Direct C-4 Alkylation of Quinazoline N-Oxides with Ethers viaOxidative Cross-Coupling Reaction under Transition-Metal-free Conditions

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Supporting Information

General experimental methods:

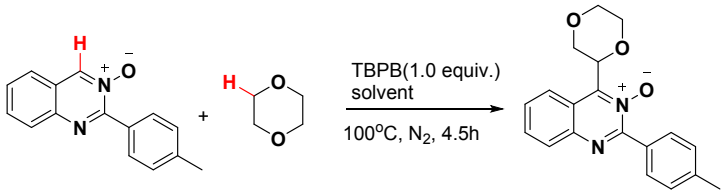
Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32-63μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230-400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr at 25-35°C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument.

General experimental procedure

A Schlenk tube was charged withquinazoline 3-oxide **1** (0.20 mmol) and *t*-butyl peroxybenzoate (TBPB, 0.2 mmol, 1.0 equiv.) under N_2. Then 1,4-dioxane **2a**(1.0 mL) was injected into the bottom of the tube using a long needle syringe. The mixture was stirred at 100 °C for 4.5 h. After completion of the reaction (monitored by TLC), Et_3N (2.0 mL) was added to remove the benzoic acid. Then, the mixture was concentrated in vacuum and the residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give the desired product.

The reactions of quinazoline-3-oxide and 1,4-dioxane in various of solvents

Screening of the solvents revealed that the reaction could be performed at a loading of 10 equivalents **2a** in PhCl (entry 2), but the yield was lowered to 25%.

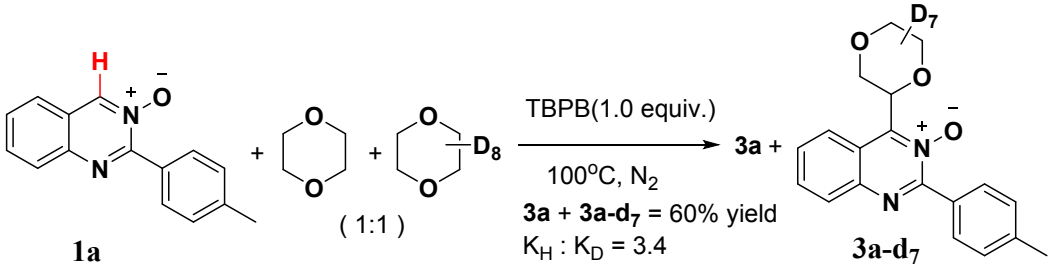


1a **2a** **3a**

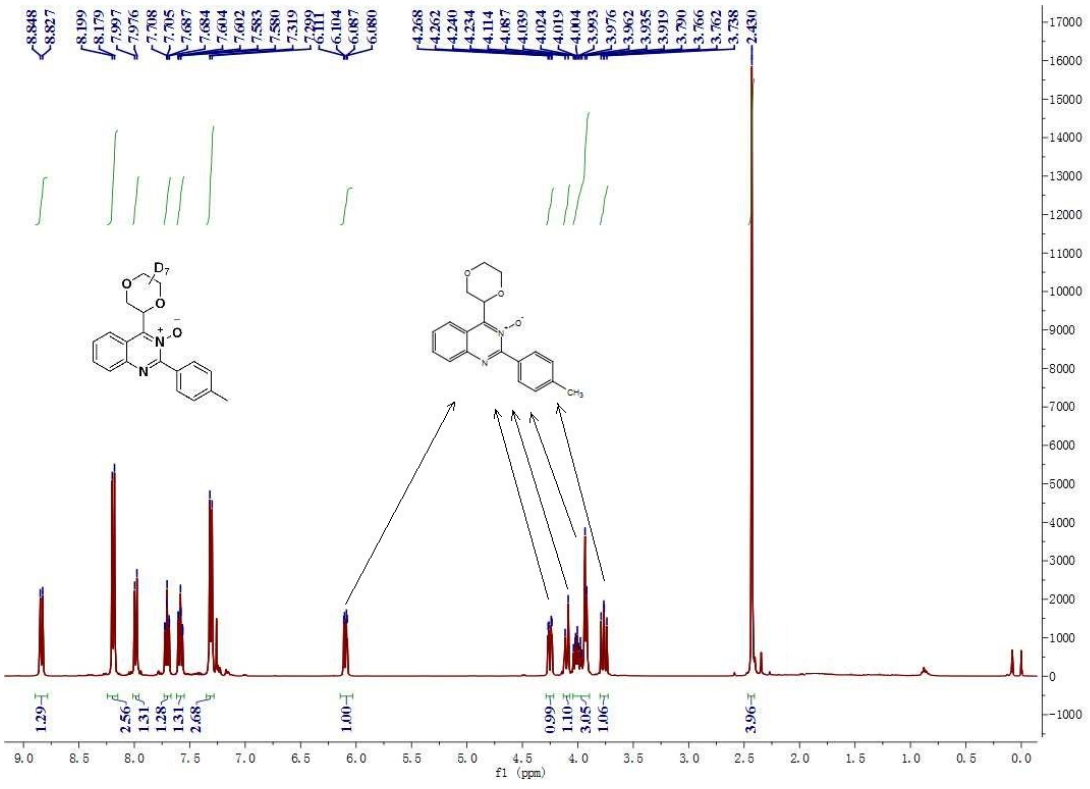
Entry[a]	solvert	T/°C	Time/h	Yield/%[b]
1	toluene	100	4.5	16
2	PhCl	100	4.5	25
3	THF	100	4.5	6
4	DCE	100	4.5	trace
5	EA	100	4.5	15

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (10.0 equiv), solvent 1.0mL, under N_2 , sealed tube. [b] Isolated yield.

The KIE studies on ethers:

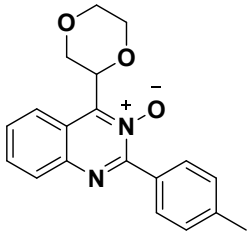


In a Schlenk tube, the mixture of **1a** (0.2 mmol), 1,4-dioxane and 1,4-dioxane-d_8 (1:1, 1.0 mL) was treated by standard condition for 4.5 h. After completion of the reaction, Et_3N (2.0 mL) was added to remove the benzoic acid. Then, the mixture was concentrated in vacuum and the residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give product **3a** and **3a**-d_7. The mixture was analyzed using ^{1}H NMR spectrometer. The data analysis method is explained as follows:

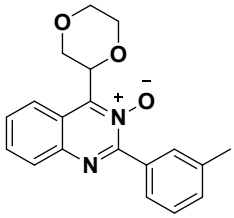


The 1,4-dioxane part of **3a**-d_7 are silent on ^{1}H NMR while that of **3a** are responding. So the ether proton adjacent toquinazoline skeleton was integrated as 1.00. Both **3a** and **3a**-d_7 have signal in the region of 7.28–8.85 ppm. The ratio of 3a / 3a-d7 was calculated to be 1/(1.29-1) = 1/0.29 = 3.4.

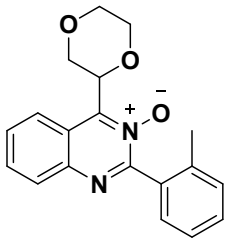
Characterization data for the cross-coupled products:



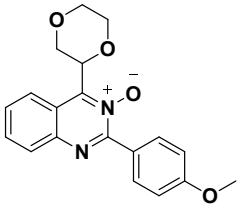
4-(1,4-dioxan-2-yl)-2-(p-tolyl)quinazoline 3-oxide (**3a**). Compound was obtained as a faint yellow solid: yield 76%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.84 (d, *J* = 8.8 Hz, 1H), 8.18 (d, *J* = 8.4 Hz, 2H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.77 – 7.67 (m, 1H), 7.62 – 7.55 (m, 1H), 7.31 (d, *J* = 8.4 Hz, 2H), 6.10 (dd, *J* = 9.6, 2.8 Hz, 1H), 4.25 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.10 (d, *J* = 10.8 Hz, 1H), 4.05 – 3.90 (m, 3H), 3.77 (t, *J* = 10.8, 1H), 2.43 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 154.3, 149.8, 141.3, 140.9, 131.1, 130.3, 130.0, 129.3, 129.1, 128.7, 128.5, 128.3, 124.3, 122.8, 75.2, 67.6, 67.0, 66.6, 21.6.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{19}H_{18}N_2O_3$: 323.1396, found 323.1397.



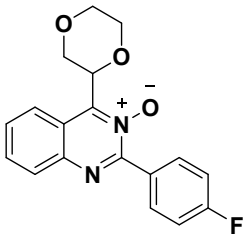
4-(1,4-dioxan-2-yl)-2-(m-tolyl)quinazoline 3-oxide (**3b**). Compound was obtained as a faint yellow solid: yield 78%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (d, *J* = 8.4 Hz, 1H), 8.10 – 7.95 (m, 3H), 7.76-7.69 (m, 1H), 7.64-7.56 (m, 1H), 7.40 (t, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 6.10 (dd, *J* = 9.6, 2.6 Hz, 1H), 4.26 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.11 (d, *J* = 11.2 Hz, 1H), 4.07 – 3.88 (m, 3H), 3.77 (t, *J* = 10.0, 1H), 2.45 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 154.5, 149.8, 140.9, 137.7, 132.1, 131.7, 131.2, 130.7, 129.1, 128.7, 127.9, 127.4, 124.3, 123.0, 75.2, 67.6, 67.0, 66.6, 21.5.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{19}H_{18}N_2O_3$: 323.1396, found 323.1394.



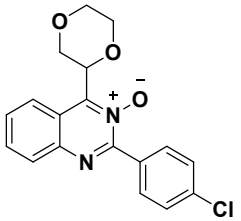
4-(1,4-dioxan-2-yl)-2-(o-tolyl)quinazoline 3-oxide(**3c**). Compound was obtained as a white solid: yield 71%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.90 (d, *J* = 8.4 Hz, 1H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.75 (t, *J* = 7.6 Hz, 1H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 7.2 Hz, 1H), 7.42 (t, *J* = 7.4 Hz, 1H), 7.33 (t, *J* = 7.2 Hz, 2H), 6.06 (dd, *J* = 9.6, 2.4 Hz, 1H), 4.24 (dd, *J* = 11.2, 2.0 Hz, 1H), 4.11 (d, *J* = 11.2 Hz, 1H), 4.06 – 3.89 (m, 3H), 3.79 (t, *J* = 10.4 Hz, 1H), 2.27 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 156.5, 149.2, 140.4, 137.4, 132.8, 131.2, 130.3, 130.0, 129.2, 129.1, 129.0, 125.9, 124.3, 123.2, 75.0, 67.6, 67.0, 66.6, 19.5.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{19}H_{18}N_2O_3$: 323.1396, found 323.1397.



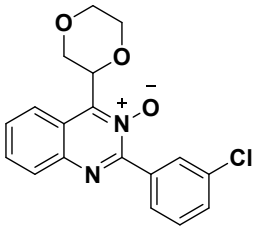
4-(1,4-dioxan-2-yl)-2-(4-methoxyphenyl)quinazoline 3-oxide(**3d**). Compound was obtained as afaint yellow solid: yield 75%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.85 (d, *J* = 8.8 Hz, 1H), 8.37 (d, *J* = 8.8 Hz, 2H), 7.99 (d, *J* = 8.4 Hz, 1H), 7.72 (t, *J* = 7.6 Hz, 1H), 7.59 (t, *J* = 7.8 Hz, 1H), 7.02 (d, *J* = 8.8 Hz, 2H), 6.11 (dd, *J* = 9.6, 2.4 Hz, 1H), 4.27 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.11 (d, *J* = 11.2 Hz, 1H), 4.02-3.96 (m, 3H), 3.89 (s, 3H), 3.77 (t, *J* = 10.4 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 161.8, 153.7, 150.0, 141.1, 132.4, 131.3, 129.0, 128.3, 124.4, 124.3, 122.6, 113.4, 75.2, 67.6, 67.1, 66.6, 55.5.HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{19}H_{18}N_2O_4$: 339.1345, found 339.1347.



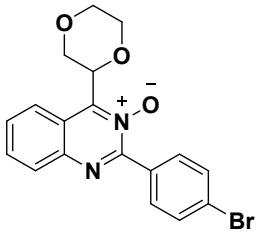
4-(1,4-dioxan-2-yl)-2-(4-fluorophenyl)quinazoline 3-oxide (**3e**). Compound was obtained as afaint yellow solid: yield 78%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (d, *J* = 8.8 Hz, 1H), 8.36 (dd, *J* = 8.8, 5.6 Hz, 2H), 8.00 (d, *J* = 8.4 Hz, 1H), 7.74 (t, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 7.8 Hz, 1H), 7.19 (t, *J* = 8.6 Hz, 2H), 6.09 (dd, *J* = 9.6, 2.6 Hz, 1H), 4.25 (dd, *J* = 11.2, 2.6 Hz, 1H), 4.12 (d, *J* = 11.2 Hz, 1H), 4.06 – 3.91 (m, 3H), 3.77 (t, *J* = 10.6 Hz,1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 164.3 (d, *J* = 250.6 Hz), 153.1, 150.1, 140.9, 132.9 (d, *J* = 8.7 Hz), 131.4, 129.1, 128.8, 128.2 (d, *J* = 3.4 Hz), 124.4, 122.9, 115.1 (d, *J* = 21.7 Hz), 75.1, 67.6, 67.0, 66.6.HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{18}H_{15}FN_2O_3$: 327.1145, found 327.1146.



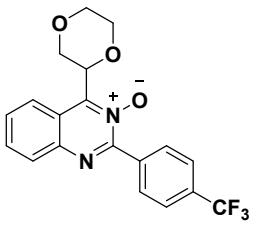
2-(4-chlorophenyl)-4-(1,4-dioxan-2-yl)quinazoline 3-oxide (**3f**). Compound was obtained as ayellow solid: yield 70%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (d, *J* = 8.4 Hz, 1H), 8.30 (d, *J* = 8.8 Hz, 2H), 7.99 (d, *J* = 8.4 Hz, 1H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.47 (d, *J* = 8.8 Hz, 2H), 6.08 (dd, *J* = 9.6, 2.4 Hz, 1H), 4.24 (dd, *J* = 11.1, 2.4 Hz, 1H), 4.11 (d, *J* = 11.2 Hz, 1H), 4.05 – 3.90 (m, 3H), 3.76 (t, *J* = 10.4, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 153.0, 150.0, 140.8, 137.1, 131.9, 131.3, 130.6, 129.1, 128.9, 128.2, 124.3, 123.0, 75.1, 67.6, 67.0, 66.6.HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{18}H_{15}ClN_2O_3$: 343.0849, found 343.0850.



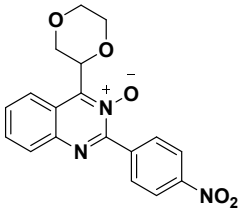
2-(3-chlorophenyl)-4-(1,4-dioxan-2-yl)quinazoline 3-oxide(**3g**). Compound was obtained as a faint yellow solid: yield 70%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.87 (d, *J* = 8.4 Hz, 1H), 8.31 (s, 1H), 8.20 (d, *J* = 7.6 Hz, 1H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.75 (t, *J* = 7.8 Hz, 1H), 7.64 (t, *J* = 7.8 Hz, 1H), 7.54-7.48 (m, 1H), 7.44 (t, *J* = 7.8 Hz, 1H), 6.08 (dd, *J* = 9.6, 2.8 Hz, 1H), 4.24 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.12 (d, *J* = 10.4 Hz, 1H), 4.07 – 3.92 (m, 3H), 3.77 (t, *J* = 10.4 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 152.7, 150.1, 140.8, 134.0, 133.8, 131.4, 131.0, 130.4, 129.2, 129.1, 128.6, 124.4, 123.1, 75.1, 67.6, 67.0, 66.6.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{15}ClN_2O_3$: 343.0849, found 343.0849.



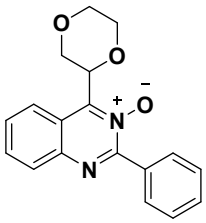
2-(4-bromophenyl)-4-(1,4-dioxan-2-yl)quinazoline 3-oxide (**3h**). Compound was obtained as ayellow solid: yield 60%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.85 (d, *J* = 8.4 Hz, 1H), 8.22 (d, *J* = 8.4 Hz, 2H), 7.99 (d, *J* = 8.4 Hz, 1H), 7.73 (t, *J* = 7.4 Hz, 1H), 7.67-7.57 (m, 3H), 6.07 (dd, *J* = 9.6, 2.8 Hz, 1H), 4.24 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.11 (d, *J* = 11.2 Hz, 1H), 4.05 – 3.90 (m, 3H), 3.76 (t, *J* = 10.6, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 153.1, 150.0, 140.8, 132.0, 131.3, 131.2, 131.1, 129.2, 128.9, 125.6, 124.4, 123.0, 75.1, 67.6, 67.0, 66.6.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{15}BrN_2O_3$: 387.0344, found 387.0343.



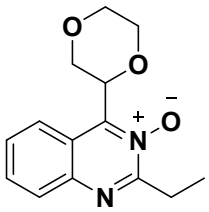
4-(1,4-dioxan-2-yl)-2-(4-(trifluoromethyl)phenyl)quinazoline 3-oxide(**3i**). Compound was obtained as ayellow solid: yield 77%;^{1}H NMR (400 MHz, $CDCl_3$) δ8.90 (d, *J* = 8.4 Hz, 1H), 8.40 (d, *J* = 8.0 Hz, 2H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.80-7.73 (m, 3H), 7.69-7.63 (m, 1H), 6.09 (dd, *J* = 9.6, 2.8 Hz, 1H), 4.25 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.12 (d, *J* = 10.8 Hz, 1H), 4.07 – 3.90 (m, 3H), 3.78 (t, *J* = 10.4,1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 152.9, 150.4, 141.0, 135.7, 132.4 (q, ^{2}J = 33.1 Hz), 131.7, 130.9, 129.4, 128.4, 125.0 (q, ^{3}J = 3.7 Hz), 124.6, 124.0 (q, *J* = 270.9 Hz), 123.3, 75.2, 67.7, 67.1, 66.7..HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{19}H_{15}F_3N_2O_3$: 377.1113, found 377.1100.



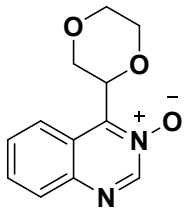
4-(1,4-dioxan-2-yl)-2-(4-nitrophenyl)quinazoline 3-oxide (3j). Compound was obtained as ayellow solid: yield 50%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.93 (d, *J* = 8.4 Hz, 1H), 8.21 (d, *J* = 8.0 Hz, 1H), 8.02 (d, *J* = 8.4 Hz, 1H), 7.85 – 7.64 (m, 5H), 5.96 (dd, *J* = 9.6, 2.4 Hz, 1H), 4.15 (dd, *J* = 11.2, 2.2 Hz, 1H), 4.09 (d, *J* = 10.0 Hz, 1H), 4.01-3.87 (m, 3H), 3.73 (t, *J* = 10.4 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 153.8, 149.0, 148.6, 140.9, 134.0, 131.9, 131.5, 131.1, 129.4, 129.1, 128.1, 124.8, 123.9, 123.4, 74.9, 67.5, 66.9, 66.6.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{15}N_3O_5$: 354.1090, found 354.1090.



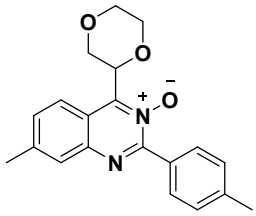
4-(1,4-dioxan-2-yl)-2-phenylquinazoline 3-oxide(**3k**). Compound was obtained as ayellow solid: yield 80%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (d, *J* = 8.4 Hz, 1H), 8.29 – 8.20 (m, 2H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.76 – 7.69 (m, 1H), 7.66 – 7.57 (m, 1H), 7.56 – 7.45 (m, 3H), 6.10 (dd, *J* = 9.6, 2.8 Hz, 1H), 4.25 (dd, *J* = 11.2, 2.8 Hz, 1H), 4.11 (d, *J* = 11.2 Hz, 1H), 4.05 – 3.90 (m, 3H), 3.77 (t, *J* = 10.4, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 154.2, 149.8, 140.9, 132.2, 131.2, 130.9, 130.3, 129.2, 128.8, 128.0, 124.3, 123.0, 75.1, 67.6, 67.0, 66.6.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{16}N_2O_3$: 309.1239, found 309.1240.



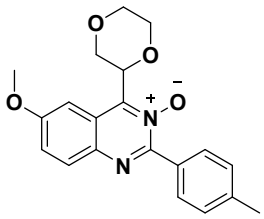
4-(1,4-dioxan-2-yl)-2-ethylquinazoline 3-oxide (**3l**). Compound was obtained as ayellow solid: yield 74%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (d, *J* = 8.4 Hz, 1H), 7.94 (d, *J* = 8.4 Hz, 1H), 7.70 (t, *J* = 8.0Hz, 1H), 7.57 (t, *J* = 7.8 Hz, 1H), 6.07 (dd, *J* = 9.6, 2.8 Hz, 1H), 4.19 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.09 (d, *J* = 10.8 Hz, 1H), 4.04 – 3.90 (m, 3H), 3.76 (t, *J* = 10.4Hz, 1H), 3.31-3.18 (m, 1H), 1.44 (t, *J* = 7.4 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 159.9, 148.1, 140.3, 130.8, 128.6, 128.0, 124.3, 122.5, 77.4, 77.1, 76.8, 74.8, 67.5, 66.9, 66.6, 25.6, 9.9.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{14}H_{16}N_2O_3$: 261.1239, found 261.1236.



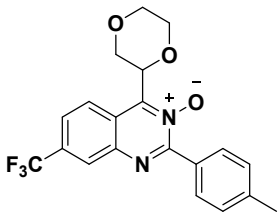
4-(1,4-dioxan-2-yl)quinazoline 3-oxide (3m). Compound was obtained as a white solid: yield 54%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.93 (s, 1H), 8.84 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 8.4 Hz, 1H), 7.79 – 7.72 (m, 1H), 7.71 – 7.63 (m, 1H), 6.02 (dd, *J* = 9.8, 2.8 Hz, 1H), 4.19 (dd, *J* = 11.2, 2.8 Hz, 1H), 4.10 (d, *J* = 10.8 Hz, 1H), 4.04 – 3.90 (m, 3H), 3.74 (t, *J* = 10.4, Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 148.4, 147.1, 141.2, 131.3, 129.5, 129.1, 124.8, 123.1, 74.3, 67.5, 66.9, 66.6.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{12}H_{12}N_2O_3$: 233.0926, found 233.0926.



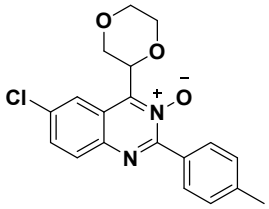
4-(1,4-dioxan-2-yl)-7-methyl-2-(p-tolyl)quinazoline 3-oxide (**3n**). Compound was obtained as a light yellow solid: yield 71%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 8.8 Hz, 1H), 8.19 (d, *J* = 8.0 Hz, 2H), 7.78 (s, 1H), 7.42 (d, *J* = 8.8 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 6.09 (dd, *J* = 9.6, 2.8 Hz, 1H), 4.25 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.10 (d, *J* = 11.2 Hz, 1H), 4.05 – 3.89 (m, 3H), 3.74 (t, *J* = 10.4,1H), 2.54 (s, 3H), 2.43 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 154.2, 149.7, 142.0, 142.3,141.2, 130.7, 130.3, 129.5, 128.6, 128.2, 123.9, 120.9, 75.2, 67.6, 67.1, 66.6, 21.8, 21. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{20}H_{20}N_2O_3$: 337.1552, found 337.1556.



