Supplementary Information

Construction of Functionalized 2,3-Dihydro-1,4-benzoxazines via [5 + 1] Annulation of 2-Halo-1,3-dicarbonyl Compounds with Imines

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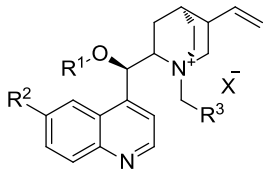
1. General Methods:

NMR spectra were recorded with tetramethylsilane as the internal standard. TLC was performed on glass-backed silica plates. Column chromatography was performed using silica gel (160-200 mesh) eluting with ethyl acetate and petroleum ether. ^{1}H NMR spectra were recorded at 400 MHz, and ^{13}C NMR spectra were recorded at 100 MHz (Bruker Avance). Chemical shifts (δ) are reported in ppm downfield from $CDCl_3$ (δ = 7.26 ppm) or DMSO (δ = 2.50 ppm) for ^{1}H NMR and relative to the central $CDCl_3$ resonance (δ = 77.0 ppm) or DMSO resonance (δ = 39.5 ppm) for ^{13}C NMR spectroscopy. Coupling constants (J) are given in Hz. ESI-HRMS spectrometer was measured with a Finnigan LCQ^{DECA} ion trap mass spectrometer. Optical rotations were measured at 589 nm at 20 °C. Enantiomeric excess was determined by HPLC analysis on Chiralpak AS, IC and OD columns.

2. Screening studies of organocatalytic [5 + 1] annulation

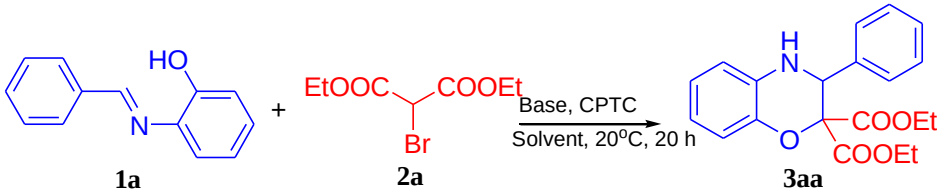


4

4a R^1 = H, R^2 =H , R^3 = Ph , X = Br
4b R^1 = H, R^2 =H , R^3 = Ph , X = Cl
4c R^1 = H, R^2 =H , R^3 = p-CF_3Ph , X = Br
4d R^1 = Allyl, R^2 =H , R^3 = p-CF_3Ph , X = Br
4e R^1 = H, R^2 =H , R^3 = 3,5-$(CF_3)_2$Ph , X = Br
4f R^1 = H, R^2 = H, R^3 = 9-Anthracyl, X = Br
4g R^1 = , R^2 = MeO, R^3 = Ph, X = Br
4h R^1 = H, R^2 = MeO, R^3 = 9-Anthracyl, X = Br

Figure 3. Structures Chiral Phase Transfer Catalysts

TABLE S1. Screening studies of organocatalytic domino reaction of 4-hydroxylcoumarin 2a to α-bromonitroalkene 3a[a].

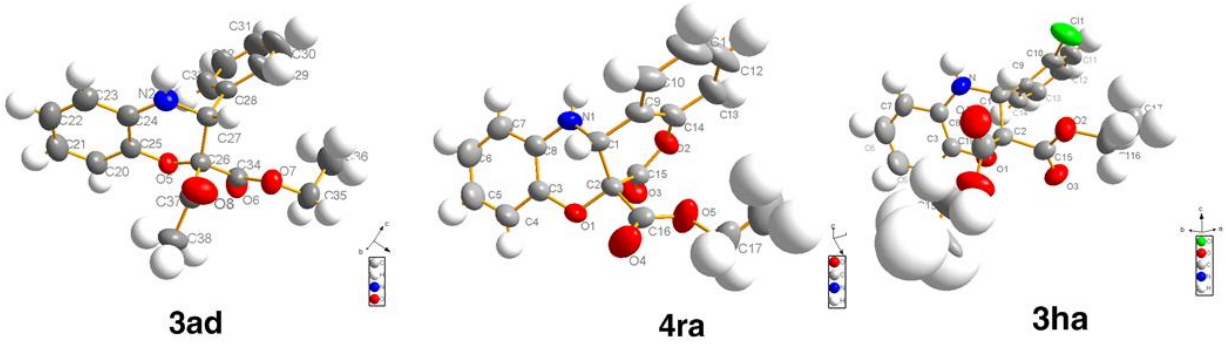


1a **2a** **3aa**

entry	catalyst	base	solvent	yield[b](%)	ee[c](%)
1	**4a**	K_2CO_3	CH_3CN	61	37
2	**4b**	K_2CO_3	CH_3CN	71	27
3	**4c**	K_2CO_3	CH_3CN	67	45
4	**4d**	K_2CO_3	CH_3CN	60	26
5	**4e**	K_2CO_3	CH_3CN	68	0
6	**4f**	K_2CO_3	CH_3CN	73	5
7	**4g**	K_2CO_3	CH_3CN	62	33
8	**4h**	K_2CO_3	CH_3CN	69	0
9	**4c**	**K_2CO_3**	**$CHCl_3$**	**66**	**81**
10	**4c**	Cs_2CO_3	$CHCl_3$	59	71
11	**4c**	CsOH	$CHCl_3$	46	35
12	**4c**	LiOH	$CHCl_3$	61	0
13	**4c**	KOH	$CHCl_3$	42	70

[a] Otherwise noted, reactions performed with 0.10 mmol of **1a**, 0.20 mmol of **2a**, 10 mol% of **4**, 100 mol% of Base in 1 mL solvent at 20 °C. [b] Isolated yield. [c] Determined by chiral HPLC analysis.

3. X-ray data of racemic 3ad, 4ra and enantiopure 3ha



Molecular Structure of 3ad, 4ra and enantiopure 3ha, (ellipsoids with 50% probability)

Crystal data for 3ad (No. CCDC 881882) $C_{19}H_{19}NO_4$ (325.35), Triclinic, space group P-1, *a* = 8.9397(6) Å, *b* = 11.7671(8)) Å, *c* = 17.0494(12) Å, *U* = 1711.5(2) Å^3, Z = 2, specimen 0.254 x 0.0177 x 0.123 mm^3, *T* = 296(2) K, SIEMENS P4 diffractometer, absorption coefficient 0.089 mm^{-1}, reflections collected 27342, independent 7780 [R(int) = 0.0278], refinement by Full-matrix least-squares on F^2, data/restraints/parameters 7780 / 0 / 434, goodness-of-fit on F^2 = 1.065, final *R* indices [$I>2\sigma(I)$] R1 = 0.0550, wR2 = 0.1548, *R* indices (all data) R1 = 0.0842, wR2 = 0.1770, largest diff. peak and hole 0.495 and - 0.412 Å^{-3}.

Crystal data for 4ra (No. CCDC 881884) $C_{18}H_{15}NO_5$ (325.31), Monoclinic, space group Cc, *a* = 12.4785 Å, *b* = 13.3392(5) Å, *c* = 10.0137(4) Å, *U* = 1623.97(10) Å^3, Z = 10, specimen 0.48 x 0.32 x 0.28 mm^3, *T* = 296(2) K, SIEMENS P4 diffractometer, absorption coefficient 0.098 mm^{-1}, reflections collected 6854, independent 3230 [R(int) = 0.0179], refinement by Full-matrix least-squares on F^2, data/restraints/parameters 3230 / 2 / 218, goodness-of-fit on F^2 = 1.046, final *R* indices [$I>2\sigma(I)$] R1 = 0.0386, wR2 = 0.1052, *R* indices (all data) R1 = 0.0447, wR2 = 0.1113, largest diff. peak and hole 0.204 and -0.178 Å^{-3}.

Crystal data for 3ha (No. CCDC 881883**)** $C_{20}H_{20}ClNO_5$ (389.82), Tetragonal, space group P4(3), *a* = 9.0853(7) Å, *b* = 9.0853(7) Å, *c* = 23.592(4) Å, *U* = 1947.4(4) Å^3, Z = 12, specimen 0.46 x 0.38 x 0.28 mm^3, *T* = 296(2) K, SIEMENS P4 diffractometer, absorption coefficient 0.226 mm^{-1}, reflections collected 8605, independent 3959 [R(int) = 0.0243], refinement by Full-matrix least-squares on F^2, data/restraints /parameters 3959/1/245, goodness-of-fit on F^2 = 1.047, final *R* indices [$I>2\sigma(I)$] R1 = 0.0573, wR2 = 01517, *R* indices (all data) R1 = 0.0975, wR2 = 0.1780, largest diff. peak and hole 0.402 and -0.306 Å^{-3}.

4. ^{1}H NMR and ^{13}C NMR data of 3ra-3za

Synthesis of 2,3-Dihydro-1,4-Benzoxazines **3ra-3za**

General procedure: **1r** (21.3 mg, 0.10 mmol), **2a** (47.4 mg, 0.20 mmol), TBAB (6.44 mg, 0.02 mmol) and KOH (5.6 mg, 0.10 mmol) were stirred in CH_3CN (1 mL) at room temperature for 20 h. Then flash chromatography on silica gel (10% ethylacetate/ petroleum ether) gave product **3ra** as a white solid (24.1 mg, 65% yield).

Diethyl 3-(2-hydroxyphenyl)-3,4-dihydro-2H-benzo[b][1,4] oxazine-2,2-dicarboxylate (3ra): white solid, mp: 168 – 170 °C; 24.1 mg, yield 65%; ^{1}H NMR (400 MHz, DMSO) δ 9.70 (s, 1H), 6.99 (t, *J* = 7.6 Hz, 2H), 6.88 (d, *J* = 7.9 Hz, 1H), 6.78 – 6.67 (m, 2H), 6.58 (t, *J* = 8.8 Hz, 2H), 6.52 (t, *J* = 5.5 Hz, 2H), 5.64 (d, *J* = 4.2 Hz, 1H), 4.16–4.05 (m, 2H), 4.03 – 3.90 (m, 2H), 1.03 (dd, *J* = 8.8, 5.3 Hz, 3H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 166.9, 165.0, 155.0, 140.5, 133.1, 128.7, 128.1, 126.7, 122.9, 119.2, 117.0, 116.9, 115.0, 114.7, 82.6, 62.3, 62.1, 47.9, 14.1, 13.8; IR (KBr) cm^{-1} 3419, 2978, 1749, 1723, 1615, 1450, 1443, 1274, 1238, 1200, 1154, 837, 739; ESI-HRMS: calcd. for $C_{20}H_{21}NO_6$+H 372.1442, found 372.1445.

Diethyl 3-(2-hydroxyphenyl)-6-methyl-3,4-dihydro-2H-benzo[b][1,4]oxazine-2,2-dicarboxylate (3sa): white solid, mp: 176 – 178 °C; 27.3 mg, yield 71%; ^{1}H NMR (400 MHz, DMSO) δ 9.67 (s, 1H), 6.99 (t, *J* = 7.7 Hz, 2H), 6.75 (d, *J* = 8.0 Hz, 2H), 6.58 (t, *J* = 7.5 Hz, 1H), 6.44 (d, *J* = 4.1 Hz, 1H), 6.37 (s, 1H), 6.31 (d, *J* = 8.0 Hz, 1H), 5.61 (d, *J* = 4.2 Hz, 1H), 4.09 (q, *J* = 6.8 Hz, 2H), 4.03-3.90 (m, 2H), 2.09 (s, 3H), 1.04 (t, *J* = 7.0 Hz, 3H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 167.0, 165.0, 155.0, 138.4, 132.8, 131.6, 128.7, 128.2, 126.7, 119.2, 117.6, 116.6, 115.0, 110.0, 82.6, 62.2, 62.0, 47.8, 21.0, 14.2, 13.8; IR (KBr) cm^{-1} 3434, 2987, 1749, 1726, 1623, 1452, 1438, 1277, 1235, 1205, 1151, 837, 742; ESI-HRMS: calcd. for $C_{21}H_{23}NO_6$+H 386.1598, found 386.1594.

Diethyl 3-(2-hydroxyphenyl)-7-methyl-3,4-dihydro-2H-benzo[b][1,4]oxazine-2,2-dicarboxylate (3ta): white solid, mp: 92 – 94 °C; 21.9 mg, yield 57%; ^{1}H NMR (400 MHz, DMSO) δ 9.68 (s, 1H), 6.98 (t, *J* = 7.1 Hz, 2H), 6.76 (d, *J* = 7.7 Hz, 1H), 6.71 (s,1H), 6.59 – 6.51 (m, 2H), 6.46 (d, *J* = 8.0 Hz, 1H), 6.25 (d, *J* = 4.1 Hz, 1H), 5.59 (d, *J* = 4.1 Hz, 1H), 4.15 – 4.05 (m, 2H), 4.03 – 3.93 (m, 2H), 2.14 (s, 3H), 1.05 (t, *J* = 7.1 Hz, 3H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 166.9, 165.0, 155.0, 140.5, 130.4, 128.6, 128.0, 126.7, 126.1, 123.3, 119.1, 117.2, 115.0, 114.8, 82.7, 62.3, 56.4, 47.9, 20.6,

14.2, 13.8; IR (KBr) cm^{-1} 3432, 2986, 1741, 1730, 1625, 1454, 1434, 1276, 1240, 1200, 1158, 836, 739; ESI-HRMS: calcd. for $C_{21}H_{23}NO_6$+H 386.1598, found 386.1592.

Diethyl 6-chloro-3-(2-hydroxyphenyl)-3,4-dihydro-2H-benzo[b][1,4]oxazine-2,2-dicarboxylate (3ua): white solid, mp: 112 – 114 °C; 25.9 mg, yield 64%; ^{1}H NMR (400 MHz, DMSO) δ 9.76 (s, 1H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.96 – 6.90 (m, 3H), 6.78 (d, *J* = 7.9 Hz, 1H), 6.64 – 6.59 (m, 2H), 6.53 – 6.51 (m, 1H), 5.64 (d, *J* = 4.0 Hz, 1H), 4.16 – 4.08 (m, 2H), 4.02 – 3.93 (m, 2H), 1.05 (t, *J* = 7.0 Hz, 3H), 0.93 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 166.5, 164.6, 155.1, 139.2, 134.5, 128.9, 127.8, 126.6, 126.1, 119.3, 118.2, 116.2, 115.1, 113.5, 82.6, 62.5, 62.2, 47.6, 14.2, 13.8; IR (KBr) cm^{-1} 3414, 2987, 1748, 1729, 1614, 1501, 1447, 1281, 1229, 1201, 1154, 846, 744; ESI-HRMS: calcd. for $C_{20}H_{20}ClNO_6$+H 406.1052, found 406.1047.

Diethyl 7-chloro-3-(2-hydroxyphenyl)-3,4-dihydro-2H-benzo[b][1,4]oxazine-2,2-dicarboxylate (3va): white solid, mp: 134 – 136 °C; 21.8 mg, yield 54%; ^{1}H NMR (400 MHz, DMSO) δ 9.75 (s, 1H), 7.03 – 6.95 (m, 3H), 6.77 (dd, *J* = 10.2, 5.0 Hz, 3H), 6.60 (dd, *J* = 12.8, 8.0 Hz, 2H), 5.65 (d, *J* = 4.1 Hz, 1H), 4.19 – 4.06 (m, 2H), 4.05 – 3.92 (m, 2H), 1.05 (t, *J* = 7.0 Hz, 3H), 0.94 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 166.5, 164.5, 155.1, 141.0, 132.3, 128.9, 127.8, 126.2, 122.7, 119.9, 119.3, 116.7, 115.5, 115.1, 82.7, 62.5, 62.2, 47.9, 14.1, 13.8; IR (KBr) cm^{-1} 3413, 2985, 1750, 1730, 1612, 1500, 1449, 1281, 1230, 1202, 1154, 846, 744; ESI-HRMS: calcd. for $C_{20}H_{20}ClNO_6$+H 406.1052, found 406.1045.

Diethyl 3-(2-hydroxy-4-methoxyphenyl)-3,4-dihydro-2H-benzo[b][1,4]oxazine-2,2-dicarboxylate (3wa): white solid, mp: 176 – 178 °C; 28.8 mg, yield 72%; ^{1}H NMR (400 MHz, DMSO) δ 9.72 (s, 1H), 6.88 (t, *J* = 7.5 Hz, 2H), 6.70 (t, *J* = 7.6 Hz, 1H), 6.56 – 6.48 (m, 2H), 6.44 (d, *J* = 4.0 Hz, 1H), 6.32 (s, 1H), 6.20 (d, *J* = 8.7 Hz, 1H), 5.54 (d, *J* = 4.0 Hz, 1H), 4.12 – 4.04 (m, 2H), 4.03 – 3.91 (m, 2H), 3.60 (s, 3H), 1.03 (t, *J* = 7.0 Hz, 3H), 0.96 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 166.9, 164.9, 159.7, 156.1, 140.5, 133.2, 128.8, 122.8, 119.3, 116.9, 114.7, 104.7, 100.8, 82.8, 62.2, 62.0, 55.2, 47.6, 14.1, 13.9; IR (KBr) cm^{-1} 3425, 2983, 1752, 1727, 1615, 1498, 1440, 1278, 1238, 1208, 1145, 843, 749; ESI-HRMS: calcd. for $C_{21}H_{23}NO_7$+H 402.1547, found 402.1563.

Diethyl 3-(2-hydroxy-5-methylphenyl)-3,4-dihydro-2H-benzo[b][1,4]oxazine-2,2-dicarboxylate (3xa): white solid, mp: 168 – 170 °C; 30.4 mg, yield 79%; ^{1}H NMR (400 MHz, DMSO) δ 9.44 (s, 1H),

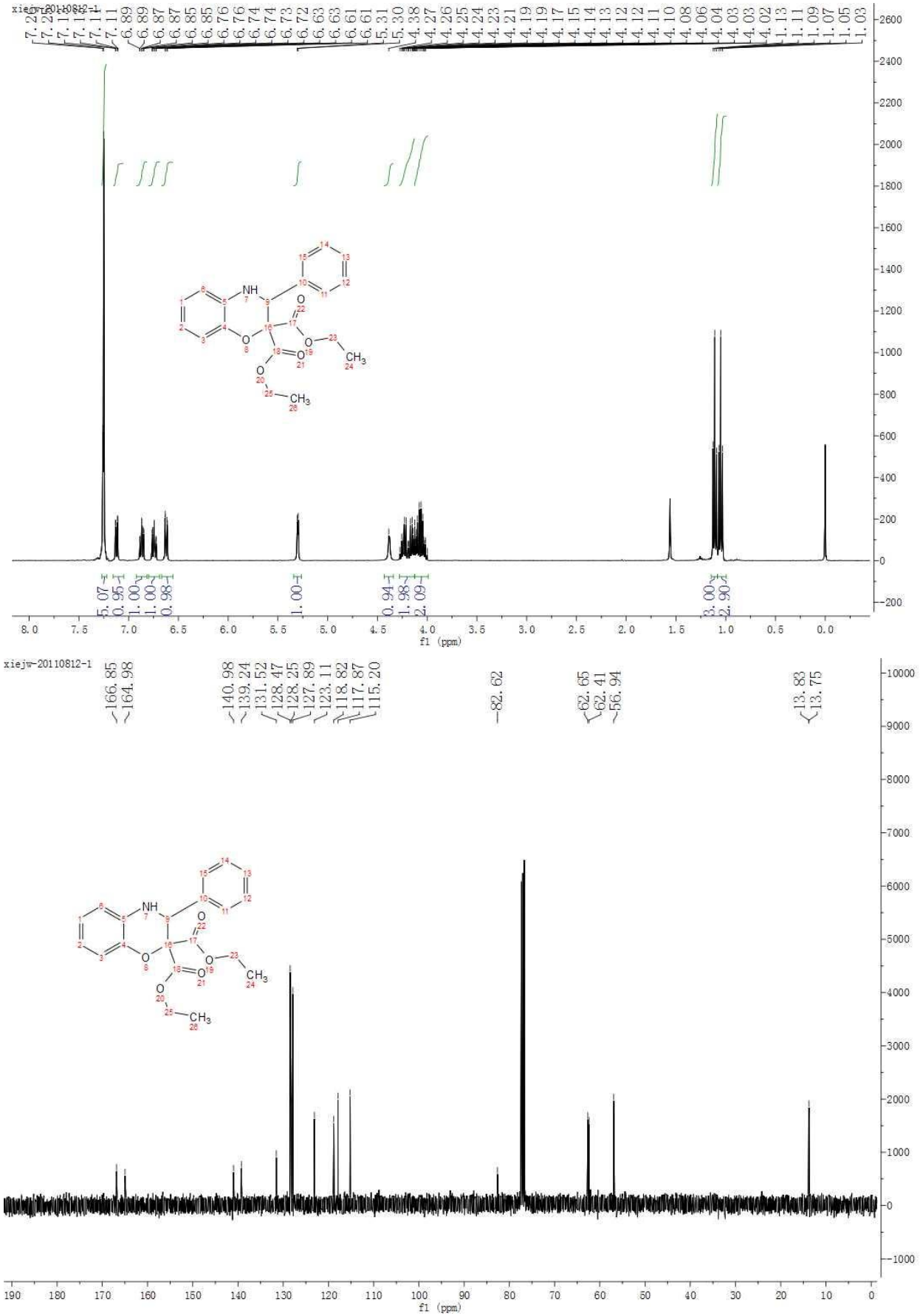
6.88 (d, *J* = 7.8 Hz, 1H), 6.80 (d, *J* = 7.1 Hz, 2H), 6.71 (t, *J* = 7.1 Hz, 1H), 6.65 (d, *J* = 8.1 Hz, 1H), 6.56 (d, *J* = 7.6 Hz, 1H), 6.53 – 6.48 (m, 2H), 5.59 (s, 1H), 4.12 – 3.95 (m, 2H), 3.98 (dd, *J* = 16.6, 8.3 Hz, 2H), 1.98 (s, 3H), 1.01 (t, *J* = 6.9 Hz, 3H), 0.94 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 166.8, 165.0, 152.8, 140.4, 133.2, 129.1, 128.4, 127.1, 126.3, 122.9, 116.9, 114.9, 114.6, 82.6, 62.3, 62.0, 48.2, 20.9, 14.1, 13.8; IR (KBr) cm^{-1} 3426, 2985, 1748, 1723, 1616, 1451, 1441, 1277, 1240, 1203, 1151, 846, 733; ESI-HRMS: calcd. for $C_{21}H_{23}NO_6$+H 386.1598, found 386.1583.

Diethyl 3-(5-chloro-2-hydroxyphenyl)-3,4-dihydro-2H-benzo[b][1,4]oxazine-2,2-dicarboxylate (3ya): white solid, mp: 162 – 164 °C; 22.6 mg, yield 56%; ^{1}H NMR (400 MHz, DMSO) δ 10.08 (s, 1H), 7.07 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.92 – 6.91 (m, 2H), 6.76 (dd, *J* = 17.5, 8.2 Hz, 2H), 6.66 (d, *J* = 4.4 Hz, 1H), 6.60 – 6.53 (m, 2H), 5.58 (d, *J* = 4.4 Hz, 1H), 4.15 – 4.06 (m, 2H), 4.04 – 3.94 (m, 2H), 1.01 (t, *J* = 7.2 Hz, 3H), 0.97 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 166.6, 164.9, 154.0, 140.2, 132.7, 128.6, 127.7, 123.2, 122.6, 117.1, 116.7, 114.8, 82.2, 62.4, 62.3, 56.4, 48.1, 19.0, 14.1, 13.8; IR (KBr) cm^{-1} 3423, 2980, 1750, 1727, 1612, 1500, 1451, 1279, 1225, 1204, 1161, 841, 737; ESI-HRMS: calcd. for $C_{20}H_{20}ClNO_6$+H 406.1052, found 406.1077.

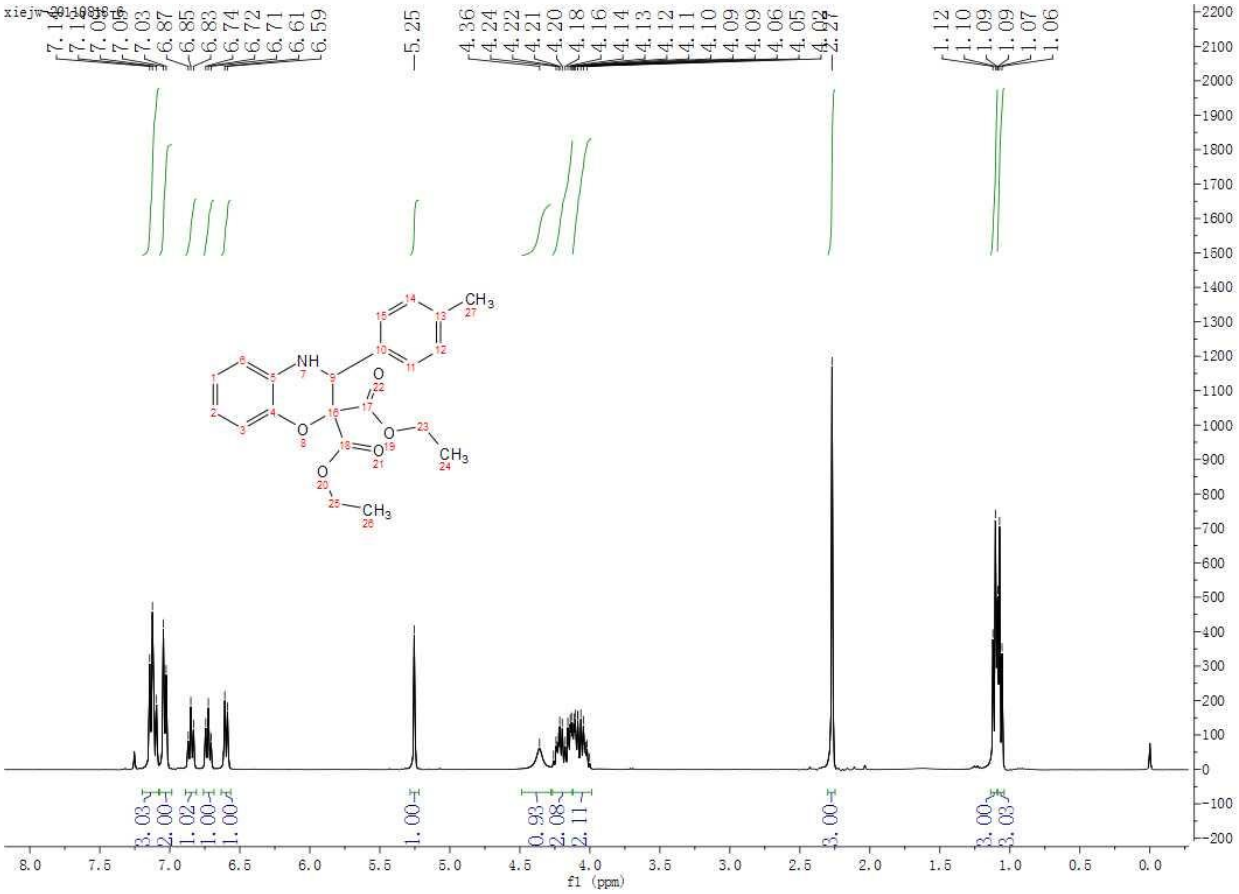
Diethyl 3-(5-bromo-2-hydroxyphenyl)-3,4-dihydro-2H-benzo[b][1,4]oxazine-2,2-dicarboxylate (3za): white solid, mp: 159 – 160 °C; 22.9 mg, yield 51%; ^{1}H NMR (400 MHz, DMSO) δ 10.11 (s, 1H), 7.18 (dd, *J* = 8.6, 2.3 Hz, 1H), 7.05 (d, *J* = 2.1 Hz, 1H), 6.91 (d, *J* = 7.9 Hz, 1H), 6.75 (t, *J* = 8.1 Hz, 2H), 6.65 (d, *J* = 4.4 Hz, 1H), 6.60 – 6.53 (m, 2H), 5.57 (d, *J* = 4.4 Hz, 1H), 4.15 – 3.94 (m, 4H), 1.03 (t, *J* = 7.5 Hz, 6H); ^{13}C NMR (100 MHz, DMSO) δ 166.6, 154.5, 140.2, 132.7, 131.4, 130.5, 129.2, 123.2, 117.3, 117.1, 114.8, 110.3, 82.2, 62.4, 62.3, 56.4, 48.2, 19.0, 14.1, 13.8; IR (KBr) cm^{-1} 3426, 2981, 1750, 1725, 1612, 1450, 1447, 1276, 1233, 1207, 1159, 844, 745; ESI-HRMS: calcd. for $C_{20}H_{20}BrNO_6$+H 450.0547, found 450.0533.

5. ^{1}H NMR and ^{13}C NMR spectra

3aa



3ba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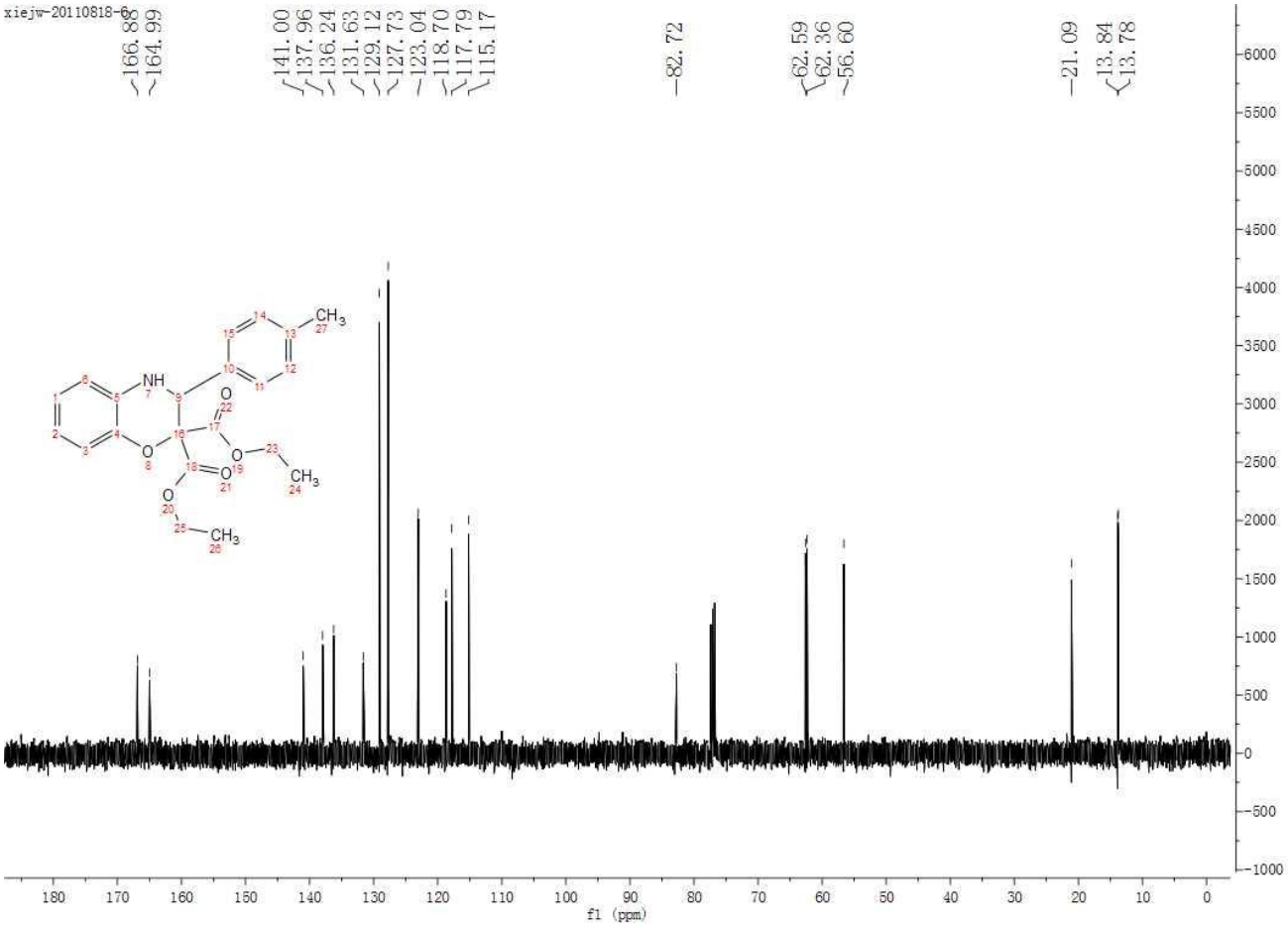
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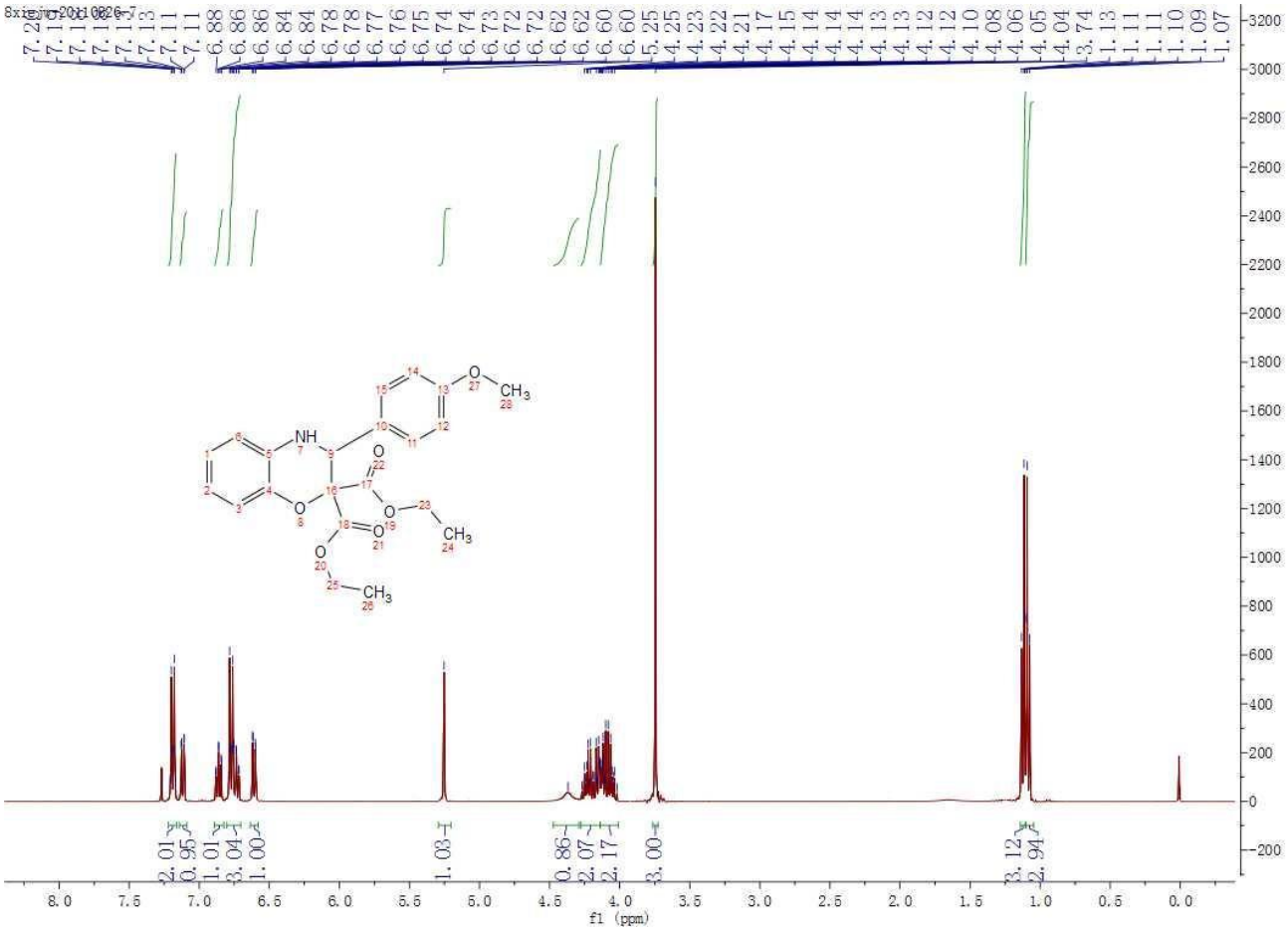
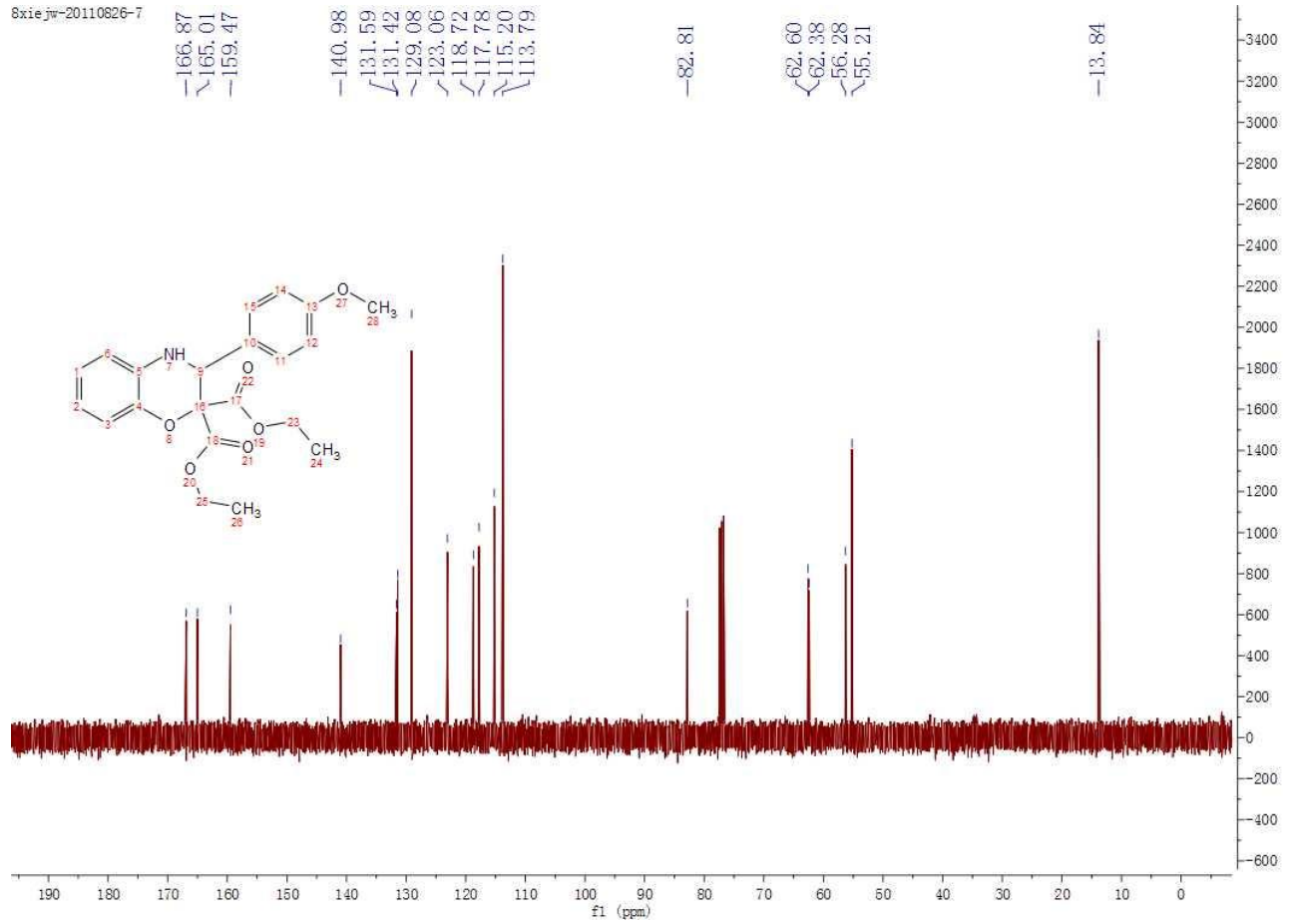
f1 (ppm)

