Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Cu-Catalyzed Site-Selective Cross-Dehydrogenative Aminations: An Approach of C2-Site Functionalization to *p*-Aminophenols

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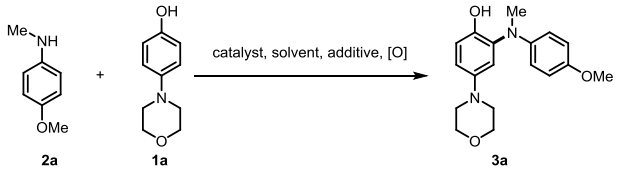
Contents

1. General Information

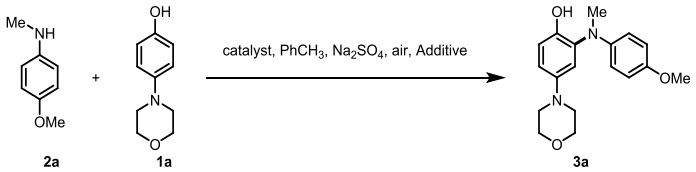
Unless otherwise noted, all reactions were carried out in a flamedried, sealed Schlenk reaction tube under an air conditions. Materials, unless otherwise noted, were purchased from Energy Chemical. Solvents were purchased from Tansoole. Solvents were purified by standard procedures as specified in Purification of Laboratory Chemicals, 4th Ed (W. L. F. Armarego, D. D. Perrin, Butterworth-Heinemann: 1997). Analytical thin layer chromatography (TLC) was performed on silica gel plates with F-254 indicator and compounds were visualized by irradiation with UV (254 and 365 nm) light. Flash column chromatography was carried out using silica gel (200-300 mesh) at increased pressure. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Advance spectrometer at 500 MHz(^{1}H, 500 MHz; ^{13}C, 125 MHz) or Bruker Advance spectrometer at 400MHz (^{1}H, 400 MHz; ^{13}C, 100 MHz). Chemical shift values are reported in δ (ppm) relative to $CHCl_3$ (^{1}H NMR, δ = 7.26; ^{13}C NMR, δ = 77.16) or methanol (^{1}H NMR, δ = 3.31; ^{13}C NMR, δ =49.00). Signal shapes are shown as s (singlet), d (doublet), t (triplet), dd (doublet of doublets), td (triplet double), m (multiplet). High resolution mass spectrometry (HRMS) was performed with a Thermo Scientific LTQ Orbitrap XL. Among them, **3a-3t** and **5a** were analyzed by KeeCloud Biotech; and the parts of Mechanism were analyzed by Life science Research Core Services of NWAFU.

2. Screening Conditions



All of the conditions screening reactions were carried out under room temperature. **2a** (0.33 mmol), **1a** (0.3 mmol), catalysts, solvents, additives and oxidants were selected as described.

Table S1 Screening of catalysts[a]

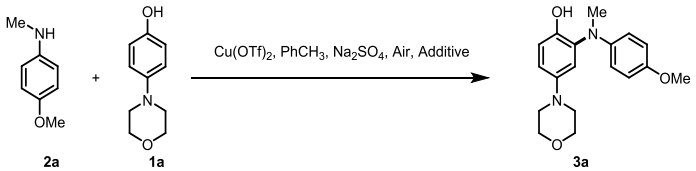


Entry	Catalyst (mol%)	Additive (eq.)	Oxidant	Solvent	Time (h)	Yield(%)[b]
1	-	Na_2SO_4 (2)	Air	$PhCH_3$	10	0
2	$Cu(OAc)_2$ (10)	Na_2SO_4 (2)	Air	$PhCH_3$	10	10
3	$Cu(OTf)_2$ (10)	Na_2SO_4 (2)	Air	$PhCH_3$	10	33
4	$Fe(OTf)_3$ (10)	Na_2SO_4 (2)	Air	$PhCH_3$	10	30
5	CuCl (10)	Na_2SO_4 (2)	Air	$PhCH_3$	10	14
6	CuI	Na_2SO_4 (2)	Air	$PhCH_3$	10	12
7	CuOTf	Na_2SO_4 (2)	Air	$PhCH_3$	10	24
8	$CuCl_2$	Na_2SO_4 (2)	Air	$PhCH_3$	10	17
9	Cu-TMEDA Catalyst(10)	Na_2SO_4 (2)	Air	$PhCH_3$	7	5<
10	$Cu(OTf)_2$ (15)	Na_2SO_4 (2)	Air	$PhCH_3$	7	50

11	$Cu(OTf)_2$ (20)	Na_2SO_4 (2)	Air	$PhCH_3$	7	32
12	Cu-TMEDA Catalyst(15)	Na_2SO_4 (2)	Air	$PhCH_3$	7	5<

[a] Reaction conditions: **2a** (0.33 mmol), **1a** (0.3 mmol), solvent (3 mL). [b] Isolated yields.

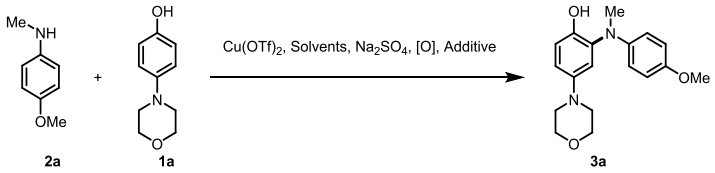
Table S2 Optimization of the additives[a]



Entry	Catalyst (mol%)	Additive (eq.)	Oxidant	Solvent	Time (h)	Yield (%)[b]
1	$Cu(OTf)_2$ (15)	Na_2SO_4 (2) + En.(0.15)	Air	$PhCH_3$	7	<5
2	$Cu(OTf)_2$(15)	Na_2SO_4 (2) + Phen.(0.15)	Air	$PhCH_3$	7	<5
3	$Cu(OTf)_2$(15)	Na_2SO_4 (2) + Bipy(0.15)	Air	$PhCH_3$	7	<5
4	$Cu(OTf)_2$(15)	Na_2SO_4(2)+TEMEDA(0.15)	Air	$PhCH_3$	7	<5
5	$Cu(OTf)_2$(15)	Na_2SO_4 (2) + HOTf (0.15)	Air	$PhCH_3$	7	12
6	$Cu(OTf)_2$(15)	Na_2SO_4 (2)+$Ti(OiPr)_4$ (0.2)	Air	$PhCH_3$	7	65
7	$Cu(OTf)_2$(15)	Na_2SO_4 (2)+$Ti(OiPr)_4$ (0.3)	Air	$PhCH_3$	7	78
8[c]	$Cu(OTf)_2$(15)	Na_2SO_4 (2)+$Ti(OiPr)_4$ (0.3)	Air	$PhCH_3$	7	NR
9	$Cu(OTf)_2$(15)	-	Air	$PhCH_3$	7	37
10	$Cu(OTf)_2$(15)	$Ti(O^iPr)_4$(0.3)	Air	$PhCH_3$	7	42
11	$Cu(OTf)_2$(15)	$MgSO_4$ (2)+ $Ti(OiPr)_4$ (0.3)	Air	$PhCH_3$	7	69

[a] Reaction conditions: **2a** (0.33 mmol), **1a** (0.3 mmol), solvent (3 mL). [b] Isolated yields. [c] No **2a**

Table S3 Optimization of the solvents and oxidant[a]

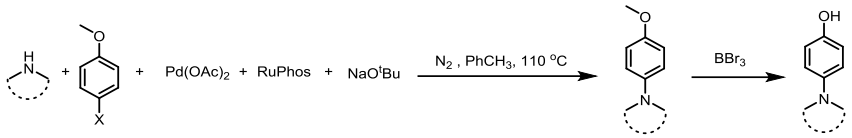


Entry	Catalyst (mol%)	Additive (eq.)	Oxidant	Solvent	Time (h)	Yield (%)[b]
1	$Cu(OTf)_2$ (15)	Na_2SO_4 (2)+ $Ti(OiPr)_4$ (0.3)	Air	$PhCF_3$	7	66
2	$Cu(OTf)_2$(15)	Na_2SO_4 (2)+ $Ti(OiPr)_4$ (0.3)	Air	PhCl	7	73
3	$Cu(OTf)_2$(15)	Na_2SO_4 (2)+ $Ti(OiPr)_4$ (0.3)	Air	HFIP	7	5
4	$Cu(OTf)_2$(15)	Na_2SO_4 (2)+ $Ti(OiPr)_4$ (0.3)	Air	DCM	7	44
5	$Cu(OTf)_2$(15)	Na_2SO_4 (2)+ $Ti(OiPr)_4$ (0.3)	Air	THF	7	34
6	$Cu(OTf)_2$(15)	Na_2SO_4 (2)+ $Ti(OiPr)_4$ (0.3)	Air	MeOH	4	0
7	$Cu(OTf)_2$ (15)	Na_2SO_4 (2)+ $Ti(OiPr)_4$ (0.3)	O_2	$PhCH_3$	7	27

[a] Reaction conditions: **2a** (0.33 mmol), **1a** (0.3 mmol), solvent (3 mL). [b] Isolated yields.

3. Preparation of Starting Materials

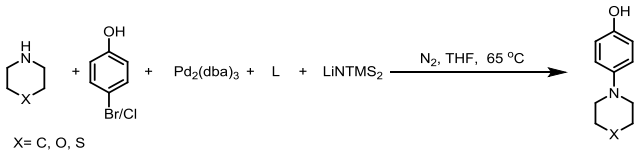
General Procedure A:



A screw-cap vial equipped with a magnetic stir bar was charged with the aryl halide (5.05 mmol), secondary amine (5 mmol), $Pd(OAc)_2$ (0.05 mmol), RuPhos (0.1 mmol), and powdered NaO^tBu (6 mmol). The vial was transferred to a preheated oil bath (110 °C). After 12 h, the reaction mixture was cooled and dissolved in CH_2Cl_2/H_2O mixture (1:1). The organic phase was separated, the solvent was evaporated in vacuo, and the product was isolated by flash chromatography on a silica gel column (CH_2Cl_2 or CH_2Cl_2/methyl tert-butyl ether).

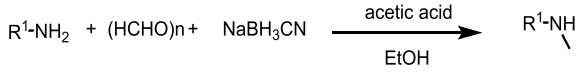
In a three-necked round-bottomed flask was placed the protected phenol compound (2.23 mmol) in anhydrous CH_2Cl_2 (30 mL). A solution of 1 M boron tribromide (3 mL, 3.00 mmol) was added dropwise at -78 °C, and the mixture was stirred at room temperature for 3 h. The mixture was poured onto ice and extracted with $NaHCO_3$ (3 × 30 mL). The organic layers were dried with anhydrous sodium sulfate, filtered and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography.

General Procedure B:



An oven-dried Schlenk tube was charged with $Pd_2(dba)_3$ (2 mol % Pd), Davephose(2.4 mol %), aryl halide (5 mmol), and amine (6 mmol). The Schlenk tube was evacuated and back-filled with argon, and the Teflon screwcap was replaced with a rubber septa. The $LiN(TMS)_2$ solution (1 M in THF, 5.5 mL) was added via syringe (substrates that are liquids at room temperature were added at this point). The rubber septum was replaced with the Teflon screwcap, and the reaction vessel was sealed. The reaction mixture was heated at 65 °C with stirring until the aryl halide had been consumed as judged by GC analysis. The reaction mixture was then allowed to cool to room temperature. To the reaction mixture was added 1 M HCl (5-10 mL), and the mixture was stirred at room temperature for 5 min, followed by neutralization with a saturated $NaHCO_3$ solution (5-10 mL), and the reaction mixture was diluted with ethyl acetate. The organic layer was dried with $MgSO_4$, filtered through a pad of celite, and concentrated in vacuo. The crude residue was purified by flash chromatography on silica gel using mixtures of ethyl acetate/hexanes or methanol/ dichloromethane (for very polar compounds) as the eluent.

General Procedure C (procedure for Synthesis of *N*-alkylaniline): all of the *N*-alkylaniline were reported compounds.



To a solution of aniline (10 mmol) in 40 mL of water-free ethanol, paraformaldehyde (12 mmol) and a drop of acetic acid as the catalyst were added. After the reaction was carried out for 2 h at room temperature, sodium cyanoborohydride (12 mmol) was added and the reaction was continued until the material disappeared as monitored by TLC. The solvent was removed in vacuum, and 50 mL of ethanol was added to the mixture. The resulting mixture was washed by saturated sodium carbohydrate and water subsequently, dried over sodium

sulfate, concentrated in vacuum to afford *N*-alkylaniline.

4. General Reaction Procedure

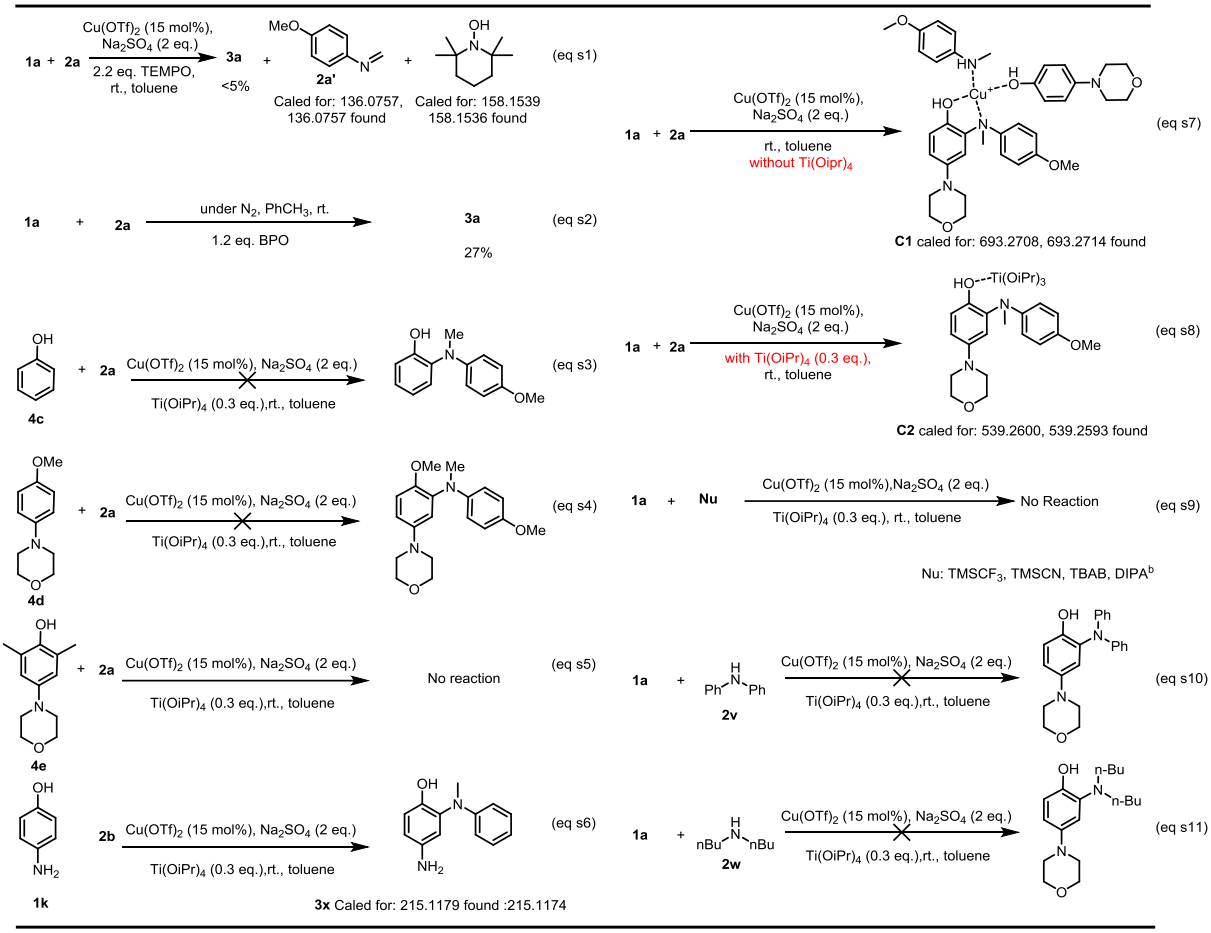
For 0.3 mmol sacles:

To a reaction oven-dried tube equipped with stir bar, Na_2SO_4 (85.2 mg, 2.0 eq., 0.6 mmol) and $Cu(OTf)_2$ (16.2 mg, 0.045 mmol,15 mol%) were added. A solution of *N*-alkylaniline (35.3mg, 0.33 mmol, 1.1 eq) and *p*-aminophenol (0.3 mmol, 1.0 eq.) were added totally 3mL toluene. The tube was capped with a rubber stopper, then, $Ti(OiPr)_4$ (25 mg, 0.09mmol, 30 mol%) was added dropwise via syringe. The resulting mixture was stirred 7-10 h at the corresponding temperature. After the reaction completed, quenched by 0.1 M HCl (1 mL), then extract with $NaHCO_3$ (5mL, aq.) and EA for 3 times, pH > 8, and the organic layer was concentrated under vacuum. The subsequent residue was purified by column chromatography to get the desired compounds.

For 3 mmol sacles (3o):

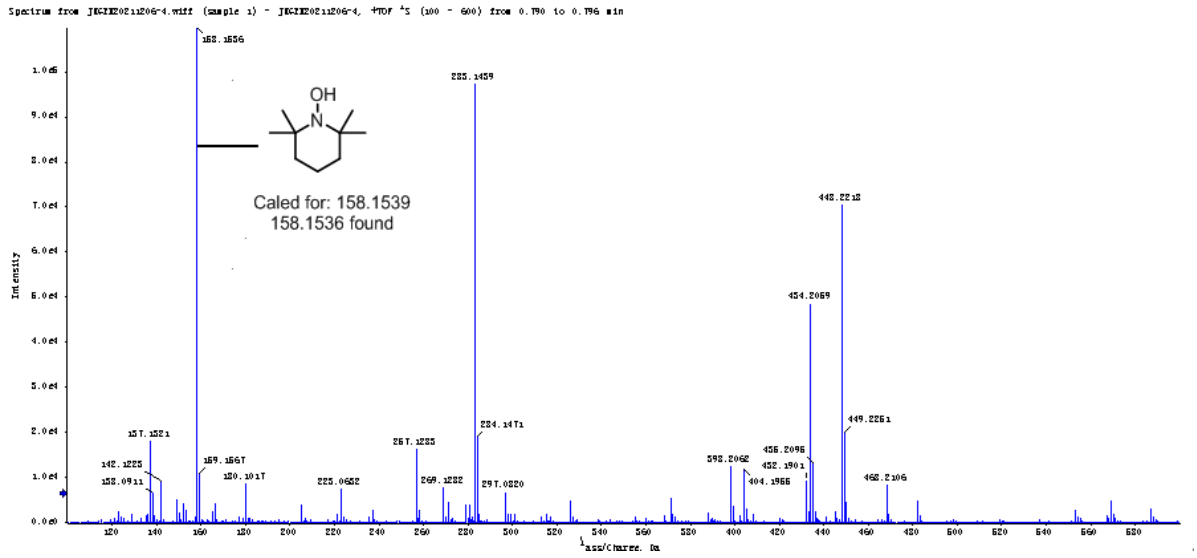
To a reaction oven-dried tube equipped with stir bar, Na_2SO_4 (852 mg, 2.0 eq., 6 mmol) and $Cu(OTf)_2$ (162 mg, 0.45 mmol,15 mol%) were added. A solution of *N*-alkylaniline (353 mg, 3.3 mmol, 1.1 eq) and *p*-aminophenol (3 mmol, 1.0 eq.) were added totally 22mL toluene. The tube was capped with a rubber stopper, then, $Ti(OiPr)_4$ (250 mg, 0.9mmol, 30 mol%) was added dropwise via syringe. The resulting mixture was stirred 24 h at the room temperature. After the reaction completed, quenched by 0.1 M HCl (5 mL), then extract with saturated $NaHCO_3$ (15mL, aq.) and EA for 3 times, pH > 8, and the organic layer was concentrated under vacuum. The subsequent residue was purified by column chromatography to get the desired compounds.

5. Mechanistic Study



[a] 1a (4c ,4d or 4e) (0.3 mmol), 2a (0.33 mmol); [b] No $Cu(OTf)_2$

The reactants **1a**, **4c**, **4d** and **4e** were conducted in 0.3 mmol, **2a** 0.33 mmol, $Cu(OTf)_2$ 0.045 mmol, Na_2SO_4 0.6 mmol, and 0.09 mmol $Ti(O^iPr)_4$ was added. We invested the radical scavenger test (eq.s1), radical initiator experiments (eq. s2), which shows that the process of radicals is most likely. Eq. s3 – eq. s6 suggesst that the phenolic hydroxyl-, protected-amino and the active site of the substrate are very vital. The reaction mixture were detected by HRMS, and two key species **C1** and **C2** were detected (eq. s7- eq. s8). In order to preclude the S_{NR1} mechanism, we replaced **2a** with various nucleophiles, there were no reaction (eq. s9). Diarylamines and aliphatic amines were tried in the reaction. When monoarylamine is replaced by diphenylamine **2v** in the reaction (eq. s10), there was no reaction; when aliphatic amines (di-n-butylamine) **2w** participated in the reaction (eq. s11), however, the products were extremely complicated accompany with precipitation.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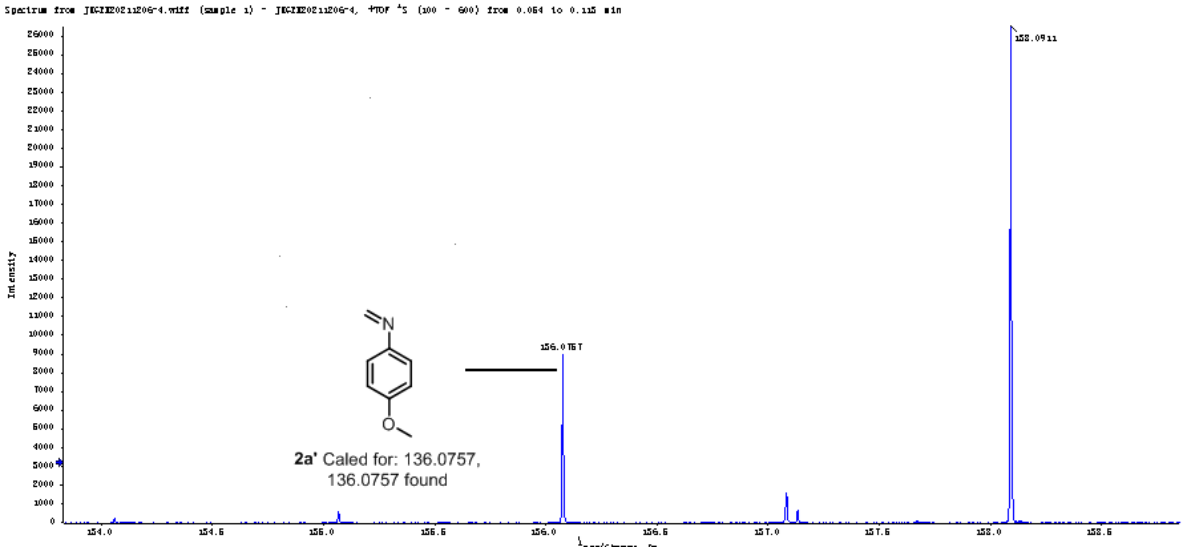
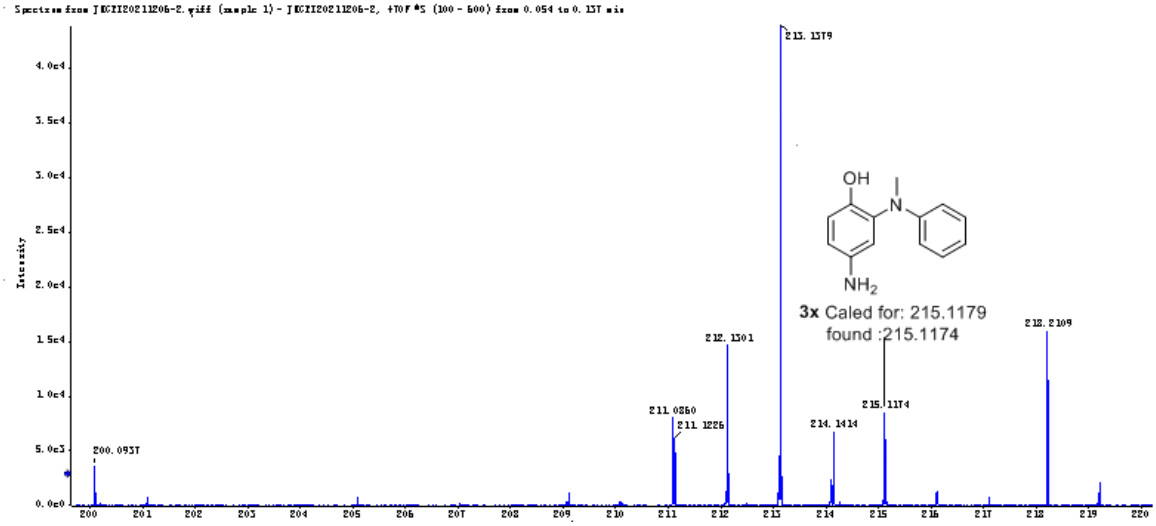
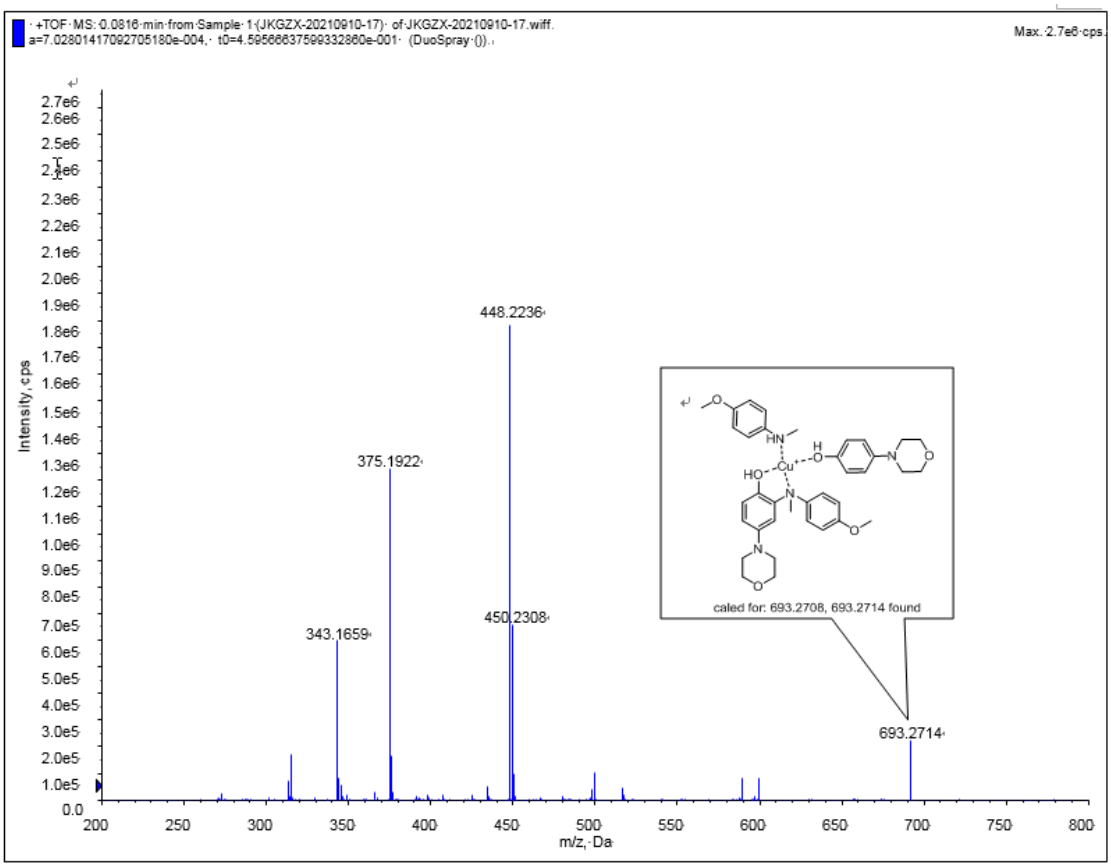
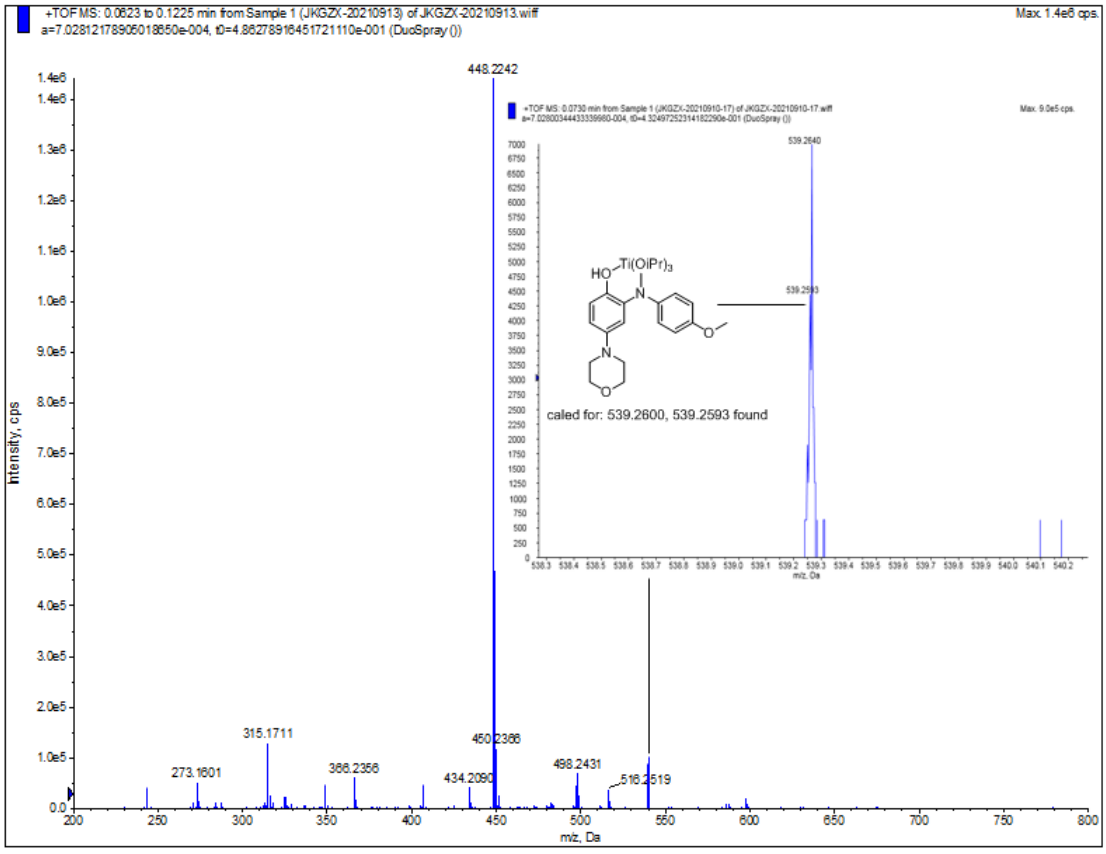
N
Caled for: 158.1539
158.1536 found
Intensity
m/z


N
O
2a' Caled for: 136.0757,
136.0757 found
Intensity


OH
N
NH2
3x Caled for: 215.1179
found :215.1174
Intensity


+TOF MS: 0.0816 min from Sample 1 (JKGZX-20210910-17) of JKGZX-20210910-17.wiff
a=7.028014170927051 80e-004, t0=4.595666375993328 60e-001 (DuoSpray ())
Max. 2.7e6 cps.
Intensity, cps
m/z, Da
448.2236
375.1922
450.2308
343.1659
693.2714
caled for: 693.2708, 693.2714 found


+TOF MS: 0.0823 to 0.1225 min from Sample 1 (JKGZX-20210913) of JKGZX-20210913.wiff
a=7.028121789050186 50e-004, t0=4.862789164517211 10e-001 (DuoSpray ())
Max. 1.4e6 cps.
Intensity, cps
m/z, Da
448.2242
315.1711
450.2366
273.1601
366.2356
434.2090
498.2431
516.2519
caled for: 539.2600, 539.2593 found

EPR expeiriments

EPR spectra recorded at room temperature using DMPO as trapping agent. 0.1 mmol scales, 0.1M. The mixture was filtered by syringe filter (0.22 μm). Microwave frequency = 9.6457. Mircowave power = 6.325 mW. Mod. ampl. = 14 G.

