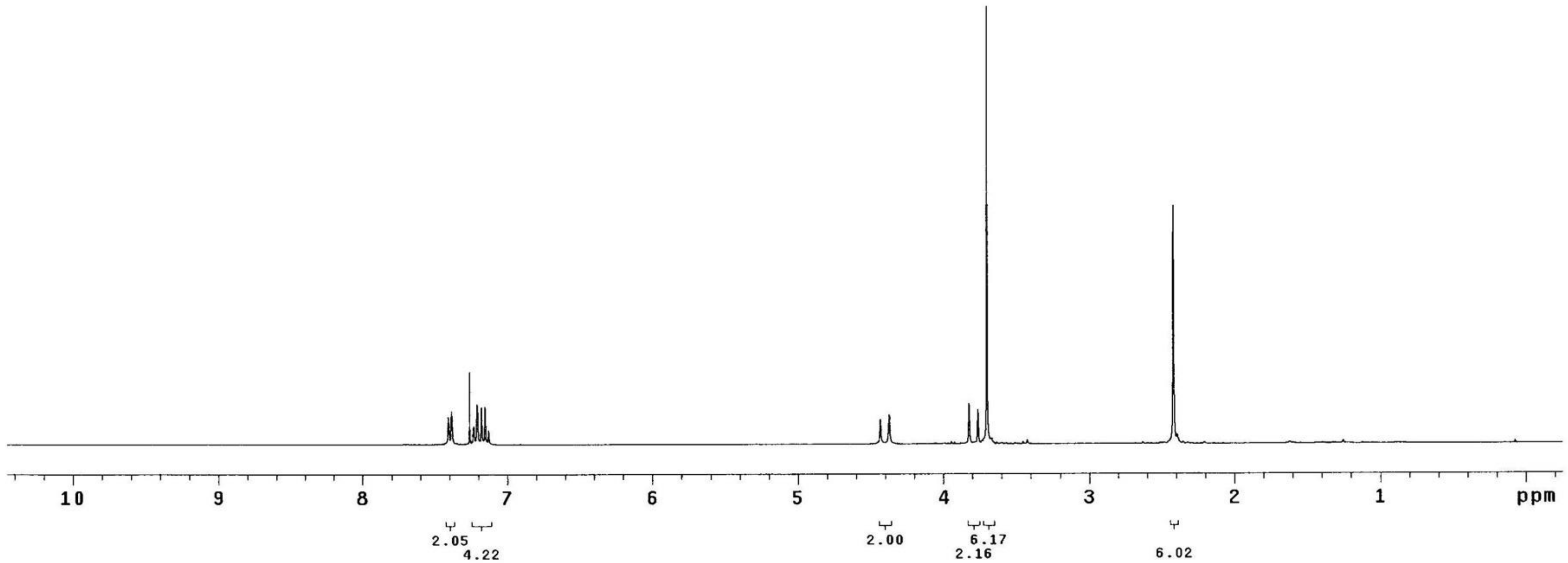


Table 2, entry 2



3b



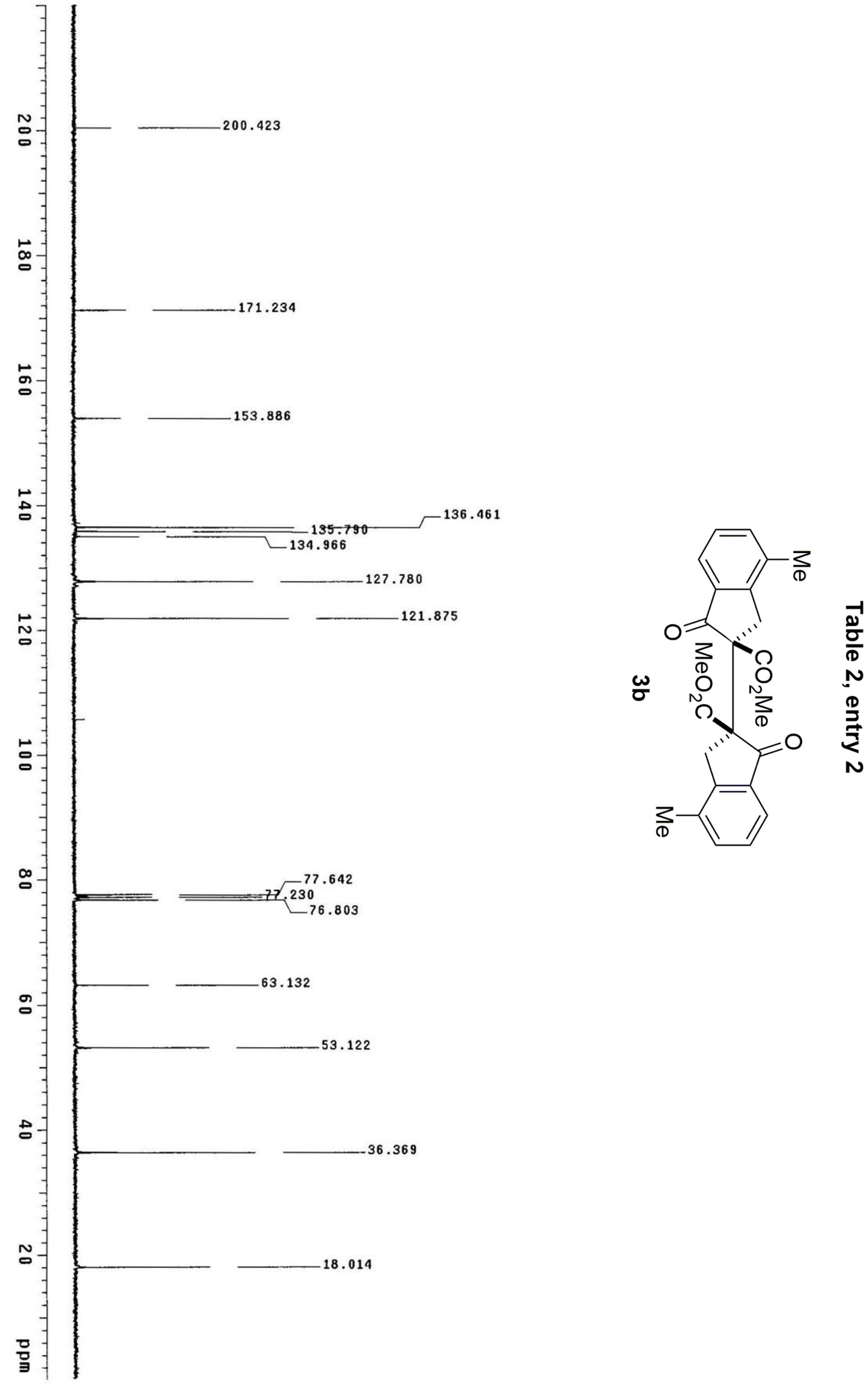
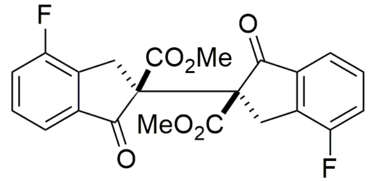
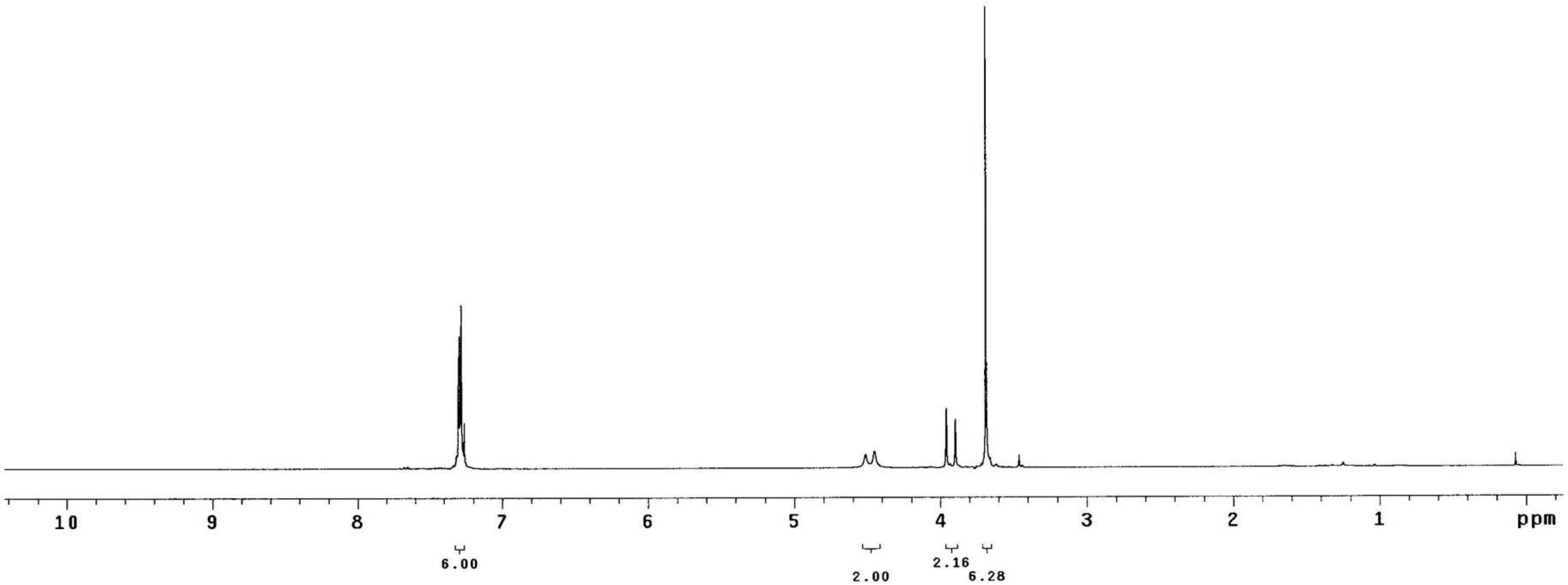