3ca





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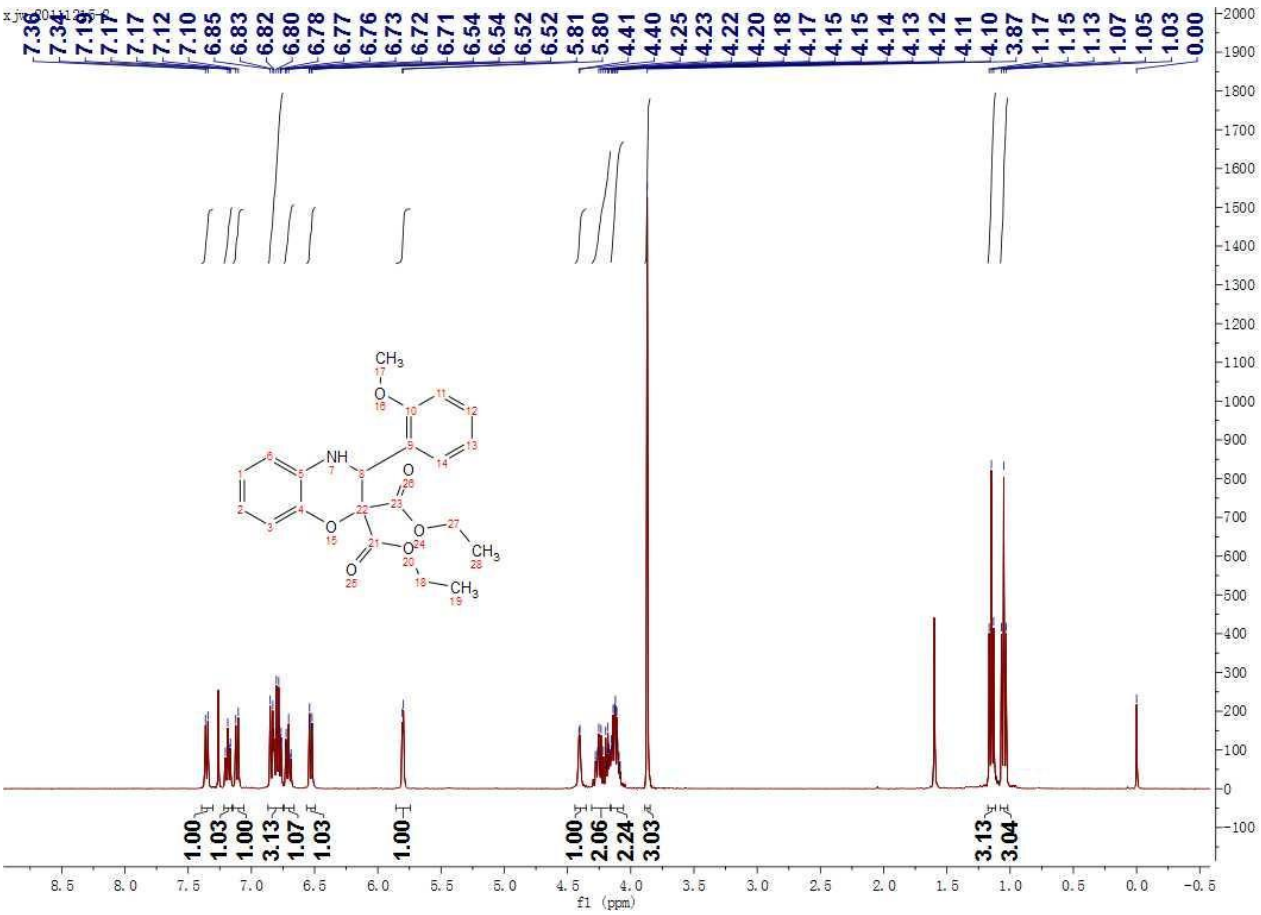
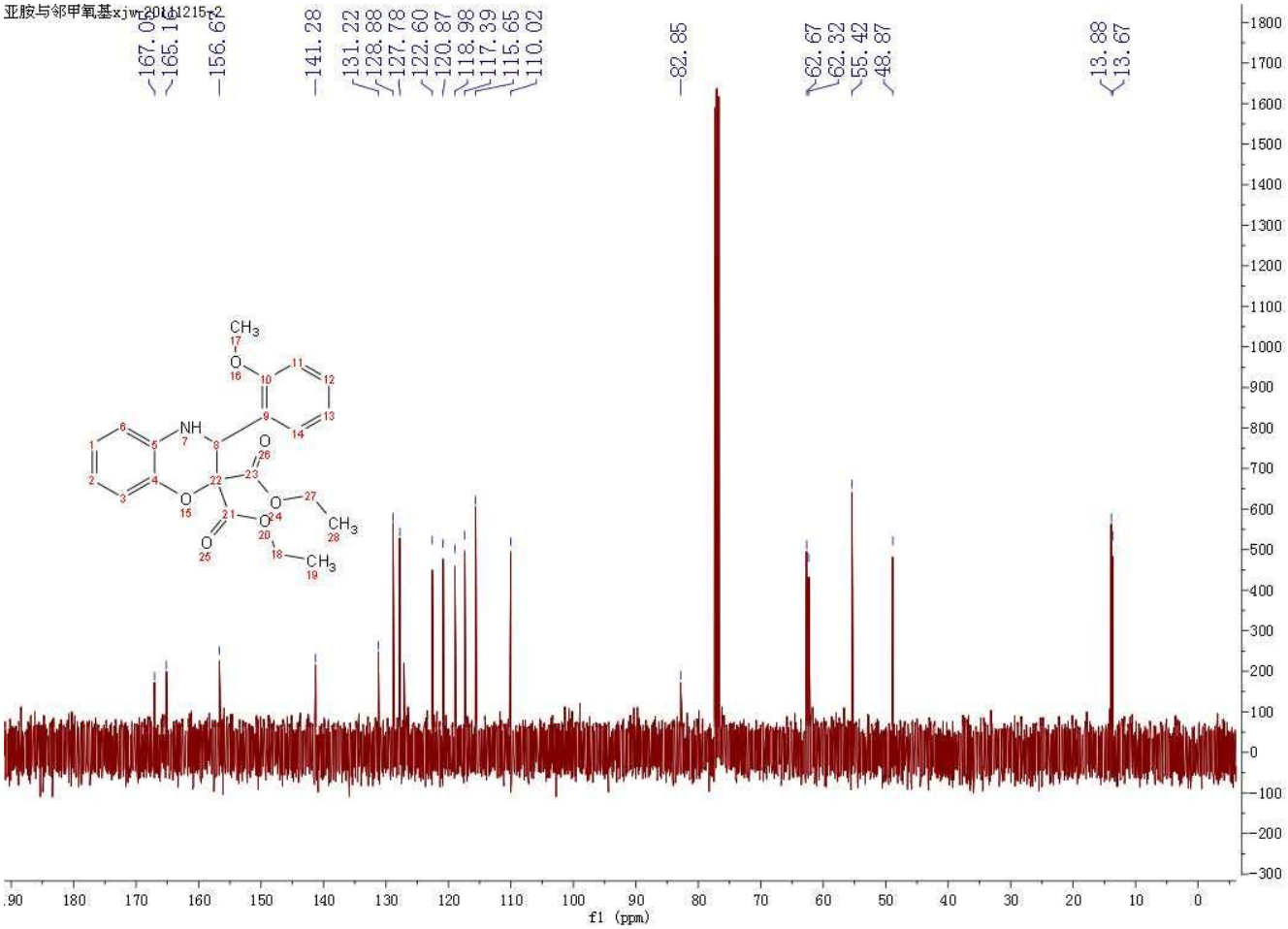
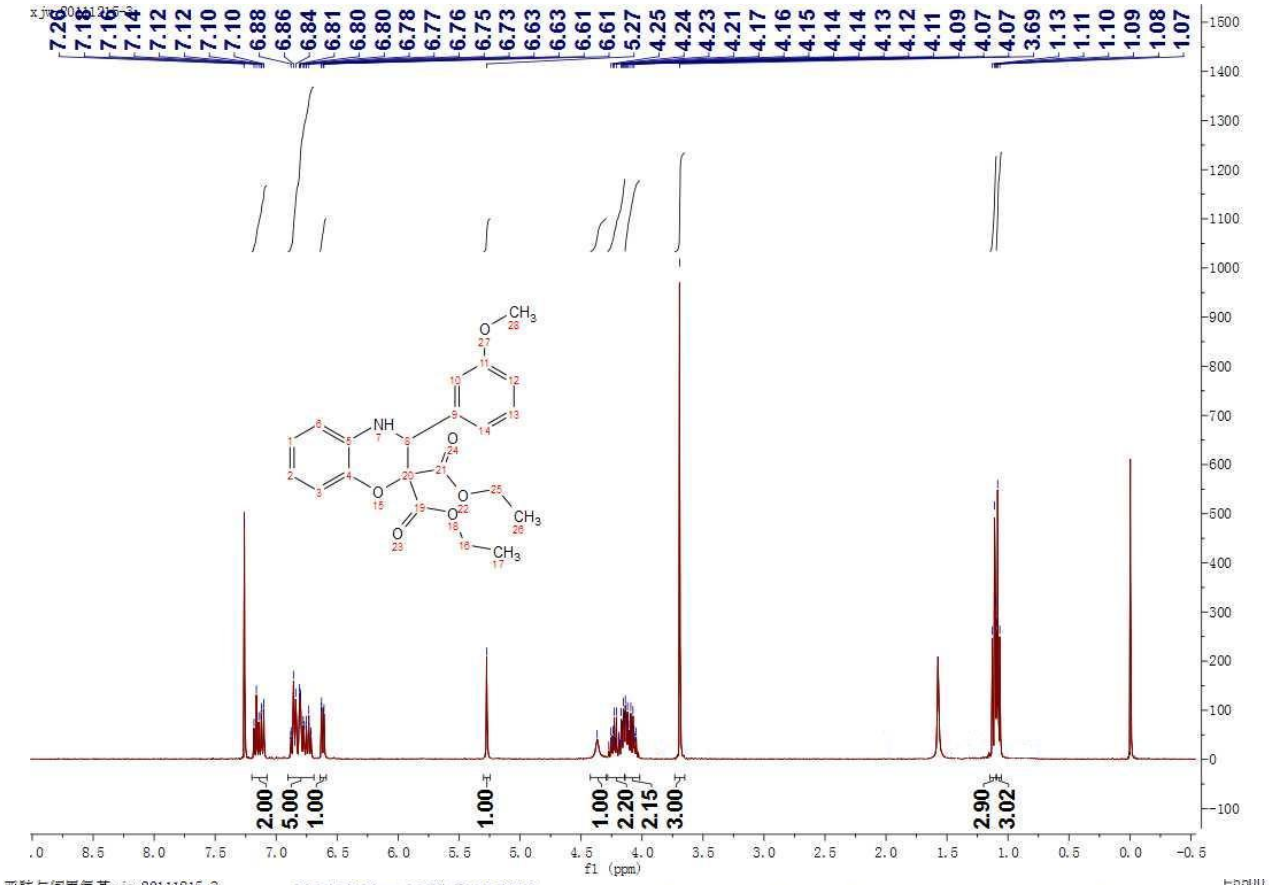




3ea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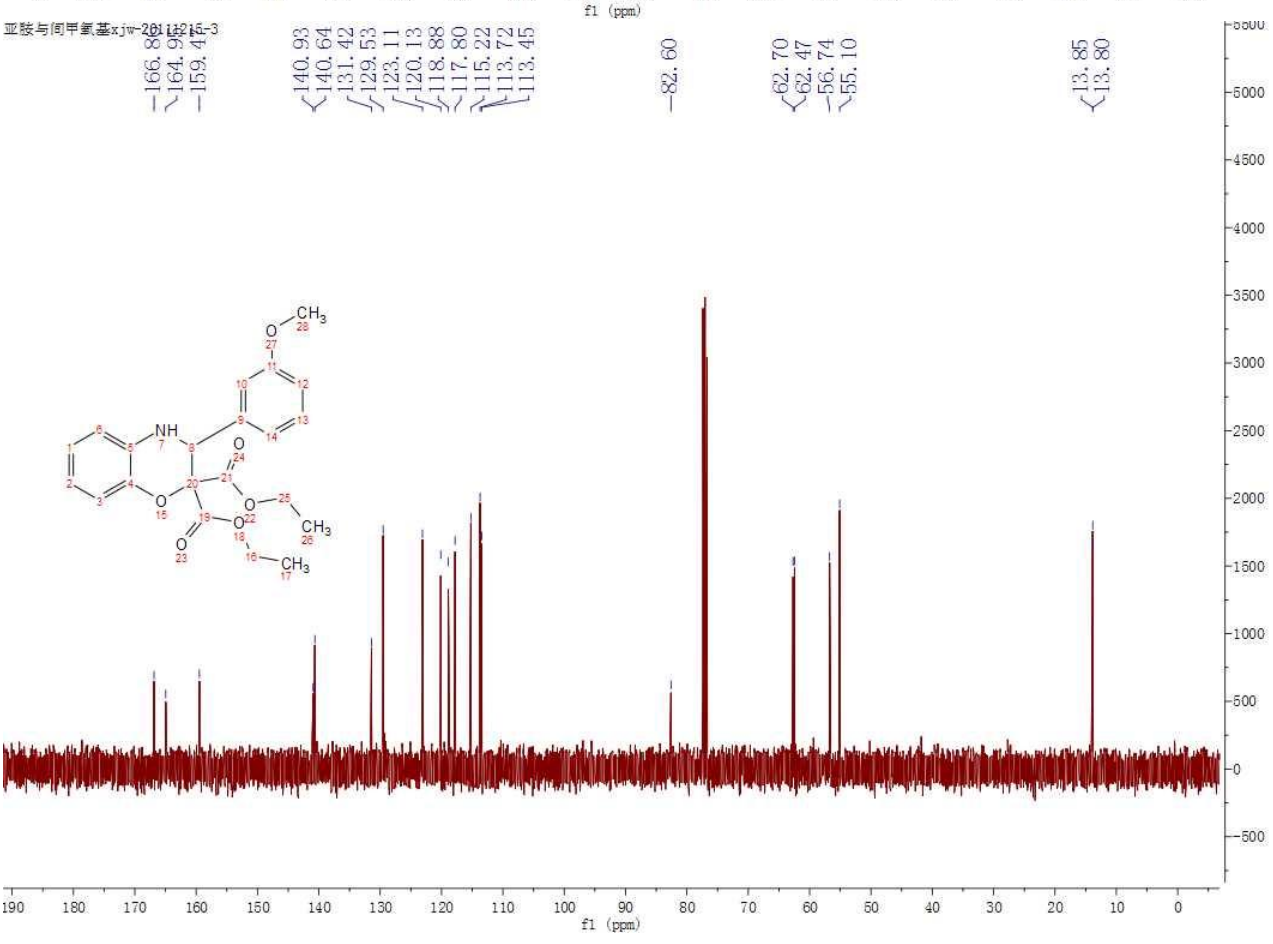
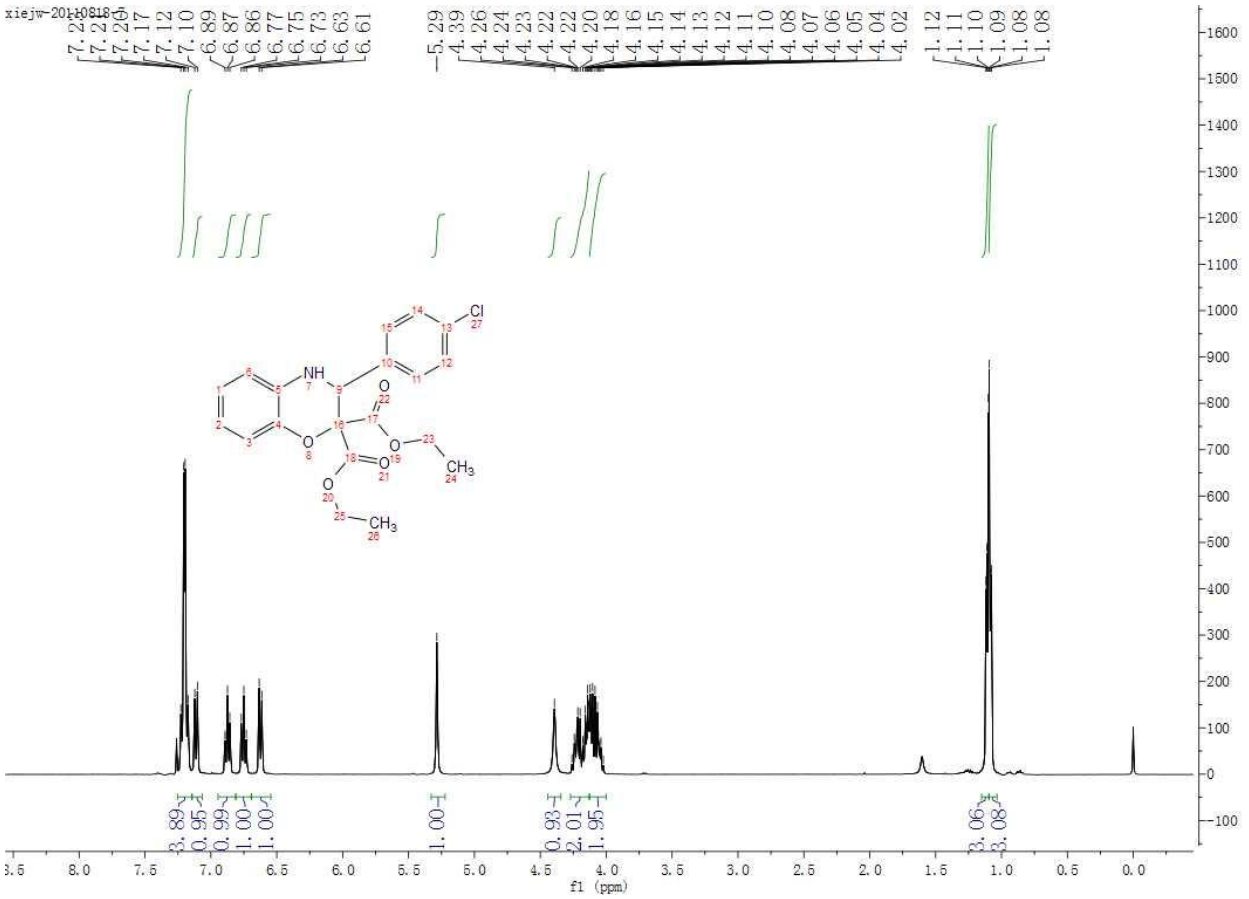
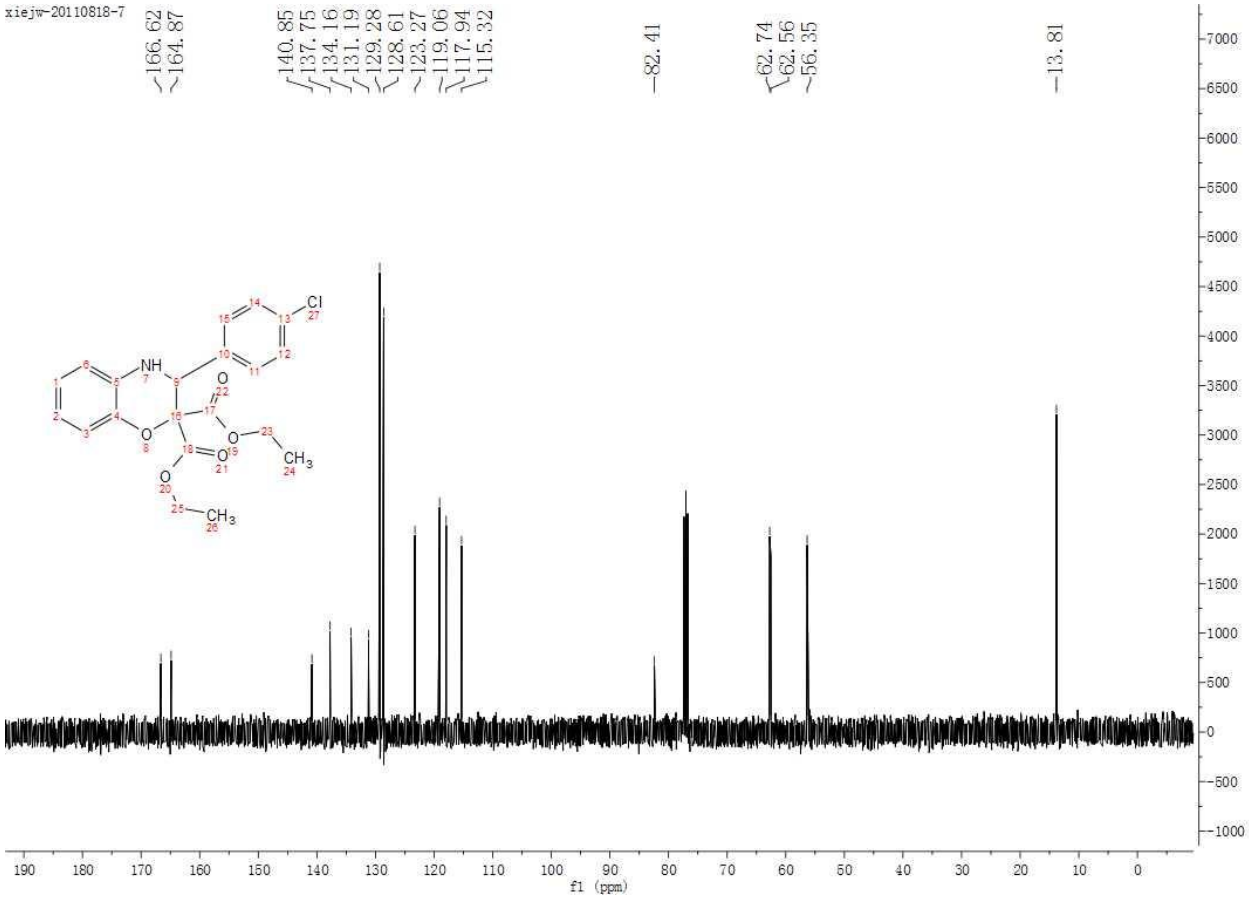
6.78
6.77
6.76
6.75
6.73
6.63
6.63
6.61
6.61
5.27
4.25
4.24
4.23
4.21
4.17
4.16
4.15
4.14
4.14
4.13
4.12
4.11
4.09
4.07
4.07
3.69
1.13
1.11
1.10
1.09
1.08
1.07
2.00
5.00
1.00
1.00
1.00
2.20
2.15
3.00
2.90
3.02
f1 (ppm)

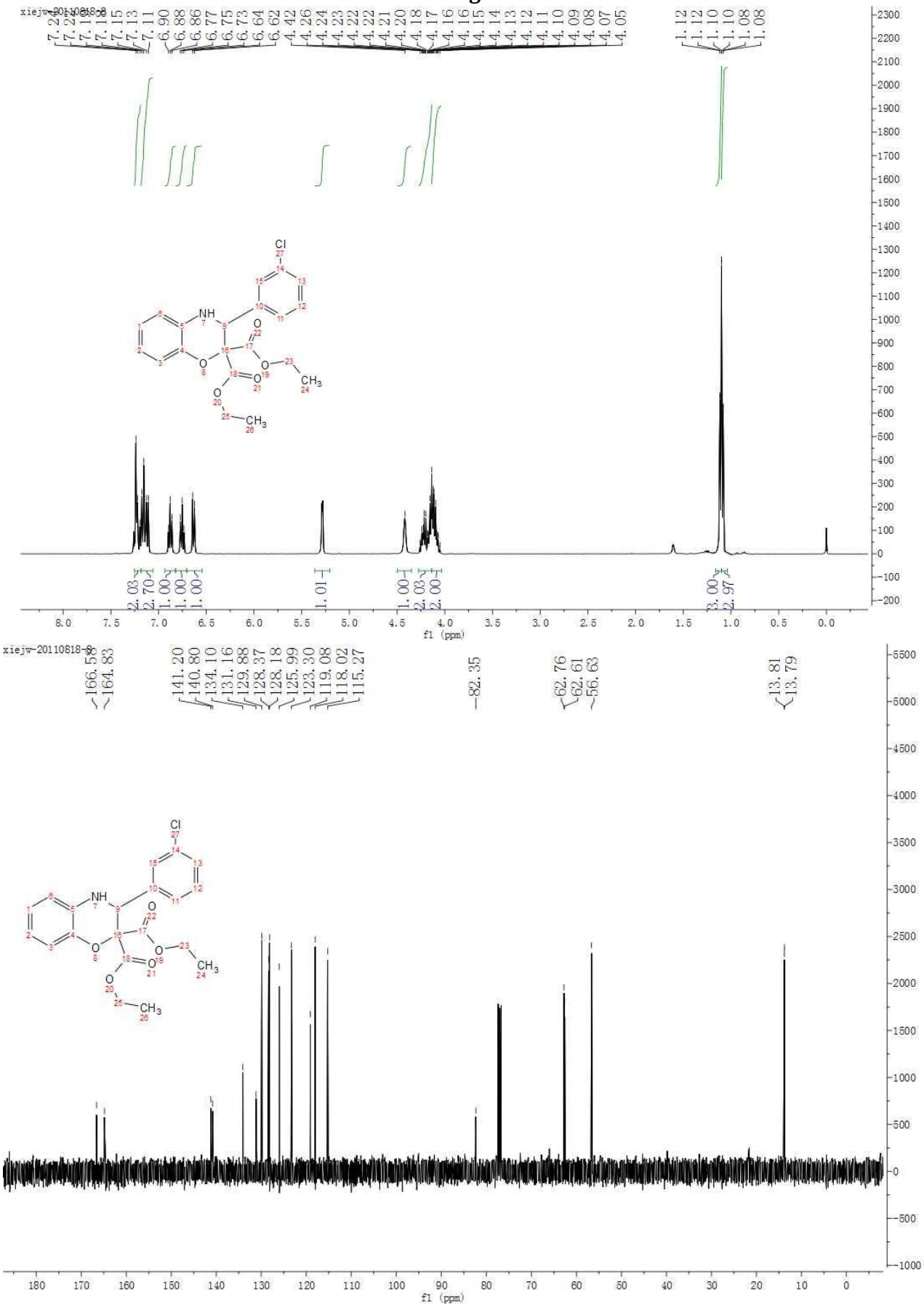

166.88
164.99
159.45
140.93
140.64
131.42
129.53
123.11
120.13
118.88
117.80
115.22
113.72
113.45
82.60
62.70
62.47
56.74
55.10
13.85
13.80
f1 (ppm)

3fa

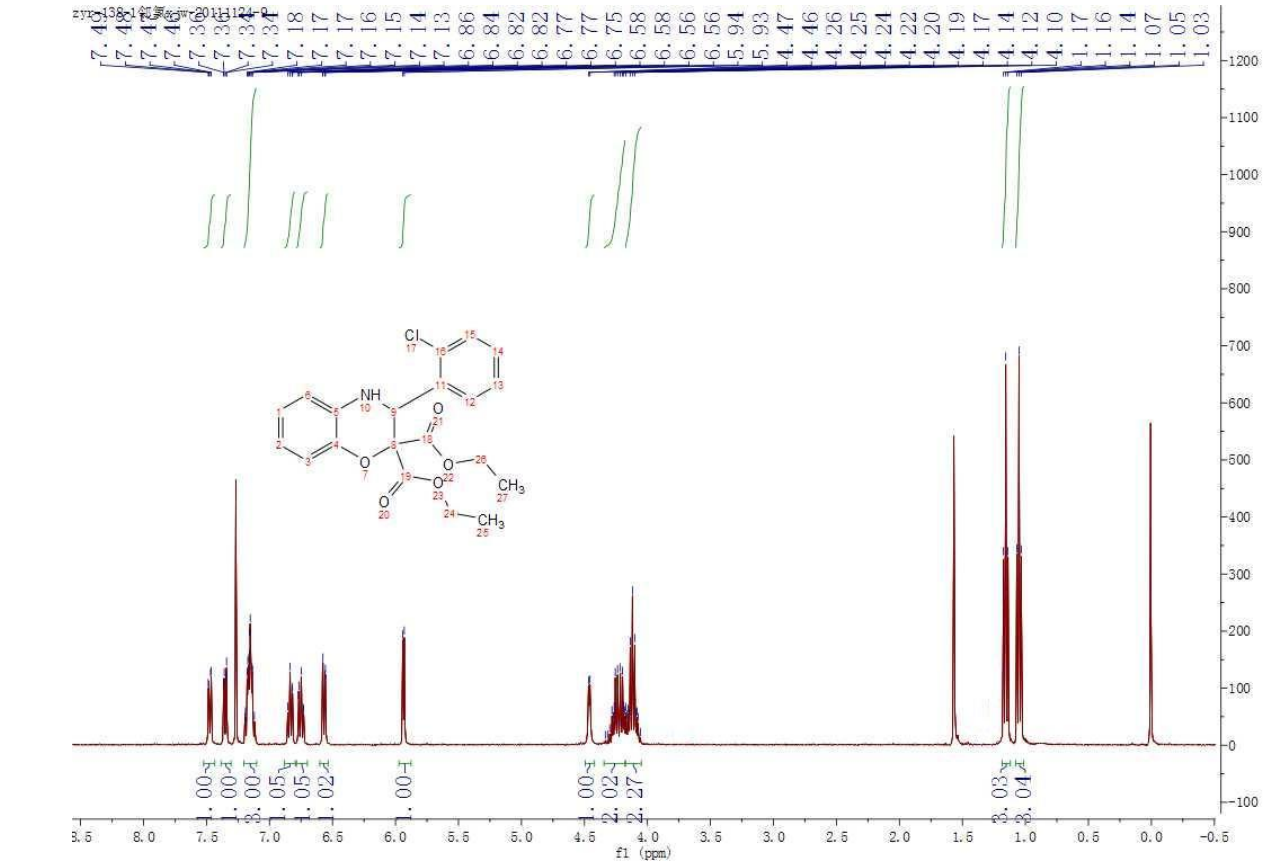


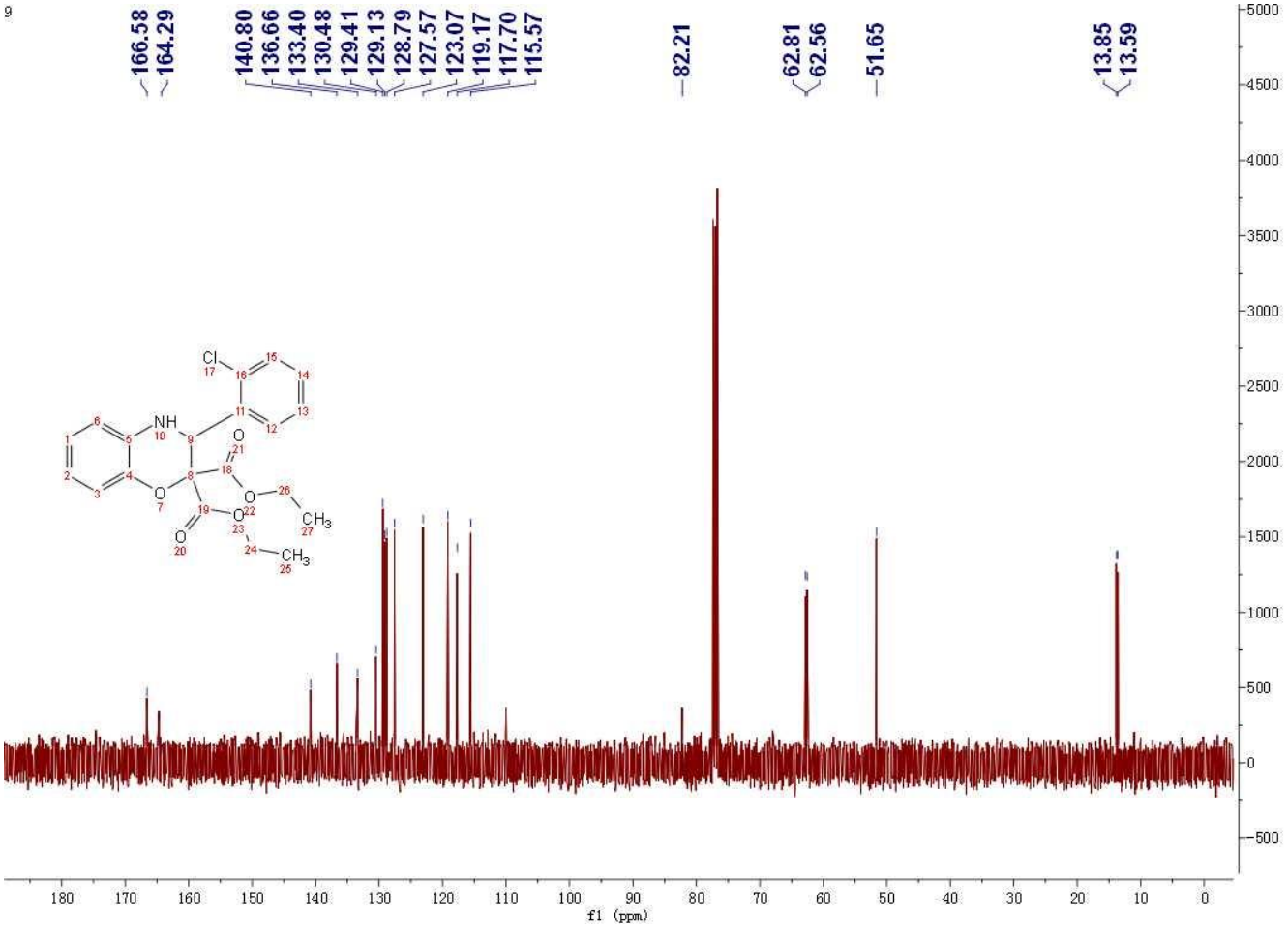


3ga

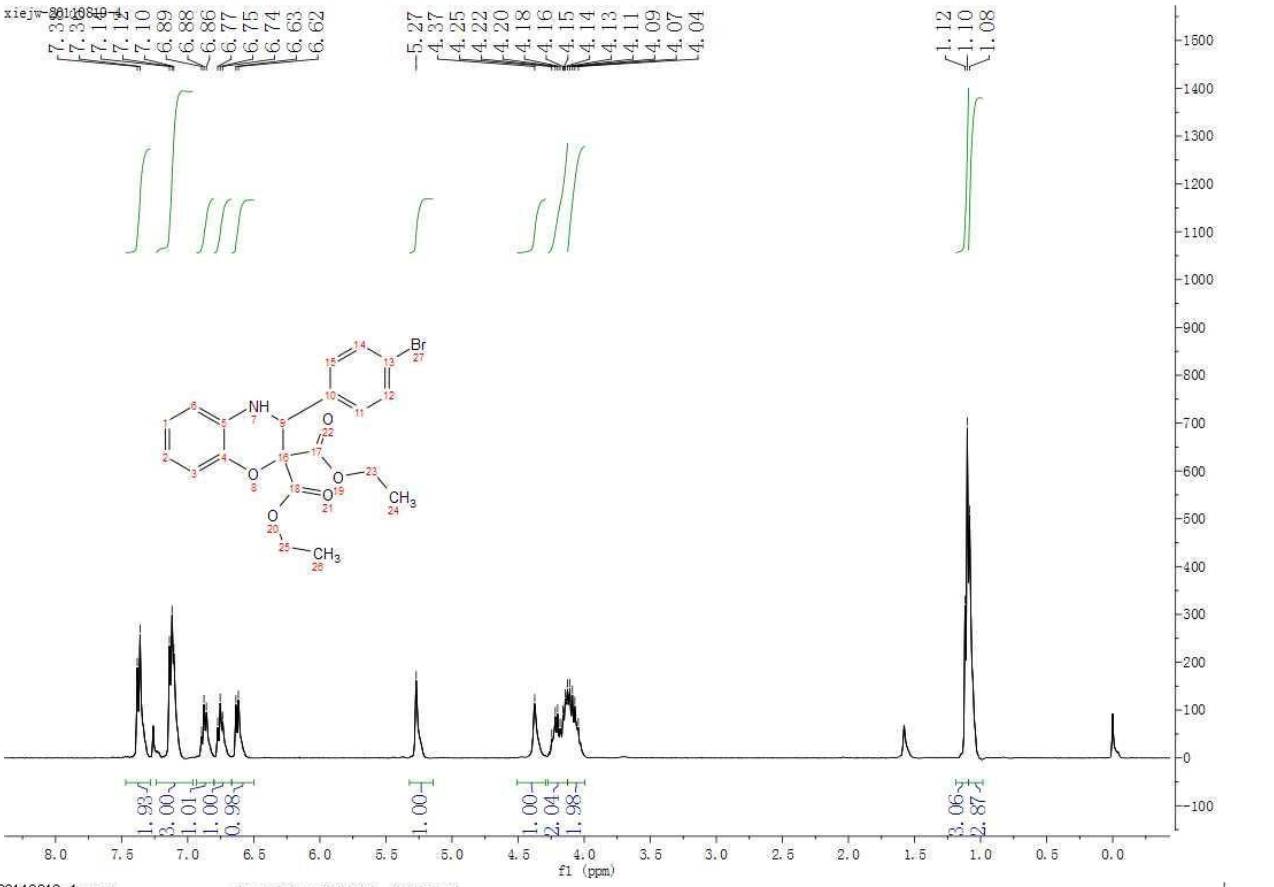

7.24
7.15
7.13
7.11
6.90
6.88
6.86
6.77
6.75
6.73
6.64
6.62
4.42
4.26
4.24
4.23
4.22
4.22
4.21
4.20
4.18
4.17
4.16
4.16
4.15
4.14
4.13
4.12
4.11
4.10
4.09
4.08
4.07
4.05
1.12
1.12
1.10
1.10
1.08
1.08
Cl
NH
O
CH3
2.03
2.70
1.00
1.00
1.00
1.01
1.00
2.03
2.00
3.00
2.97
f1 (ppm)
xiejw-20110818-8
166.58
164.83
141.20
140.80
134.10
131.16
129.88
128.37
128.18
125.99
123.30
119.08
118.02
115.27
82.35
62.76
62.61
56.63
13.81
13.79
f1 (ppm)

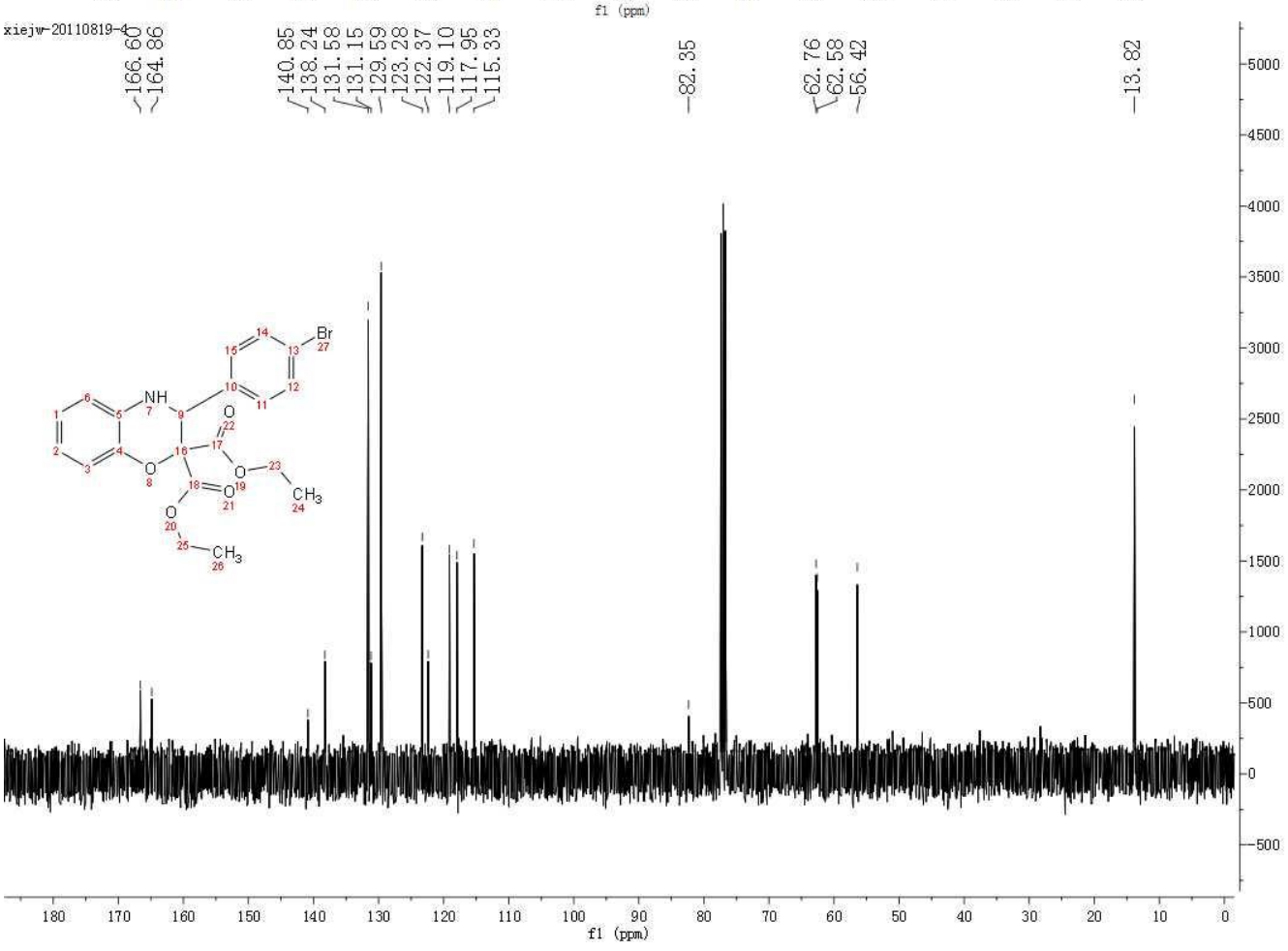
3ha


7.40 7.38 7.36 7.34 7.18 7.17 7.16 7.15 7.14 7.13 6.86 6.84 6.82 6.82 6.77 6.77 6.75 6.58 6.58 6.56 6.56 5.94 5.93 4.47 4.46 4.26 4.25 4.24 4.22 4.20 4.19 4.17 4.14 4.12 4.10 1.17 1.16 1.14 1.07 1.05 1.03
f1 (ppm)