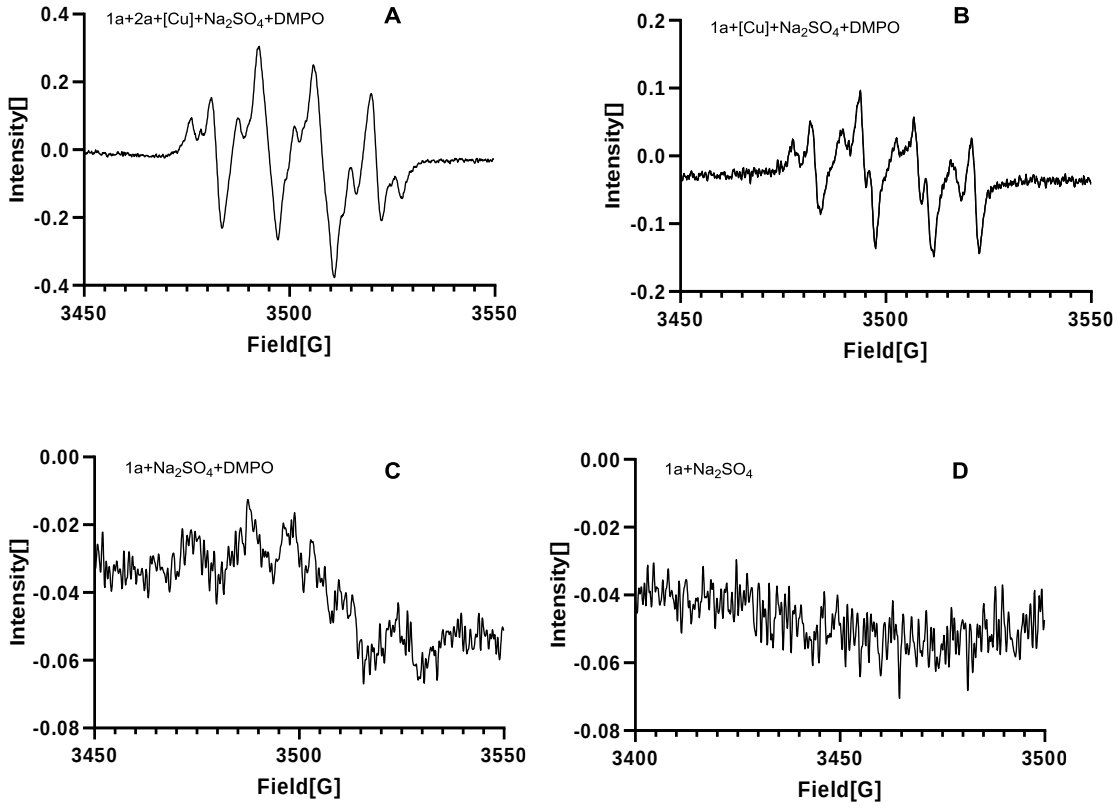


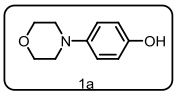
Figure S1 EPR spectra of mixture.

6. Characterization of Compounds and X-ray Structure of 5a.

6.1 Characterization of Compounds

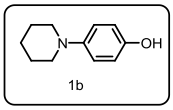
1a[1]**, 1b**[1], **1c**[2], **4a**[3], **4b**[1] and **4d**[4] and are known compounds**. 1d** and **4c** were commercial available. **4e** was synthesized through known literature[1].

*4-morpholinophenol***(1a):** known compounds,[1] general procedure B, purified by SiO_2 column chromatography (hexane/EtOAc = 5:1) to give red solid, 1.66 g (10 mmol), 92 % yield, which perfect consistent with the literature.[2] **^{1}H NMR** (400 MHz, Methanol-d_4) δ 6.86 - 6.81 (m, 2H), 6.76 - 6.66 (m, 2H), 3.79 (t, *J* = 4.0 Hz, 4H), 2.97 (t, *J* = 4.0 Hz, 4H).



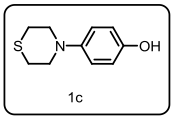
1a

*4-(piperidin-1-yl)phenol***(1b):** known compounds,[1] general procedure B, purified by SiO_2 column chromatography (hexane/EtOAc = 5:1) to give gray solid, 779 mg, 88 %, yield, which perfect consistent with the literature.[2] **^{1}H NMR** (500 MHz, Methanol-d_4) δ 6.85 (d, *J* = 9.0 Hz, 2H), 6.67 (d, *J* = 8.8 Hz, 2H), 2.91 (t, 4H), 1.74 - 1.63 (m, 4H), 1.56 - 1.43 (m, 2H).



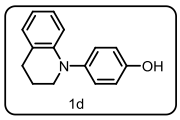
1b

4-thiomorpholinophenol **(1c):** known compounds,[2] general procedure B, purified by SiO_2 column chromatography (hexane/EtOAc = 5:1) to give white solid, 780 mg, 82 % yield. **^{1}H NMR** (500 MHz, Methanol-d_4) δ 7.35 (d, *J* = 8.3 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 3.96 - 3.47 (m, 4H), 3.16 (d, *J* = 6.8 Hz, 4H). **^{13}C NMR** (125 MHz, MeOD) δ 121.8, 116.8, 56.5, 27.1.



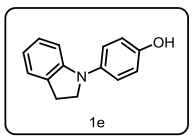
1c

4-(3,4-dihydroquinolin-1(2H)-yl)phenol **(1d):** commercial available. **^{1}H NMR** (500 MHz, Methanol-d_4) δ 6.97 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 9.0 Hz, 1H), 6.78 (d, *J* = 8.7 Hz, 2H), 6.74 (t, *J* = 8.5 Hz, 1H), 6.50 (t, *J* = 7.3 Hz, 1H), 6.32 (d, *J* = 9.3 Hz, 1H), 3.47 - 3.39 (m, 2H), 2.74 (t, *J* = 6.4 Hz, 2H), 1.98 - 1.84 (m, 2H). **^{13}C NMR** (125 MHz, MeOD) δ 153.7, 145.0, 139.9, 128.1, 126.9, 125.2, 122.0, 116.1, 115.2, 113.4, 51.0, 26.8, 21.6.



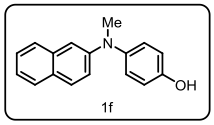
1d

4-(indolin-1-yl)phenol **(1e):** general procedure A, purified by SiO_2 column chromatography (hexane/EtOAc = 8:1) to give dark red solid, 122 mg (1mmol), 59% yield. **^{1}H NMR** (500 MHz, Methanol-d_4) δ 6.97 - 6.91 (m, 3H), 6.87 - 6.80 (m, 1H), 6.77 - 6.70 (m, 2H), 6.65 (d, *J* = 7.9 Hz, 1H), 6.53 (t, *J* = 7.3 Hz, 1H), 3.56 (t, *J* = 8.4 Hz, 2H), 2.83 (t, *J* = 8.4 Hz, 2H). **^{13}C NMR** (125MHz, MeOD) δ 151.4, 148.1, 136.4, 129.7, 125.8, 123.6, 120.3, 117.0, 114.7, 106.2, 52.3, 27.0. **HRMS (ESI) m/z** calcd for $C_{14}H_{14}NO^+$ $(M+H)^+$: 212.1069, found 212.1068.



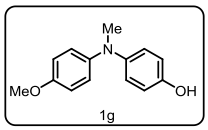
1e

4-(methyl(naphthalen-2-yl)amino)phenol **(1f):** general procedure A, purified by SiO_2 column chromatography (hexane/EtOAc = 8:1) to give black solid**,** 958mg, 77% yield, **^{1}H NMR** (500 MHz, Methanol-d_4) δ 7.85 (d, *J* = 8.5 Hz, 1H), 7.72 (d, *J* = 8.2 Hz, 1H), 7.56 (d, *J* = 8.2 Hz, 1H), 7.30 (t, *J* = 7.9 Hz, 2H), 7.21 (t, *J* = 7.6 Hz, 1H), 7.12 (d, *J* = 7.4 Hz, 1H), 6.63 (d, *J* = 9.0 Hz, 2H), 6.49 (d, *J* = 9.0 Hz, 2H), 3.15 (s, 3H). **^{13}C NMR** (125 MHz, MeOD) δ 148.6, 145.9, 143.7, 134.3, 129.9, 127.3, 125.2, 124.8, 124.6, 124.3, 123.0, 121.8, 116.1, 114.6, 39.8. **HRMS** (ESI) m/z calcd for $C_{17}H_{16}NO^+$ $[M+H]^+$: 250.1226, found 250.1227.



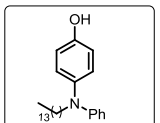
1f

4-((4-methoxyphenyl)(methyl)amino)phenol **(1g):** general procedure A, purified by SiO_2 column chromatography (hexane/EtOAc = 8:1) to give yellowish brown solid, 213 (1 mmol) mg, 93% yield. **^{1}H NMR** (500MHz, Methanol-d4):δ 6.82-6.76(m, 6H), 6.73-6.69(m, 2H), 3.71(s, 3H), 3.12(s, 3H). **^{13}C NMR** (125 MHz, Methanol-d4): δ 155.3, 153.6, 145.6, 144.2, 124.2, 121.3, 116.8, 115.4, 56.0, 41.6. **HRMS** (ESI) m/z calcd for $C_{14}H_{16}NO_2^+$ $[M+H]^+$: 230.1175, found 230.1174.



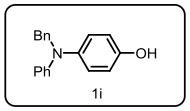
1g

4-(phenyl(tetradecyl)amino)phenol **(1h):** general procedure A, purified by SiO_2 column chromatography (hexane/EtOAc = 10:1) to give yellowish brown solid, 236 mg (1 mmol), 53% yield. **^{1}H NMR** (500MHz, Methanol-d4): δ 7.09 (t, *J* = 7.9 Hz, 2H), 6.97 (d, *J* = 8.4 Hz, 2H), 6.81 (d, *J* = 8.5 Hz, 2H), 6.66 (d, *J* = 8.1 Hz, 3H), 3.58 (t, *J* = 7.6 Hz, 2H), 1.63 (t, *J* = 7.4 Hz, 2H), 1.32 - 1.27 (m, 22H), 0.92 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR** (125 MHz, Methanol-d4): δ 155.6, 150.7, 141.1, 129.7, 129.0, 118.4, 117.2, 116.2, 53.4, 33.1, 30.8, 30.8, 30.8, 30.7, 30.7, 30.6, 30.5, 28.6, 28.1, 23.8, 23.7, 14.5. **HRMS** (ESI) m/z calcd for $C_{26}H_{40}NO^+$ $[M+H]^+$: 382.3104, found 382.3100.



1h

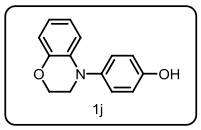
4-(benzyl(phenyl)amino)phenol **(1i):** general procedure A, purified by SiO_2 column chromatography (hexane/EtOAc = 8:1) to give white solid, 205 mg (1mmol), 75% yield. **^{1}H NMR** (500 MHz, Methanol-d4) δ 7.31 (d, *J* = 7.17 Hz, 2H), 7.25 (t, *J* = 7.65 Hz, 2H), 7.17 (t, *J* = 7.14 Hz, 1H), 7.07 - 7.01 (m, 4H), 6.77 (d, *J* = 8.83 Hz, 2H), 6.71 - 6.68 (m, 2H), 6.65 (t, *J* = 7.29 Hz, 1H), 4.84 (s, 2H). **^{13}C NMR**



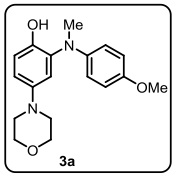
1i

(125 MHz, Methanol-d4) δ 155.5, 150.5, 141.5, 140.9, 129.7, 129.4, 128.3, 127.8, 127.6, 118.9, 117.2, 116.6, 57.6. **HRMS** (ESI) m/z calcd for $C_{19}H_{18}NO^+$ $[M+H]^+$: 276.1382, found 276.1381.

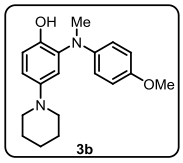
*4-(2,3-dihydro-4H-benzo[b][1,4]oxazin-4-yl)phenol***(1j):** general procedure A, purified by SiO_2 column chromatography (hexane/EtOAc = 8:1) to give brown solid, 737mg, 65% yield. **^{1}H NMR** (500 MHz, Methanol-d_4) δ 7.01 (d, *J* = 8.8 Hz, 2H), 6.80 (d, *J* = 8.7 Hz, 2H), 6.75 - 6.70 (m, 1H), 6.61 - 6.54 (m, 2H), 6.53 - 6.46 (m, 1H), 4.19 (t, 2H), 3.51 (t, 2H). **^{13}C NMR** (125 MHz, MeOD) δ 155.7, 145.7, 140.5, 135.6, 127.8, 121.8, 119.8, 117.5, 117.1, 116.6, 65.5, 50.6. **HRMS** (ESI) m/z calcd for $C_{14}H_{14}NO_2^+$ $[M+H]^+$ 228.1019, found 228.1019.



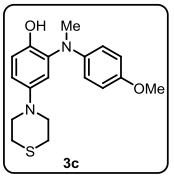
*2-((4-methoxyphenyl)(methyl)amino)-4-morpholinophenol***(3a):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give black oil, 73 mg, 78% yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 6.96 (d, *J* = 8.7 Hz, 1H), 6.84 - 6.75 (m, 3H), 6.73 - 6.61 (m, 3H), 5.87 (s, 1H), 3.81 (d, *J* = 4.6 Hz, 4H), 3.75 (s, 3H), 3.14 (s, 3H), 2.99 (t, *J* = 4.8 Hz, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.3, 146.9, 146.1, 143.5, 116.6, 116.0, 115.5, 114.8, 114.6, 67.0, 55.7, 50.8, 40.4. **HRMS** (ESI) m/z calcd for $C_{18}H_{23}N_2O_3^+$ $[M+H]^+$ 315.1703, found 315.1698.



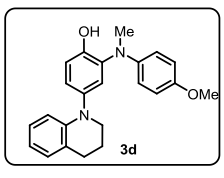
*2-((4-methoxyphenyl)(methyl)amino)-4-(piperidin-1-yl)phenol***(3b)**: purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give black oil, 66 mg, 71 % yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 6.94 (d, *J* = 8.8 Hz, 1H), 6.85 - 6.77 (m, 3H), 6.72 - 6.66 (m, 3H), 5.81 (s, 1H), 3.76 (s, 3H), 3.14 (s, 3H), 3.00 - 2.91 (m, 4H), 1.69 (p, *J* = 5.8 Hz, 4H), 1.51 (t, *J* = 6.0 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.2, 147.3, 146.4, 143.6, 136.3, 117.0, 116.4, 115.7, 115.1, 114.5, 55.6, 52.3, 40.4, 26.0, 24.1. **HRMS** (ESI) m/z calcd for $C_{19}H_{25}N_2O_2^+$ $[M+H]^+$ 313.1911, found 313.1906.



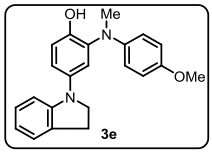
*2-((4-methoxyphenyl)(methyl)amino)-4-thiomorpholinophenol***(3c):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give black solid, 56 mg, 57 % yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ = 6.95 (d, *J* = 8.6 Hz, 1H), 6.80 (d, *J* = 8.2 Hz, 3H), 6.72 - 6.60 (m, 3H), 5.83 (s, 1H), 3.76 (s, 3H), 3.35 - 3.26 (m, 4H), 3.14 (s, 3H), 2.95 - 2.54 (m, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.4, 147.2, 147.0, 143.5, 136.6, 118.1, 116.9, 116.6, 115.4, 114.6, 55.7, 53.8, 40.5, 27.7. **HRMS** (ESI) m/z calcd for $C_{18}H_{23}N_2O_2S^+$ $[M+H]^+$ 331.1475, found 331.1475.



*4-(3,4-dihydroquinolin-1(2H)-yl)-2-((4 methoxyphenyl)(methyl)amino)phenol***(3d):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give black solid, 67 mg, 62 % yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 7.12 - 7.03 (m, 2H), 7.00 (d, *J* = 2.3 Hz, 2H), 6.90 (d, *J* = 7.5 Hz, 1H), 6.84 (d, *J* = 9.2 Hz, 2H), 6.75 (d, *J* = 9.1 Hz, 2H), 6.64 (t, *J* = 6.7 Hz, 1H), 6.53 (d, *J* = 7.8 Hz, 1H), 6.12 (s, 1H), 3.78 (s, 3H), 3.55 (t, *J* = 5.6 Hz, 2H), 3.17 (s, 3H), 2.86 (t, *J* = 6.4 Hz, 2H), 2.05 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.5, 149.7, 145.3, 143.3, 141.5, 137.2, 129.3, 126.4, 125.8, 124.3, 123.0, 117.2, 116.8, 115.8, 114.6, 114.1, 55.6, 51.6, 40.6, 27.8, 22.5. **HRMS** (ESI) m/z calcd for $C_{23}H_{25}N_2O_2^+$ $[M+H]^+$ 361.1911, found 361.1908.

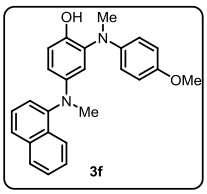


*4-(indolin-1-yl)-2-((4-methoxyphenyl)(methyl)amino)phenol***(3e):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give brown solid, 52 mg, 51 % yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 7.16 - 6.93 (m, 5H), 6.83 (d, *J* = 8.4 Hz, 3H), 6.75 (d, *J* = 8.5 Hz, 2H), 6.70 (t, *J* = 7.3 Hz, 1H), 5.97 (s, 1H), 3.83 (t, *J* = 8.5 Hz, 2H), 3.78 (s, 3H), 3.18 (s, 3H), 3.09 (t, *J* = 8.5

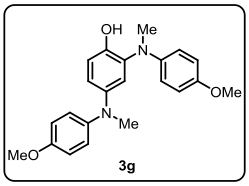


Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.5, 148.3, 147.6, 143.4, 138.2, 136.7, 130.6, 127.1, 124.8, 118.8, 118.2, 117.2, 116.7, 115.5, 114.9, 114.6, 113.7, 107.2, 55.6, 53.0, 40.5, 28.2. **HRMS** (ESI) m/z calcd for $C_{22}H_{23}N_2O_2^+$ $[M+H]^+$ 347.1754, found 347.1751.

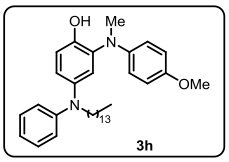
*4-(4H-benzo[b][1,4]oxazin-4-yl)-2-((4-methoxyphenyl)(methyl)amino)phenol***(3f):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give black solid, 60 mg, 56 % yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 7.13 (d, *J* = 8.8 Hz, 3H), 6.93 - 6.81 (m, 5H), 6.77 - 6.64 (m, 5H), 5.37 (s, 1H), 4.34 (t, *J* = 4.4 Hz, 3H), 3.65 (t, *J* = 4.4 Hz, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 152.8, 144.3, 140.1, 134.1, 126.6, 121.1, 119.2, 119.2, 116.8, 116.4, 116.3, 115.7, 64.5, 49.4. **HRMS** (ESI) m/z calcd for $C_{22}H_{21}N_2O_3^+$ $[M+H]^+$ 361.1547, found 361.1547.



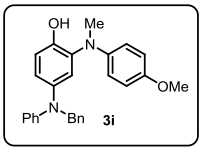
*2,4-bis((4-methoxyphenyl)(methyl)amino)phenol***(3g)**: purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give light red solid, 76 mg, 70 % yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 6.96 - 6.89 (m, 3H), 6.85 - 6.78 (m, 5H), 6.75 - 6.67 (m, 3H), 5.84 (s, 1H), 3.78 (s, 3H), 3.76 (s, 3H), 3.17 (s, 3H), 3.14 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 154.3, 153.2, 147.1, 143.9, 143.4, 143.4, 136.5, 121.6, 119.8, 118.0, 116.4, 115.4, 114.5, 55.6, 55.5, 41.0, 40.4. **HRMS** (ESI) m/z calcd for $C_{22}H_{25}N_2O_3^+$ $[M+H]^+$ 365.1860, found 365.1858.



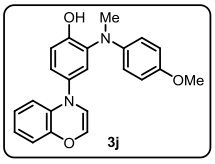
*2-((4-methoxyphenyl)(methyl)amino)-4-(phenyl(tetradecyl)amino)phenol***(3h):** purified by SiO_2 column chromatography (hexane/EtOAc = 20:1) to give bromn solid, 70 mg, 45 % yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.20 – 7.11 (m, 2H), 7.06 – 6.94 (m, 2H), 6.89 (d, *J* = 2.4 Hz, 1H), 6.81 (d, *J* = 9.1 Hz, 2H), 6.76 – 6.68 (m, 5H), 6.07 (s, 1H), 3.77 (s, 3H), 3.66 – 3.49 (m, 2H), 3.15 (s, 3H), 1.61 (t, *J* = 7.5 Hz, 2H), 1.38 – 1.09 (m, 24H), 0.90 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.5, 149.5, 148.9, 143.4, 141.0, 137.2, 131.7, 129.0, 125.7, 124.3, 117.8, 116.8, 115.9, 115.7, 115.4, 114.6, 55.9, 55.7, 52.5, 40.7, 32.0, 29.8, 29.7, 29.7, 29.7, 29.7, 29.5, 29.4, 27.5, 27.1, 22.8, 14.2. **HRMS** (ESI) m/z $C_{34}H_{49}N_2O_2^+$ $[M+H]^+$ 517.3789, found 517.3790.



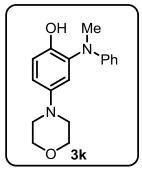
*4-(benzyl(phenyl)amino)-2-((4-methoxyphenyl)(methyl)amino)phenol***(3i)**: purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give black solid, 79 mg, 65 % yield.**^{1}H NMR** (500 MHz, Chloroform-*d*) δ 7.28 - 7.20 (m, 4H), 7.15 (t, *J* = 7.1 Hz, 1H), 7.06 (t, 2H), 6.93 - 6.86 (m, 2H), 6.83 (d, *J* = 2.5 Hz, 1H), 6.78 - 6.71 (m, 4H), 6.70 - 6.59 (m, 3H), 4.80 (s, 2H), 3.69 (s, 3H), 3.06 (s, 3H). **^{13}C NMR** (125 MHz, MeOD) δ 154.0, 151.2, 150.4, 145.2, 142.0, 140.7, 138.8, 129.8, 129.4, 128.0, 127.7, 125.8, 124.9, 119.4, 118.1, 117.2, 117.2, 115.8, 115.5, 115.3, 57.4, 56.1, 40.0. **HRMS** (ESI) m/z calcd for $C_{27}H_{27}N_2O_2^+$ $[M+H]^+$ 411.2067, found 411.2066.



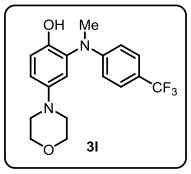
*2-((4-methoxyphenyl)(methyl)amino)-4-(methyl(naphthalen-1-yl)amino)phenol***(3j):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give black solid, 58 mg, 51 % yield. **^{1}H NMR** (500 MHz, Methanol-d_4) δ 7.83 (dd, *J* = 14.5, 8.3 Hz, 2H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.47 - 7.31 (m, 3H), 7.22 (d, *J* = 7.3 Hz, 1H), 6.76 (d, *J* = 8.8 Hz, 1H), 6.61 (d, *J* = 9.2 Hz, 2H), 6.52 (d, *J* = 9.1 Hz, 2H), 6.47 (dd, *J* = 8.8, 2.9 Hz, 1H), 6.30 (d, *J* = 2.9 Hz, 1H), 3.65 (s, 3H), 3.24 (s, 3H), 2.99 (s, 3H). **^{13}C NMR** (125 MHz, MeOD) δ 153.7, 147.7, 147.0, 146.5, 138.0, 136.5, 131.9, 129.4, 127.3, 126.9, 126.7, 126.5, 125.2, 123.8, 117.8, 116.8, 116.1, 115.6, 115.2, 56.1, 41.7, 39.9. **HRMS** (ESI) m/z calcd for $C_{25}H_{25}N_2O_2^+$ $[M+H]^+$ 385.1911, found 385.1908.