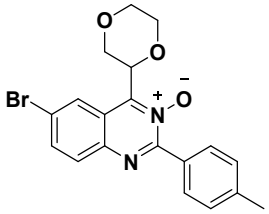
4-(1,4-dioxan-2-yl)-6-methoxy-2-(p-tolyl)quinazoline 3-oxide(3o). Compound was obtained as a light yellow solid: yield 68%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.0 Hz, 3H), 7.89 (d, *J* = 9.2 Hz, 1H), 7.35 (dd, *J* = 9.2, 2.8 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.10 (dd, *J* = 9.6, 2.4 Hz, 1H), 4.24 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.09 (d, *J* = 11.6 Hz, 1H), 4.05-3.99 (m, 1H), 3.96 (s, 3H), 3.93-3.85 (m, 2H), 3.77 (t, *J* = 10.4 Hz, 1H), 2.42 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 158.9, 152.3, 148.3, 140.9, 137.0, 130.5, 130.1, 129.5, 128.6, 124.1, 123.1, 102.7, 75.1, 67.61, 66.74, 66.69, 55.6, 21.5.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{20}H_{20}N_2O_4$: 353.1501, found 353.1499.



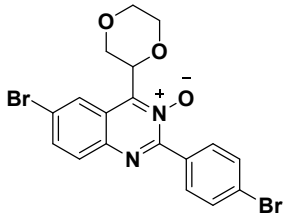
4-(1,4-dioxan-2-yl)-2-(p-tolyl)-7-(trifluoromethyl)quinazoline 3-oxide (3p). Compound was obtained as ayellow oil liquid: yield 77%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.98 (d, J = 8.8 Hz, 1H), 8.28 (s, 1H), 8.22 (d, J = 8.4 Hz, 2H), 7.74 (d, J = 9.2 Hz, 1H), 7.32 (d, J = 8.4 Hz, 2H), 6.03 (dd, J = 9.6, 2.4 Hz, 1H), 4.26 (dd, J = 11.2, 2.4 Hz, 1H), 4.12 (d, J = 10.8 Hz, 1H), 4.06 – 3.90 (m, 3H), 3.71 (t, J = 10.6, 1H), 2.45 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 155.5, 149.7, 142.0, 139.7, 132.3 (q, ^{2}J = 33.0 Hz), 130.4, 128.8, 128.6, 126.6 (q, ^{3}J = 4.4 Hz),125.5, 124.4, 124.1 (q, ^{3}J = 3.0 Hz), 123.4 (q, ^{1}J = 270.9 Hz),75.2, 67.6, 66.9, 66.6, 21.6.HRMS (ESI): m/z [M + H]$^{+}$ calcd for $C_{20}H_{17}F_3N_2O_3$: 391.1270, found 391.1266.



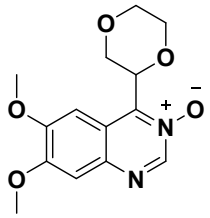
6-chloro-4-(1,4-dioxan-2-yl)-2-(p-tolyl)quinazoline 3-oxide(3q). Compound was obtained as ayellow solid: yield 82%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.82 (d, J = 2.0 Hz, 1H), 8.18 (d, J = 8.0 Hz, 2H), 7.91 (d, J = 8.8 Hz, 1H), 7.63 (dd, J = 8.8, 2.4 Hz, 1H), 7.31 (d, J = 8.0 Hz, 2H), 6.01 (dd, J = 9.6, 2.8 Hz, 1H), 4.25 (dd, J = 11.2, 2.8 Hz, 1H), 4.14 (d, J = 9.6 Hz, 1H), 4.04 – 3.91 (m, 3H), 3.70 (t, J = 10.4 Hz, 1H), 2.43 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ154.4, 148.9, 141.7, 139.1, 134.4, 131.7, 130.5, 130.3, 128.9, 128.7, 123.5, 123.0, 75.3, 67.6, 66.8, 66.6, 21.6.HRMS (ESI): m/z [M + H]$^{+}$ calcd for $C_{19}H_{17}ClN_2O_3$: 357.1006, found 357.1005.



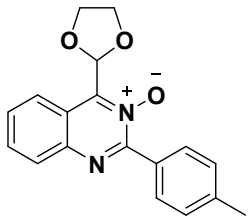
6-bromo-4-(1,4-dioxan-2-yl)-2-(p-tolyl)quinazoline 3-oxide(3r). Compound was obtained as ayellow solid: yield 53%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.99 (d, J = 2.0 Hz, 1H), 8.18 (d, J = 8.4 Hz, 2H), 7.84 (d, J = 8.8 Hz, 1H), 7.76 (dd, J = 9.0, 2.2 Hz, 1H), 7.31 (d, J = 8.4 Hz, 2H), 6.00 (dd, J = 9.6, 2.4 Hz, 1H), 4.25 (dd, J = 11.2, 2.8 Hz, 1H), 4.14 (d, J = 9.6 Hz, 1H), 4.04 – 3.89 (m, 3H), 3.70 (t, J = 10.0, 1H), 2.43 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 154.5, 148.8, 141.7, 139.3, 134.3, 130.6, 130.3, 128.9, 128.7, 126.2, 123.9, 122.7, 75.3, 67.6, 66.8, 66.6, 21.6.HRMS (ESI): m/z [M + H]$^{+}$ calcd for $C_{19}H_{17}BrN_2O_3$: 401.0501, found 401.0498.



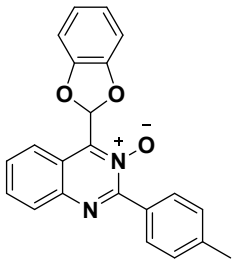
6-bromo-2-(4-bromophenyl)-4-(1,4-dioxan-2-yl)quinazoline 3-oxide(3s).
Compound was obtained as ayellow solid: yield 67%;^{1}H NMR (400 MHz, $CDCl_3$) δ 9.00 (d, *J* = 2.0 Hz, 1H), 8.21 (d, *J* = 8.8 Hz, 2H), 7.84 (d, *J* = 8.8 Hz, 1H), 7.78 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.63 (d, *J* = 8.8 Hz, 2H), 5.98 (dd, *J* = 9.6, 2.8 Hz, 1H), 4.23 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.14 (d, *J* = 9.2 Hz, 1H), 4.04 – 3.89 (m, 3H), 3.69 (t, *J* = 10.0, Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 153.3, 149.1, 139.2, 134.7, 132.0, 131.3, 130.6, 126.4, 126.0, 124.1, 123.3, 75.2, 67.6, 66.8, 66.6.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{14}Br_2N_2O_3$: 464.9449, found 464.9448.



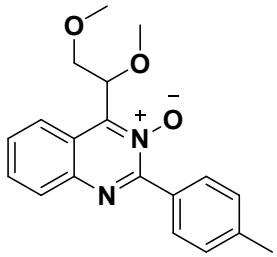
4-(1,4-dioxan-2-yl)-6,7-dimethoxyquinazoline 3-oxide(3t). Compound was obtained as a white solid: yield 59%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.87 (s, 1H), 8.18 (s, 1H), 7.28 (s, 1H), 6.05 (dd, *J* = 9.8, 2.6 Hz, 1H), 4.23 (dd, *J* = 11.8, 2.6 Hz, 1H), 4.04 (s, 6H), 4.02 – 3.92 (m, 2H), 3.89-3.84 (m, 1H), 3.70 (t, *J* = 10.6 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 153.8, 151.3, 147.2, 145.4, 139.6, 119.1, 107.8, 102.7, 74.3, 67.6, 66.9, 66.7, 56.4, 56.1.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{14}H_{16}N_2O_5$: 293.1137, found 293.1136.



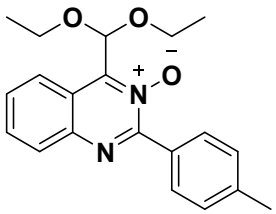
4-(1,3-dioxolan-2-yl)-2-(p-tolyl)quinazoline 3-oxide (3u). Compound was obtained as ayellow solid: yield 65%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (d, *J* = 8.8 Hz, 1H), 8.21 (d, *J* = 8.4 Hz, 2H), 7.98 (d, *J* = 8.0 Hz, 1H), 7.73-7.64 (m, 1H), 7.62-7.53 (m, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 6.88 (s, 1H), 4.43-4.31 (m, 2H), 4.27-4.13 (m, 2H), 2.43 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 154.9, 147.0, 141.3, 140.7, 130.9, 130.4, 129.2, 129.1, 129.0, 128.6, 123.0, 122.3, 98.8, 65.6, 21.6.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{16}N_2O_3$: 309.1239, found 309.1242.



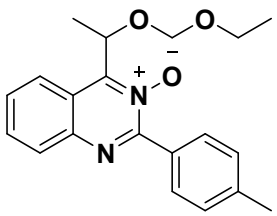
4-(benzo[d][1,3]dioxol-2-yl)-2-(p-tolyl)quinazoline 3-oxide (3v). Compound was obtained as a white solid: yield 51%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (d, *J* = 8.4 Hz, 2H), 8.05 (s, 1H), 8.02 (d, *J* = 8.8 Hz, 2H), 7.68 (t, *J* = 7.8 Hz, 1H), 7.49 (t, *J* = 8.0, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.04 – 6.92 (m, 4H), 2.45 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 154.7, 146.8, 145.4, 141.7, 140.7, 131.1, 130.5, 129.7, 129.4, 128.8, 122.7, 122.2, 121.9, 109.4, 104.7, 21.7.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{22}H_{16}N_2O_3$: 356.1239, found 357.1237.



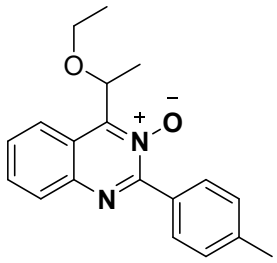
4-(1,2-dimethoxyethyl)-2-(p-tolyl)quinazoline 3-oxide(3w). Compound was obtained as a yellow liquid: yield 41%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (d, *J* = 8.8 Hz, 1H), 8.21 (d, *J* = 8.0 Hz, 2H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.73 (t, *J* = 7.8 Hz, 1H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 5.92 (dd, *J* = 6.4, 2.8 Hz, 1H), 4.03 (dd, *J* = 10.8, 6.4 Hz, 1H), 3.81 (dd, *J* = 10.8, 2.8 Hz, 1H), 3.45 (s, 3H), 3.43 (s, 3H),2.44 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 154.5, 151.2, 141.3, 140.7, 131.2, 130.4, 129.3, 129.3, 129.0, 128.9, 128.7, 128.3, 124.0, 123.1, 79.0, 72.2, 59.3, 58.6, 21.6.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{19}H_{20}N_2O_3$: 325.1552, found 325.1552.



4-(diethoxymethyl)-2-(p-tolyl)quinazoline 3-oxide(3x). Compound was obtained as a white solid: yield 15%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, *J* = 8.8 Hz, 1H), 8.17 (d, *J* = 8.0 Hz, 2H), 7.97 (d, *J* = 8.4 Hz, 1H), 7.72-7.68 (m, 1H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 6.80 (s, 1H), 3.95-4.03 (m, 2H), 3.78-3.71 (m, 2H), 2.43 (s, 3H), 1.28 (t, *J* = 7.0 Hz, 6H).^{13}C NMR (101 MHz, $CDCl_3$) δ 154.6, 141.3, 141.2, 131.2, 130.3, 129.5, 128.7, 128.6, 128.6, 125.2, 121.6, 100.0, 98.3, 65.3, 21.6, 15.4.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{20}H_{22}N_2O_3$: 339.1709, found 339.1701.

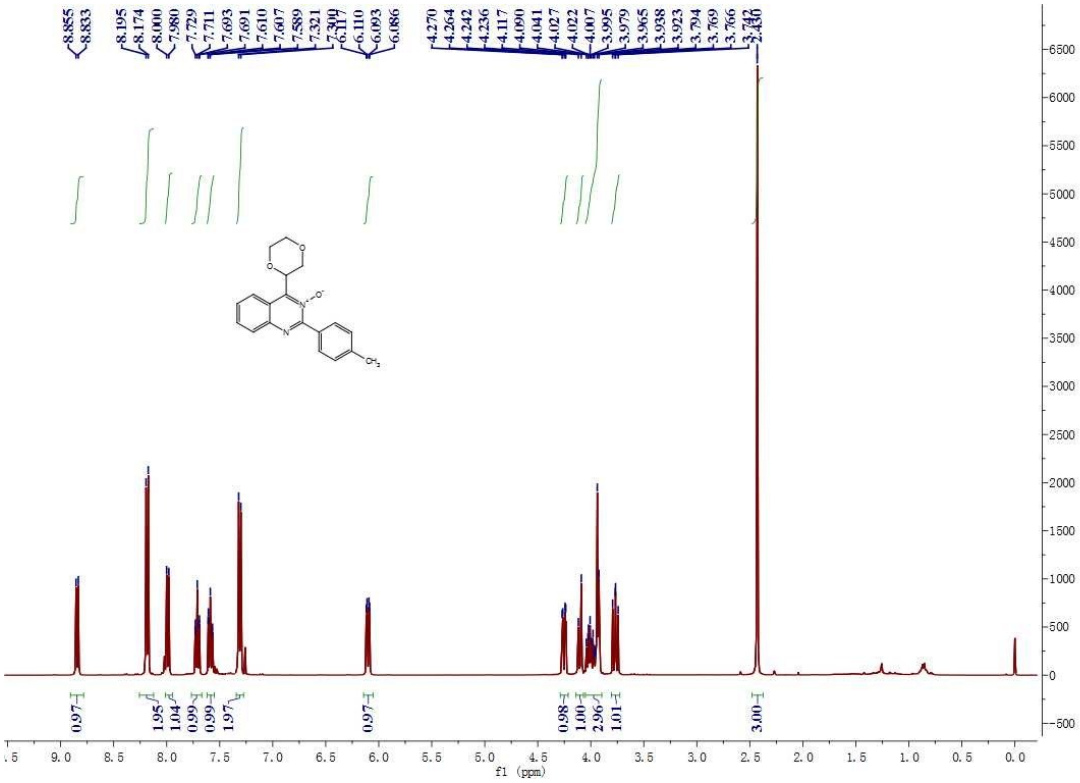


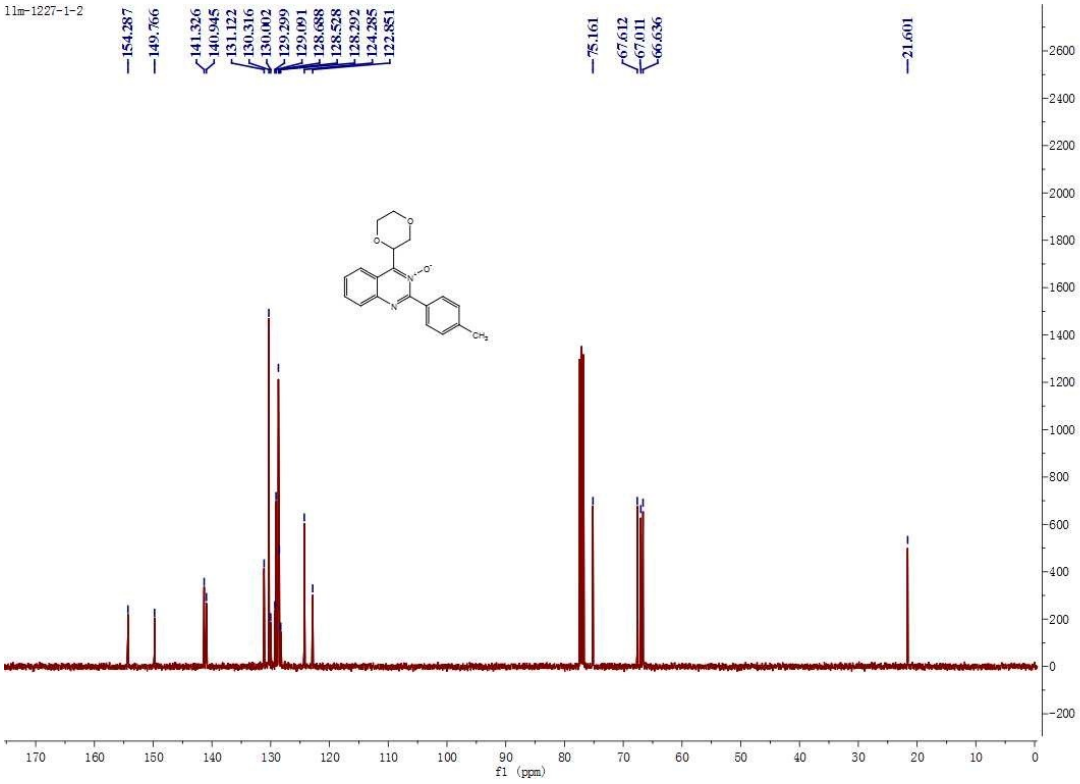
4-(1-(ethoxymethoxy)ethyl)-2-(p-tolyl)quinazoline 3-oxide(3x′). Compound was obtained as ayellow solid: yield 32%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (d, *J* = 8.8 Hz, 1H), 8.23 (d, *J* = 8.4 Hz, 2H), 8.00 (d, *J* = 8.4 Hz, 1H), 7.74 – 7.66 (m, 1H), 7.62 – 7.55 (m, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 6.16 (q, *J* = 6.8 Hz, 1H), 4.79 (d, *J* = 6.8 Hz, 1H), 4.68 (d, *J* = 6.8 Hz, 1H), 3.61-3.53 (m, 1H), 3.43-3.35 (m, 1H), 2.43 (s, 3H), 1.75 (d, *J* = 6.8 Hz, 3H), 0.99 (t, *J* = 7.0 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 155.0, 154.5, 141.2, 140.9, 130.8, 130.4, 129.5, 129.2, 128.6, 128.4, 123.9, 122.0, 95.2, 70.7, 64.1, 21.6, 19.3, 14.9.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{20}H_{22}N_2O_3$: 339.1709, found 339.1709.

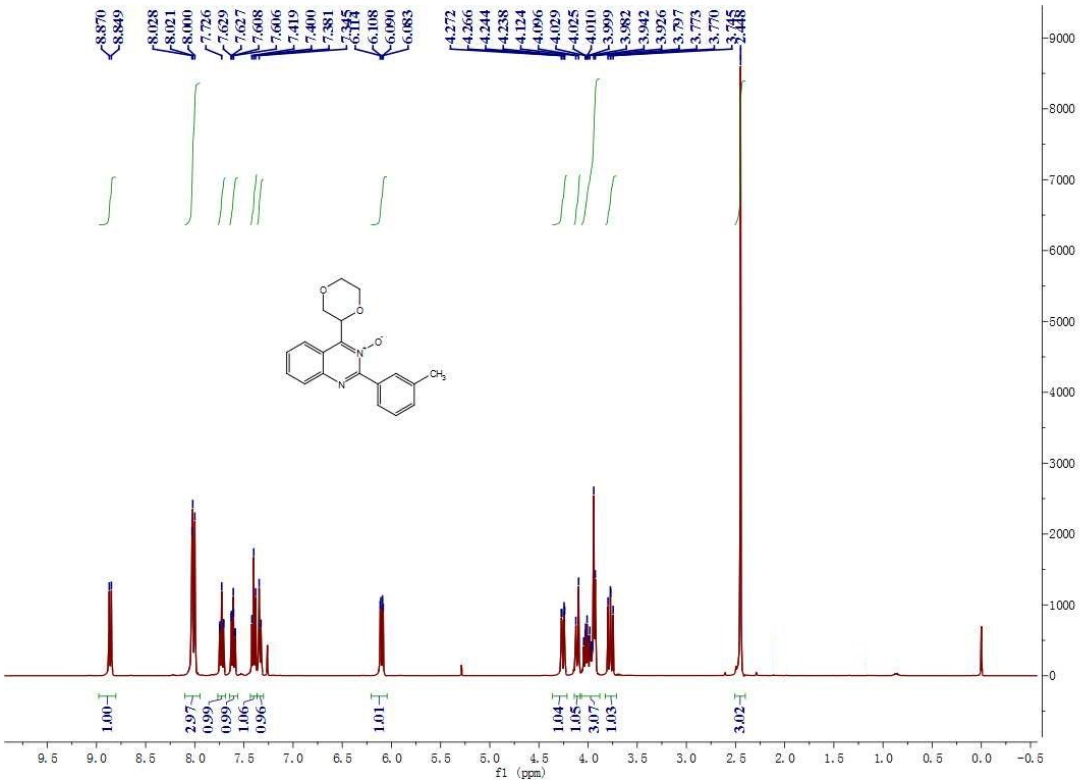


4-(1-ethoxyethyl)-2-(p-tolyl)quinazoline 3-oxide(3y). Compound was obtained as ayellow solid: yield 91%;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.84 (d, *J* = 8.8 Hz, 1H), 8.23 (d, *J* = 8.4 Hz, 2H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.77 - 7.66 (m, 1H), 7.60-7.57 (m, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 5.89 (q, *J* = 6.8 Hz, 1H), 3.60-3.53 (m, 1H), 3.48-3.31 (m, 1H), 2.43 (s, 3H), 1.72 (d, *J* = 6.8 Hz, 3H), 1.21 (t, *J* = 7.0 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 155.3, 154.5, 141.2, 140.8, 131.0, 130.5, 129.5, 129.2, 128.6, 128.6, 123.5, 122.1, 73.1, 65.7, 21.6, 19.2, 15.3.HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{19}H_{20}N_2O_2$: 309.1603, found 309.1603.