Table 2, entry 2
3b
Me
O
CO2Me
MeO2C
O
Me
200.423
171.234
153.886
136.461
135.790
134.966
127.780
121.875
77.642
77.230
76.803
63.132
53.122
36.369
18.014
200
180
160
140
120
100
80
60
40
20
ppm

Table 2, entry 3



3c



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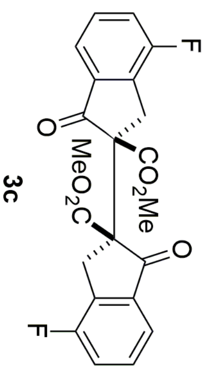
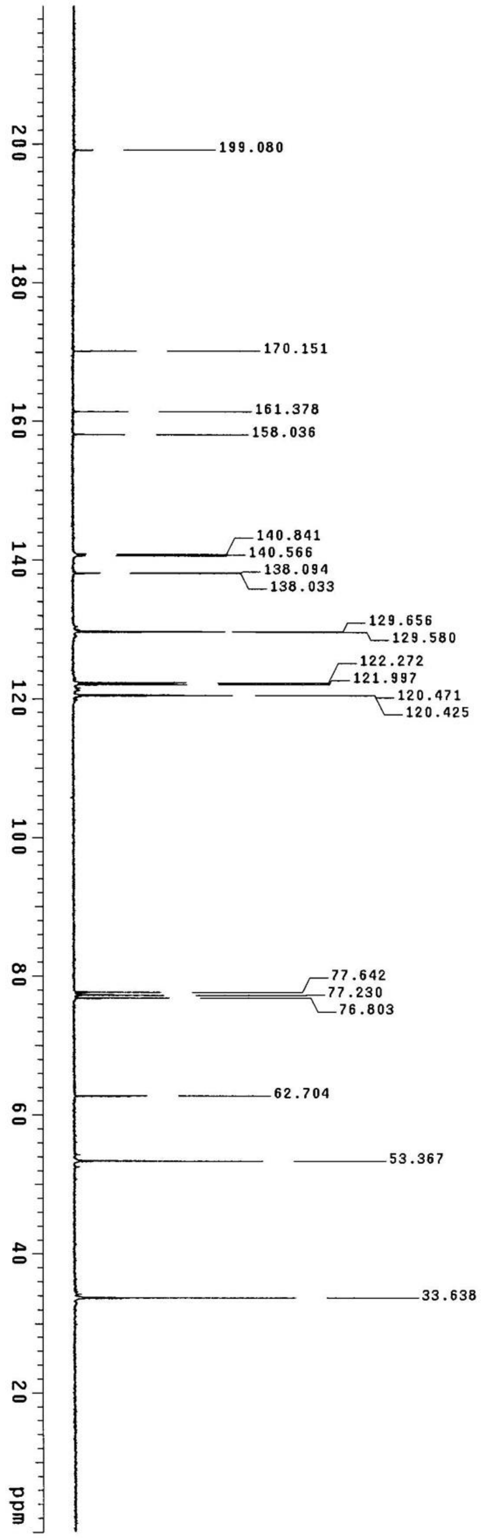


3c



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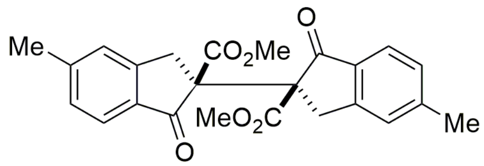
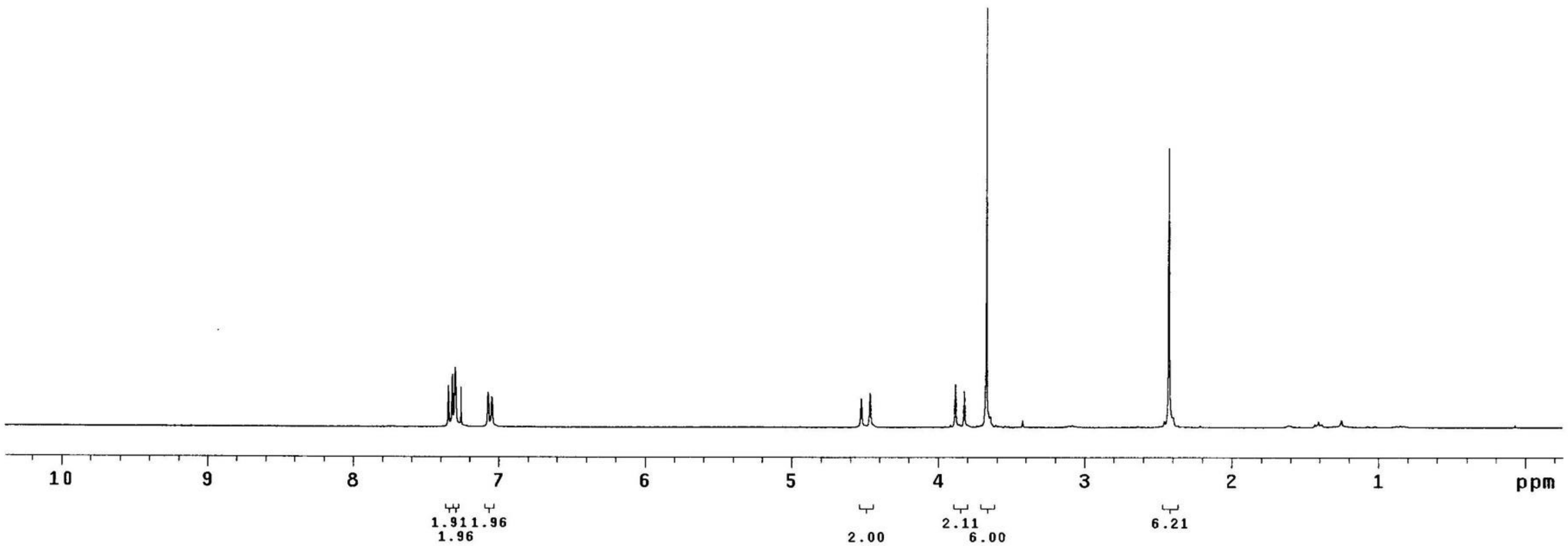


3d



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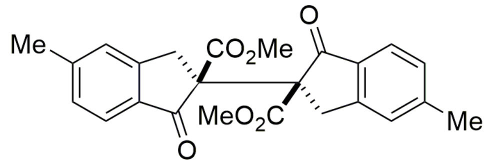
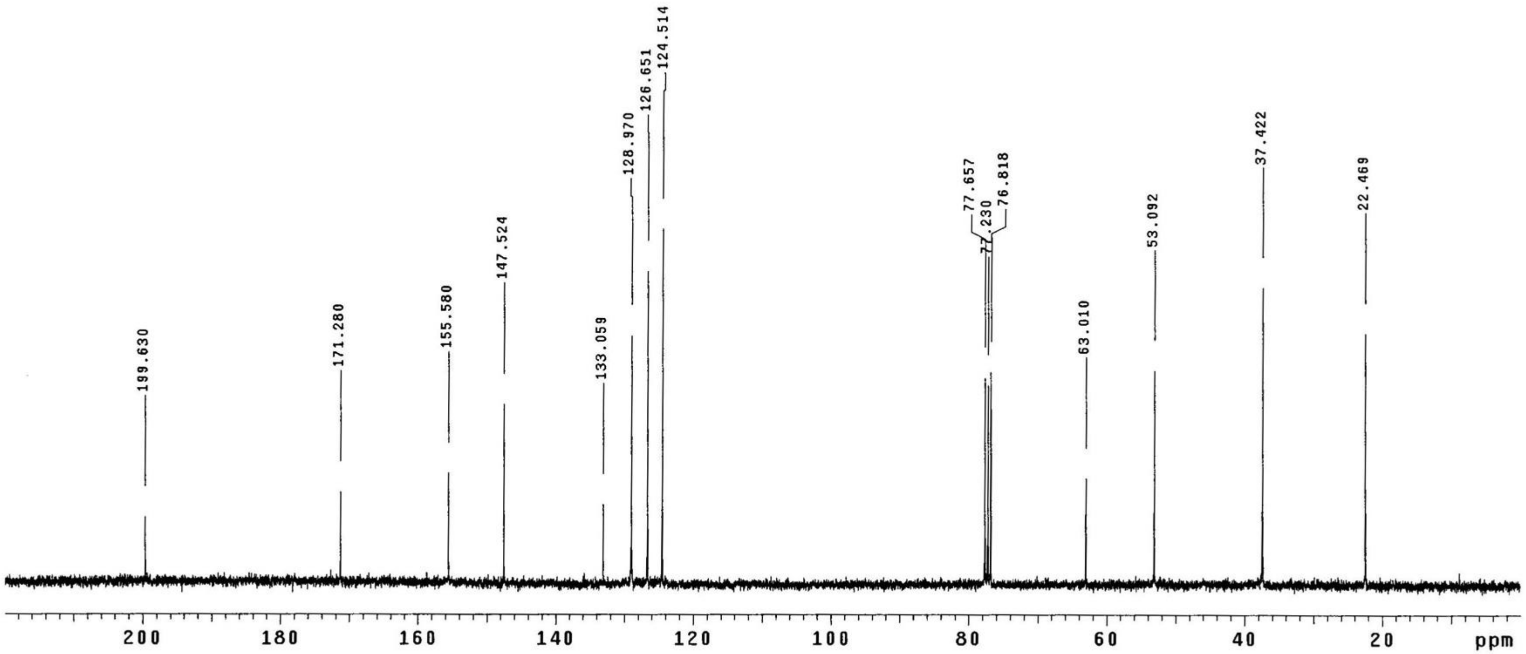