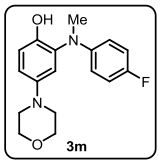
2-(methyl(phenyl)amino)-4-morpholinophenol **(3k):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give black oil, 53 mg**,** 62 % yield **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 7.23 (t, *J* = 7.8 Hz, 2H), 6.99 (d, *J* = 8.9 Hz, 1H), 6.86 - 6.81 (m, 2H), 6.72 (d, *J* = 8.2 Hz, 2H), 6.68 (d, *J* = 2.8 Hz, 1H), 5.71 (s, 1H), 3.82 (t, *J* = 4.7 Hz, 4H), 3.20 (s, 3H), 3.01 (t, *J* = 4.7 Hz, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 149.1, 146.9, 146.2, 135.5, 129.1, 119.0, 116.5, 115.7, 115.2, 114.5, 66.9, 50.6, 39.7. **HRMS** (ESI) m/z calcd for $C_{17}H_{21}N_2O_2^+$ $[M+H]^+$ 285.1598, found 285.1599.



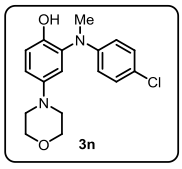
2-(methyl(4-(trifluoromethyl)phenyl)amino)-4-morpholinophenol **(3l)**: purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give light yellow solid, 52 mg, 50 % yield. **^{1}H NMR** (500 MHz, Methanol-d_4) δ 7.34 (d, *J* = 8.6 Hz, 2H), 6.95 - 6.80 (m, 2H), 6.72 (d, *J* = 2.8 Hz, 1H), 6.65 (d, *J* = 8.6 Hz, 2H), 3.84 - 3.67 (m, 4H), 3.22 (s, 3H), 3.05 - 2.76 (m, 4H). **^{13}C NMR** (125 MHz, MeOD) δ 153.3, 149.2, 147.1, 135.2, 127.7 (d, J_{CF} = 267.5 Hz), 127.0 (q, J_{CF} = 3.75 Hz), 119.2 (d, J_{CF} = 32.5Hz), 118.5, 118.4, 118.2, 113.5, 68.0, 52.1, 39.4. **HRMS** (ESI) m/z calcd for $C_{18}H_{20}F_3N_2O_2^+$ $[M+H]^+$ 353.1471, found 353.1472.



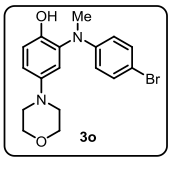
2-((4-fluorophenyl)(methyl)amino)-4-morpholinophenol **(3m):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give red oil, 42 mg, 47 % yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 6.98 (d, *J* = 8.8 Hz, 1H), 6.91 (t, *J* = 8.7 Hz, 2H), 6.81 (dd, *J* = 8.9, 2.9 Hz, 1H), 6.70 - 6.60 (m, 3H), 5.78 (s, 1H), 3.98 - 3.53 (m, 4H), 3.16 (s, 3H), 3.06 - 2.92 (m, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 157.7 (d, J_{CF} = 236.3 Hz), 146.8, 146.2, 145.6(d, J_{CF} = 1.3 Hz), 135.9, 116.4, 115.9(d, J_{CF} = 7.5 Hz), 115.7, 115.6(d, J_{CF} = 22.5 Hz), 114.8, 66.9, 50.6, 40.2. **HRMS** (ESI) m/z calcd for $C_{17}H_{20}FN_2O_2^+$ $[M+H]^+$ 303.1503, found 303.1503.



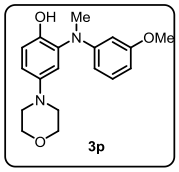
2-((4-chlorophenyl)(methyl)amino)-4-morpholinophenol **(3n):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give yellow solid, 53 mg, 55 % yield. **^{1}H NMR** (500 MHz, Methanol-d_4) δ 7.05 (d, *J* = 9.1 Hz, 2H), 6.88 (d, *J* = 8.9 Hz, 1H), 6.80 (dd, *J* = 8.9, 2.9 Hz, 1H), 6.69 (d, *J* = 3.0 Hz, 1H), 6.56 (d, *J* = 9.1 Hz, 2H), 3.81 - 3.67 (m, 4H), 3.15 (s, 3H), 2.99 - 2.83 (m, 4H). **^{13}C NMR** (125 MHz, MeOD) δ 149.6, 149.2, 147.0, 136.3, 129.4, 122.9, 118.3, 118.1, 117.7, 115.8, 67.9, 52.1, 39.6. **HRMS** (ESI) m/z calcd for $C_{17}H_{20}ClN_2O_2^+$ $[M+H]^+$ 319.1208, found 319.1205.



2-((4-bromophenyl)(methyl)amino)-4-morpholinophenol **(3o):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give light yellow solid, 62 mg, 57 % yield. 434.4 mg, 40% yield 3mmol scale. **^{1}H NMR** (500 MHz, Methanol-d_4) δ 7.21 (d, *J* = 9.0 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 1H), 6.87 - 6.79 (m, 1H), 6.72 (d, *J* = 2.9 Hz, 1H), 6.54 (d, *J* = 9.1 Hz, 2H), 3.85 - 3.74 (m, 4H), 3.17 (s, 3H), 3.02 - 2.92 (m, 4H). **^{13}C NMR** (125 MHz, MeOD) δ 150.1, 149.2, 147.1, 136.2, 132.4, 118.3, 118.2, 117.8, 116.3, 110.0, 68.0, 52.2, 39.5. **HRMS** (ESI) m/z calcd for $C_{17}H_{20}BrN_2O_2^+$ $[M+H]^+$ 363.0703, found 363.0703.

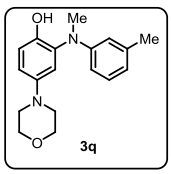


2-((3-methoxyphenyl)(methyl)amino)-4-morpholinophenol **(3p):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1) to give yellow solid, 62 mg, 66 % yield. **^{1}H NMR** (500 MHz, Methanol-d_4) δ 7.02 (t, *J* = 8.2 Hz, 1H), 6.88 (d, *J* = 8.7 Hz, 1H), 6.81 (dd, *J* = 8.8, 2.9 Hz, 1H), 6.71 (d, *J* = 2.9 Hz, 1H), 6.30 - 6.23 (m, 2H), 6.18 (t, *J* = 2.4 Hz, 1H), 3.79 - 3.74 (m, 4H), 3.67 (s, 3H), 3.15 (s, 3H), 2.97 - 2.90 (m, 4H). **^{13}C NMR** (125 MHz, MeOD) δ 160.5, 150.9, 147.9, 145.6, 135.4, 129.0, 117.0, 116.7, 116.2, 106.6, 102.2, 99.8, 66.6, 54.0, 50.9, 38.2. **HRMS** (ESI) m/z calcd for



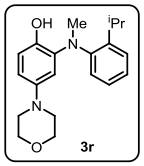
$C_{18}H_{23}N_2O_3^+$ [M+H]$^+$ 315.1703, found 315.1698.

2-(methyl(m-tolyl)amino)-4-morpholinophenol **(3q):** purified by SiO_2 column chromatography (hexane/EtOAc = 15:1)



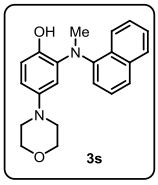
to give brown oil, 59 mg, 66 % yield.**^{1}H NMR** (500 MHz, Methanol-d_4) δ 6.99 (t, *J* = 7.8 Hz, 1H), 6.88 (d, *J* = 8.8 Hz, 1H), 6.84 - 6.77 (m, 1H), 6.69 (d, *J* = 2.9 Hz, 1H), 6.51 (d, *J* = 8.2 Hz, 1H), 6.49 - 6.42 (m, 2H), 3.80 - 3.64 (m, 4H), 3.14 (s, 3H), 3.03 - 2.83 (m, 4H), 2.21 (s, 3H). **^{13}C NMR** (125 MHz, MeOD) δ 151.0, 149.3, 147.0, 139.3, 137.2, 129.6, 119.4, 118.2, 118.0, 117.4, 115.6, 112.3, 68.0, 52.3, 39.6, 21.9. **HRMS** (ESI) m/z calcd for $C_{18}H_{23}N_2O_2$+ [M+H]+ 299.1754, found 299.1757.

2-((2-isopropylphenyl)(methyl)amino)-4-morpholinophenol **(3r):** purified by SiO_2 column chromatography



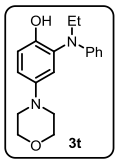
(hexane/EtOAc = 15:1) to give bromn solid, 38 mg, 39 % yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 6.95 - 6.84 (m, 5H), 6.79 (dd, *J* = 8.6, 2.8 Hz, 1H), 6.59 (d, *J* = 8.6 Hz, 1H), 3.92 - 3.83 (m, 4H), 3.59 (s, 1H), 3.09 (dd, *J* = 5.7, 3.9 Hz, 4H), 2.88 (s, 3H), 2.87 - 2.82 (m, 1H), 1.23 (d, *J* = 6.8 Hz, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 152.6, 148.6, 146.6, 142.1, 134.1, 118.3, 117.4, 117.1, 116.8, 110.6, 67.0, 50.4, 31.4, 27.2, 22.2. **HRMS** (ESI) m/z calcd for $C_{20}H_{27}N_2O_2^+$ [M+H]$^+$ 327.2067, found 327.2065.

2-(methyl(naphthalen-1-yl)amino)-4-morpholinophenol **(3s):** purified by SiO_2 column chromatography



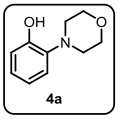
(hexane/EtOAc = 15:1) to give dark green solid, 69 mg, 69 % yield. **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 8.16 - 8.09 (m, 1H), 7.88 - 7.75 (m, 1H), 7.52 - 7.41 (m, 2H), 6.99 (d, *J* = 8.2 Hz, 1H), 6.93 (d, *J* = 9.1 Hz, 2H), 6.86 (d, *J* = 9.1 Hz, 2H), 6.52 (d, *J* = 8.2 Hz, 1H), 4.32 (s, 1H), 4.06 - 3.72 (m, 4H), 3.17 - 3.03 (m, 4H), 3.00 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.2, 146.5, 144.2, 141.2, 127.7, 125.8, 125.3, 124.5, 122.8, 120.1, 117.8, 117.4, 115.7, 103.3, 66.9, 50.4, 31.2. **HRMS** (ESI) m/z calcd for $C_{21}H_{23}N_2O_2^+$ [M+H]$^+$ 335.1754, found 335.1753.

2-(ethyl(phenyl)amino)-4-morpholinophenol **(3t)**, purified by SiO_2 column chromatography (hexane/EtOAc = 15:1)



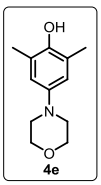
to give black oil, 56mg, 63 % yield. **^{1}H NMR** (400 MHz, Methanol-d_4) δ 7.14 – 7.01 (m, 2H), 6.93 – 6.79 (m, 2H), 6.69 (d, *J* = 2.8 Hz, 1H), 6.61 (t, *J* = 9.1 Hz, 3H), 3.81 – 3.71 (m, 4H), 3.61 (q, *J* = 7.1 Hz, 2H), 3.01 – 2.87 (m, 4H), 1.16 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 148.3, 147.8, 146.2, 133.2, 129.3, 118.7, 116.8, 116.7, 115.8, 114.6, 67.0, 50.8, 45.8, 12.7. **HRMS** (ESI) m/z calcd for $C_{18}H_{23}N_2O_2^+$ [M+H]$^+$ 299.1754, found 299.1755.

2-morpholinophenol (**4a**). Known compound,[3] General Procedure A, purified by SiO_2 column chromatography



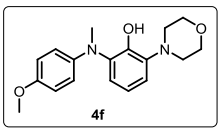
(hexane/EtOAc = 20:1), yellow solid, 134 mg 75% yield (1 mmol). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.17 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.09 (td, *J* = 7.8, 1.5 Hz, 1H), 6.96 (dd, *J* = 8.1, 1.5 Hz, 1H), 6.88 (td, *J* = 7.6, 1.5 Hz, 1H), 3.87 (dd, *J* = 5.7, 3.6 Hz, 4H), 3.02 – 2.65 (m, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ151.47, 138.69, 126.71, 126.69, 121.45, 120.21, 114.35, 67.58, 52.83.

2,6-dimethyl-4-morpholinophenol (**4e**). Known compound,[1] General Procedure B, purified by SiO_2 column



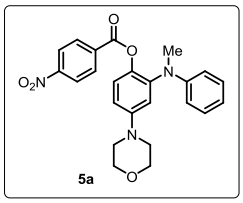
chromatography (hexane/EtOAc = 20:1), orange solid, 137 mg 66% yield (1 mmol). **^{1}H NMR** (400 MHz, Methanol-d_4) δ 6.70 (d, J = 3.2 Hz, 2H), 3.90 (d, J = 4.7 Hz, 4H), 3.08 (d, J = 4.7 Hz, 4H), 2.32 (d, J = 3.4 Hz, 6H). **^{13}C NMR** (125 MHz, MeOD) δ 147.48, 144.49, 125.33, 117.28, 66.77, 51.20, 16.09, 16.07.

2-((4-methoxyphenyl)(methyl)amino)-6-morpholinophenol (**4f**), purified by SiO_2 column chromatography



(hexane/EtOAc = 20:1) to give yellow oil, 16mg, 17 % yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.84 – 6.74 (m, 3H), 6.71 – 6.55 (m, 4H), 4.02 (br, 1H), 3.76 (s, 4H), 3.72 (s, 3H), 3.13 (s, 4H), 2.80 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 152.50, 151.84, 143.54, 140.78, 135.98, 115.58, 114.57, 113.12, 112.31, 111.32, 55.88, 55.75, 39.46, 31.28. **HRMS** (ESI) m/z calcd for $C_{18}H_{23}N_2O_3^+$ $[M+H]^+$ 315.1703, found 315.1699.

*2-(methyl(phenyl)amino)-4-morpholinophenyl 4-nitrobenzoate***(5a):** purified by SiO_2 column chromatography



(hexane/EtOAc = 20:1) to give red solid**. ^{1}H NMR** (400 MHz, Chloroform-*d*) δ = 8.13 (d, *J* = 8.7 Hz, 2H), 7.81 (d, *J* = 8.9 Hz, 2H), 7.17 (d, *J* = 8.8 Hz, 1H), 7.14 - 7.08 (m, 2H), 6.90 (d, *J* = 2.9 Hz, 1H), 6.81 (dd, *J* = 8.9, 2.9 Hz, 1H), 6.74 (t, *J* = 7.3 Hz, 1H), 6.68 (d, *J* = 7.6 Hz, 2H), 3.96 - 3.82 (t, 4H), 3.25 (s, *J* = 6.0, 3H), 3.22 - 3.12 (t, *J* = 6.0, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.3, 150.8, 150.4, 148.3, 140.5, 139.2, 134.5, 130.9, 128.8, 123.5, 123.2, 118.6, 114.9, 114.8, 112.6, 66.7, 49.2, 39.6. **HRMS** (ESI) m/z calcd for $C_{24}H_{24}N_3O_5^+$ $[M+H]^+$ 434.17105, found 434.17108.

6.2 X-ray structure of **5a.**

Suitable crystals of compound **5a** was obtained by slowly evaporating a mixture of $CHCl_3$ and n-hexane solution ($CHCl_3$: n-hexane = 1:8) at ambient temperature. For 4 days, a colorless crystal of **5a** was mounted on a glass fiber at a random orientation. The data were collected by a diffractometer Rigaku Oxford Diffraction Supernova Dual Source, Cu at Zero equipped with an AtlasS2 CCD using Cu Kα radiation (1.54178 Å) by using a w scan mode. The structures were solved by direct methods using Olex2 software, and the nonhydrogen atoms were located from the trial structure and then refined anisotropically with XL using a full-matrix least squares procedure based on F2. The weighted R factor, wR and goodness-of-fit S values were obtained based on F2.The hydrogen atom positions were fixed geometrically at the calculated distances and allowed to ride on the parent atoms.

Compound **5a** was collected at 100 K on a Rigaku Oxford Diffraction Supernova Dual Source, Cu at Zero equipped with an AtlasS2 CCD using Cu Kα radiation. The data were collected and processed using CrysAlisPro. The structures were solved by direct methods using Olex2 software, and the non-hydrogen atoms were located from the trial structure and then refined anisotropically with SHELXL-2018 using a full-matrix least squares procedure based on F2. The weighted R factor, wR and goodness-of-fit S values were obtained based on F2.The hydrogen atom positions were fixed geometrically at the calculated distances and allowed to ride on their parent atoms. Crystallographic data for the structure reported in this paper have been deposited at the Cambridge Crystallographic Data Center and allocated with the deposition numbers: CCDC 2090410.

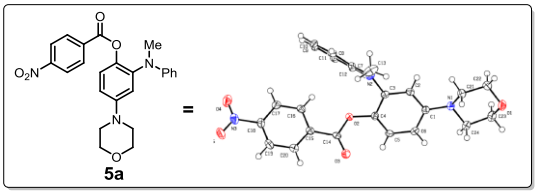


Table S4 Crystal data and structure refinement for 5a.	
Identification code	2090410
Empirical formula	$C_{24}H_{23}N_3O_5$
Formula weight	433.45
Temperature/K	100.00(10)

Crystal system	orthorhombic
Space group	Pbca
a/Å	15.1398(8)
b/Å	11.4003(6)
c/Å	24.1150(15)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	4162.2(4)
Z	8
ρ_{calc}g/cm^3	1.383
μ/mm^{-1}	0.098
F(000)	1824.0
Crystal size/mm^3	0.14 × 0.13 × 0.12
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.318 to 49.99
Index ranges	-17 ≤ h ≤ 18, -13 ≤ k ≤ 9, -28 ≤ l ≤ 23
Reflections collected	10556
Independent reflections	3660 [R_{int} = 0.0299, R_{sigma} = 0.0357]
Data/restraints/parameters	3660/0/290
Goodness-of-fit on F^2	1.079
Final R indexes [I>=2σ (I)]	R_1 = 0.0415, wR_2 = 0.0907
Final R indexes [all data]	R_1 = 0.0512, wR_2 = 0.0953
Largest diff. peak/hole / e Å^{-3}	0.17/-0.24

Crystal structure determination of 5a.

Crystal Data for $C_{24}H_{23}N_3O_5$ (*M* =433.45 g/mol): orthorhombic, space group Pbca (no. 61), *a* = 15.1398(8) Å, *b* = 11.4003(6) Å, *c* = 24.1150(15) Å, *V* = 4162.2(4) Å^3, *Z* = 8, *T* = 100.00(10) K, μ(MoKα) = 0.098 mm^{-1}, *Dcalc* = 1.383 g/cm^3, 10556 reflections measured (4.318° ≤ 2Θ ≤ 49.99°), 3660 unique (R_{int} = 0.0299, R_{sigma} = 0.0357) which were used in all calculations. The final R_1 was 0.0415 (I > 2σ(I)) and wR_2 was 0.0953 (all data).

Table S5 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 5a. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ}tensor.

Atom	*x*	*y*	*z*	U(eq)
O2	6260.0(7)	3849.8(10)	6342.3(4)	22.8(3)
O3	5425.2(7)	4967.3(10)	6913.6(5)	27.1(3)
O1	8209.8(8)	-866.0(11)	8752.9(5)	30.4(3)
O4	5445.1(8)	7299.8(12)	4103.4(5)	37.3(3)
O5	4778.1(8)	8591.0(12)	4616.7(6)	41.5(4)
N1	7550.8(8)	500.4(12)	7844.6(5)	20.9(3)
N2	8043.4(8)	3340.1(12)	6378.2(5)	21.6(3)

N3	5173.2(9)	7659.6(14)	4553.8(6)	29.4(4)
C3	7445.4(10)	2783.0(14)	6746.7(6)	17.6(3)
C14	5738.3(10)	4777.0(14)	6462.5(7)	18.8(4)
C7	7889.4(9)	3328.6(13)	5807.0(6)	18.2(4)
C4	6540.9(10)	3048.4(14)	6748.5(6)	18.6(4)
C12	7368.0(10)	2443.2(14)	5570.5(6)	19.9(4)
C6	6295.4(10)	1637.2(14)	7472.3(6)	19.3(4)
C1	7203.2(10)	1370.3(13)	7493.7(6)	18.1(3)
C15	5610.8(9)	5521.0(14)	5959.4(7)	18.5(4)
C2	7754.9(10)	1950.6(14)	7117.4(6)	19.4(4)
C16	6010.2(10)	5246.7(14)	5457.4(7)	20.4(4)
C5	5973.6(10)	2459.4(14)	7097.2(6)	21.2(4)
C17	5870.7(10)	5950.9(14)	4996.7(7)	22.4(4)
C18	5334.8(10)	6924.6(15)	5049.8(7)	22.4(4)
C8	8268.6(11)	4164.1(14)	5453.7(7)	24.5(4)
C11	7213.5(11)	2423.2(16)	5006.5(7)	25.4(4)
C20	5080.3(10)	6518.7(14)	6002.6(7)	23.3(4)
C19	4941.4(10)	7230.2(15)	5546.7(7)	25.8(4)
C24	6955.0(11)	-62.1(15)	8240.6(7)	24.2(4)
C21	8418.7(10)	734.4(15)	8096.8(7)	25.6(4)
C9	8100.0(12)	4130.6(16)	4887.3(7)	31.3(4)
C10	7567.7(12)	3271.2(16)	4661.4(7)	30.4(4)
C23	7391.5(12)	-1131.6(15)	8489.8(7)	30.2(4)
C22	8794.6(11)	-356.7(16)	8357.3(7)	30.4(4)
C13	8672.4(12)	4162.0(18)	6611.2(8)	39.8(5)

Table S6 Anisotropic Displacement Parameters ($Å^2×10^3$) for 5a. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O2	24.4(6)	22.8(6)	21.4(6)	4.6(5)	4.0(5)	8.5(5)
O3	31.7(6)	28.0(7)	21.6(6)	0.2(5)	4.1(5)	7.8(5)
O1	38.4(7)	32.4(7)	20.4(6)	4.7(6)	-2.4(5)	7.3(6)
O4	37.6(7)	45.2(8)	29.2(7)	11.1(6)	-6.5(6)	-3.6(6)
O5	33.3(7)	35.0(8)	56.2(9)	18.6(7)	-2.9(6)	9.7(6)
N1	22.0(7)	22.1(7)	18.7(7)	2.5(6)	-1.3(6)	1.0(6)
N2	21.2(7)	23.5(8)	20.1(7)	0.4(6)	0.3(6)	-7.3(6)
N3	19.5(7)	31.3(9)	37.3(9)	10.6(7)	-7.1(7)	-4.2(7)
C3	19.1(8)	18.8(9)	14.9(8)	-3.1(7)	0.3(7)	-1.9(7)
C14	13.5(7)	18.6(9)	24.2(9)	-2.6(7)	-1.2(7)	-0.7(7)

C7	16.7(7)	17.5(9)	20.5(8)	2.1(7)	5.0(7)	3.9(6)
C4	23.1(8)	17.5(8)	15.1(8)	0.3(7)	-0.4(7)	2.3(7)
C12	20.2(8)	19.1(9)	20.4(8)	1.9(7)	2.9(7)	1.4(7)
C6	21.0(8)	20.6(9)	16.3(8)	-0.6(7)	3.4(7)	-2.3(7)
C1	22.2(8)	17.3(8)	15.0(8)	-2.4(7)	-0.5(7)	0.5(7)
C15	12.8(7)	18.4(9)	24.2(8)	-0.2(7)	-1.9(7)	-2.4(6)
C2	16.6(7)	22.0(9)	19.5(8)	-3.4(7)	-1.7(7)	1.0(7)
C16	15.4(7)	18.8(9)	27.1(9)	0.1(7)	-0.9(7)	-0.9(6)
C5	17.7(8)	25.0(9)	20.9(8)	-1.8(7)	1.7(7)	3.7(7)
C17	17.4(8)	26.8(10)	23.0(9)	1.6(7)	-0.6(7)	-4.1(7)
C18	16.7(7)	22.8(9)	27.7(9)	7.5(7)	-6.7(7)	-5.3(7)
C8	26.0(9)	15.7(9)	31.9(10)	2.0(7)	9.3(8)	3.4(7)
C11	28.1(9)	24.9(9)	23.3(9)	-5.1(7)	-2.0(8)	9.6(8)
C20	20.4(8)	24.2(9)	25.3(9)	-1.9(7)	0.5(7)	1.4(7)
C19	20.3(8)	21.2(9)	35.8(10)	2.3(8)	-4.3(8)	2.9(7)
C24	28.3(9)	24.4(10)	19.7(8)	2.2(7)	0.3(7)	-2.2(7)
C21	23.3(8)	30.0(10)	23.4(9)	2.8(8)	-2.0(7)	2.8(7)
C9	41.0(10)	23.5(10)	29.6(10)	11.0(8)	19.1(9)	13.7(8)
C10	41.3(10)	32.0(11)	17.9(8)	2.5(8)	4.0(8)	18.8(9)
C23	42.8(10)	24.2(10)	23.7(9)	2.2(8)	0.4(8)	0.0(8)
C22	29.3(9)	35.8(11)	26.0(9)	3.3(8)	1.5(8)	10.0(8)
C13	38.2(10)	49.2(13)	32.0(10)	-12.6(9)	7.2(9)	-24.8(10)

Table S7 Bond Lengths for 5a.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O2	C14	1.3510(19)	C7	C8	1.401(2)
O2	C4	1.4053(18)	C4	C5	1.377(2)
O3	C14	1.2063(19)	C12	C11	1.380(2)
O1	C23	1.424(2)	C6	C1	1.409(2)
O1	C22	1.425(2)	C6	C5	1.391(2)
O4	N3	1.2317(19)	C1	C2	1.399(2)
O5	N3	1.2282(19)	C15	C16	1.389(2)
N1	C1	1.406(2)	C15	C20	1.396(2)
N1	C24	1.462(2)	C16	C17	1.387(2)
N1	C21	1.472(2)	C17	C18	1.381(2)
N2	C3	1.4188(19)	C18	C19	1.383(2)
N2	C7	1.397(2)	C8	C9	1.390(2)
N2	C13	1.449(2)	C11	C10	1.384(2)
N3	C18	1.481(2)	C20	C19	1.382(2)

C3	C4	1.402(2)	C24	C23	1.511(2)
C3	C2	1.385(2)	C21	C22	1.505(2)
C14	C15	1.493(2)	C9	C10	1.381(3)
C7	C12	1.403(2)			

Table S8 Bond Angles for 5a.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C14	O2	C4	122.42(12)	C5	C6	C1	120.75(14)
C23	O1	C22	109.19(13)	N1	C1	C6	122.66(14)
C1	N1	C24	118.15(12)	C2	C1	N1	120.02(13)
C1	N1	C21	117.06(13)	C2	C1	C6	117.16(14)
C24	N1	C21	111.13(12)	C16	C15	C14	121.61(14)
C3	N2	C13	117.77(13)	C16	C15	C20	119.93(15)
C7	N2	C3	120.46(12)	C20	C15	C14	118.46(14)
C7	N2	C13	119.87(14)	C3	C2	C1	122.71(14)
O4	N3	C18	117.95(15)	C17	C16	C15	120.10(15)
O5	N3	O4	124.02(15)	C4	C5	C6	120.49(14)
O5	N3	C18	118.03(15)	C18	C17	C16	118.73(15)
C4	C3	N2	121.90(14)	C17	C18	N3	118.49(15)
C2	C3	N2	119.67(13)	C17	C18	C19	122.41(15)
C2	C3	C4	118.43(14)	C19	C18	N3	119.10(15)
O2	C14	C15	110.23(13)	C9	C8	C7	120.24(16)
O3	C14	O2	124.33(15)	C12	C11	C10	121.02(16)
O3	C14	C15	125.44(14)	C19	C20	C15	120.40(15)
N2	C7	C12	120.11(14)	C20	C19	C18	118.42(15)
N2	C7	C8	121.66(14)	N1	C24	C23	110.11(13)
C8	C7	C12	118.20(15)	N1	C21	C22	111.11(14)
C3	C4	O2	115.70(13)	C10	C9	C8	120.95(16)
C5	C4	O2	123.64(14)	C9	C10	C11	119.00(16)
C5	C4	C3	120.38(14)	O1	C23	C24	112.70(14)
C11	C12	C7	120.53(15)	O1	C22	C21	112.41(13)

Table S9 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 5a.