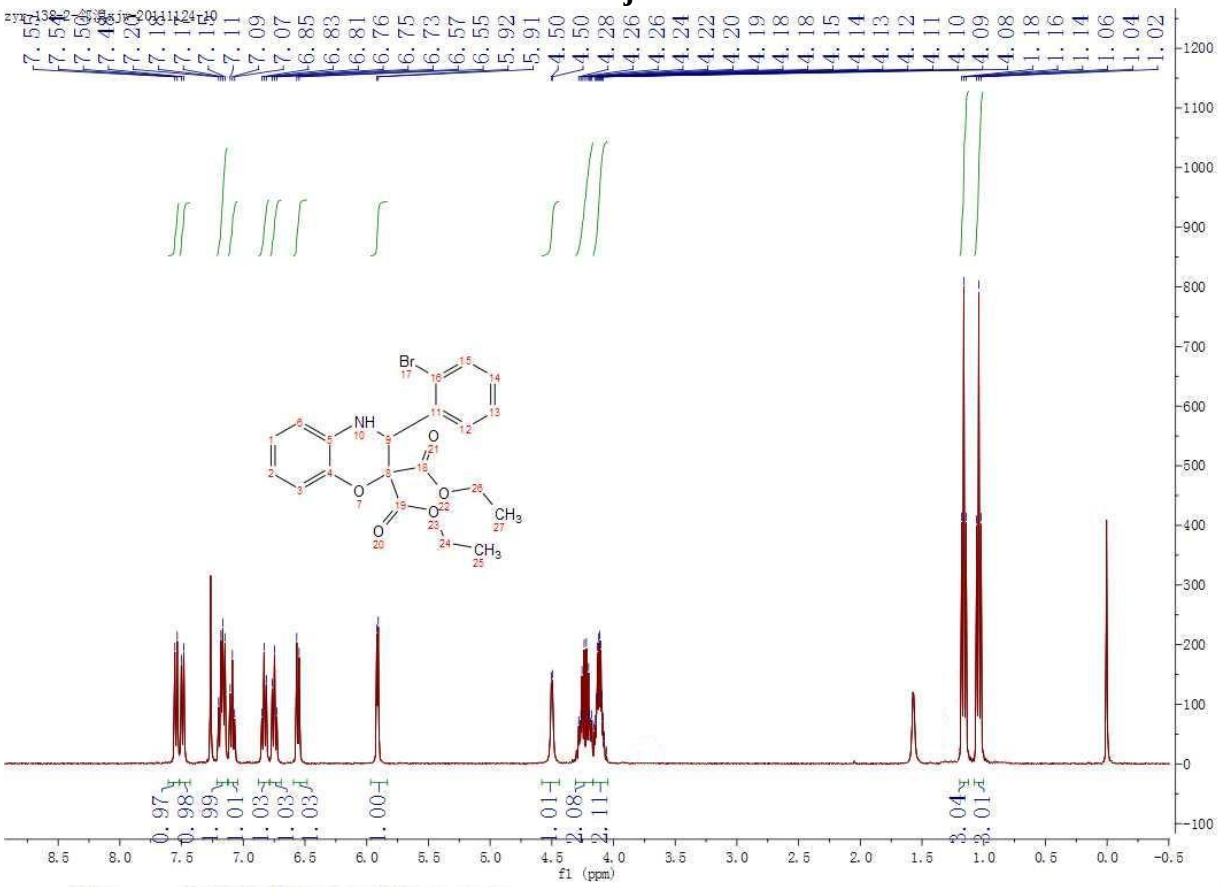

166.58 164.29 140.80 136.66 133.40 130.48 129.41 129.13 128.79 127.57 123.07 119.17 117.70 115.57 82.21 62.81 62.56 51.65 13.85 13.59
f1 (ppm)

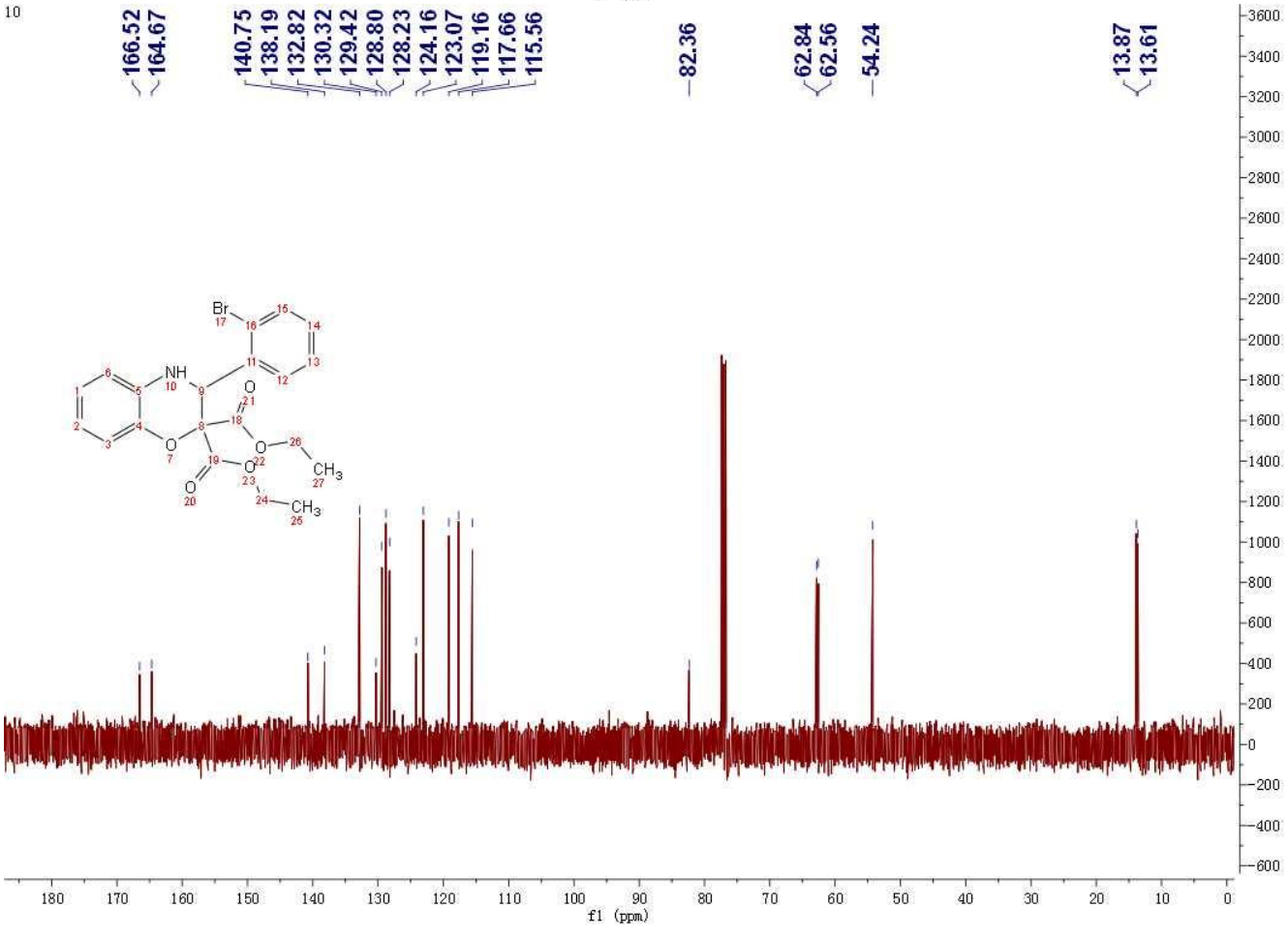
3ia


7.32
7.14
7.12
7.10
6.89
6.88
6.86
6.77
6.75
6.74
6.63
6.62
5.27
4.37
4.25
4.22
4.20
4.18
4.16
4.15
4.14
4.13
4.11
4.09
4.07
4.04
1.12
1.10
1.08
Br
NH
O
CH3
1.93
3.00
1.01
1.00
0.98
1.00
1.00
2.04
1.98
3.06
2.87
f1 (ppm)

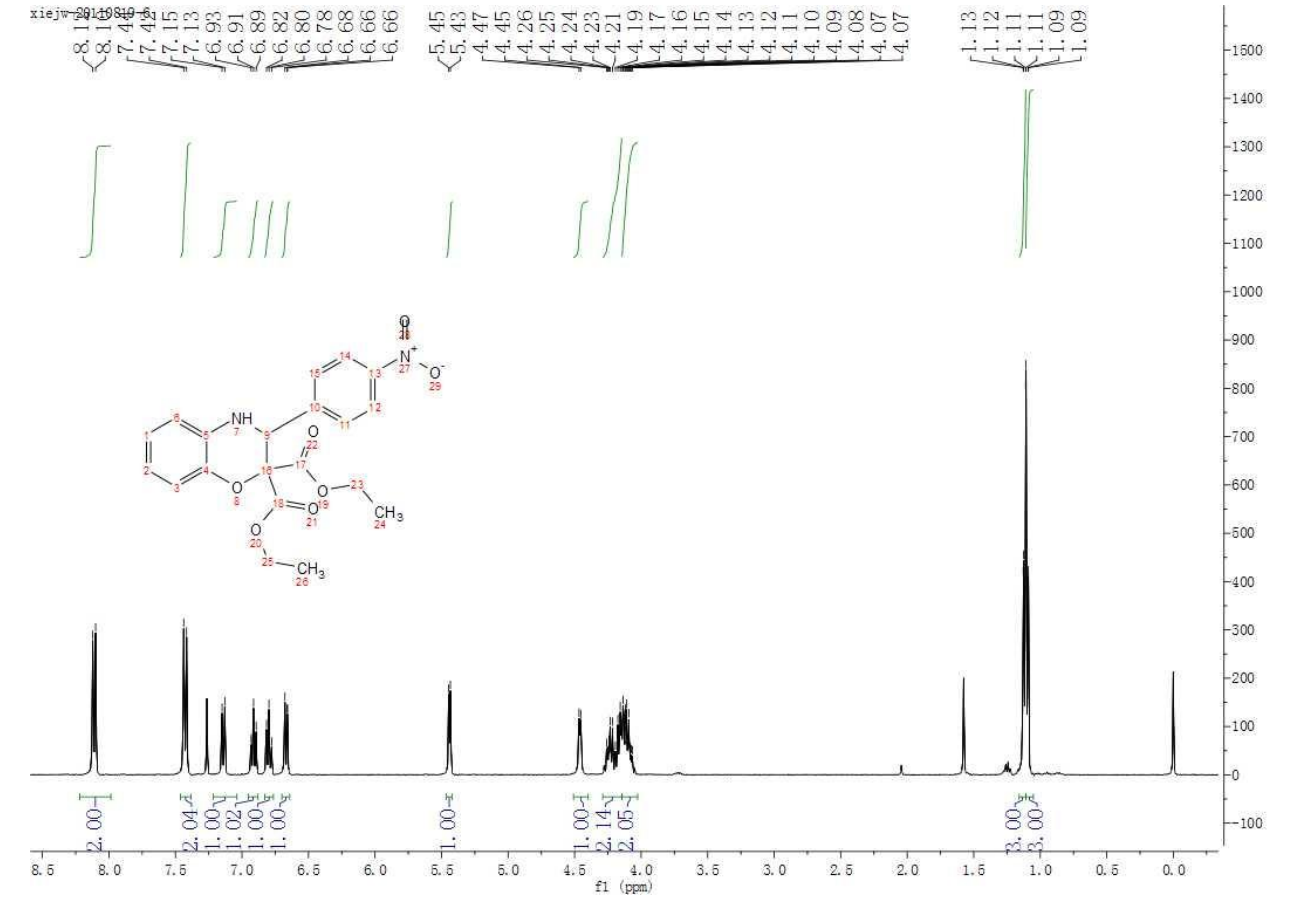

xiejw-20110819-4
166.60
164.86
140.85
138.24
131.58
131.15
129.59
123.28
122.37
119.10
117.95
115.33
82.35
62.76
62.58
56.42
13.82
Br
NH
O
CH3
f1 (ppm)

3ja





3ka


xiejw-20110819-6
8.17
8.15
7.44
7.42
7.15
7.13
6.93
6.91
6.89
6.82
6.80
6.78
6.68
6.66
6.66
5.45
5.43
4.47
4.45
4.26
4.25
4.24
4.23
4.21
4.19
4.17
4.16
4.15
4.14
4.13
4.12
4.11
4.10
4.09
4.08
4.07
4.07
1.13
1.12
1.11
1.11
1.09
1.09
2.00
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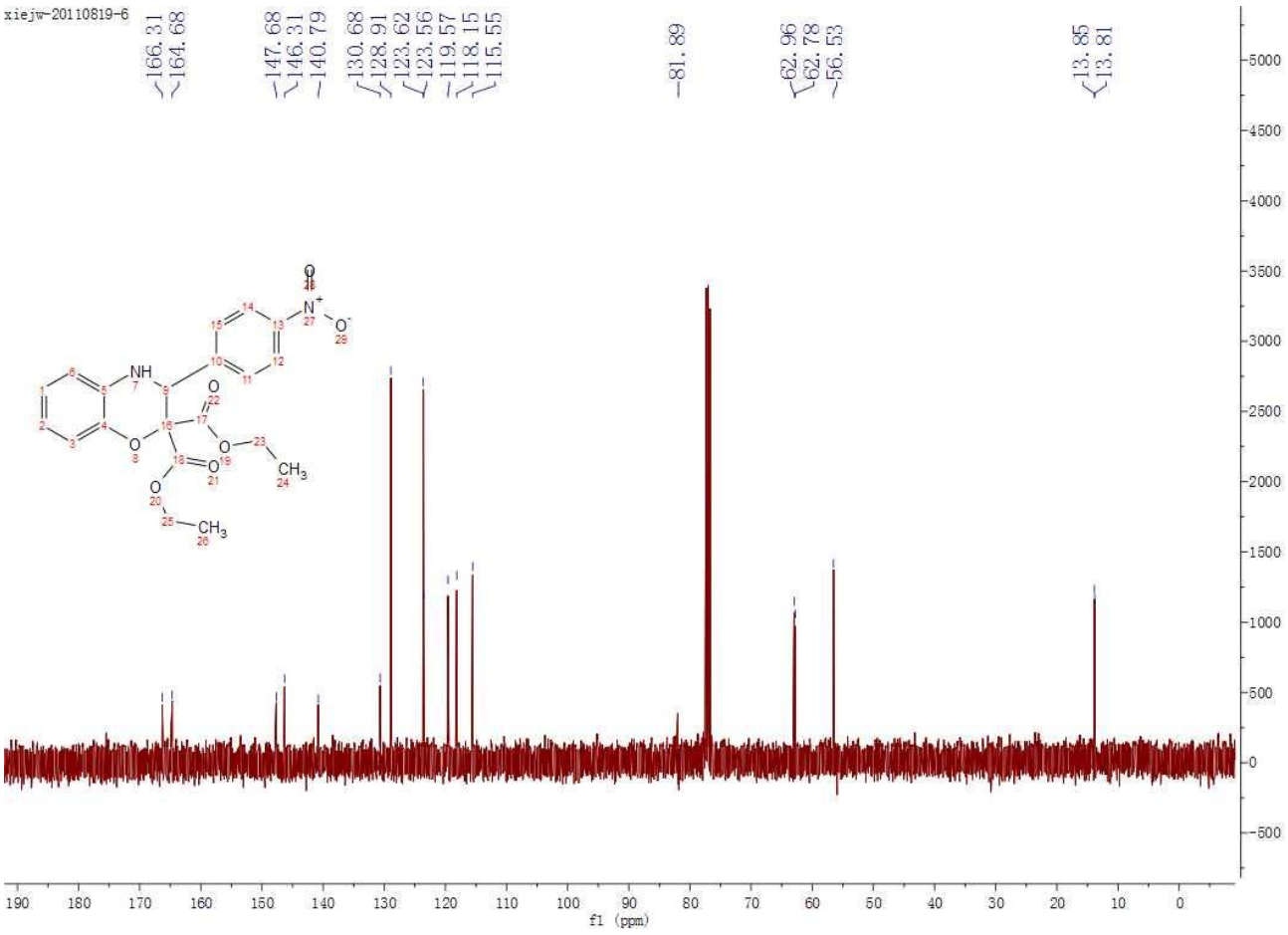
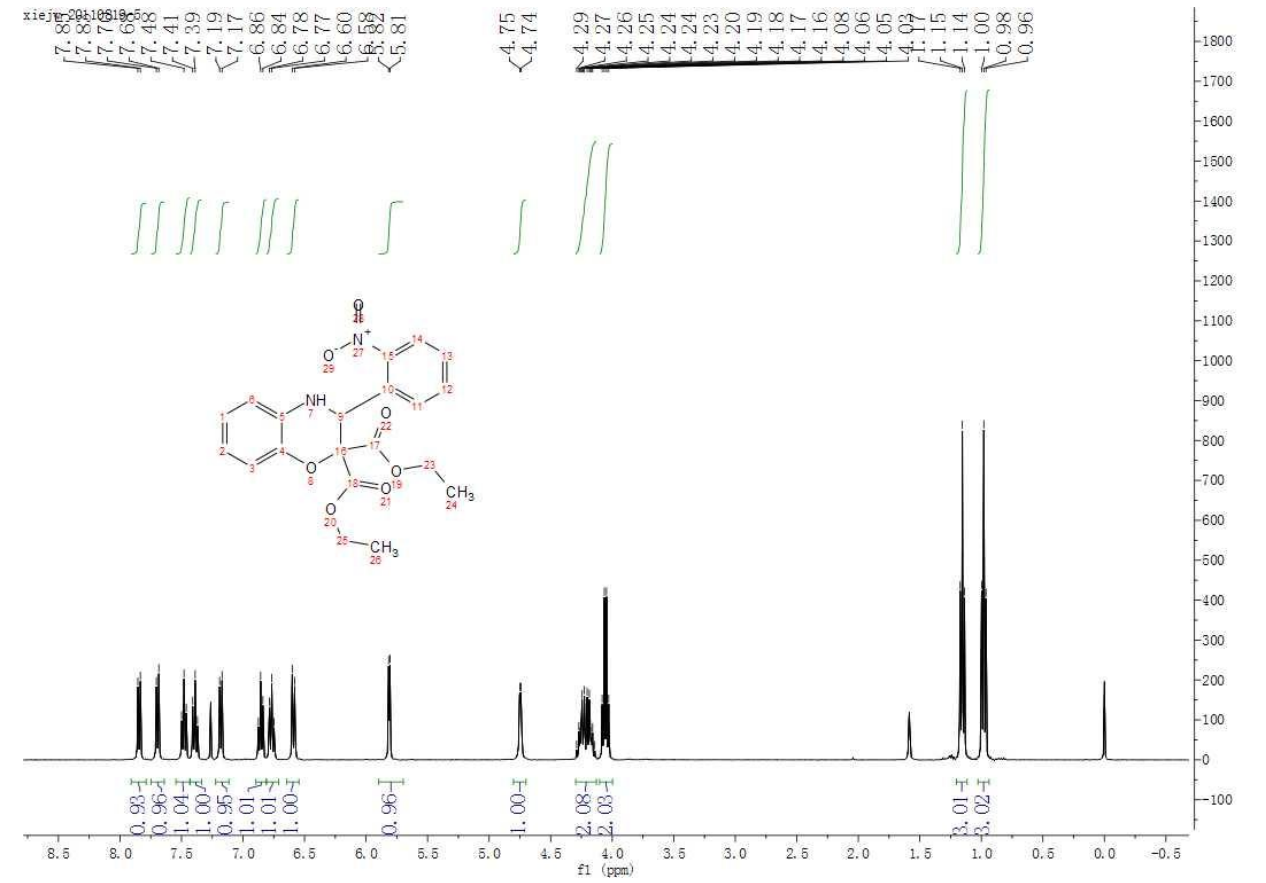
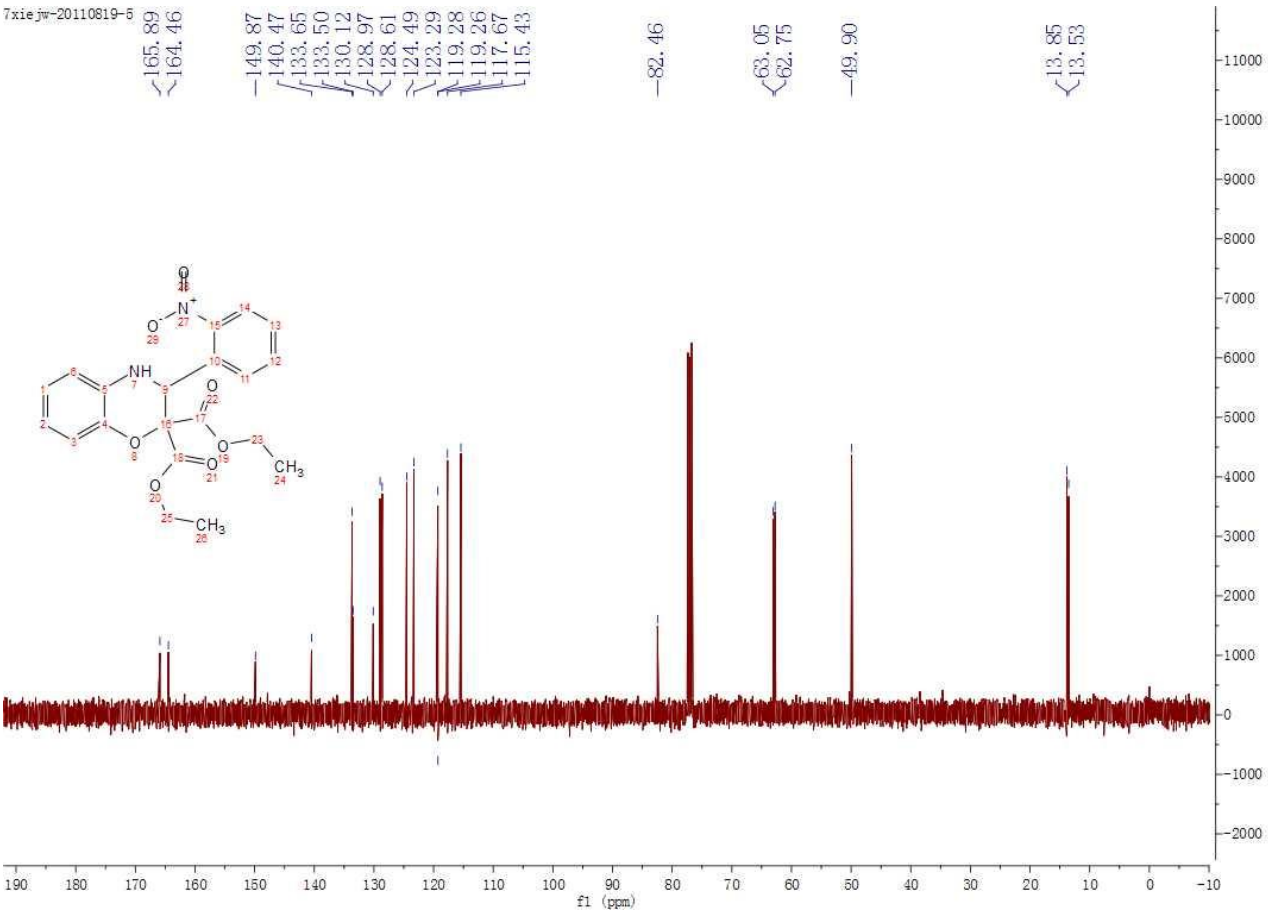
2.14
2.05
3.00
3.00
f1 (ppm)

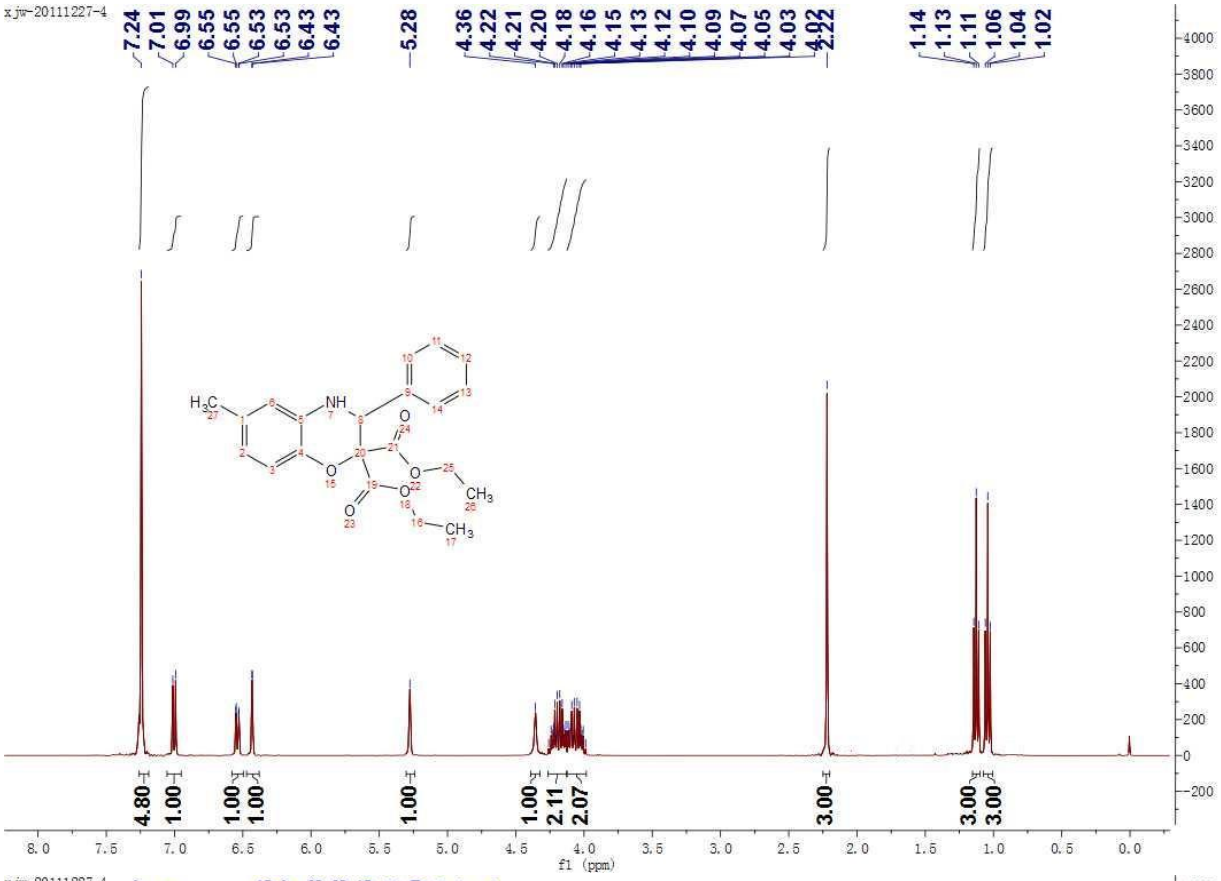

xiejw-20110819-6
166.31
164.68
147.68
146.31
140.79
130.68
128.91
123.62
123.56
119.57
118.15
115.55
81.89
62.96
62.78
56.53
13.85
13.81
f1 (ppm)

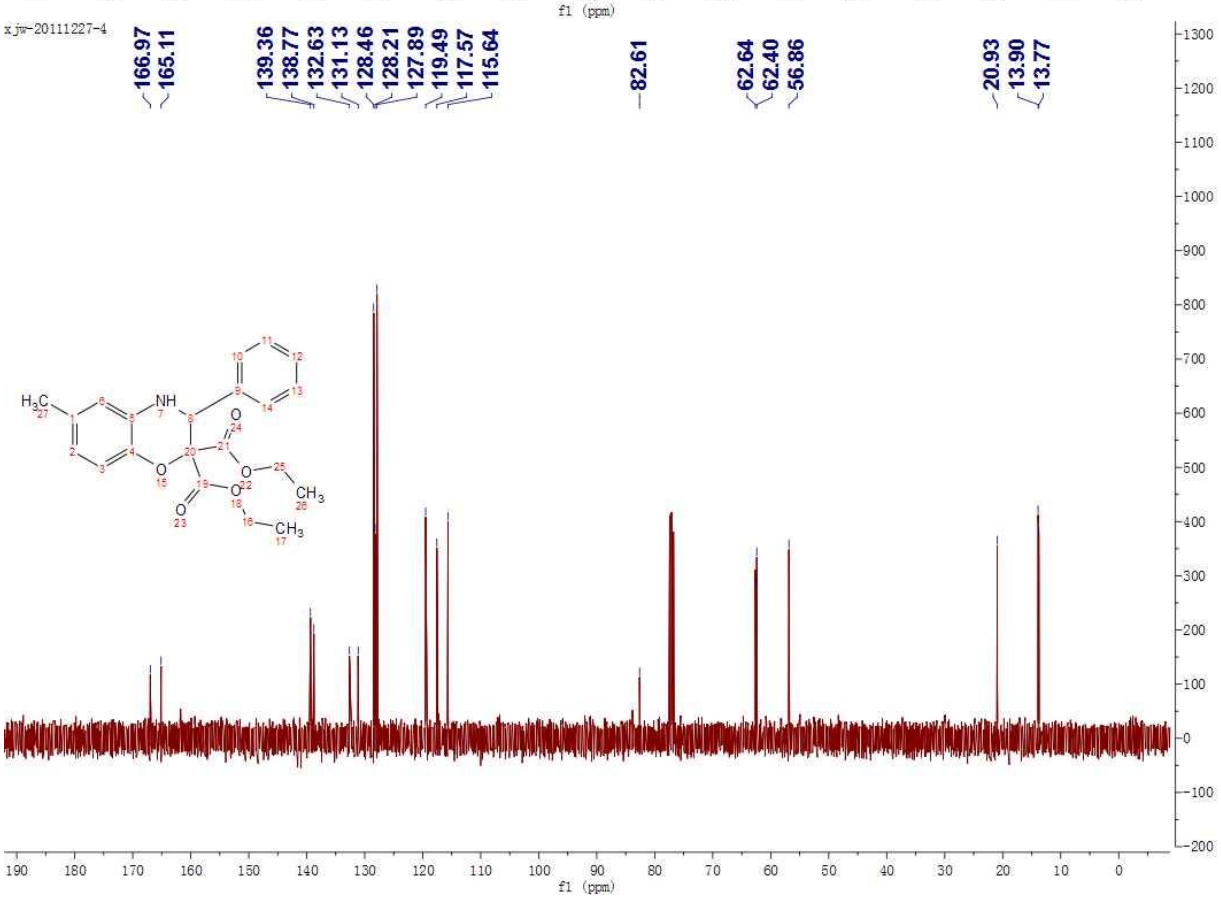
3la



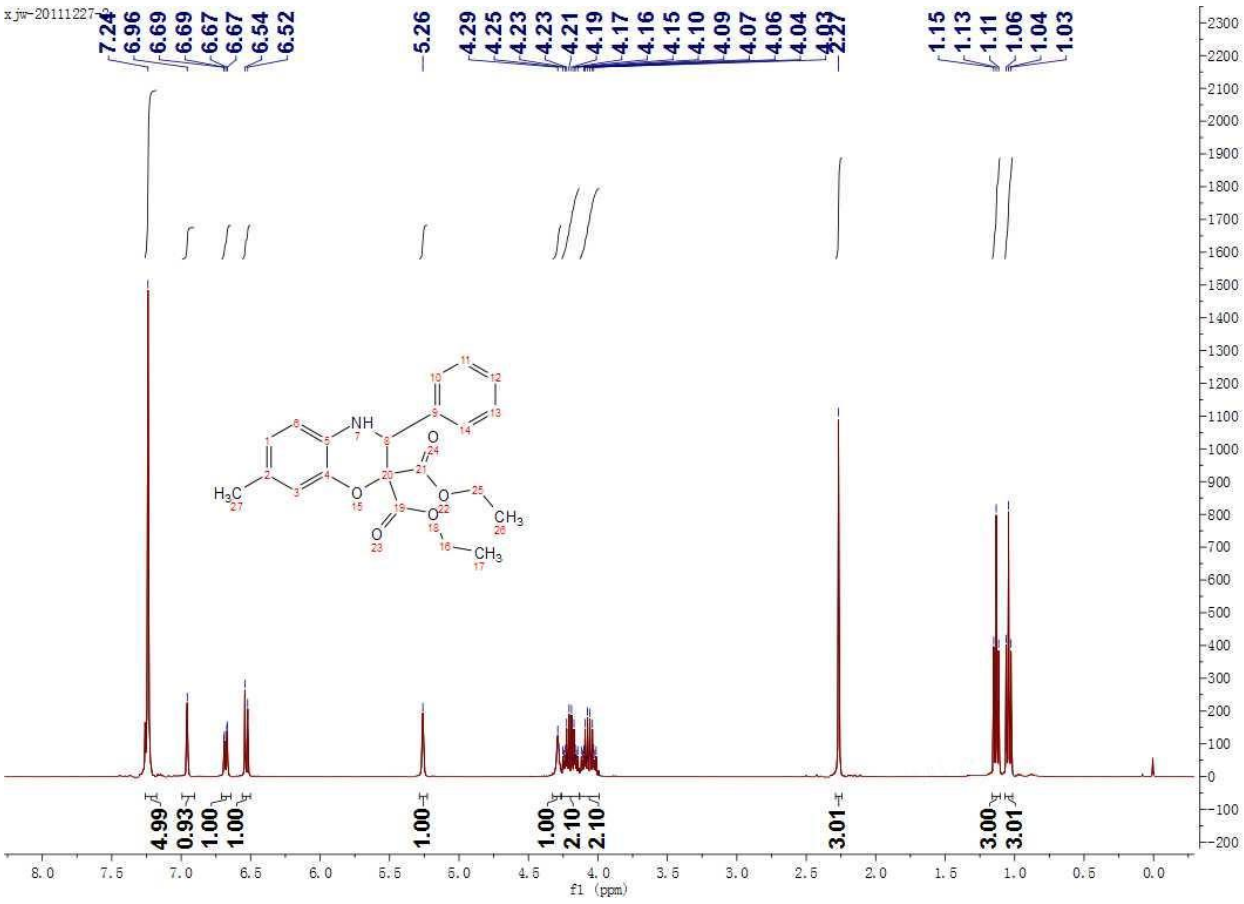


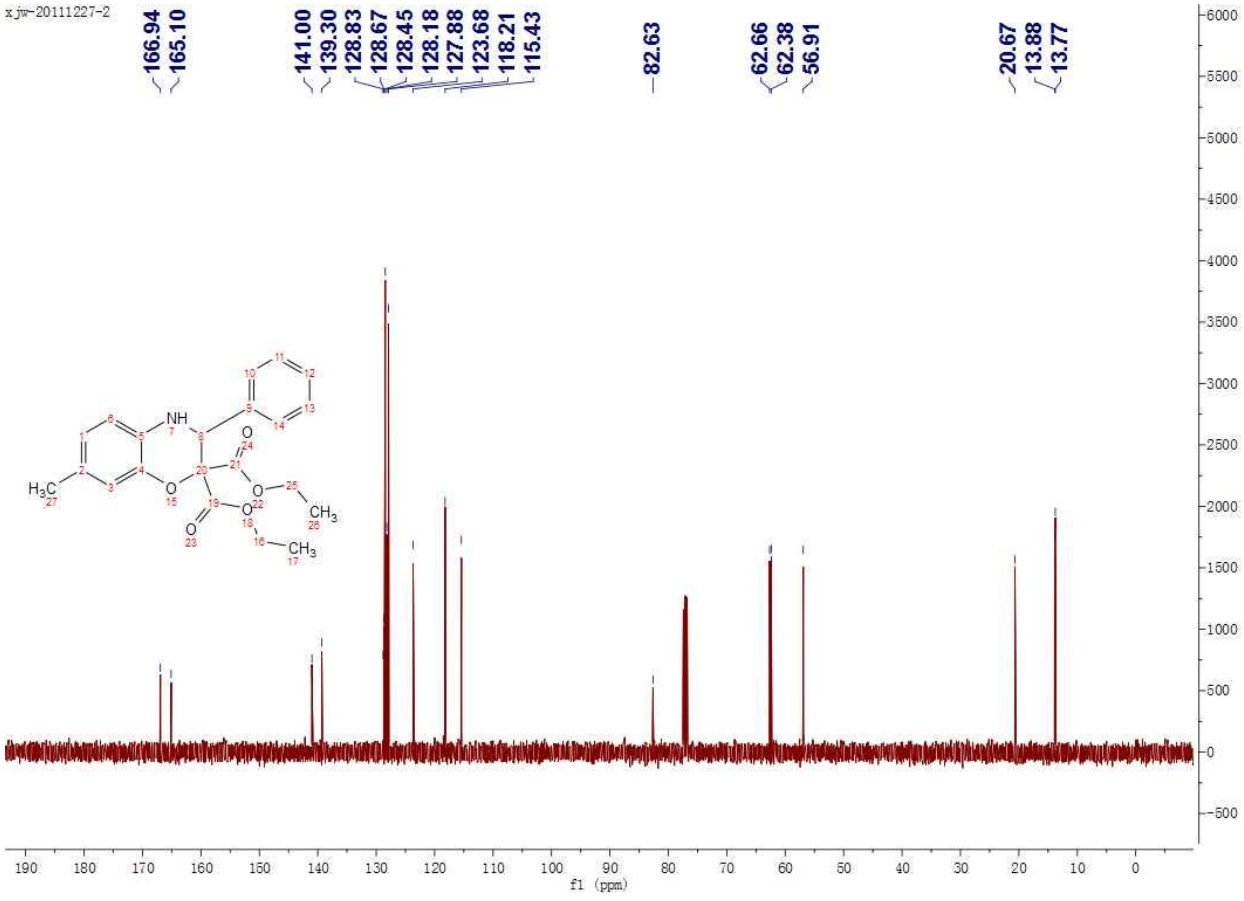
3ma





3na


xjw-20111227-1
7.24
6.96
6.69
6.69
6.67
6.67
6.54
6.52
5.26
4.29
4.25
4.23
4.23
4.21
4.19
4.17
4.16
4.15
4.10
4.09
4.07
4.06
4.04
4.03
2.27
1.15
1.13
1.11
1.06
1.04
1.03
4.99
0.93
1.00
1.00
1.00
1.00
2.10
2.10
3.01
3.00
3.01
f1 (ppm)