3d



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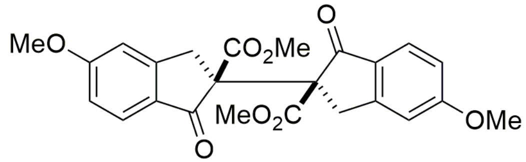


3e

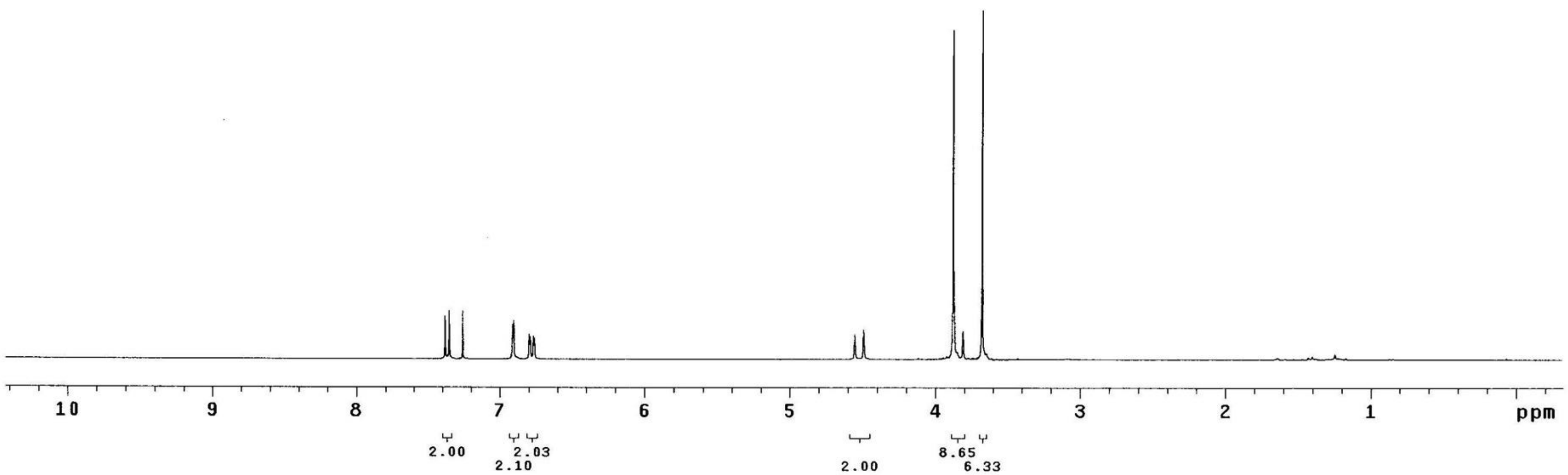
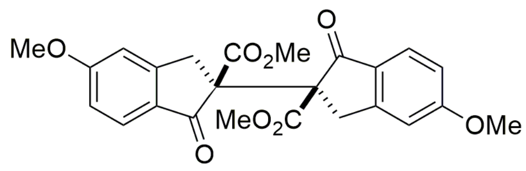
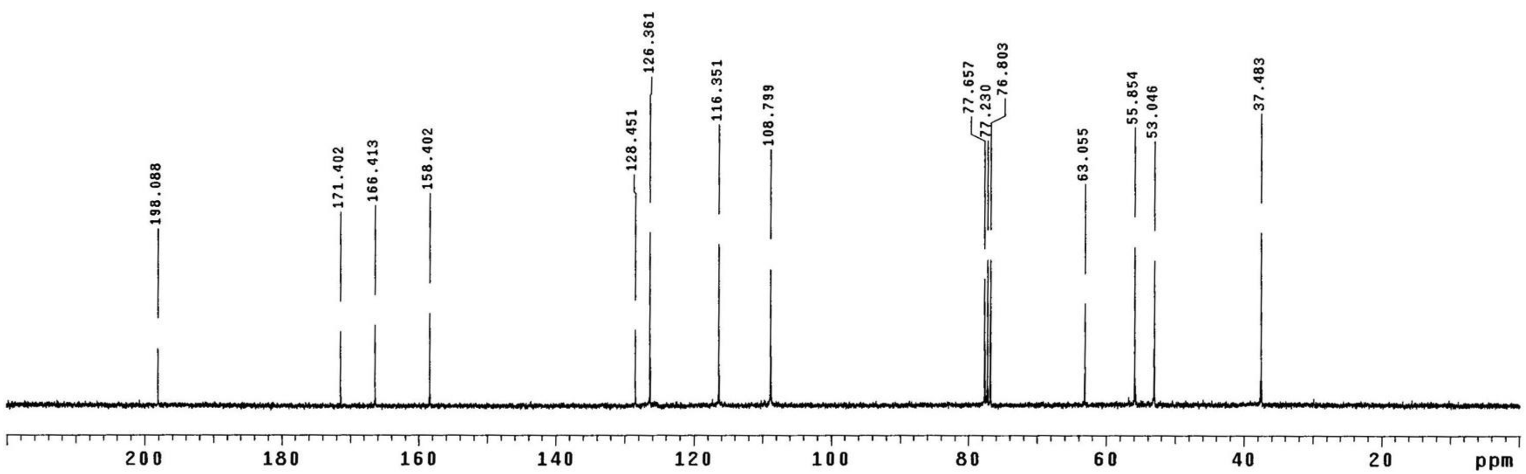


Table 2, entry 5



3e



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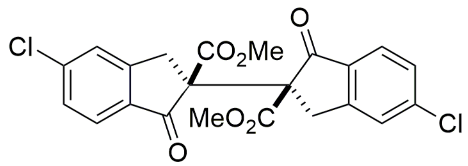


3f

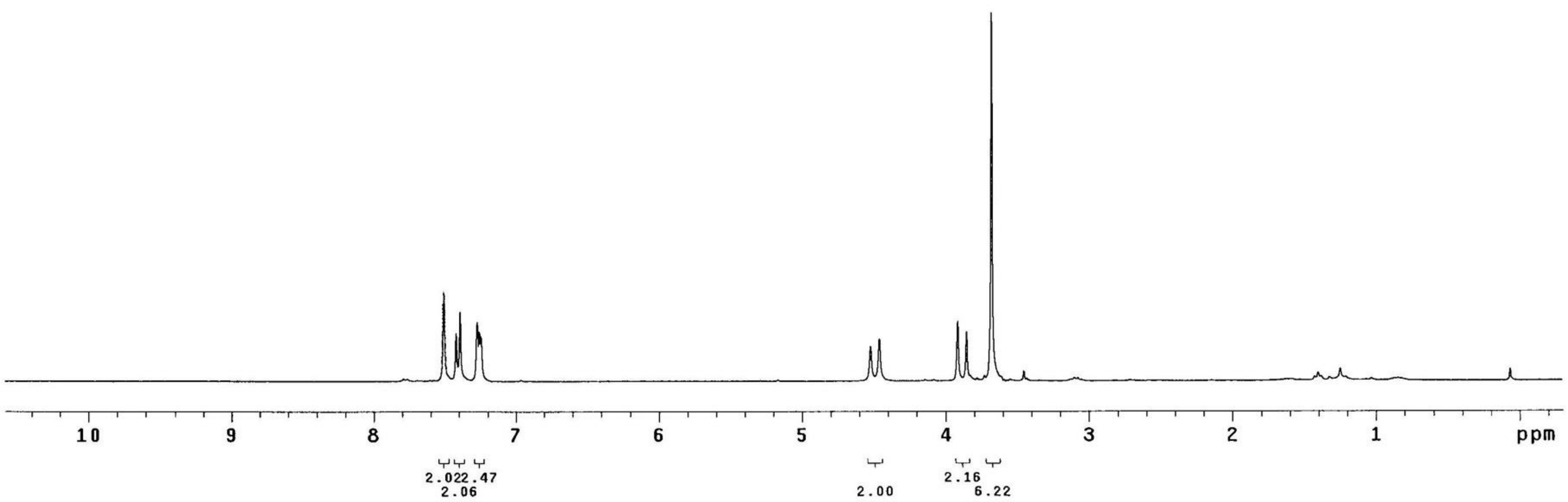
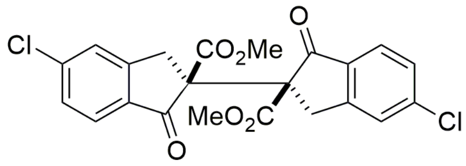


