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Supplementary Material

Facile Domino Access to Unexpected Alkyl 3-Substituted-2-Hydroxy-3,4-Dihydro-2H-1,4-Benzoxazine-2-Carboxylates

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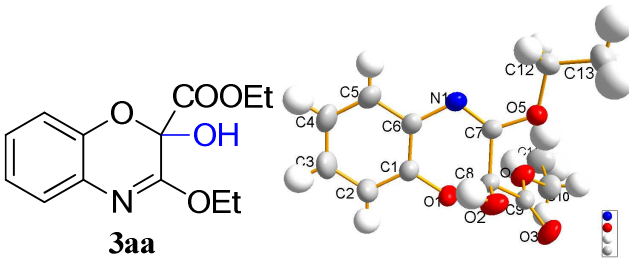
1. General Methods:

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) for ^{1}H NMR and relative to the central $CDCl_3$ resonance (δ = 77.0 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.

2. X-ray data of 3aa



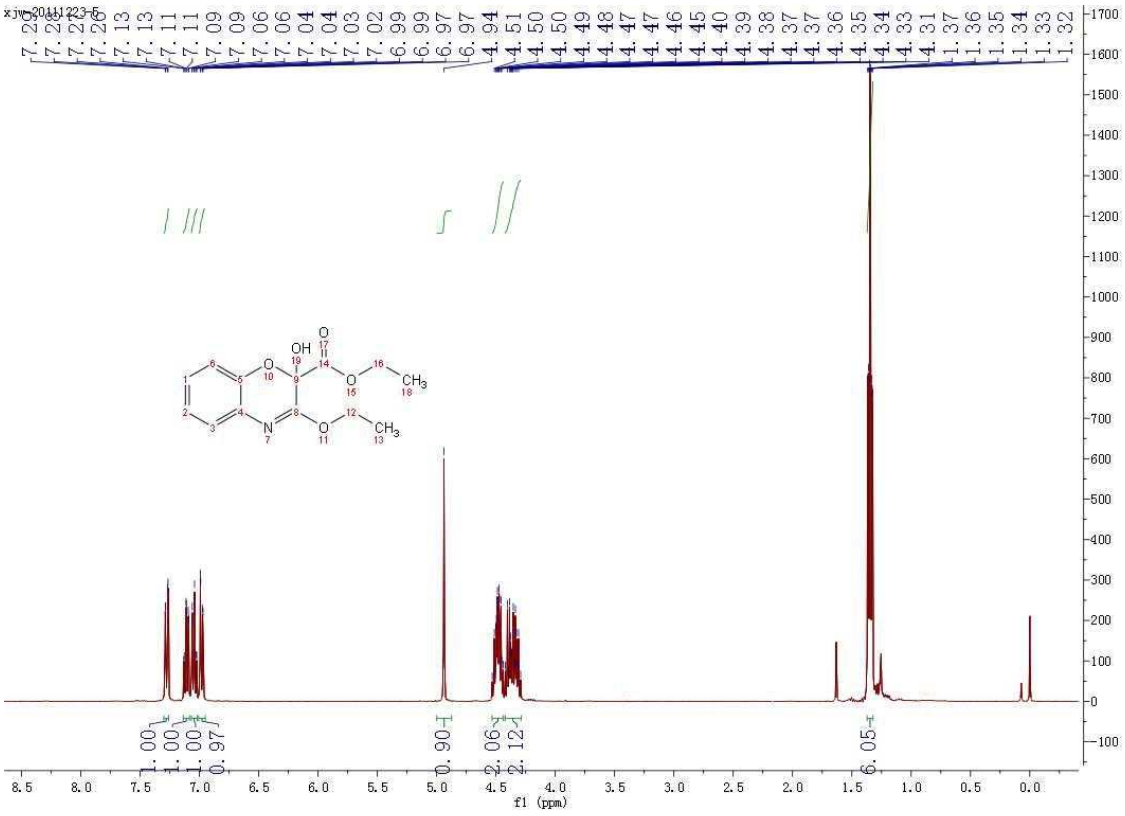
3aa

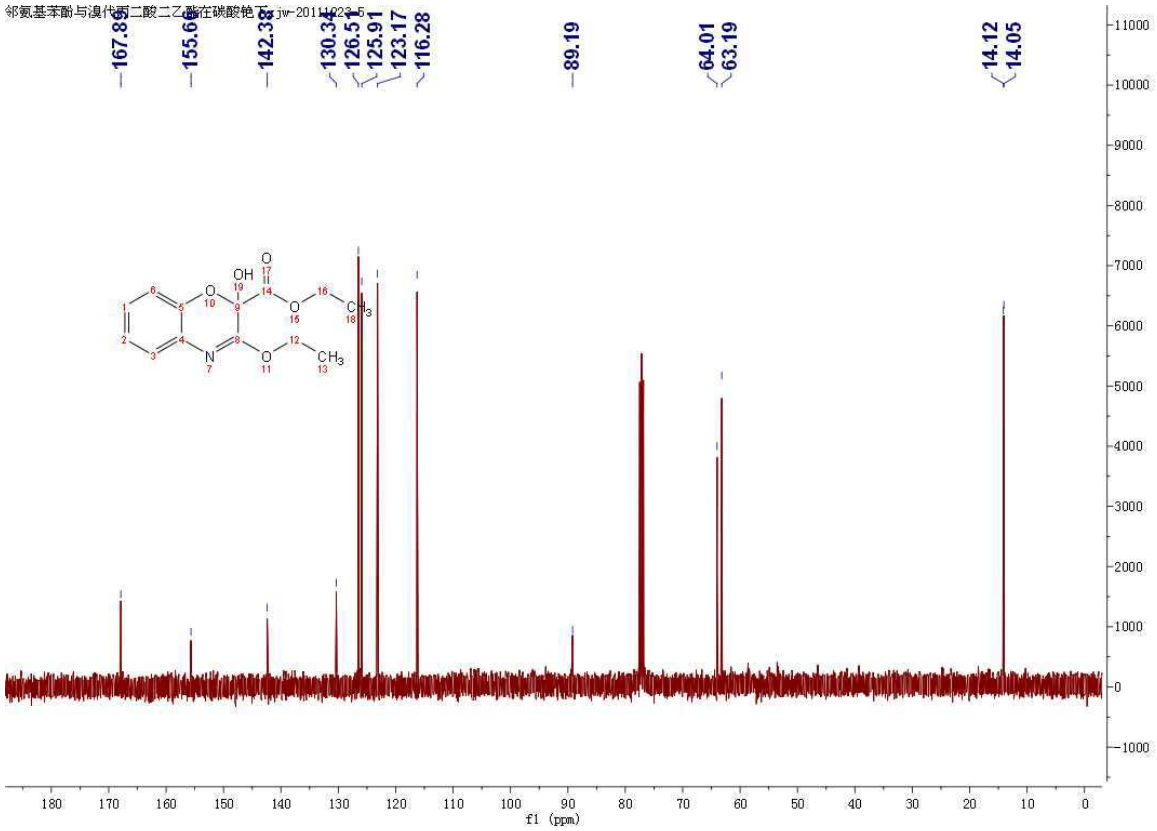
Molecular Structure of 3aa (ellipsoids with 50% probability)

Crystal data for 3aa $C_{13}H_{15}NO_5$ (265.26), Triclinic, space group P-1, a = 5.8989(4) Å, b = 9.2987(6) Å, c = 13.0803(9) Å, V = 695.86(8) $Å^3$, Z = 2, specimen 0.251 x 0.0175 x 0.121 mm^3, T = 296(2) K, SIEMENS P4 diffractometer, absorption coefficient 0.098 mm^{-1}, reflections collected 11416, independent 3210 [R(int) = 0.0222], refinement by Full-matrix least-squares on F^2, data/restraints/parameters 3210 / 0 / 173, goodness-of-fit on F^2 = 1.038, final R indices [I>2σ(I)] R1 = 0.0459, wR2 = 0.1413, R indices (all data) R1 = 0.0545, wR2 = 0.1497, largest diff. peak and hole 0.602 and - 0.509 $Å^{-3}$.

3. ^{1}H NMR and ^{13}C NMR spectra

3aa

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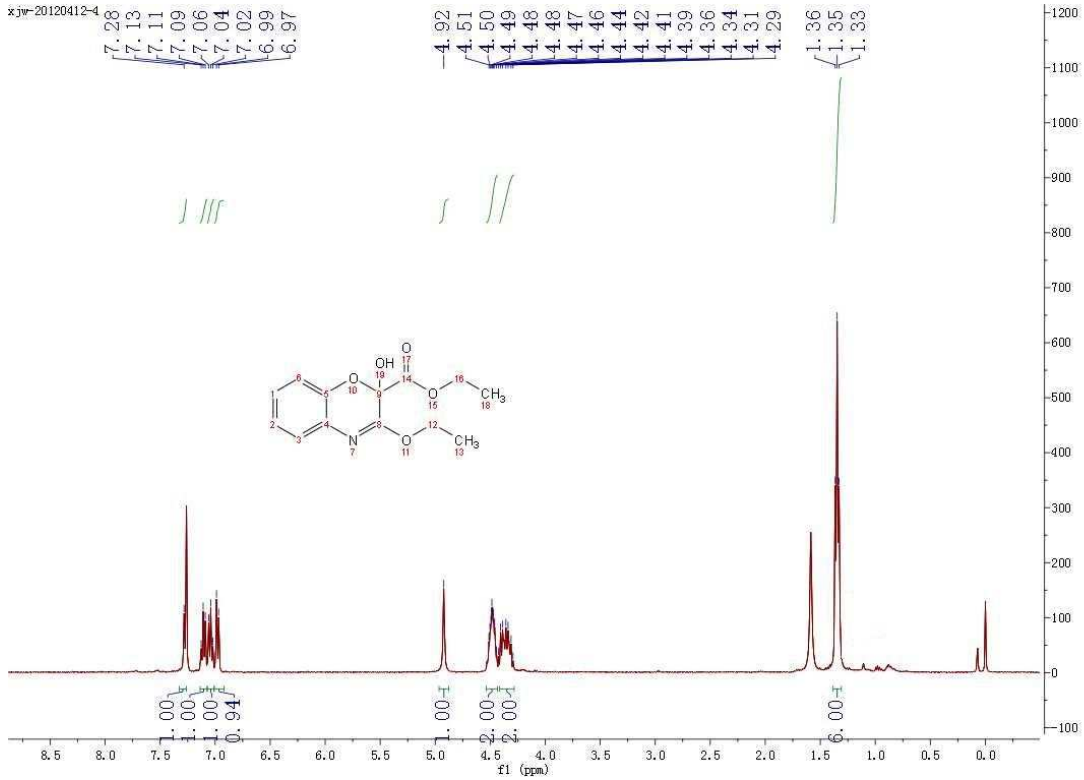
63.19
14.12
14.05
f1 (ppm)

3ab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4.50