^{1}H and $^{13}CNMR$ spectraof compounds



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3.07
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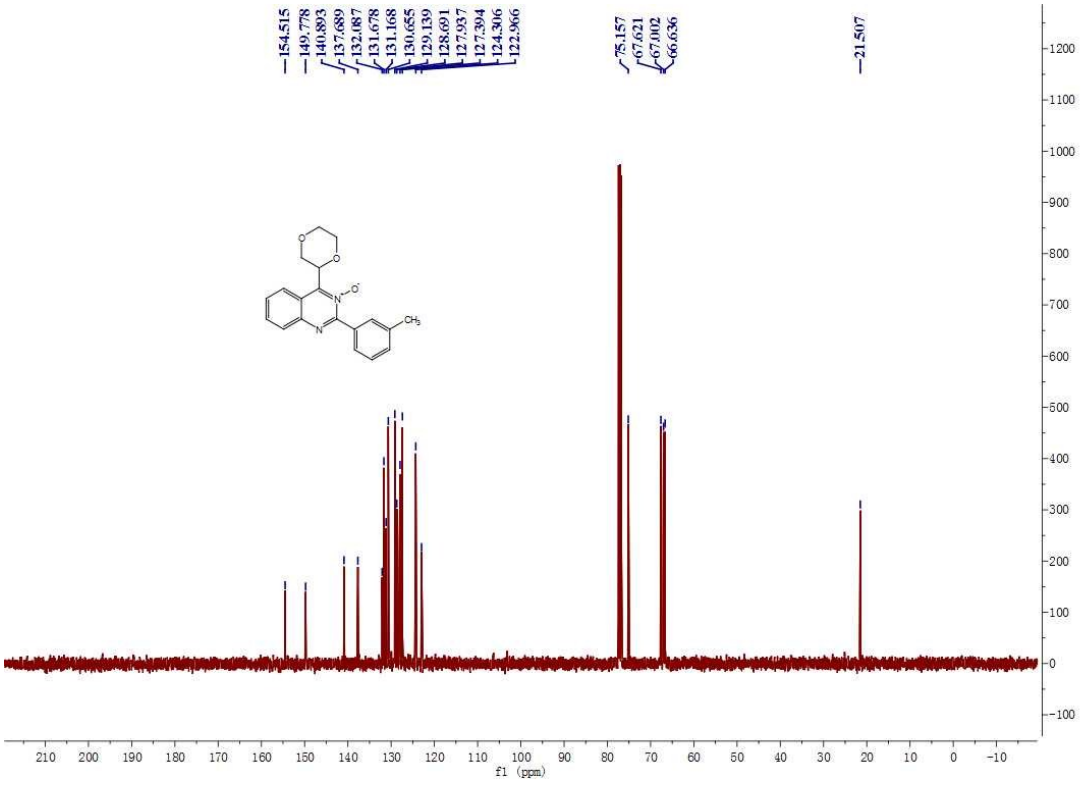
3.02
f1 (ppm)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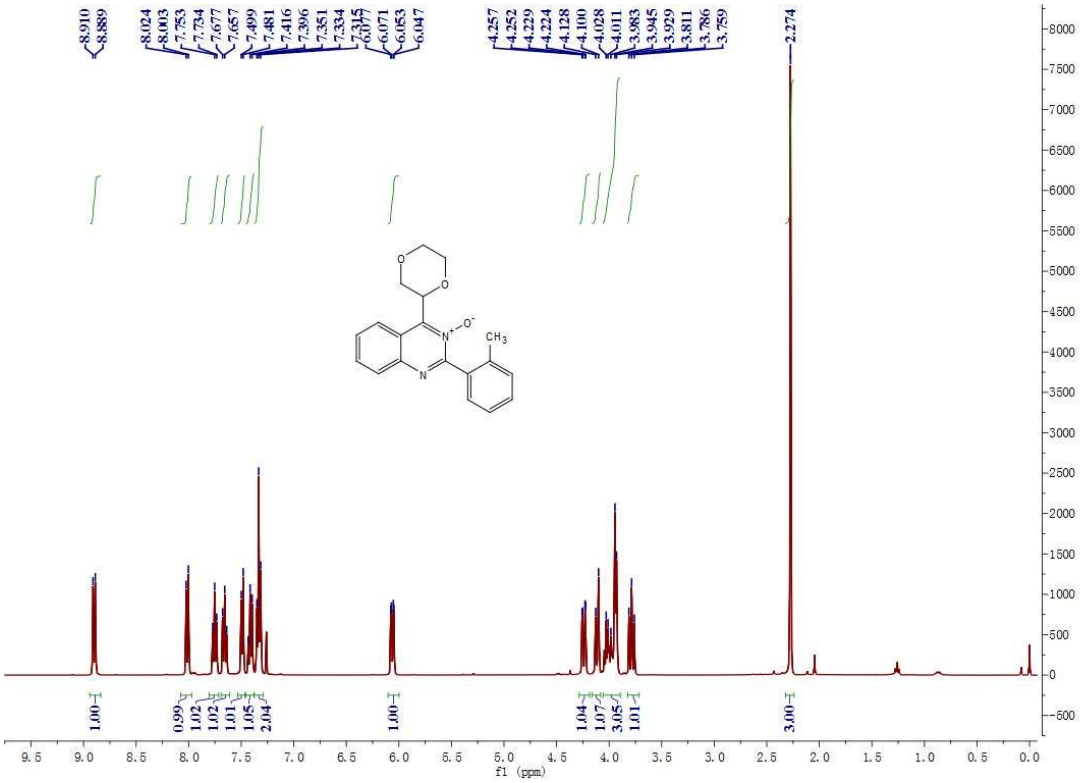
f1 (ppm)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3.945
3.929
3.811
3.786
3.759
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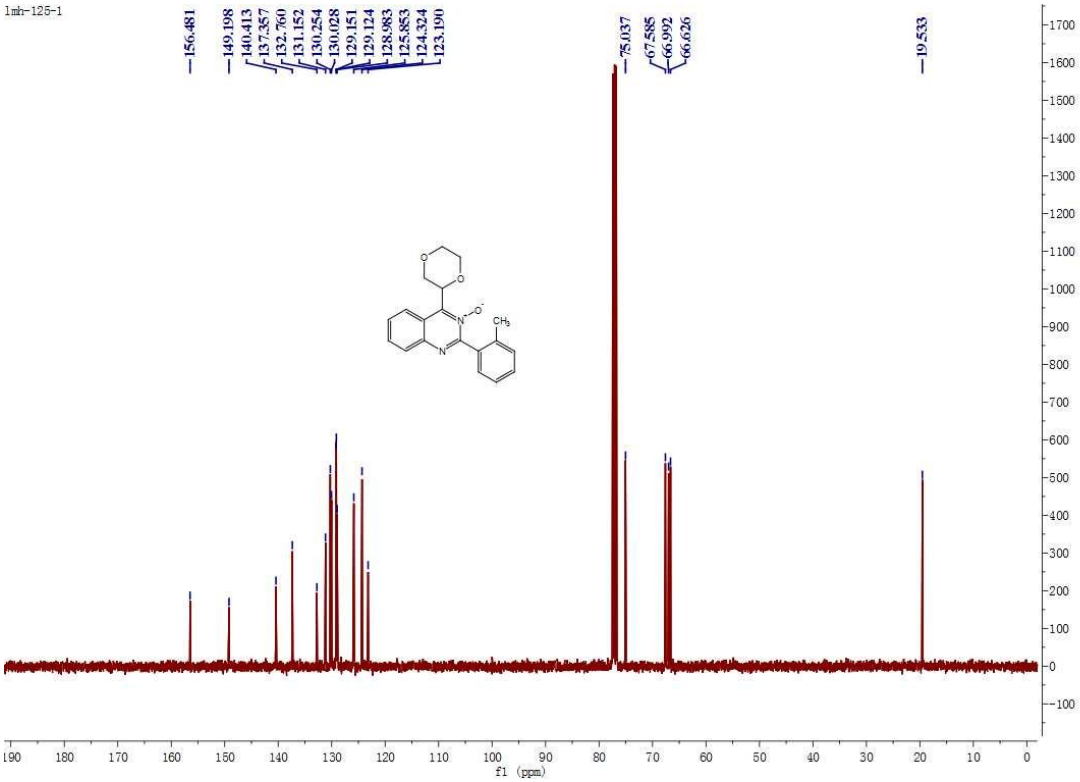
0.99
1.02
1.02
1.01
1.05
2.04
1.00
1.04
1.07
3.05
1.01
3.00
f1 (ppm)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137.357
132.760
131.152
130.254
130.028
129.151
129.124
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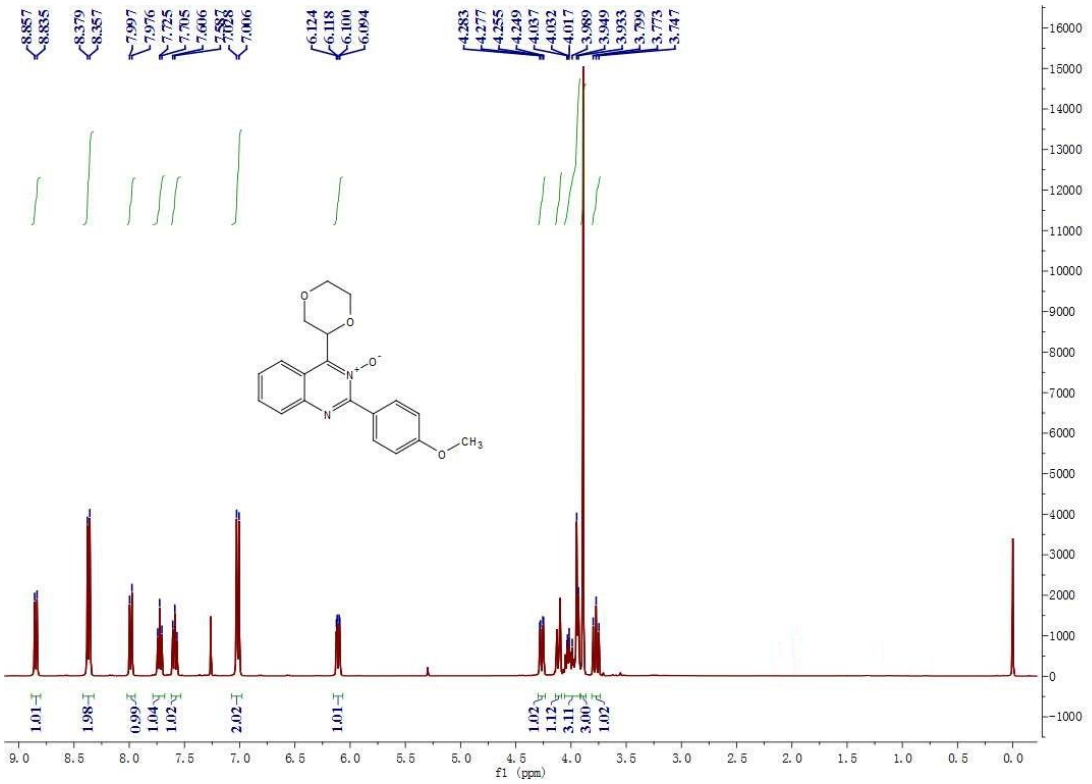
125.853
124.324
123.190
75.037
67.585
66.992
66.626
19.533
f1 (ppm)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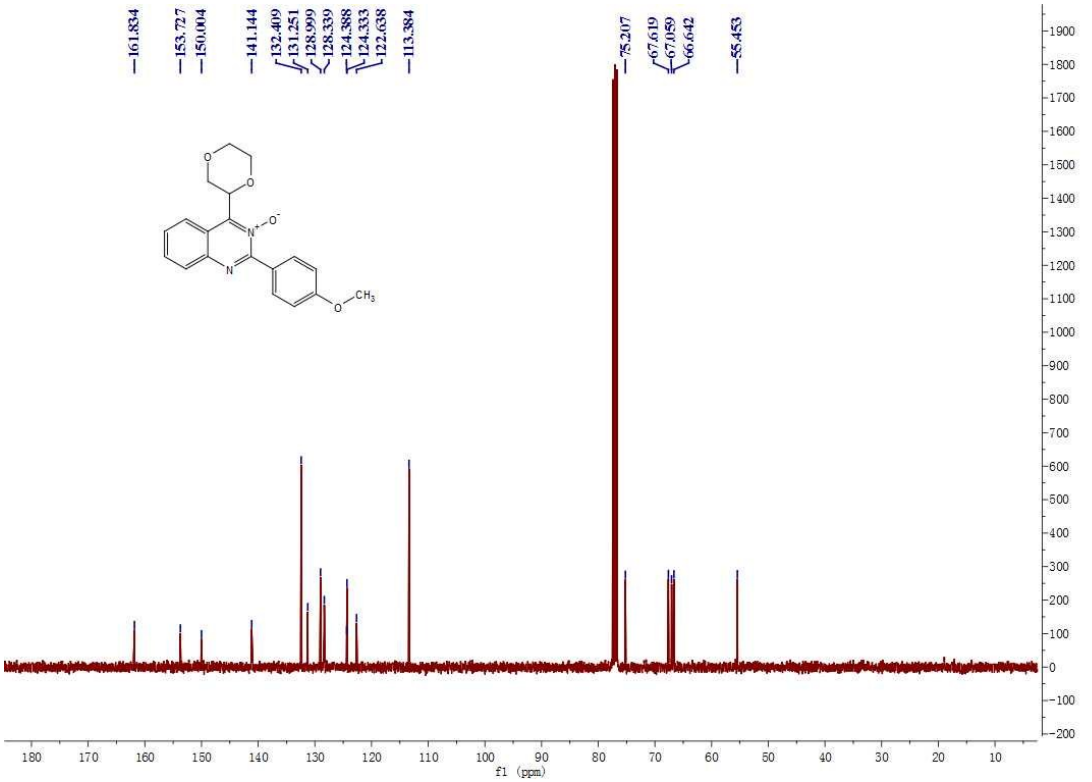
8.379
8.357
7.997
7.976
7.725
7.705
7.606
7.028
7.006
6.124
6.118
6.100
6.094
4.283
4.277
4.255
4.249
4.037
4.032
4.017
3.989
3.949
3.933
3.799
3.773
3.747
1.01
1.98
0.99
1.04
1.02
2.02
1.01
1.02
1.12
3.11
3.00
1.02
f1 (ppm)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131.251
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128.339
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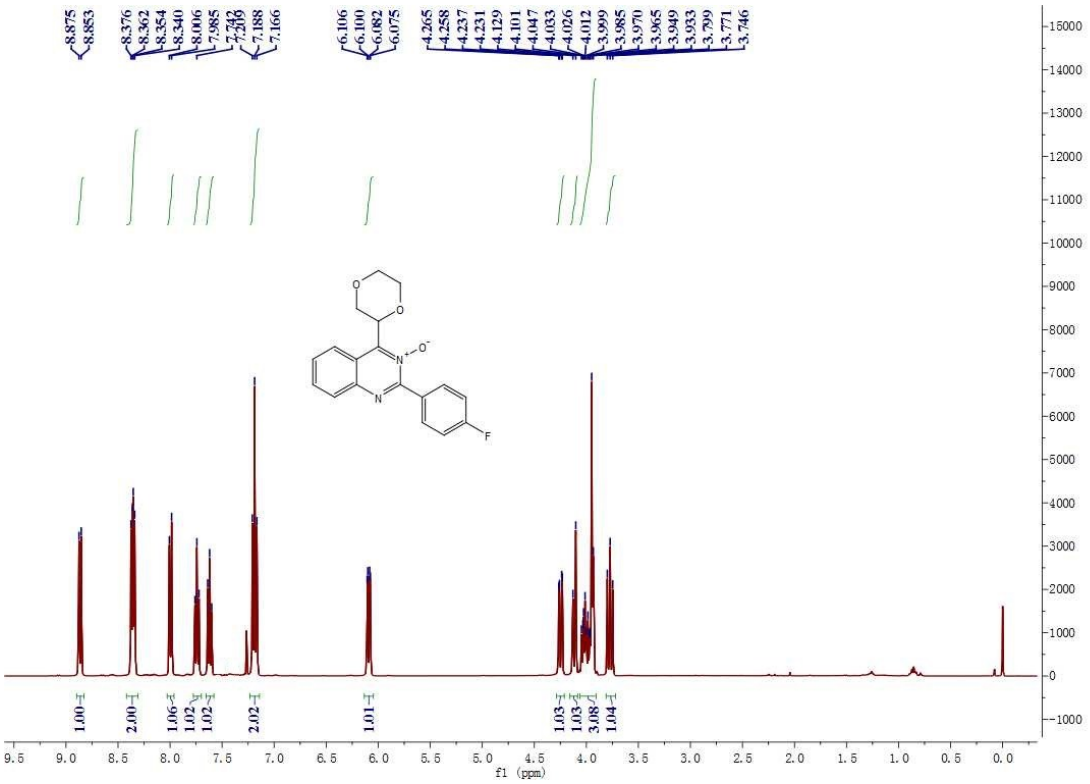
124.333
122.638
113.384
75.207
67.619
67.059
66.642
55.453
f1 (ppm)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8.006
7.985
7.240
7.209
7.188
7.166
6.106
6.100
6.082
6.075
4.265
4.258
4.237
4.231
4.129
4.101
4.047
4.033
4.026
4.012
3.999
3.985
3.970
3.965
3.949
3.933
3.799
3.771
3.746
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2.00
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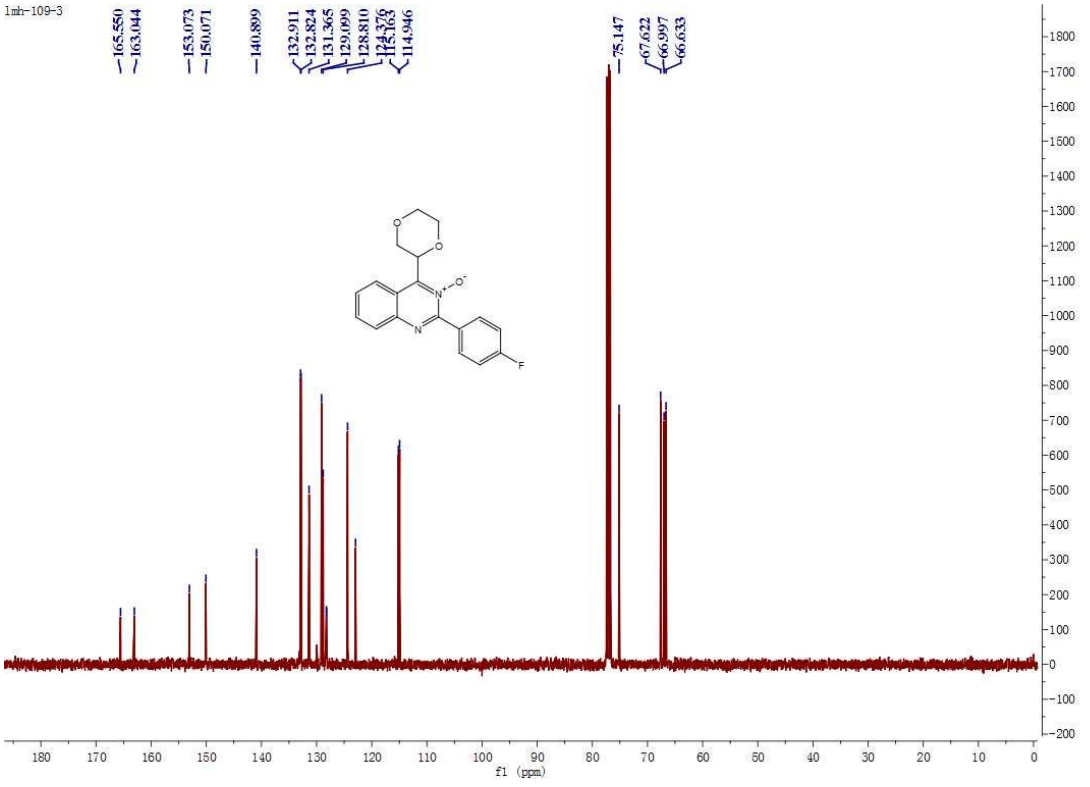
1.02
1.02
2.02
1.01
1.03
1.03
3.08
1.04
f1 (ppm)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132.824
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128.810
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124.363
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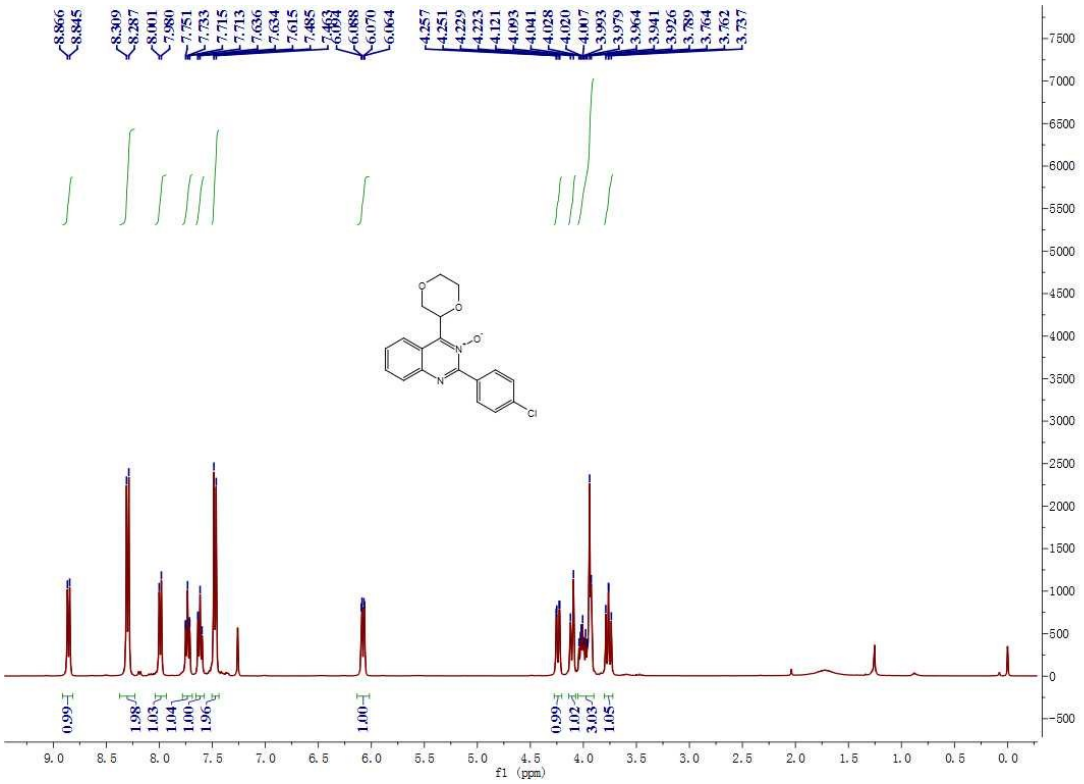
75.147
67.622
66.997
66.633
f1 (ppm)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7.733
7.715
7.713
7.636
7.634
7.615
7.485
6.088
6.070
6.064
4.257
4.251
4.229
4.223
4.121
4.093
4.041
4.028
4.020
4.007
3.993
3.979
3.964
3.941
3.926
3.789
3.764
3.762
3.737
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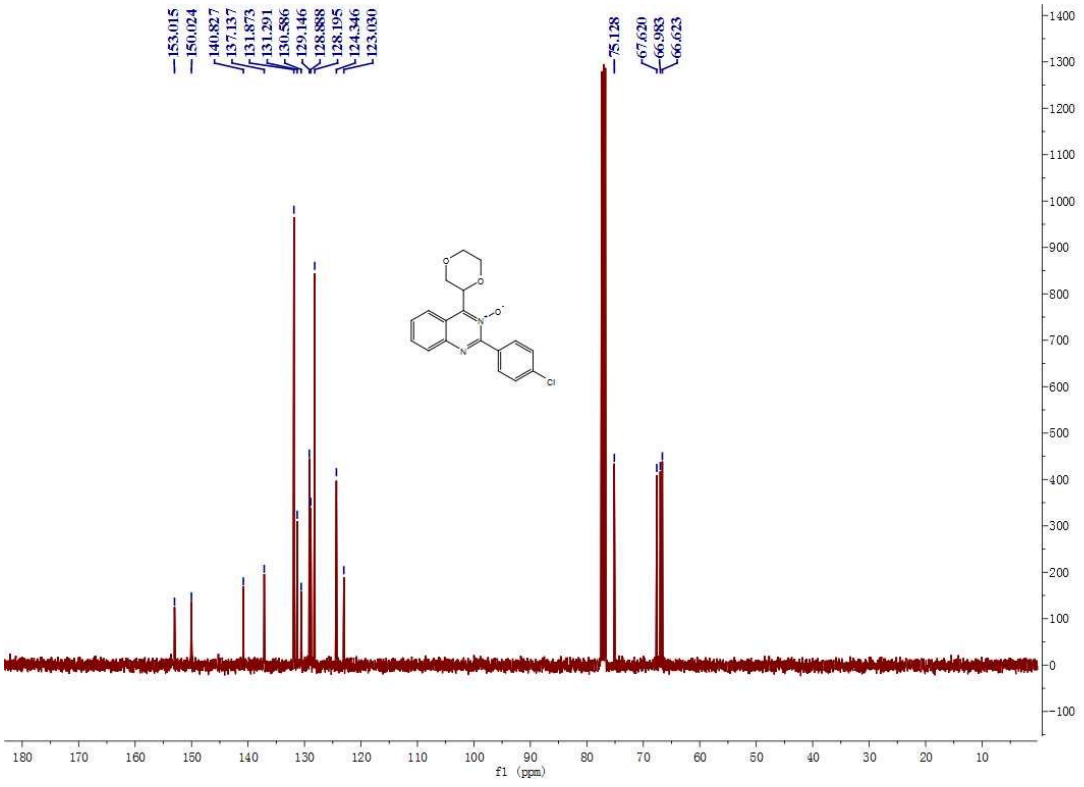
1.98
1.03
1.04
1.00
1.96
1.00
0.99
1.02
3.03
1.05
f1 (ppm)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128.195
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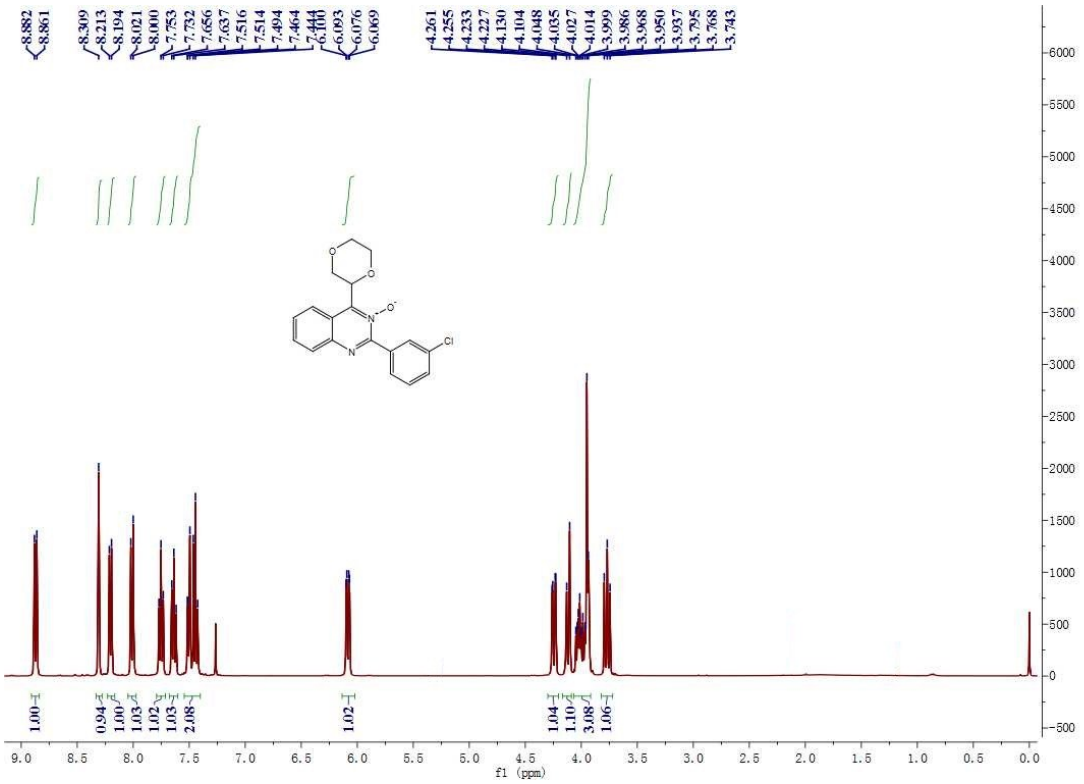
67.620
66.983
66.623
f1 (ppm)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8.861
8.309
8.213
8.194
8.021
8.000
7.753
7.732
7.656
7.637
7.516
7.514
7.494
7.464
6.093
6.076
6.069
4.261
4.255
4.233
4.227
4.130
4.104
4.048
4.035
4.027
4.014
3.999
3.986
3.968
3.950
3.937
3.795
3.768
3.743
1.00
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1.00
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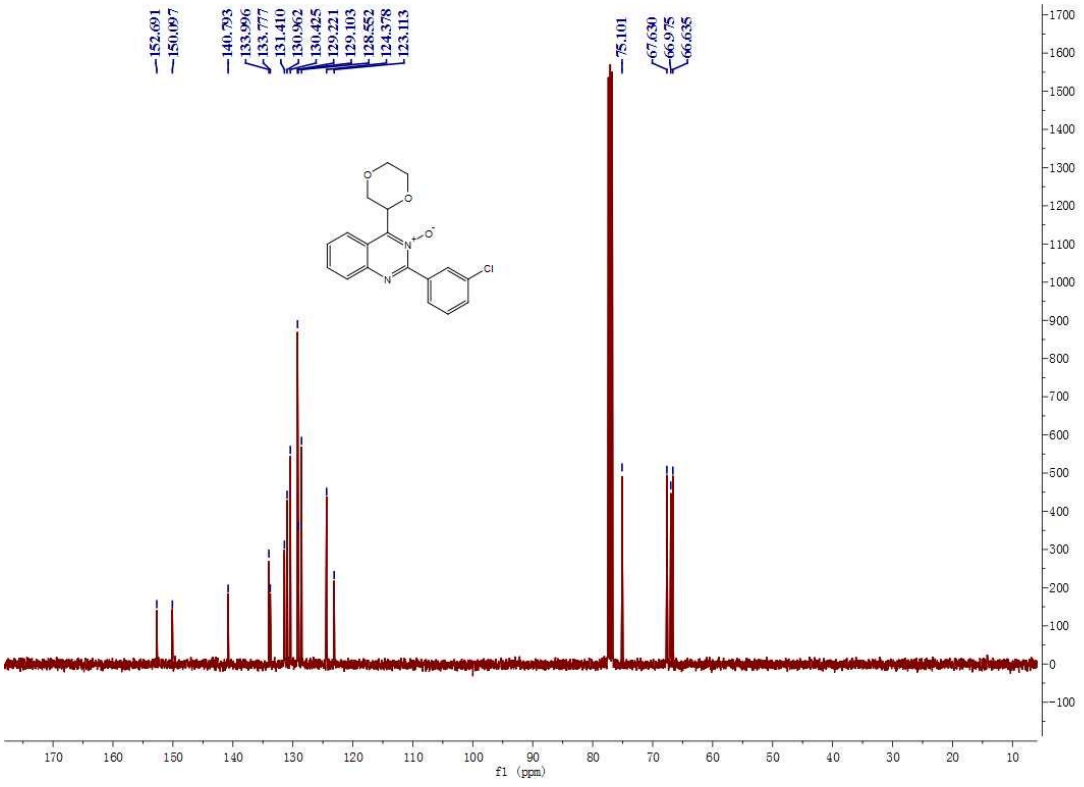
1.02
1.03
2.08
1.02
1.04
1.10
3.08
1.06
f1 (ppm)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140.793