Table 2, entry 6



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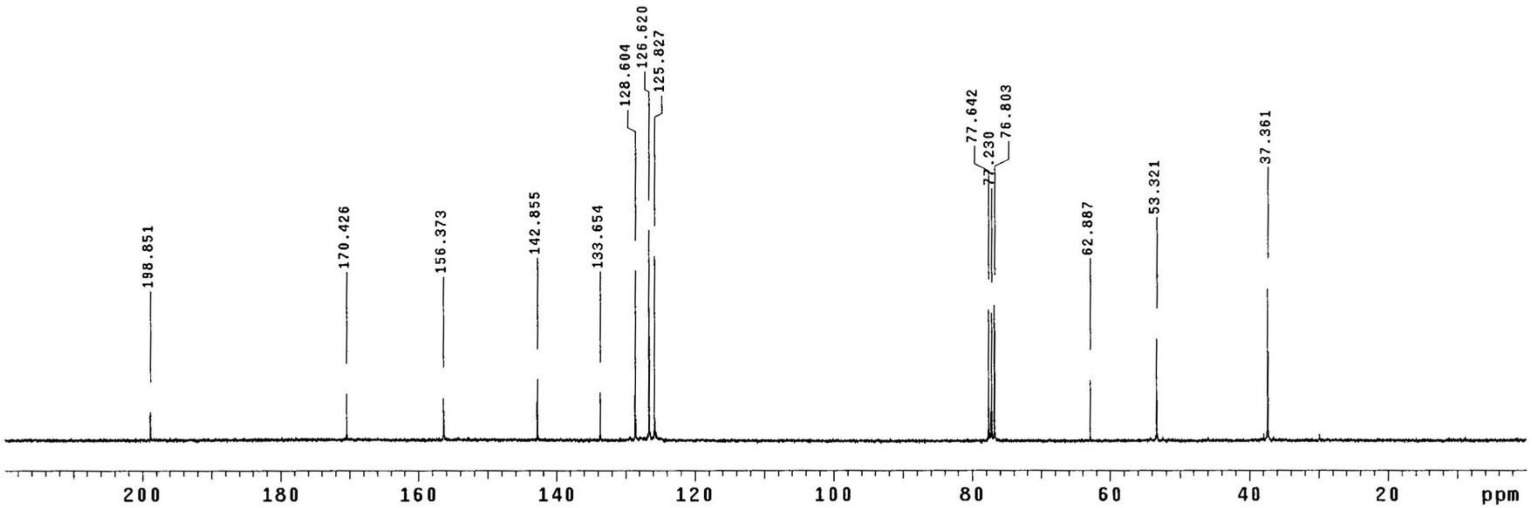

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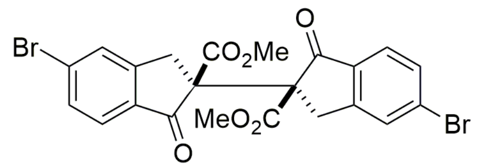


3g

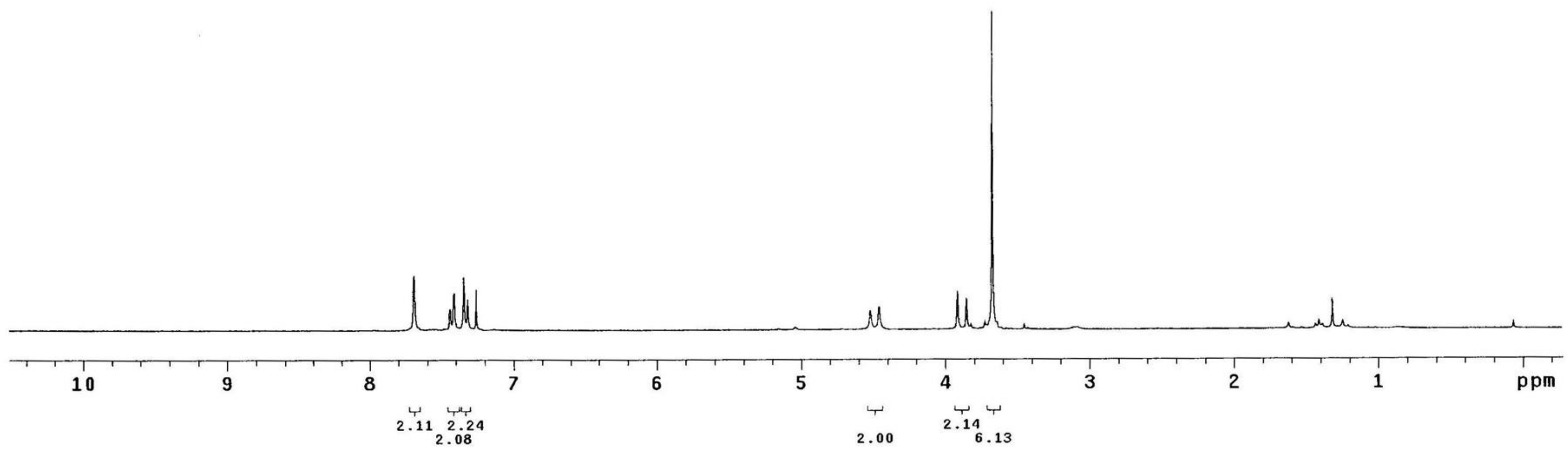
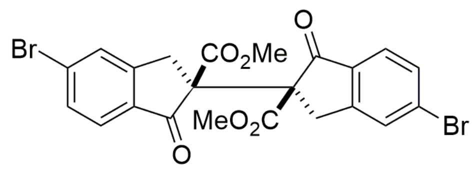
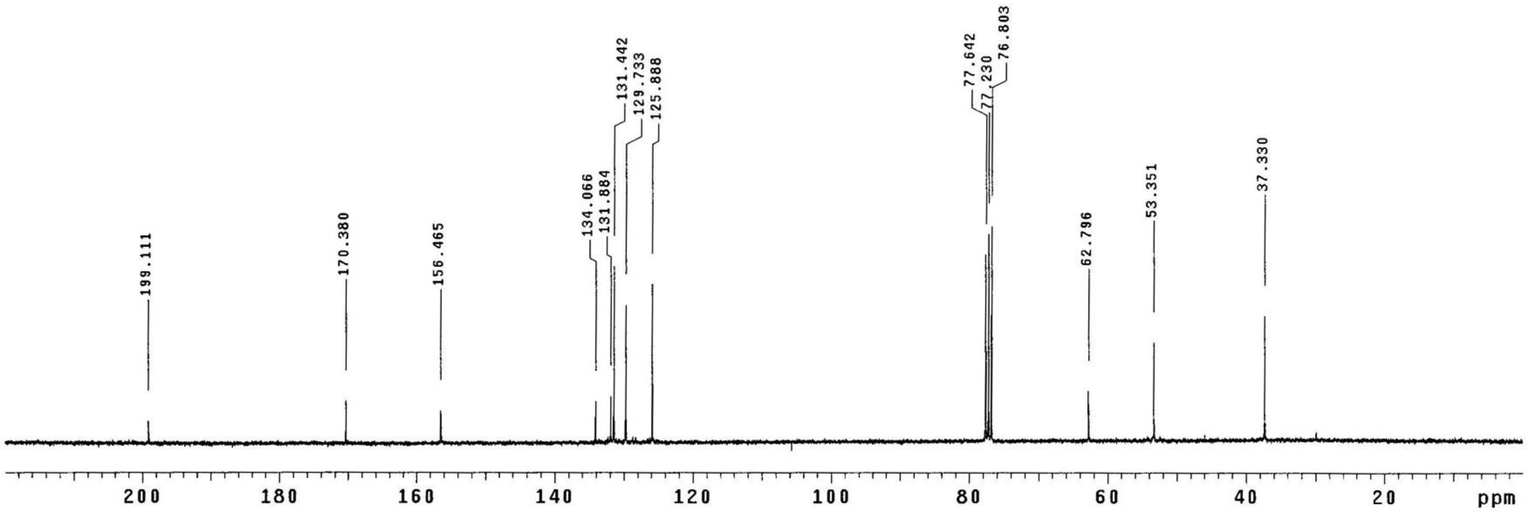