xjw-20111227-2
166.94
165.10
141.00
139.30
128.83
128.67
128.45
128.18
127.88
123.68
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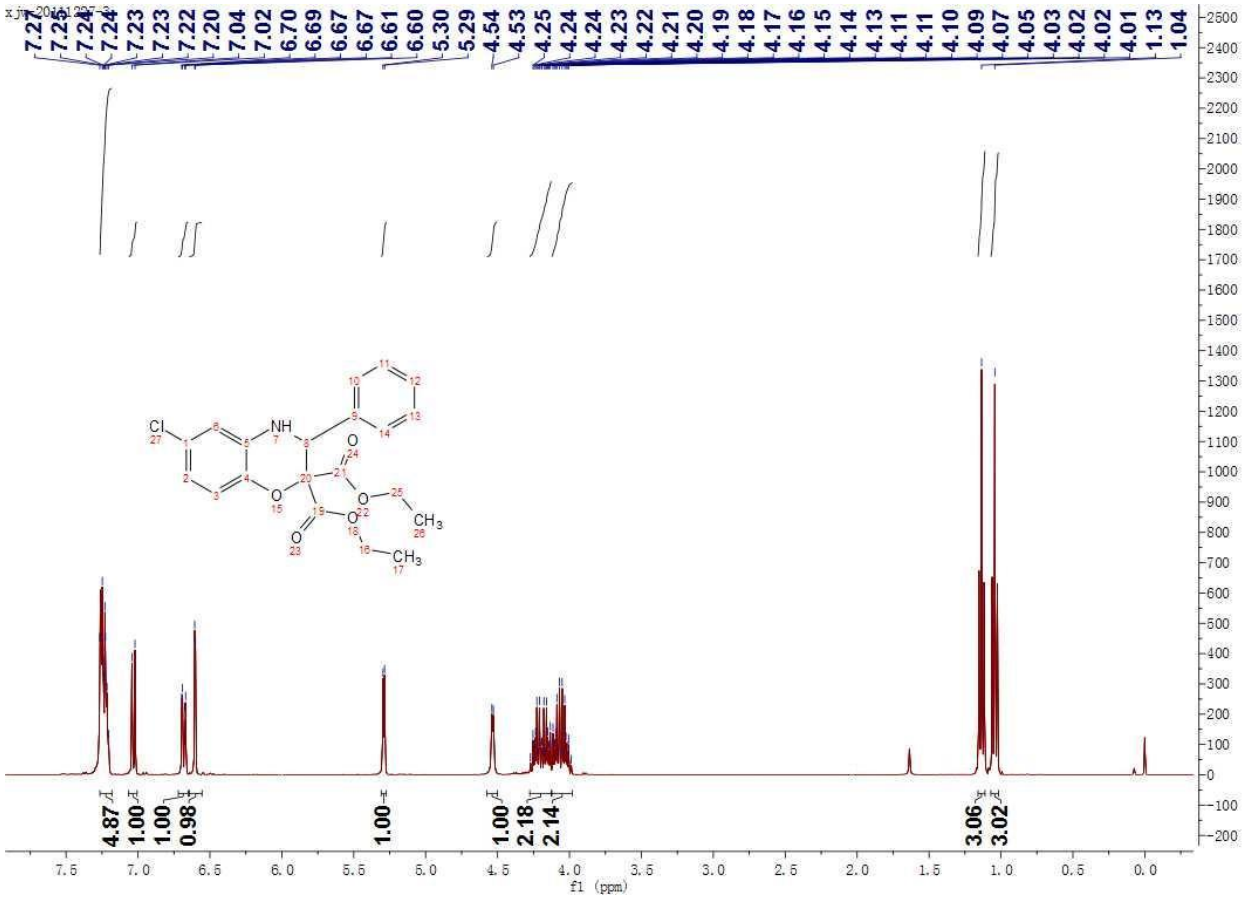
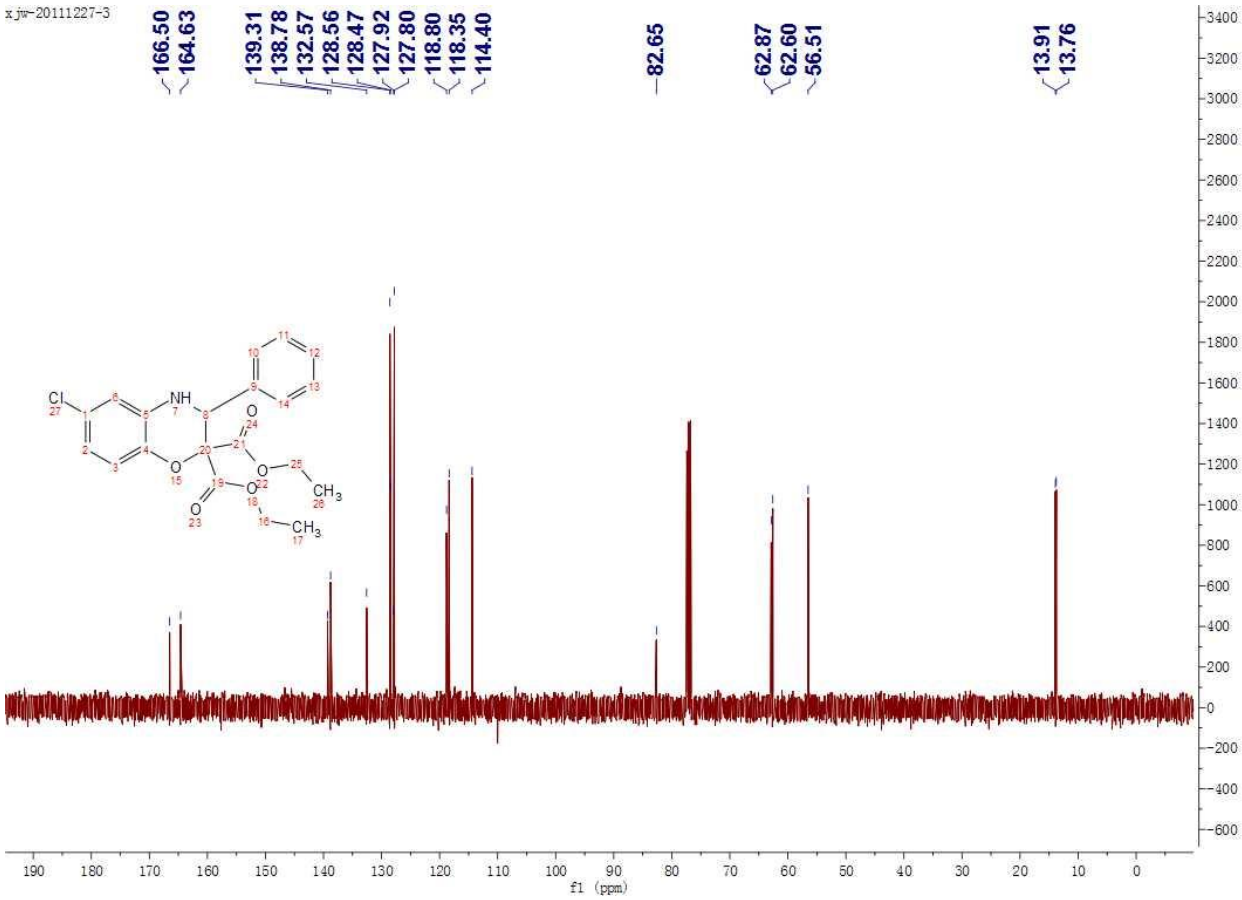
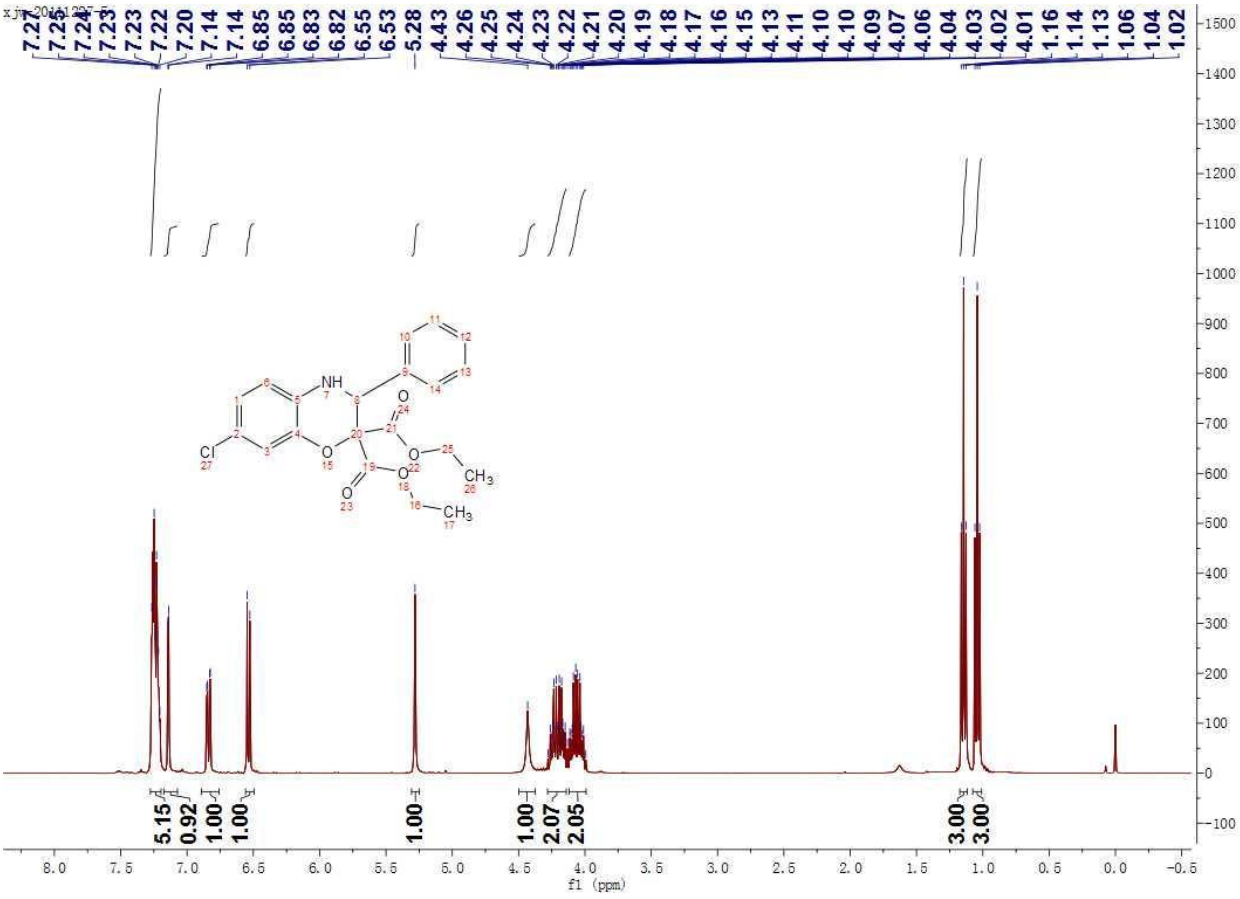
82.63
62.66
62.38
56.91
20.67
13.88
13.77
f1 (ppm)

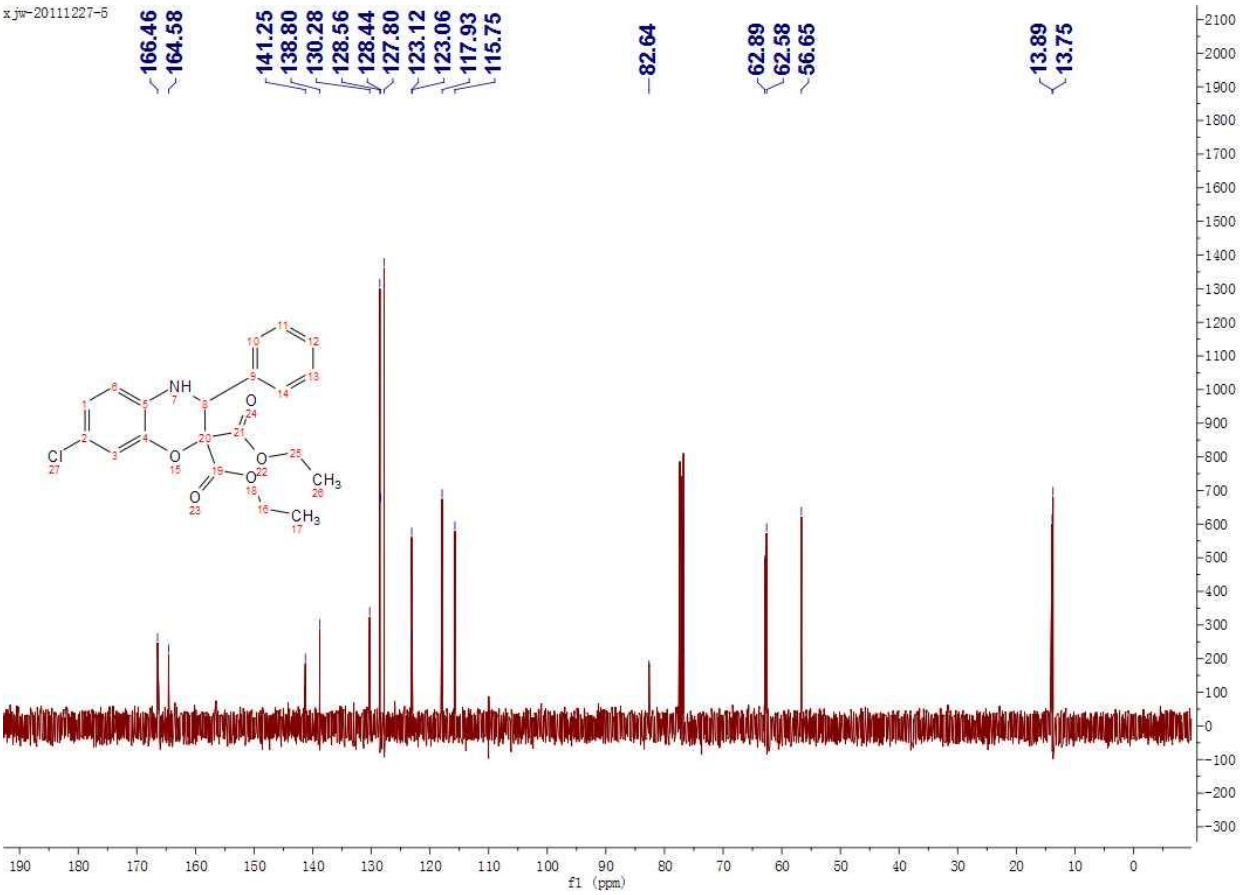
3oa





3pa


7.26
7.25
7.24
7.23
7.23
7.22
7.20
7.14
7.14
6.85
6.85
6.83
6.82
6.55
6.53
5.28
4.43
4.26
4.25
4.24
4.23
4.22
4.21
4.20
4.19
4.18
4.17
4.16
4.15
4.13
4.11
4.10
4.10
4.09
4.07
4.06
4.04
4.03
4.02
4.01
1.16
1.14
1.13
1.06
1.04
1.02
5.15
0.92
1.00
1.00
1.00
1.00
2.07
2.05
3.00
3.00
f1 (ppm)


xjw-20111227-5
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164.58
141.25
138.80
130.28
128.56
128.44
127.80
123.12
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115.75
82.64
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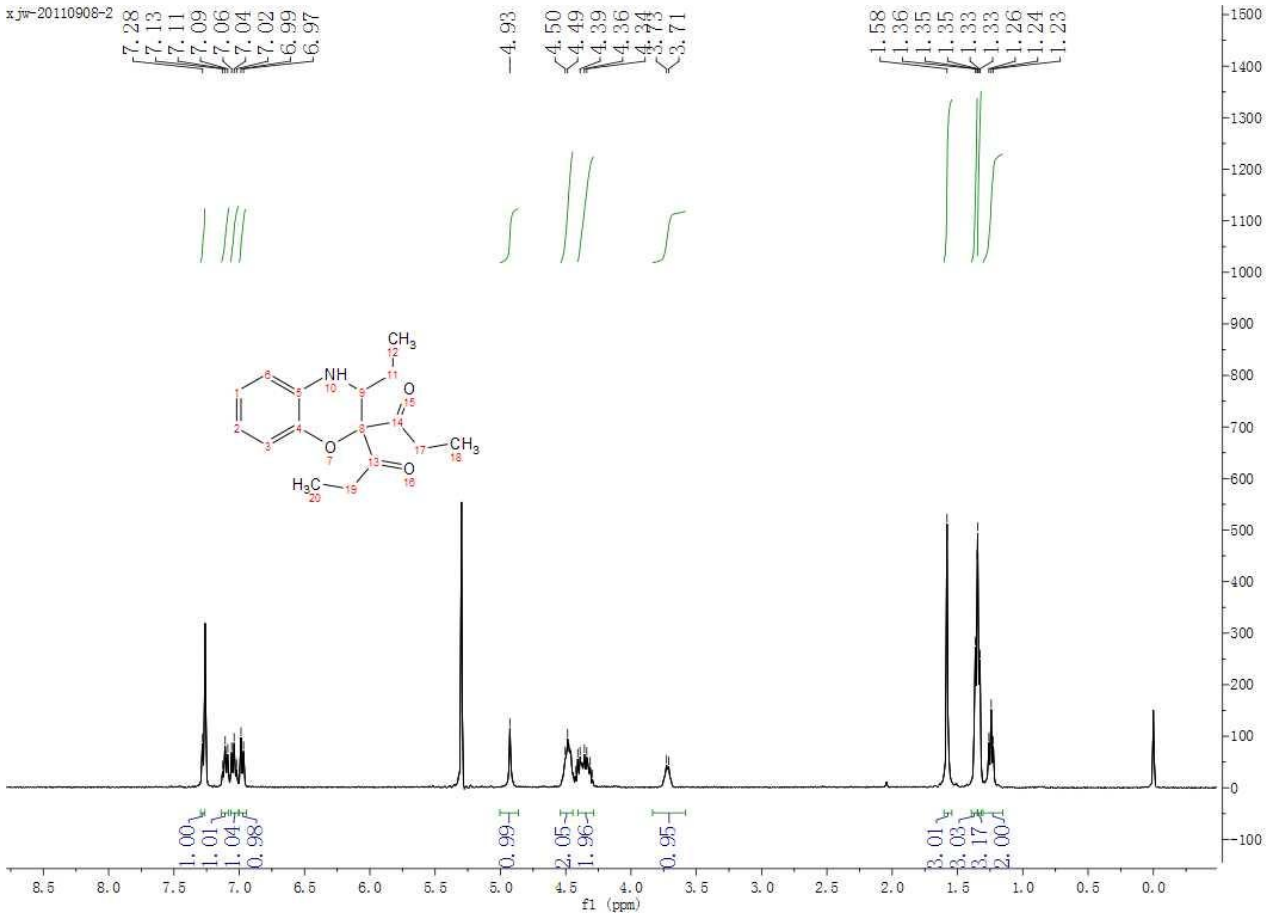
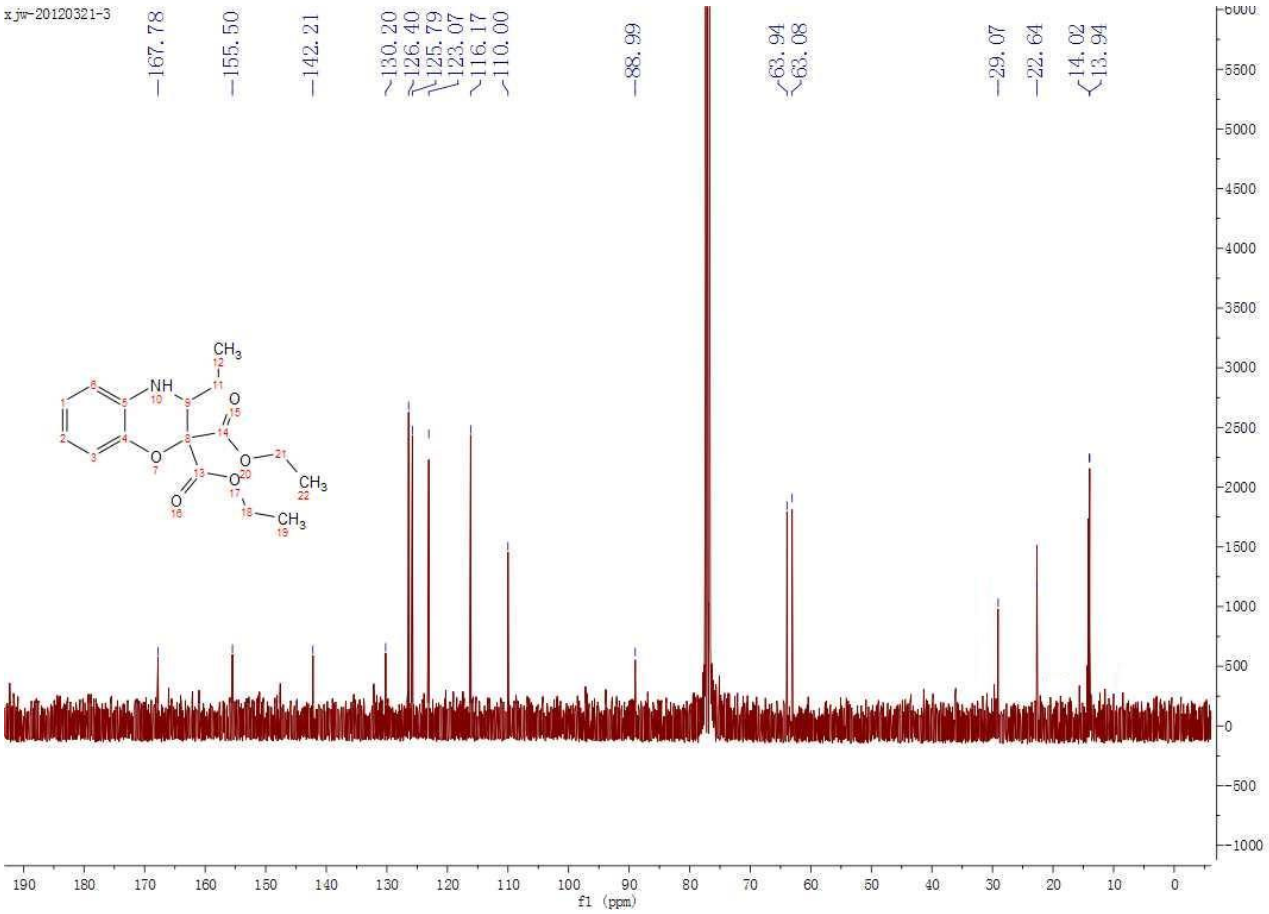
62.58
56.65
13.89
13.75
f1 (ppm)

3qa


xjw-20110908-2
7.28
7.13
7.11
7.09
7.06
7.04
7.02
6.99
6.97
4.93
4.50
4.49
4.39
4.36
4.34
3.71
1.58
1.36
1.35
1.35
1.33
1.33
1.26
1.24
1.23
1.00
1.01
1.04
0.98
0.99
2.05
1.96
0.95
3.01
3.03
3.17
2.00
f1 (ppm)


xjw-20120321-3
167.78
155.50
142.21
130.20
126.40
125.79
123.07
116.17
110.00
88.99
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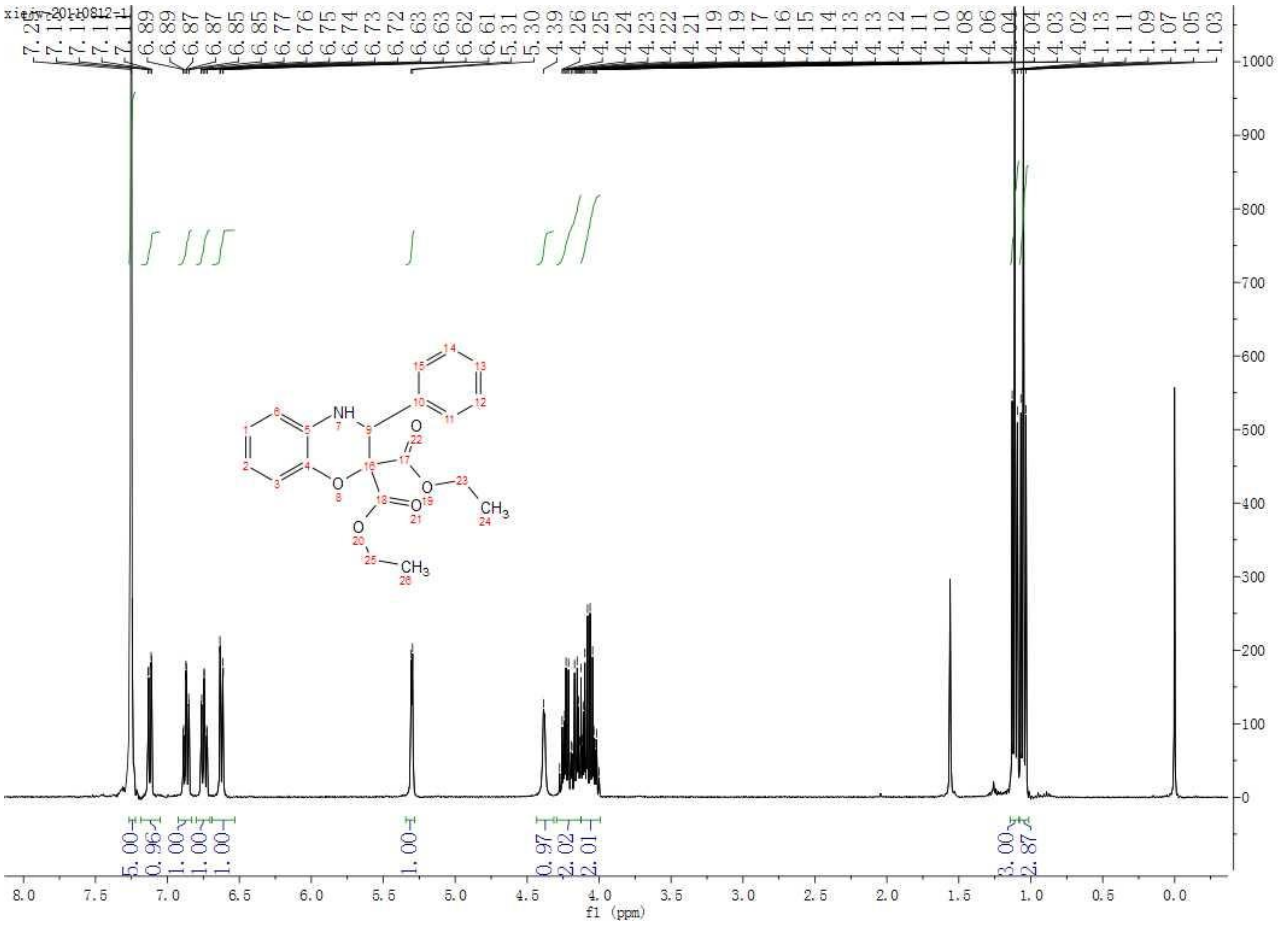
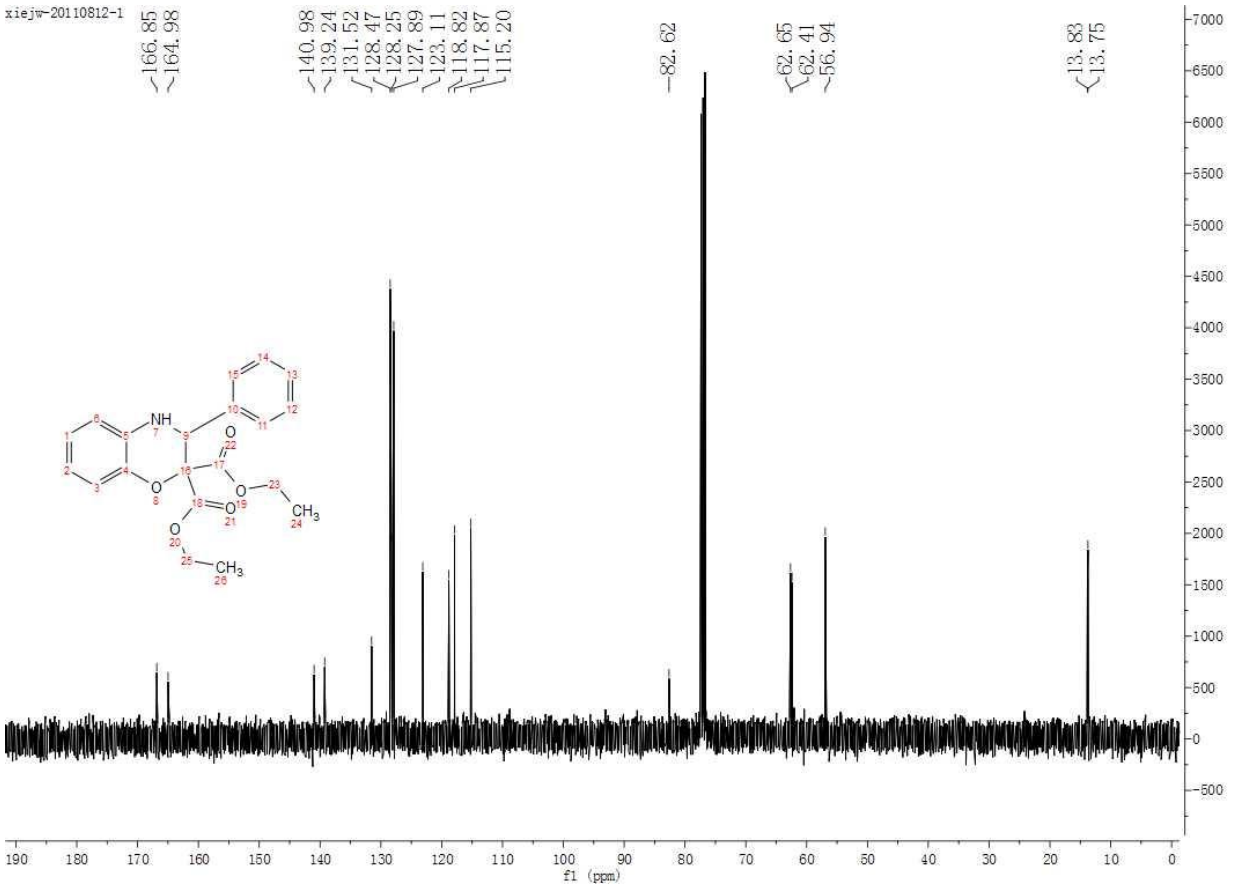
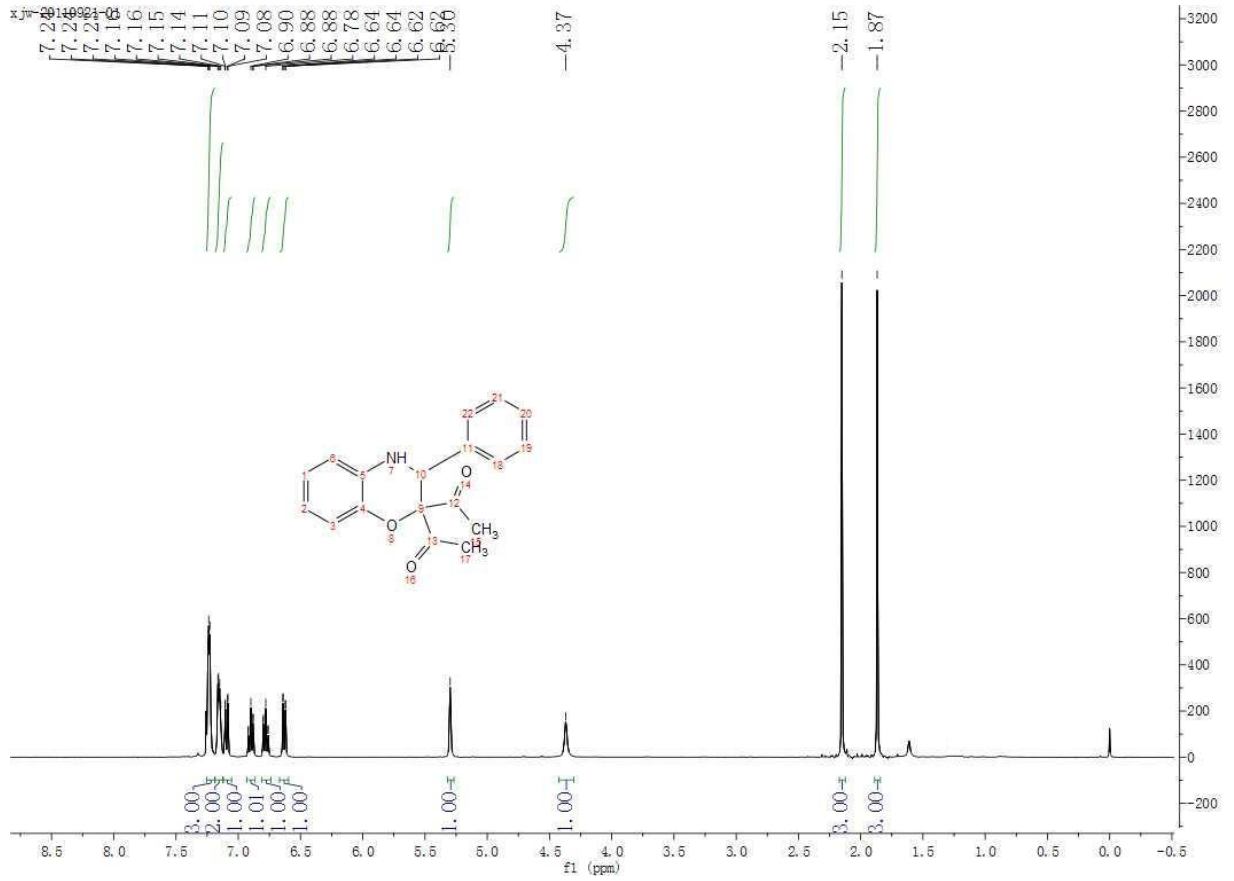
29.07
22.64
14.02
13.94
f1 (ppm)

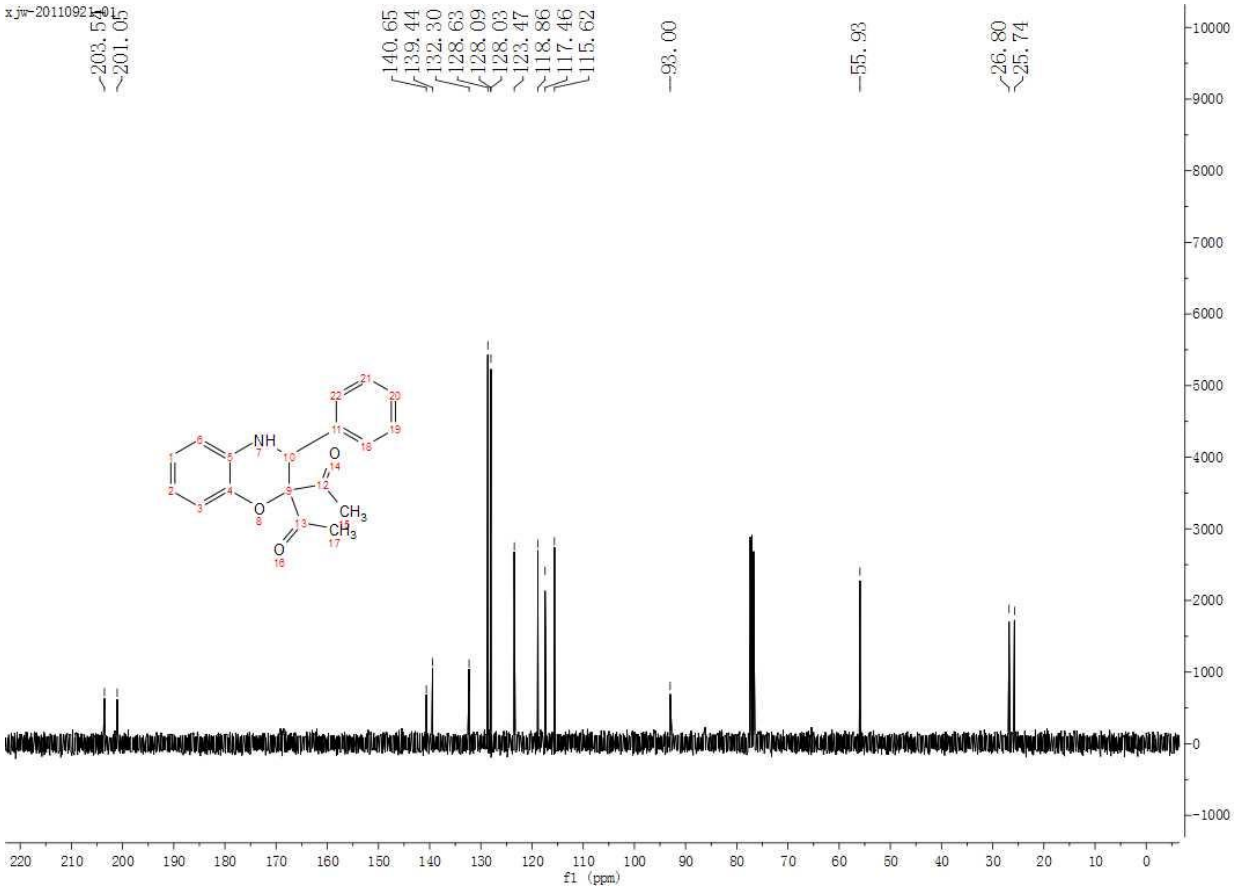
3ab





3ac


7.25
7.24
7.16
7.15
7.14
7.11
7.10
7.09
7.08
6.90
6.88
6.88
6.78
6.64
6.64
6.62
6.62
5.36
4.37
2.15
1.87
NH
O
CH3
CH3
3.00
2.00
1.00
1.01
1.00
1.00
1.00
1.00
3.00
3.00
f1 (ppm)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201.05
140.65
139.44
132.30
128.63
128.09
128.03
123.47
118.86
117.46
115.62
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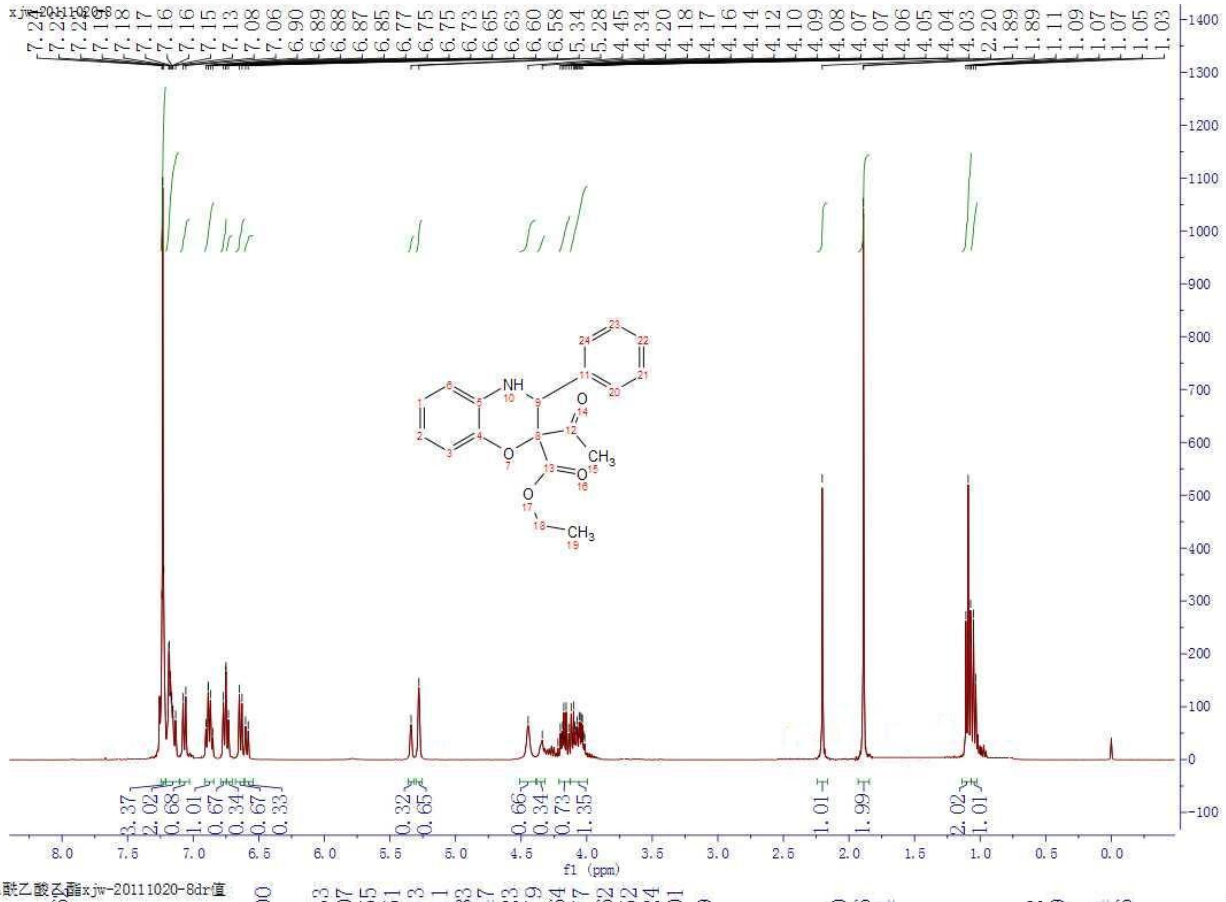
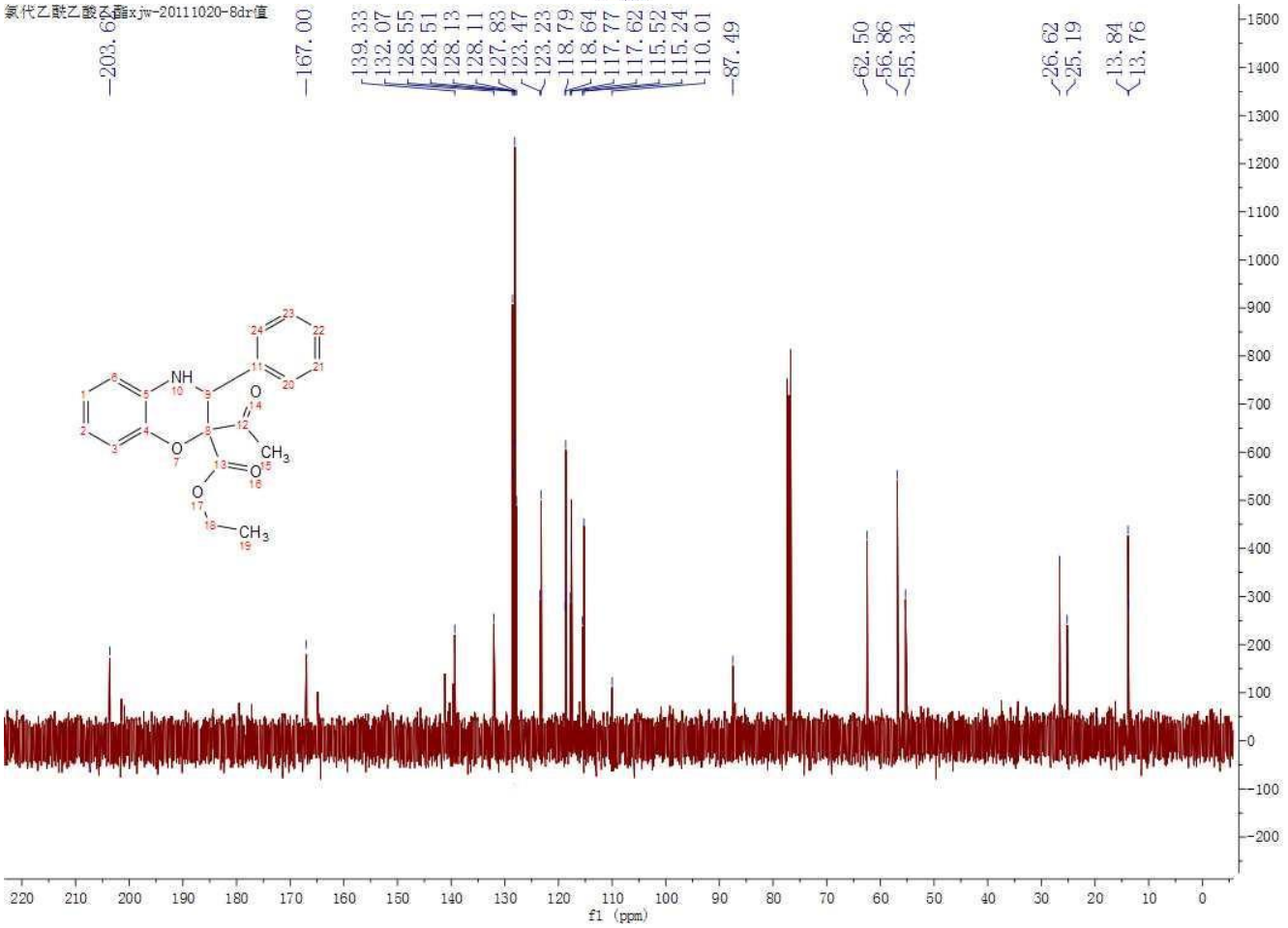
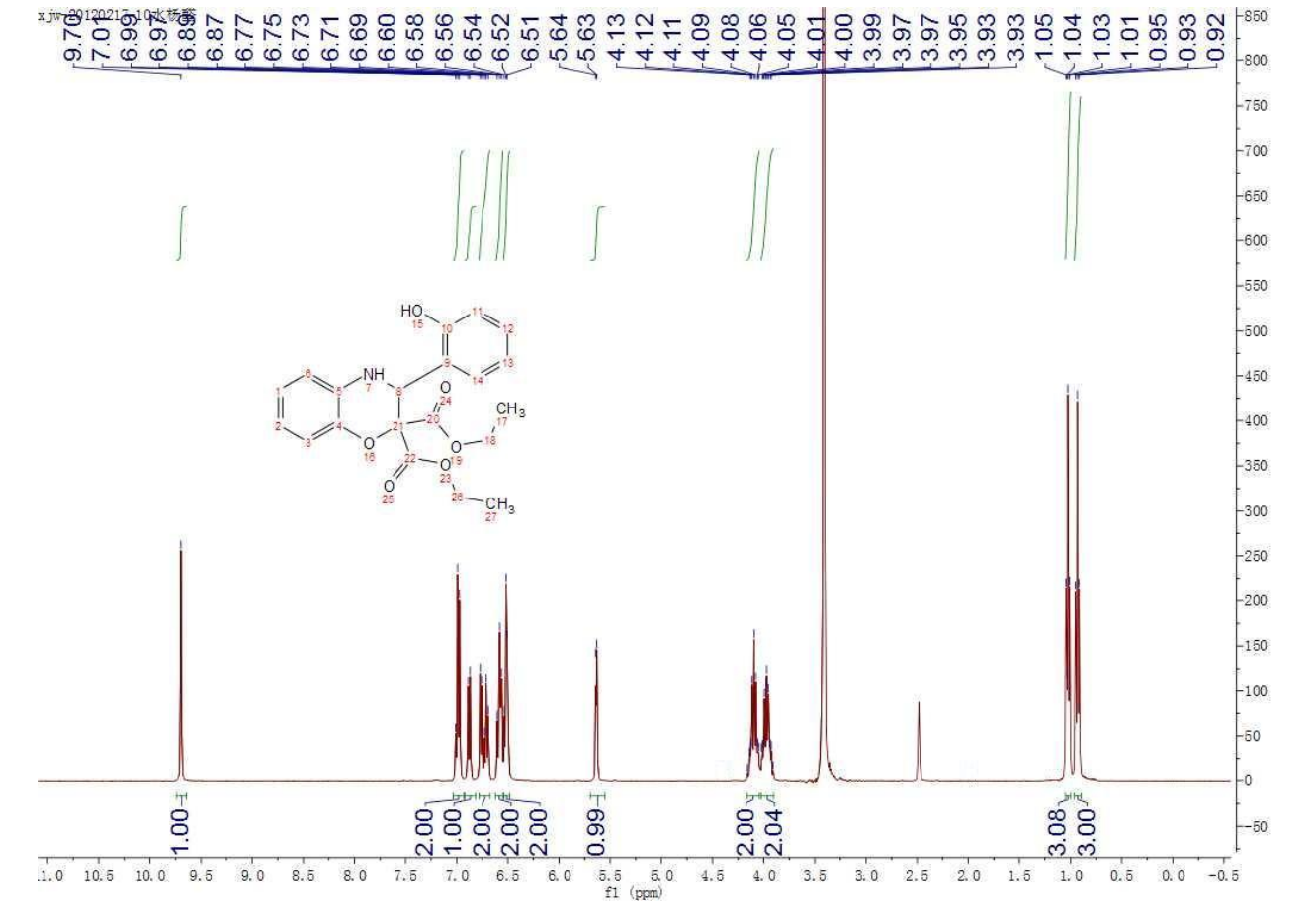
25.74
NH
O
CH3
CH3
f1 (ppm)