4.49
4.48
4.47
4.46
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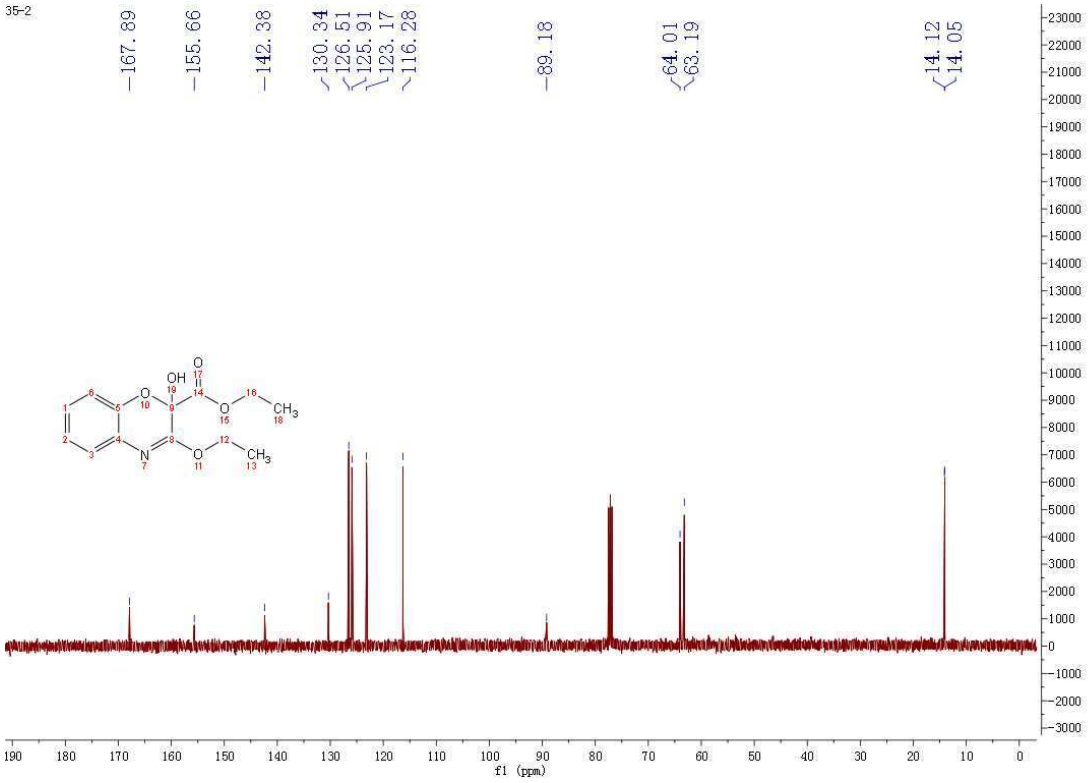
f1 (ppm)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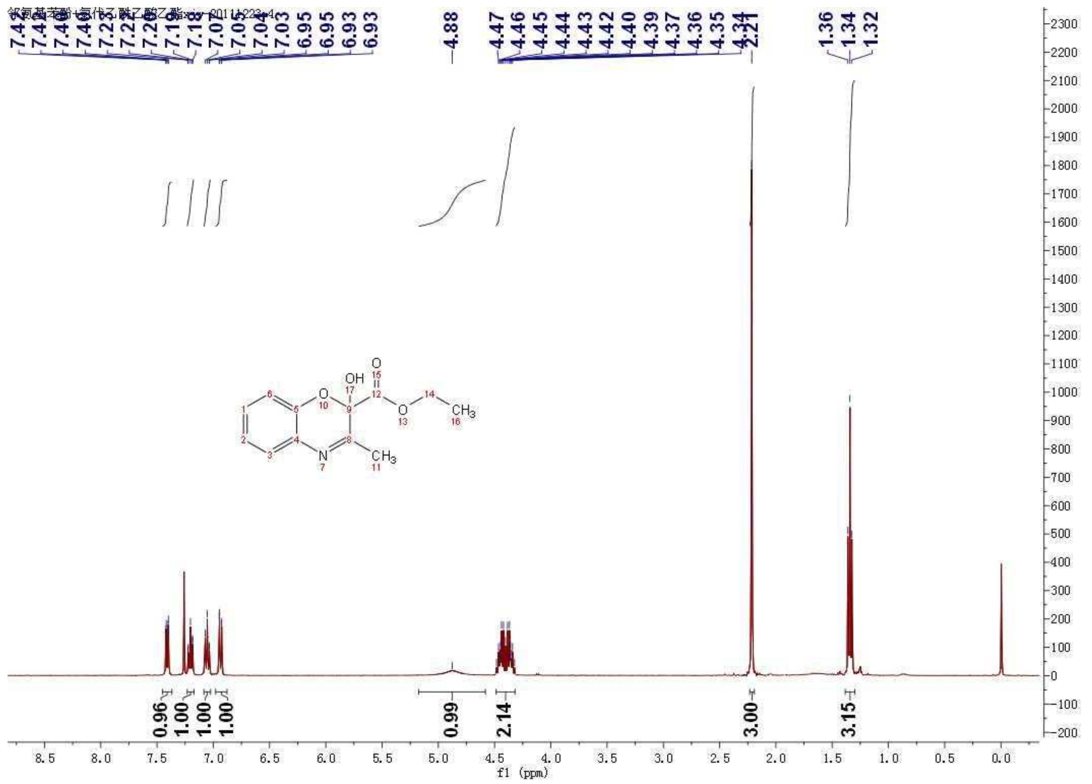
14.12
14.05
f1 (ppm)

3ac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4.42
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4.39