Table 2, entry 7



3g



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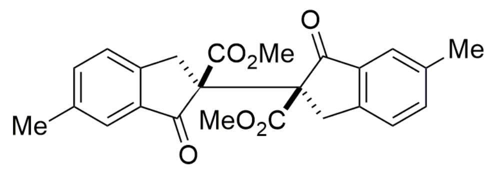
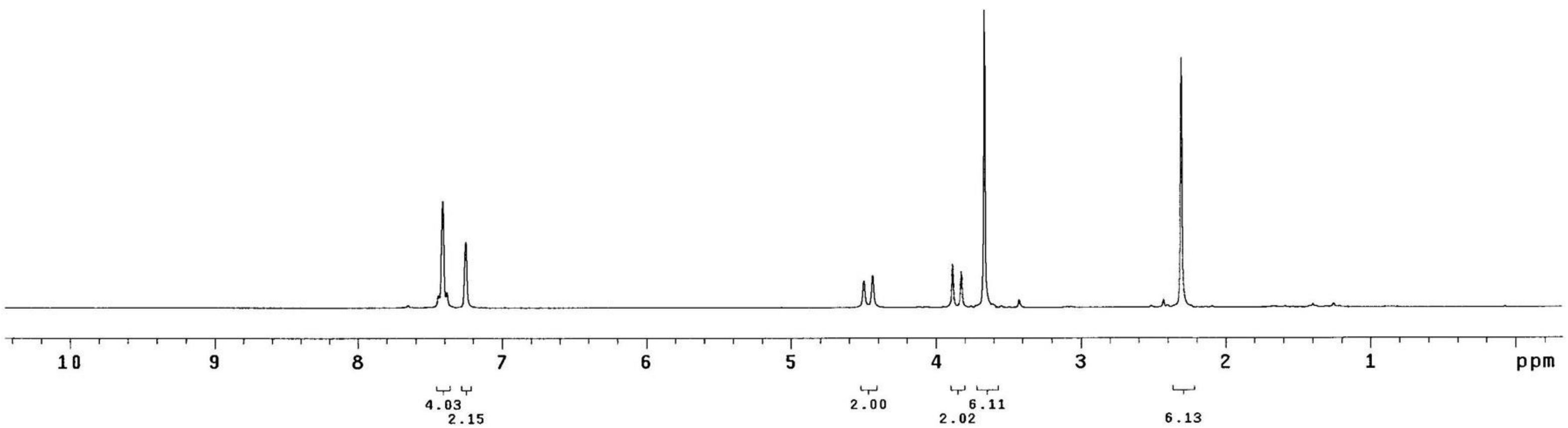


3h



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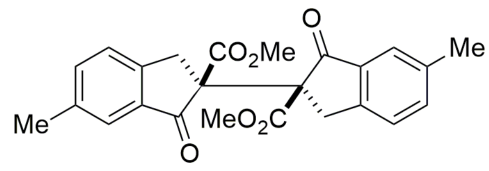
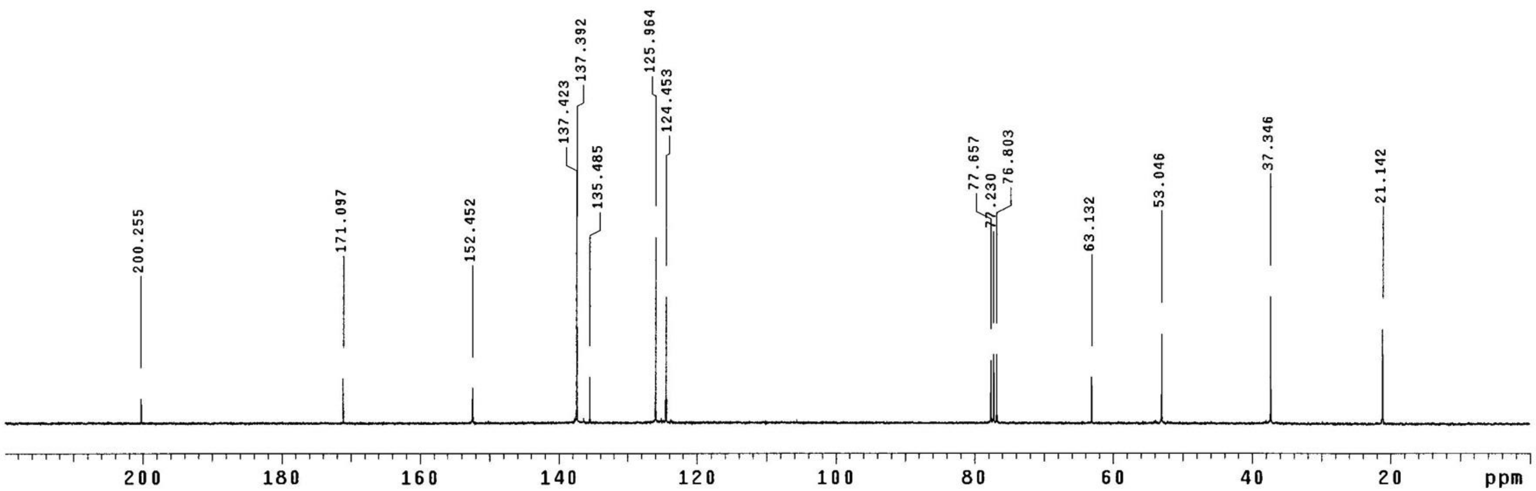


3h



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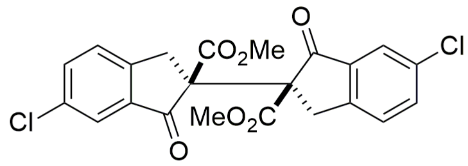
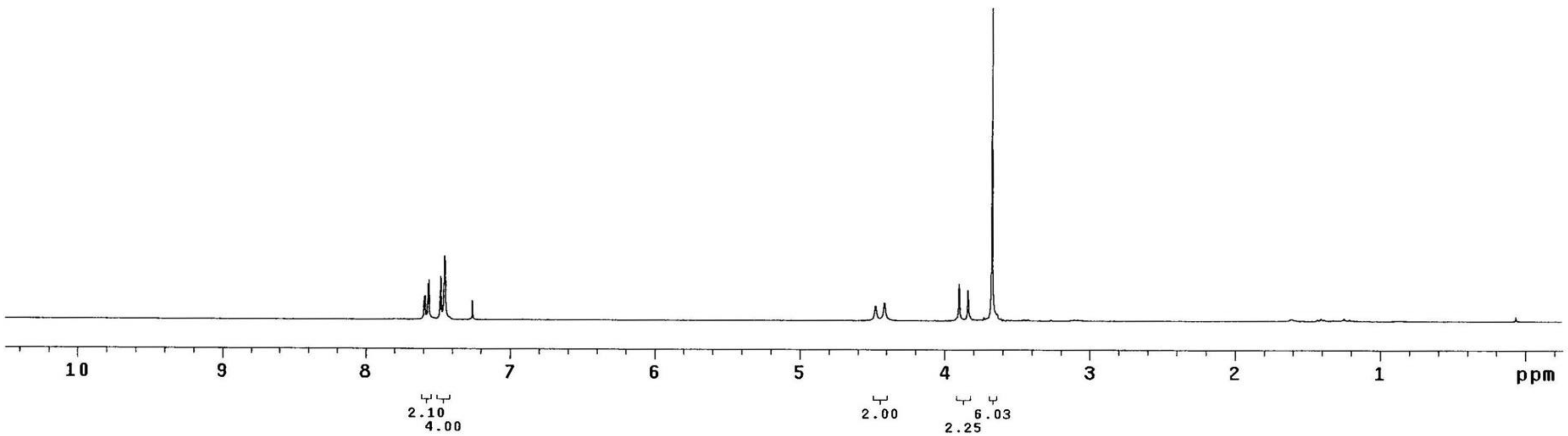