133.996
133.777
131.410
130.962
130.425
129.221
129.103
128.552
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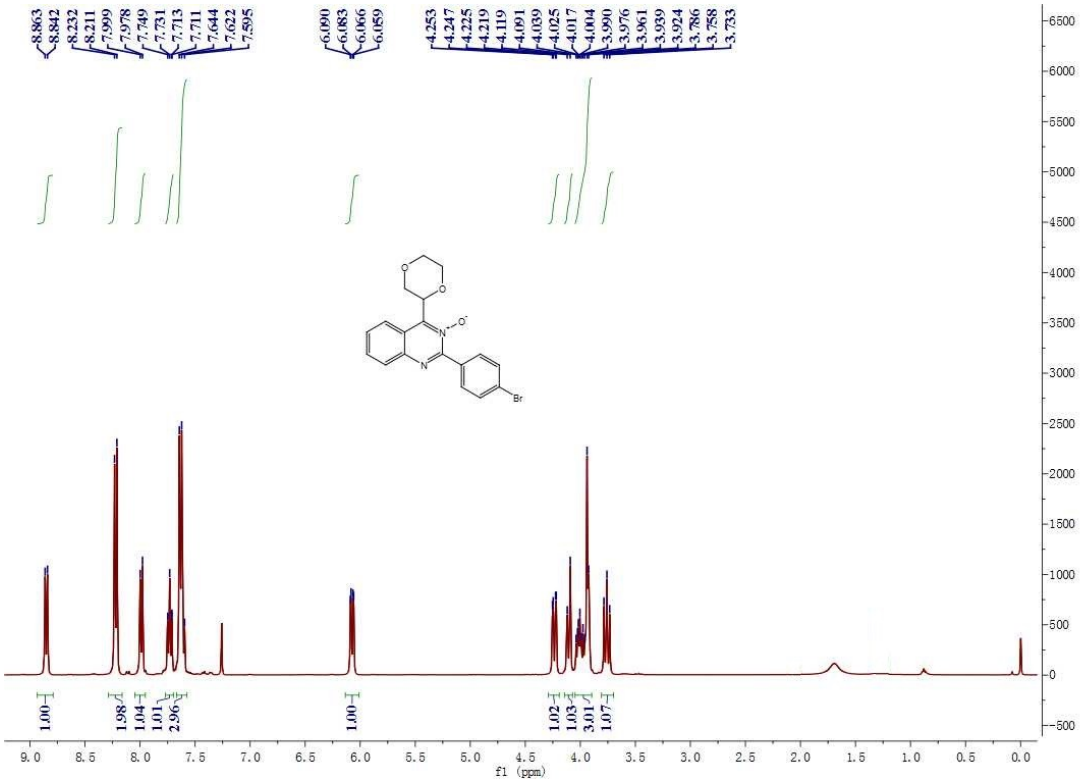
75.101
67.630
66.975
66.635
f1 (ppm)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8.232
8.211
7.999
7.978
7.749
7.731
7.713
7.711
7.644
7.622
7.595
6.090
6.083
6.066
6.059
4.253
4.247
4.225
4.219
4.119
4.091
4.039
4.025
4.017
4.004
3.990
3.976
3.961
3.939
3.924
3.786
3.758
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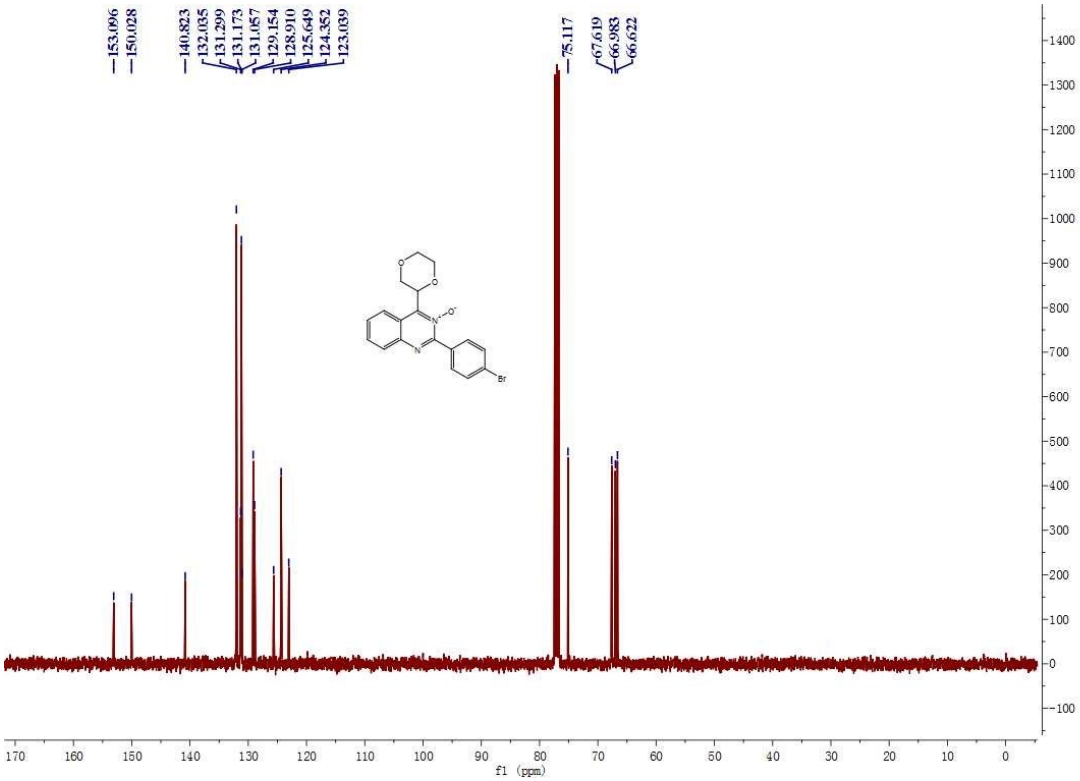
1.02
1.03
3.01
1.07
f1 (ppm)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150.028
140.823
132.035
131.299
131.173
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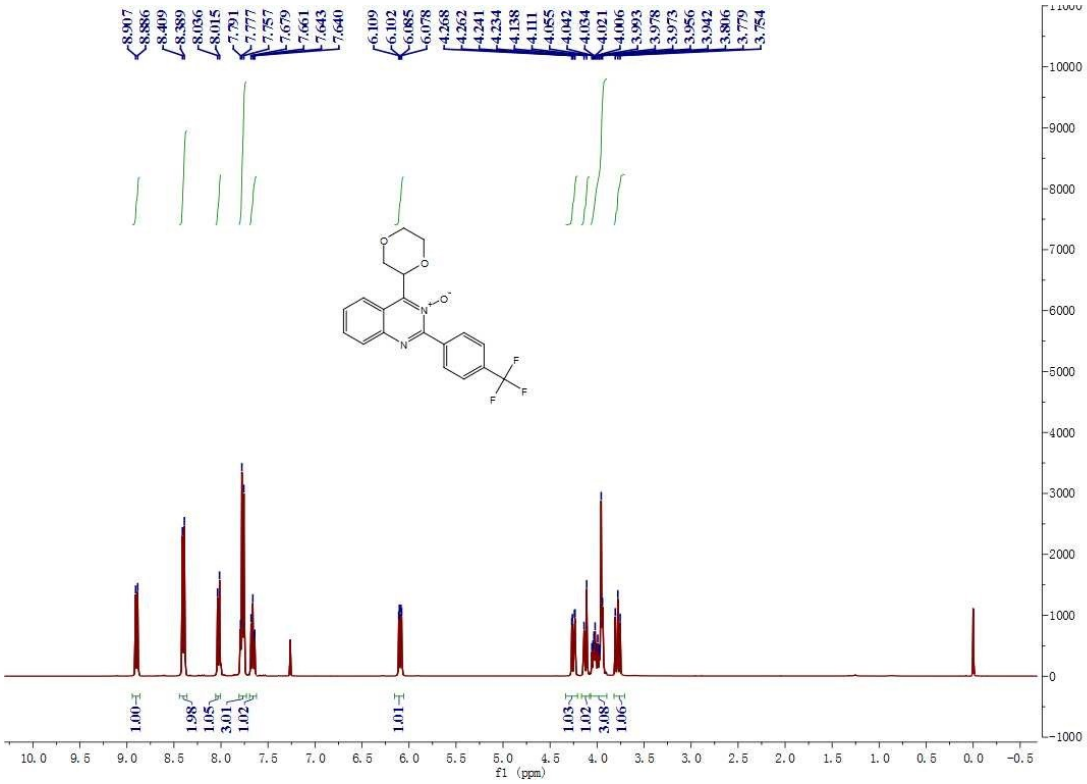
129.154
128.910
125.649
124.352
123.039
75.117
67.619
66.983
66.622
f1 (ppm)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8.015
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7.777
7.757
7.679
7.661
7.643
7.640
6.109
6.102
6.085
6.078
4.268
4.262
4.241
4.234
4.138
4.111
4.055
4.042
4.034
4.021
4.006
3.993
3.978
3.973
3.956
3.942
3.806
3.779
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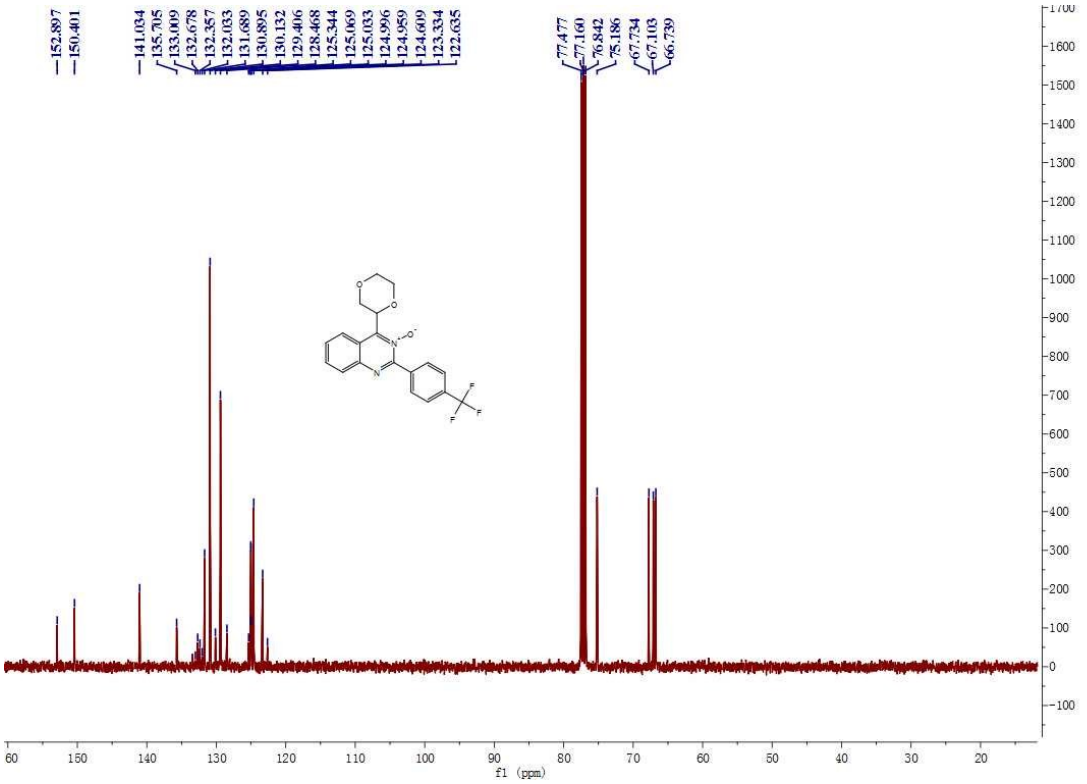
1.05
3.01
1.02
1.01
1.03
1.02
3.08
1.06
f1 (ppm)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129.406
128.468
125.344
125.069
125.033
124.996
124.959
124.609
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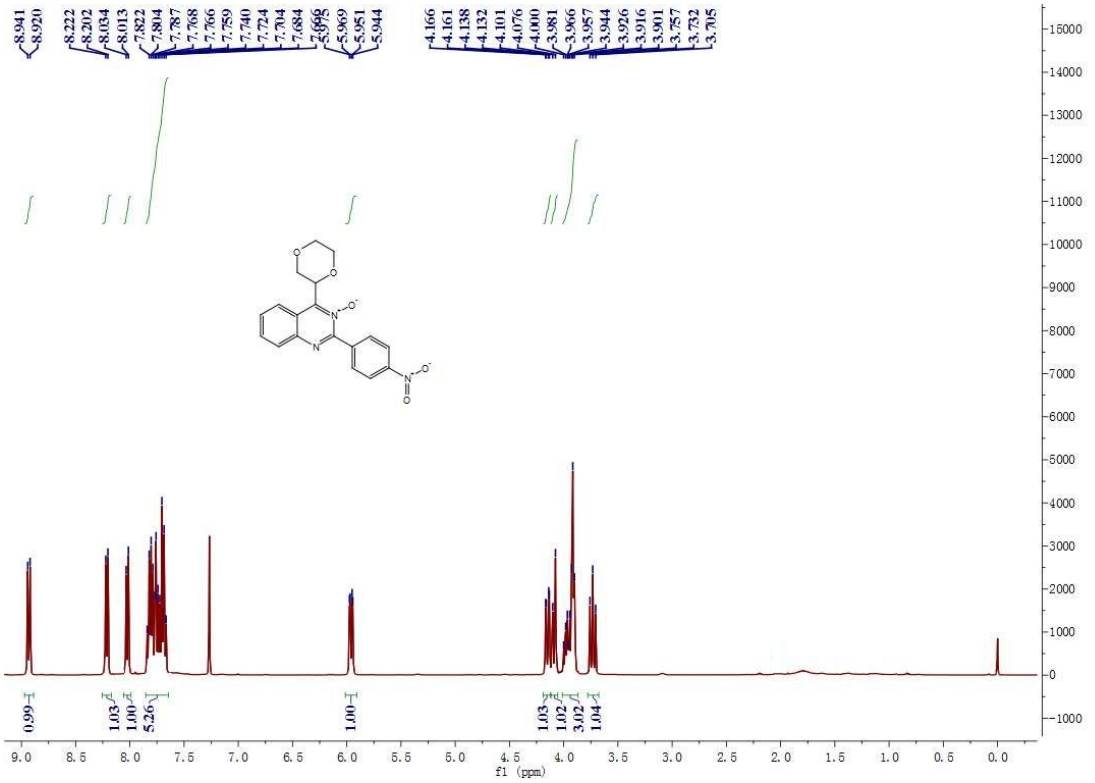
122.635
77.477
77.160
76.842
75.186
67.734
67.103
66.739
f1 (ppm)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8.013
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7.804
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7.768
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7.740
7.724
7.704
7.684
5.969
5.951
5.944
4.166
4.161
4.138
4.132
4.101
4.076
4.000
3.981
3.966
3.957
3.944
3.926
3.916
3.901
3.757
3.732
3.705
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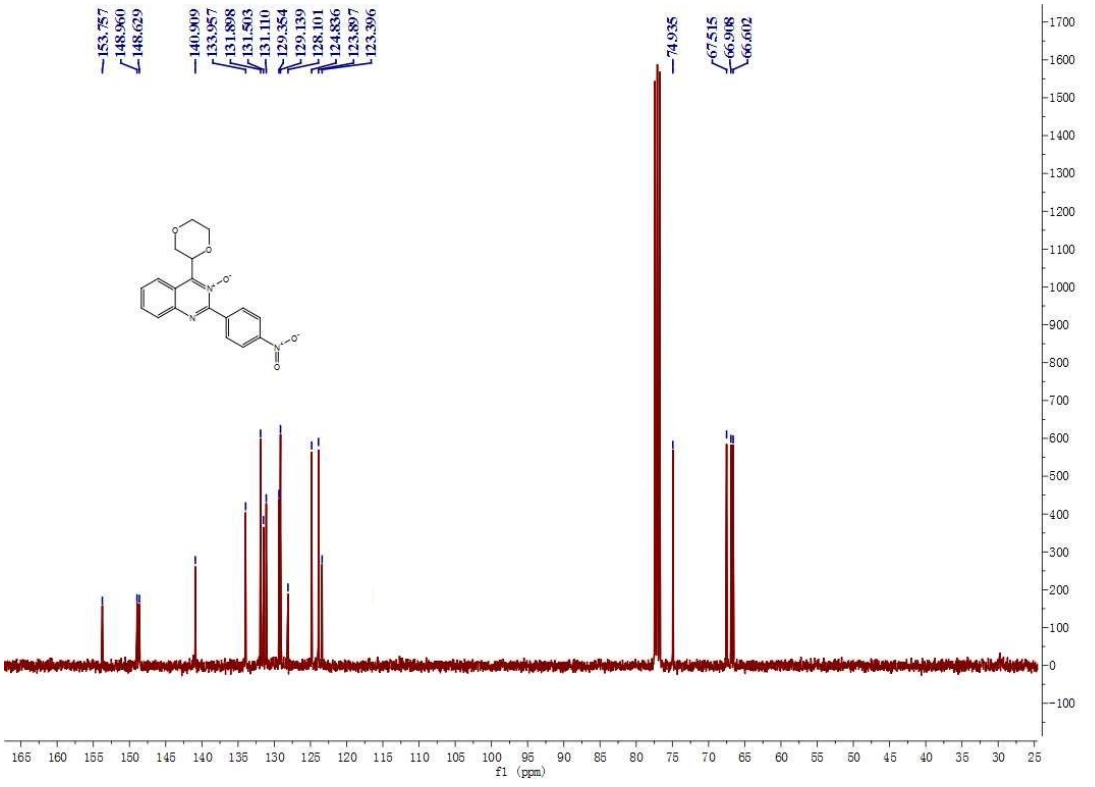
1.00
5.26
1.00
1.03
1.02
3.02
1.04
f1 (ppm)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131.110
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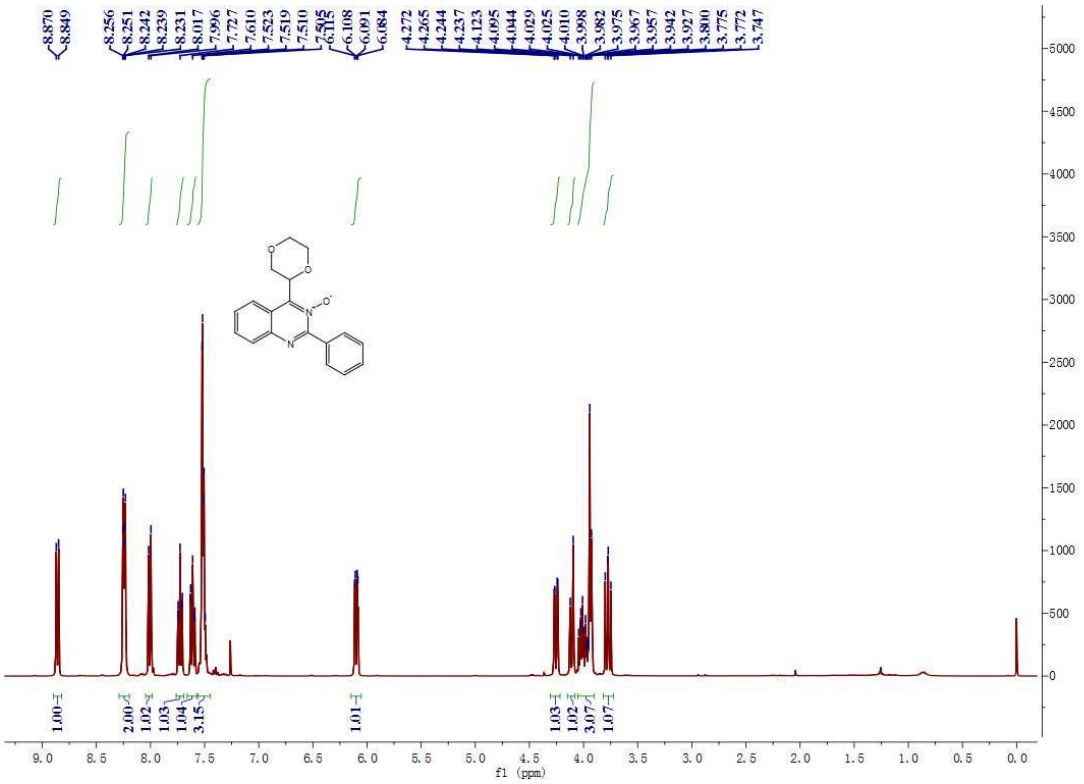
66.908
66.602
f1 (ppm)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8.251
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8.239
8.231
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7.727
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7.519
7.510
7.505
6.115
6.108
6.091
6.084
4.272
4.265
4.244
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4.029
4.025
4.010
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3.982
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3.957
3.942
3.927
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3.775
3.772
3.747
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1.04
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1.02
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1.07
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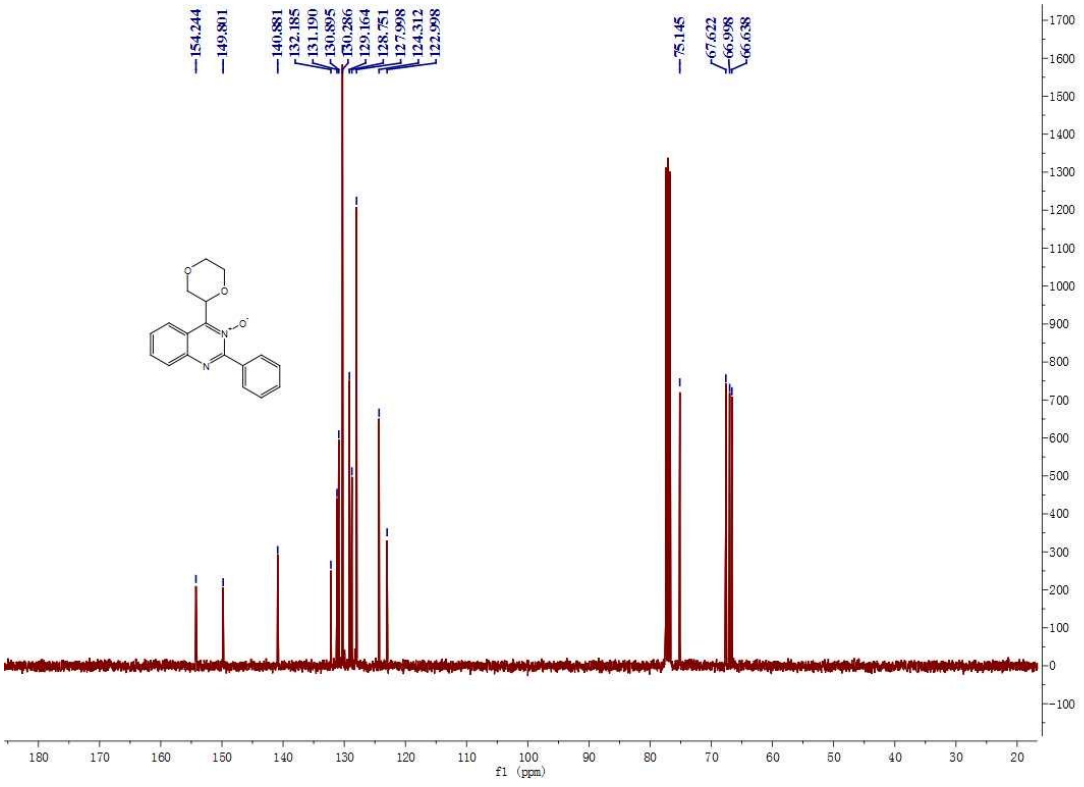
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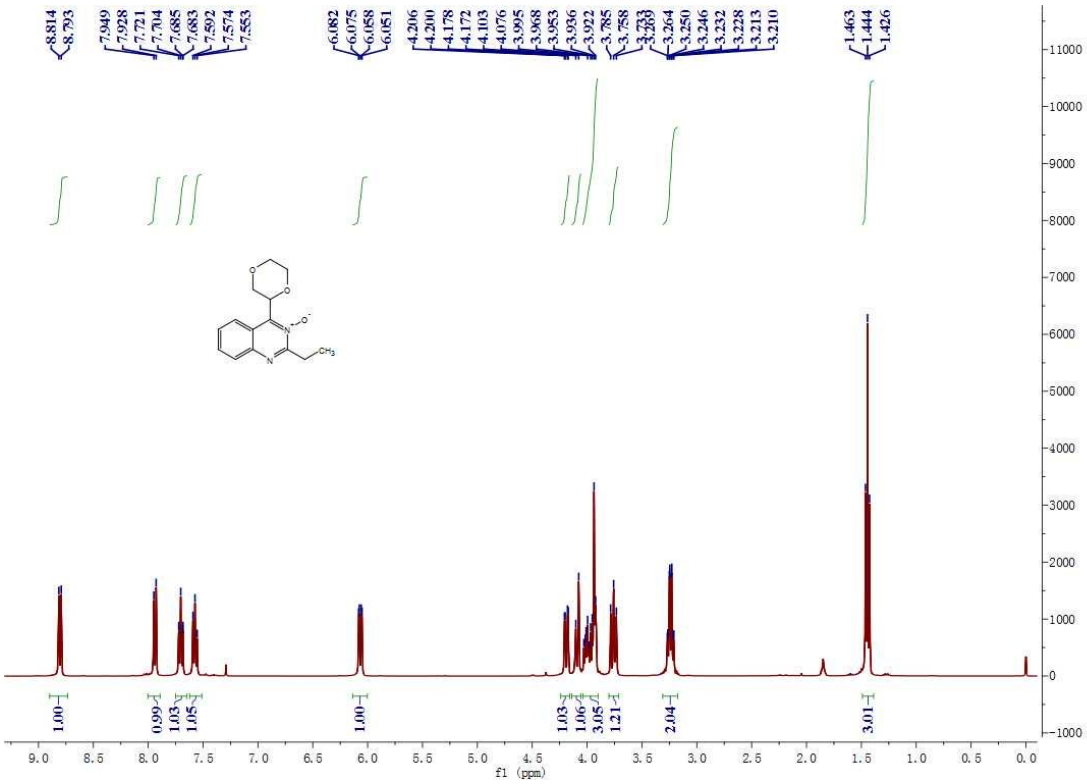
66.638
f1 (ppm)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6.075
6.058
6.051
4.206
4.200
4.178
4.172
4.103
4.076
3.995