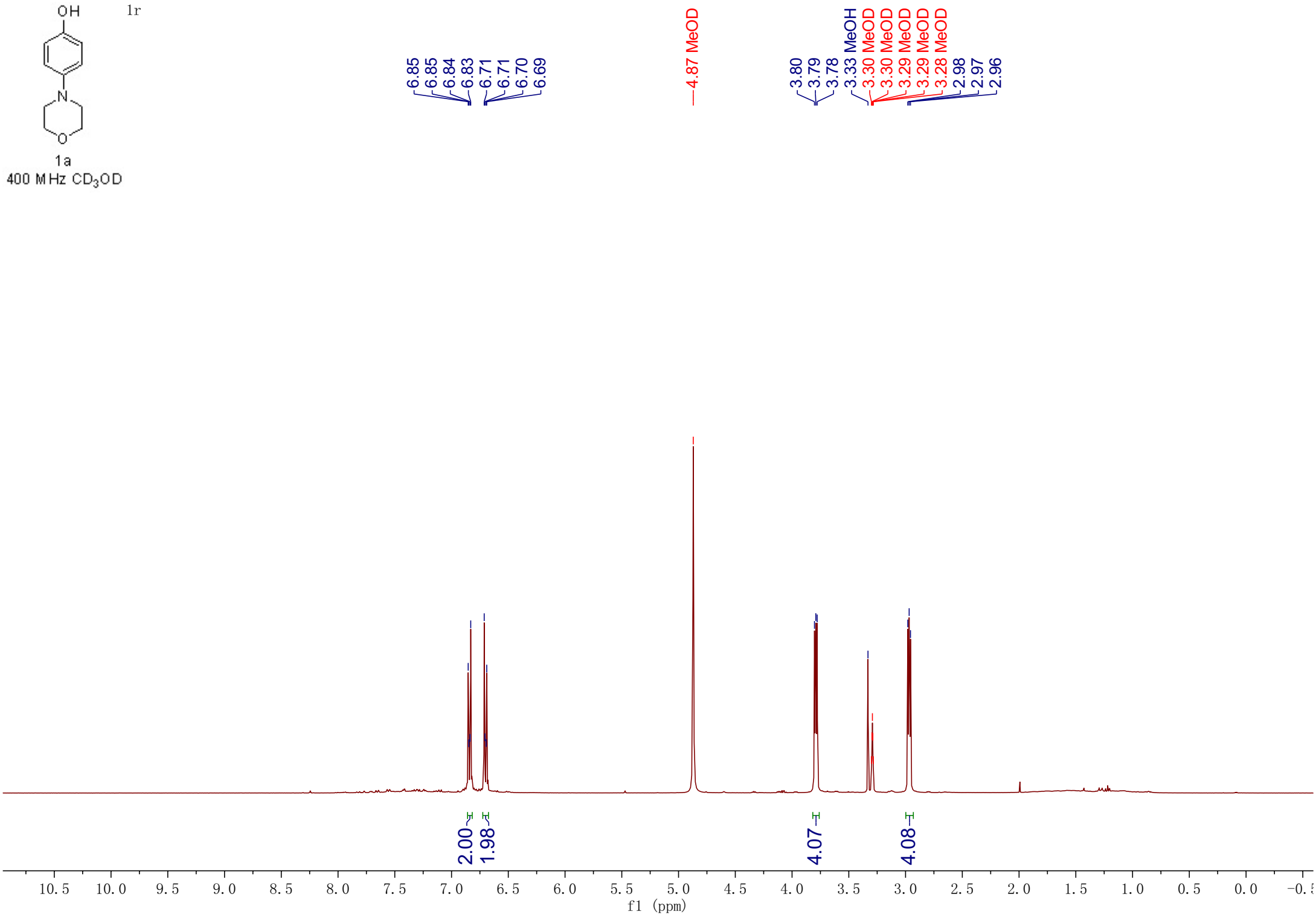
Atom	*x*	*y*	*z*	U(eq)
H12	7124.51	1864.95	5795.48	24
H6	5906.82	1259.29	7711.64	23
H2	8353.88	1769.96	7116.21	23

H16	6371.86	4589.78	5430.12	25
H5	5370.7	2612.59	7081.88	25
H17	6132.95	5770.89	4658.52	27
H8	8634.48	4743.25	5598.63	29
H11	6865.88	1830.6	4856.07	31
H20	4818.88	6705.75	6340.23	28
H19	4591.59	7898.37	5573.53	31
H24A	6414.29	-291.41	8054	29
H24B	6803.8	487.79	8532.44	29
H21A	8358.23	1339.28	8377.13	31
H21B	8821.6	1020.04	7814.72	31
H9	8349.42	4695.25	4657.14	38
H10	7449.02	3262.33	4283.06	36
H23A	6995.12	-1480.08	8759.45	36
H23B	7493.35	-1705.15	8199.6	36
H22A	8918.76	-926.54	8068.92	36
H22B	9348.06	-164.35	8538.31	36
H13A	8445.18	4945.02	6577.99	60
H13B	9221.93	4104.02	6414.49	60
H13C	8766.73	3981.37	6995.5	60

7. References

1. S. Urgaonkar and J. G. Verkade, *Adv. Synth. Catal.*, 2004, **346**, 611-616.
2. L. Li, A. Wang, B. Wang, M. Liu, K. Lv, Z. Tao, C. Ma, X. Ma, B. Han, A. Wang and Y. Lu, *Chinese Chem. Lett*., 2020, **31**, 409-412.
3. C. Dinesh, K. Vinayak, P. Sunil Kumar, Pat., WO 2014/111871 A1.
4. J. Tappen, I. Rodstein, K. McGuire, A. Großjohann, J. Löffler, T. Scherpf and V. H. Gessner, *Chem. Eur. J.,* 2020, **26**, 4281-4288.

8. NMR Spectra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3.29 MeOD
3.28 MeOD
2.98
2.97
2.96
2.00
1.98
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4.08
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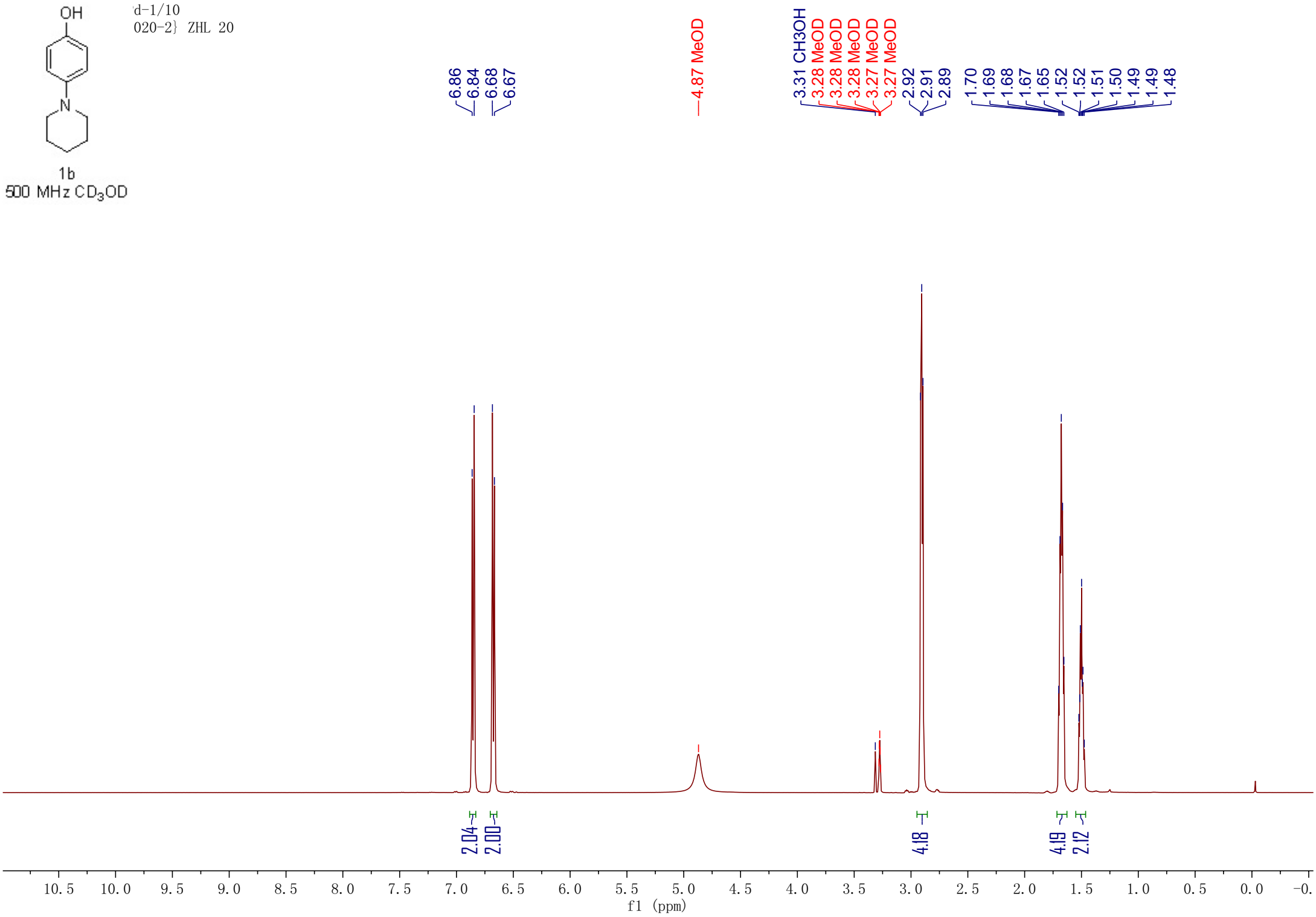
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3.28 MeOD
3.28 MeOD
3.28 MeOD
3.27 MeOD
3.27 MeOD
2.92
2.91
2.89
1.70
1.69
1.68
1.67
1.65
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1.52
1.51
1.50
1.49
1.49
1.48
2.04
2.00
4.18
4.19
2.12
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10.0
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9.0
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8.0
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5.0
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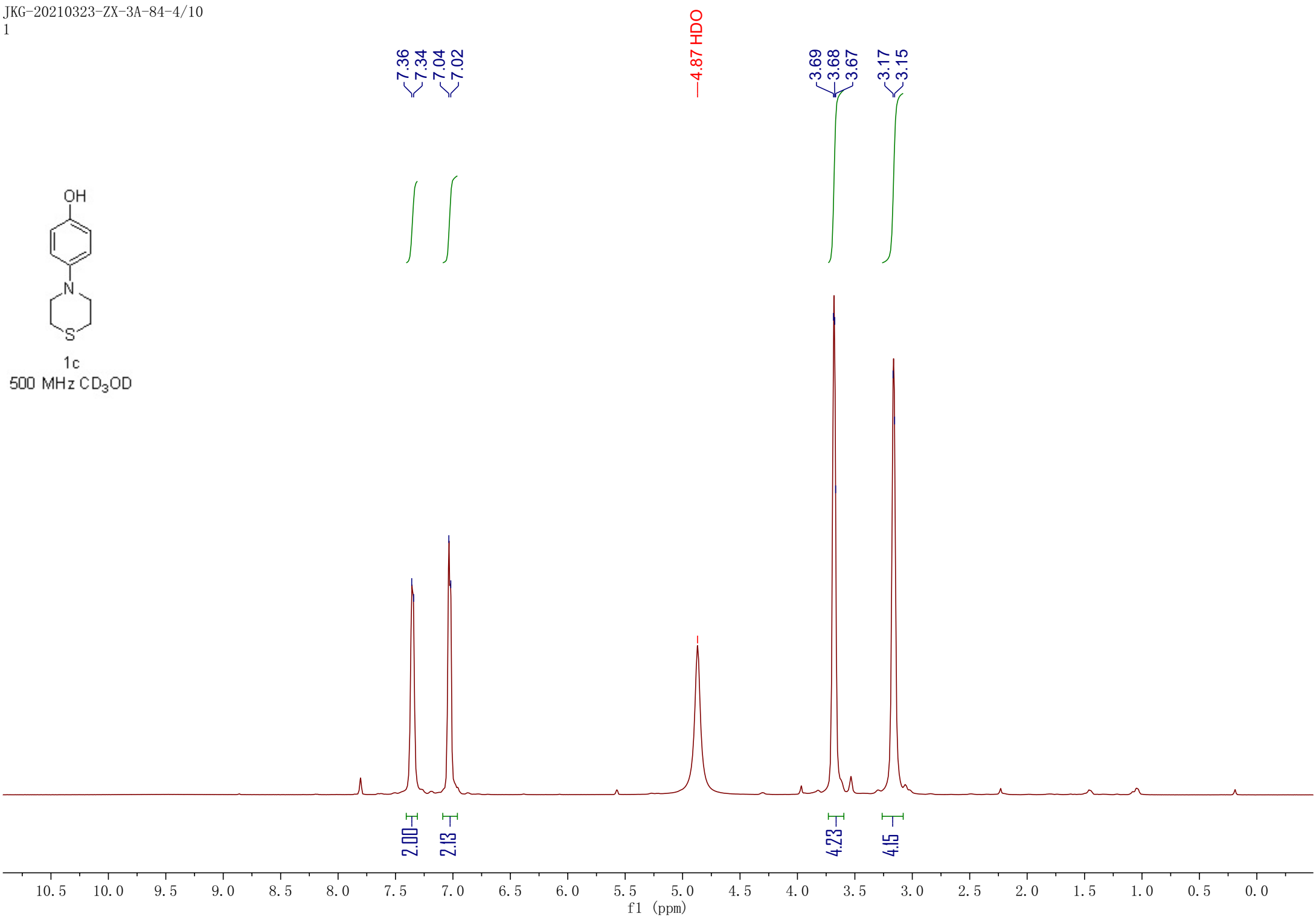

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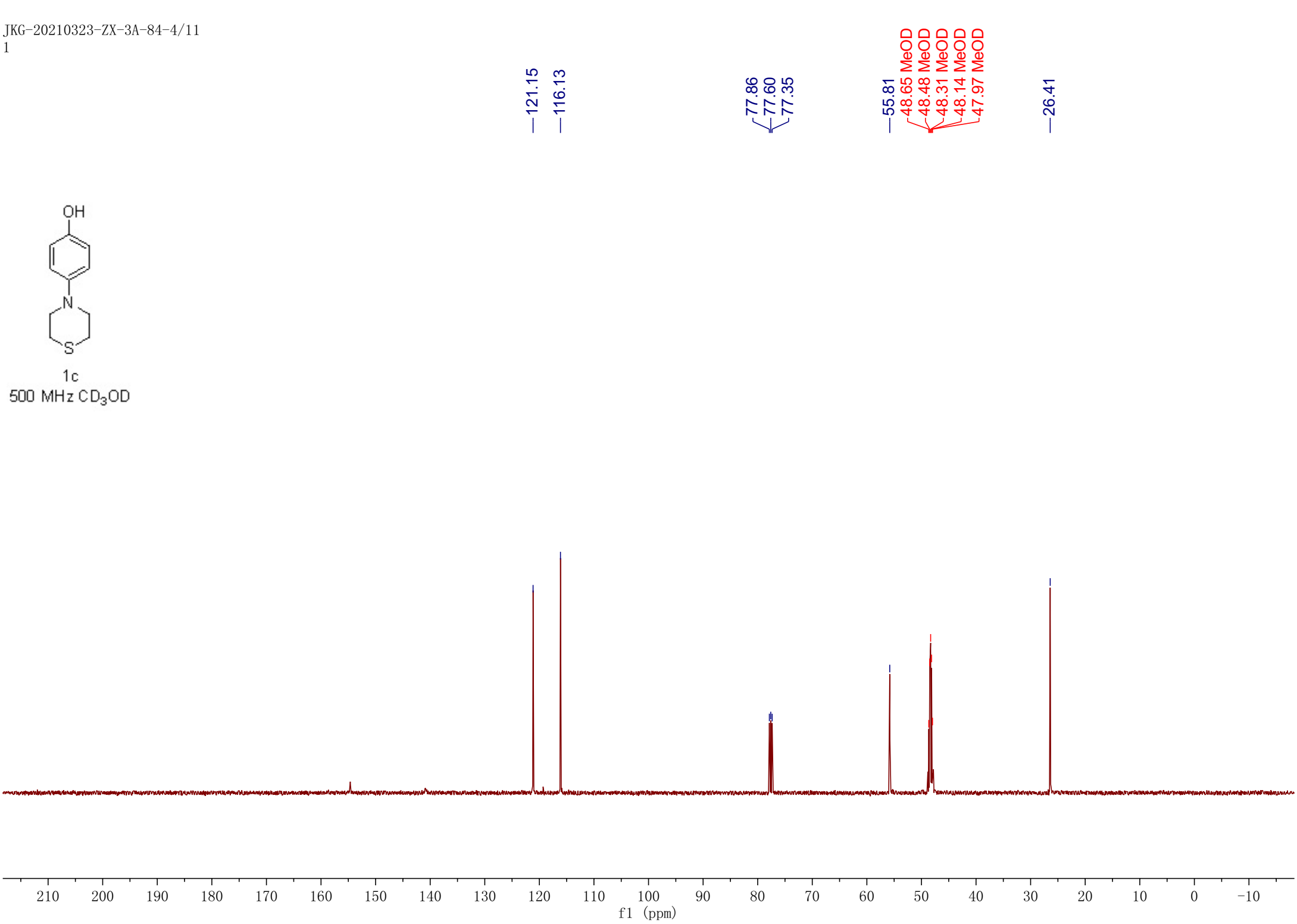

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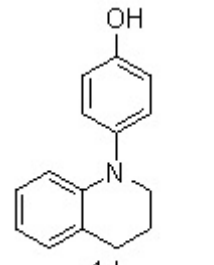

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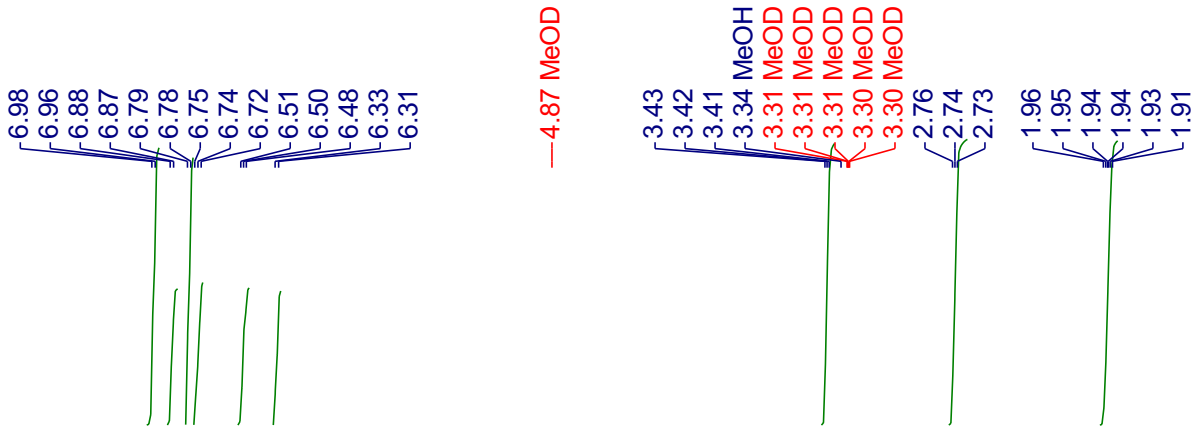

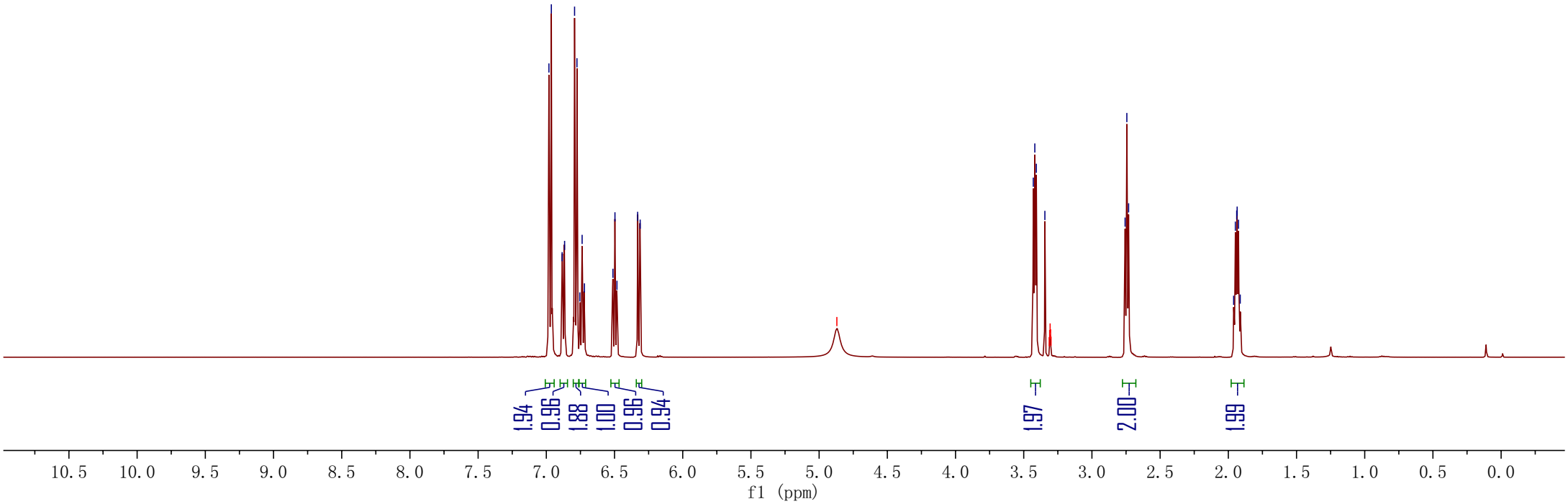

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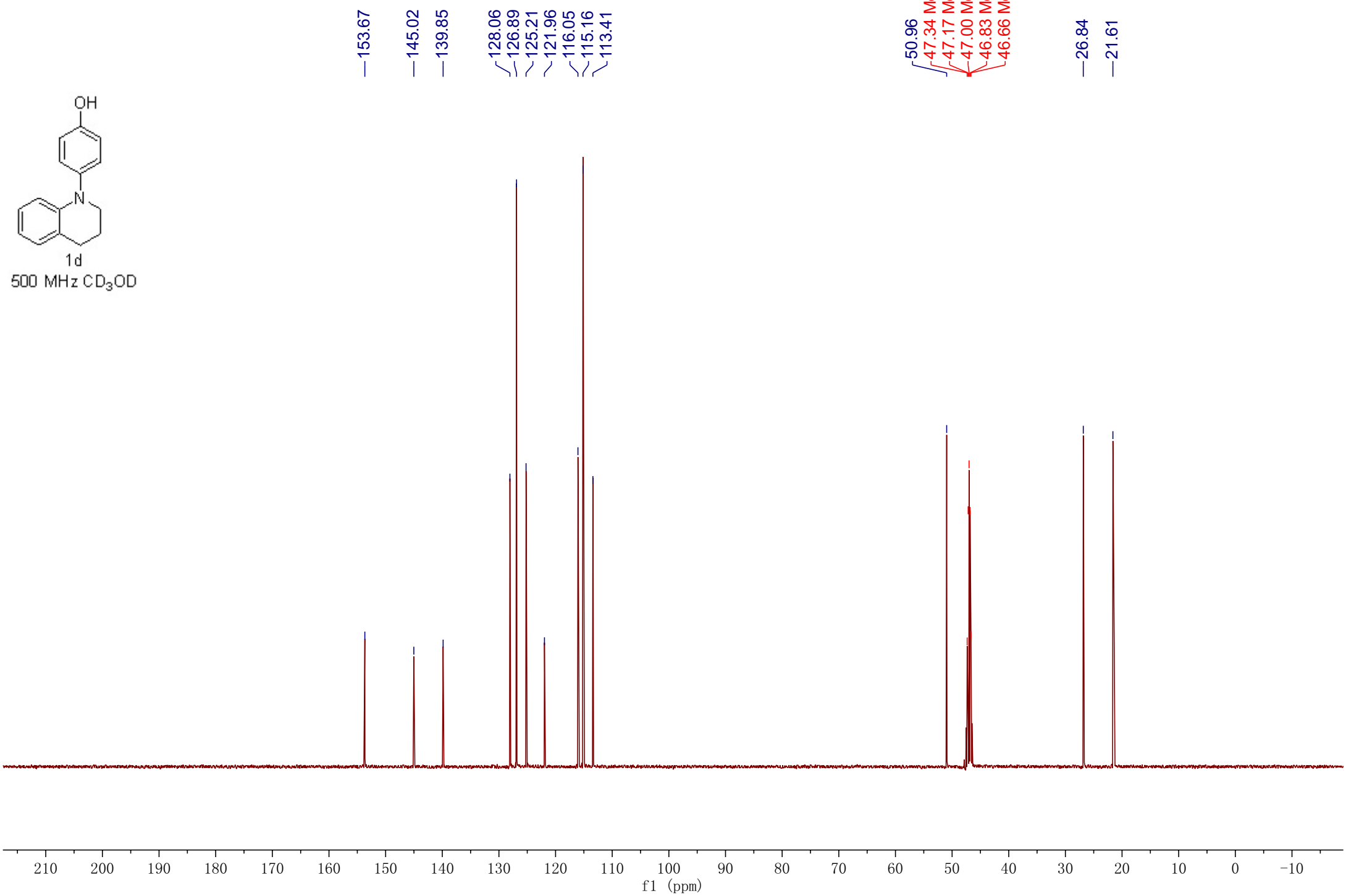

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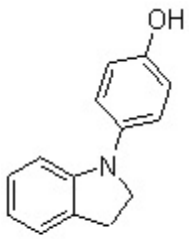

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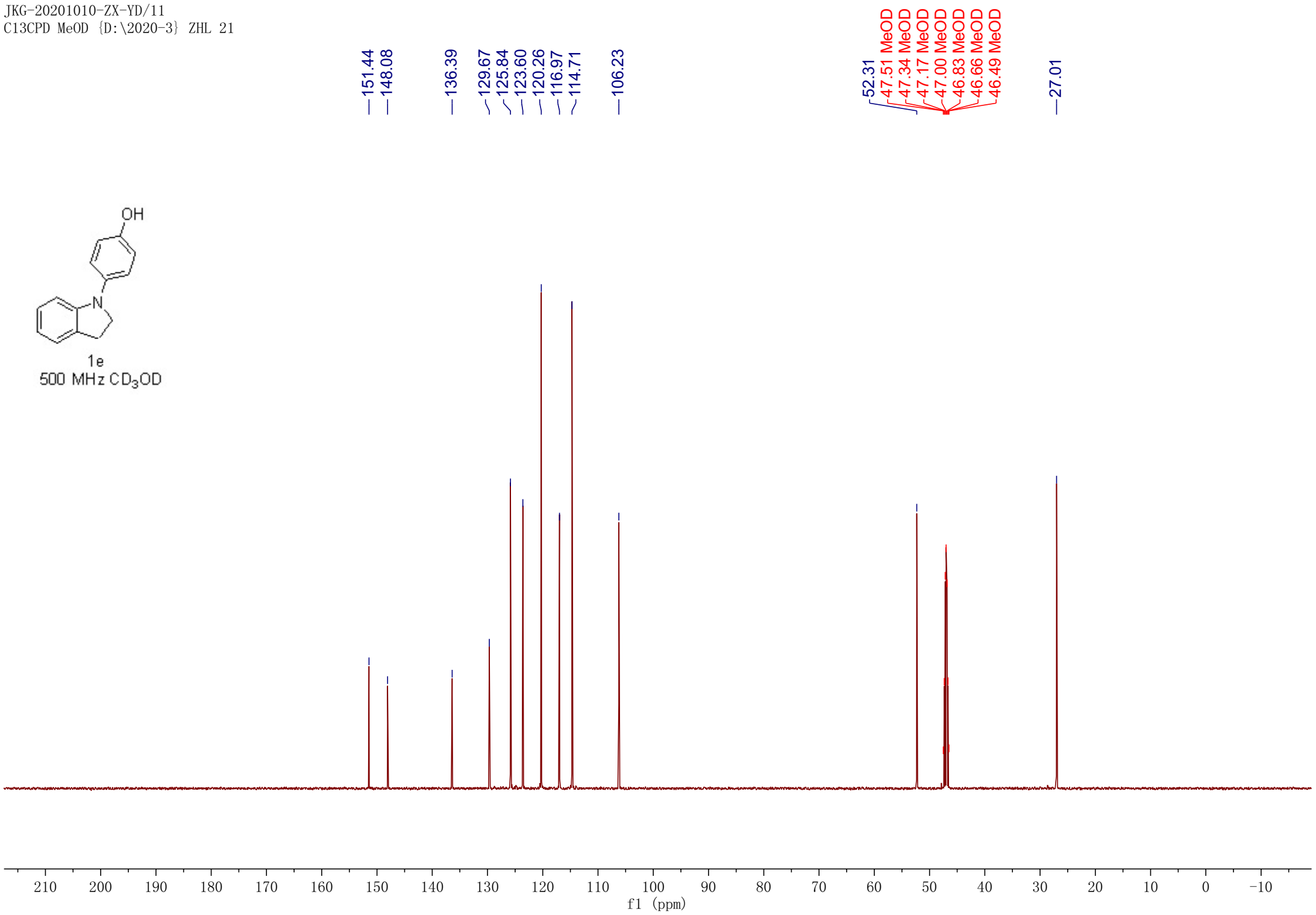