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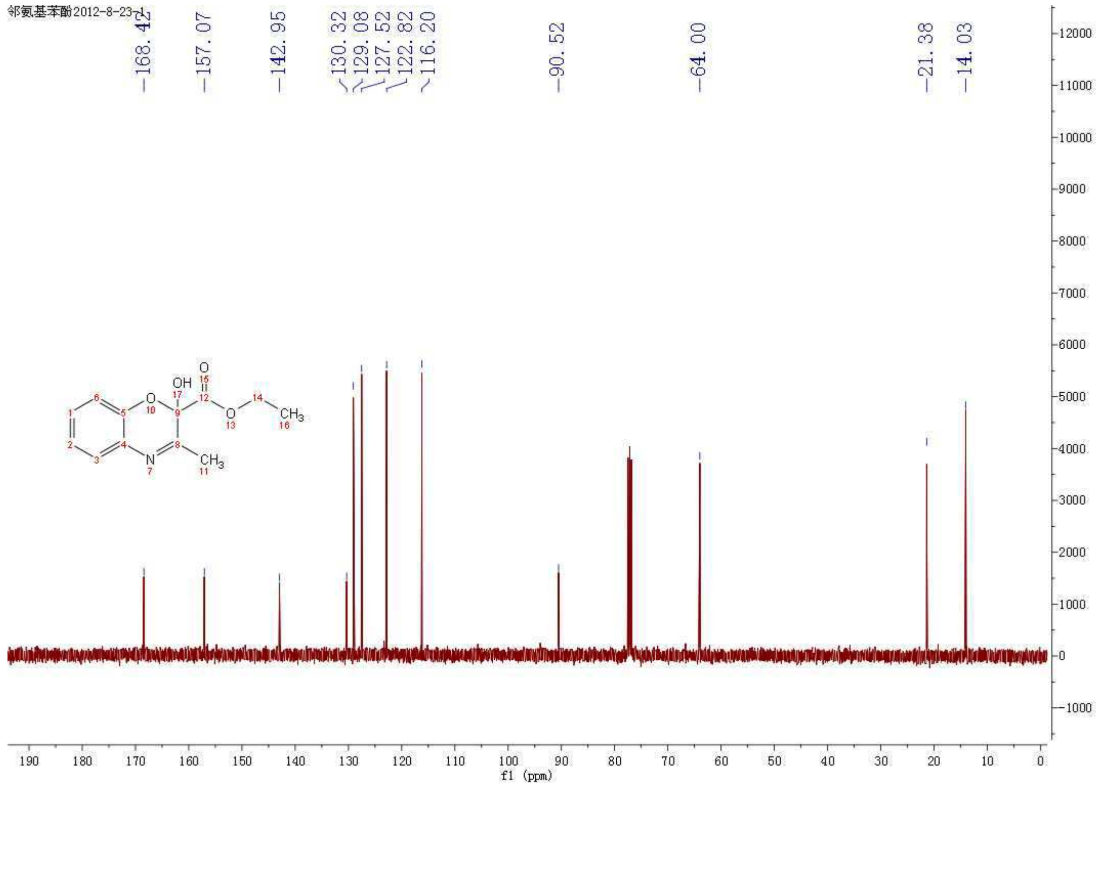
f1 (ppm)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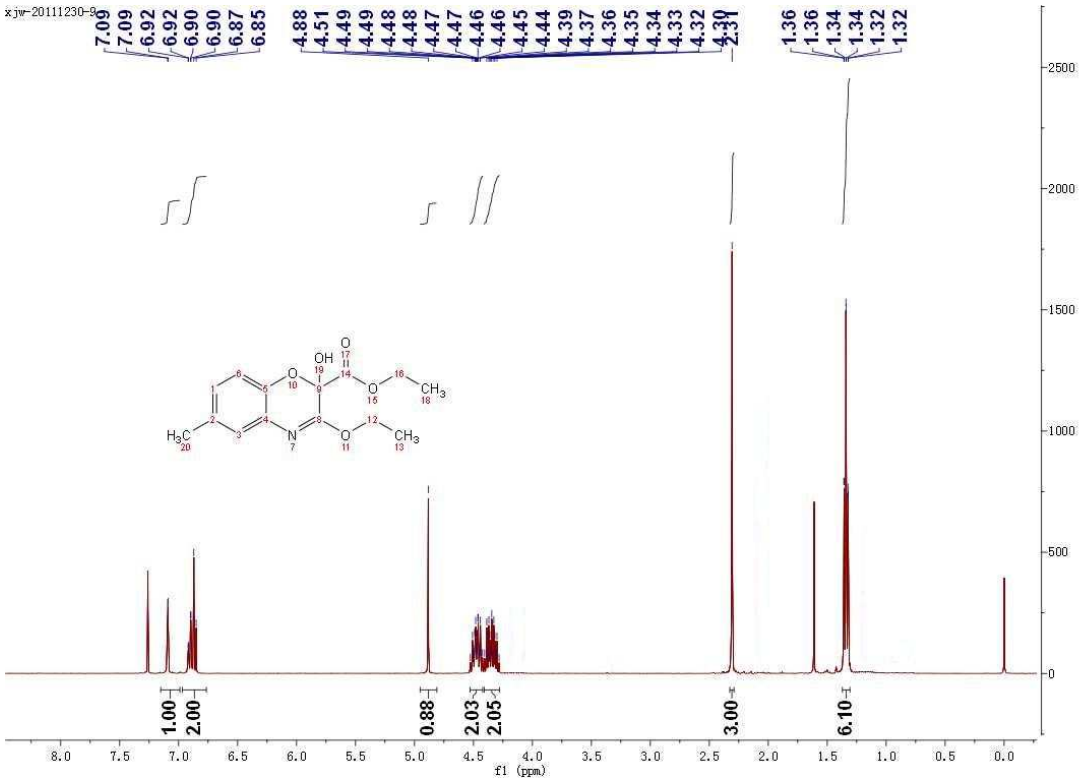
3ba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4.32
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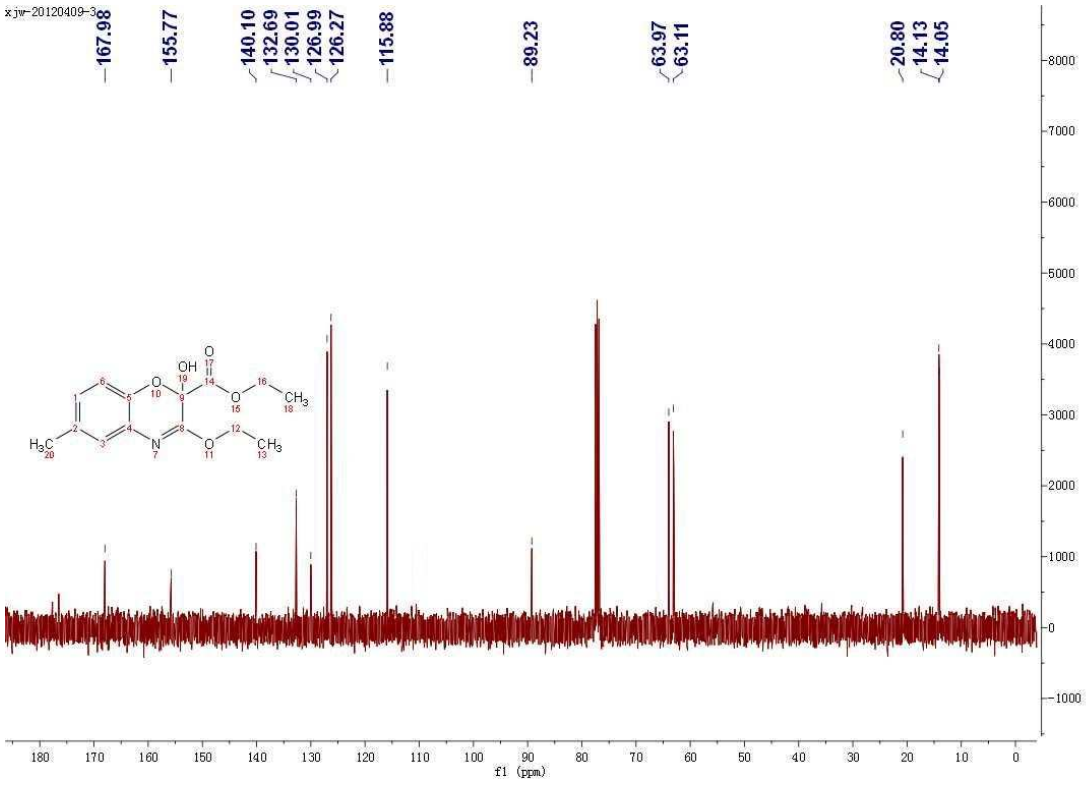