3.968
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3.785
3.758
3.733
3.703
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3.232
3.228
3.213
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1.03
1.06
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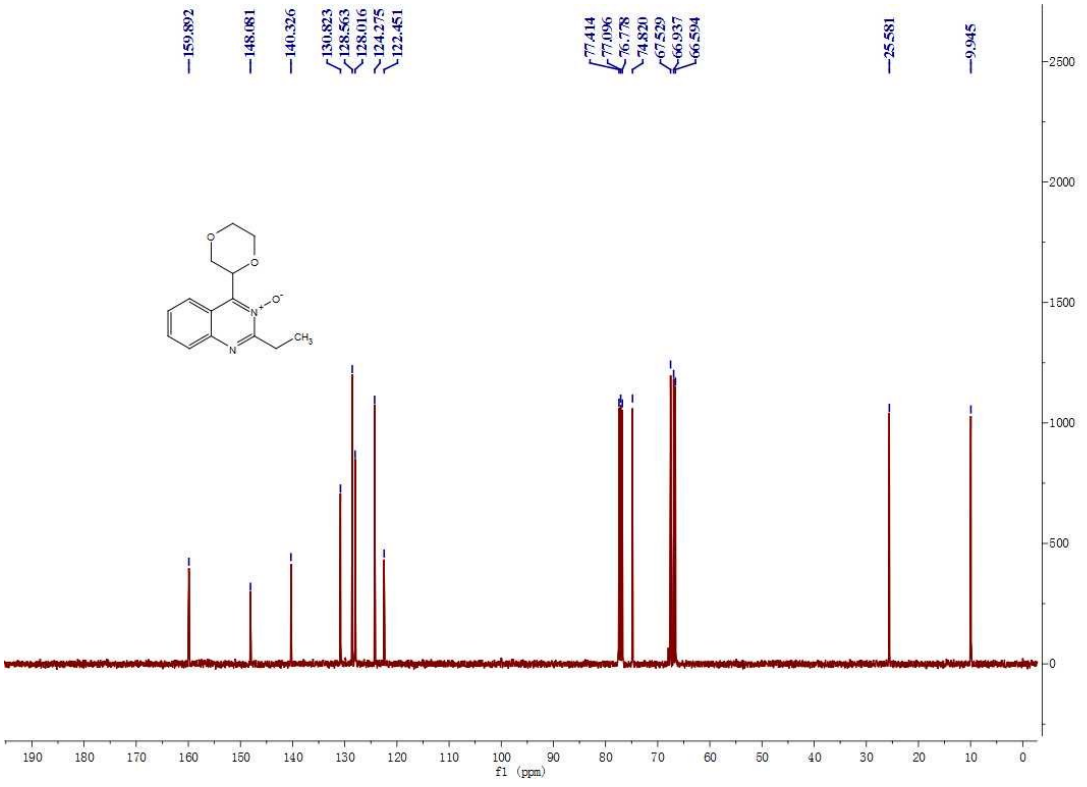
3.01
f1 (ppm)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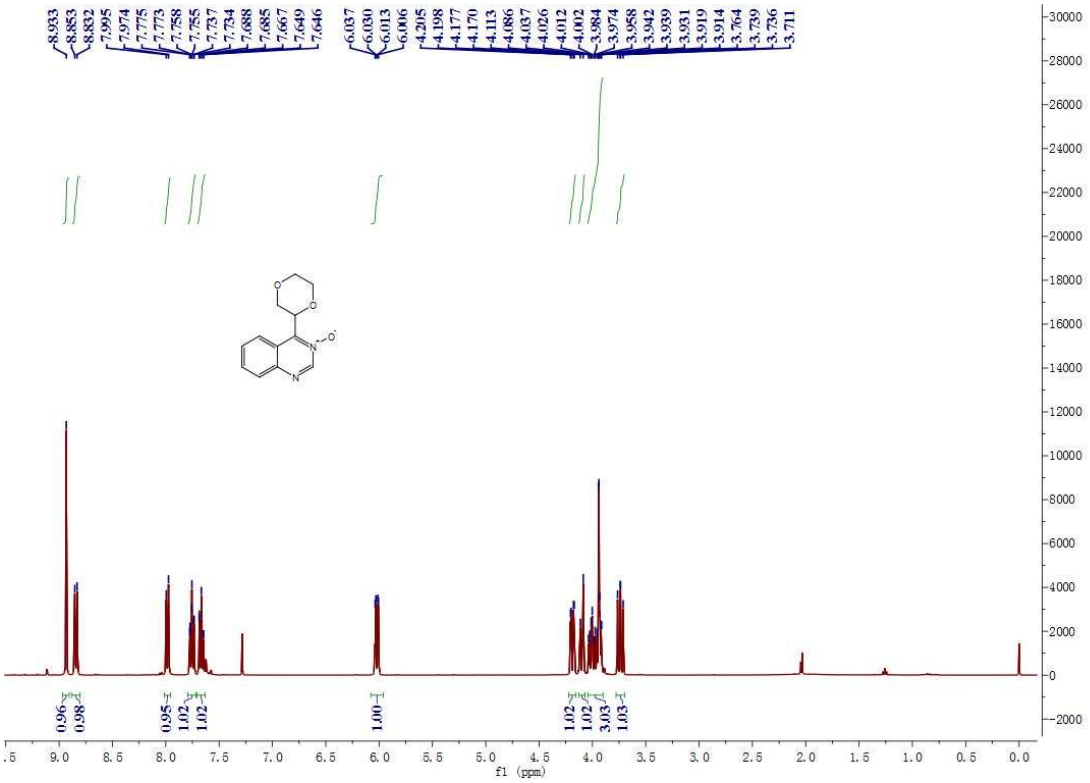
9.945
f1 (ppm)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7.758
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7.649
7.646
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6.030
6.013
6.006
4.205
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4.113
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4.012
4.002
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3.974
3.958
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3.939
3.931
3.919
3.914
3.764
3.739
3.736
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0.95
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1.02
1.00
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1.02
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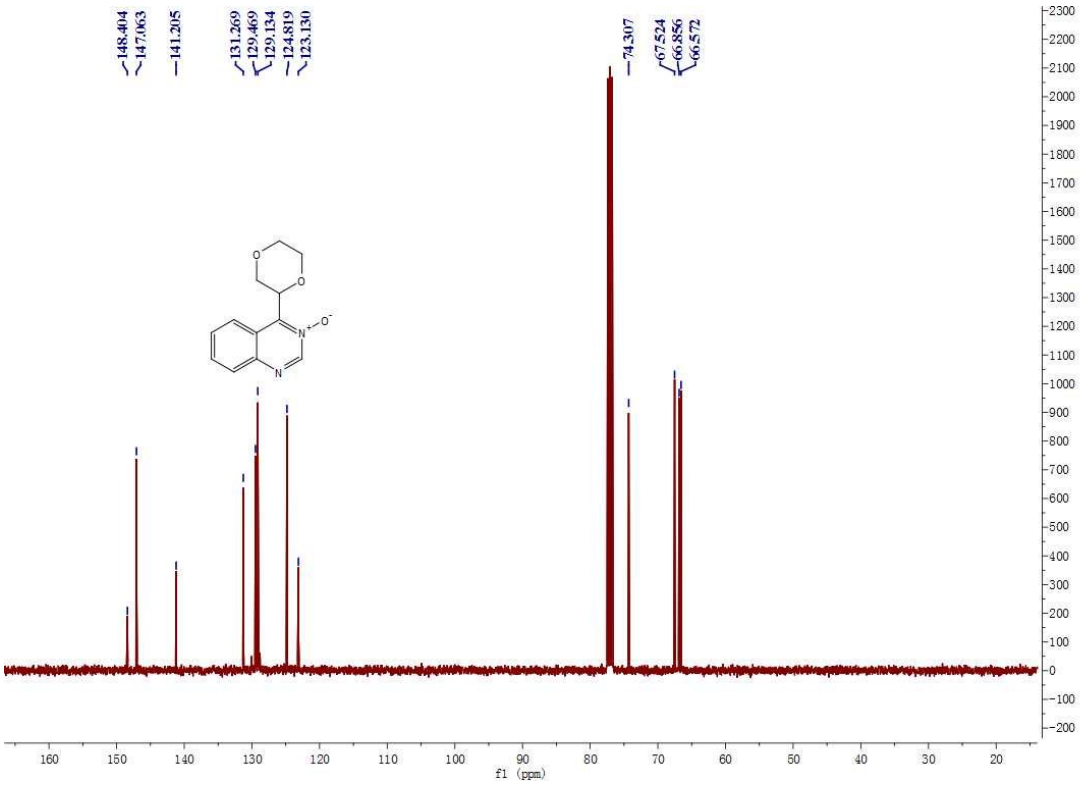
1.03
f1 (ppm)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123.130
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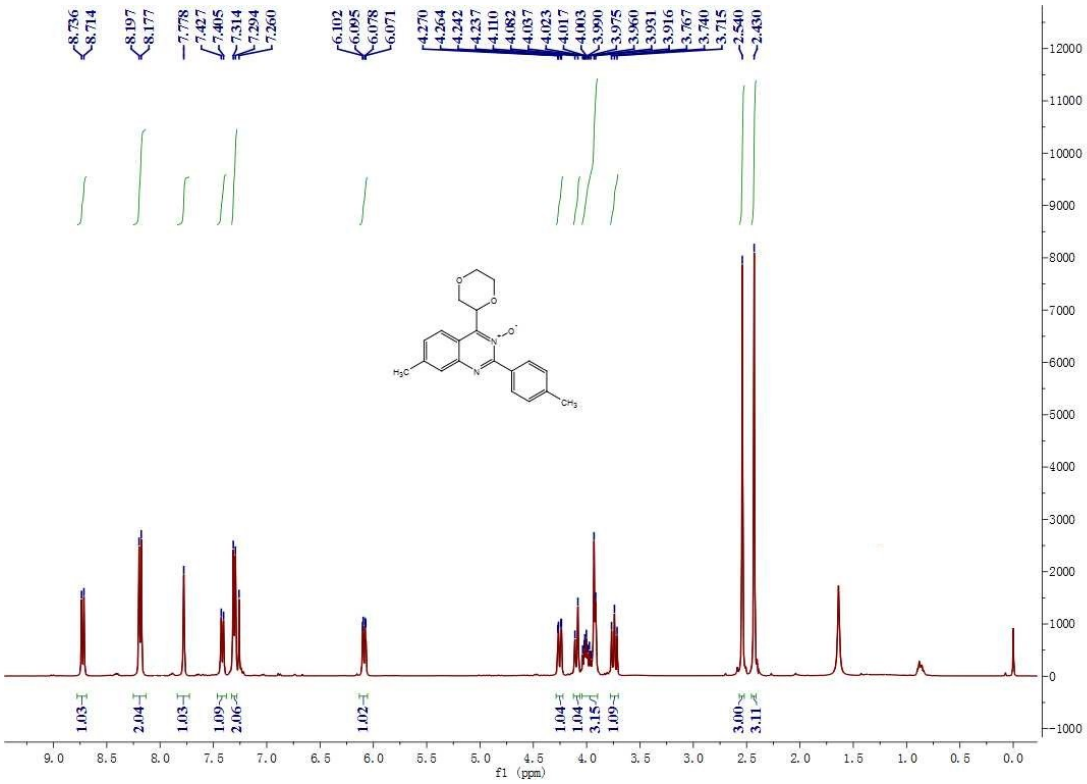
66.572
f1 (ppm)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6.071
4.270
4.264
4.242
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4.110
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2.430
1.03
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2.06
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1.04
1.04
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1.09
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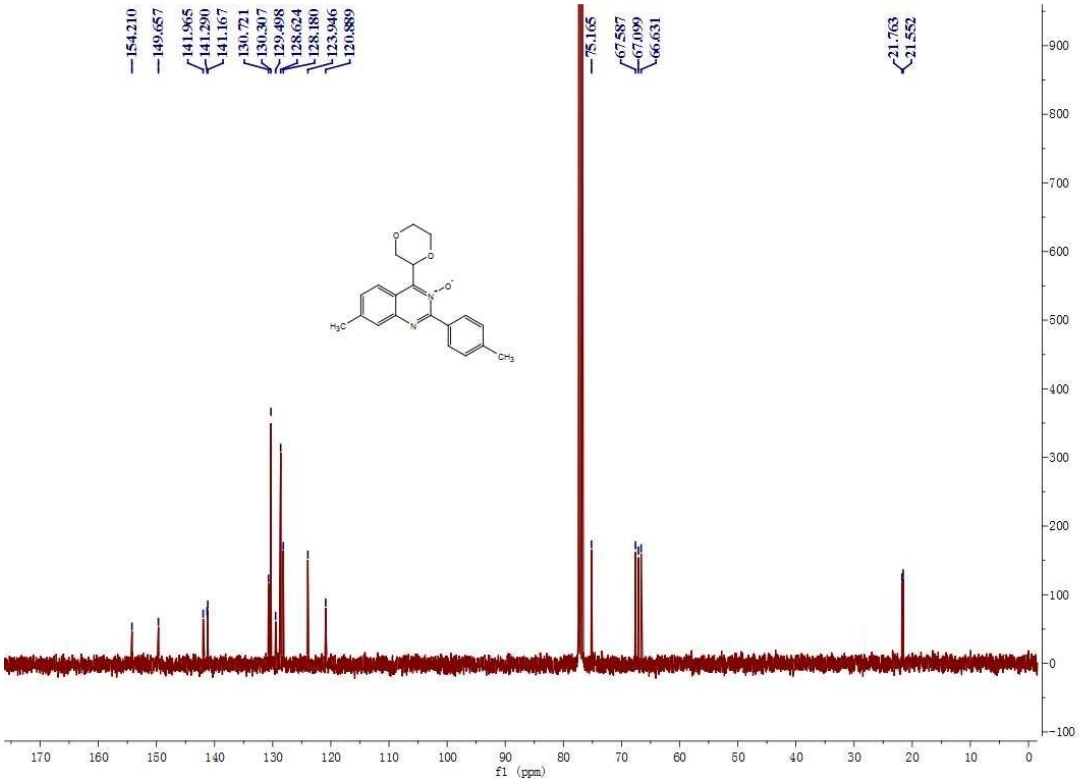
3.11
f1 (ppm)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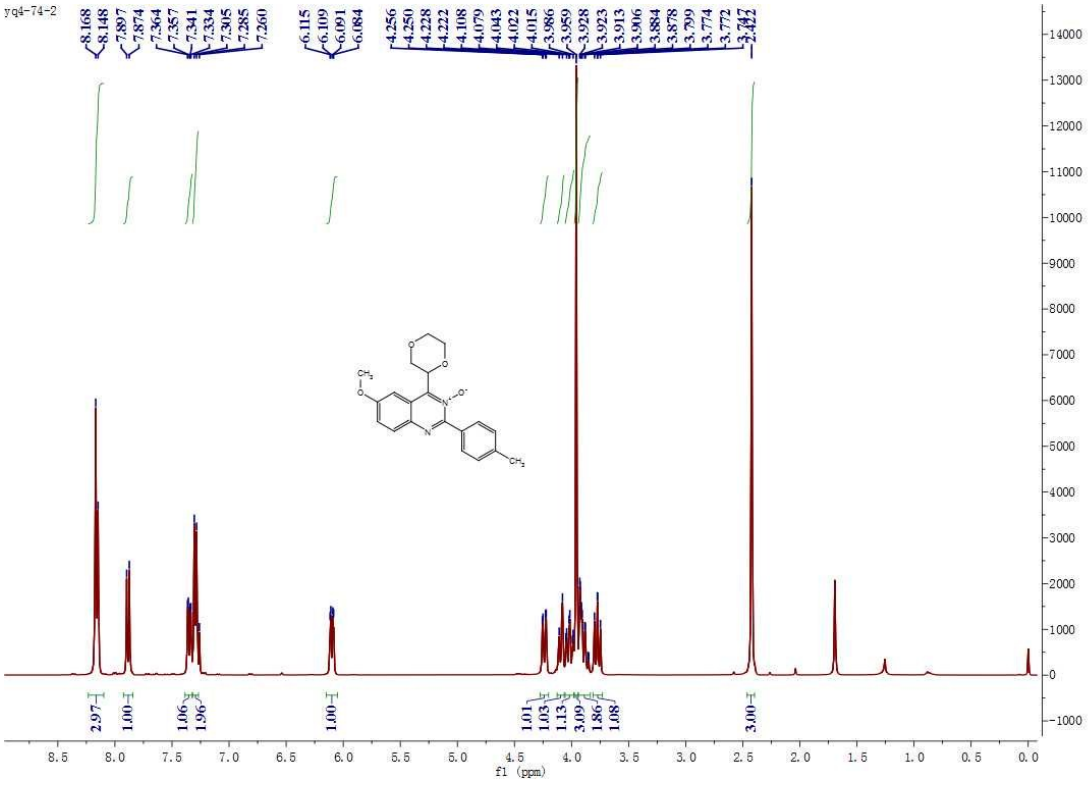
67.587
67.099
66.631
21.763
21.552
f1 (ppm)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6.109
6.091
6.084
4.256
4.250
4.228
4.222
4.108
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4.022
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3.923
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3.906
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3.774
3.772
2.442
2.97
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1.06
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1.00
1.01
1.03
1.13
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1.86
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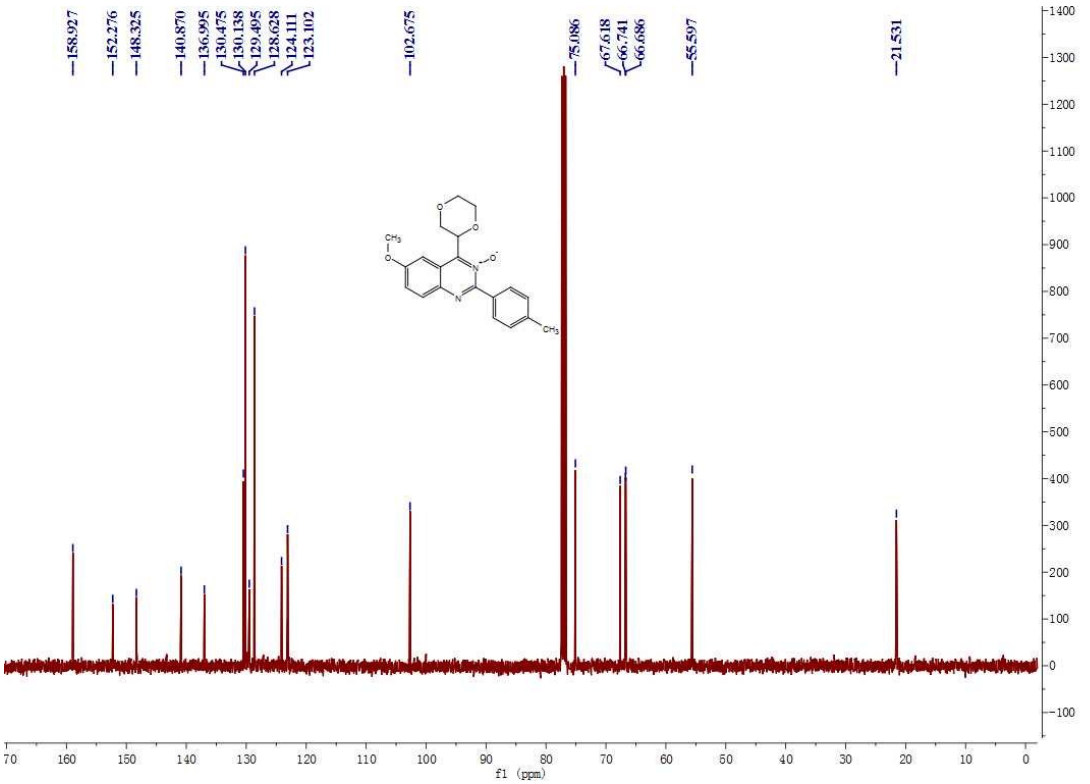
3.00
f1 (ppm)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130.138
129.495
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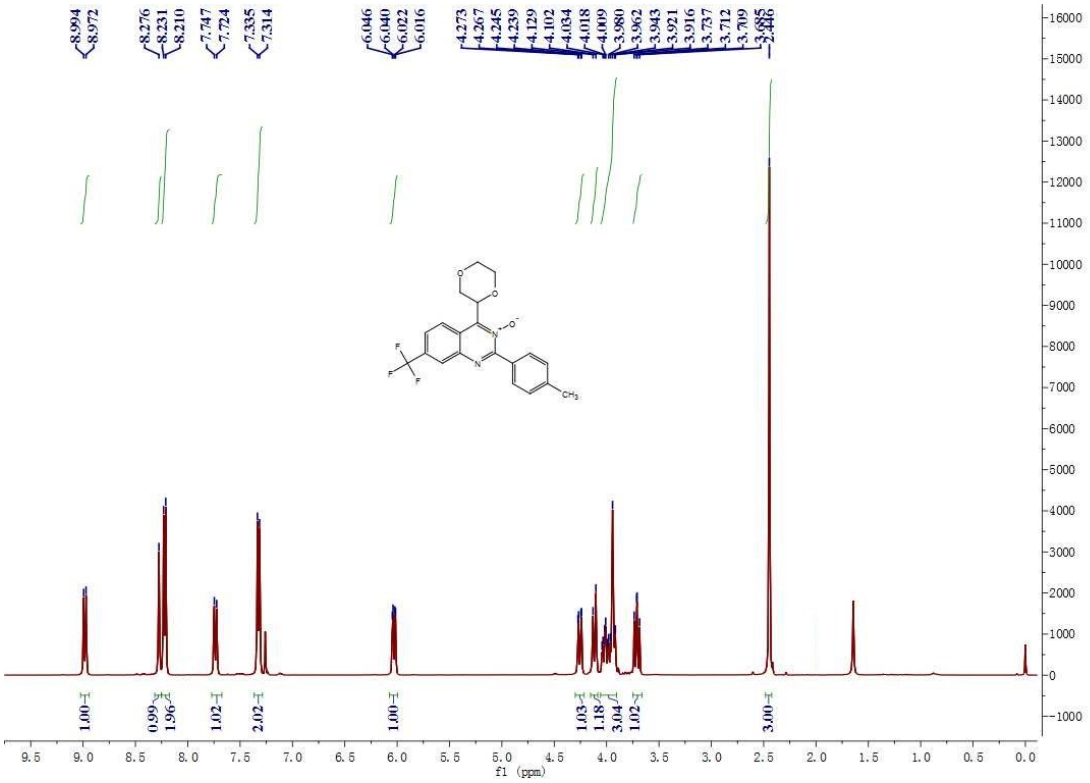
123.102
102.675
75.086
67.618
66.741
66.686
55.597
21.531
f1 (ppm)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8.276
8.231
8.210
7.747
7.724
7.335
7.314
6.046
6.040
6.022
6.016
4.273
4.267
4.245
4.239
4.129
4.102
4.034
4.018
4.009
3.980
3.962
3.943
3.921
3.916
3.737
3.712
3.709
3.685
2.446
1.00
0.99
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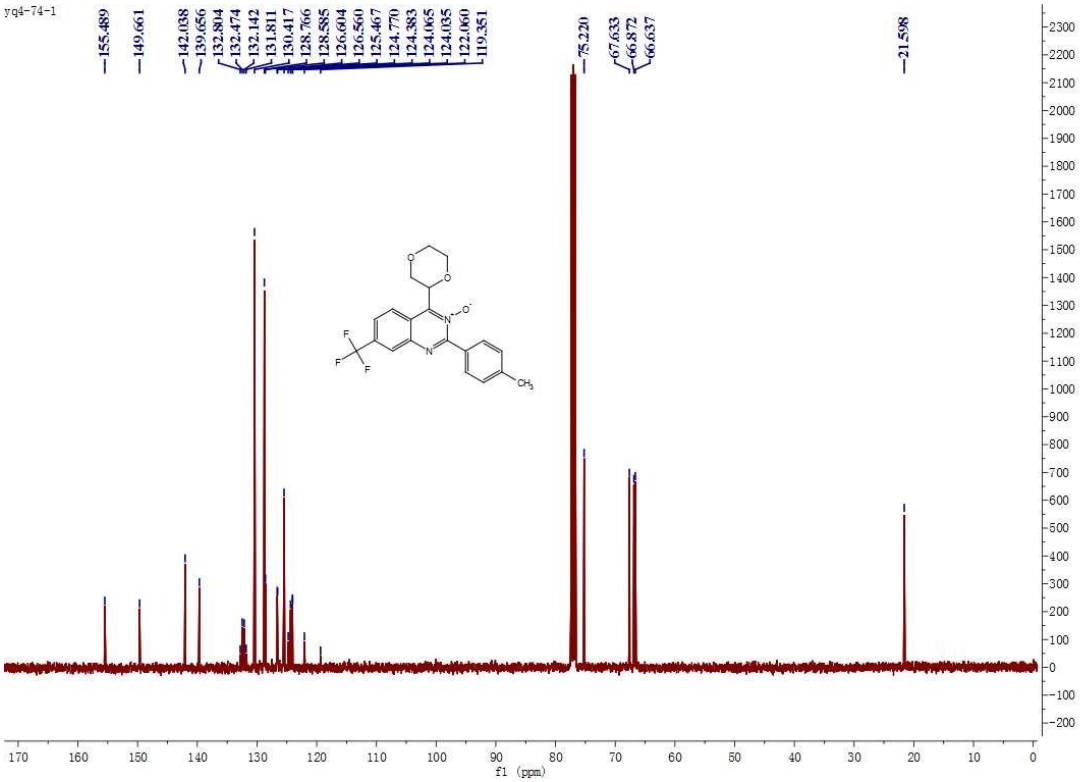
1.02
2.02
1.00
1.03
1.18
3.04
1.02
3.00
f1 (ppm)