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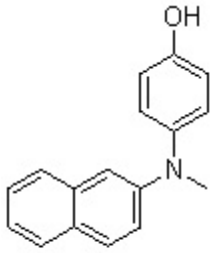

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129.92
127.25
125.19
124.83
124.63
124.25
123.01
121.82
116.05
114.57

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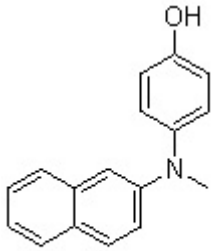
39.75



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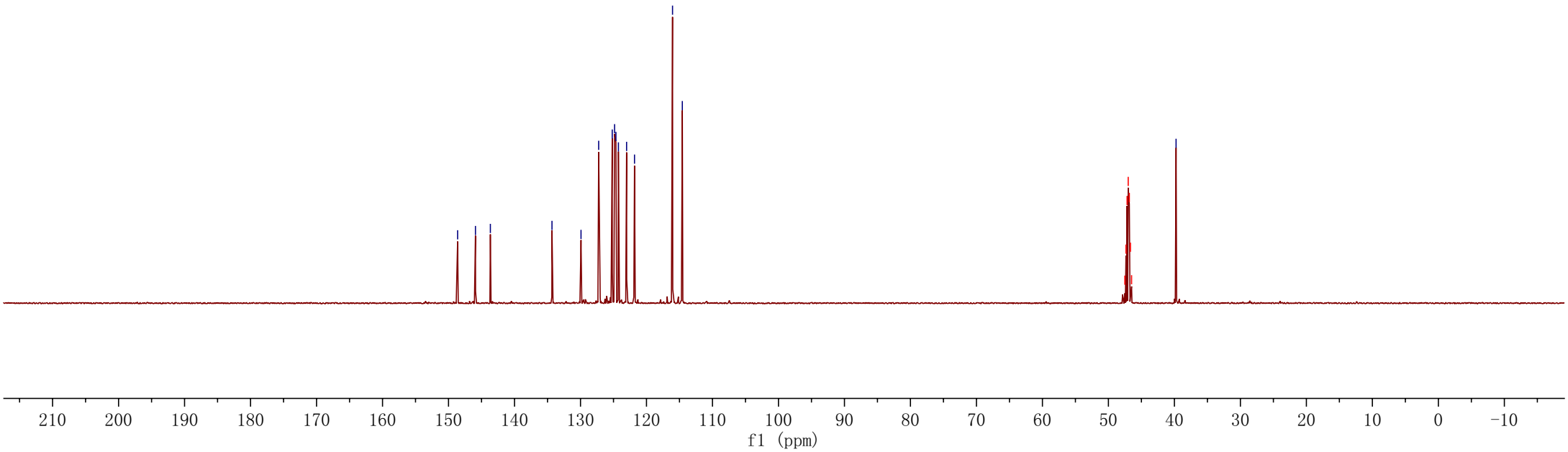

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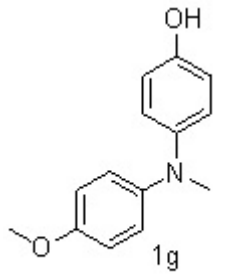

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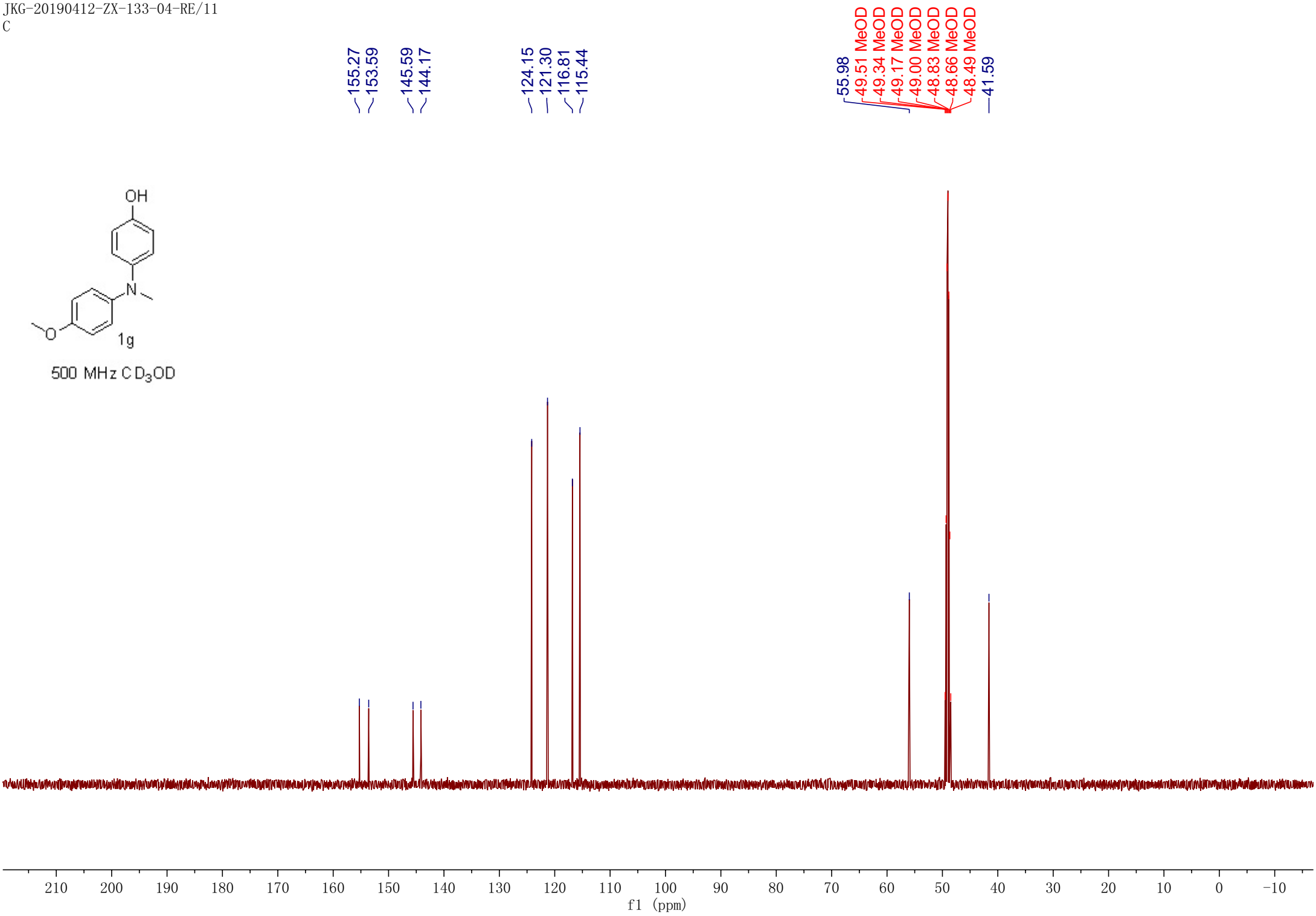

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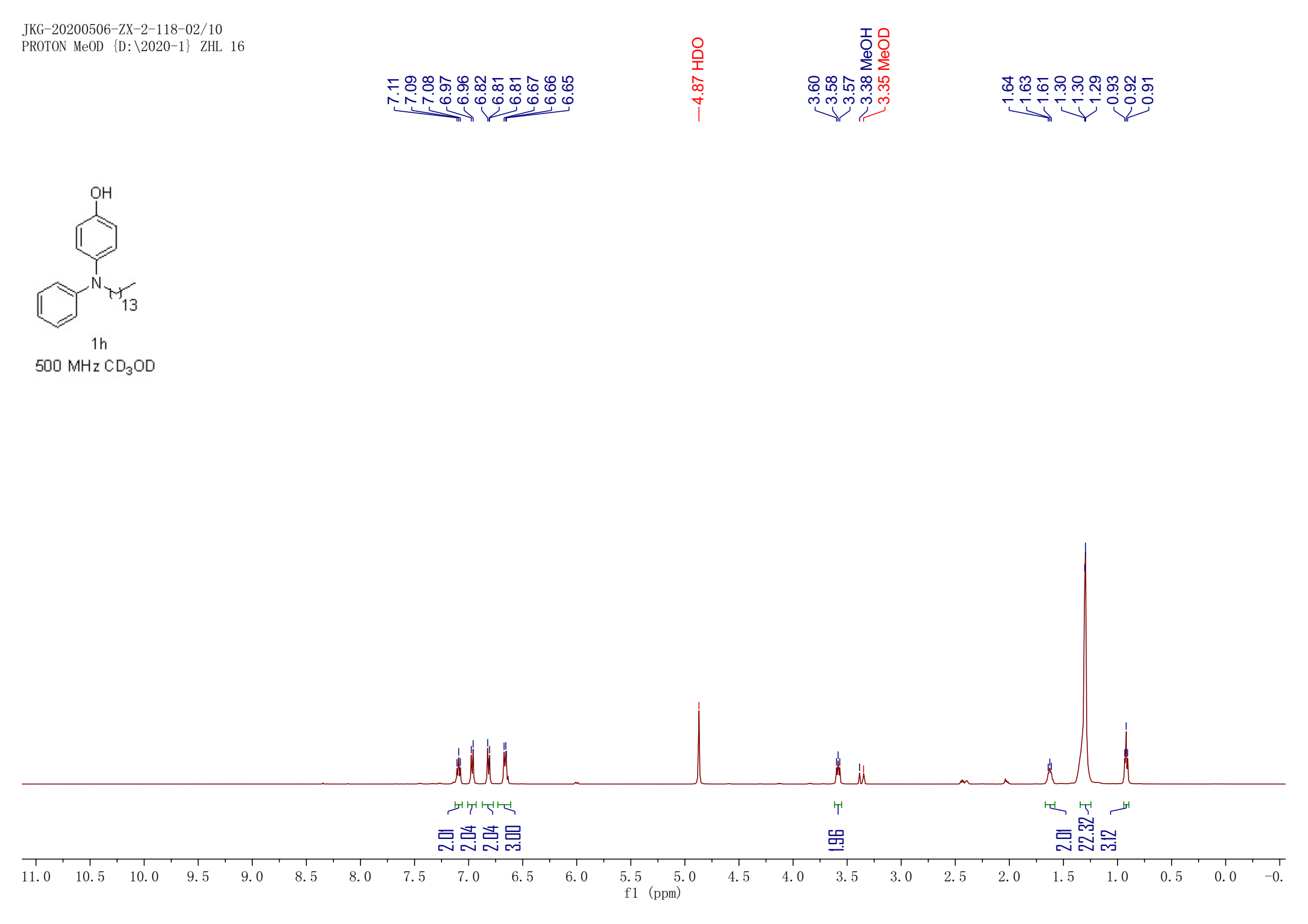

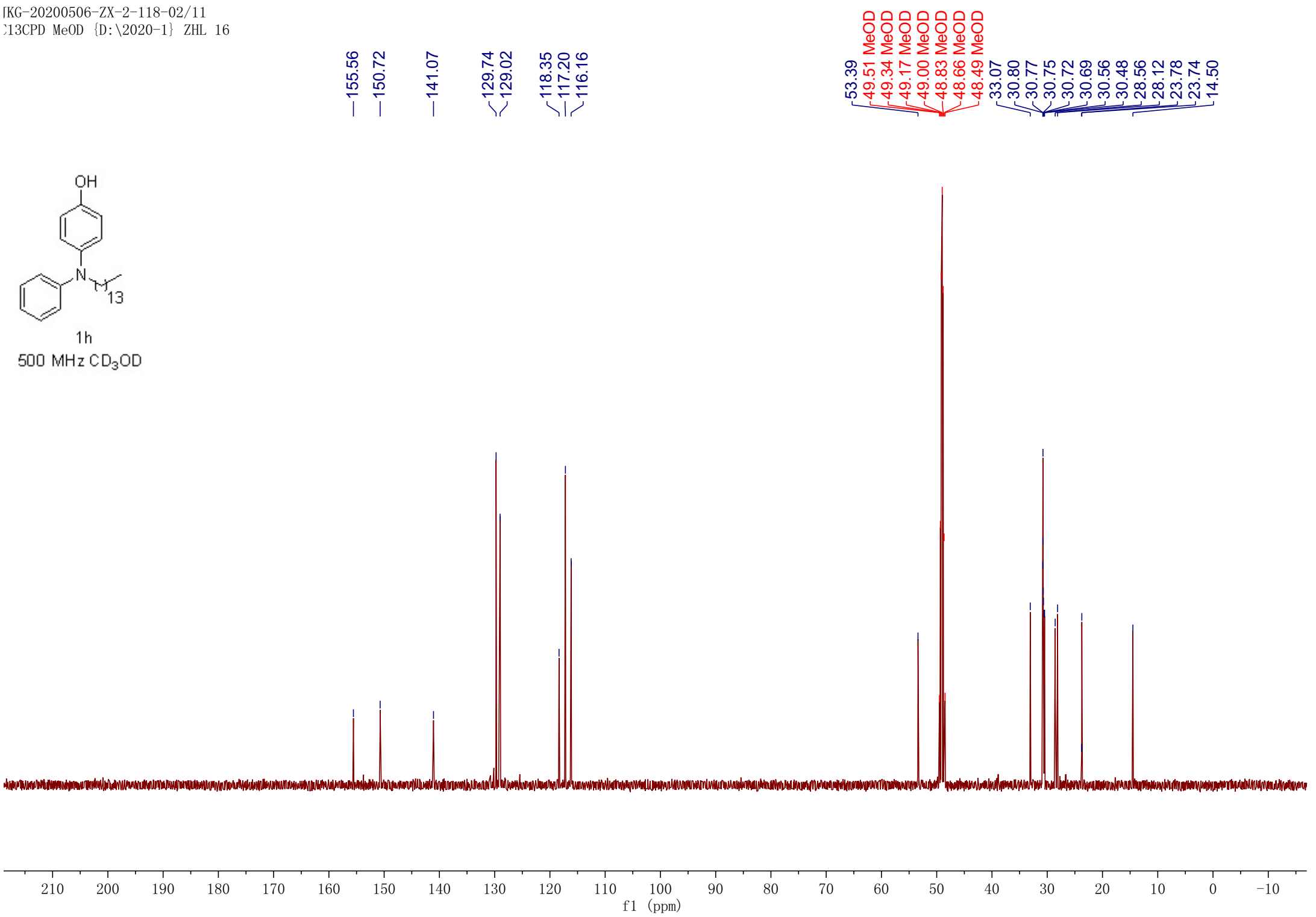
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1h
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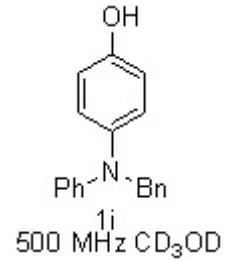

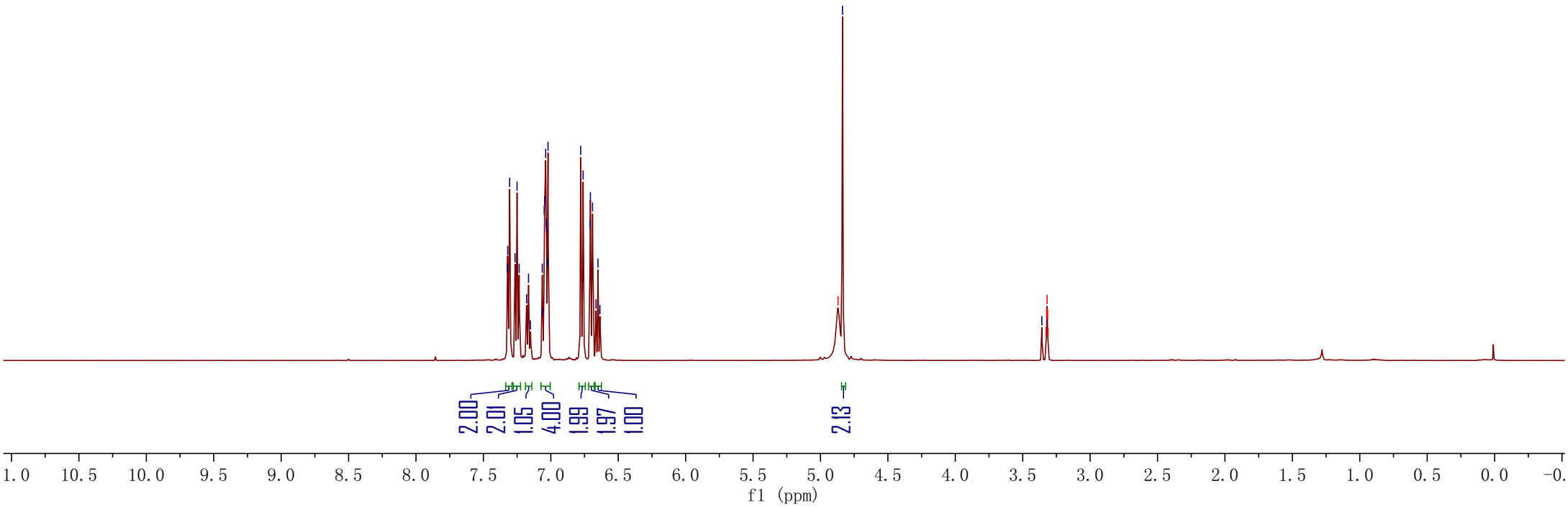

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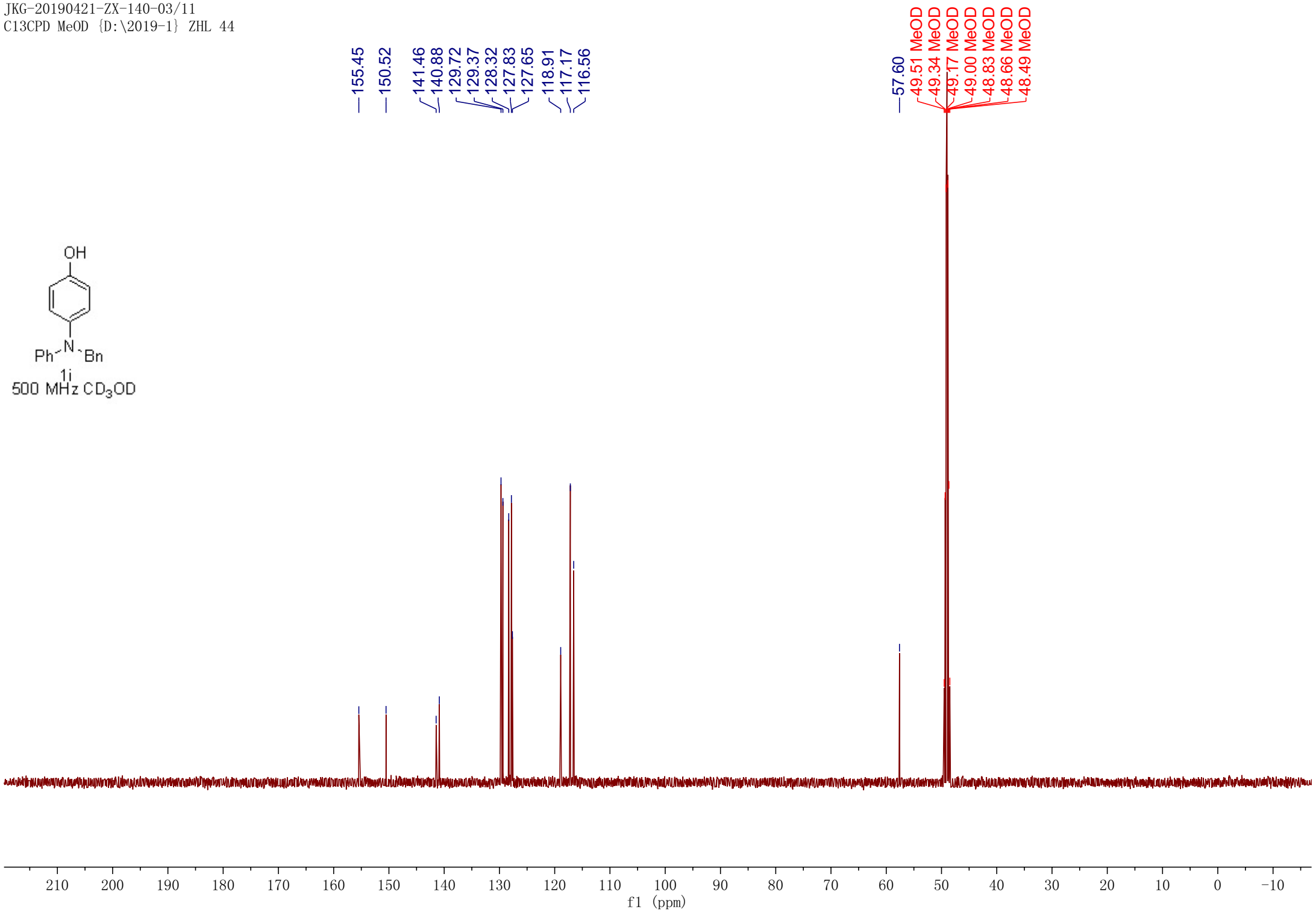
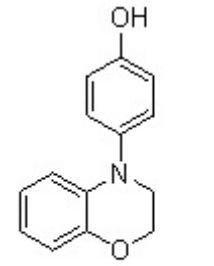
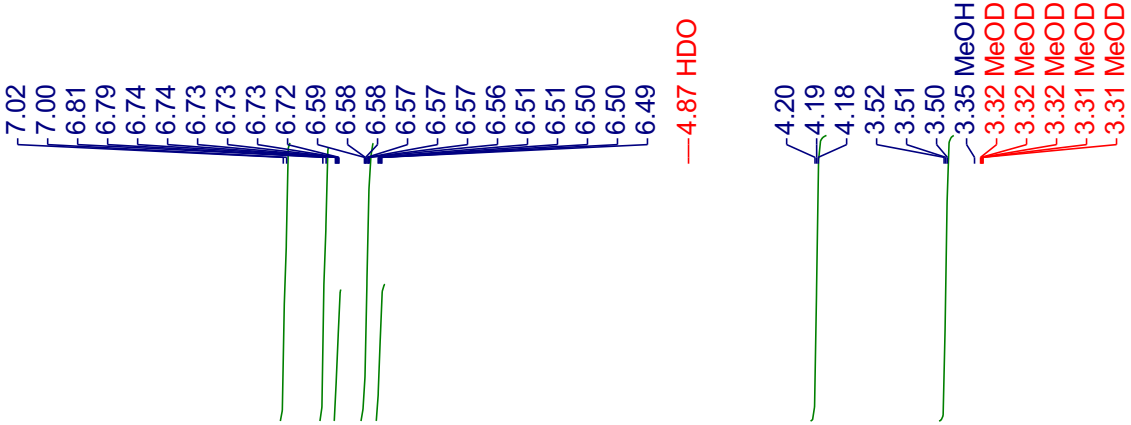
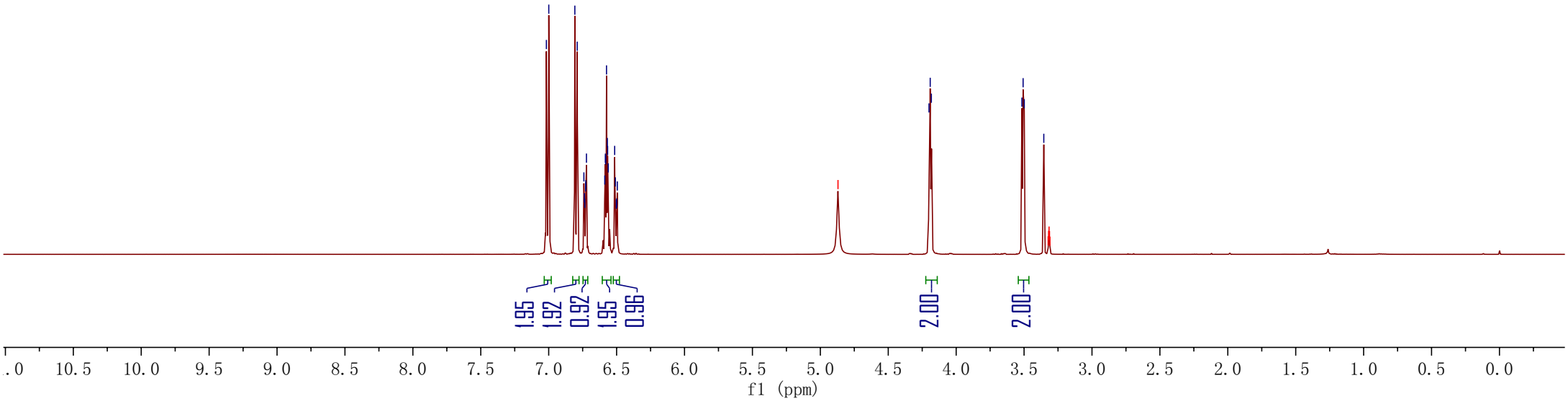


JKG-20200911-ZX-3A-6-3/10
PROTON MeOD {D:\2020-2} ZHL 21

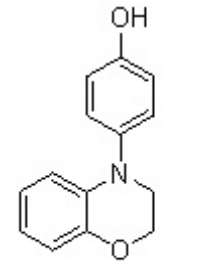


1j
500 MHz CD_3OD



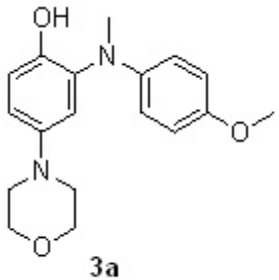


JKG-20200911-ZX-3A-6-3/11
C13CPD MeOD {D:\2020-2} ZHL 21



1j
500 MHz CD_3OD

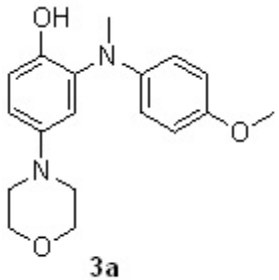
JKG20200821-ZX-3-1-4/10
PROTON CDCl3 {D:\2020-2} ZHL 17