f1 (ppm)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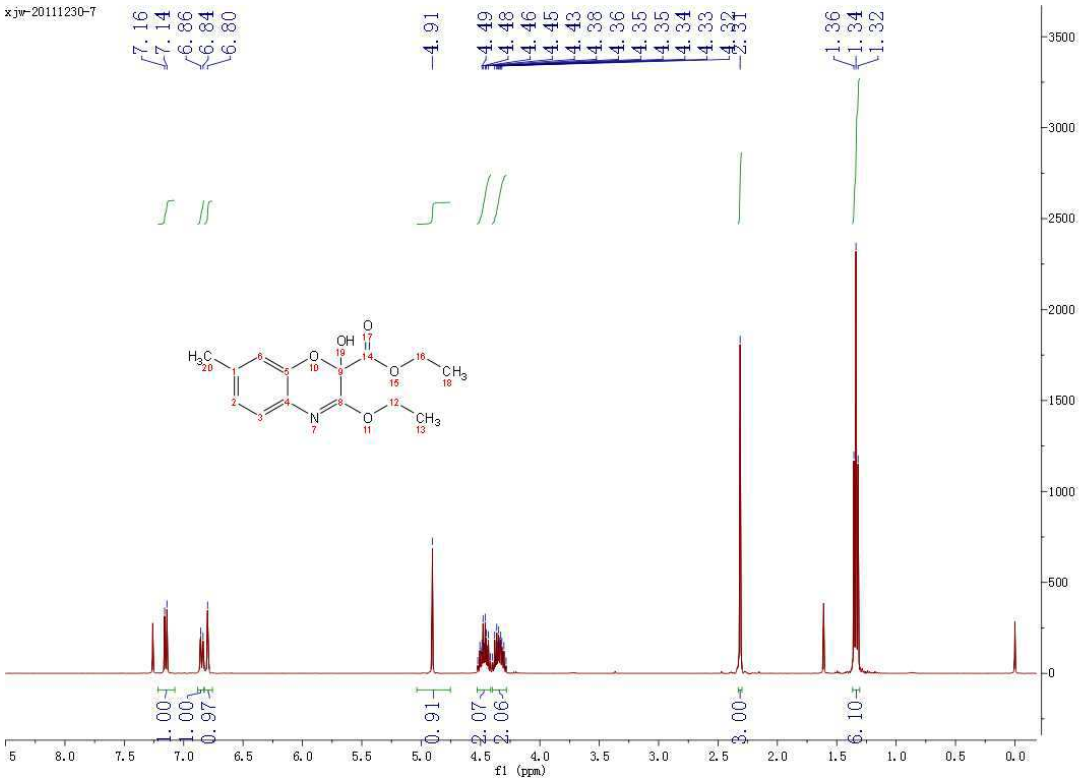
3ca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4.33
4.32
4.31
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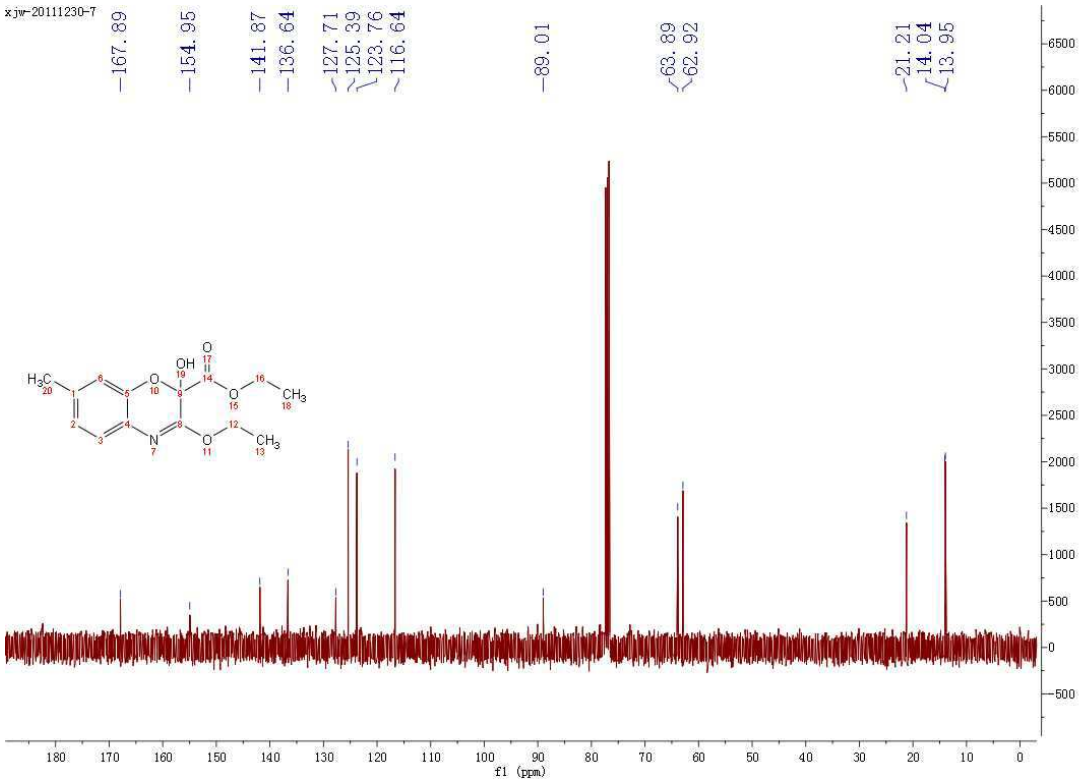
f1 (ppm)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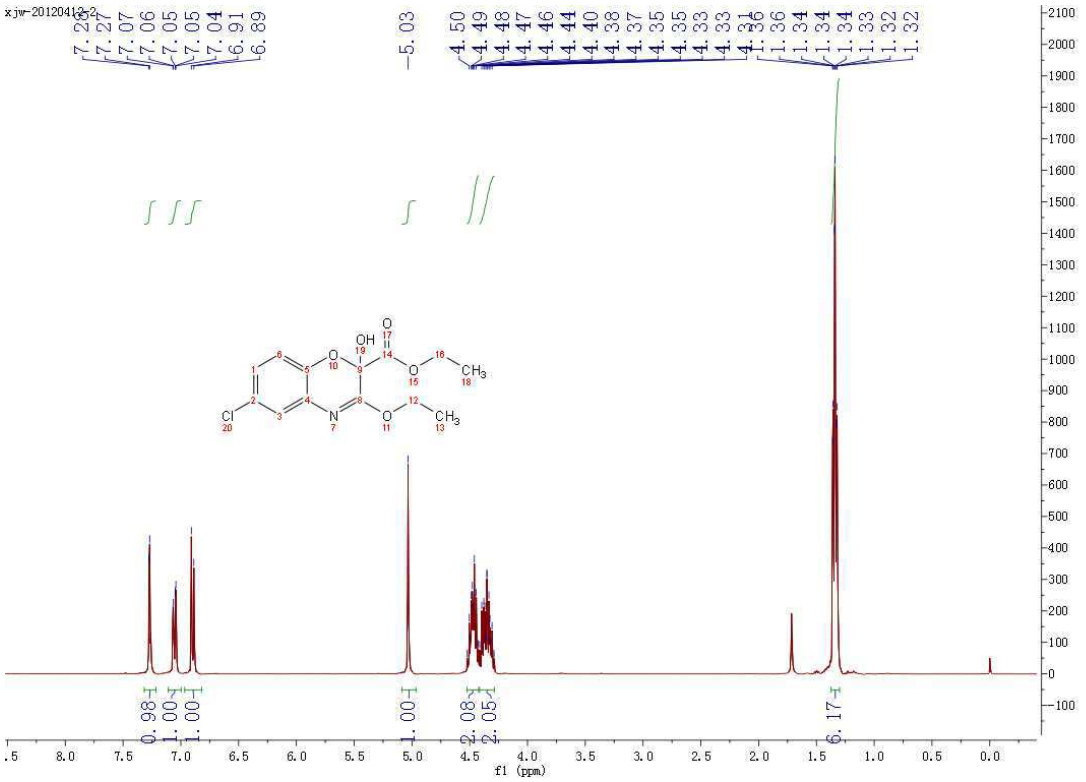
f1 (ppm)

3da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1.00