yq4-74-1
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130.417
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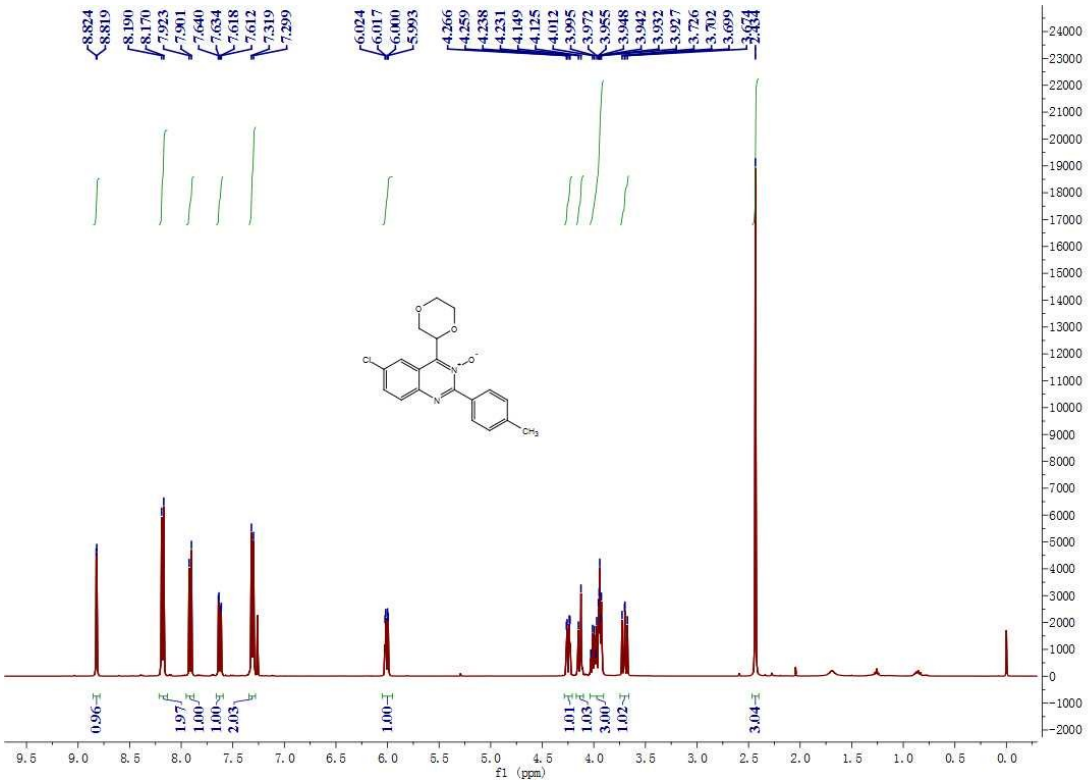
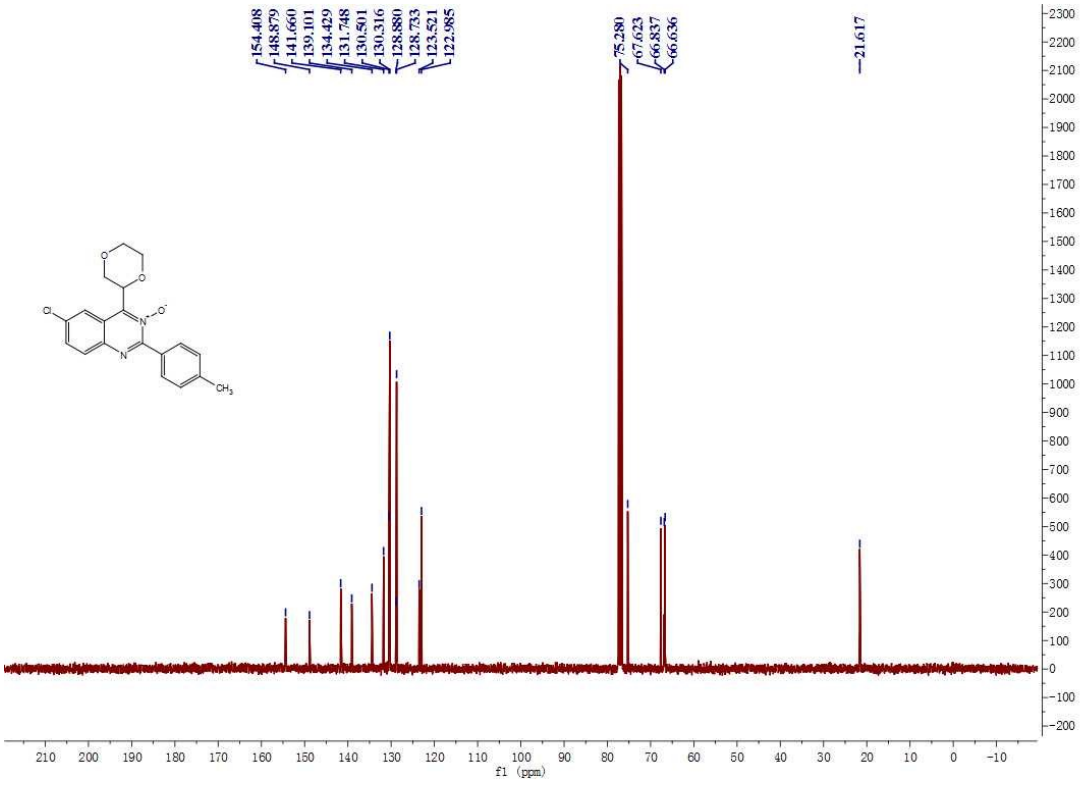
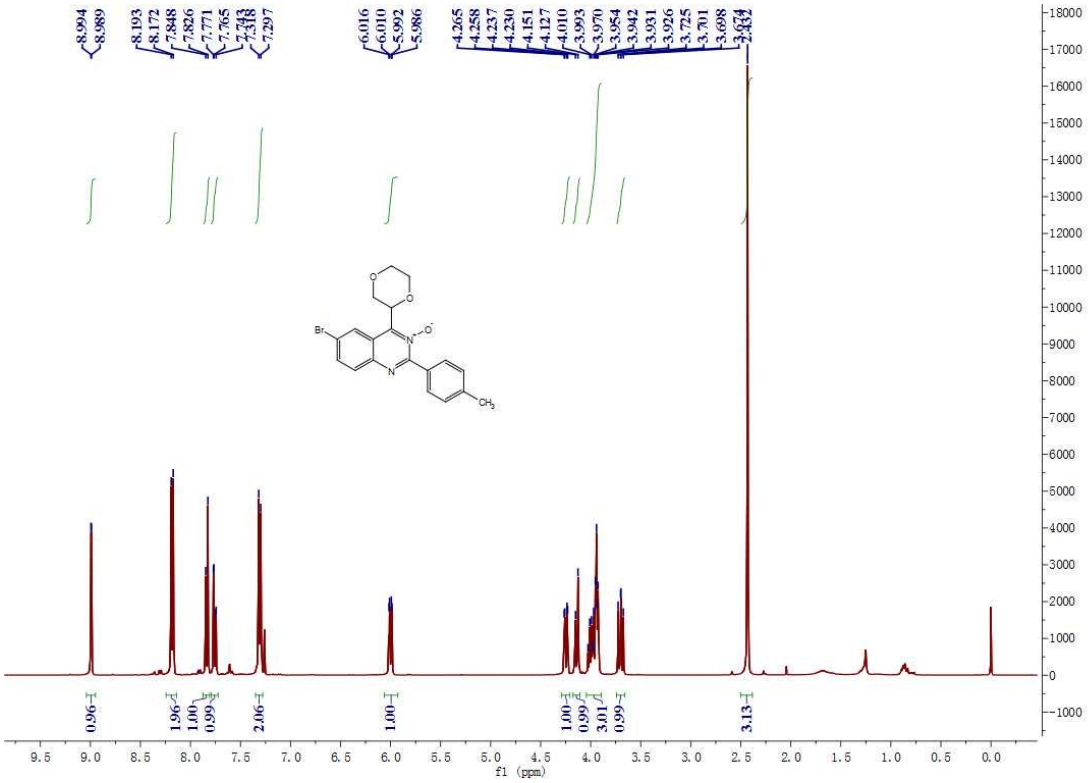
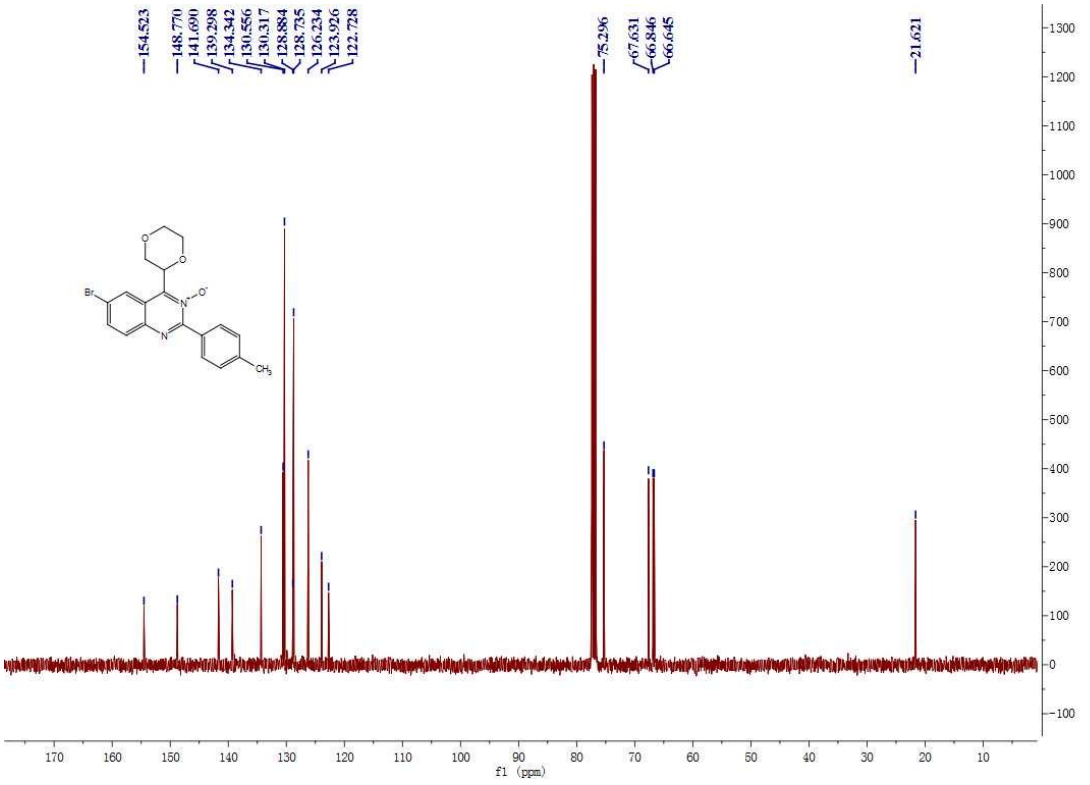
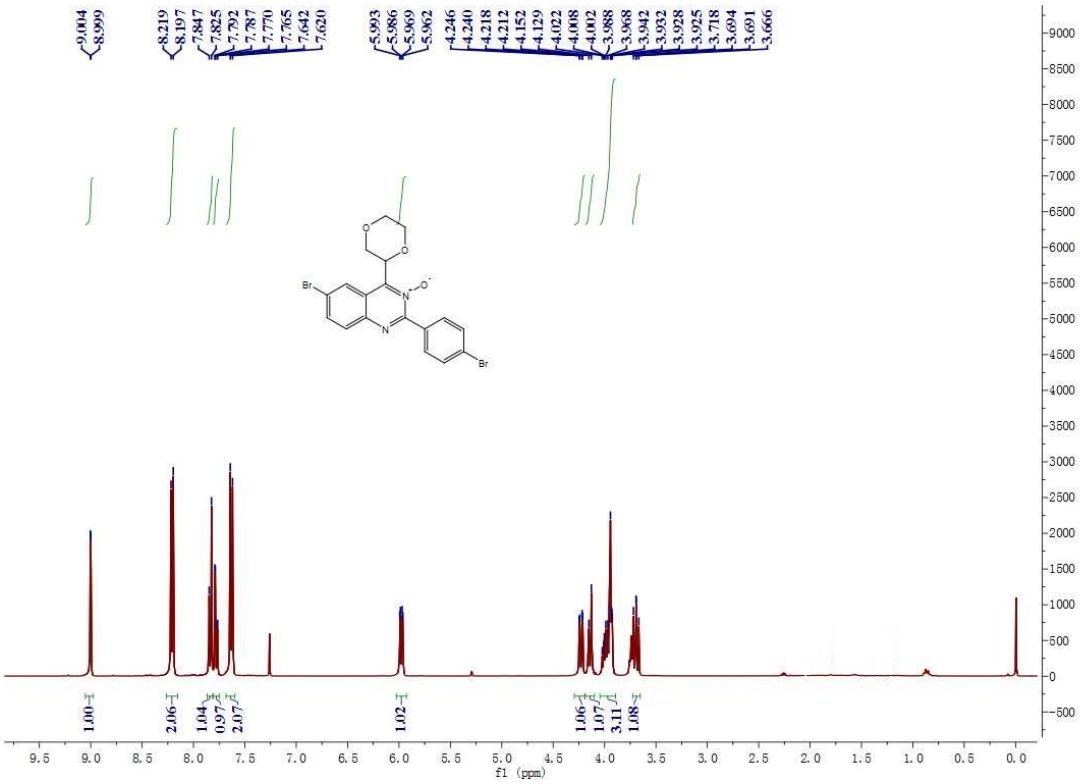
128.585
126.604
126.560
125.467
124.770
124.383
124.065
124.035
122.060
119.351
75.220
67.633
66.872
66.637
21.598
f1 (ppm)