3a

500 MHz, $CDCl_3$

JKG20200821-ZX-3-1-4/11
C13CPD CDCl3 {D:\2020-2} ZHL 17



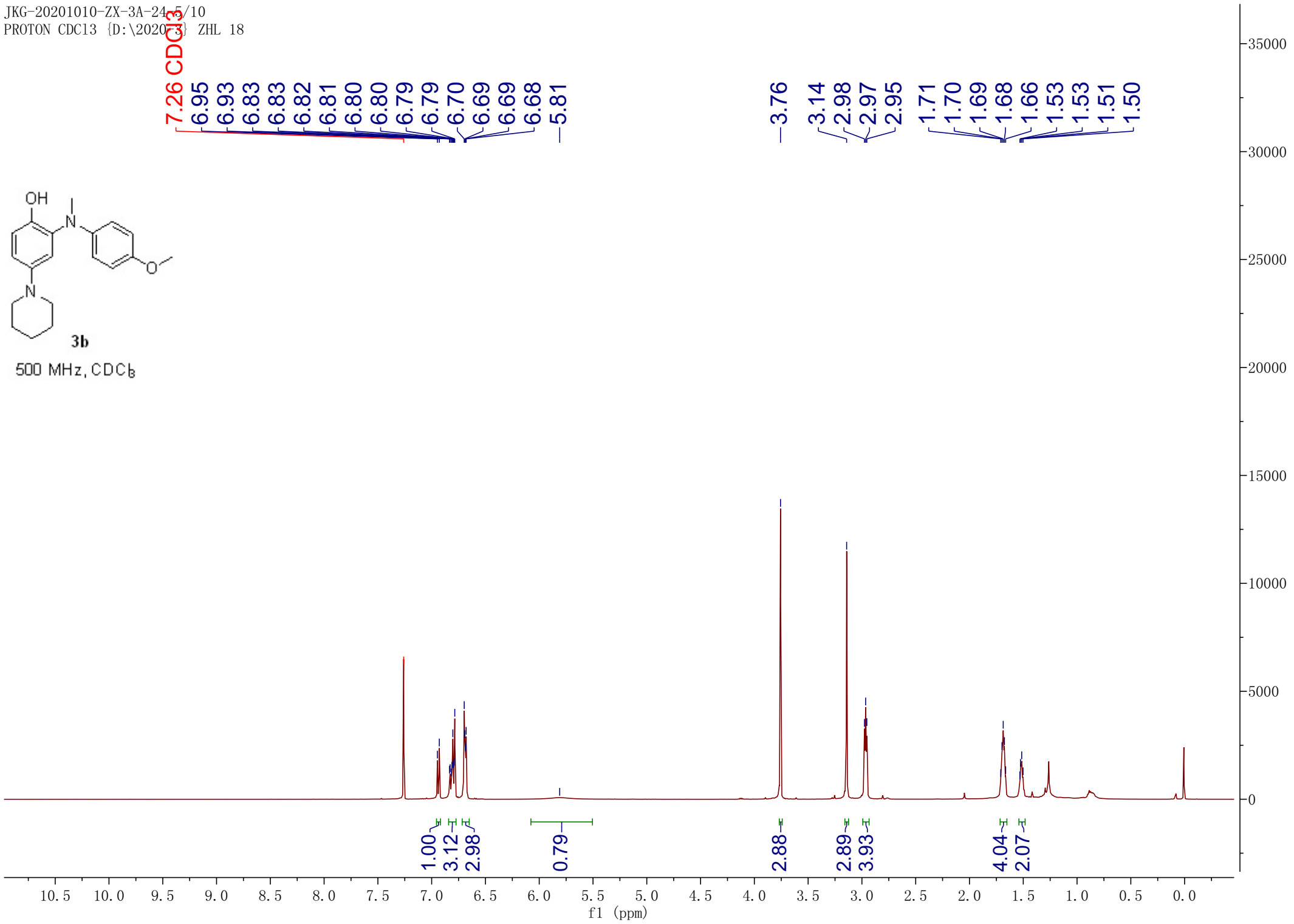
3a

500 MHz, $CDCl_3$

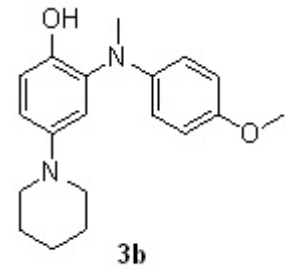
153.32
146.86
146.11
143.47
116.55
116.02
115.49
114.76
114.60
77.32 CDCl3
77.07 CDCl3
76.81 CDCl3
66.99
55.69
50.76
40.44

f1 (ppm)

JKG-20201010-ZX-3A-24_5/10
PROTON CDCl3 {D:\2020\3} ZHL 18



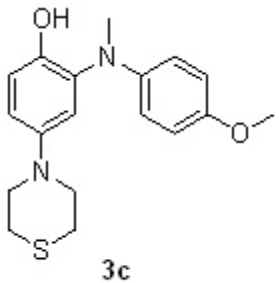
JKG-20201010-ZX-3A-24-5/11
C13CPD CDCl3 {D:\2020-3} ZHL 18



3b

500 MHz, $CDCl_3$

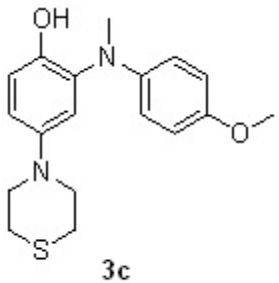
JKG-20210112-ZX-3A-71-3.10.fid
H



3c

500 MHz, $CDCl_3$

JKG-20210112-ZX-3A-71-3/11
C



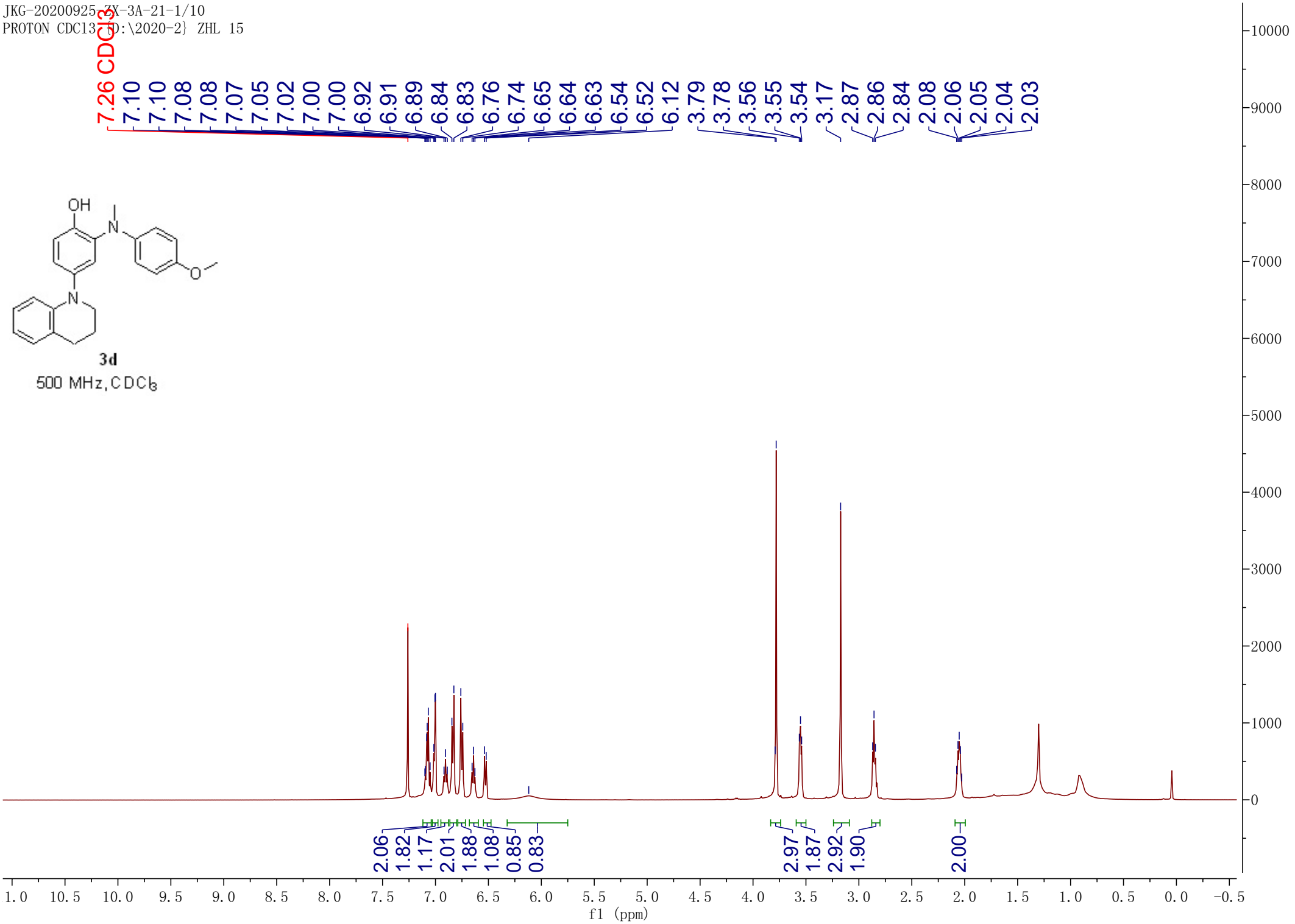
3c

500 MHz, $CDCl_3$

153.40
147.18
147.04
143.48
136.65
118.13
116.88
116.59
115.42
114.63
77.28 CDCl3
77.03 CDCl3
76.78 CDCl3
55.69
53.82
40.47
27.74

f1 (ppm)

JKG-20200925-ZX-3A-21-1/10
PROTON CDCl3 D:\2020-2} ZHL 15

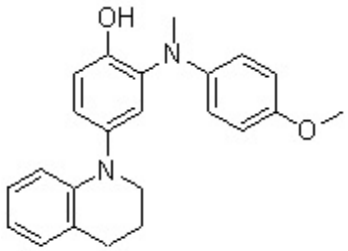


JKG-20200925-ZX-3A-21-1/11
C13CPD CDCl3 {D:\2020-2} ZHL 15

153.50
149.73
145.28
143.34
141.54
137.20
129.25
126.41
125.75
124.27
122.95
117.23
116.78
115.78
114.57
114.12

77.25 CDCl3
77.00 CDCl3
76.75 CDCl3

55.61
51.56
40.58
27.78
22.49

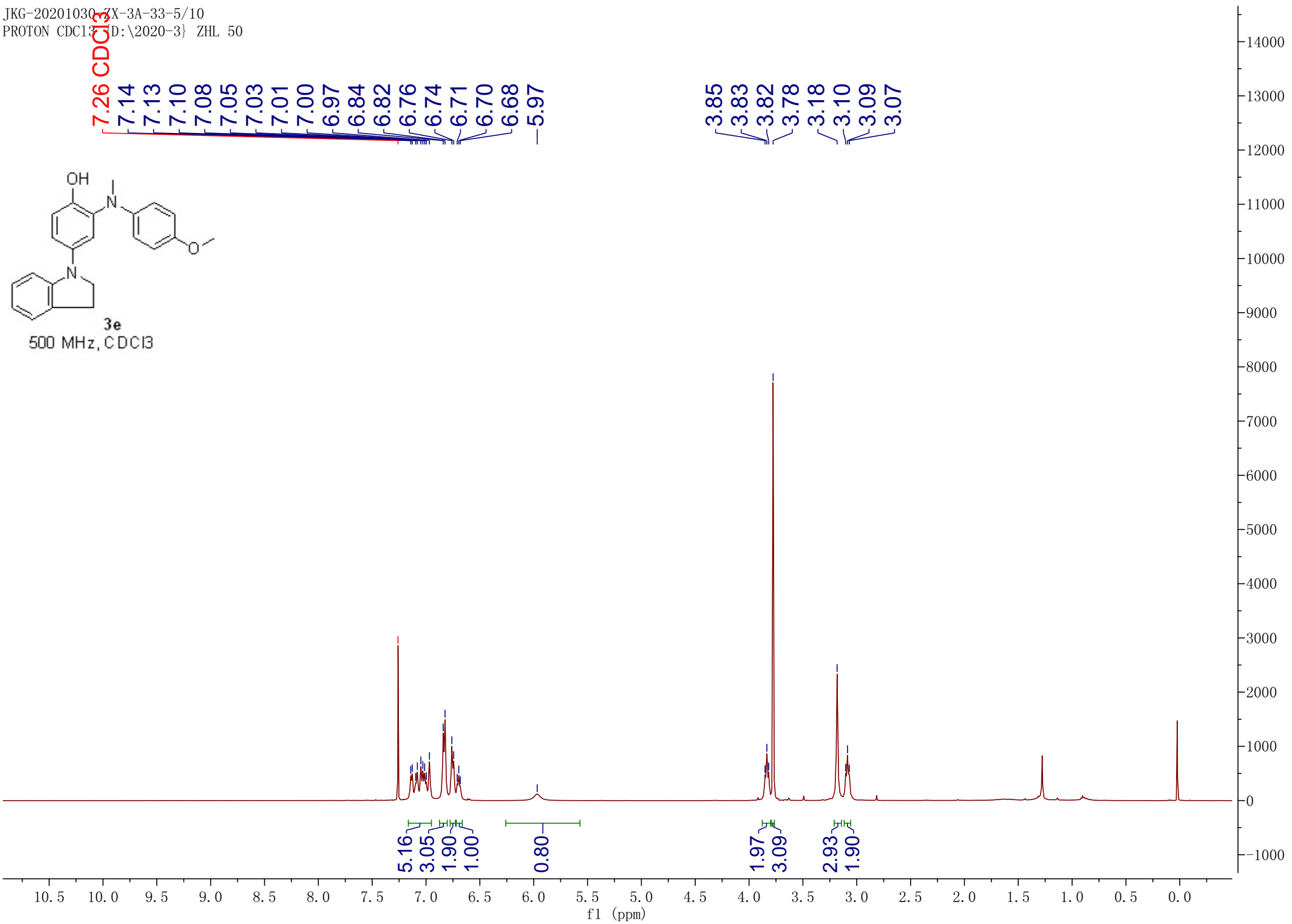


3d

500 MHz, $CDCl_3$

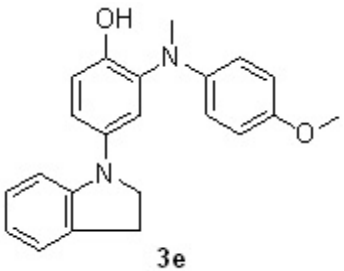
f1 (ppm)

JKG-20201030-ZX-3A-33-5/10
PROTON CDCl3 {D:\2020-3} ZHL 50



JKG-20200925-ZX-3A-21-2/11
C13CPD CDCl3 {D:\2020-2} ZHL 16

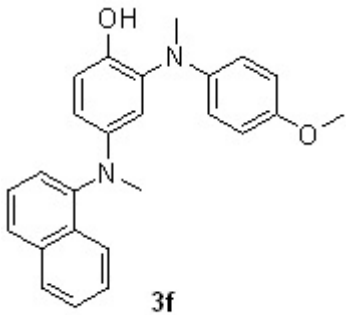
153.46
148.26
147.60
143.37
138.21
136.72
130.59
127.05
124.84
118.84
118.20
117.24
116.74
115.50
114.88
114.60
113.65
107.23
77.25 CDCl3
77.00 CDCl3
76.75 CDCl3
55.64
52.97
40.50
28.20



3e
500 MHz, CDCl3

f1 (ppm)

JKG-20201022-ZX-3A-28-3/10
PROTON MeOD {D:\2020-3} ZHL 27



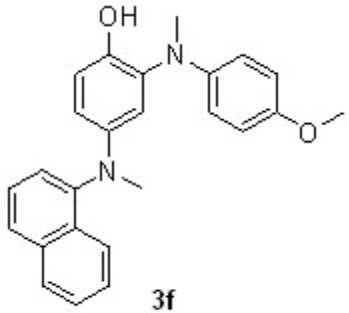
3f

500 MHz, CD_3OD

JKG-20201022-ZX-3A-28-3/11
C13CPD MeOD {D:\2020-3} ZHL 27

153.66
147.68
146.97
146.52
138.02
136.54
131.89
129.43
127.25
126.92
126.72
126.45
125.19
123.79
117.76
116.76
116.07
115.61
115.19

56.06
49.51 MeOD
49.34 MeOD
49.17 MeOD
49.00 MeOD
48.83 MeOD
48.66 MeOD
48.49 MeOD
41.70
39.90

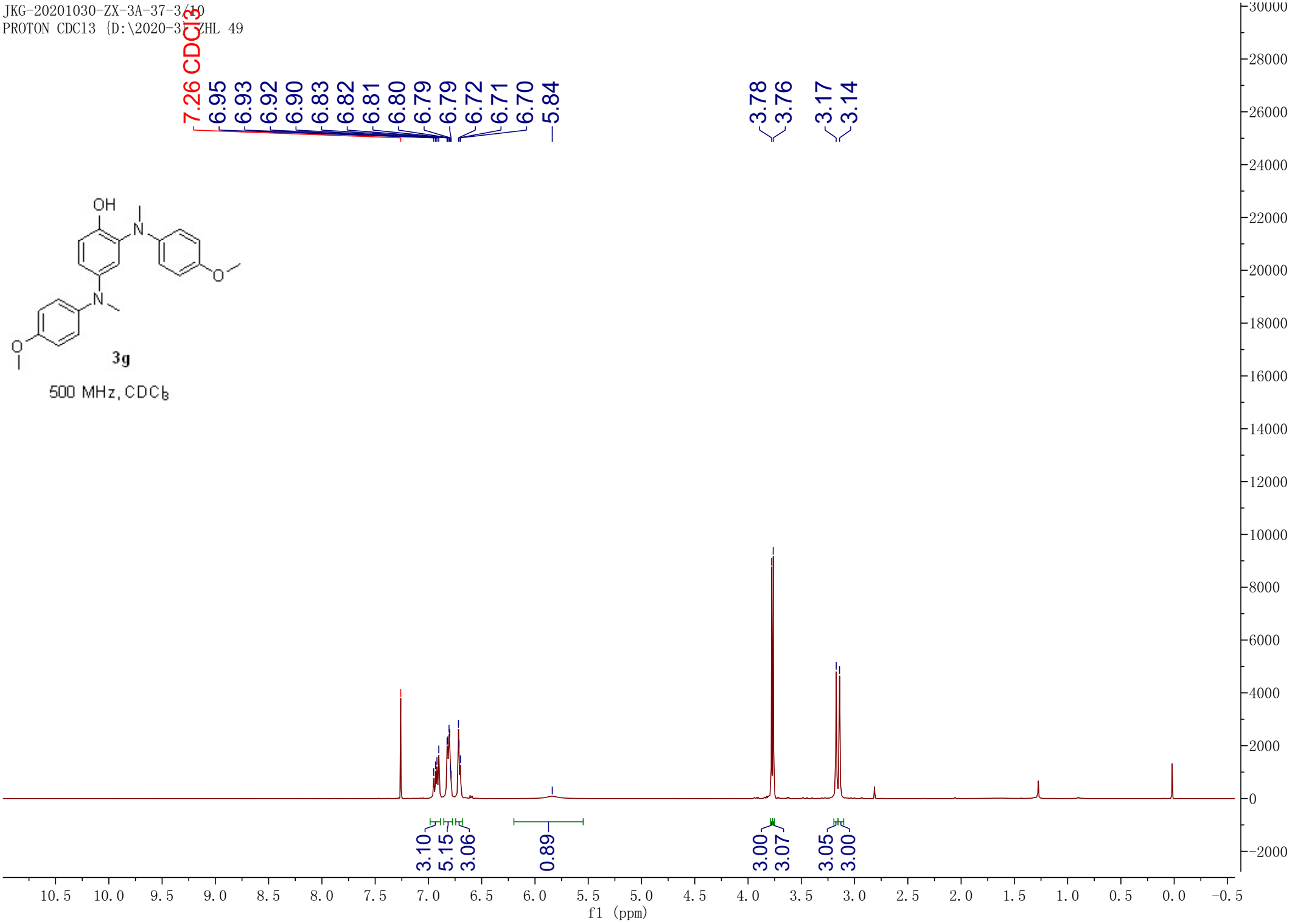


3f

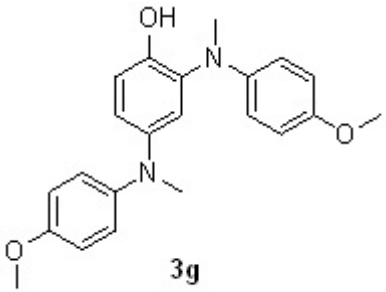
500 MHz, CD_3OD

f1 (ppm)

JKG-20201030-ZX-3A-37-3/40
PROTON CDCl3 {D:\2020-3\ZHL 49



JKG-20201030-ZX-3A-37-3/11
C13CPD CDCl3 {D:\2020-3} ZHL 49



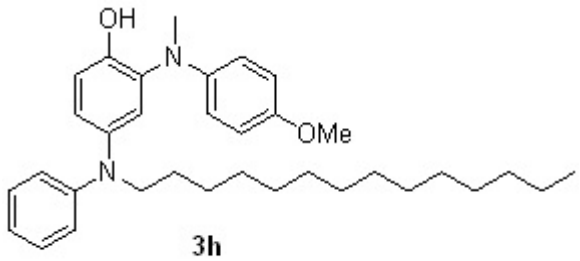
3g

500 MHz, $CDCl_3$

154.33
153.23
147.05
143.91
143.42
143.37
136.49
121.59
119.75
117.98
116.43
115.40
114.52
77.25 CDCl3
77.00 CDCl3
76.75 CDCl3
55.62
55.54
41.04
40.42

f1 (ppm)

ZX-3-83-2.10.1.1r



3h

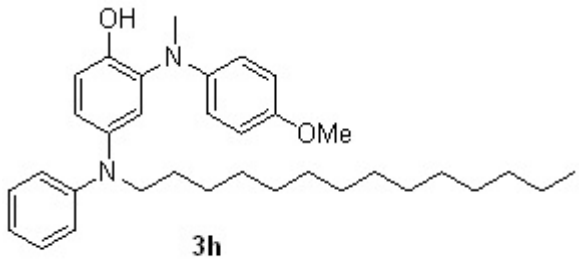
400 MHz, $CDCl_3$

7.26 CDCl3
7.18
7.17
7.16
7.15
7.14
7.04
7.02
6.99
6.98
6.97
6.96
6.89
6.89
6.82
6.81
6.80
6.79
6.74
6.73
6.73
6.72
6.71
6.07
3.77
3.59
3.57
3.55
3.15
1.61
1.59
1.32
1.30
1.27
1.26
0.91
0.90
0.88

2.04 2.07 1.00 2.08 5.11 1.05 3.16 2.07 3.18 2.26 24.13 3.08

f1 (ppm)

ZX-3-83-2.11.1.1r



3h

400 MHz, $CDCl_3$

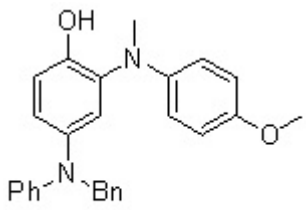
153.52
149.45
148.91
143.38
141.03
137.16
128.97
125.73
124.29
117.75
116.76
115.87
115.37
114.60
77.37 CDC13
77.06 CDC13
76.74 CDC13
55.66
52.45
52.44
40.64
31.96
29.73
29.72
29.70
29.67
29.64
29.49
29.40
27.42
27.10
22.73
14.17

1.60E+11
1.50E+11
1.40E+11
1.30E+11
1.20E+11
1.10E+11
1.00E+11
9.00E+10
8.00E+10
7.00E+10
6.00E+10
5.00E+10
4.00E+10
3.00E+10
2.00E+10
1.00E+10
0.00E+00
-1.00E+10

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

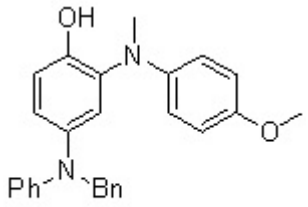
JKG-20201022-ZX-3A-21-4/10
PROTON MeOD {D:\2020-3} ZHL 28



3i

500 MHz, CD_3OD

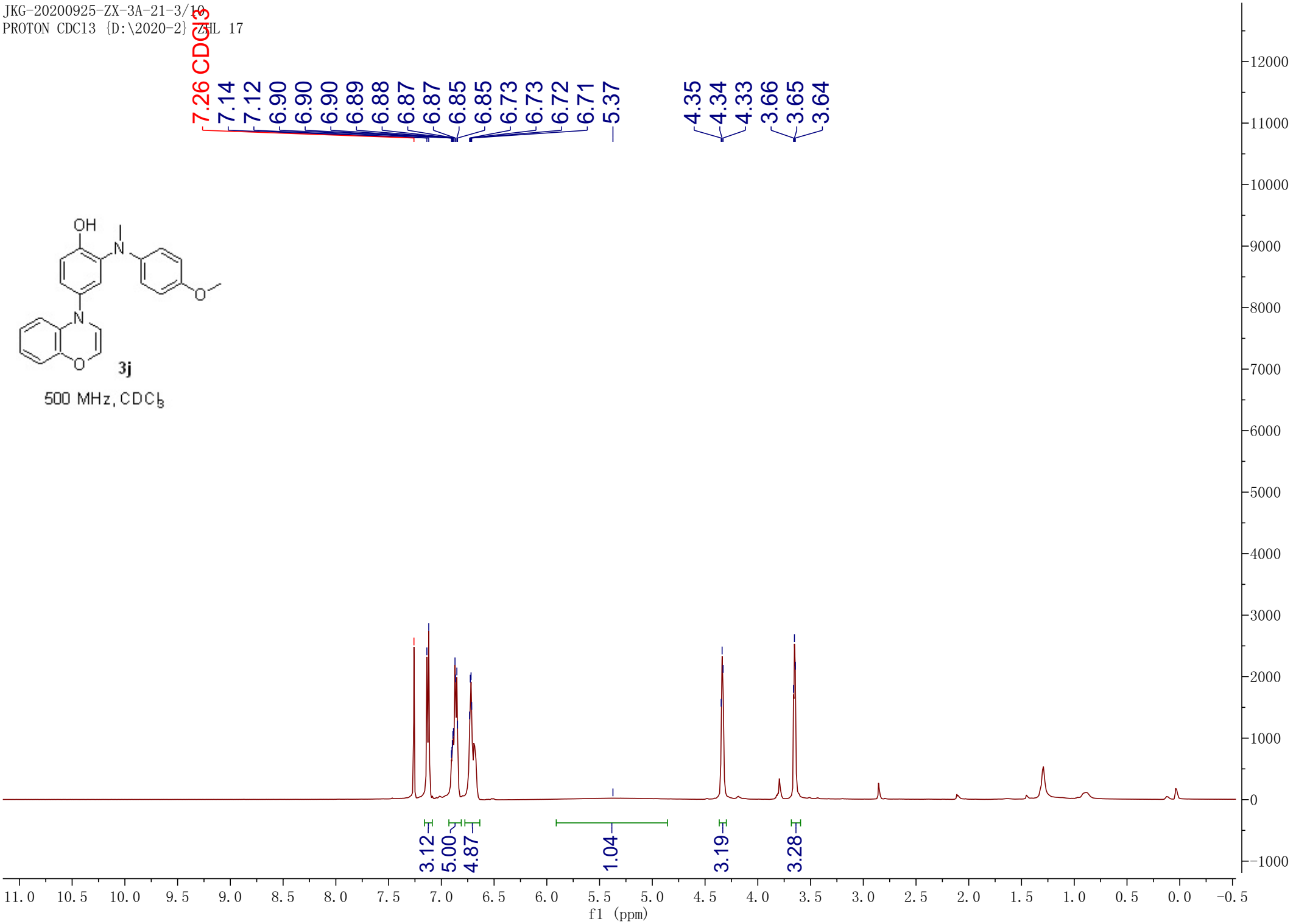
JKG-20201022-ZX-3A-21-4/11
C13CPD MeOD {D:\2020-3} ZHL 28



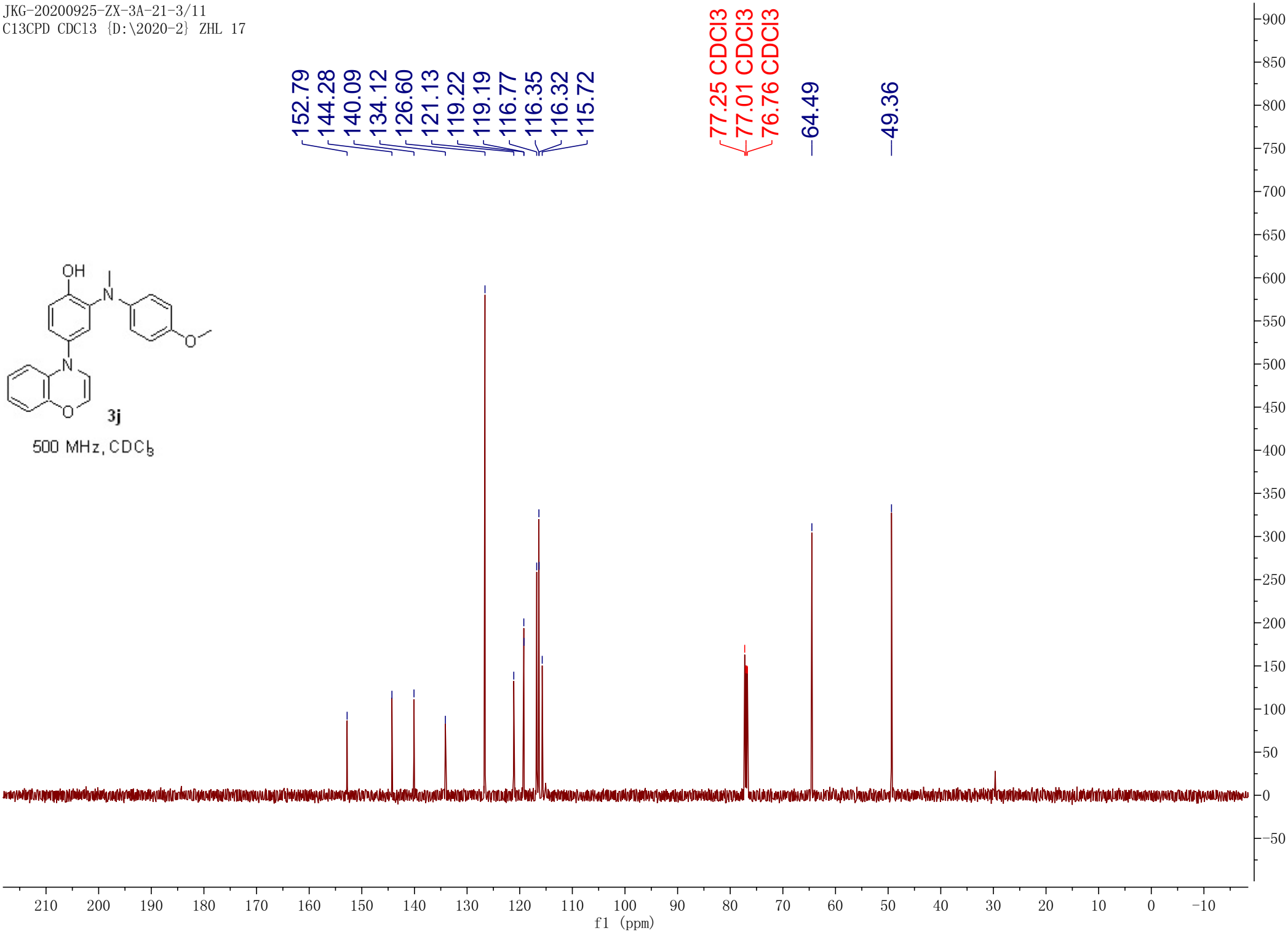
3i

500 MHz, CD_3OD

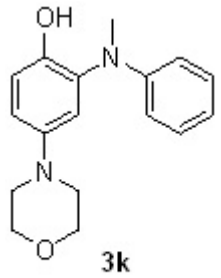
JKG-20200925-ZX-3A-21-3/10
PROTON CDCl3 {D:\2020-2} ZHL 17