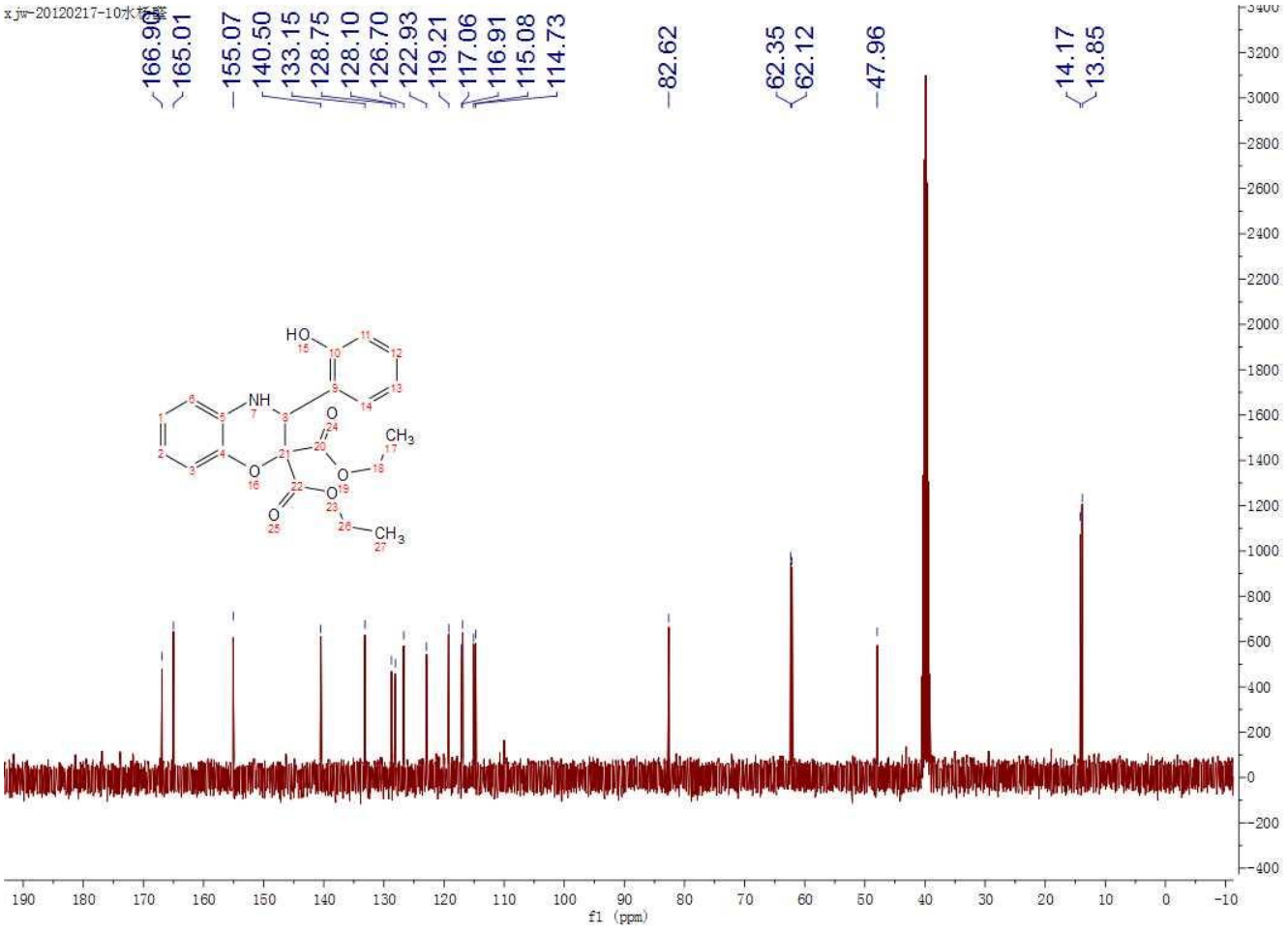
3ad



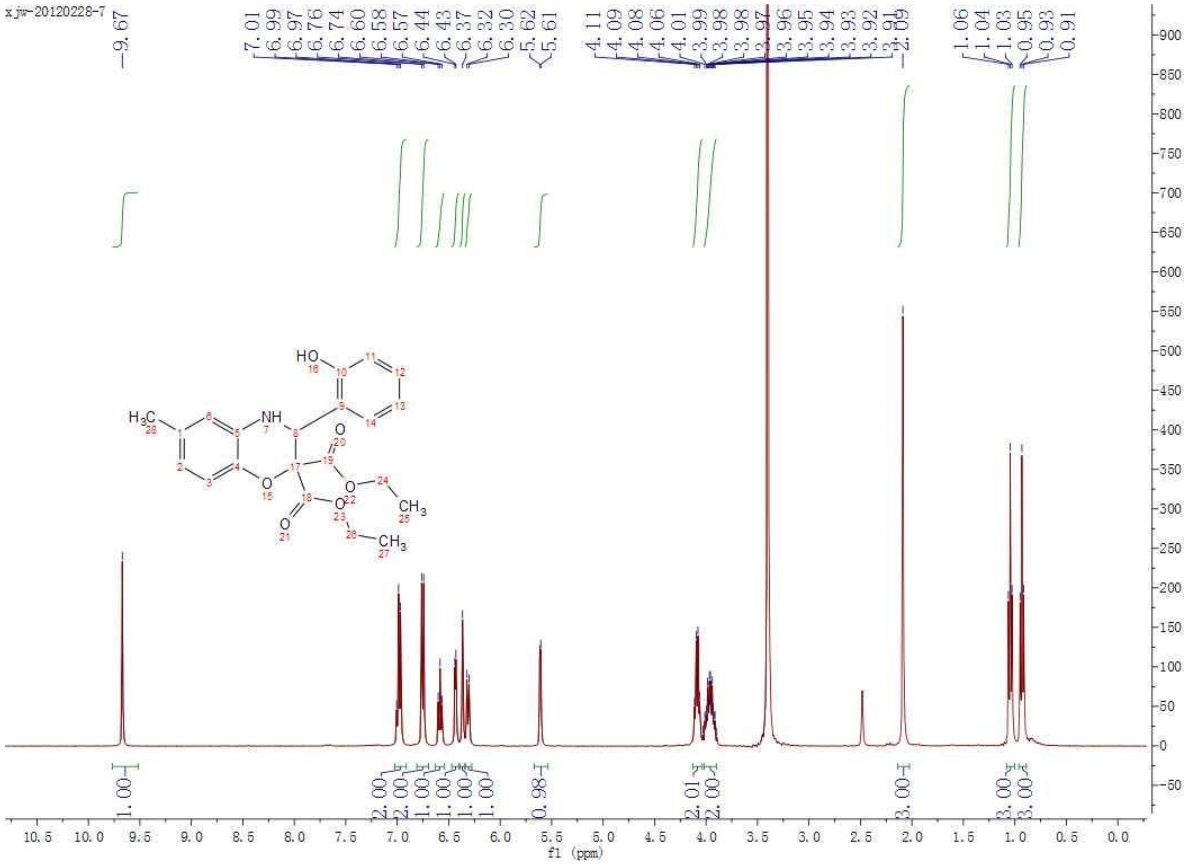


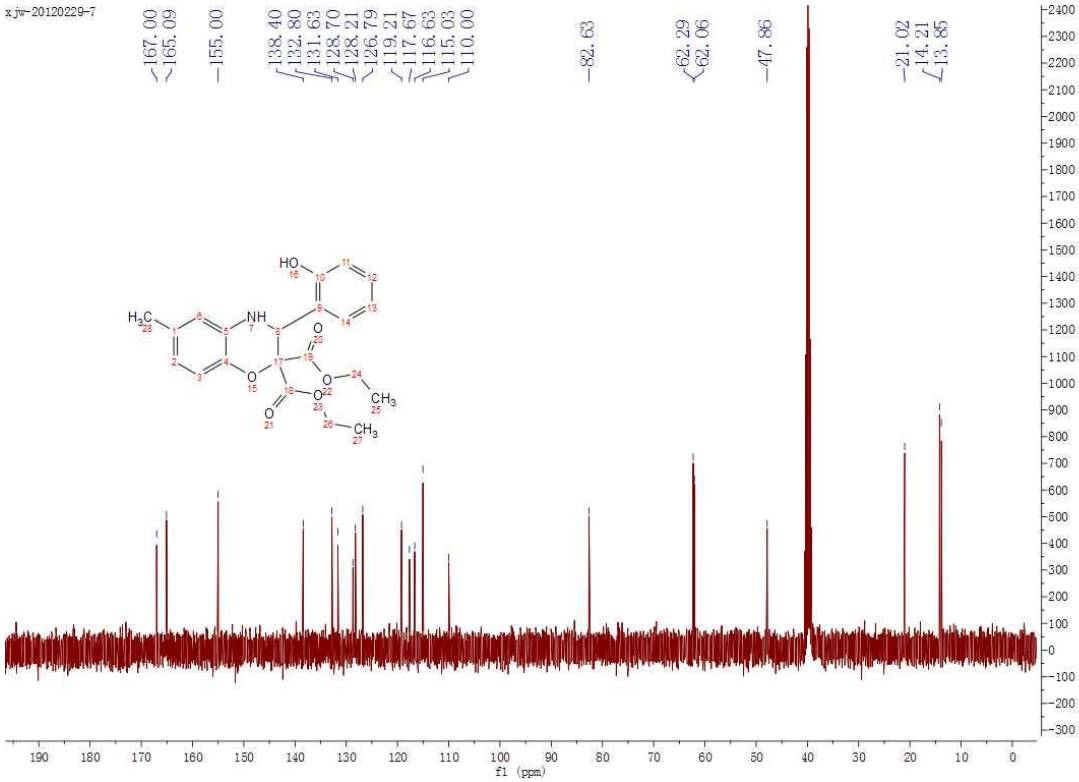
3ra



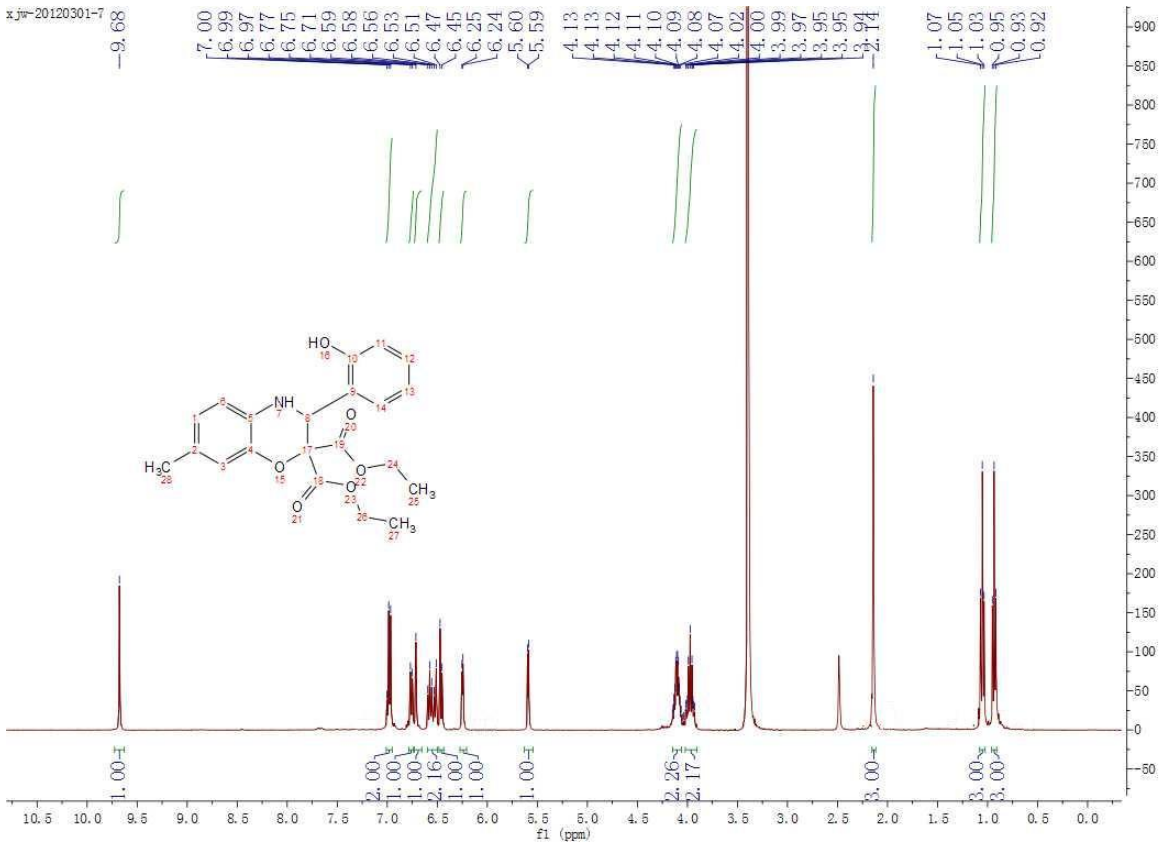


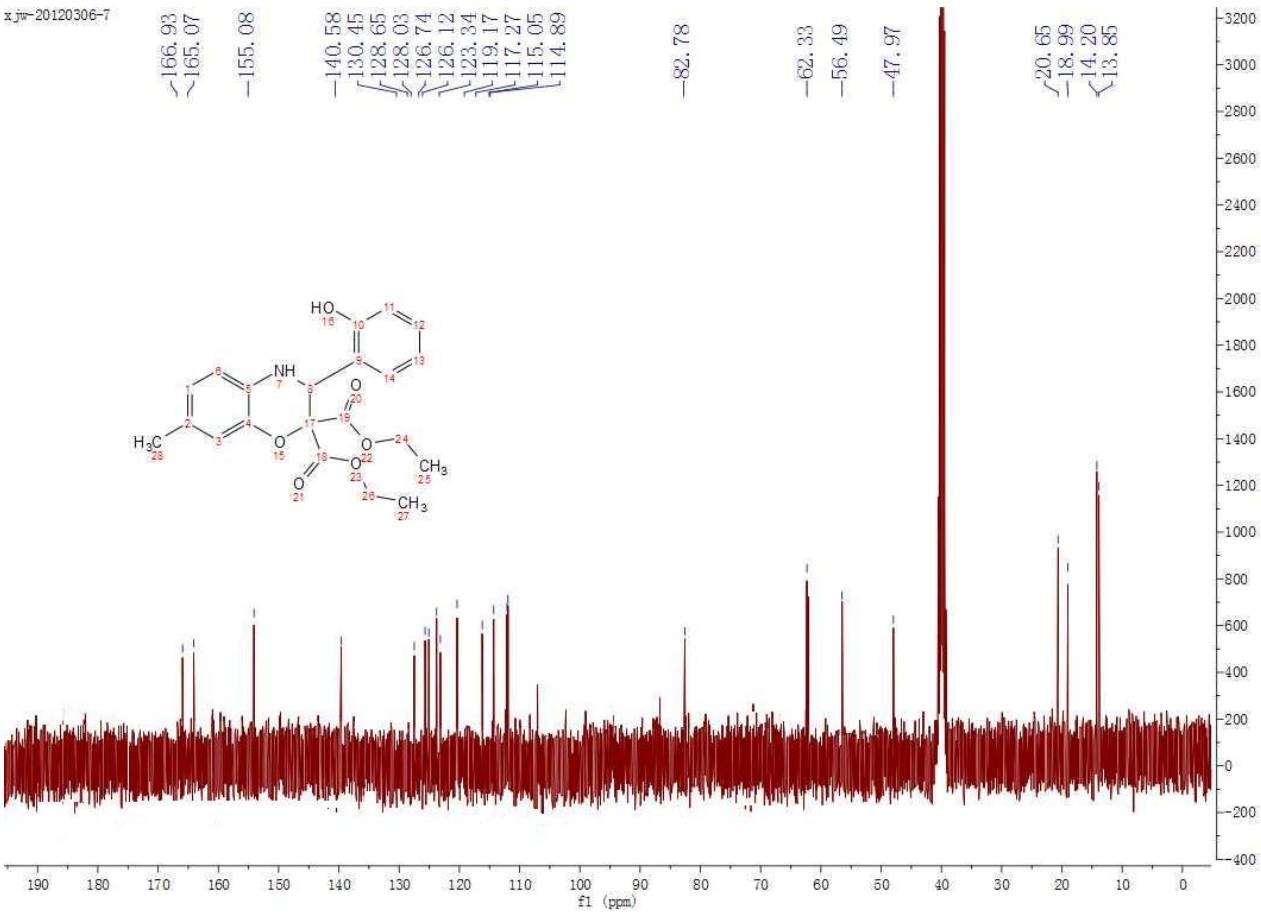
3sa



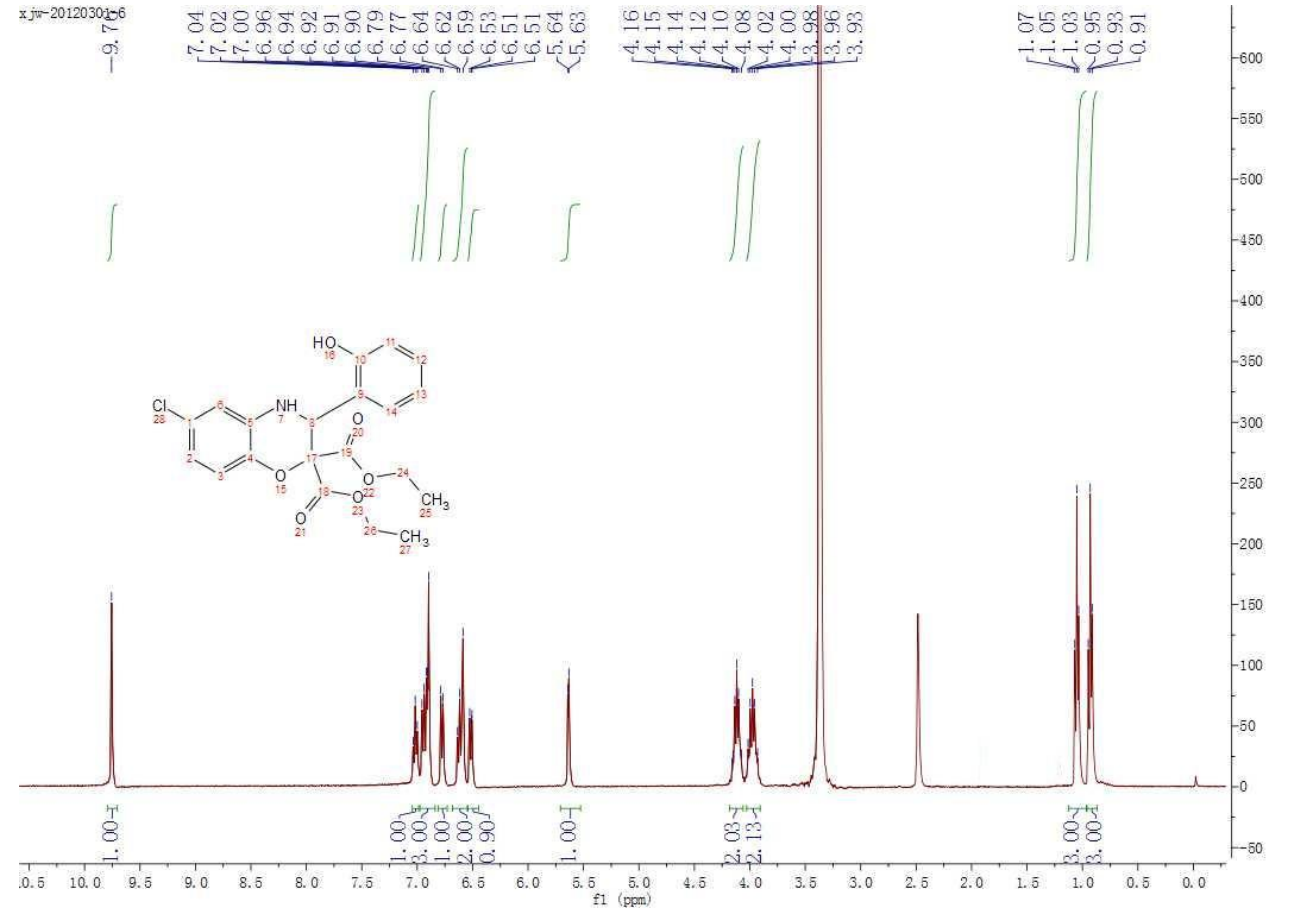


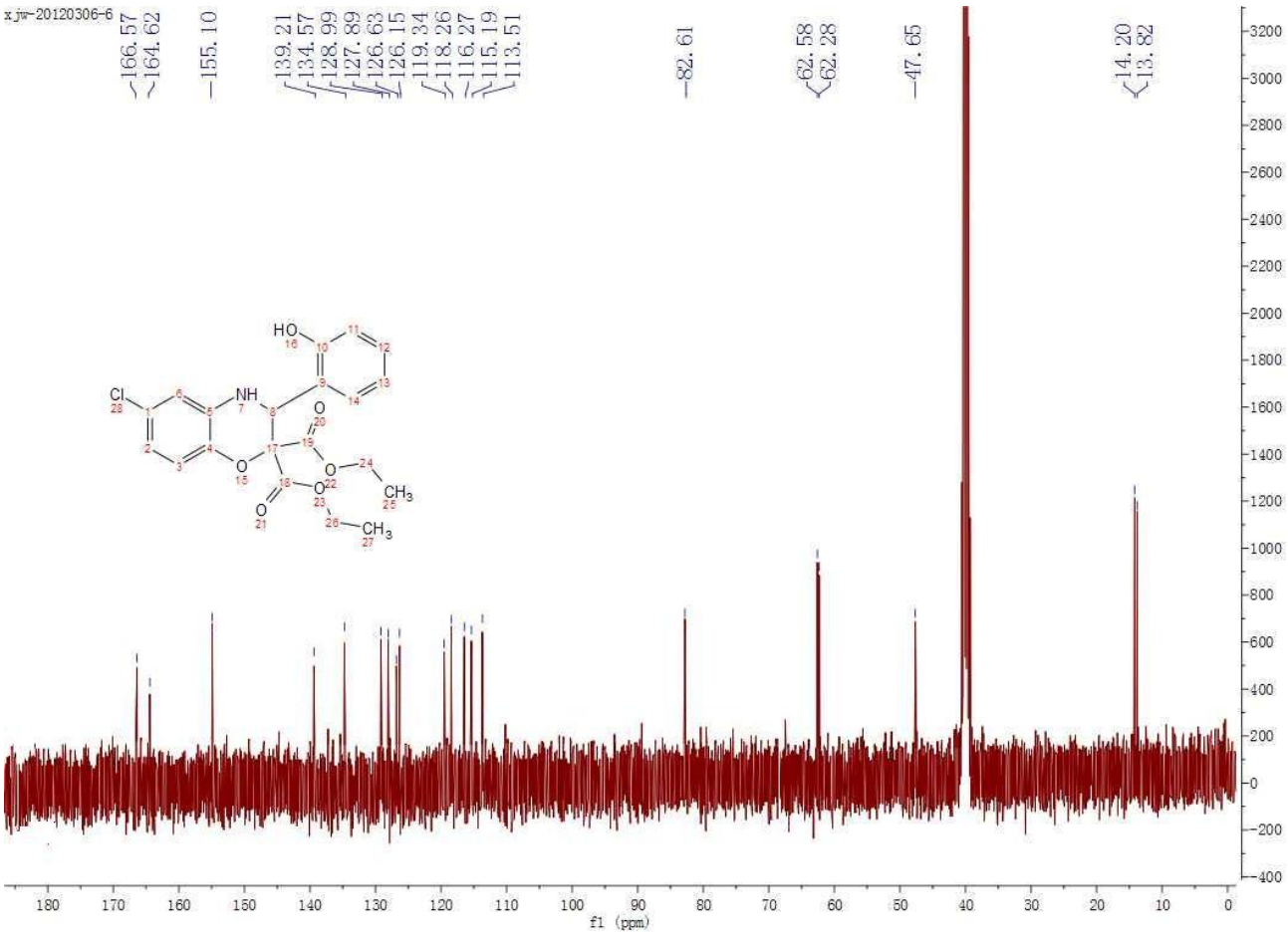
3ta



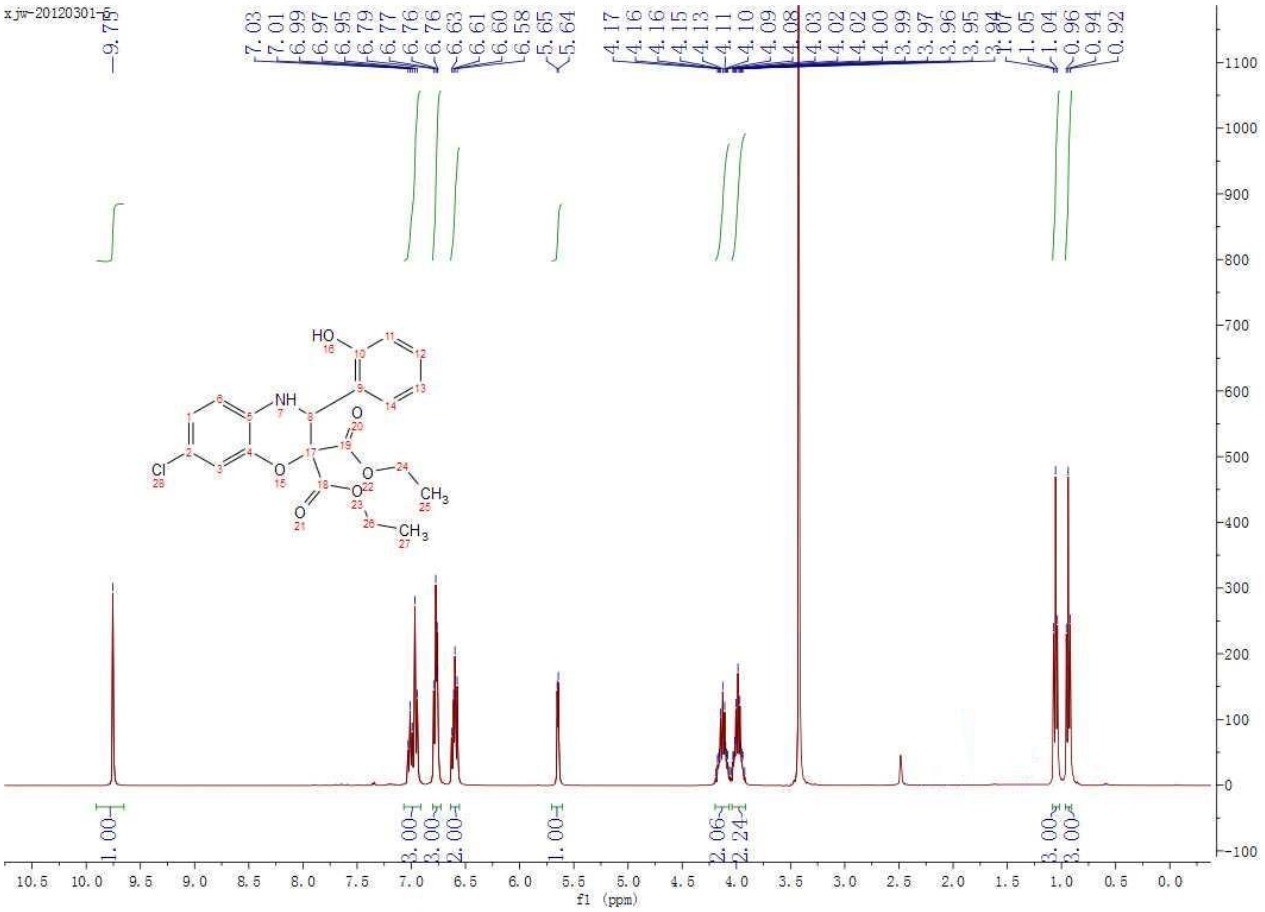


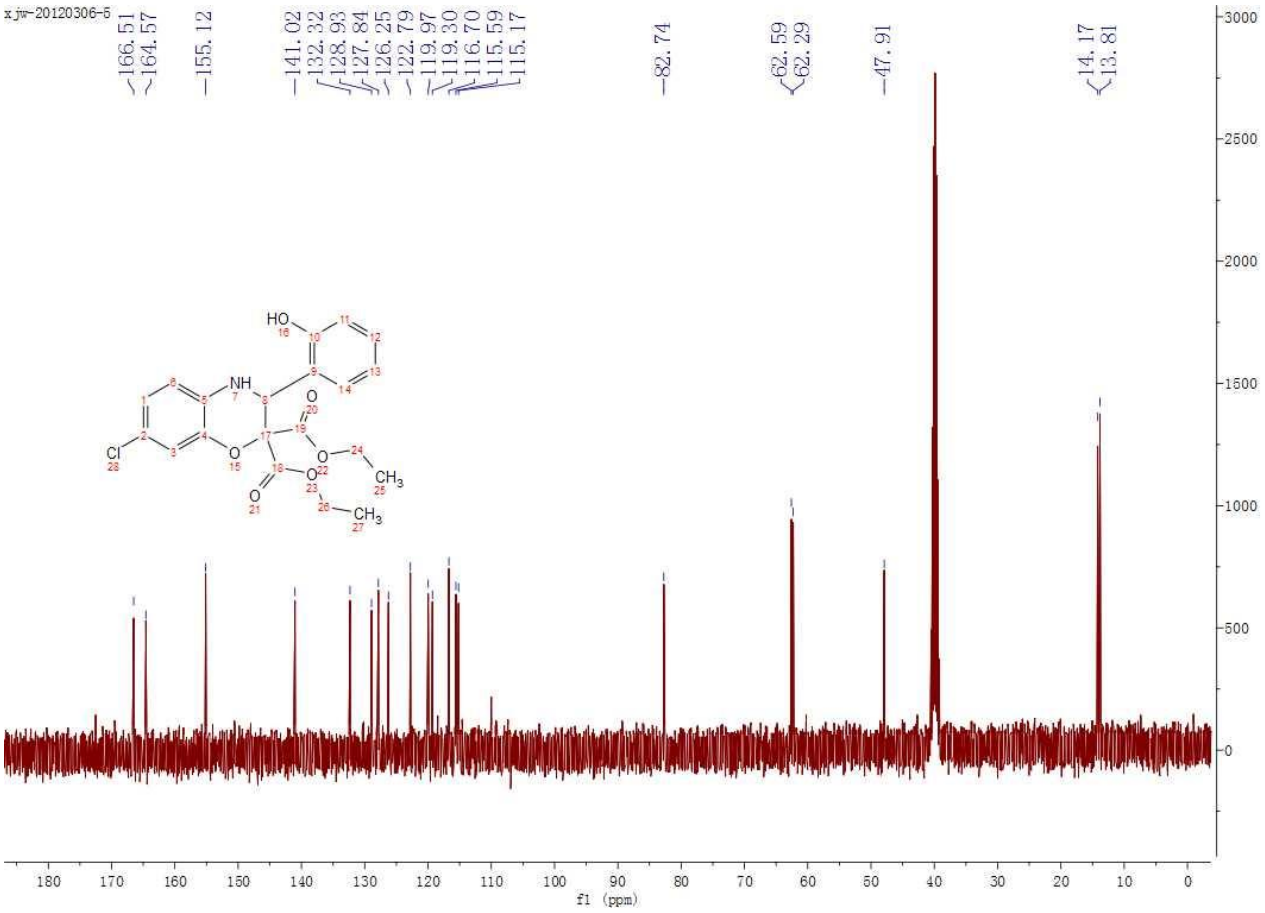
3ua



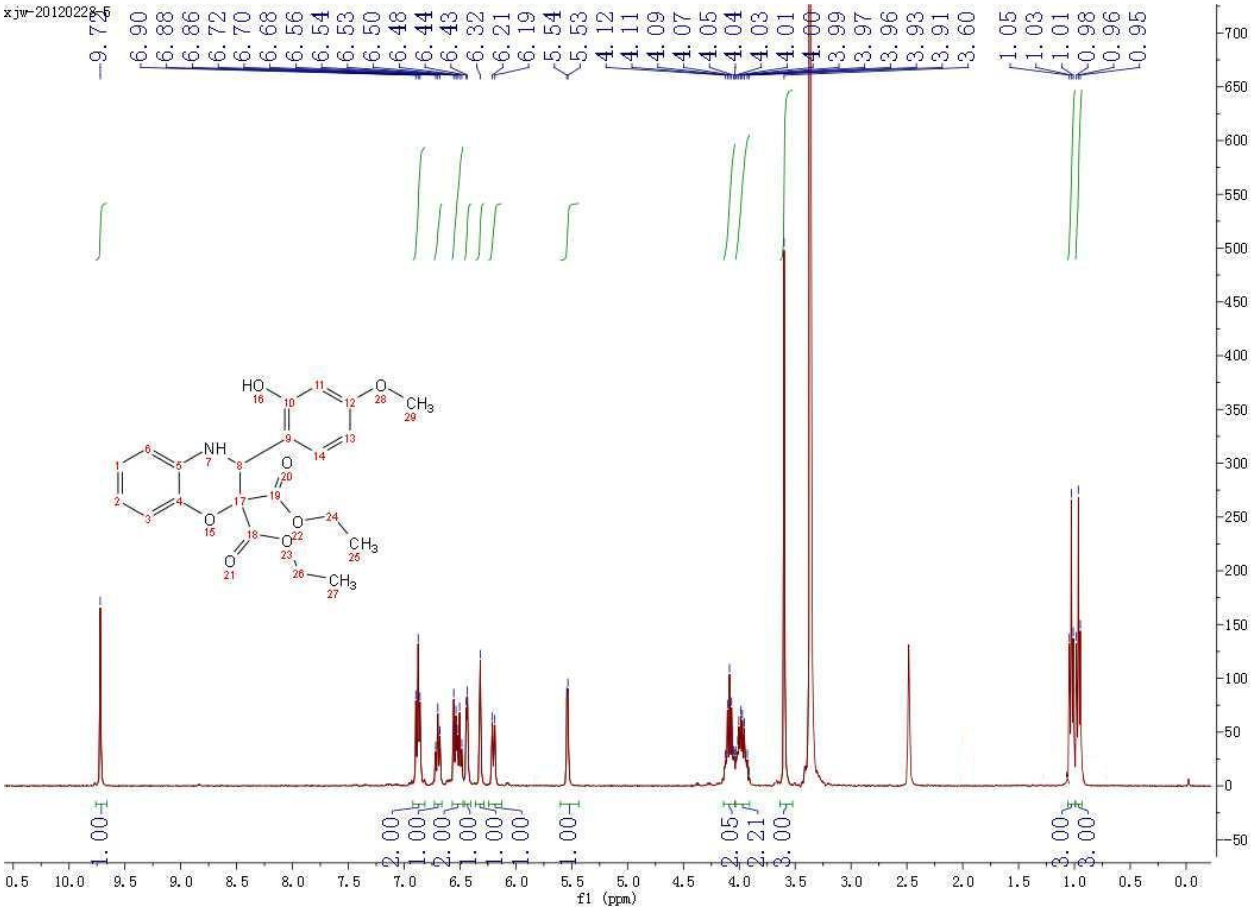


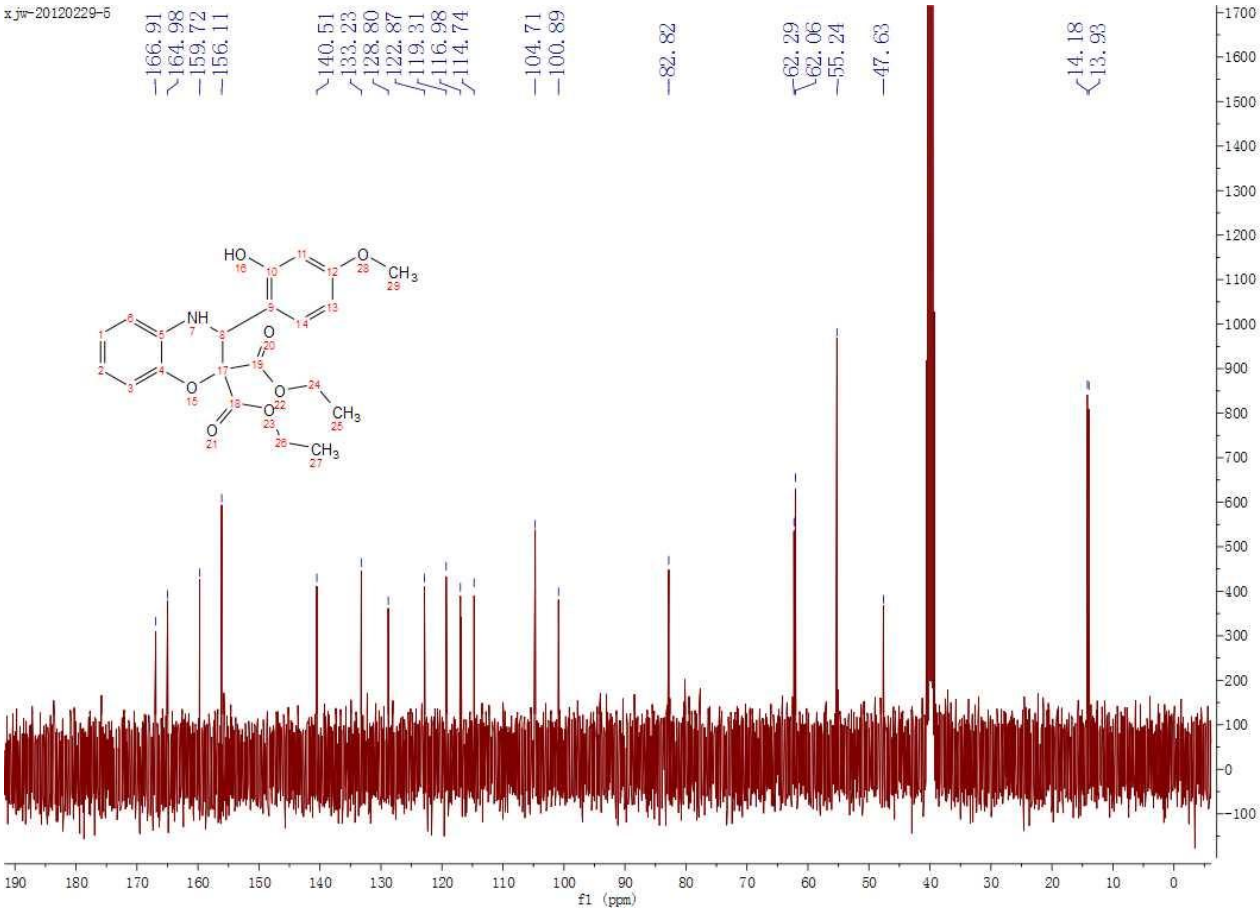
3va


xjw-20120301-6
9.75
7.03
7.01
6.99
6.97
6.95
6.79
6.77
6.76
6.76
6.63
6.61
6.60
6.58
5.65
5.64
4.17
4.16
4.16
4.15
4.13
4.11
4.10
4.09
4.08
4.03
4.02
4.02
4.00
3.99
3.97
3.96
3.95
1.07
1.05
1.04
0.96
0.94
0.92
HO
NH
Cl
O
CH3
1.00
3.00
3.00
2.00
1.00
2.06
2.24
3.00
3.00
f1 (ppm)