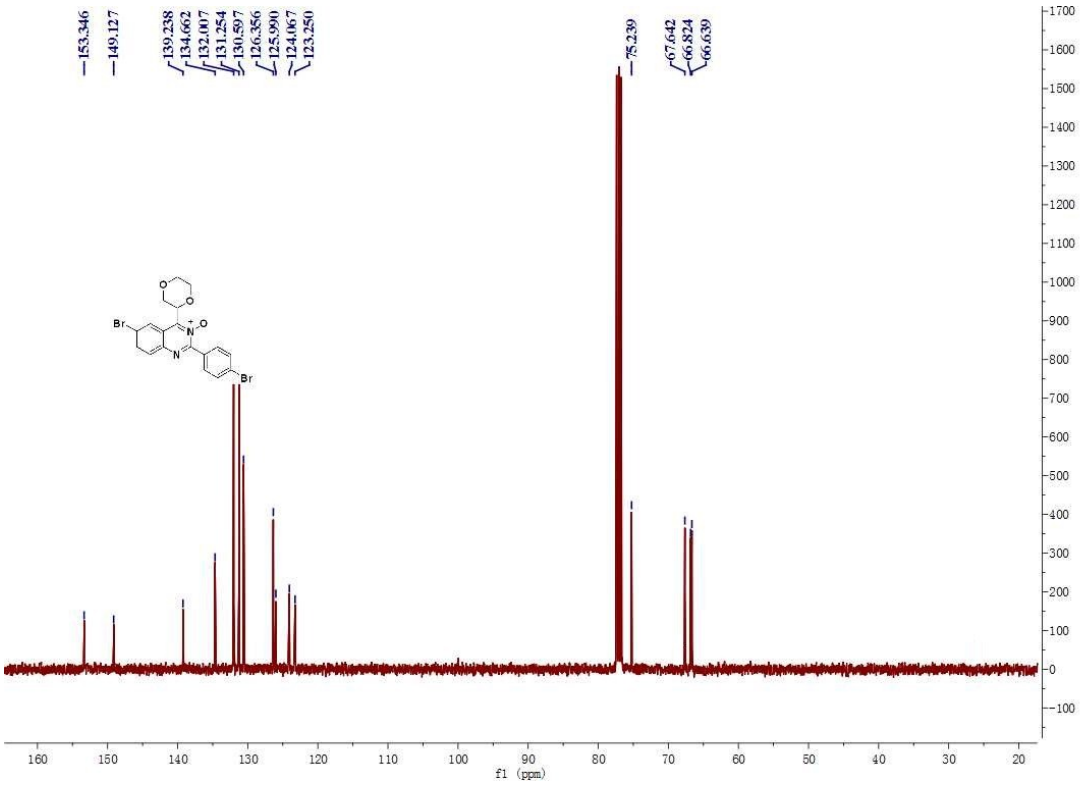

8.824
8.819
8.190
8.170
7.923
7.901
7.640
7.634
7.618
7.612
7.319
7.299
6.024
6.017
6.000
5.993
4.266
4.259
4.238
4.231
4.149
4.125
4.012
3.995
3.972
3.955
3.948
3.942
3.932
3.927
3.726
3.702
3.699
2.424
0.96
1.97
1.00
1.00
2.03
1.00
1.01
1.03
3.00
1.02
3.04
f1 (ppm)

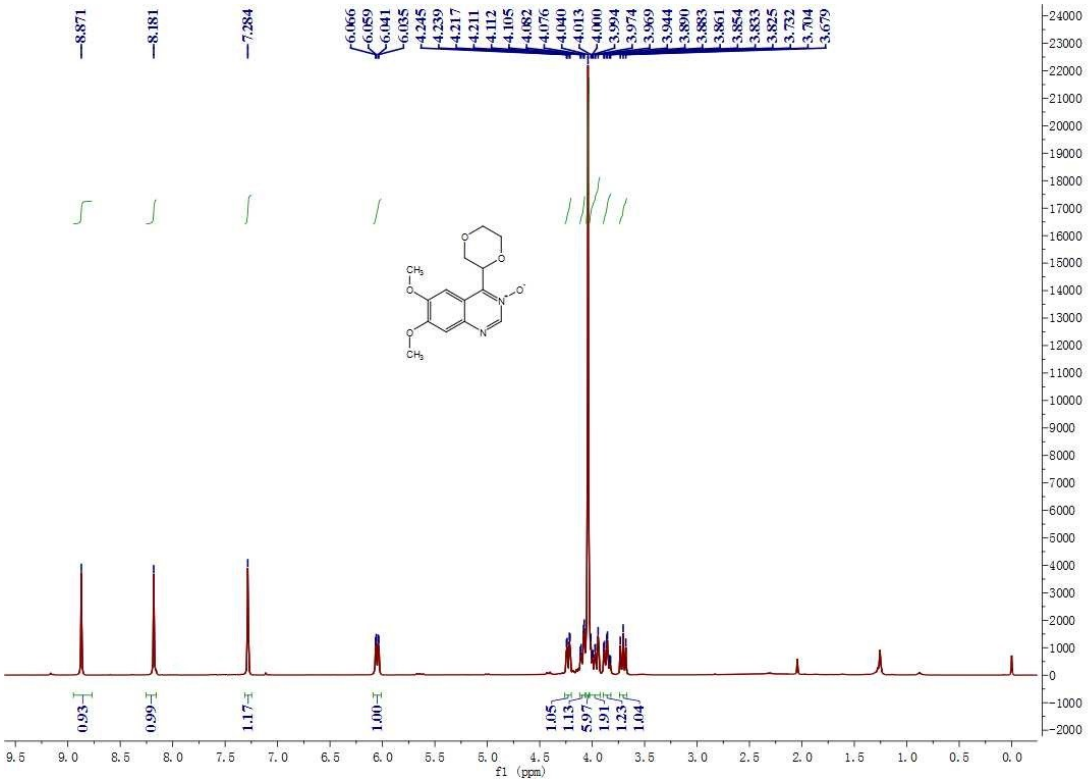

154.408
148.879
141.660
139.101
134.429
131.748
130.501
130.316
128.880
128.733
123.521
122.985
75.280
67.623
66.837
66.636
21.617
f1 (ppm)

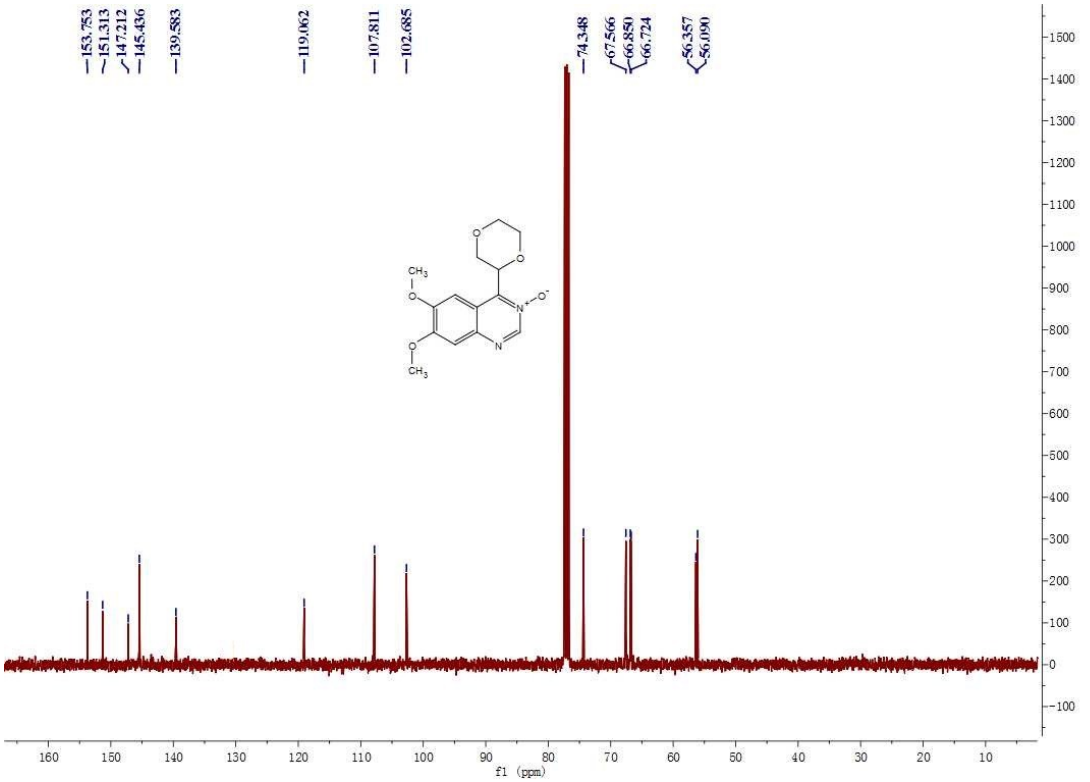

8.994
8.989
8.193
8.172
7.848
7.826
7.771
7.765
7.318
7.297
6.016
6.010
5.992
5.986
4.265
4.258
4.237
4.230
4.151
4.127
4.010
3.993
3.970
3.954
3.942
3.931
3.926
3.725
3.701
3.698
2.432
0.96
1.96
1.00
0.99
2.06
1.00
1.00
0.99
3.01
0.99
3.13
f1 (ppm)

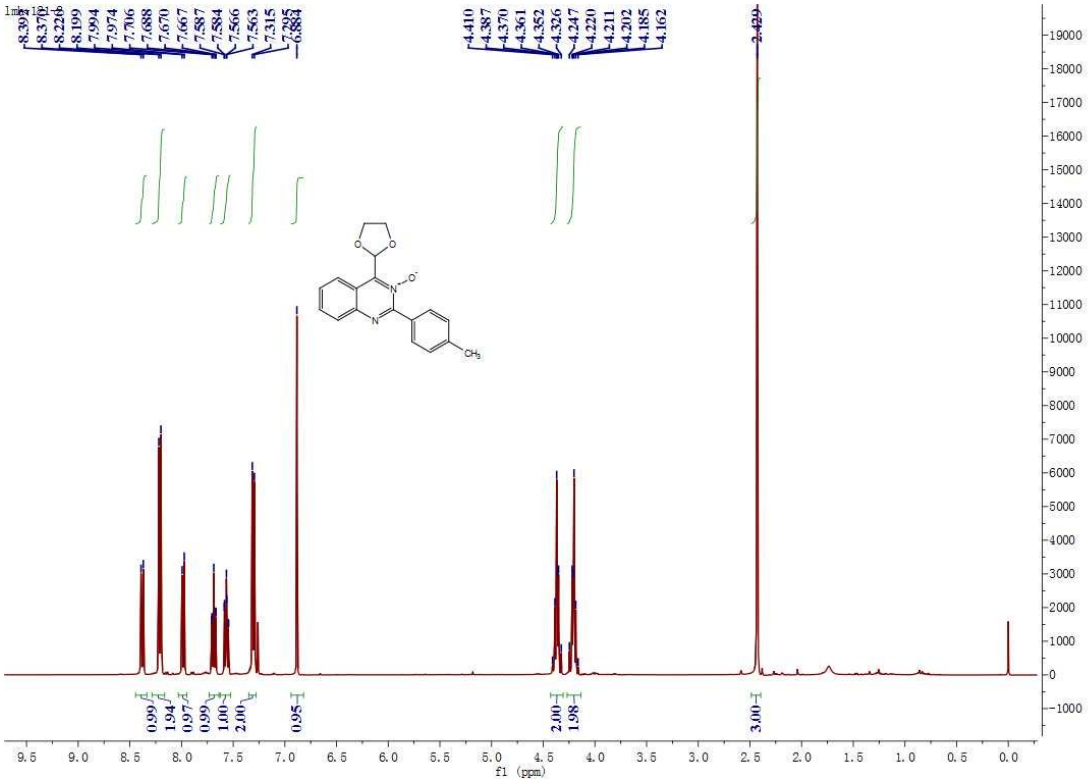

154.523
148.770
141.690
139.298
134.342
130.556
130.317
128.884
128.735
126.234
123.926
122.728
75.296
67.631
66.846
66.645
21.621
f1 (ppm)

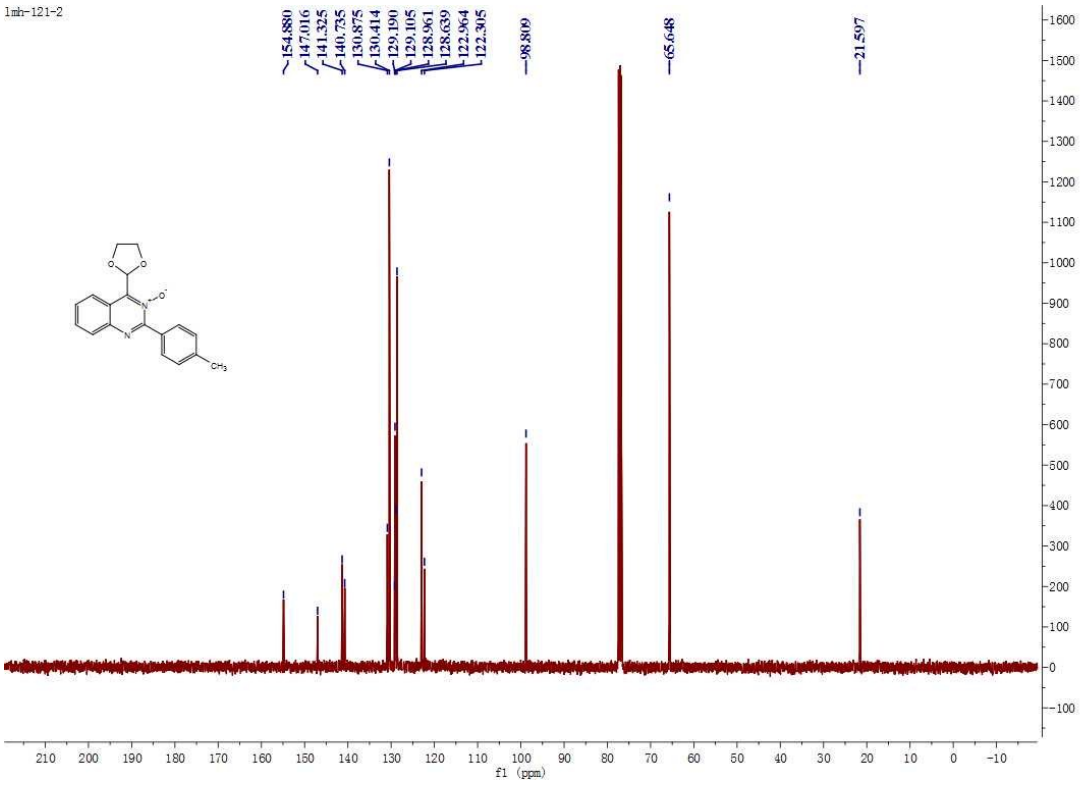

9.004
8.999
8.219
8.197
7.847
7.825
7.792
7.787
7.770
7.765
7.642
7.620
5.993
5.986
5.969
5.962
4.246
4.240
4.218
4.212
4.152
4.129
4.022
4.008
4.002
3.988
3.968
3.942
3.932
3.928
3.925
3.718
3.694
3.691
3.666
1.00
2.06
1.04
0.97
2.07
1.02
1.06
1.07
3.11
1.08
f1 (ppm)

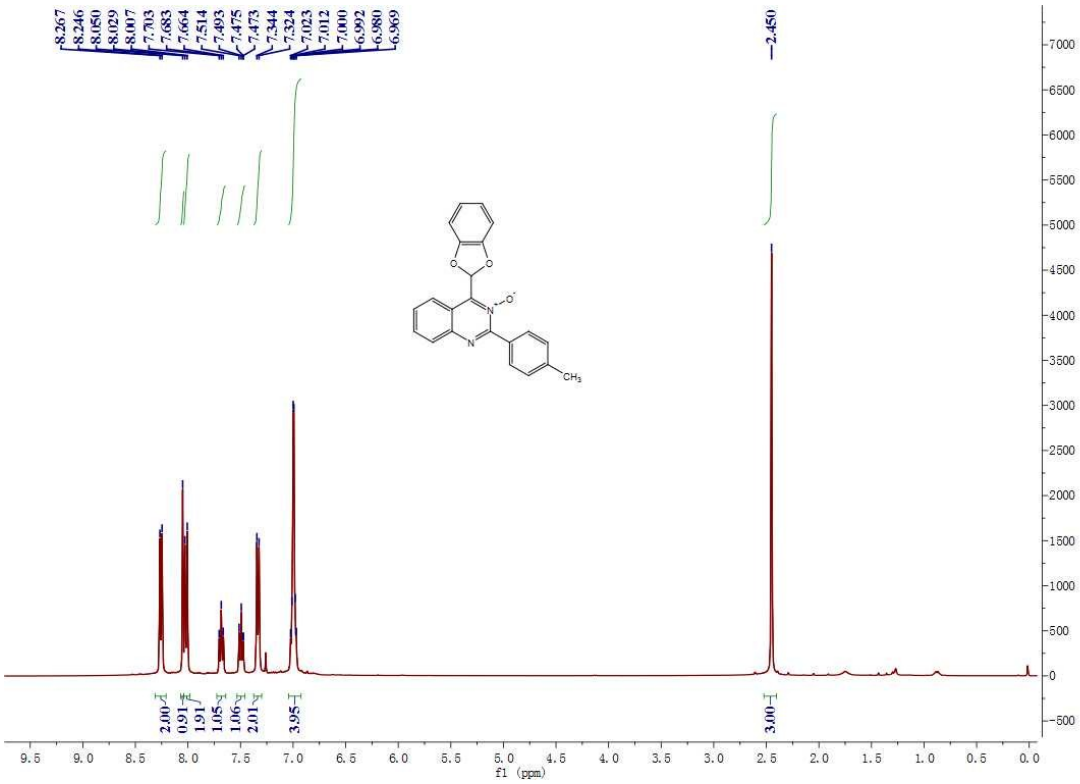

153.346
149.127
139.238
134.662
132.007
131.254
130.597
126.356
125.990
124.067
123.250
78.239
67.642
66.824
66.639
f1 (ppm)

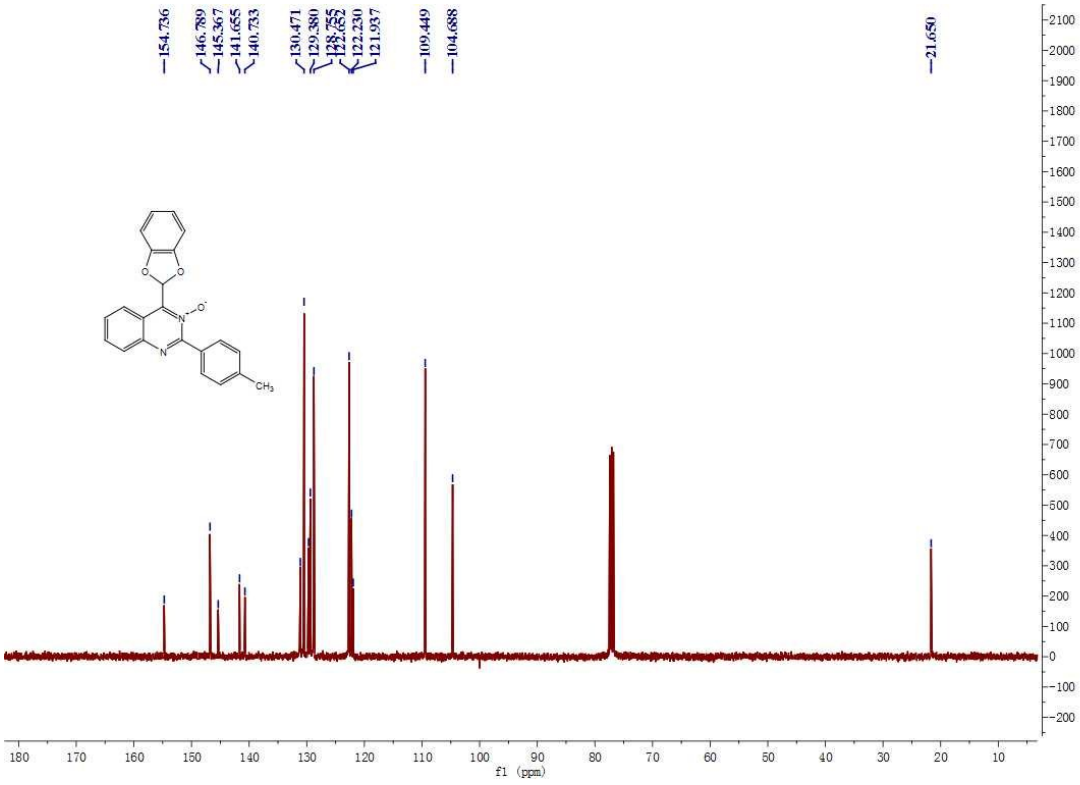

8.871
8.181
7.284
6.066
6.059
6.041
6.035
4.245
4.239
4.217
4.211
4.112
4.105
4.082
4.076
4.040
4.013
4.000
3.994
3.974
3.969
3.944
3.890
3.883
3.861
3.854
3.833
3.825
3.732
3.704
3.679
0.93
0.99
1.17
1.00
1.05
1.13
5.97
1.91
1.23
1.04
f1 (ppm)

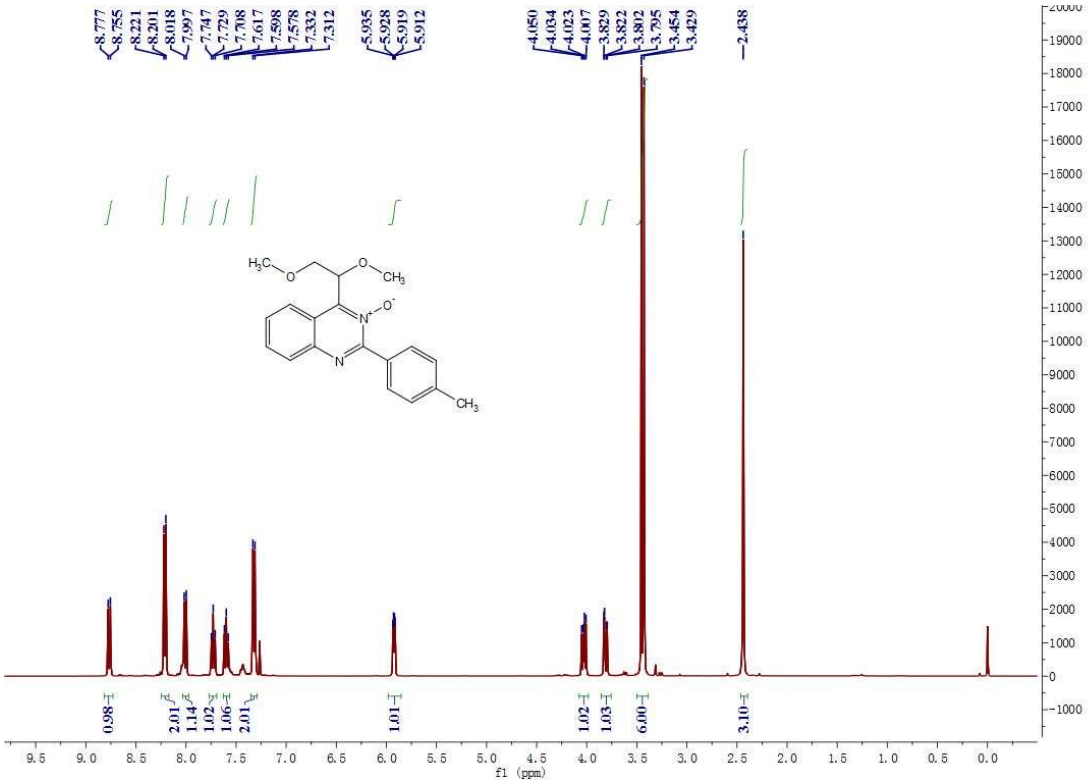

153.753
151.313
147.212
145.436
139.583
119.062
107.811
102.685
74.348
67.566
66.850
66.724
56.357
56.090
f1 (ppm)