JKG-20200925-ZX-3A-21-3/11
C13CPD CDCl3 {D:\2020-2} ZHL 17



JKG-20201010-ZX-3A-Me/10
PROTON CDCl3 {D:\2020-3} ZHL 19

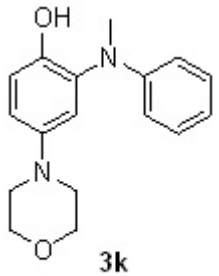


3k

500 MHz, $CDCl_3$

JKG-20201010-ZX-3A-Me/11
C13CPD CDCl3 {D:\2020-3} ZHL 19

149.08
146.90
146.18
135.51
129.13
119.00
116.46
115.72
115.18
114.52
77.25 CDCl3
77.00 CDCl3
76.75 CDCl3
66.91
50.64
39.72

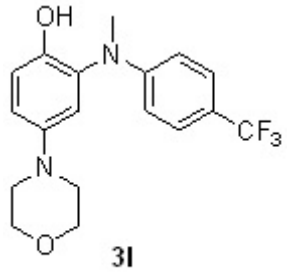


3k

500 MHz, $CDCl_3$

f1 (ppm)

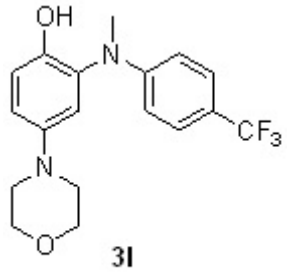
JKG-20201201-ZX-3A-49-3/10
PROTON MeOD {D:\2020-3} ZHL 12



3l

500 MHz, CD_3OD

JKG-20201201-ZX-3A-49-3/11
C13CPD MeOD {D:\2020-3} ZHL 12



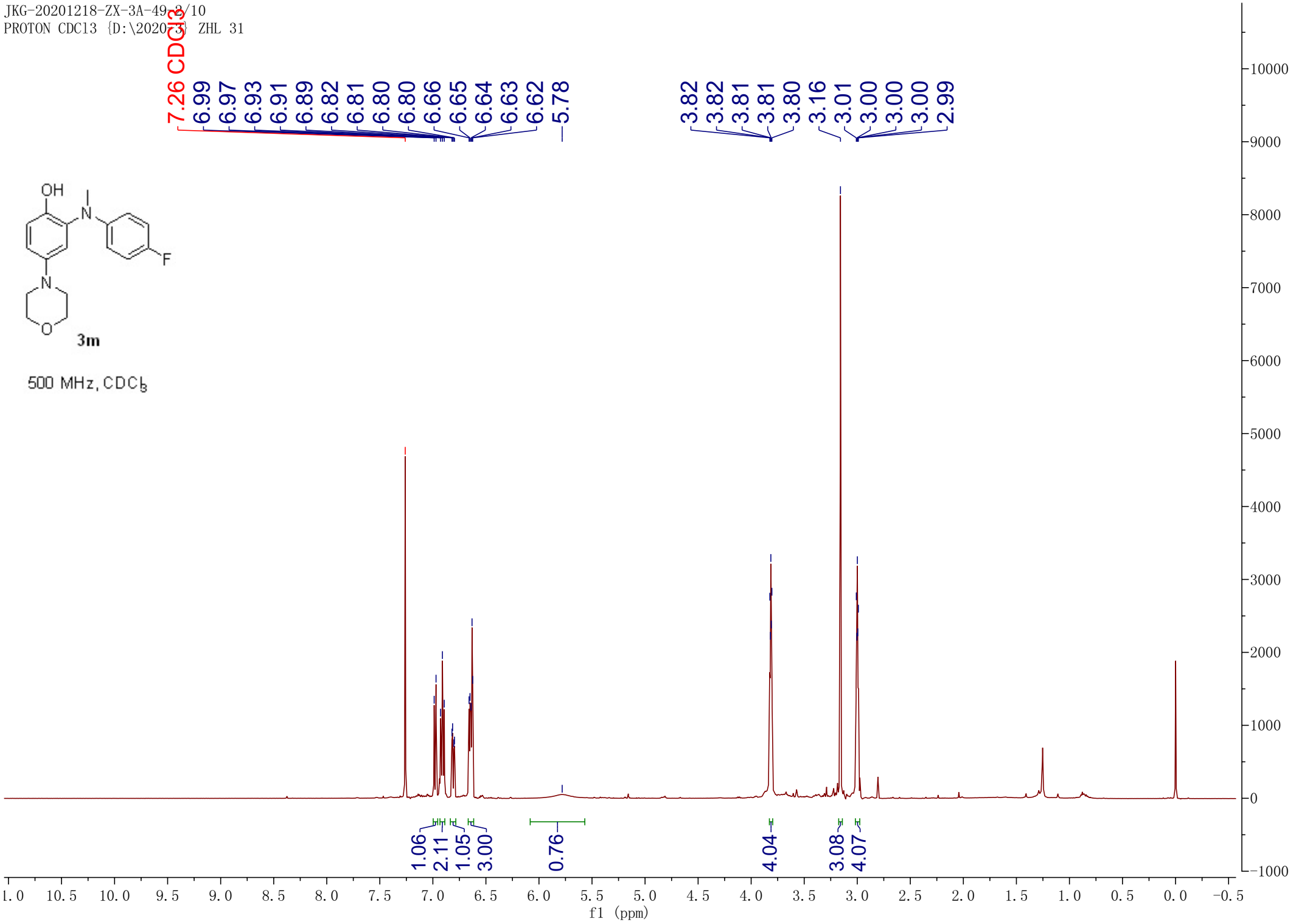
3l

500 MHz, CD_3OD

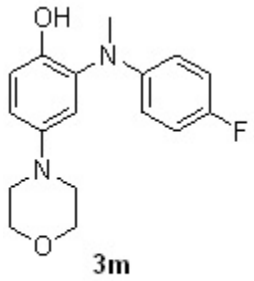
153.33
149.17
147.14
135.24
127.73
126.97
126.94
126.91
126.88
125.59
119.23
118.97
118.54
118.40
118.19
113.50
67.98
52.09
49.51 MeOD
49.34 MeOD
49.17 MeOD
49.00 MeOD
48.83 MeOD
48.66 MeOD
48.49 MeOD
39.44

f1 (ppm)

JKG-20201218-ZX-3A-49-2/10
PROTON CDC13 {D:\2020\3} ZHL 31



JKG-20201218-ZX-3A-49-2/11
C13CPD CDCl3 {D:\2020-3} ZHL 31



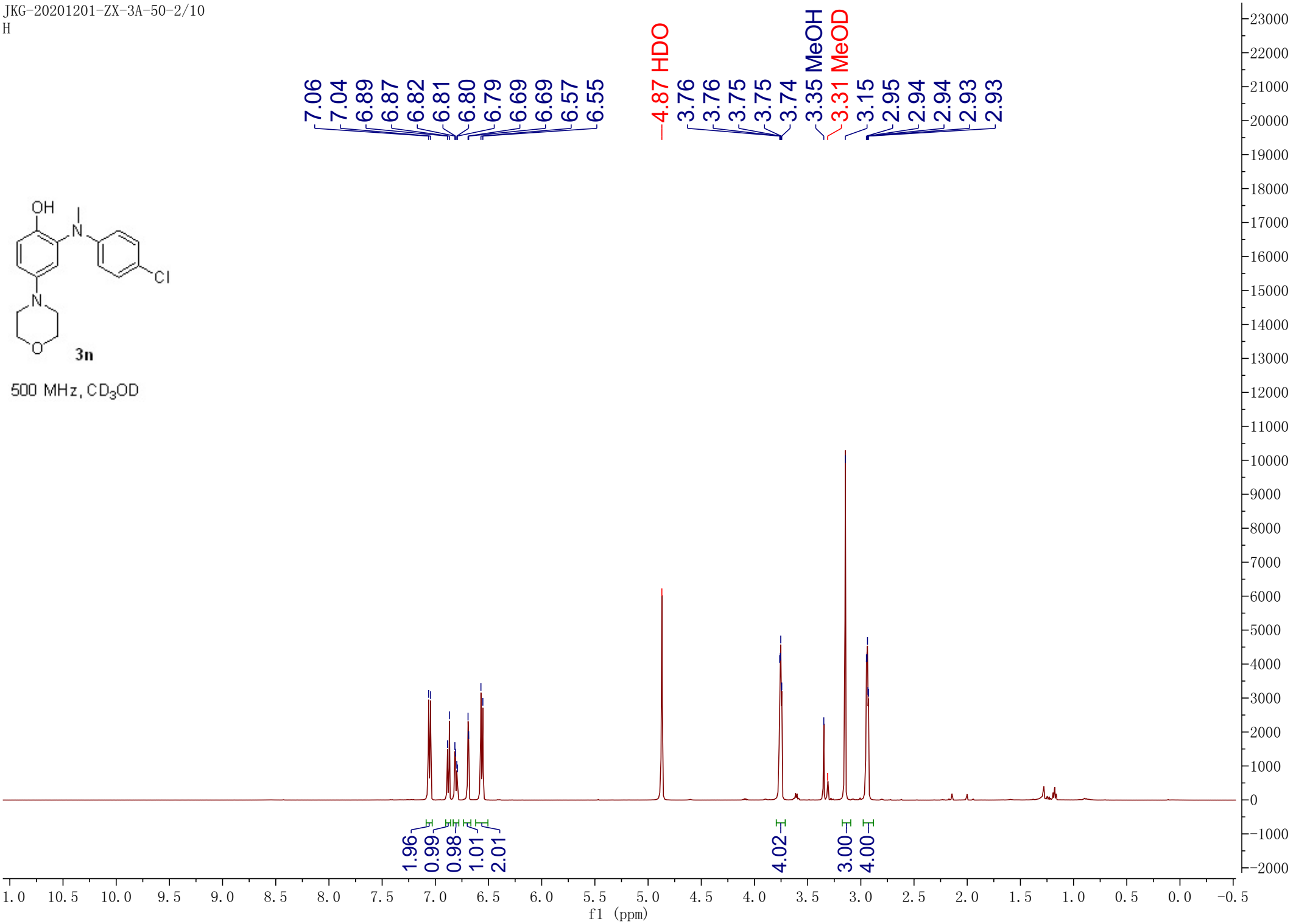
3m

500 MHz, $CDCl_3$

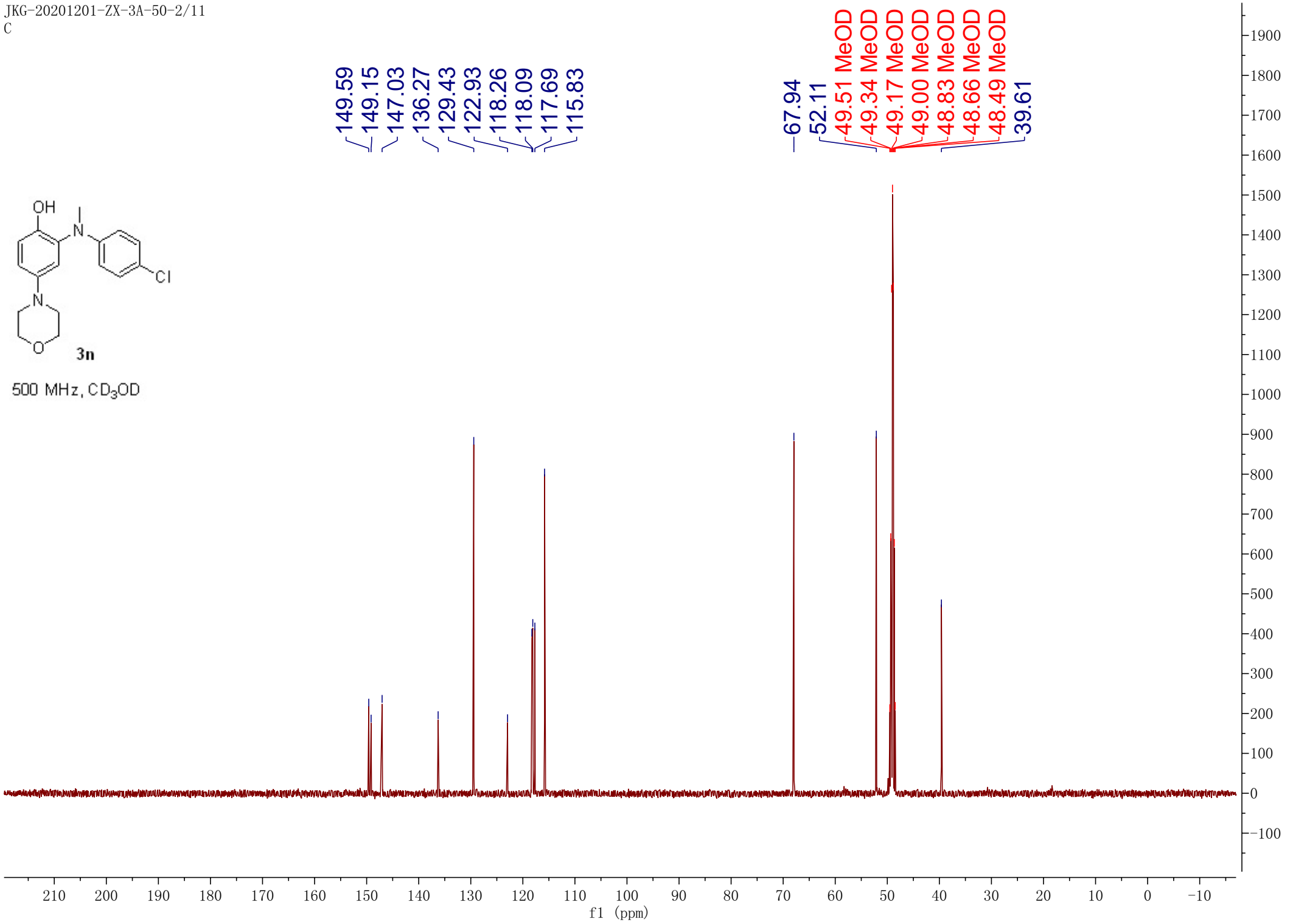
157.68
155.79
146.75
146.18
145.64
145.63
135.90
116.40
115.93
115.87
115.70
115.62
115.44
114.80
77.25 CDCl3
77.00 CDCl3
76.75 CDCl3
66.90
50.61
40.24

f1 (ppm)

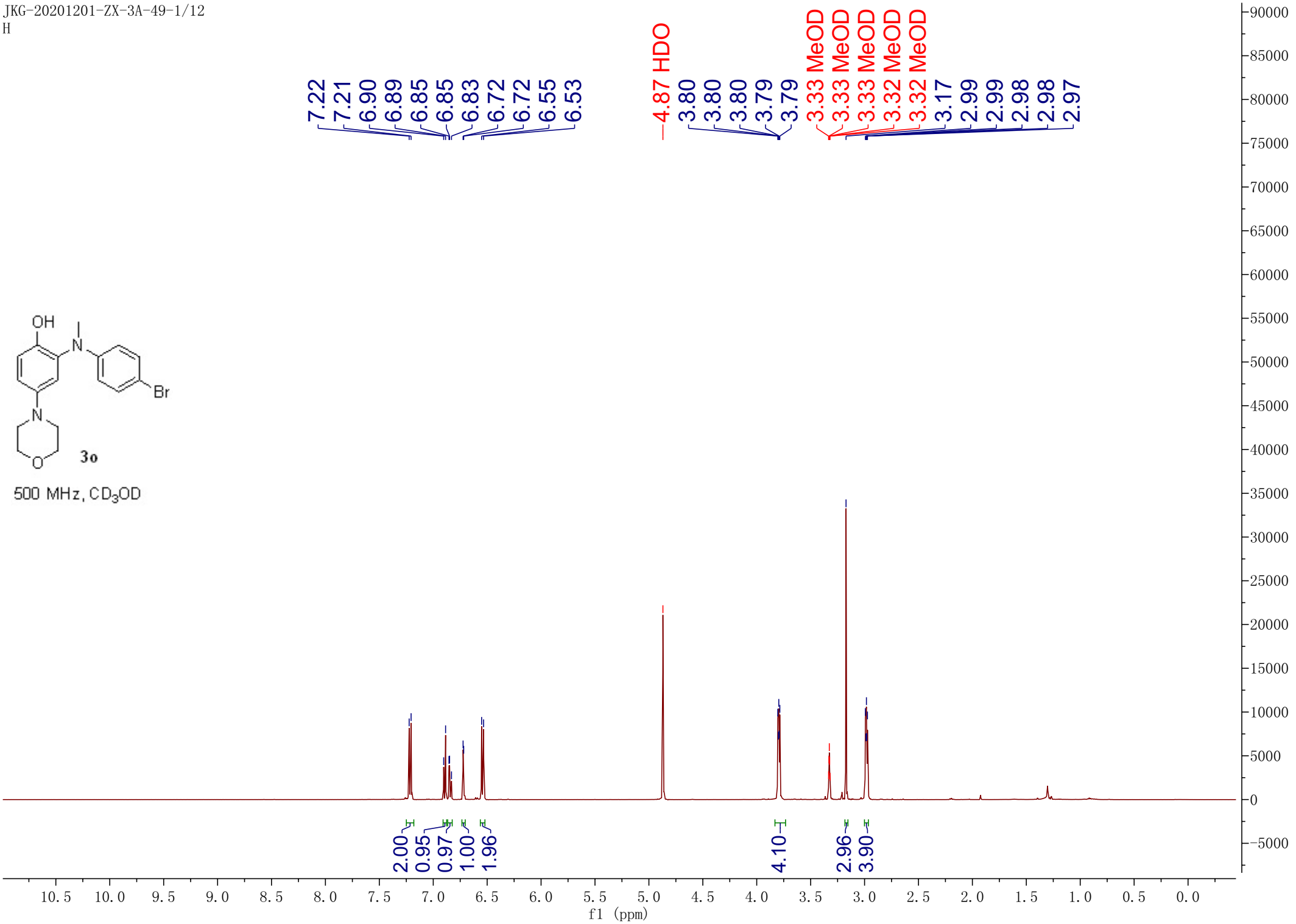
JKG-20201201-ZX-3A-50-2/10
H



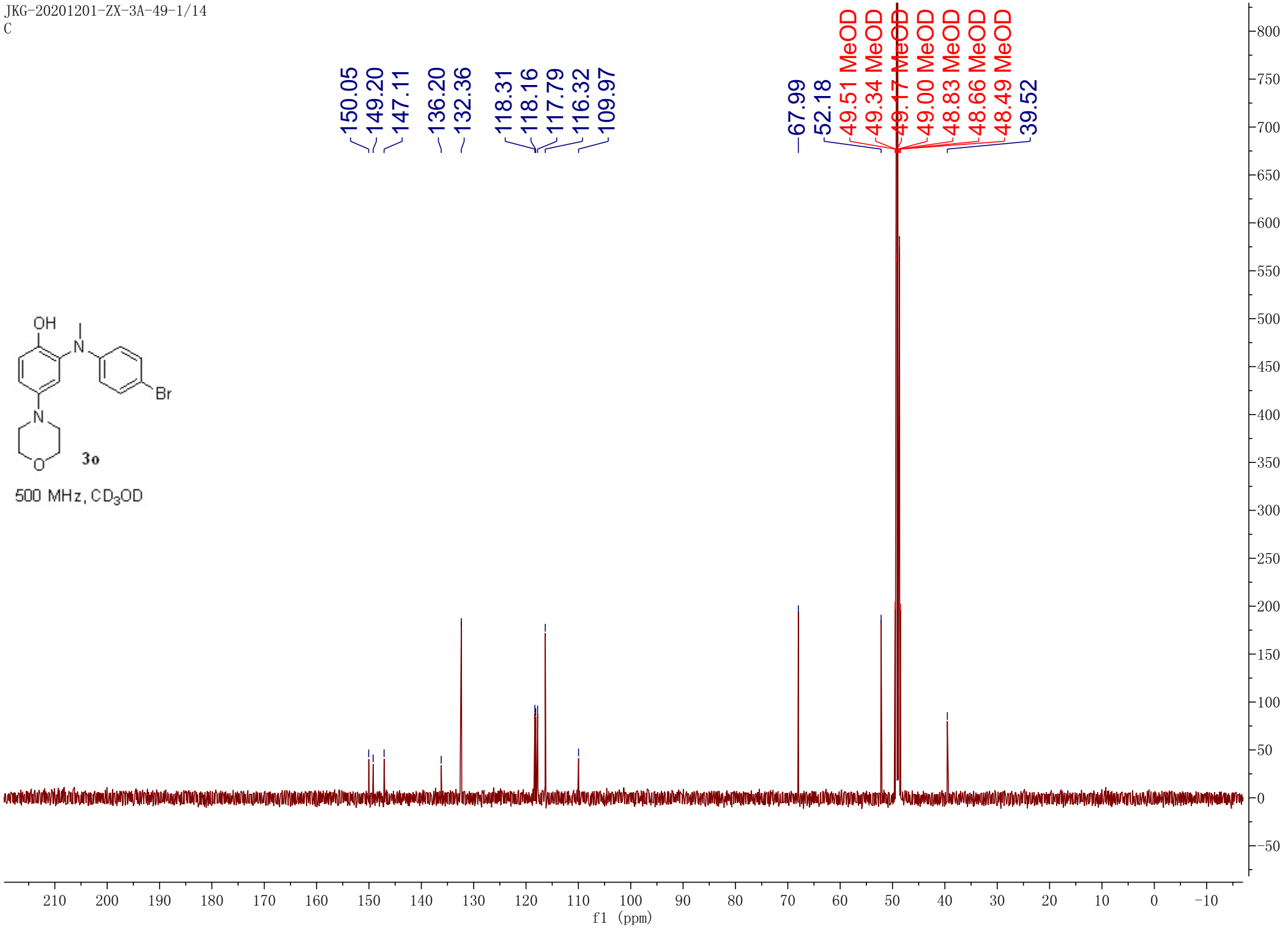
JKG-20201201-ZX-3A-50-2/11
C



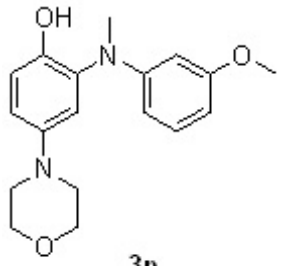
JKG-20201201-ZX-3A-49-1/12
H



JKG-20201201-ZX-3A-49-1/14
C



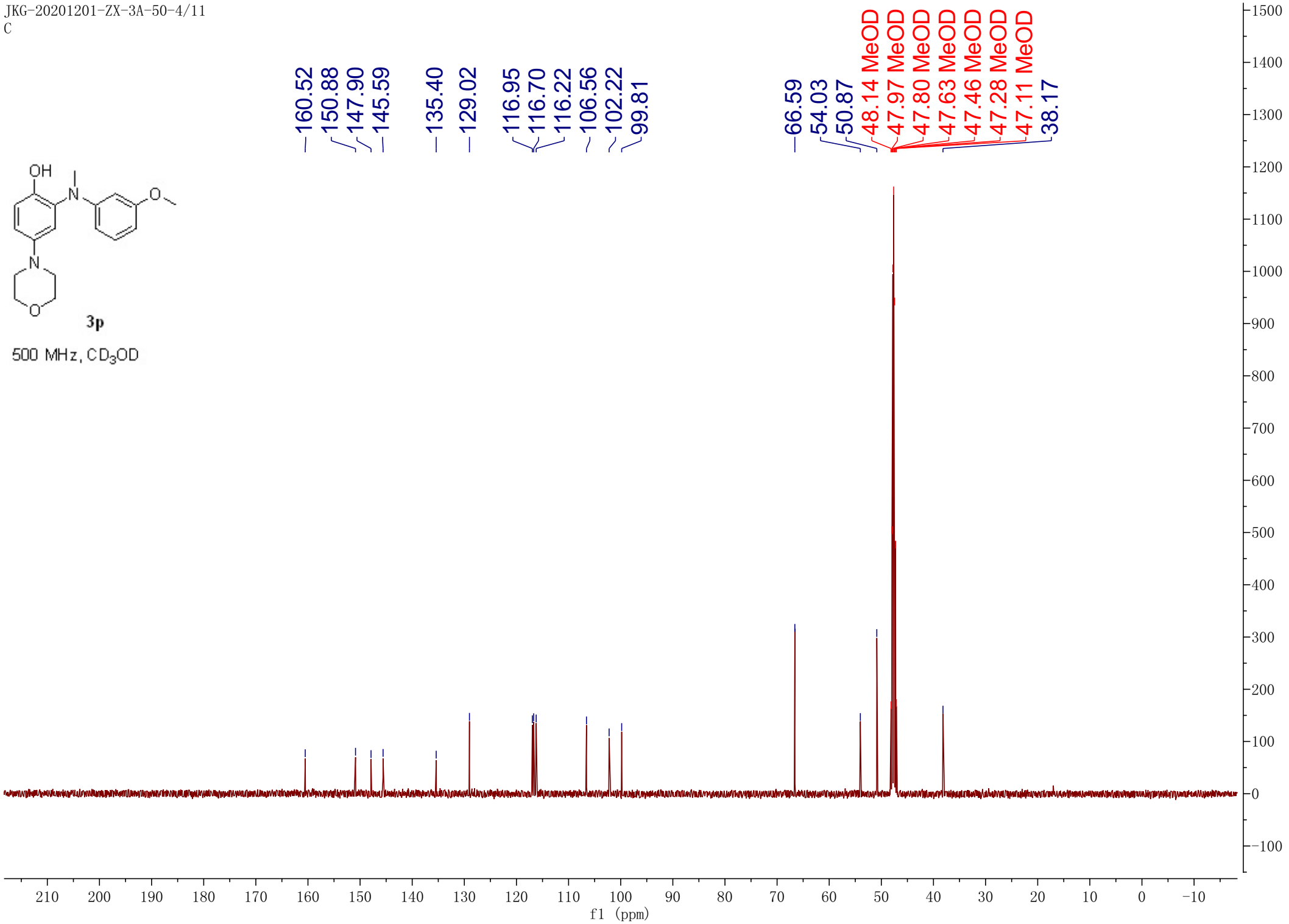
JKG-20201201-ZX-3A-50-4/10
H



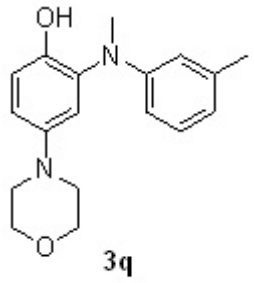
3p

500 MHz, CD_3OD

JKG-20201201-ZX-3A-50-4/11
C



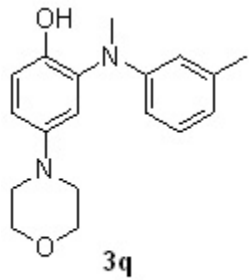
JKG-20201211-ZX-3A-54-3/10
PROTON MeOD {D:\2020-3} ZHL 45