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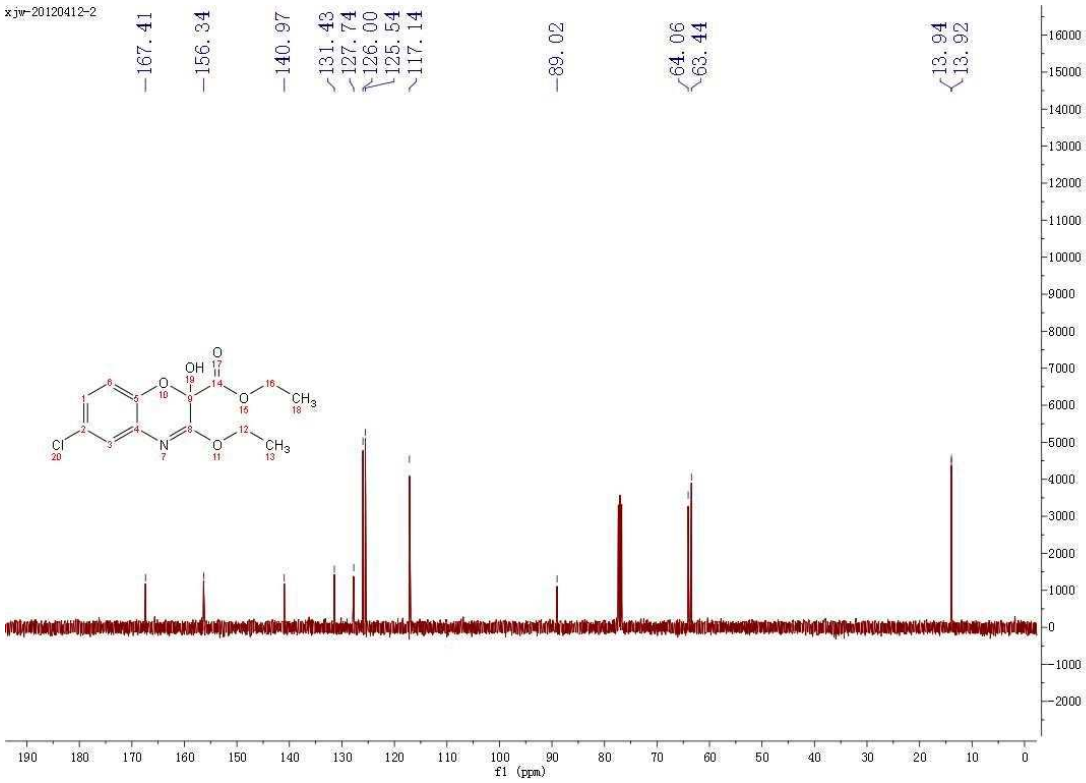
6.17
f1 (ppm)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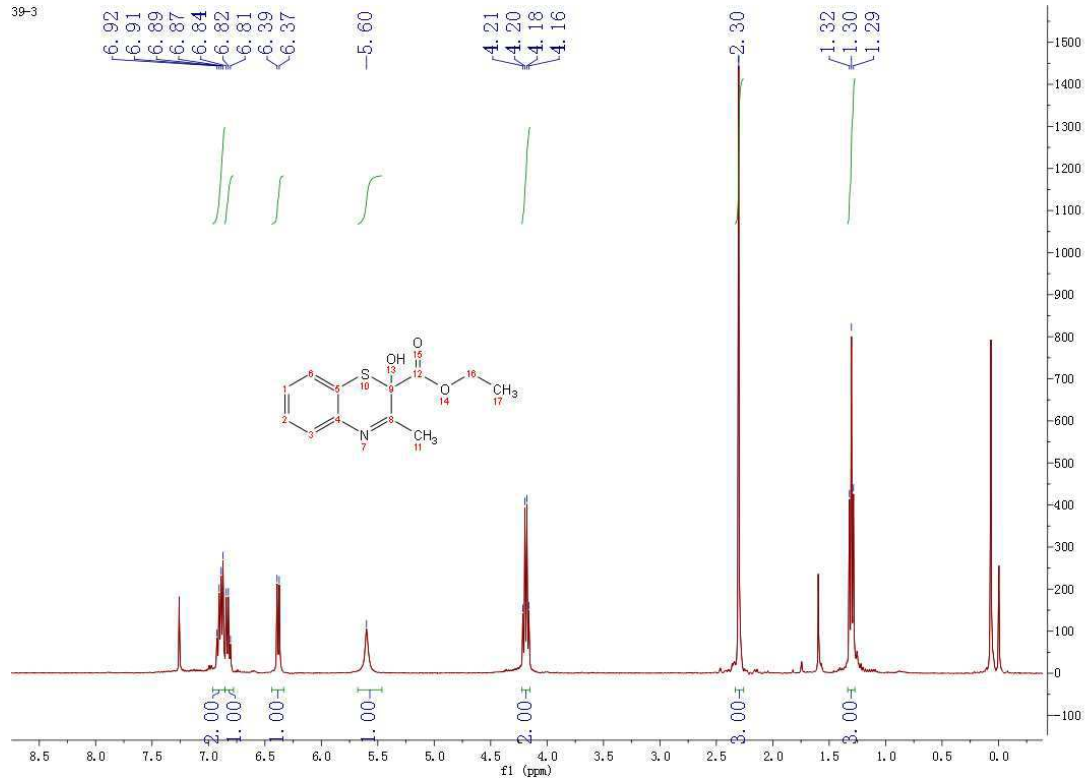
f1 (ppm)

3ec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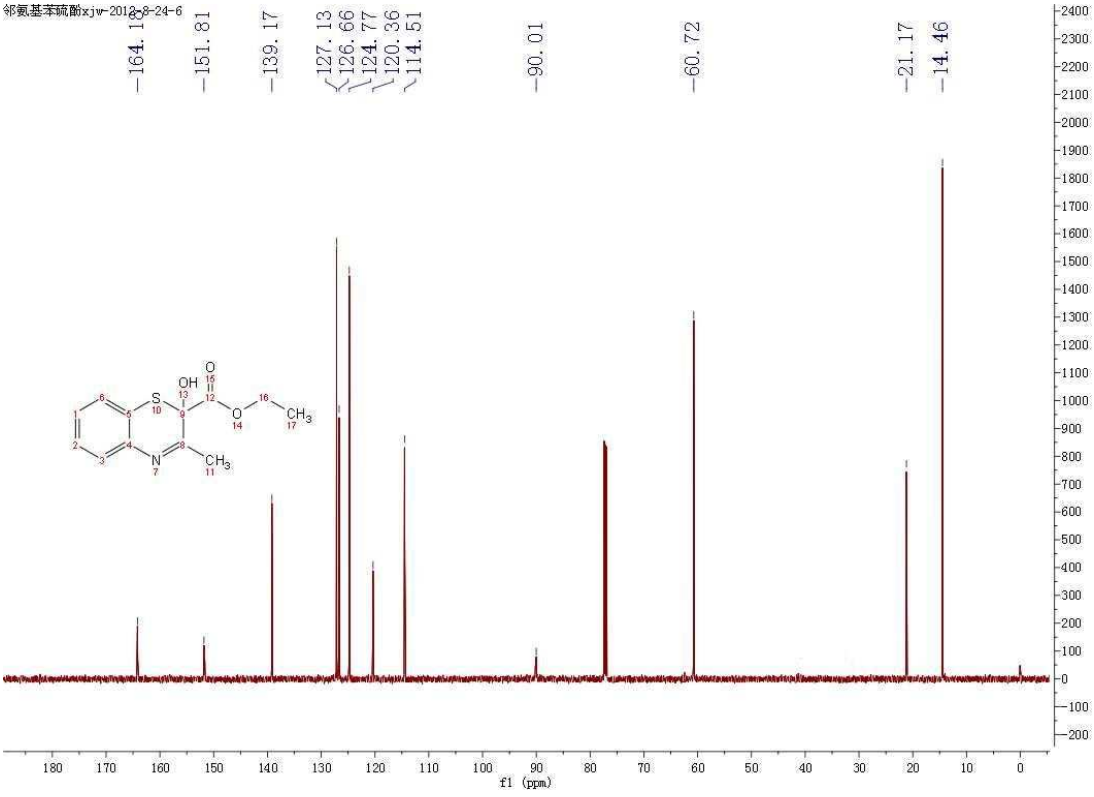
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