3i



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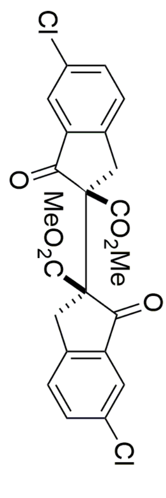
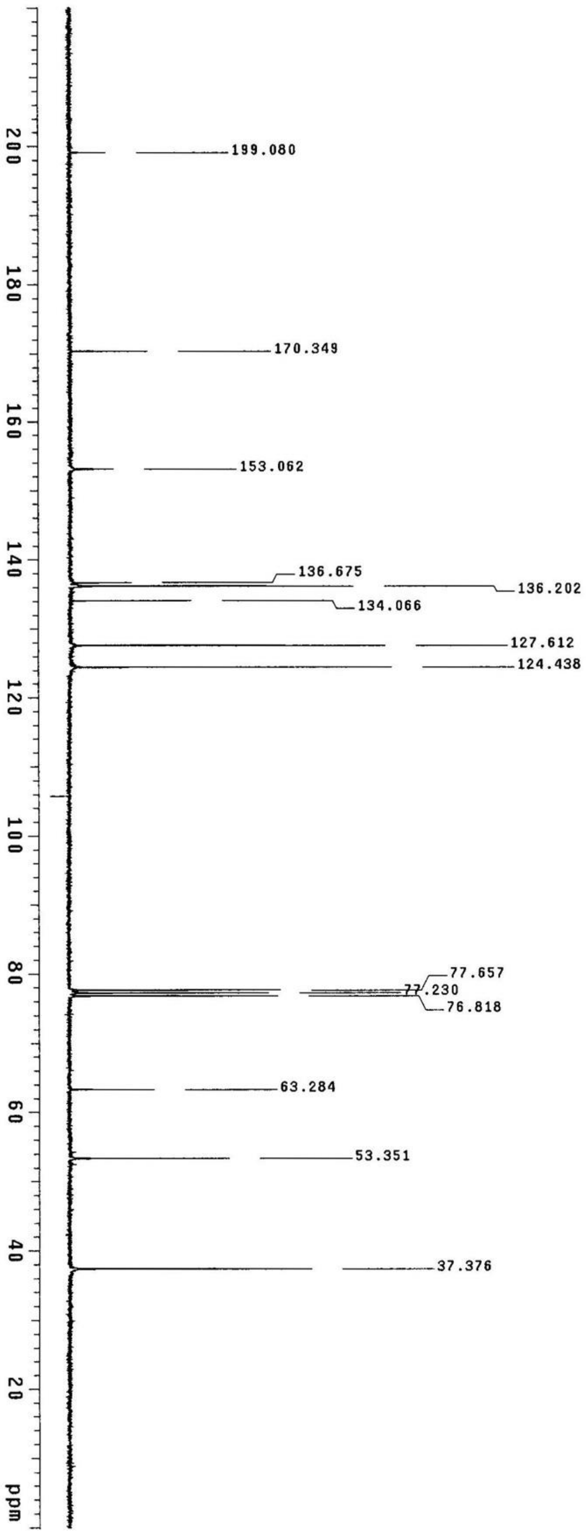


3i



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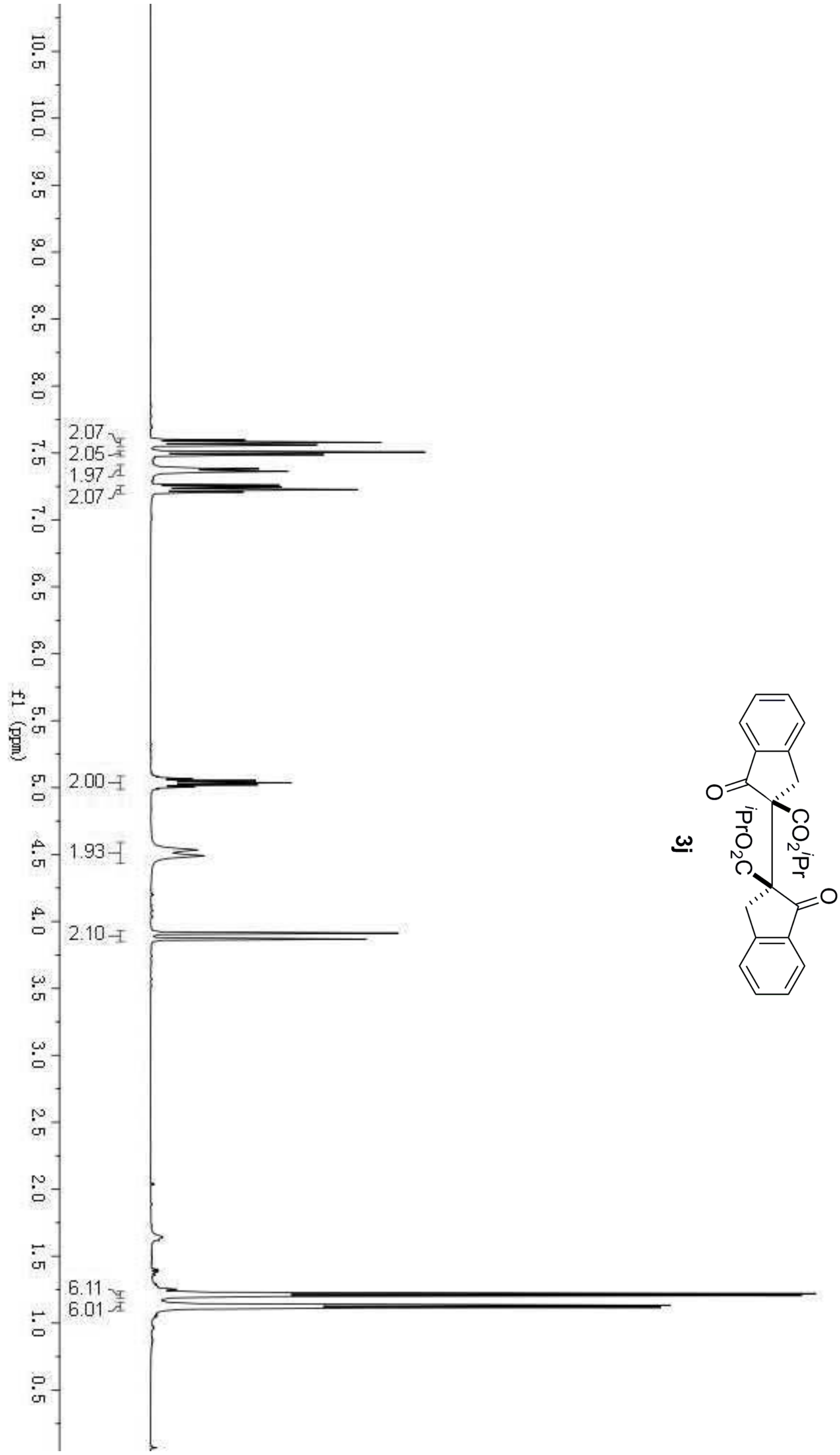


3j

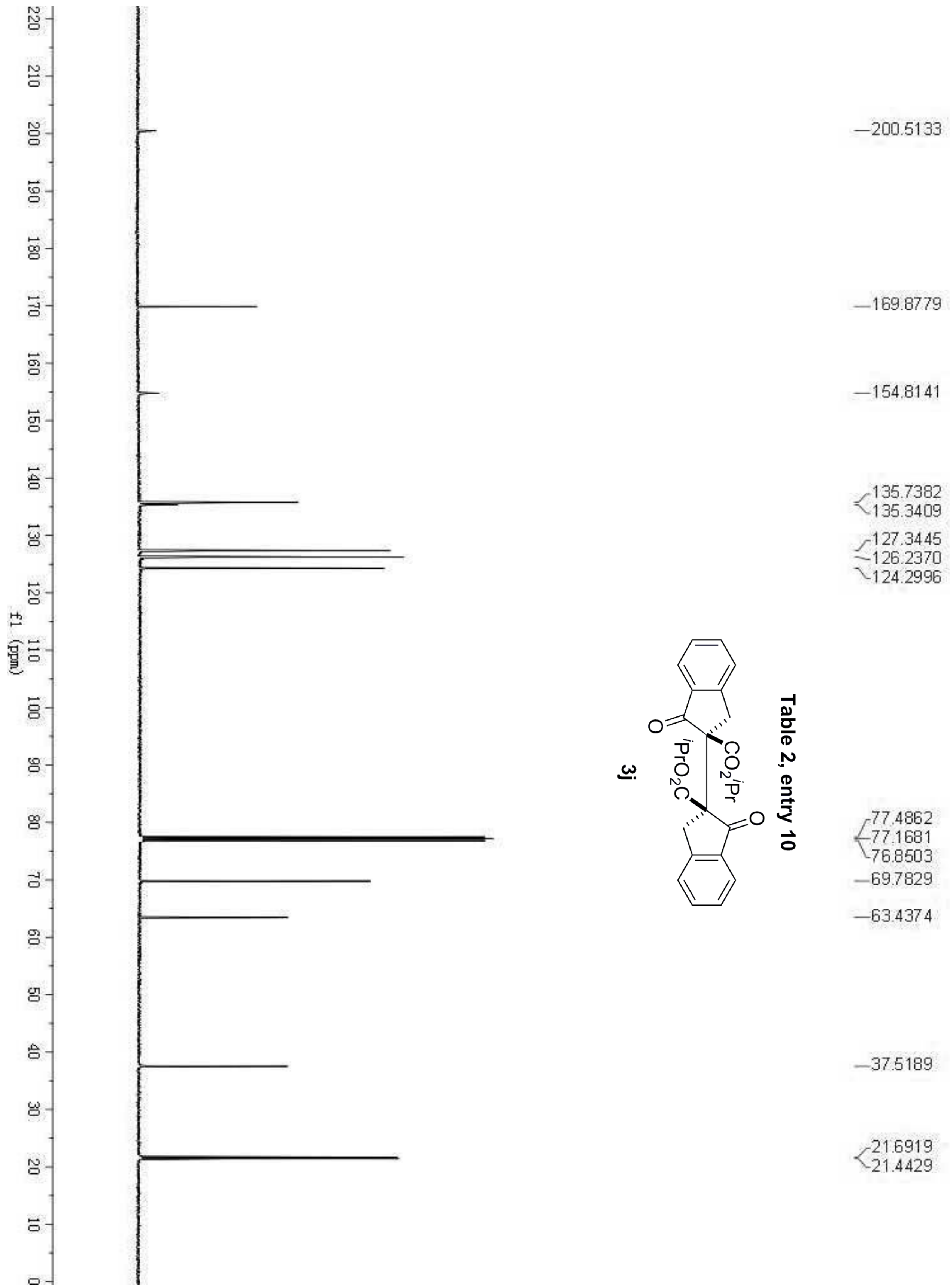
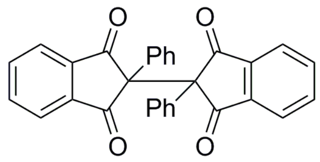
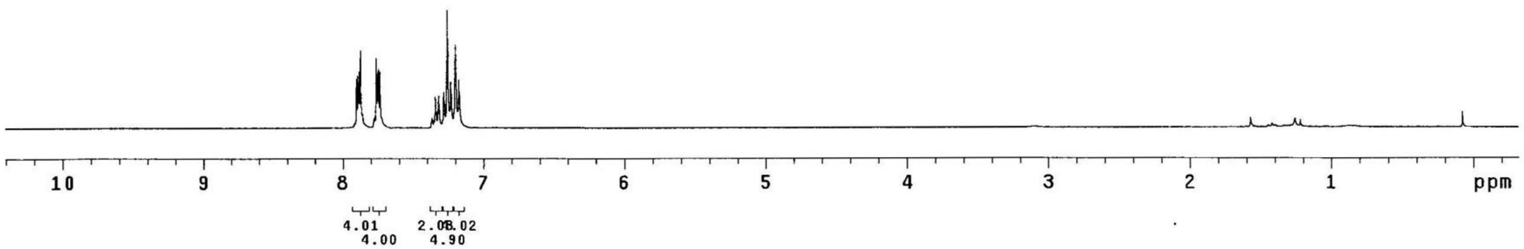