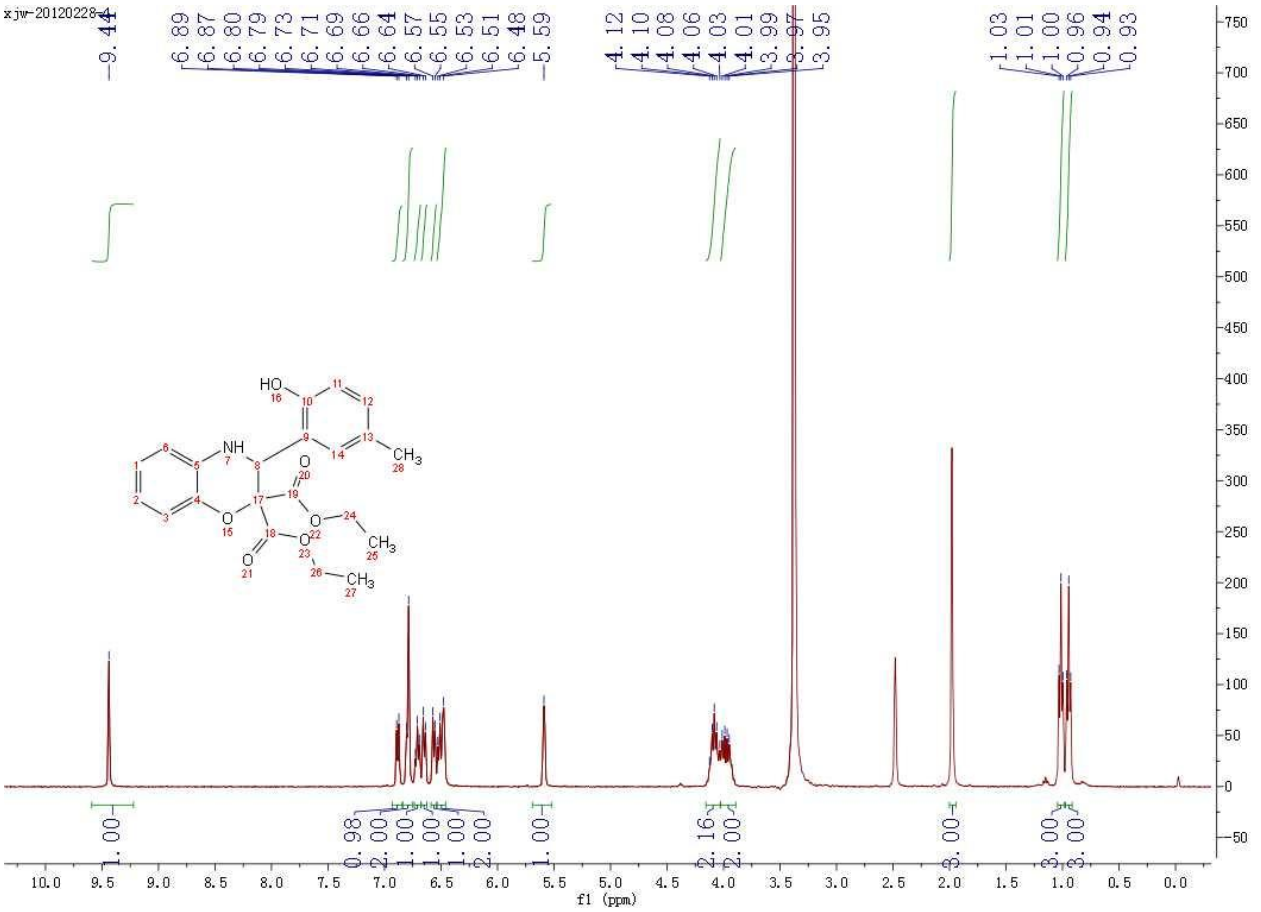

xjw-20120306-6
166.51
164.57
155.12
141.02
132.32
128.93
127.84
126.25
122.79
119.97
119.30
116.70
115.59
115.17
82.74
62.59
62.29
47.91
14.17
13.81
HO
NH
Cl
O
CH3
f1 (ppm)

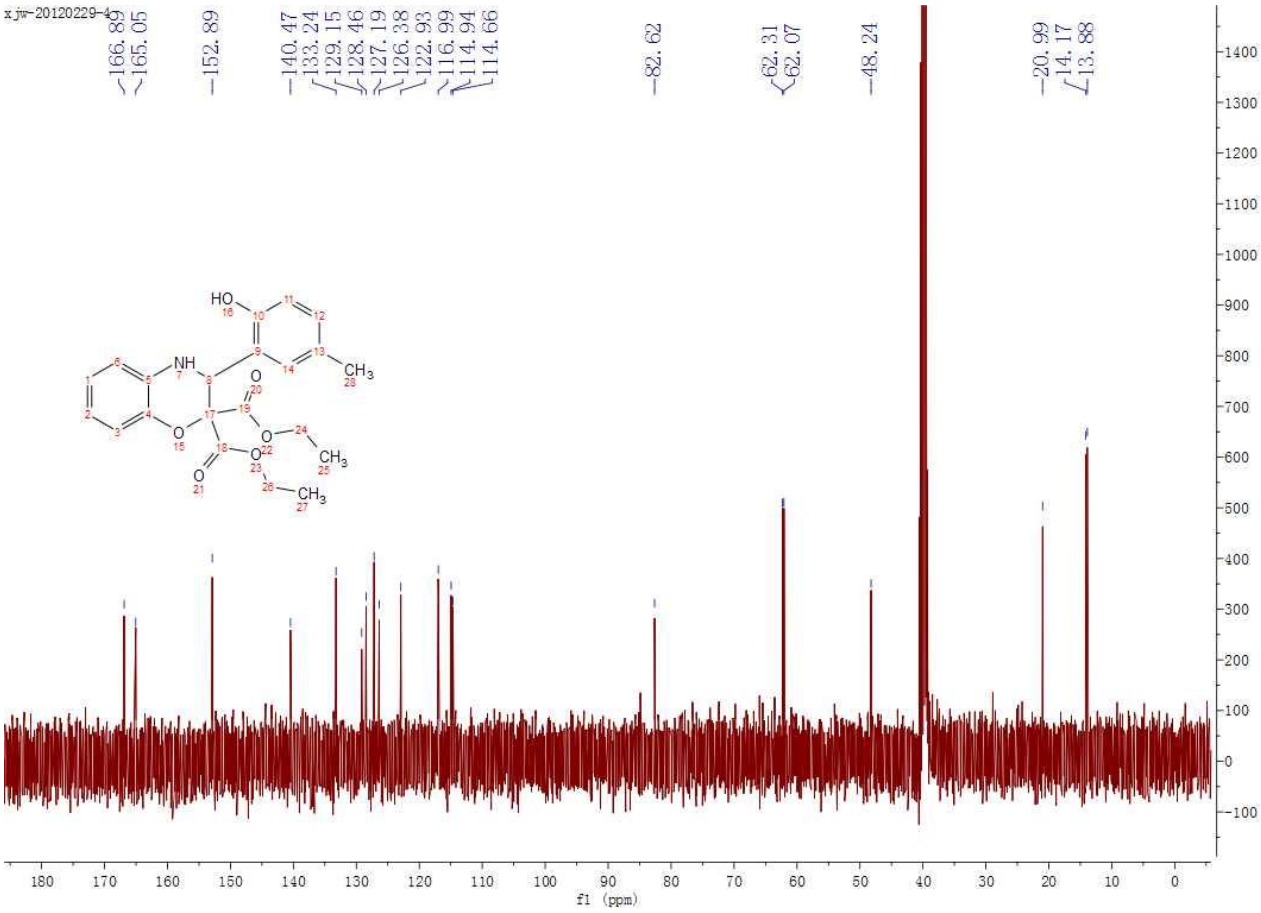
3wa


xjw-20120228-5
9.72
6.90
6.88
6.86
6.72
6.70
6.68
6.56
6.54
6.53
6.50
6.48
6.44
6.43
6.32
6.21
6.19
5.54
5.53
4.12
4.11
4.09
4.07
4.05
4.04
4.03
4.01
4.00
3.99
3.97
3.96
3.93
3.91
3.60
1.05
1.03
1.01
0.98
0.96
0.95
HO
NH
O
CH3
f1 (ppm)

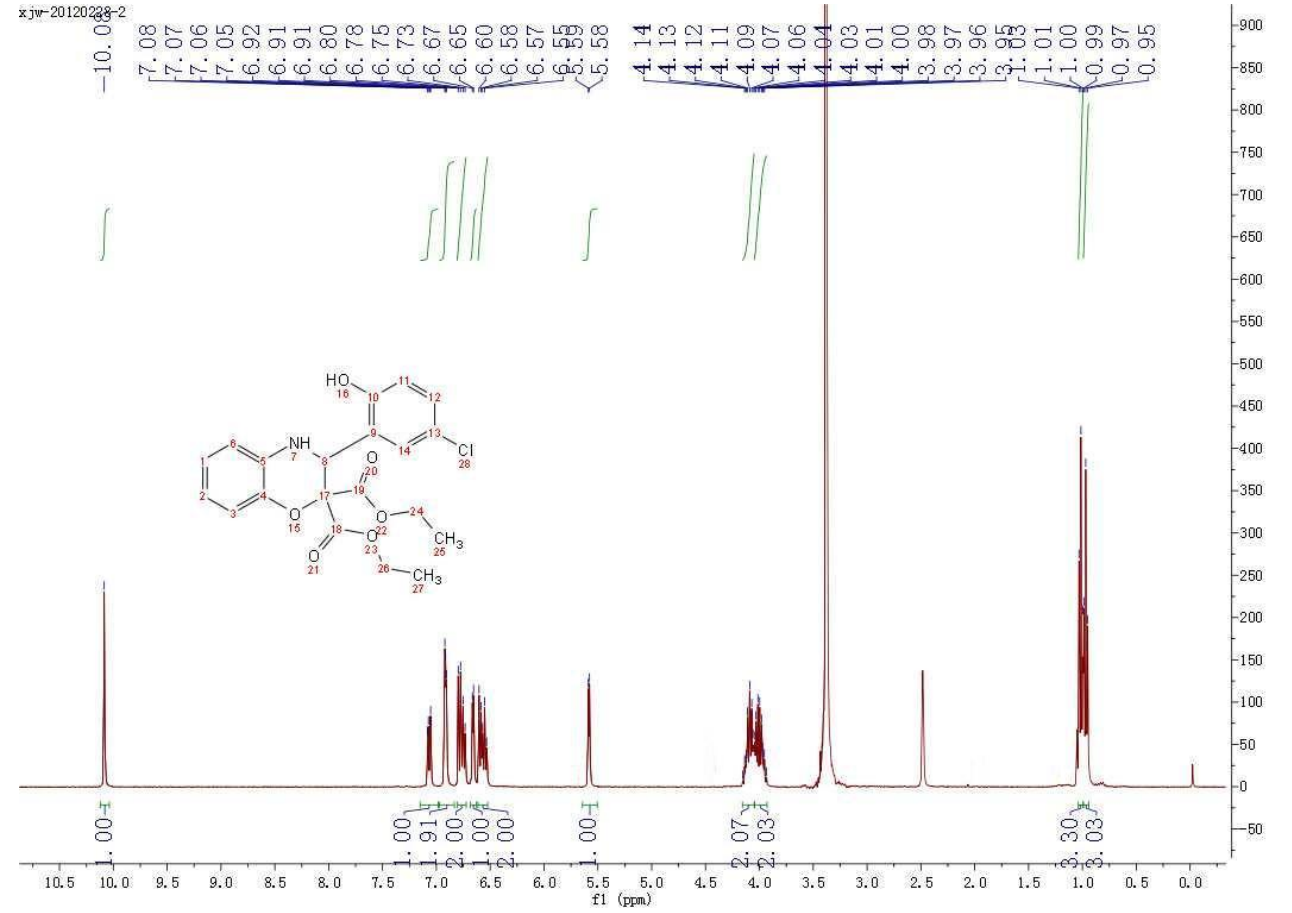

xjw-20120229-5
166.91
164.98
159.72
156.11
140.51
133.23
128.80
122.87
119.31
116.98
114.74
104.71
100.89
82.82
62.29
62.06
55.24
47.63
14.18
13.93
HO
NH
O
CH3
f1 (ppm)

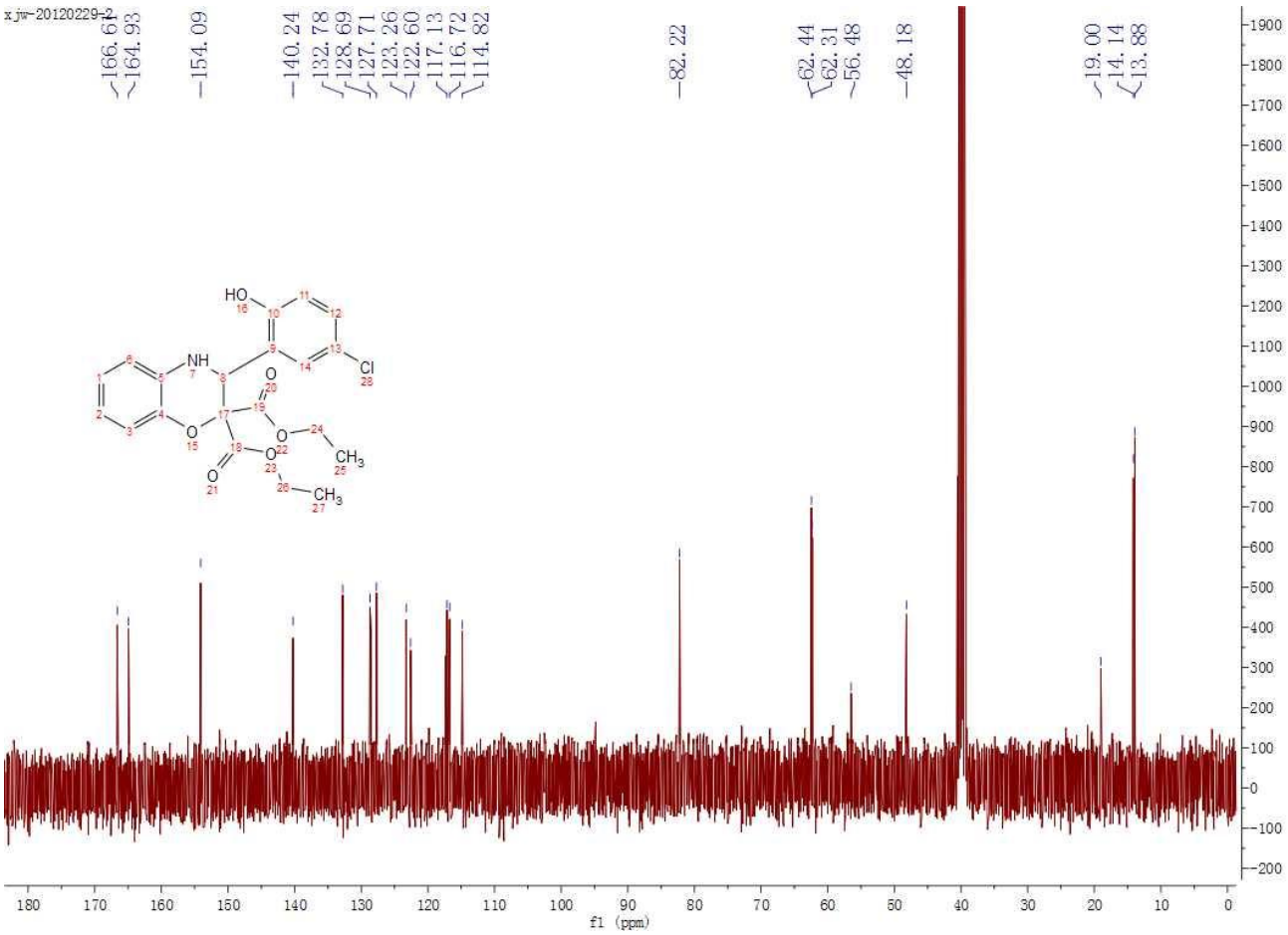
3xa



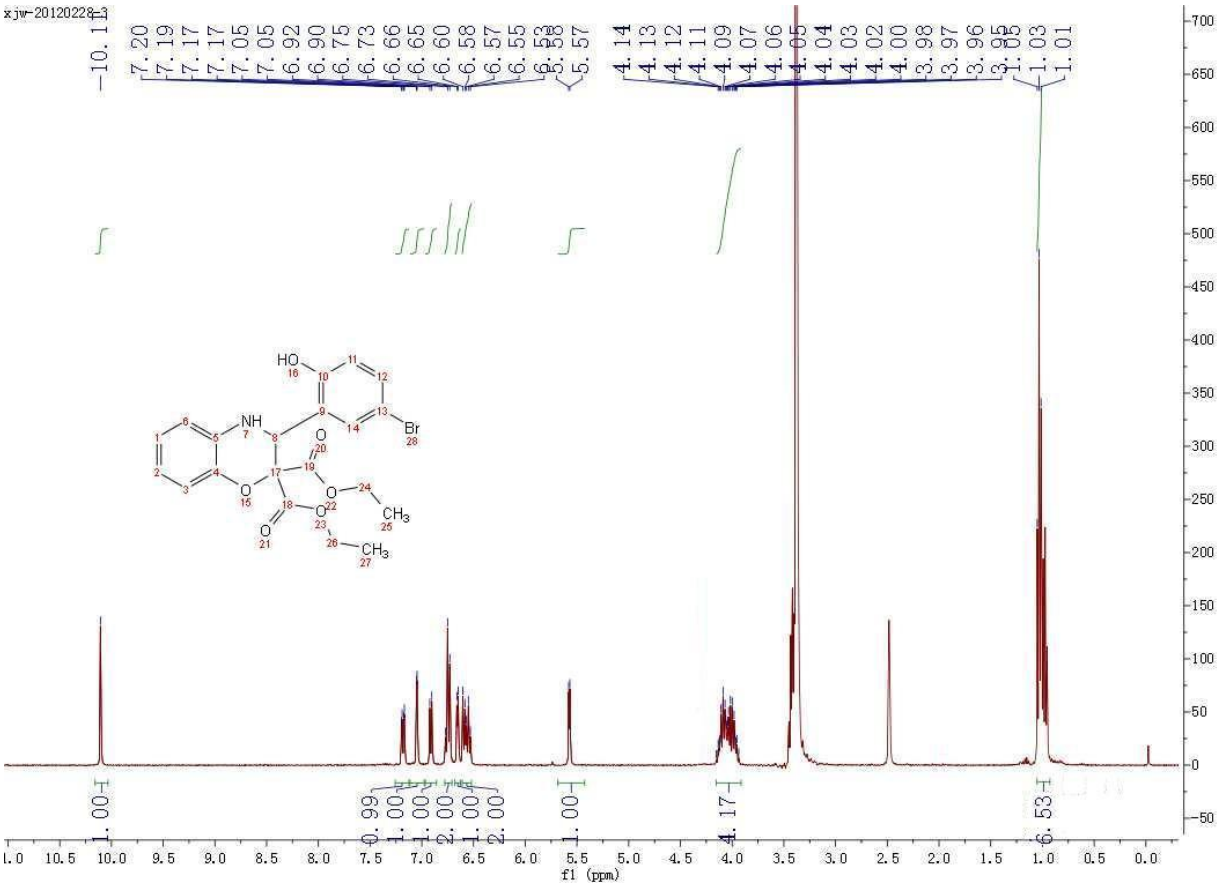


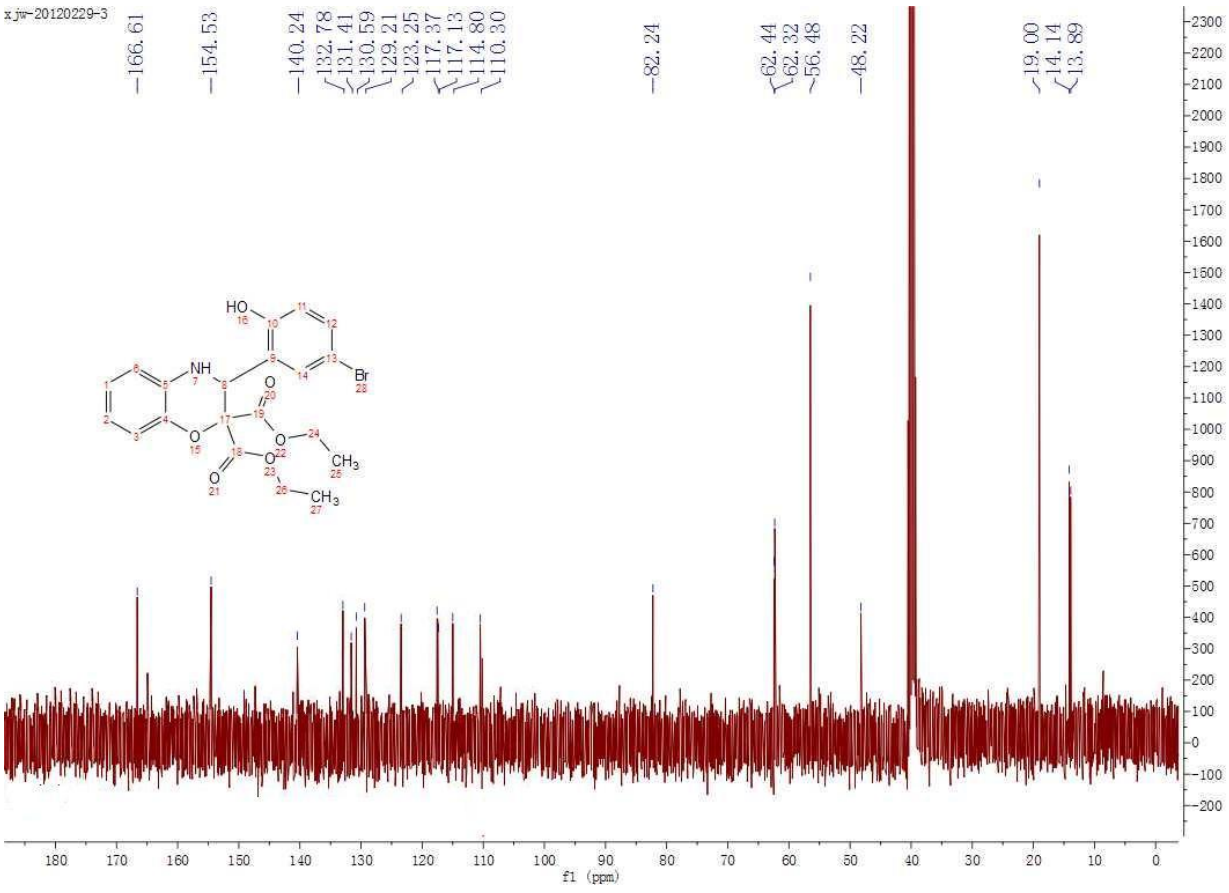
3ya



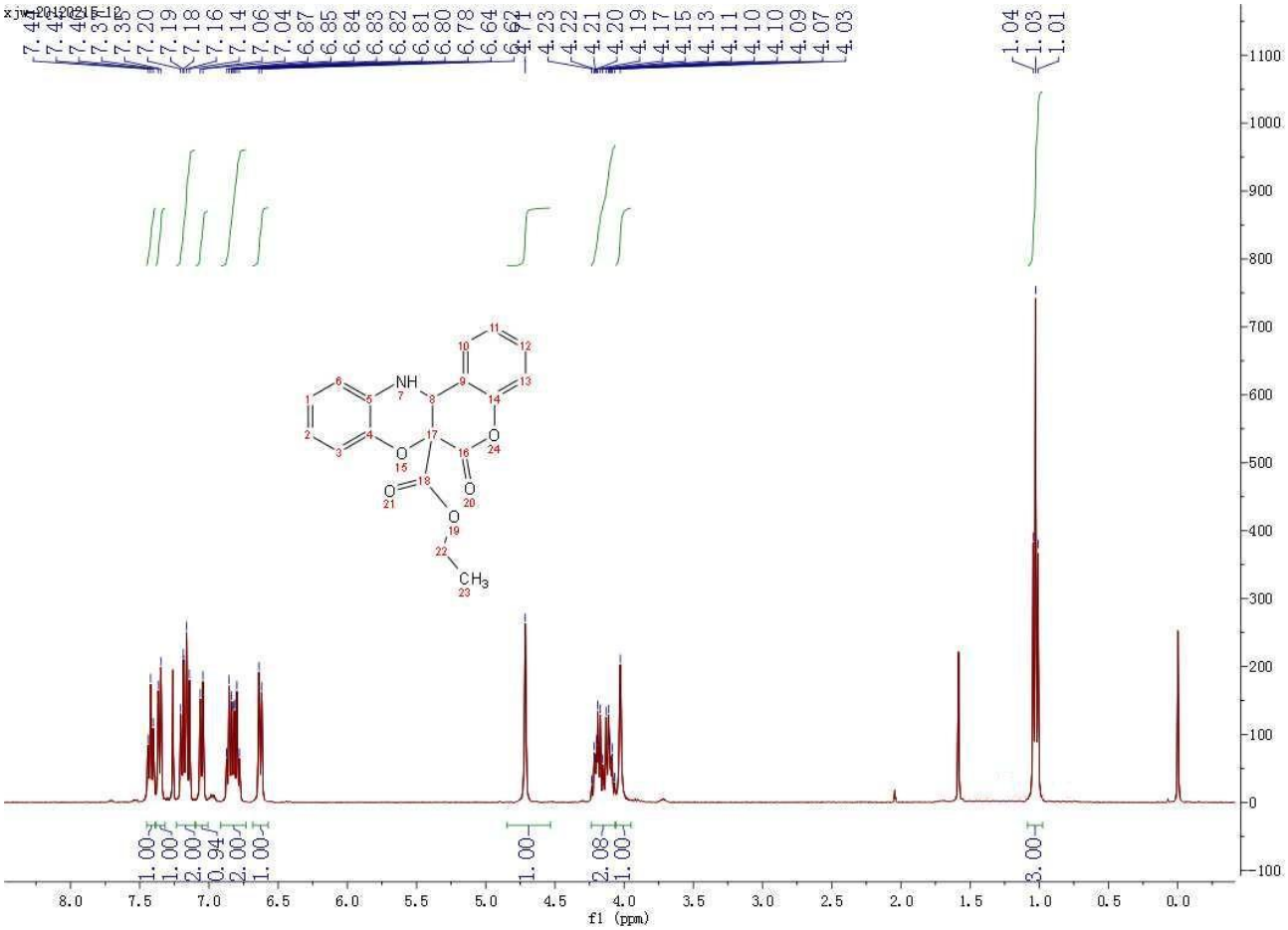


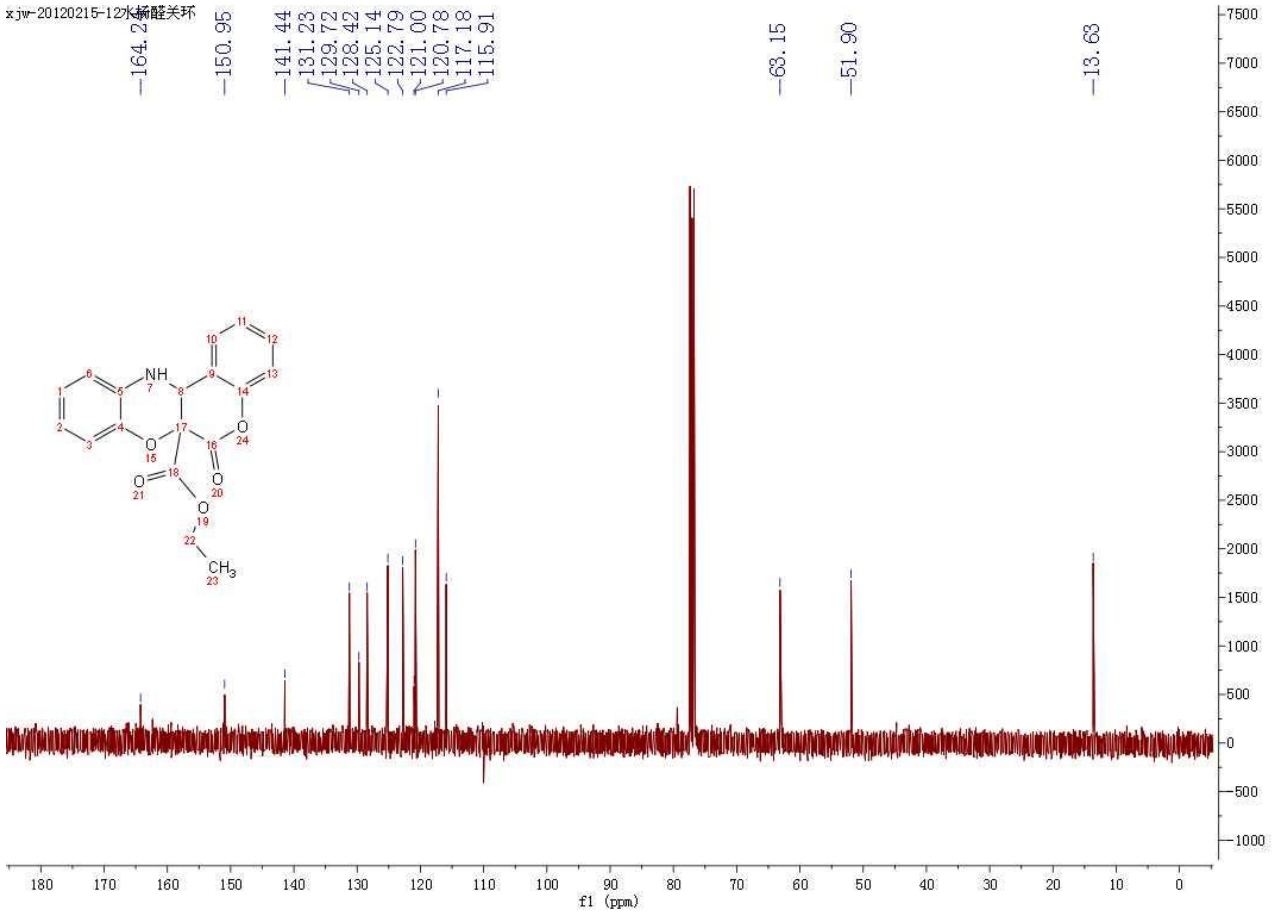
3za



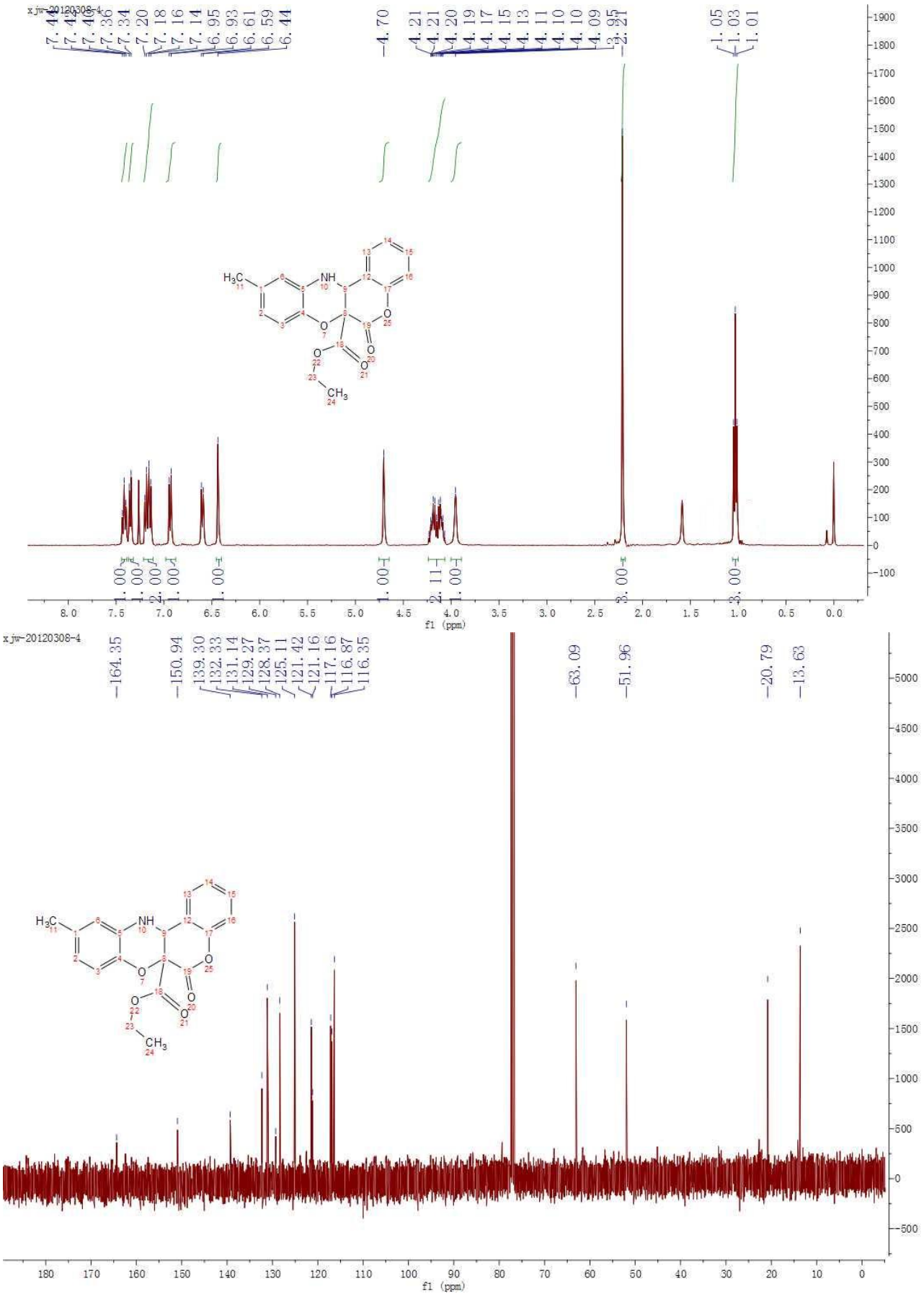


4ra

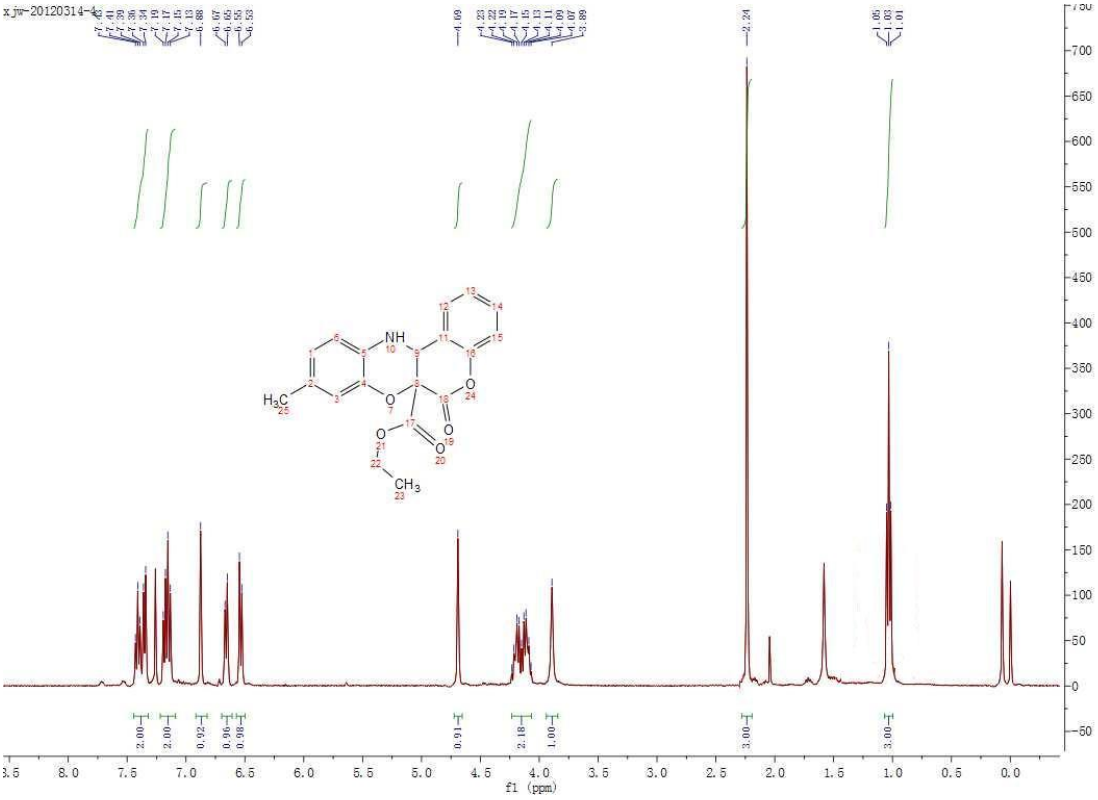

7.42
7.41
7.35
7.20
7.19
7.18
7.16
7.14
7.06
7.04
6.87
6.85
6.84
6.83
6.82
6.81
6.80
6.78
6.64
6.62
4.23
4.22
4.21
4.20
4.19
4.17
4.15
4.13
4.11
4.10
4.10
4.09
4.07
4.03
1.04
1.03
1.01
NH
O
CH3
1.00
1.00
2.00
0.94
2.00
1.00
1.00
2.08
1.00
3.00
f1 (ppm)

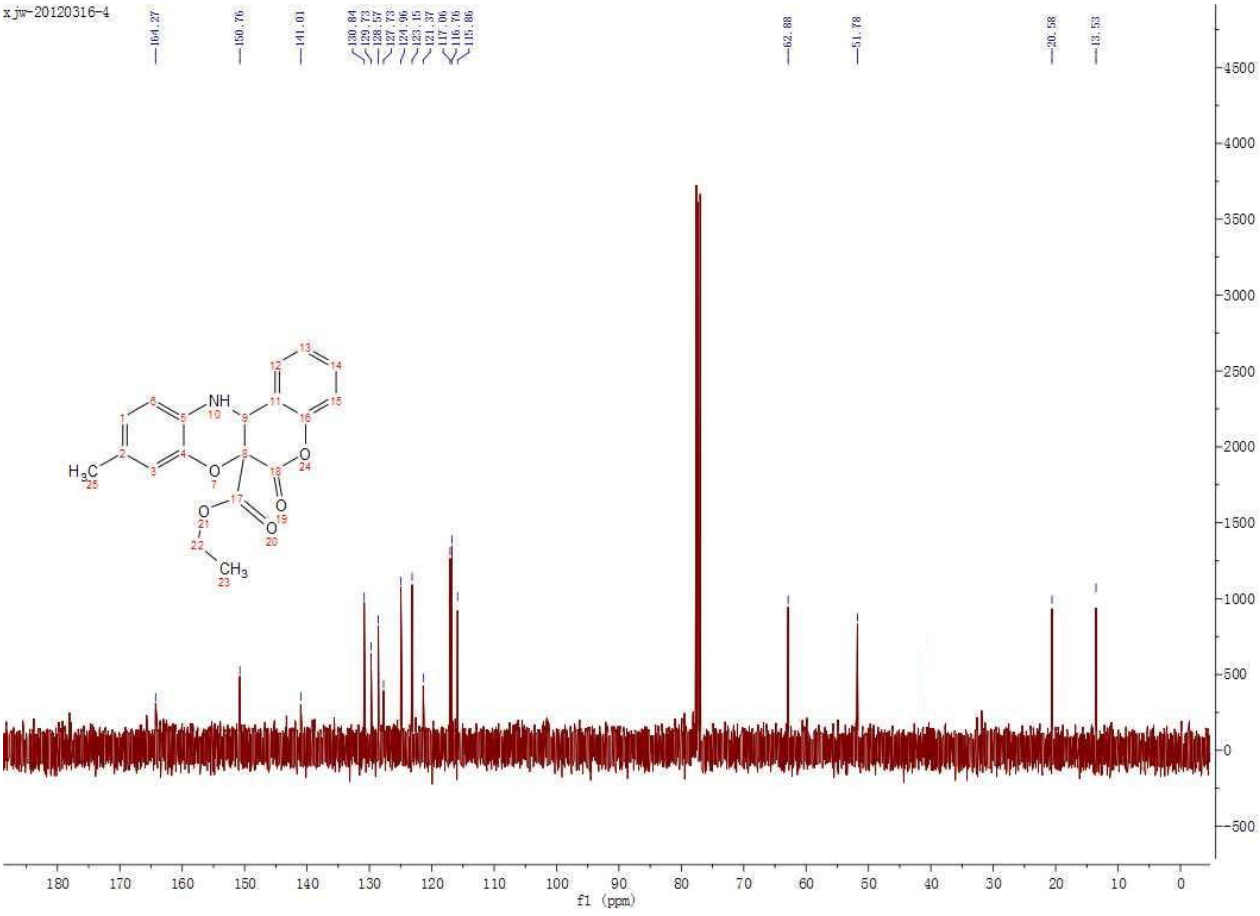

164.21
150.95
141.44
131.23
129.72
128.42
125.14
122.79
121.00
120.78
117.18
115.91
63.15
51.90
13.63
NH
O
CH3
f1 (ppm)