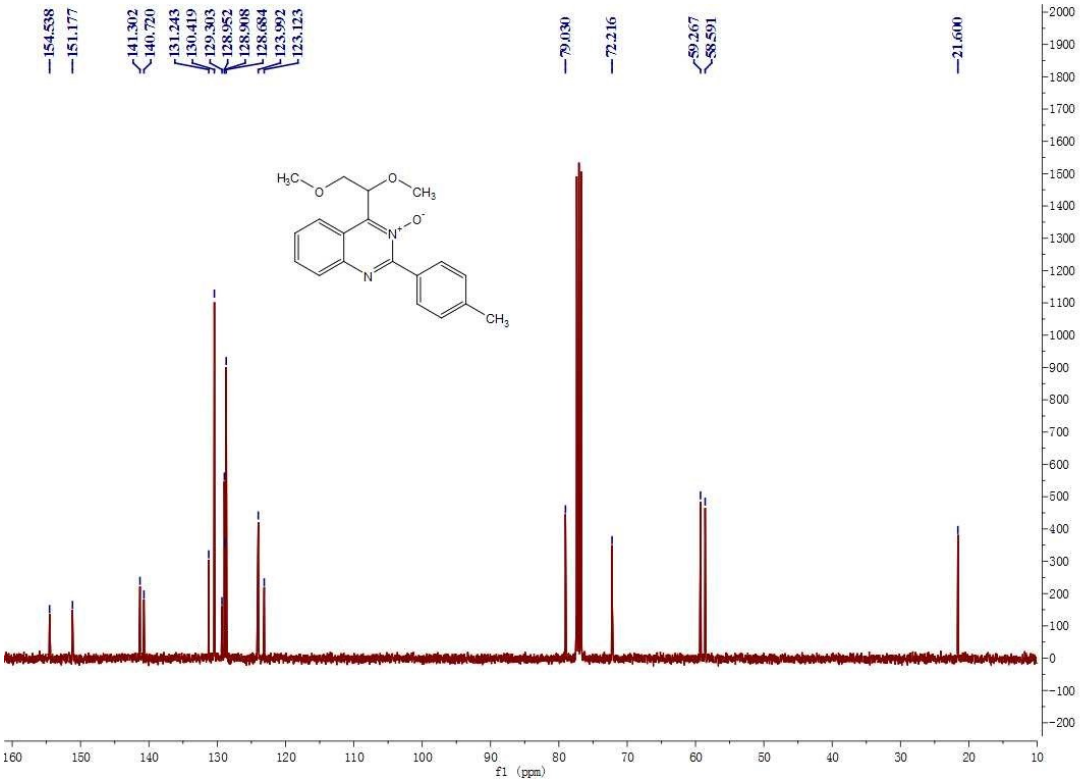

8.393
8.370
8.226
8.199
7.994
7.974
7.706
7.688
7.670
7.667
7.587
7.584
7.566
7.563
7.315
6.884
4.410
4.387
4.370
4.361
4.352
4.326
4.247
4.220
4.211
4.202
4.185
4.162
2.429
0.99
1.94
0.97
0.99
1.00
2.00
0.95
2.00
1.98
3.00
f1 (ppm)

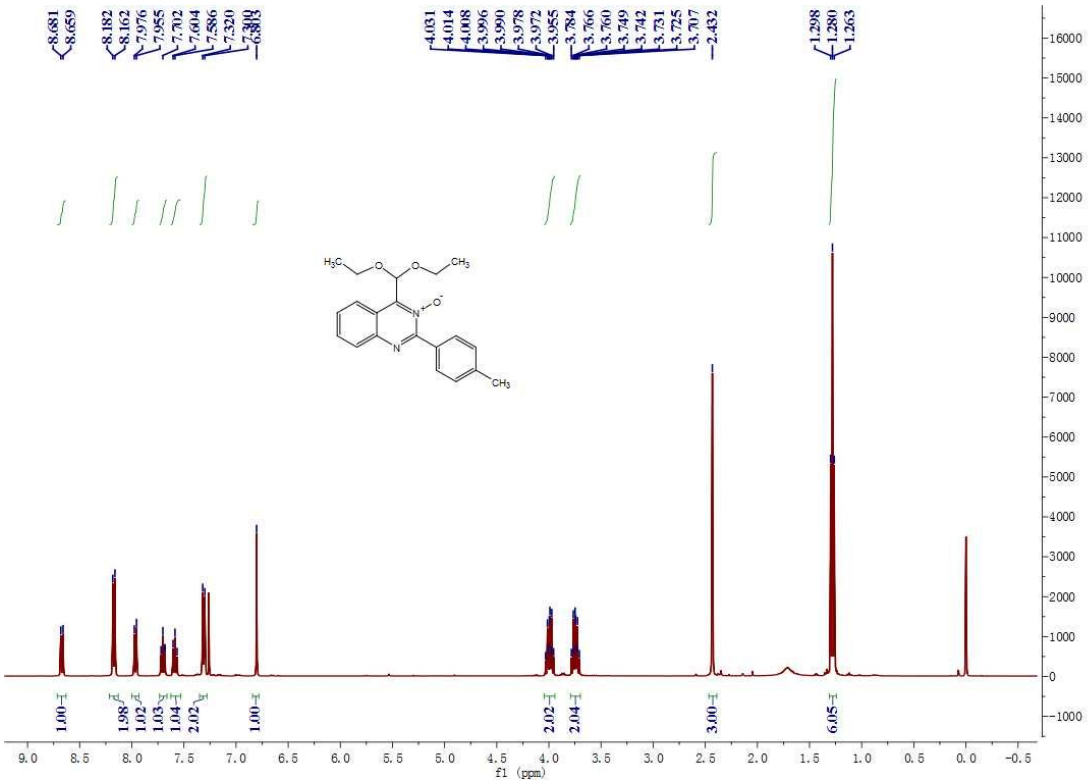

154.880
147.016
141.325
140.735
130.875
130.414
129.190
129.105
128.961
128.639
122.964
122.305
98.809
65.648
21.597
f1 (ppm)

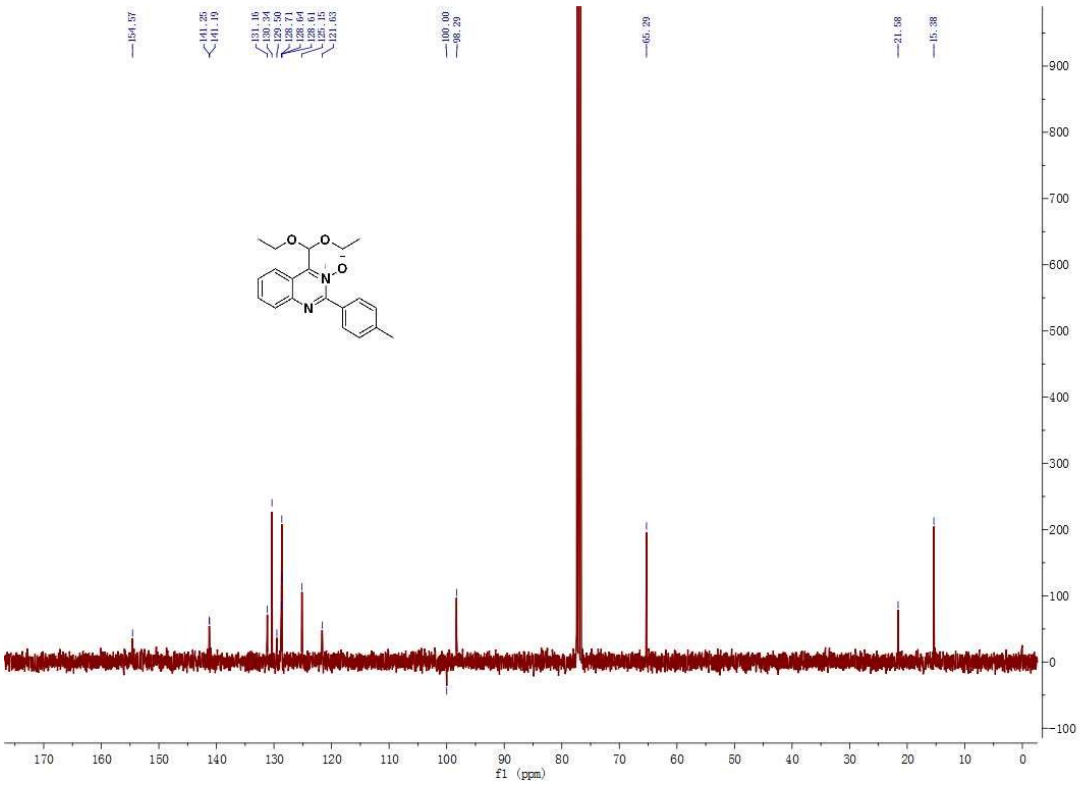

8.267
8.246
8.050
8.029
8.007
7.703
7.683
7.664
7.514
7.493
7.475
7.473
7.344
7.324
7.023
7.012
7.000
6.992
6.980
6.969
2.450
2.00
0.91
1.91
1.05
1.06
2.01
3.95
3.00
f1 (ppm)

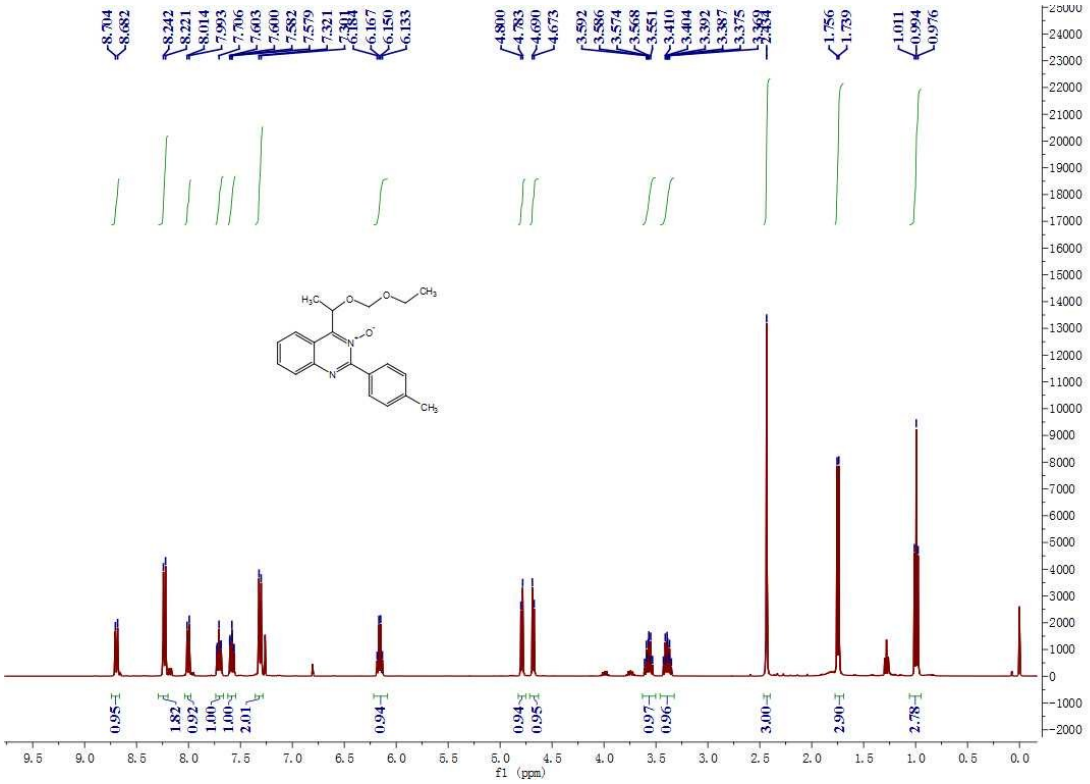

154.736
146.789
145.367
141.655
140.733
130.471
129.380
128.755
122.652
122.230
121.937
109.449
104.688
21.650
f1 (ppm)

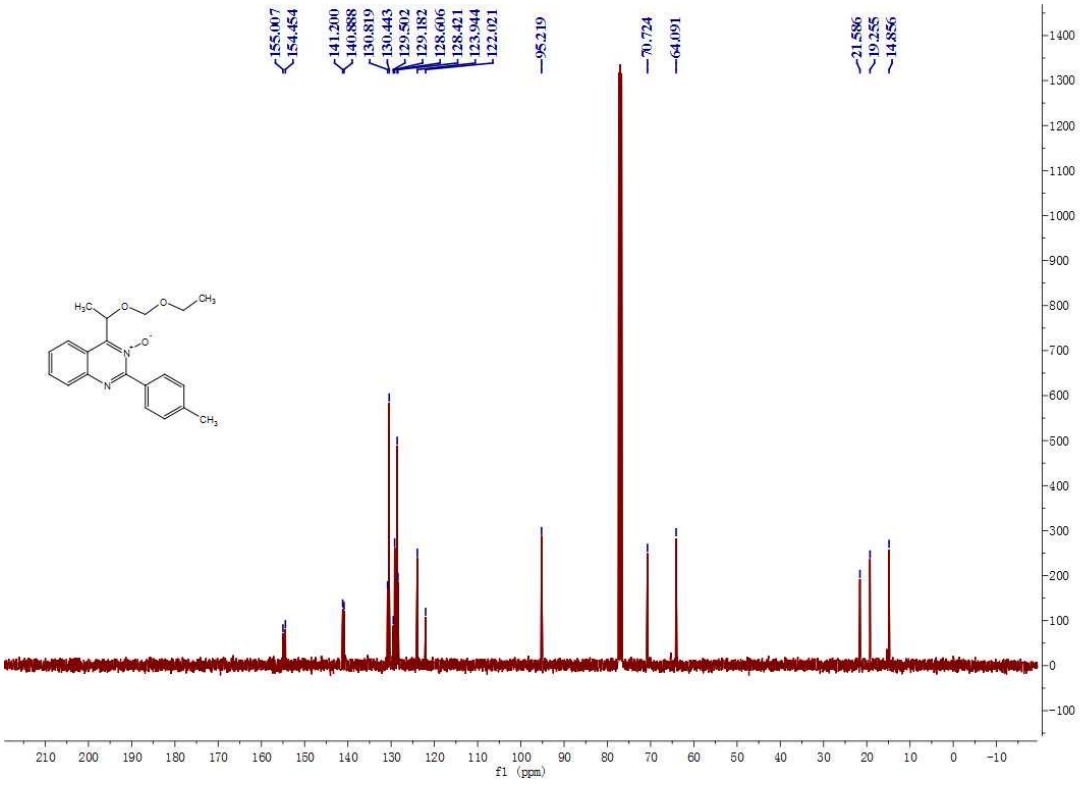

8.777
8.755
8.221
8.201
8.018
7.997
7.747
7.729
7.708
7.617
7.598
7.578
7.332
7.312
5.935
5.928
5.919
5.912
4.050
4.034
4.023
4.007
3.829
3.822
3.802
3.795
3.454
3.429
2.438
0.98
2.01
1.14
1.02
1.06
2.01
1.01
1.02
1.03
6.00
3.10
f1 (ppm)


154.538
151.177
141.302
140.720
131.243
130.419
129.303
128.952
128.908
128.684
123.992
123.123
79.030
72.216
59.267
58.591
21.600
f1 (ppm)


8.681
8.659
8.182
8.162
7.976
7.955
7.702
7.604
7.586
7.320
7.300
6.803
4.031
4.014
4.008
3.996
3.990
3.978
3.972
3.955
3.784
3.766
3.760
3.749
3.742
3.731
3.725
3.707
2.432
1.298
1.280
1.263
1.00
1.98
1.02
1.03
1.04
2.02
1.00
2.02
2.04
3.00
6.05
f1 (ppm)


154.57
141.25
141.19
131.16
130.34
129.50
128.71
128.64
126.61
125.15
121.63
100.00
98.29
65.29
21.58
15.38
f1 (ppm)


8.704
8.682
8.242
8.221
8.014
7.993
7.706
7.603
7.600
7.582
7.579
7.321
6.167
6.150
6.133
4.800
4.783
4.690
4.673
3.592
3.586
3.574
3.568
3.551
3.410
3.404
3.392
3.387
3.375
3.360
2.554
1.756
1.739
1.011
0.994
0.976
0.95
1.82
0.92
1.00
1.00
2.01
0.94
0.94
0.95
0.97
0.96
3.00
2.90
2.78
f1 (ppm)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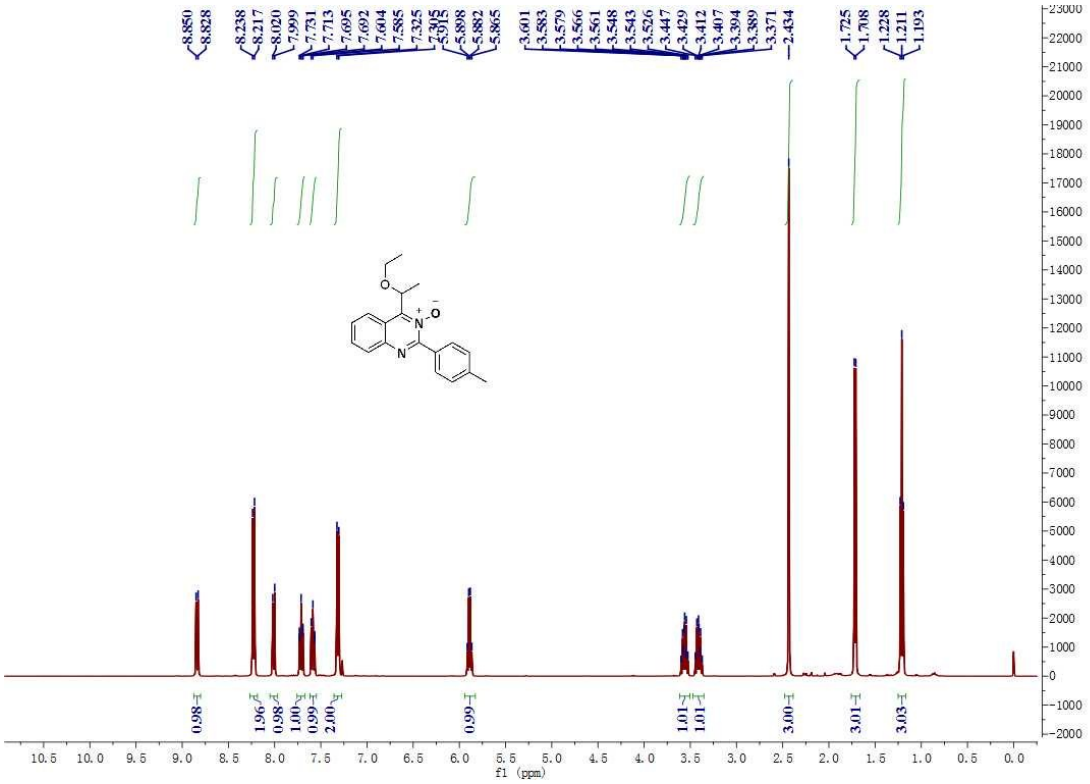
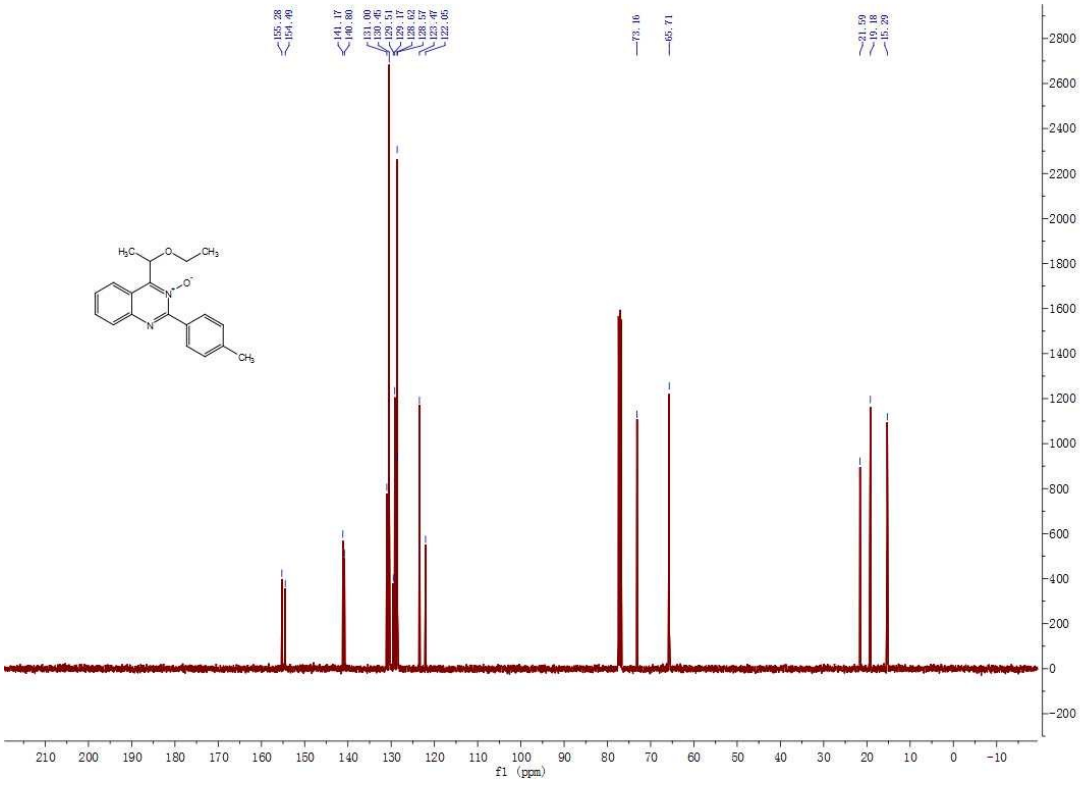
141.200
140.888
130.819
130.443
129.502
129.182
128.606
128.421
123.944
122.021
95.219
70.724
64.091
21.586
19.255
14.856
f1 (ppm)


8.850
8.828
8.238
8.217
8.020
7.999
7.731
7.713
7.695
7.692
7.604
7.585
7.325
7.305
5.915
5.898
5.882
5.865
3.601
3.583
3.579
3.566
3.561
3.548
3.543
3.526
3.447
3.429
3.412
3.407
3.394
3.389
3.371
2.434
1.725
1.708
1.228
1.211
1.193
0.98
1.96
0.98
1.00
0.99
2.00
0.99
1.01
1.01
3.00
3.01
3.03
f1 (ppm)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141.17
140.80
131.00
130.46
129.51
129.17
128.62
128.57
123.37
122.05
73.16
65.71
21.59
19.18
15.29
f1 (ppm)

^{1}H of spectra of benzoic acid concomitancy with **3q.**