200.5133
169.8779
154.8141
135.7382
135.3409
127.3445
126.2370
124.2996
77.4862
77.1681
76.8503
69.7829
63.4374
37.5189
21.6919
21.4429
f1 (ppm)
Table 2, entry 10
CO2iPr
iPrO2C
3j

Table 2, entry 11



3k



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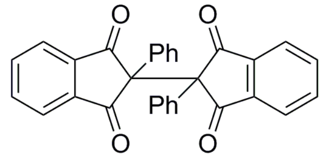
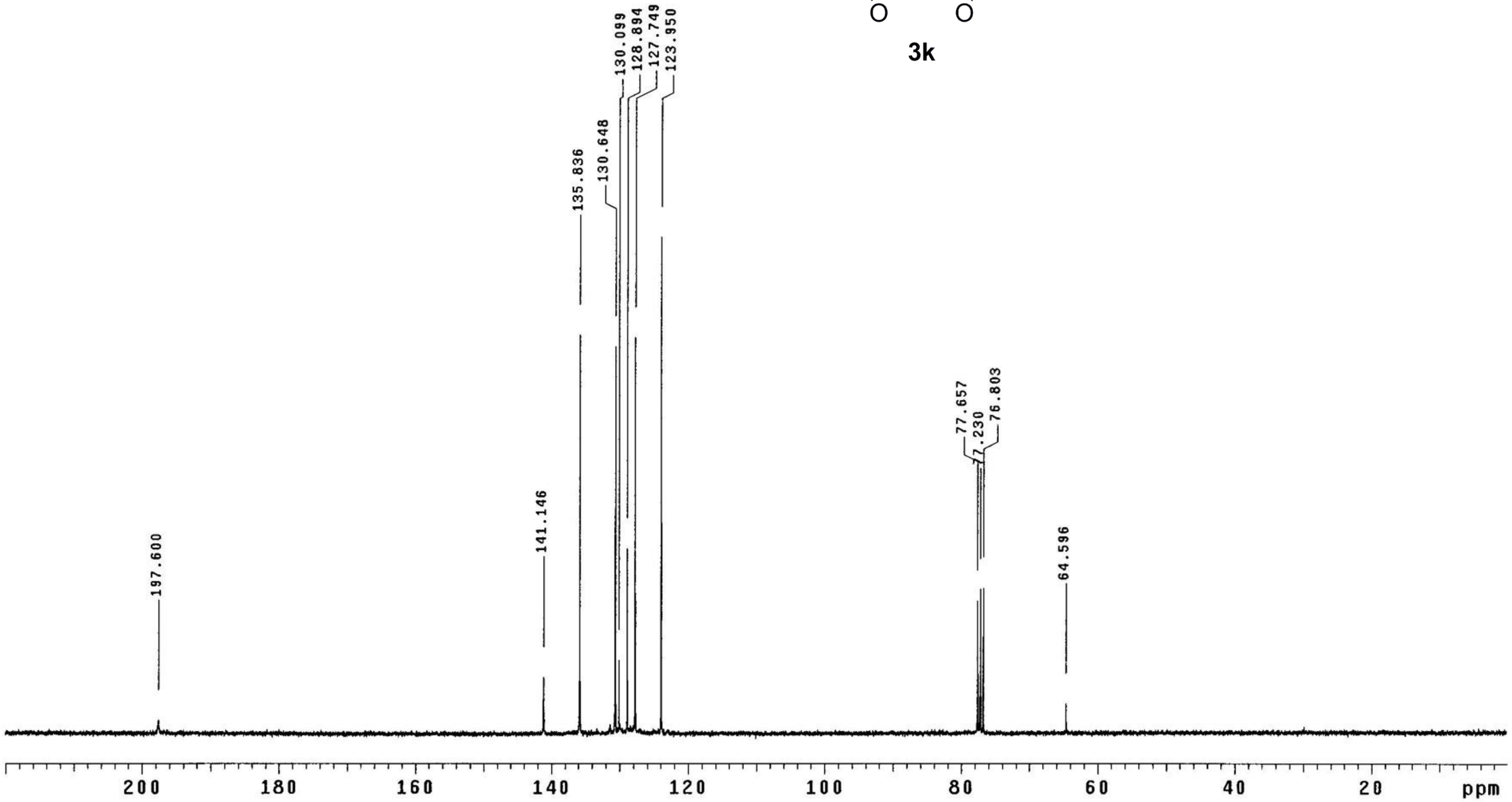
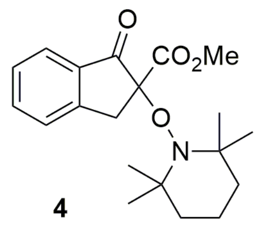