4sa

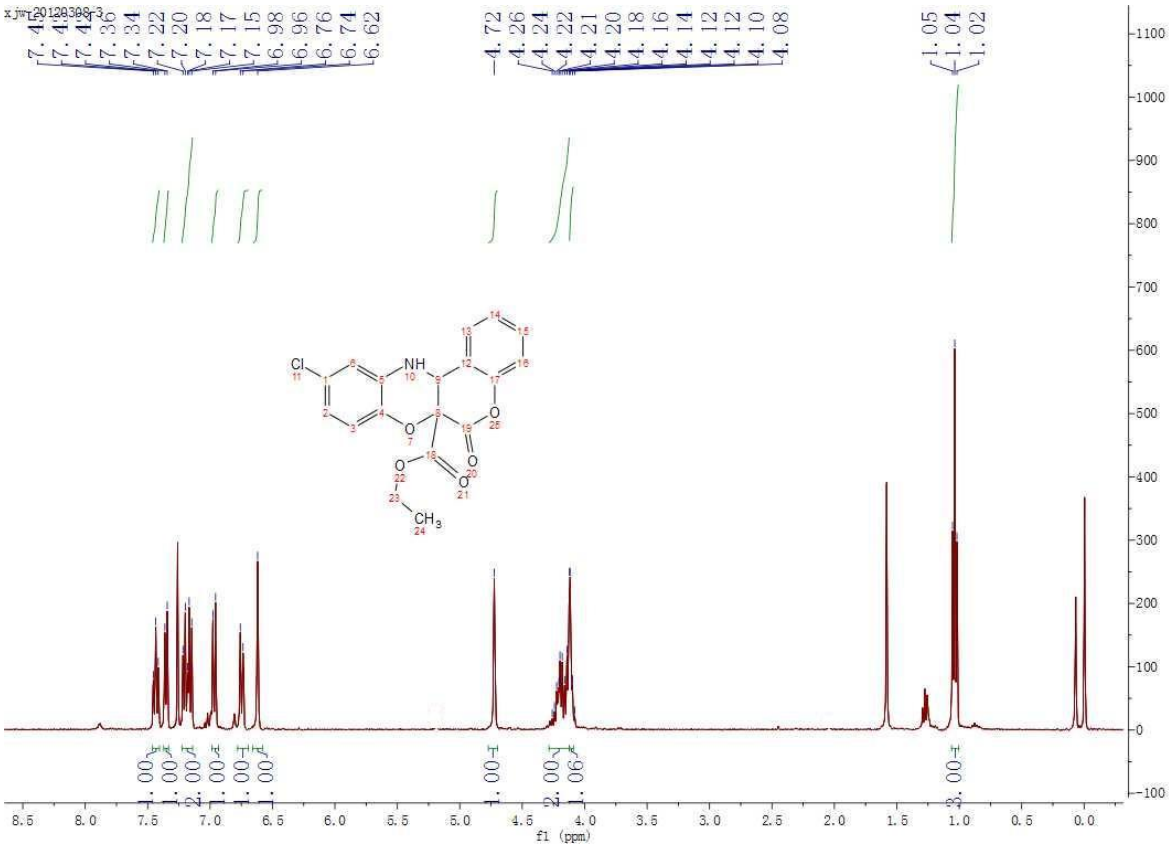

xjw-20120308-4
7.44
7.43
7.36
7.34
7.20
7.18
7.16
7.14
6.95
6.93
6.61
6.59
6.44
4.70
4.21
4.21
4.20
4.19
4.17
4.15
4.13
4.11
4.10
4.10
4.09
3.95
2.21
1.05
1.03
1.01
f1 (ppm)
xjw-20120308-4
164.35
150.94
139.30
132.33
131.14
129.27
128.37
125.11
121.42
121.16
117.16
116.87
116.35
63.09
51.96
20.79
13.63
f1 (ppm)

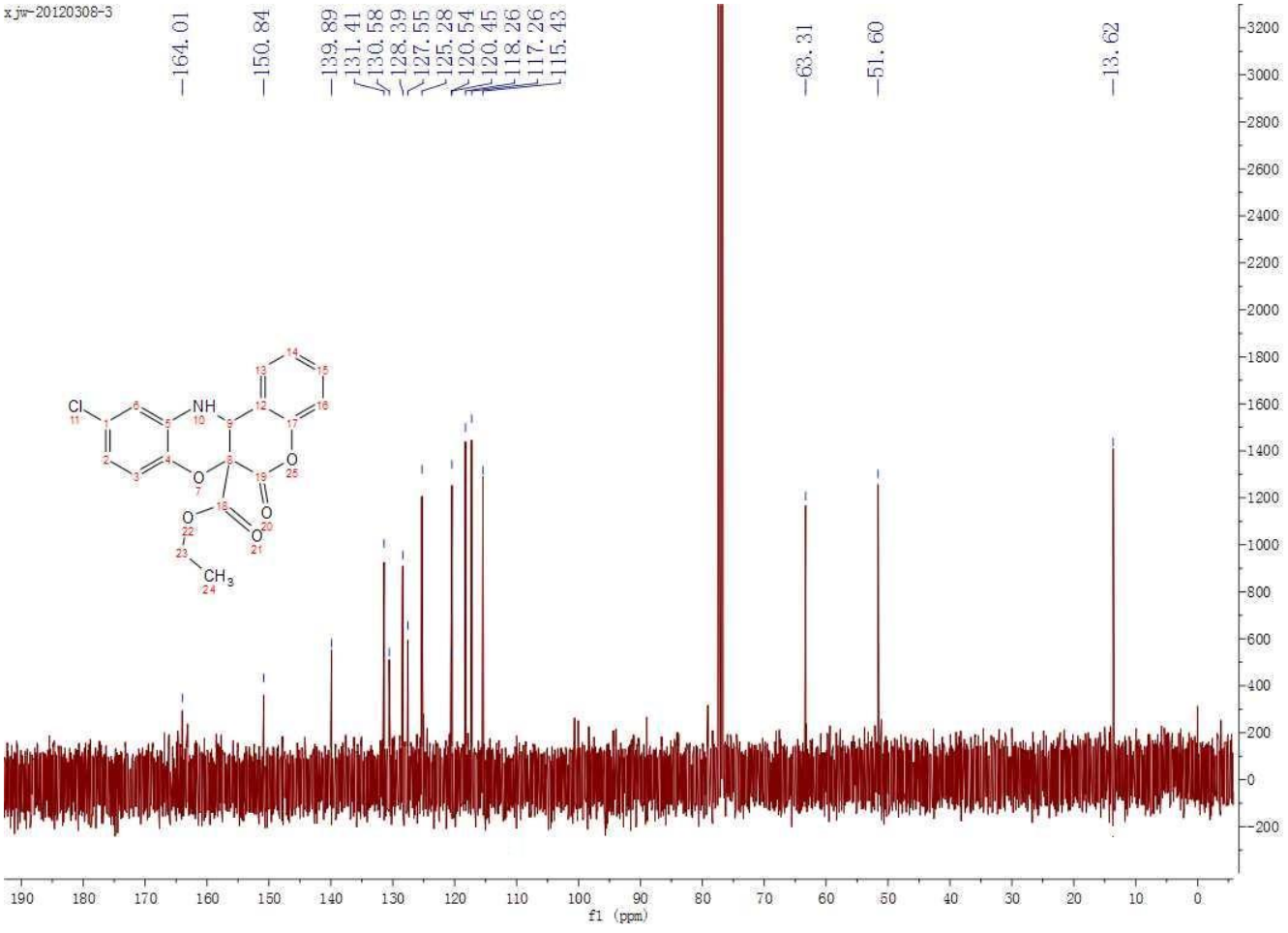
4ta



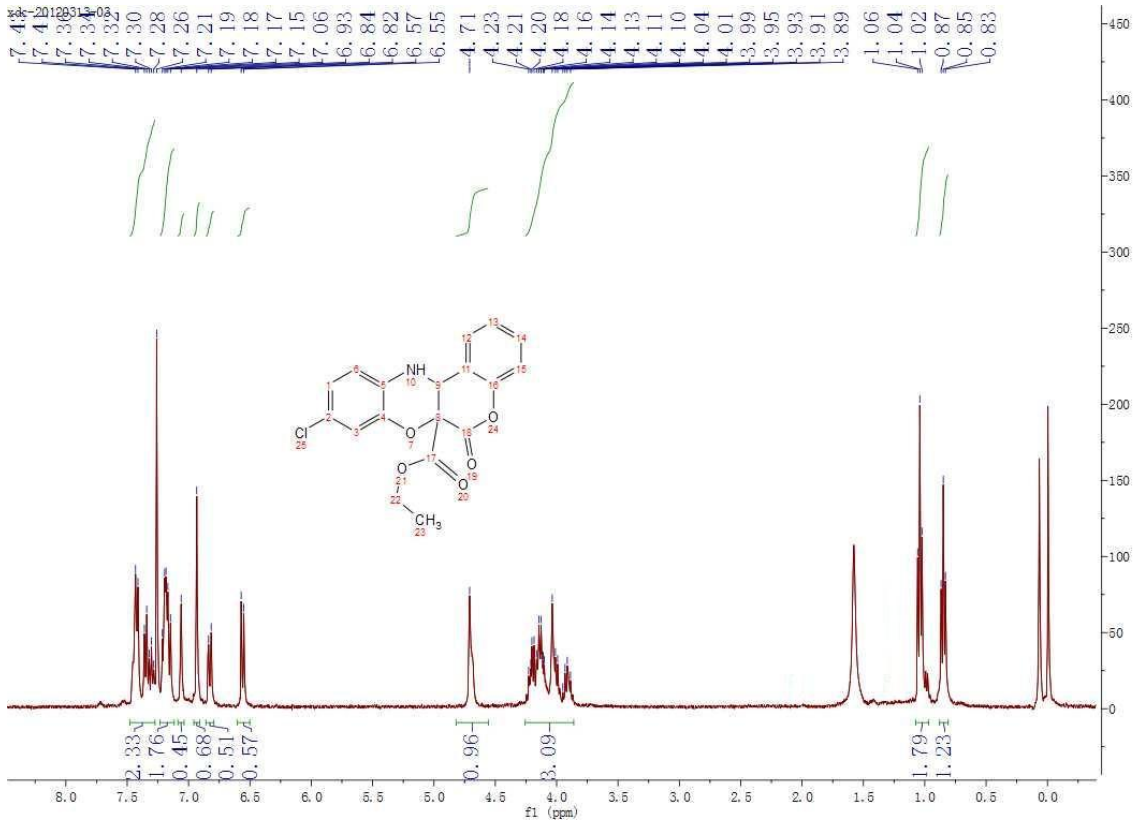


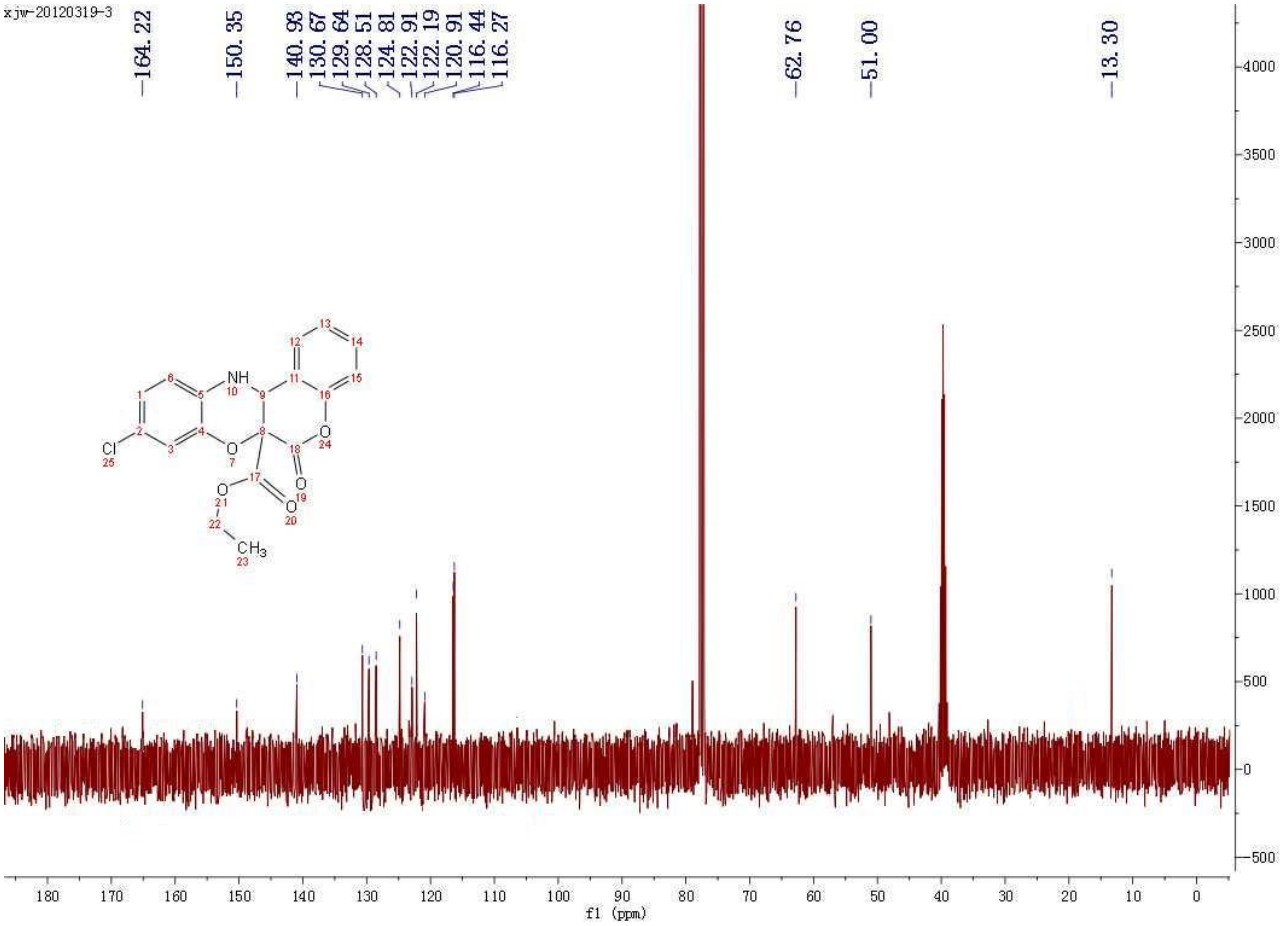
4ua


7.45
7.41
7.36
7.34
7.22
7.20
7.18
7.17
7.15
6.98
6.96
6.76
6.74
6.62
4.72
4.26
4.24
4.22
4.21
4.20
4.18
4.16
4.14
4.12
4.12
4.10
4.08
1.05
1.04
1.02
f1 (ppm)

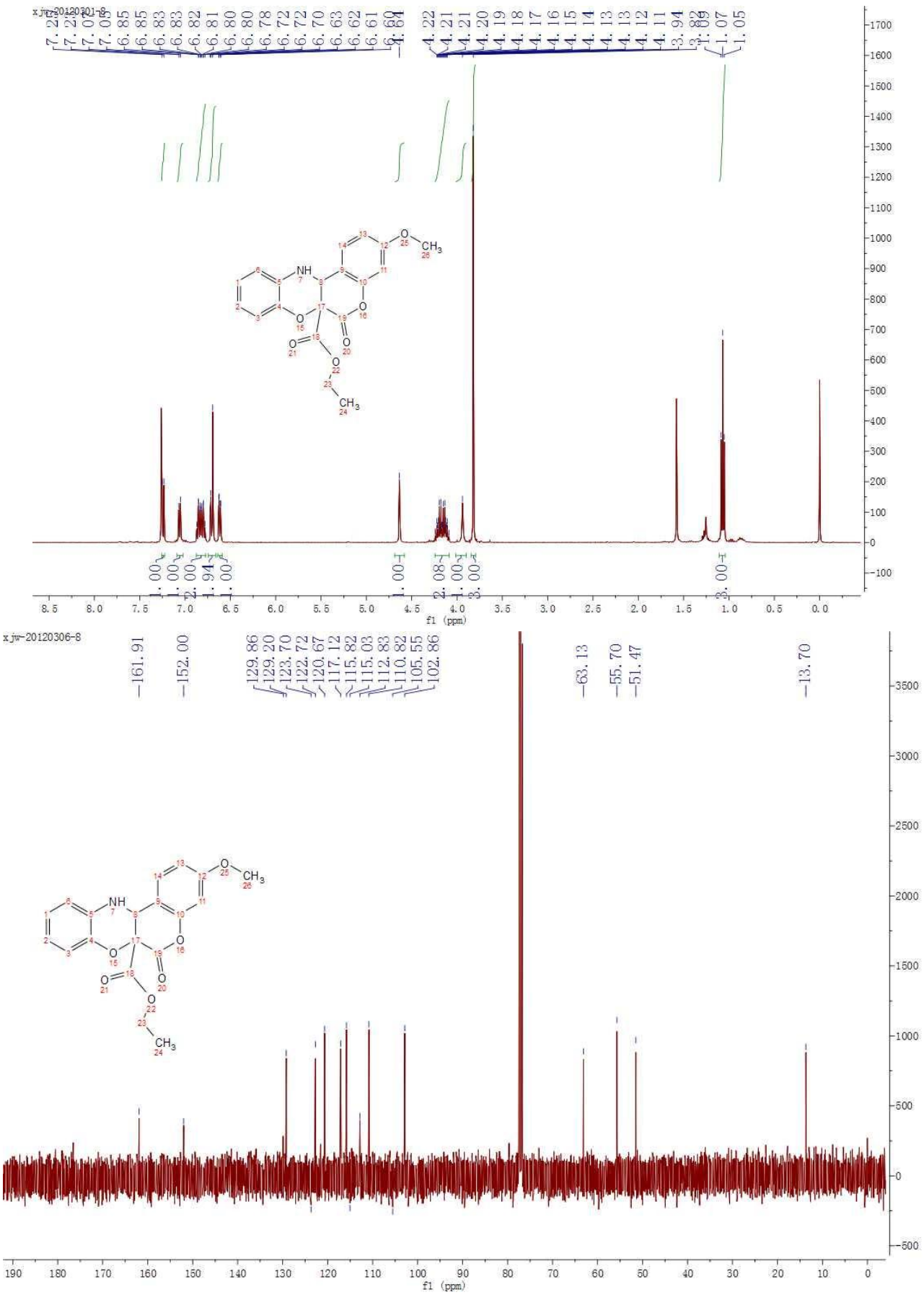

xjw-20120308-3
164.01
150.84
139.89
131.41
130.58
128.39
127.55
125.28
120.54
120.45
118.26
117.26
115.43
63.31
51.60
13.62
f1 (ppm)

4va

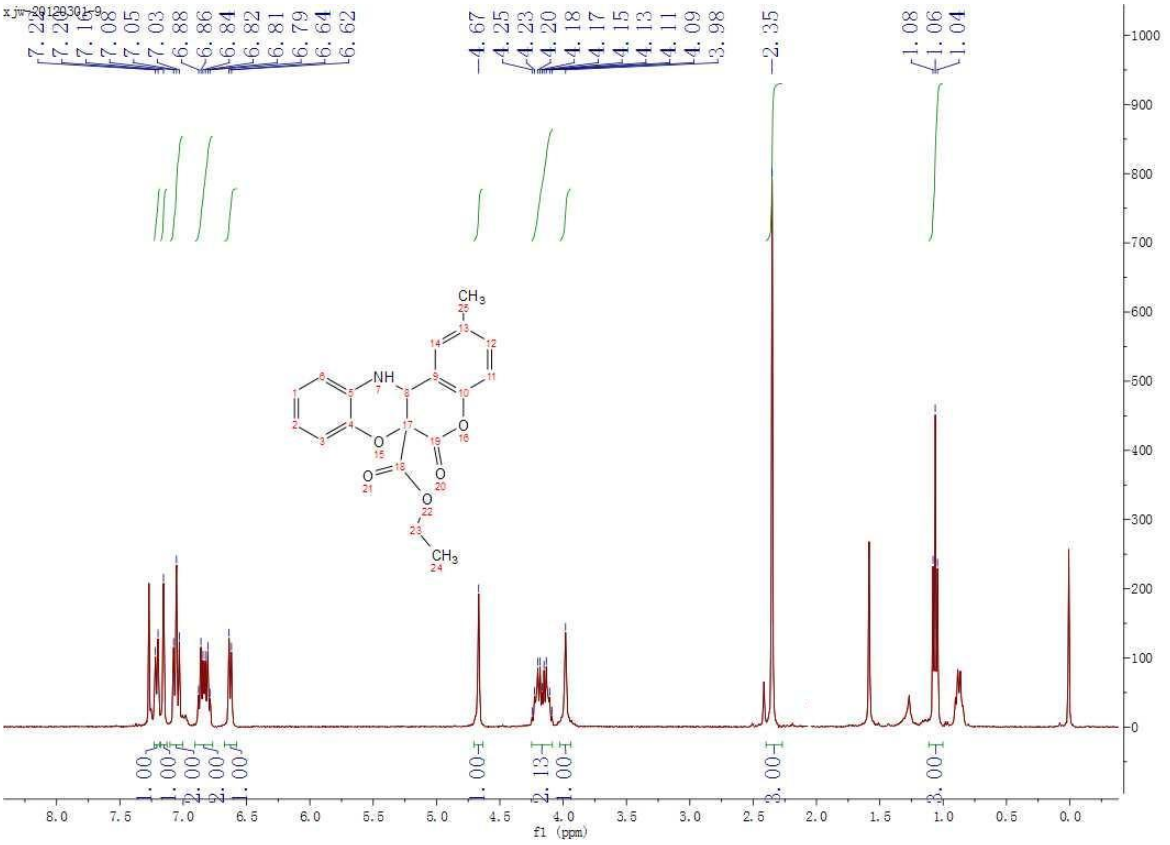

xjw-20120319-03
7.44
7.41
7.38
7.33
7.30
7.28
7.26
7.21
7.19
7.18
7.17
7.15
7.06
6.93
6.84
6.82
6.57
6.55
4.71
4.23
4.21
4.20
4.18
4.16
4.14
4.13
4.11
4.10
4.04
4.01
3.99
3.95
3.93
3.91
3.89
1.06
1.04
1.02
0.87
0.85
0.83
2.33
1.76
0.45
0.68
0.51
0.57
0.96
3.09
1.79
1.23
f1 (ppm)

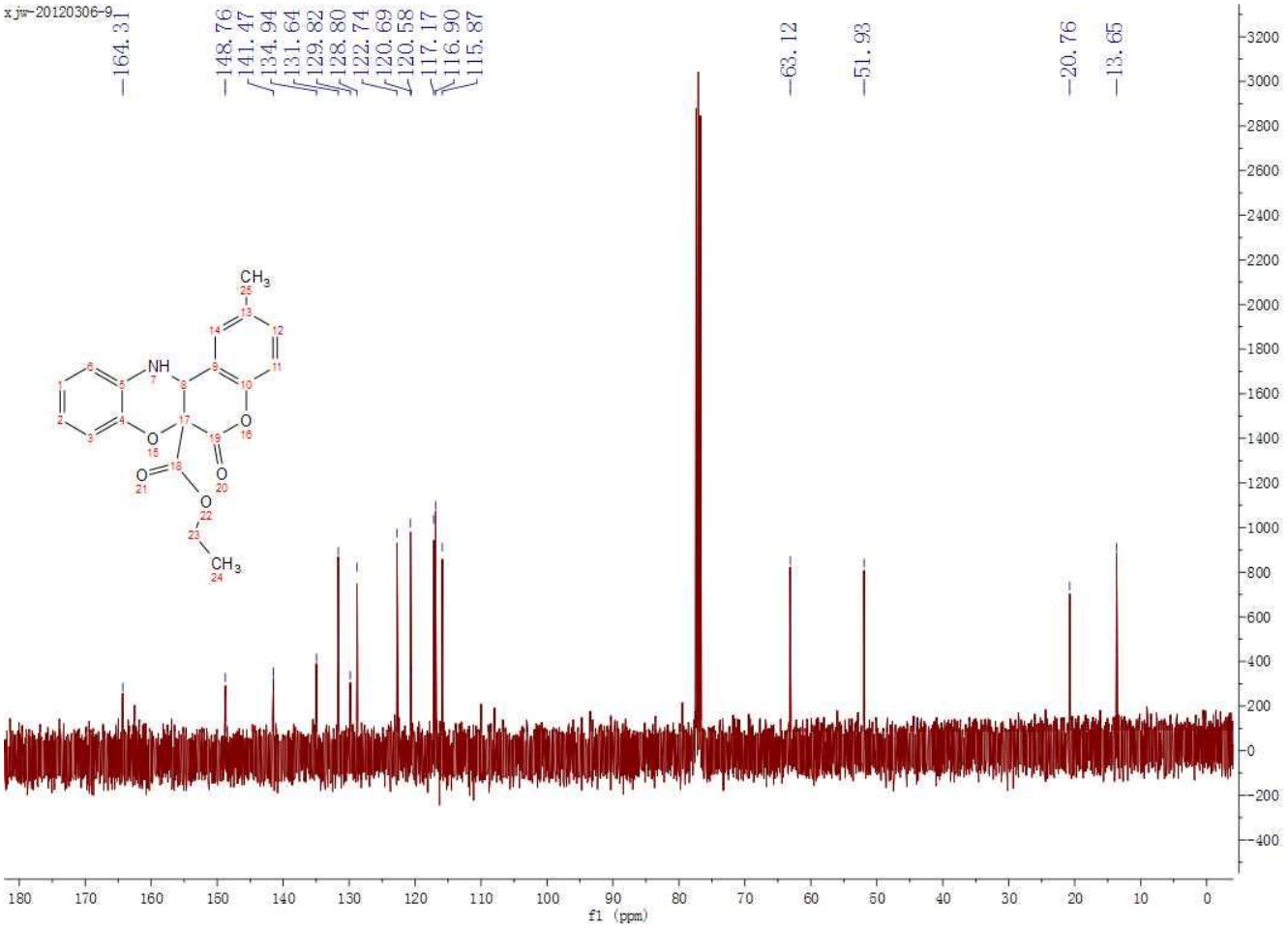

xjw-20120319-3
164.22
150.35
140.93
130.67
129.64
128.51
124.81
122.91
122.19
120.91
116.44
116.27
62.76
51.00
13.30
f1 (ppm)

4wa

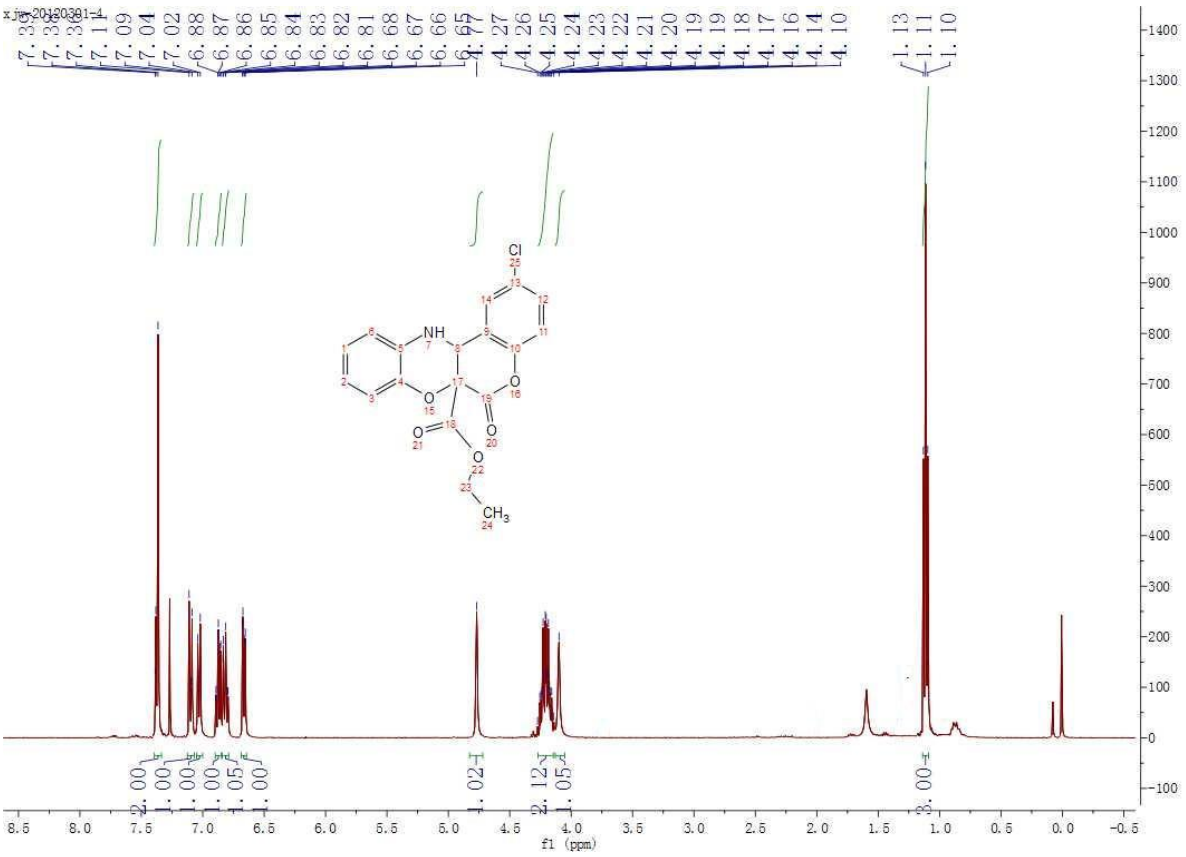

7.22
7.21
7.05
6.85
6.85
6.83
6.83
6.82
6.81
6.80
6.80
6.78
6.72
6.72
6.70
6.63
6.62
6.61
6.60
4.64
4.22
4.21
4.21
4.20
4.19
4.18
4.17
4.16
4.15
4.14
4.13
4.13
4.12
4.11
3.94
3.82
3.09
1.07
1.05
1.00
1.00
2.00
1.94
1.00
1.00
2.08
1.00
3.00
3.00
f1 (ppm)
xjw-20120306-8
161.91
152.00
129.86
129.20
123.70
122.72
120.67
117.12
115.82
115.03
112.83
110.82
105.55
102.86
63.13
55.70
51.47
13.70
f1 (ppm)

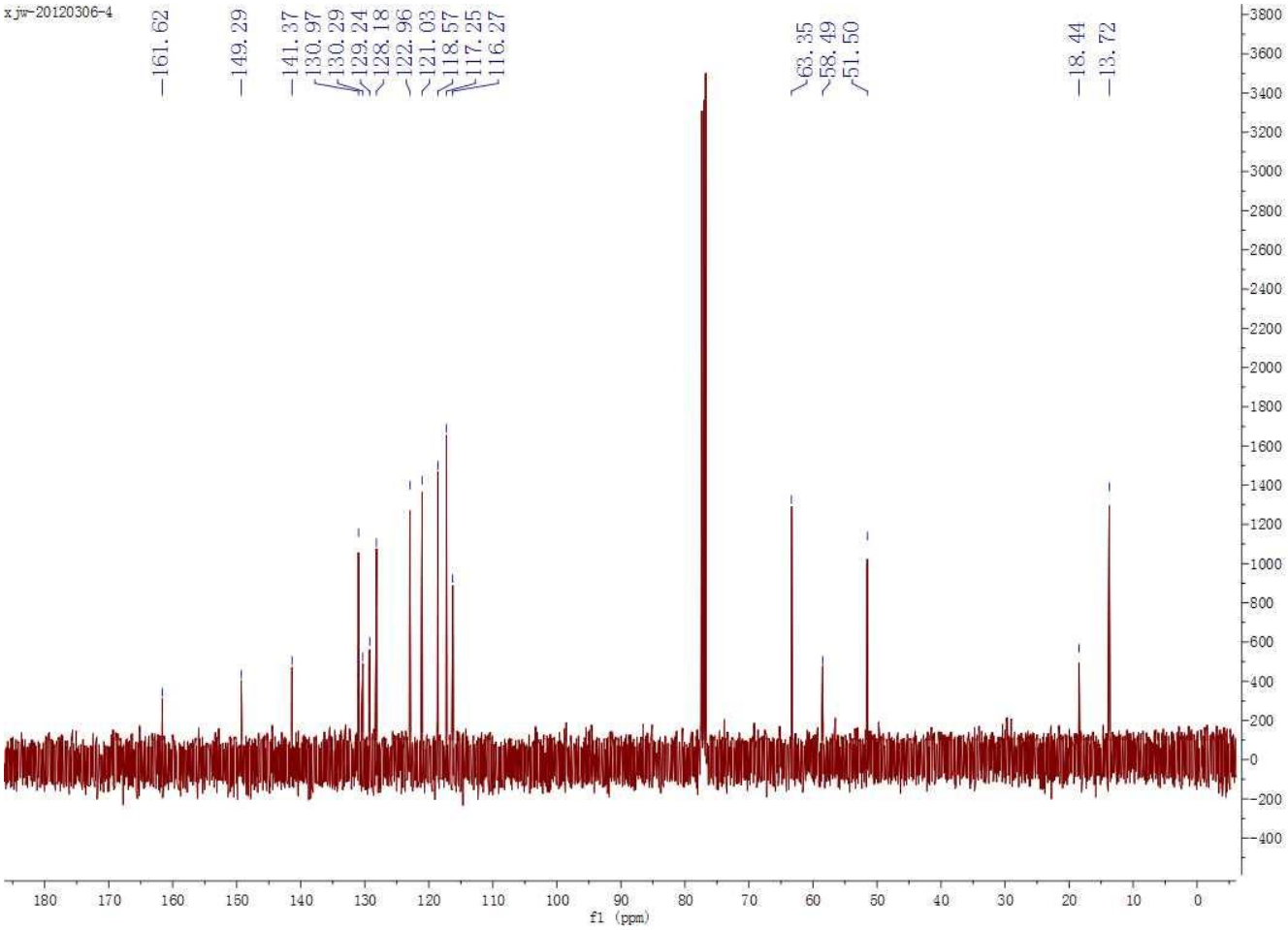
4xa


7.24
7.23
7.18
7.08
7.05
7.03
6.88
6.86
6.84
6.82
6.81
6.79
6.64
6.62
4.67
4.25
4.23
4.20
4.18
4.17
4.15
4.13
4.11
4.09
3.98
2.35
1.08
1.06
1.04
f1 (ppm)


xjw-20120306-9
164.31
148.76
141.47
134.94
131.64
129.82
128.80
122.74
120.69
120.58
117.17
116.90
115.87
63.12
51.93
20.76
13.65
f1 (ppm)

4ya


7.36
7.11
7.09
7.04
7.02
6.88
6.87
6.86
6.85
6.84
6.83
6.82
6.81
6.68
6.67
6.66
6.65
4.27
4.26
4.25
4.24
4.23
4.22
4.21
4.20
4.19
4.18
4.17
4.16
4.14
4.10
1.13
1.11
1.10
Cl
NH
O
O
O
O
O
CH3
2.00
1.00
1.00
1.05
1.00
1.02
2.12
2.05
3.00
f1 (ppm)


xjw-20120306-4
161.62
149.29
141.37
130.97
130.29
129.24
128.18
122.96
121.03
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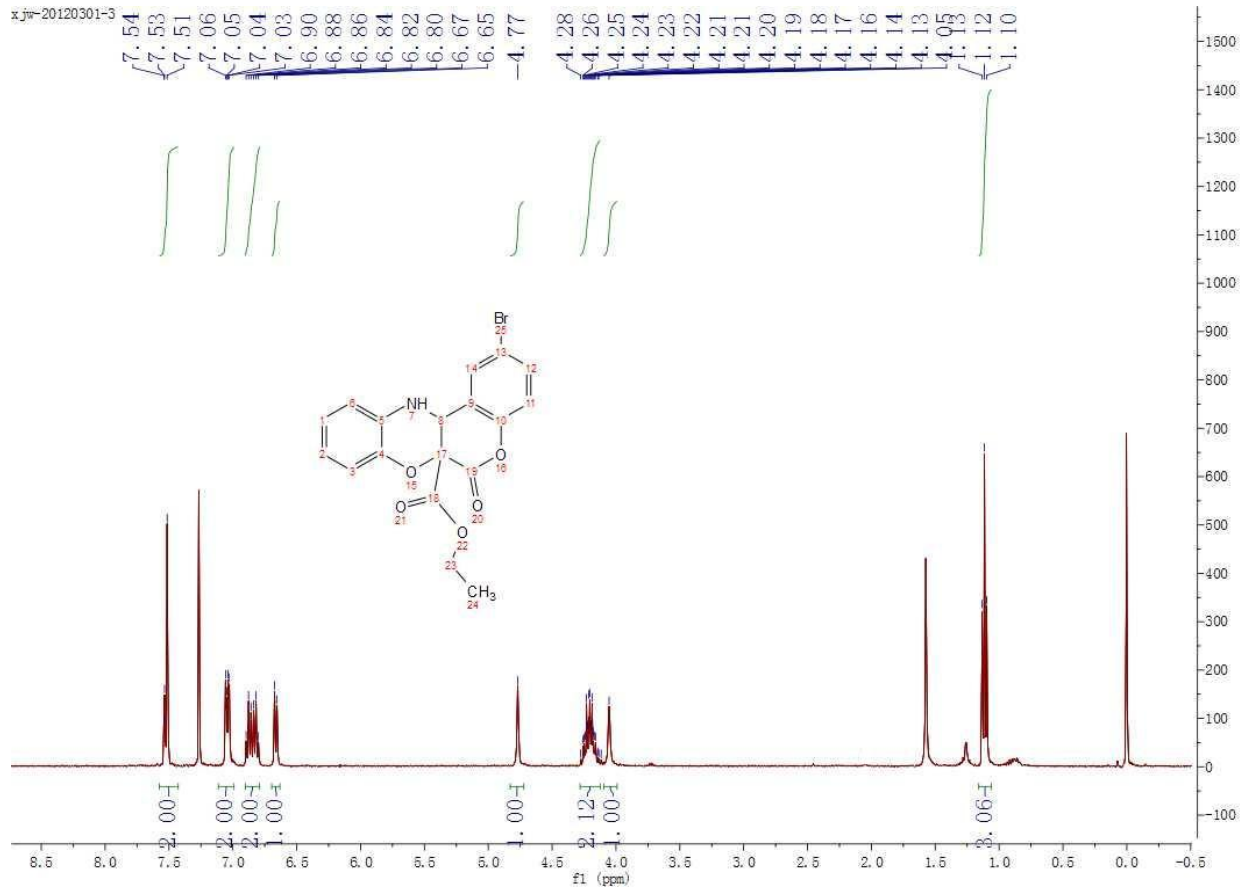
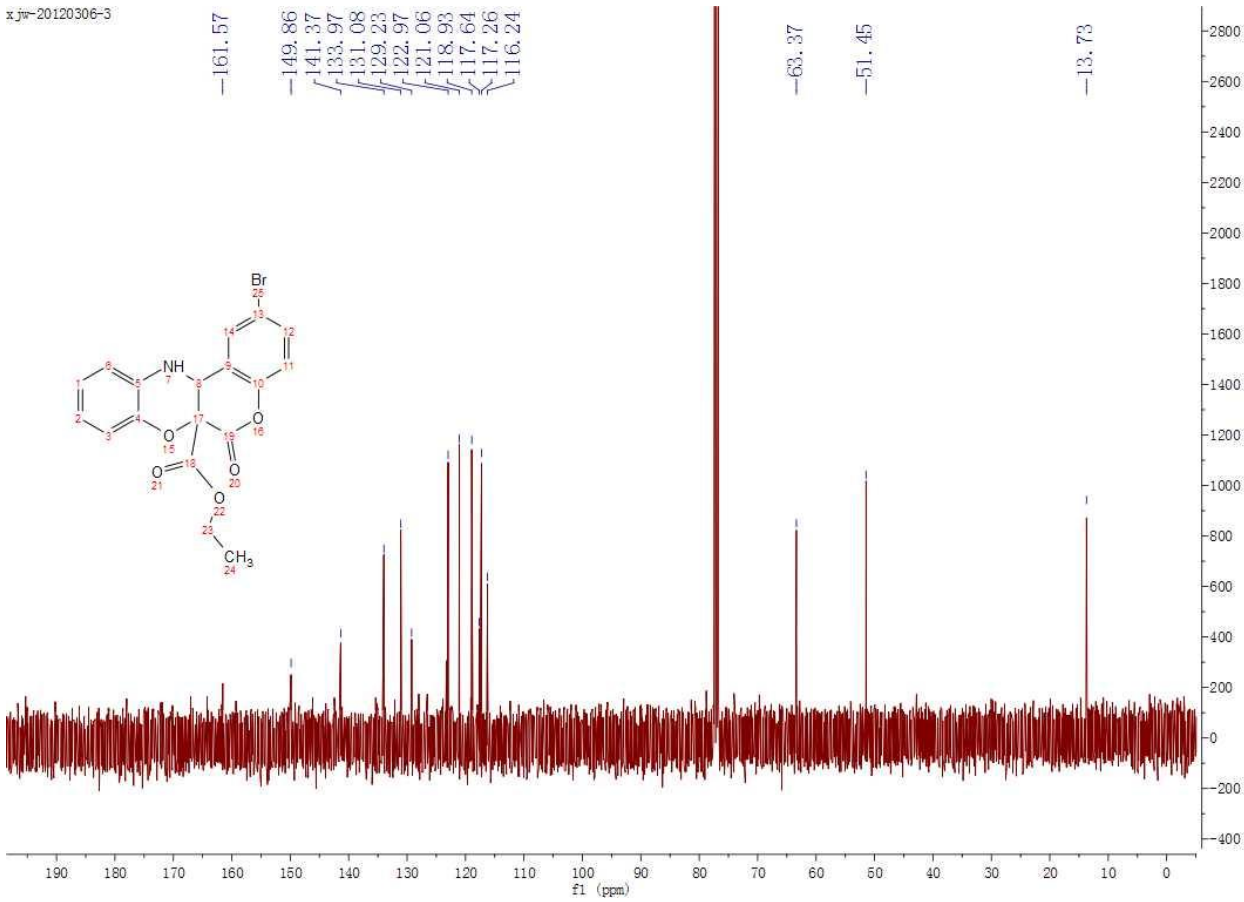
117.25
116.27
63.35
58.49
51.50
18.44
13.72
f1 (ppm)