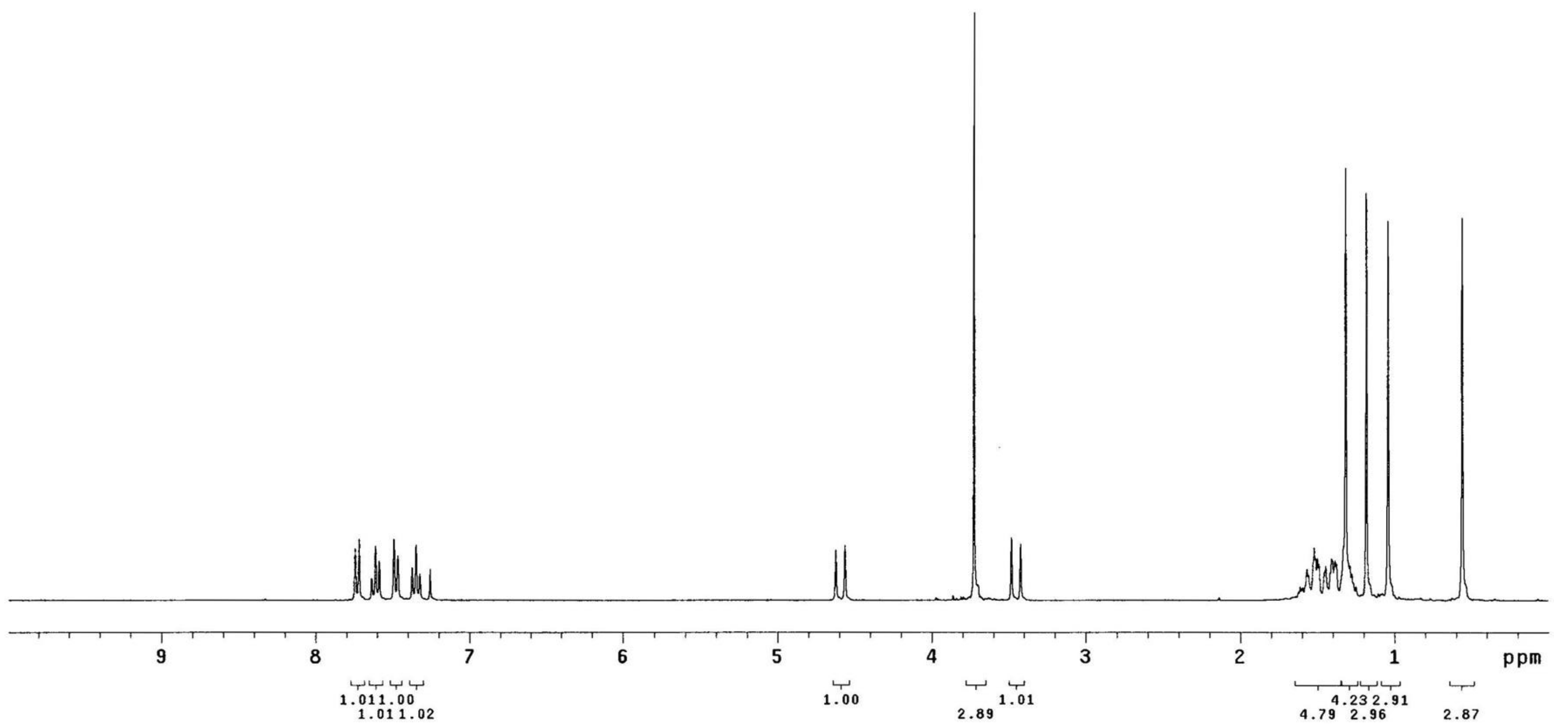
3k



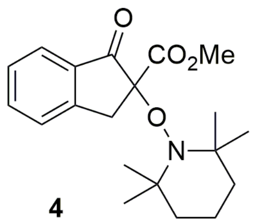
Scheme 3


O
CO2Me
O
N

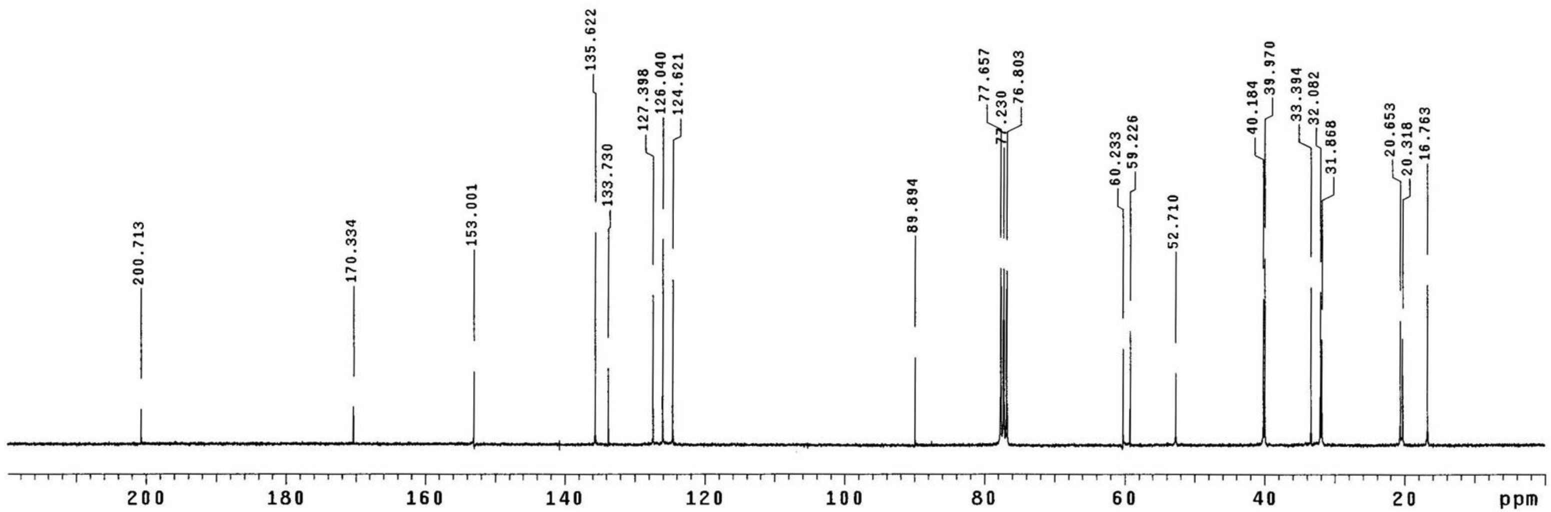
4


9
8
7
6
5
4
3
2
1
ppm
1.01
1.00
1.01
1.02
1.00
2.89
1.01
4.79
4.23
2.96
2.91
2.87

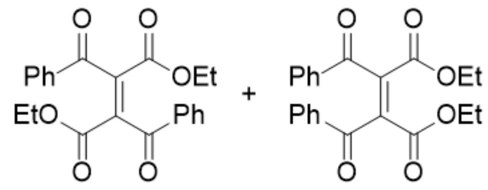
Scheme 3


O
CO_2Me
O
N

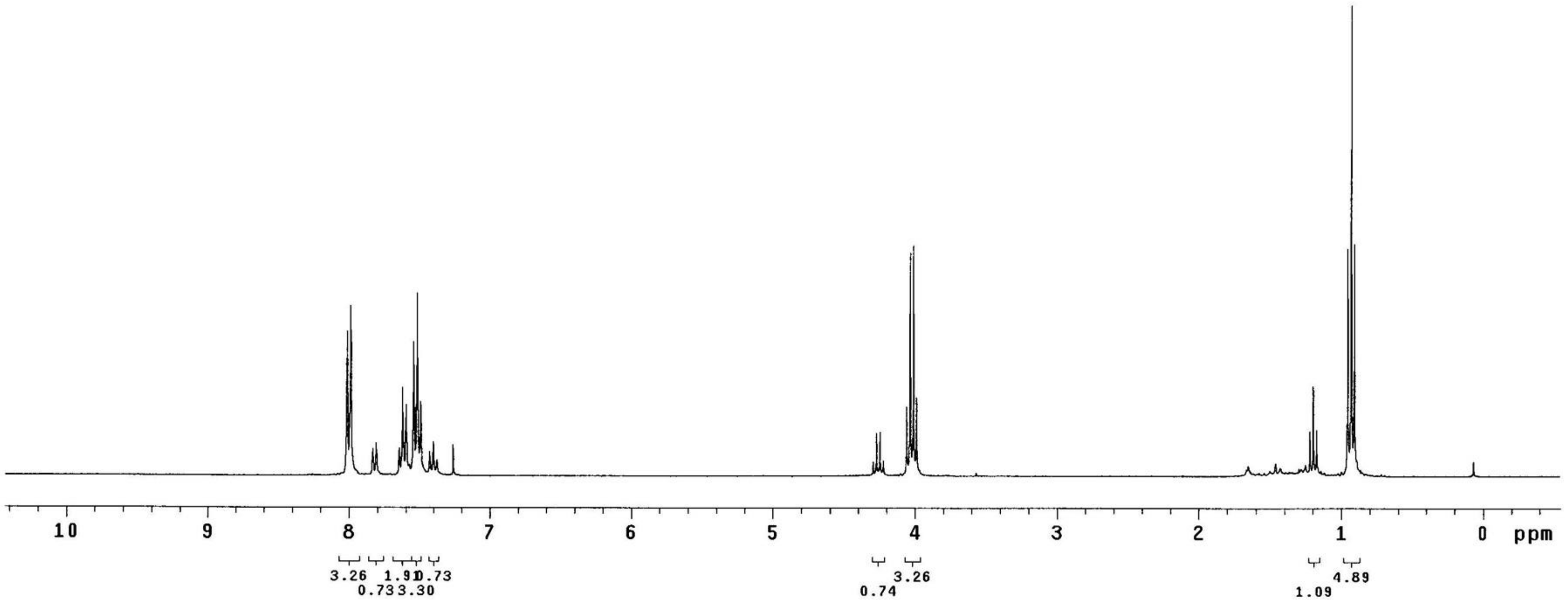
4


200.713
170.334
153.001
135.622
133.730
127.398
126.040
124.621
89.894
77.657
77.230
76.803
60.233
59.226
52.710
40.184
39.970
33.394
32.082
31.868
20.653
20.318
16.763
200
180
160
140
120
100
80
60
40
20
ppm

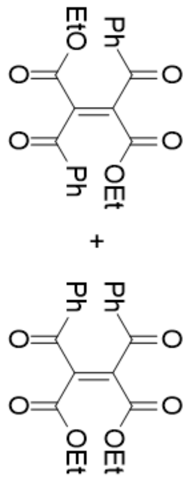
Scheme 4



E-6 *Z*-6



Scheme 4



***E*-6** ***Z*-6**