4za


xjw-20120301-3
7.54
7.53
7.51
7.06
7.05
7.04
7.03
6.90
6.88
6.86
6.84
6.82
6.80
6.67
6.65
4.77
4.28
4.26
4.25
4.24
4.23
4.22
4.21
4.21
4.20
4.19
4.18
4.17
4.16
4.14
4.13
4.05
1.13
1.12
1.10
Br
NH
O
O
O
O
O
CH3
2.00
2.00
2.00
1.00
1.00
2.12
1.00
3.06
f1 (ppm)


xjw-20120306-3
161.57
149.86
141.37
133.97
131.08
129.23
122.97
121.06
118.93
117.64
117.26
116.24
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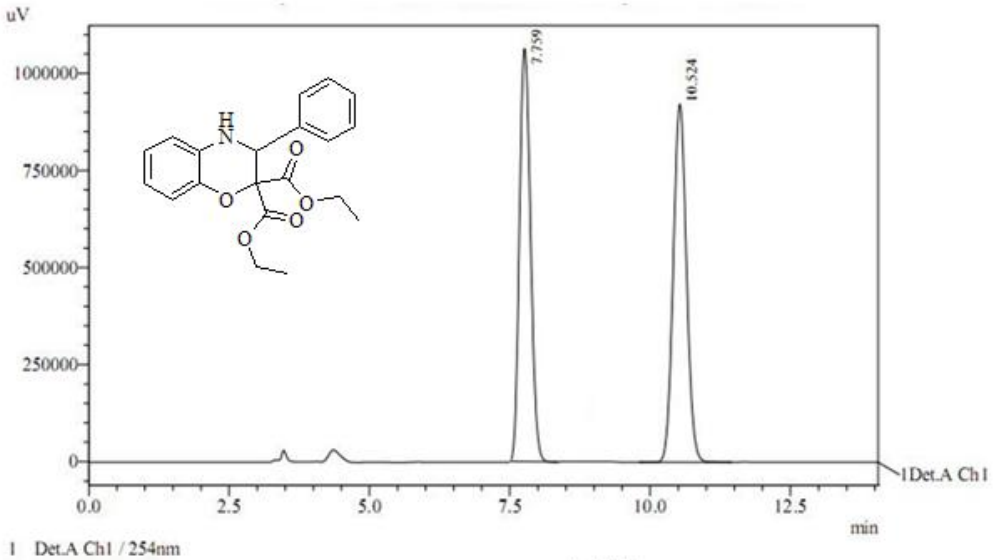
13.73
Br
NH
O
O
O
O
O
CH3
f1 (ppm)

5. HPLC spectra

3aa

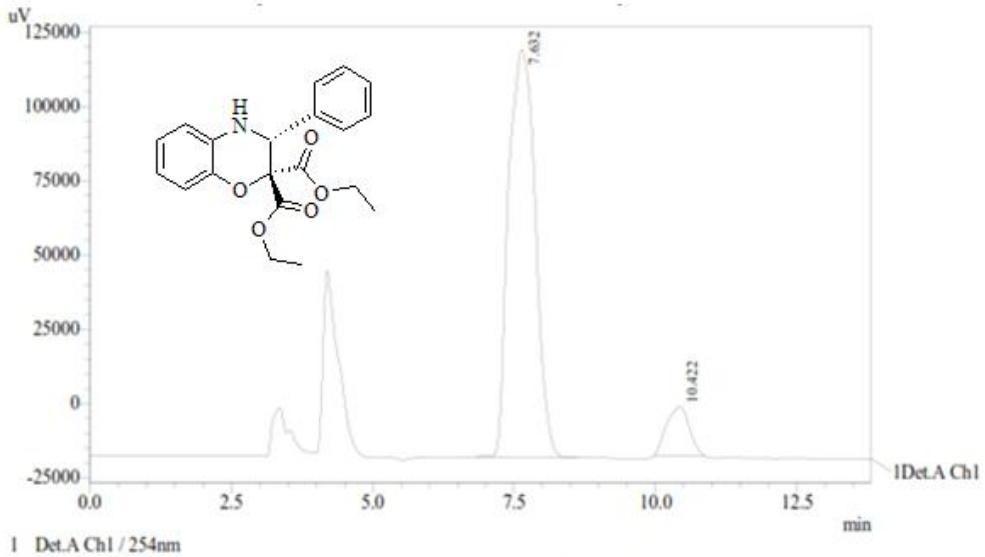


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.759	14606409	1063833	49.111	53.552
2	10.524	15135201	922722	50.889	46.448
Total		29741610	1986555	100.000	100.000



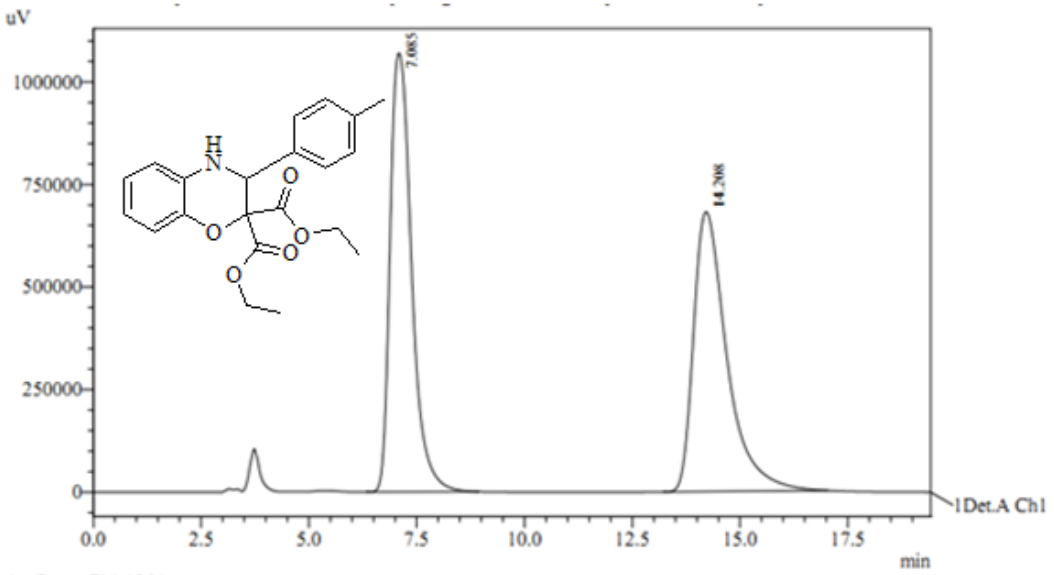
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.632	4501336	137256	90.813	89.324
2	10.422	455351	16405	9.187	10.676
Total		4956687	153661	100.000	100.000

3ba

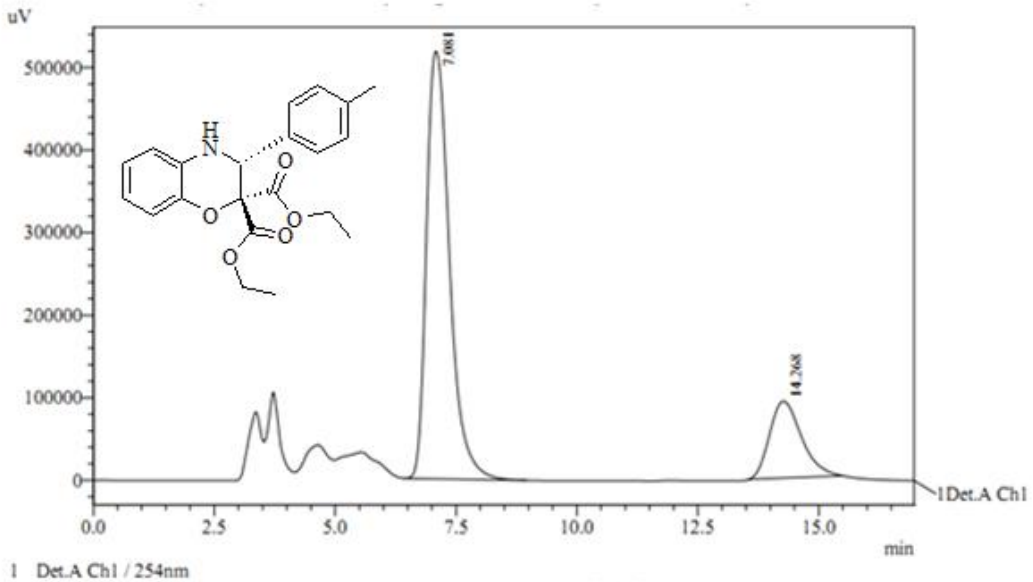


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.085	37108675	1069913	50.180	61.063
2	14.208	36842992	682221	49.820	38.937
Total		73951667	1752134	100.000	100.000



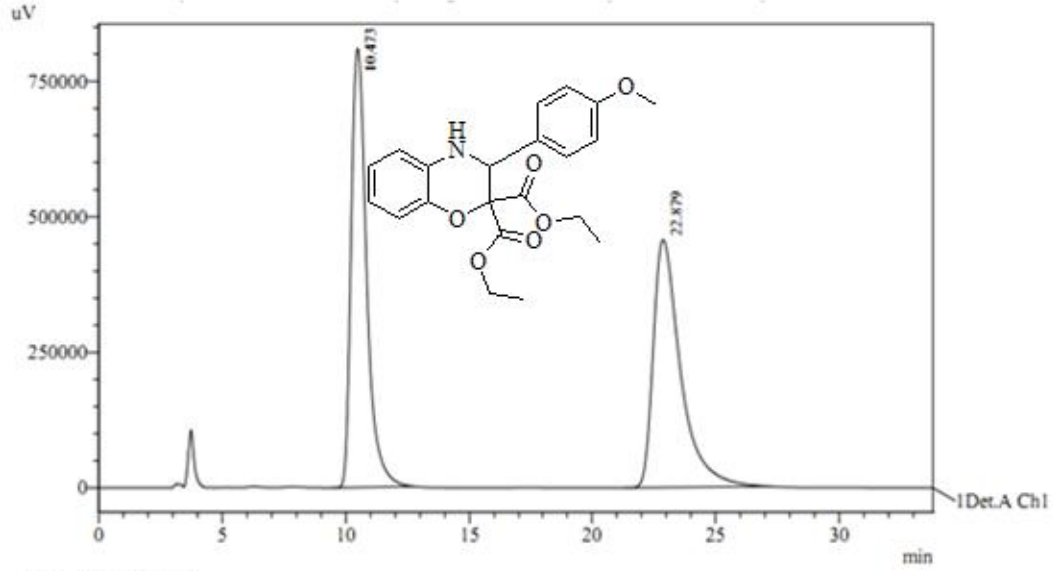
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.081	17151284	518194	80.011	84.782
2	14.268	4284752	93015	19.989	15.218
Total		21436036	611209	100.000	100.000

3ca

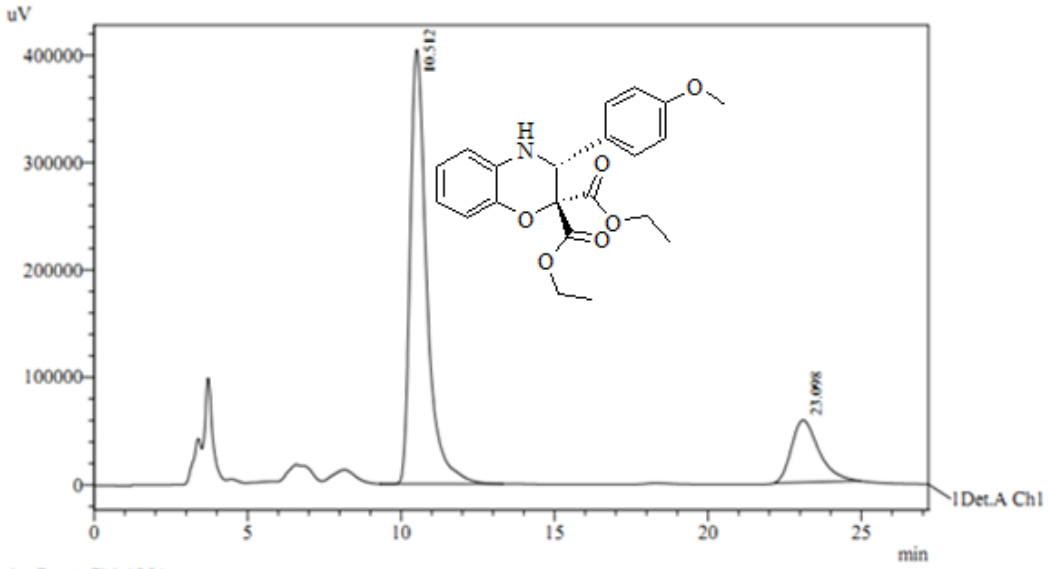


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.473	35256612	809726	50.047	63.917
2	22.879	35190618	457121	49.953	36.083
Total		70447230	1266847	100.000	100.000



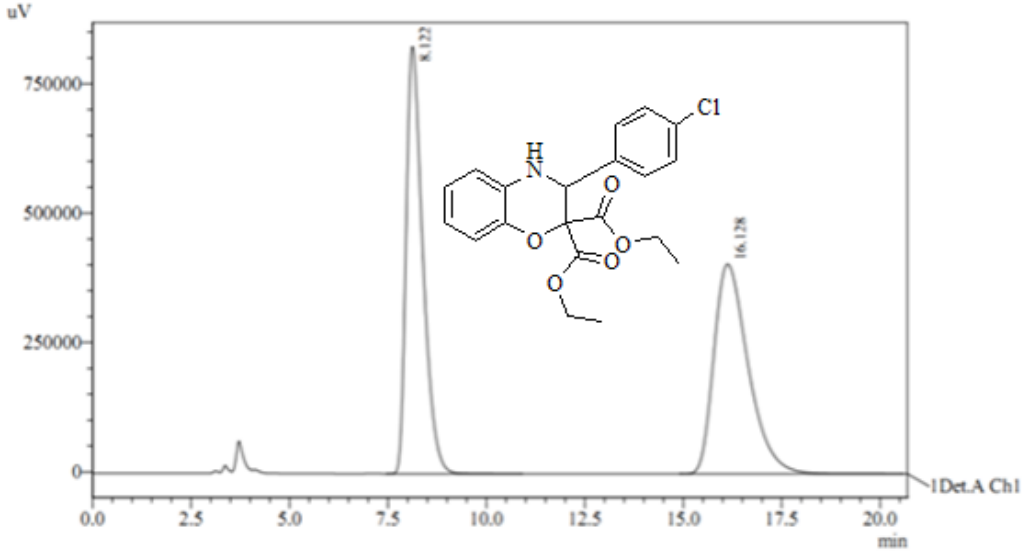
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.512	15240483	404683	80.871	87.479
2	23.098	3604904	57925	19.129	12.521
Total		18845387	462608	100.000	100.000

3fa

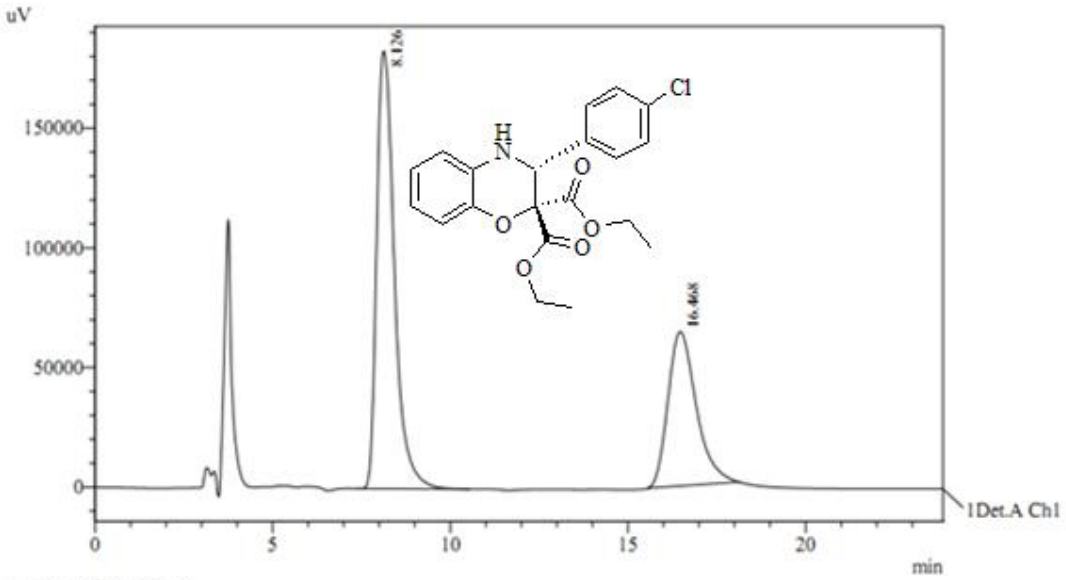


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.122	24298986	825769	49.755	67.073
2	16.128	24538211	405372	50.245	32.927
Total		48837197	1231141	100.000	100.000



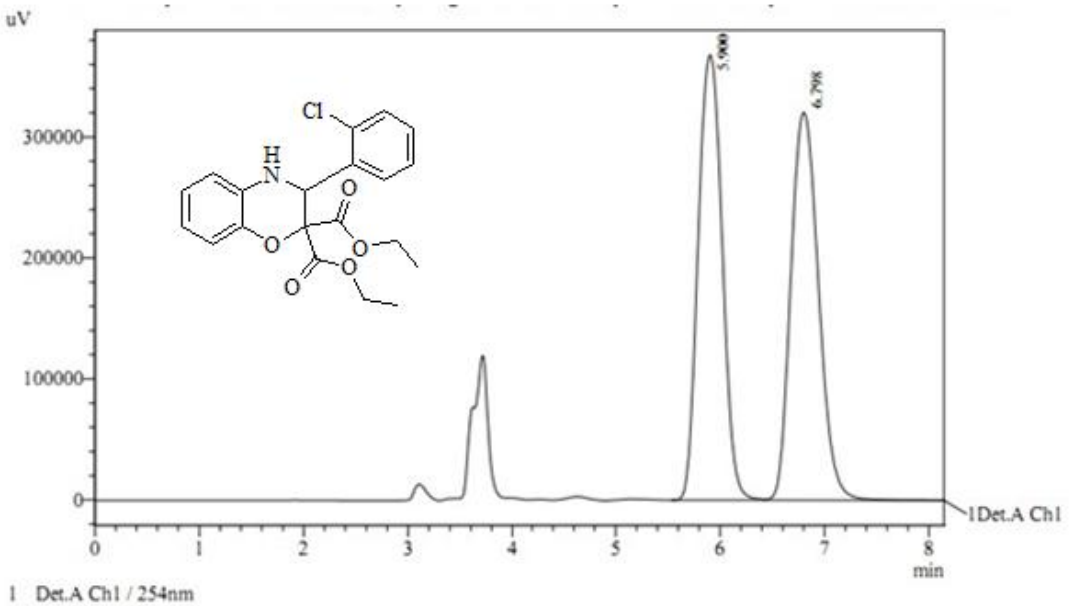
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.126	6347292	182807	64.010	73.939
2	16.468	3568839	64432	35.990	26.061
Total		9916131	247239	100.000	100.000

3ha

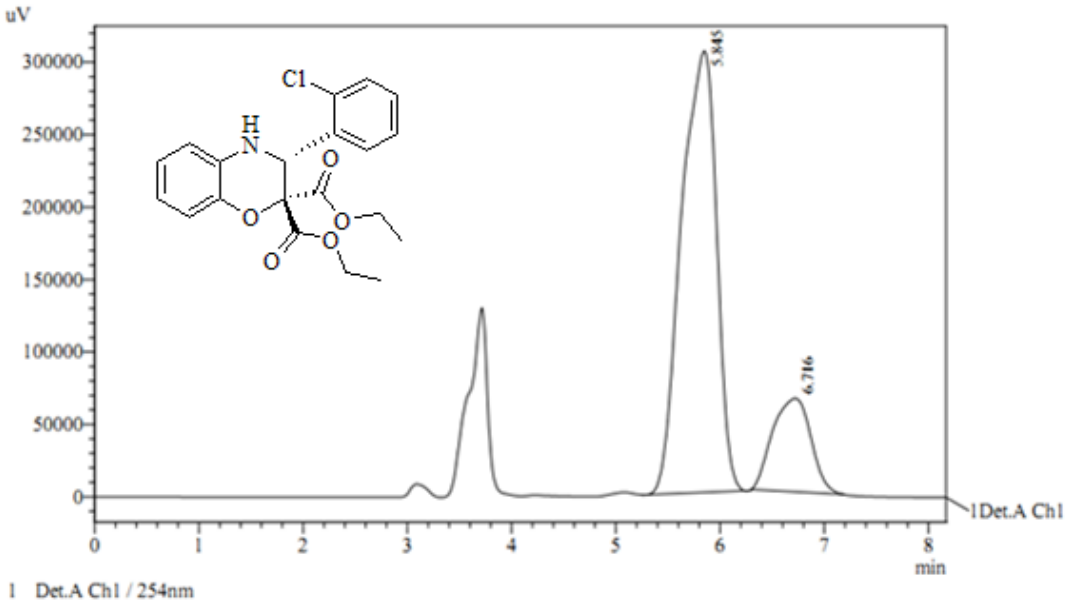


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.900	5889078	367971	49.900	53.437
2	6.798	5912672	320630	50.100	46.563
Total		11801750	688600	100.000	100.000



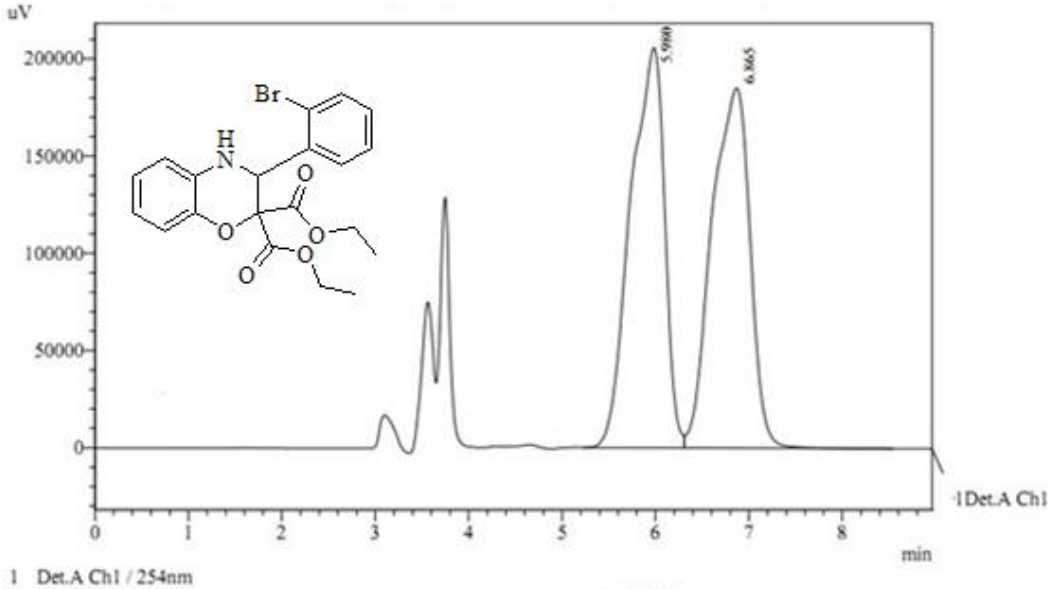
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.845	7156620	304830	81.348	82.544
2	6.716	1640963	64465	18.652	17.456
Total		8797583	369295	100.000	100.000

3ja

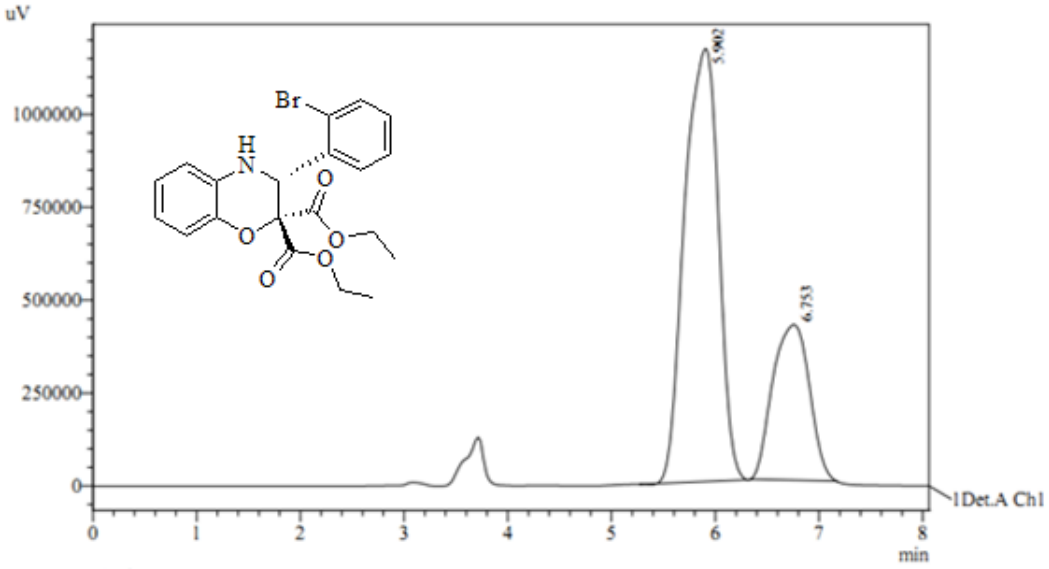


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.980	5224354	205759	49.753	52.639
2	6.865	5276245	185125	50.247	47.361
Total		10500599	390884	100.000	100.000



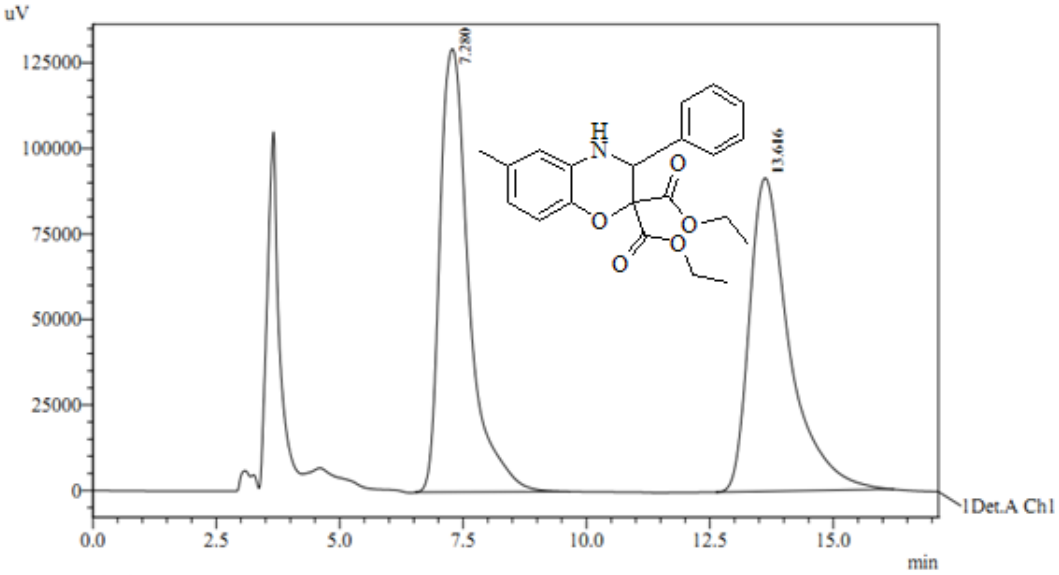
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.902	26589637	1165227	72.044	73.585
2	6.753	10317920	418279	27.956	26.415
Total		36907557	1583506	100.000	100.000

3ma

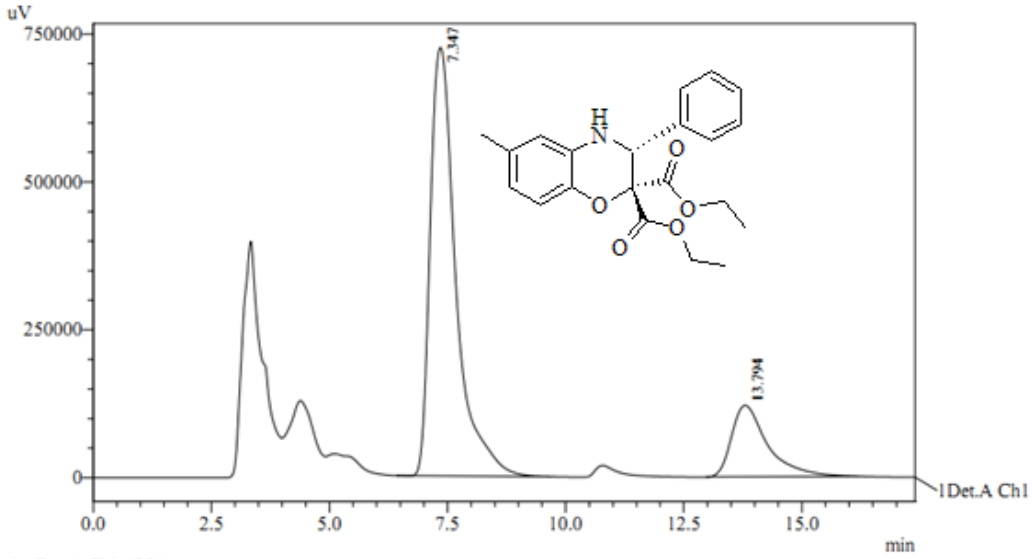


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.280	5324518	129561	50.721	58.579
2	13.616	5173051	91613	49.279	41.421
Total		10497569	221174	100.000	100.000



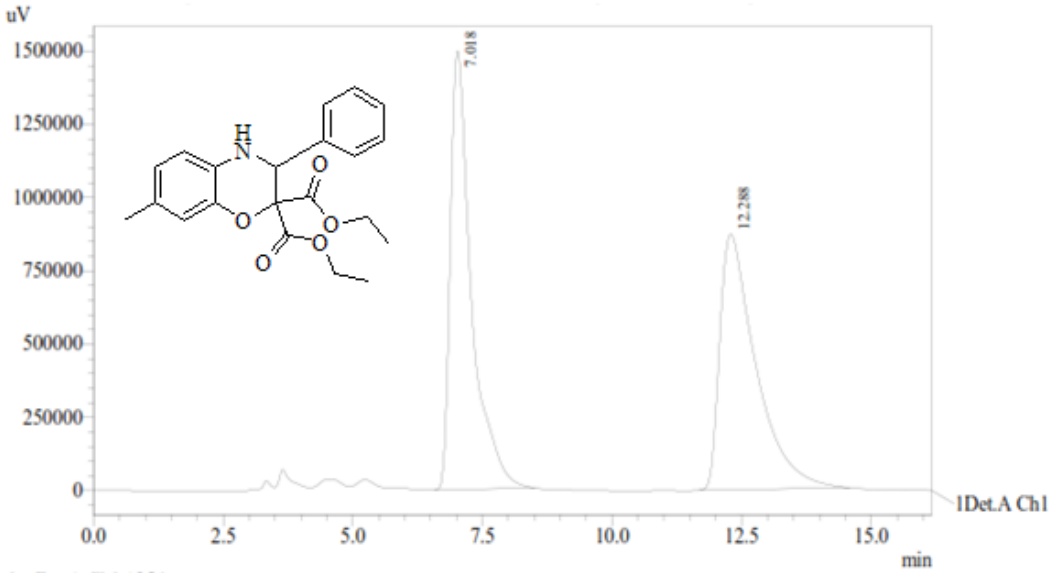
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.347	27416600	724161	81.087	85.701
2	13.794	6394636	120825	18.913	14.299
Total		33811237	844986	100.000	100.000

3na

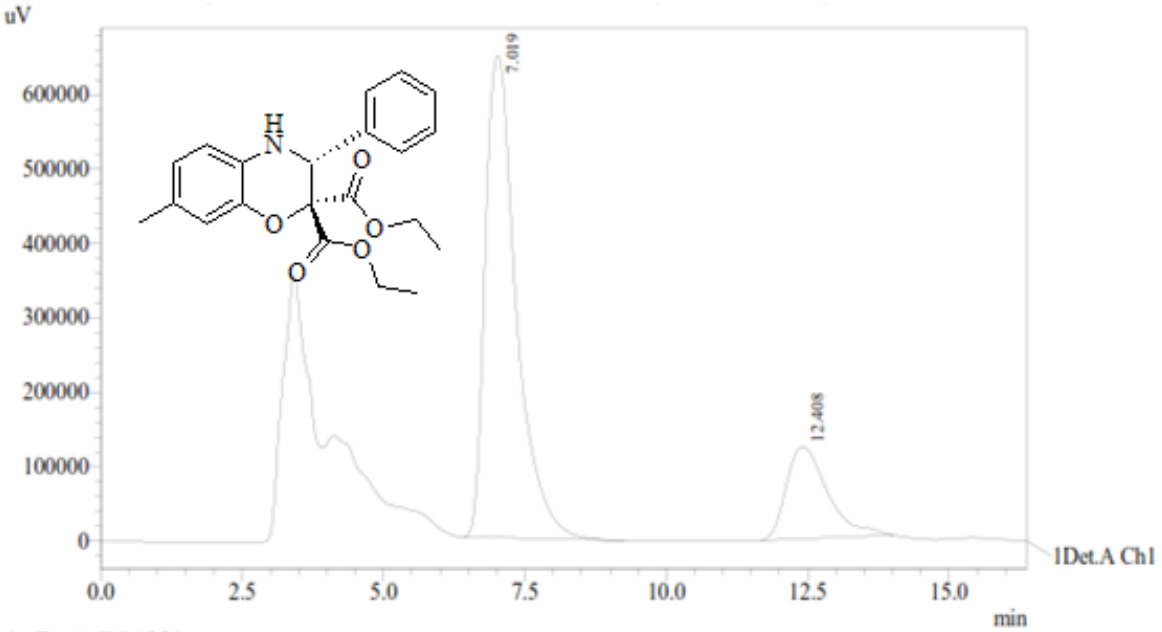


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.018	42456354	1497943	50.070	63.184
2	12.288	42336928	872822	49.930	36.816
Total		84793283	2370766	100.000	100.000



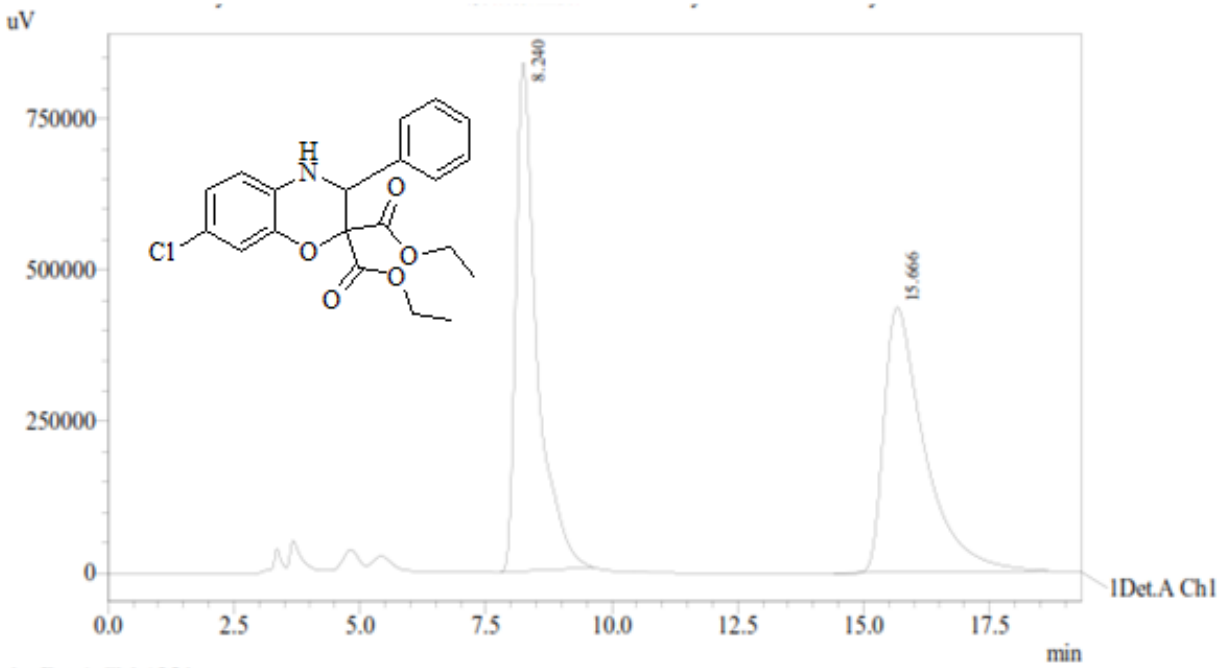
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.019	24839553	647983	79.832	83.940
2	12.408	6275261	123978	20.168	16.060
Total		31114813	771961	100.000	100.000

3pa

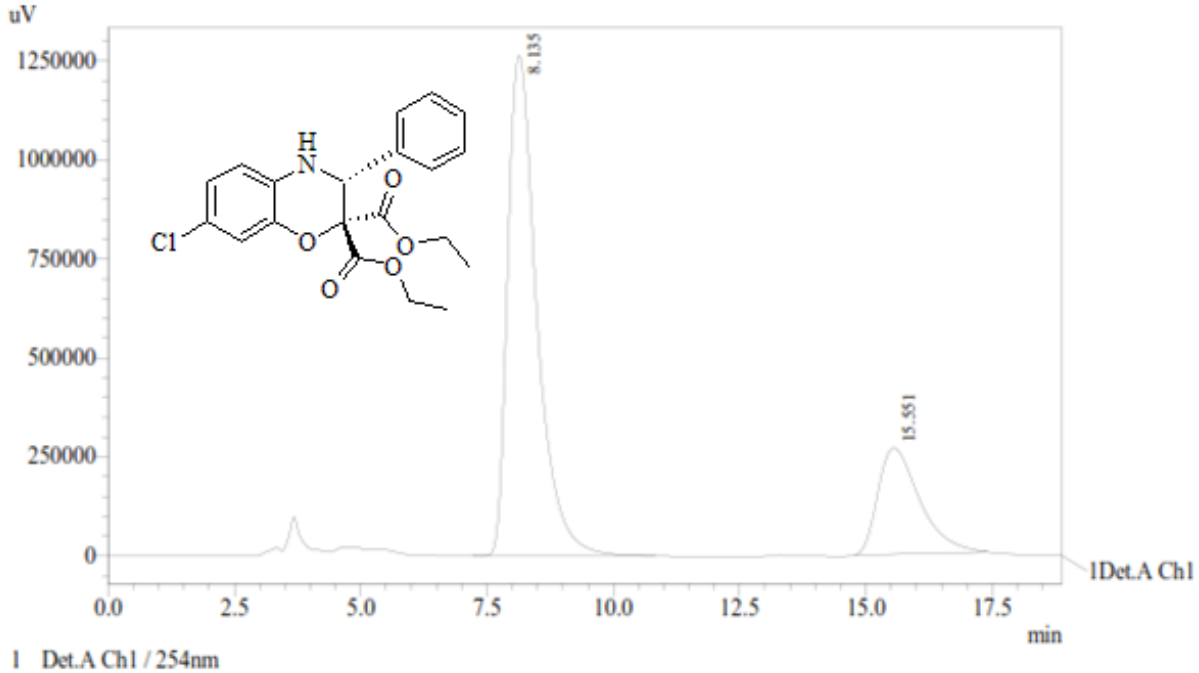


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.240	24391098	839676	49.875	65.749
2	15.666	24512872	437409	50.125	34.251
Total		48903970	1277085	100.000	100.000



1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.135	50953007	1263989	76.773	82.499
2	15.551	15415668	268140	23.227	17.501
Total		66368676	1532129	100.000	100.000