3q

500 MHz, CD_3OD

JKG-20201211-ZX-3A-54-3/11
C13CPD MeOD {D:\2020-3} ZHL 45



3q

500 MHz, CD_3OD

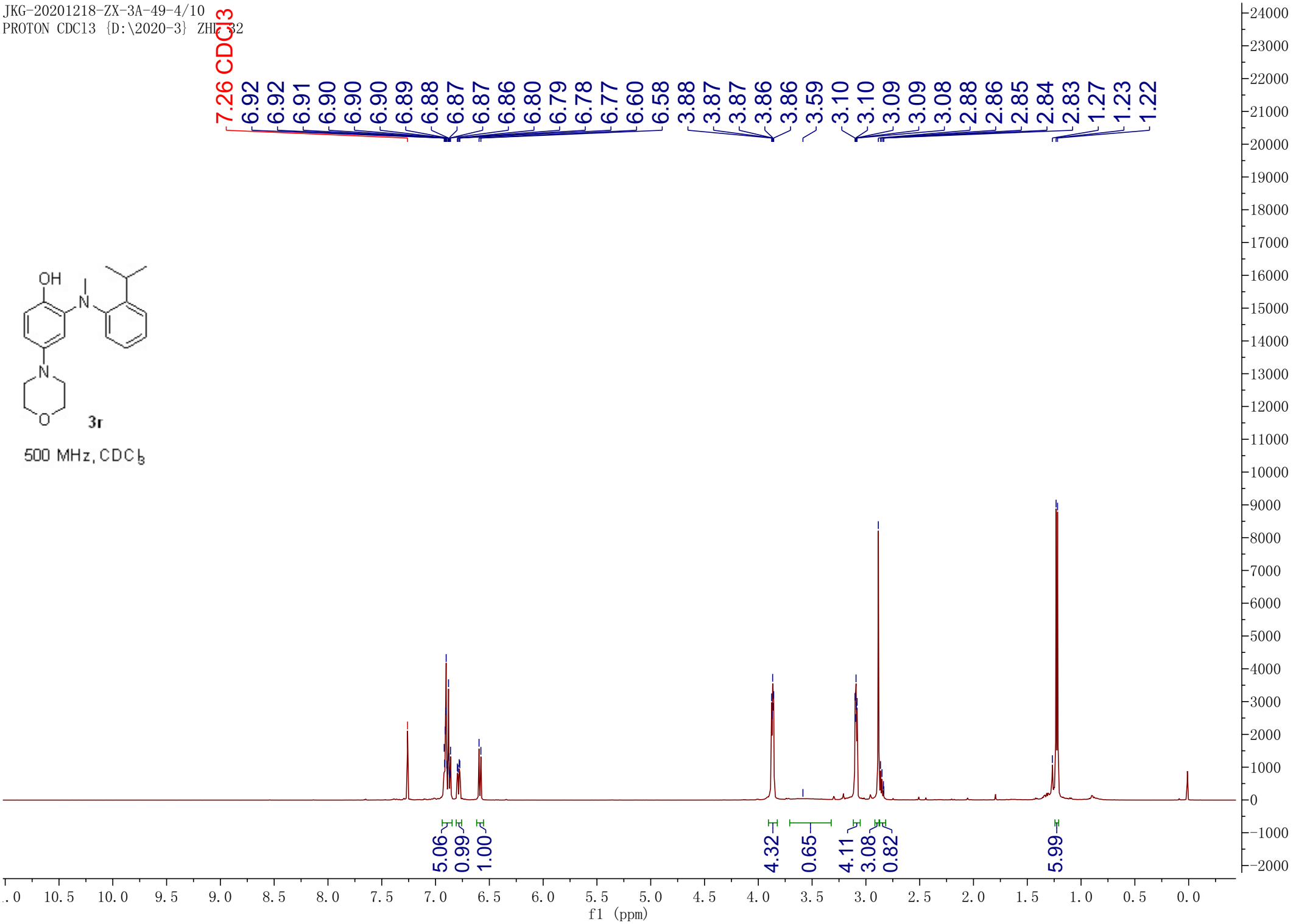
150.97
149.31
146.98
139.30
137.19
129.57
119.44
118.22
117.96
117.40
115.64
112.32

67.98
52.27
49.51 MeOD
49.34 MeOD
49.17 MeOD
49.00 MeOD
48.83 MeOD
48.66 MeOD
48.49 MeOD
39.55

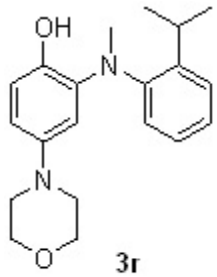
21.87

f1 (ppm)

JKG-20201218-ZX-3A-49-4/10
PROTON CDCl3 {D:\2020-3} ZHU 32



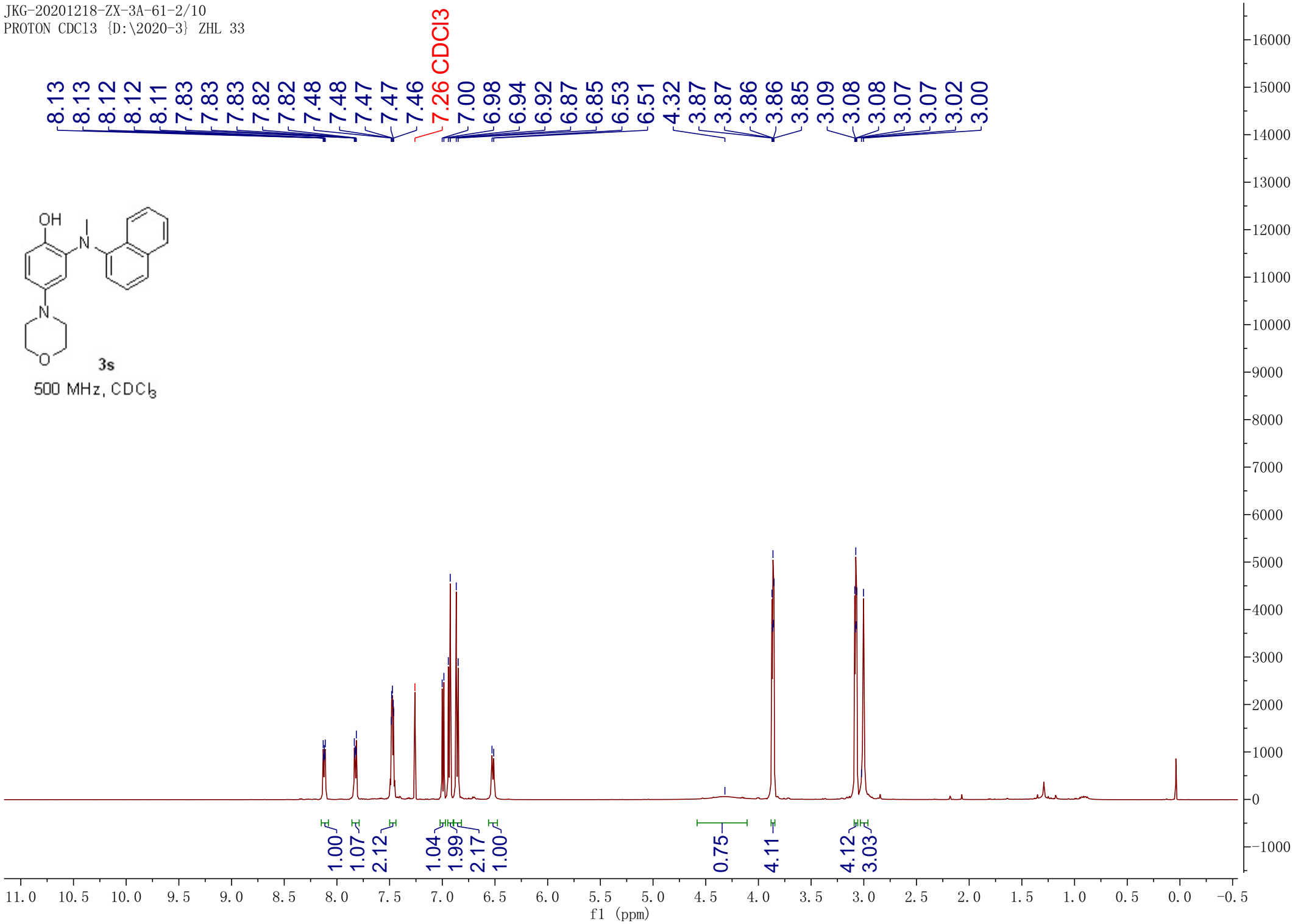
JKG-20201218-ZX-3A-49-4/11
C13CPD CDCl3 {D:\2020-3} ZHL 32



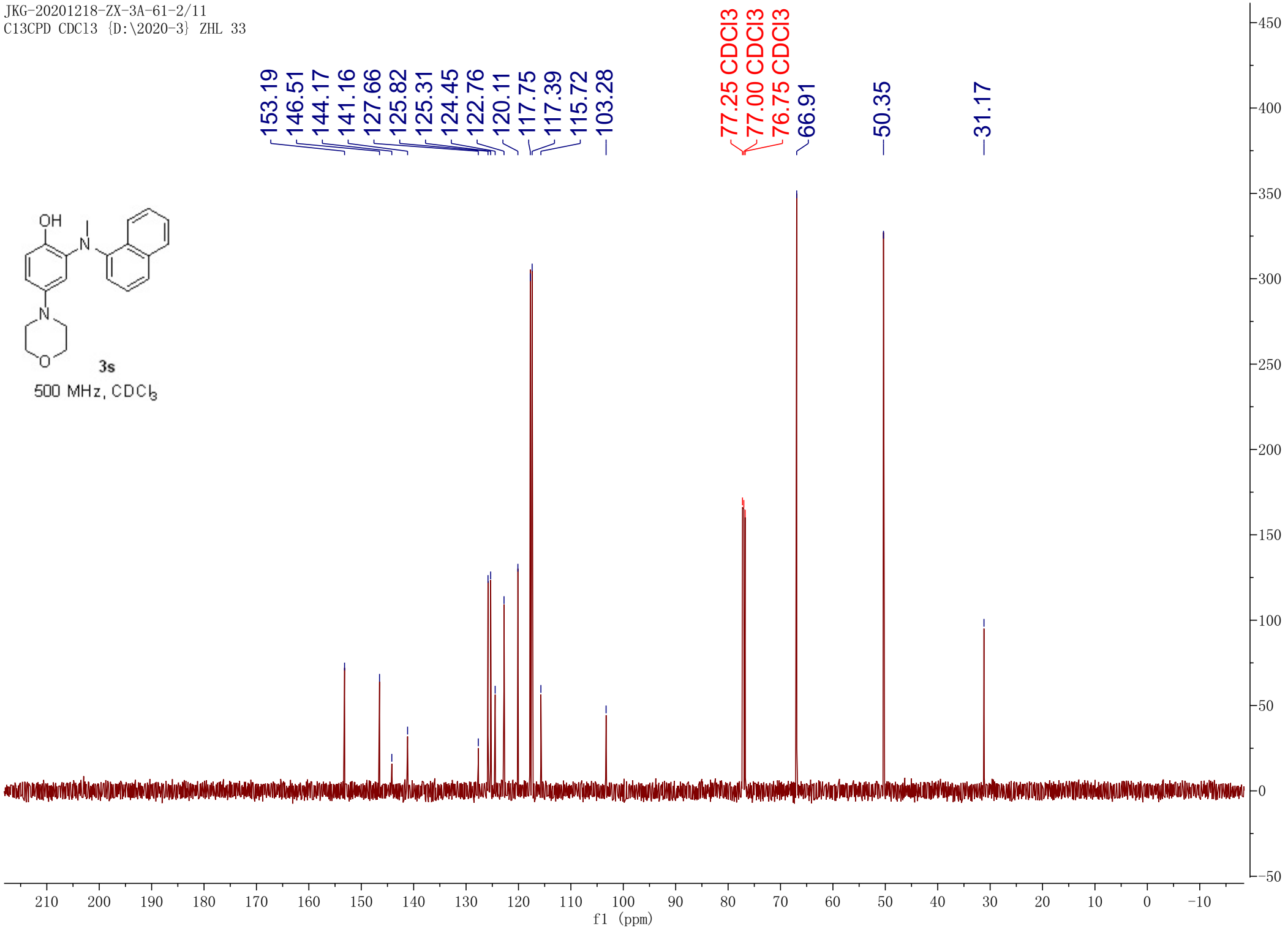
3r

500 MHz, $CDCl_3$

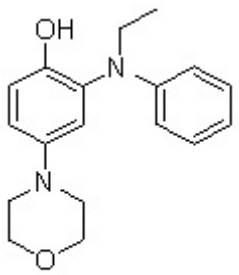
JKG-20201218-ZX-3A-61-2/10
PROTON CDCl3 {D:\2020-3} ZHL 33



JKG-20201218-ZX-3A-61-2/11
C13CPD CDCl3 {D:\2020-3} ZHL 33



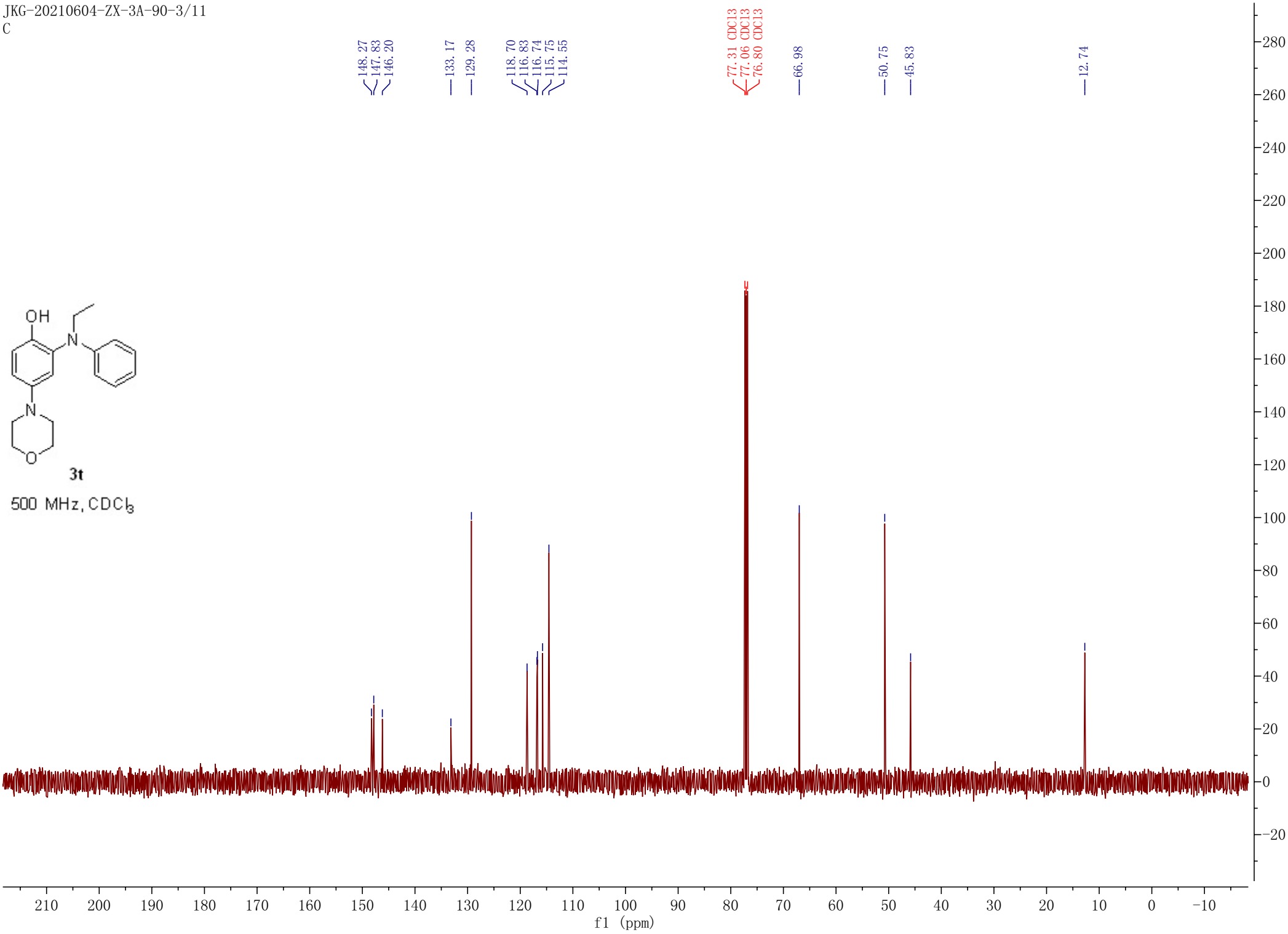
ZX-3A-91-3.20.1.1r



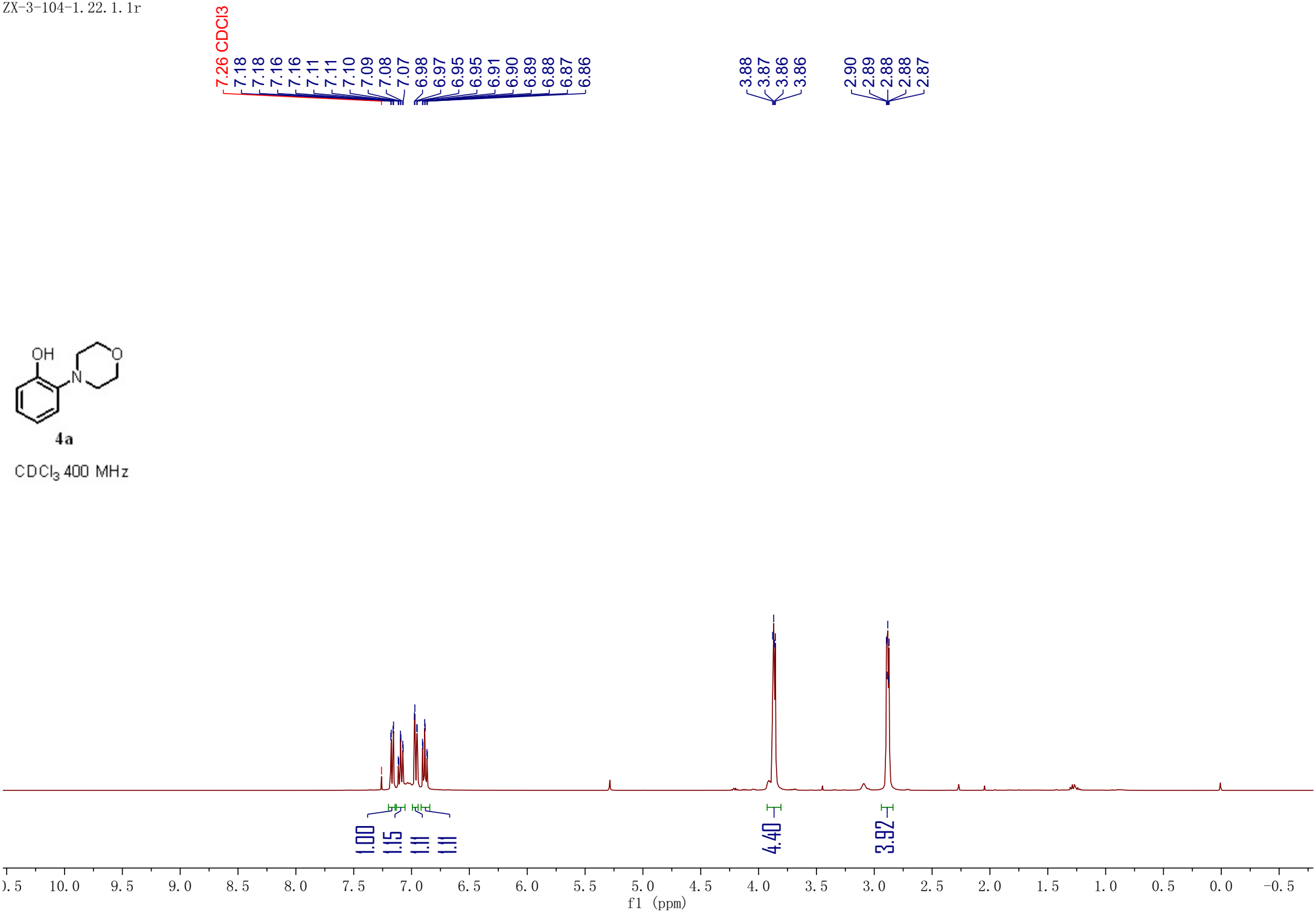
3t

400 MHz, CD_3OD

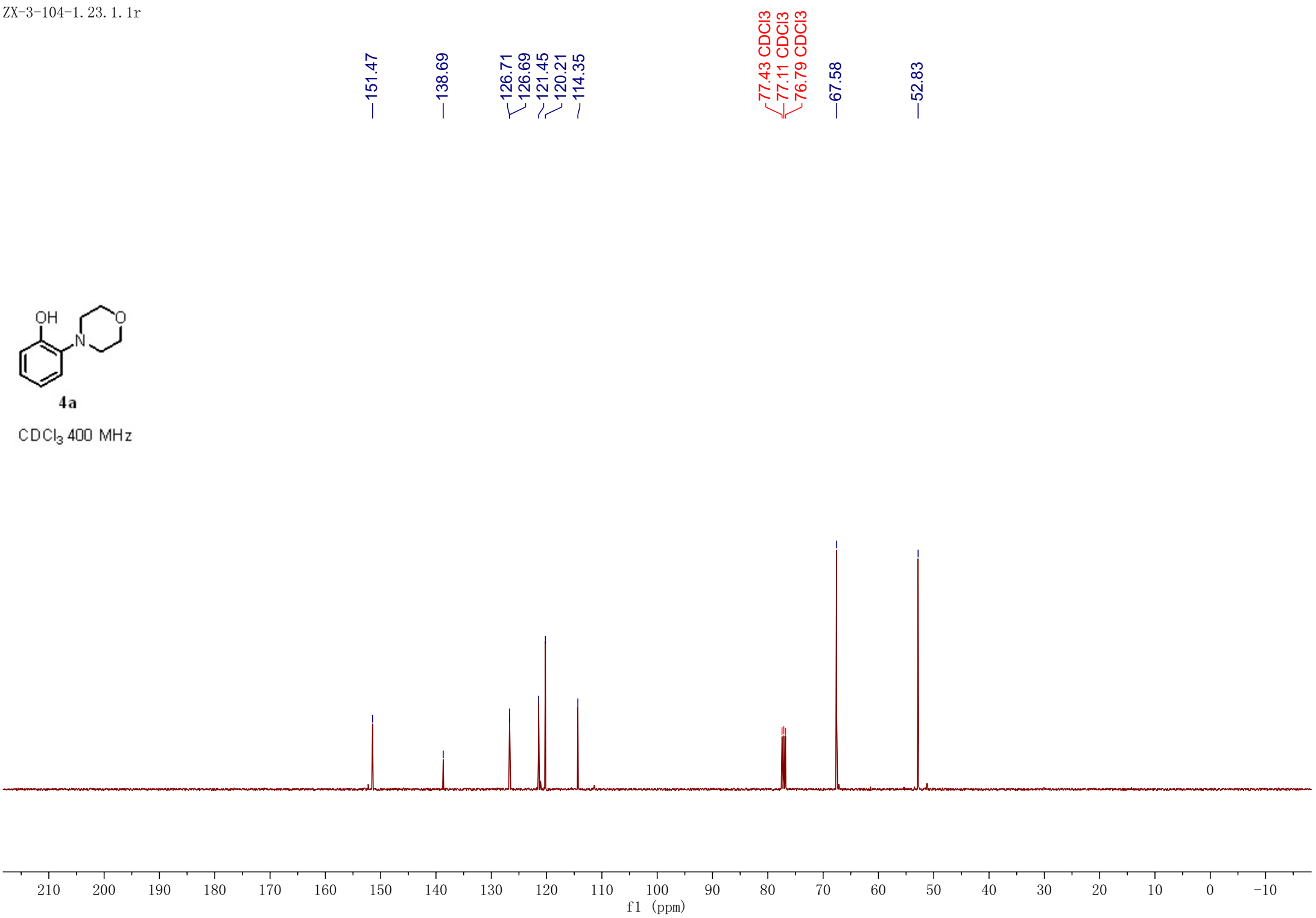
JKG-20210604-ZX-3A-90-3/11
C



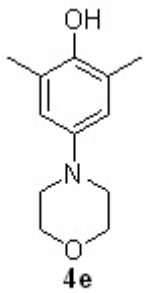
ZX-3-104-1.22.1.1r



ZX-3-104-1.23.1.1r



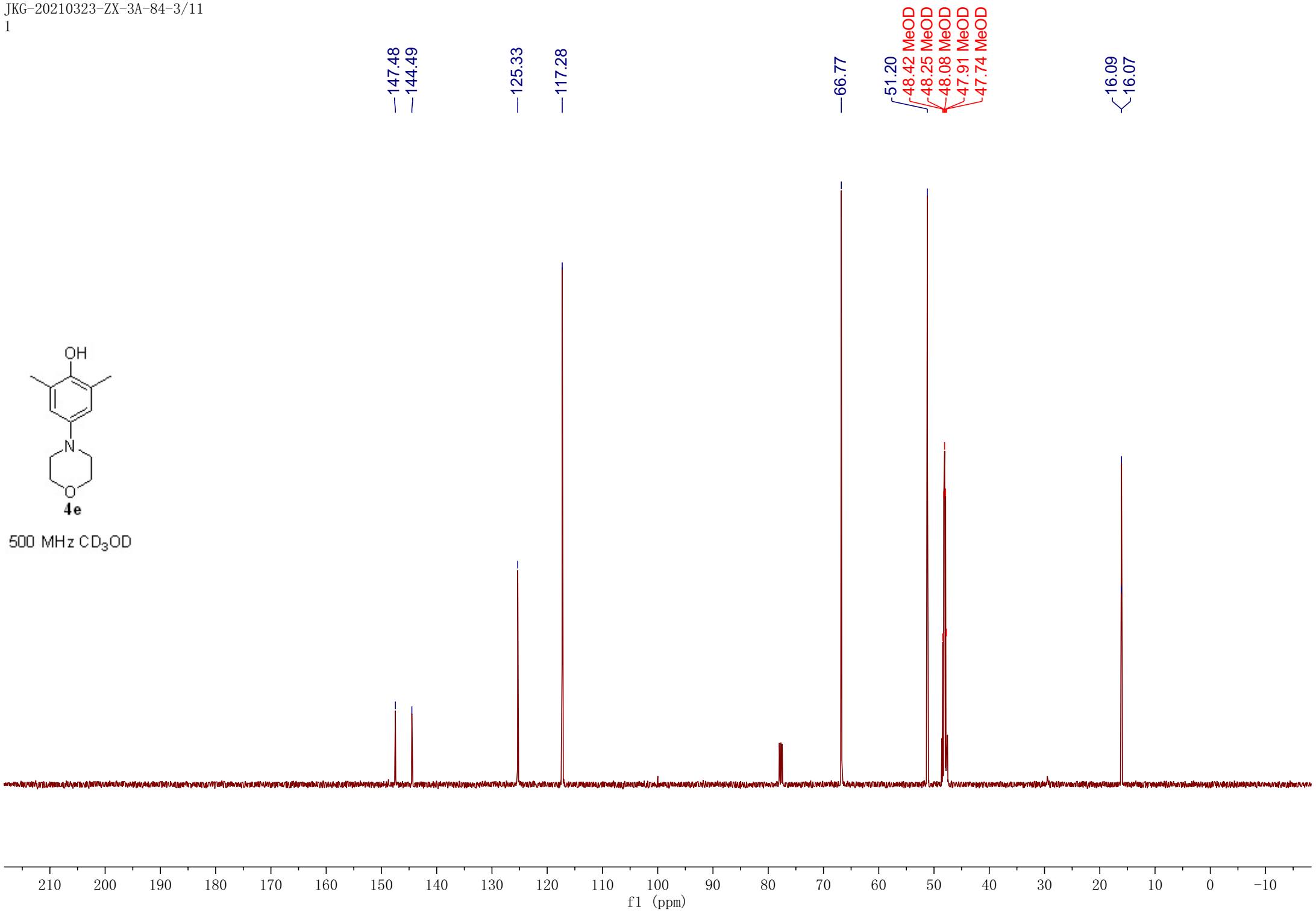
JKG-20210323-ZX-3A-84-3/10
1



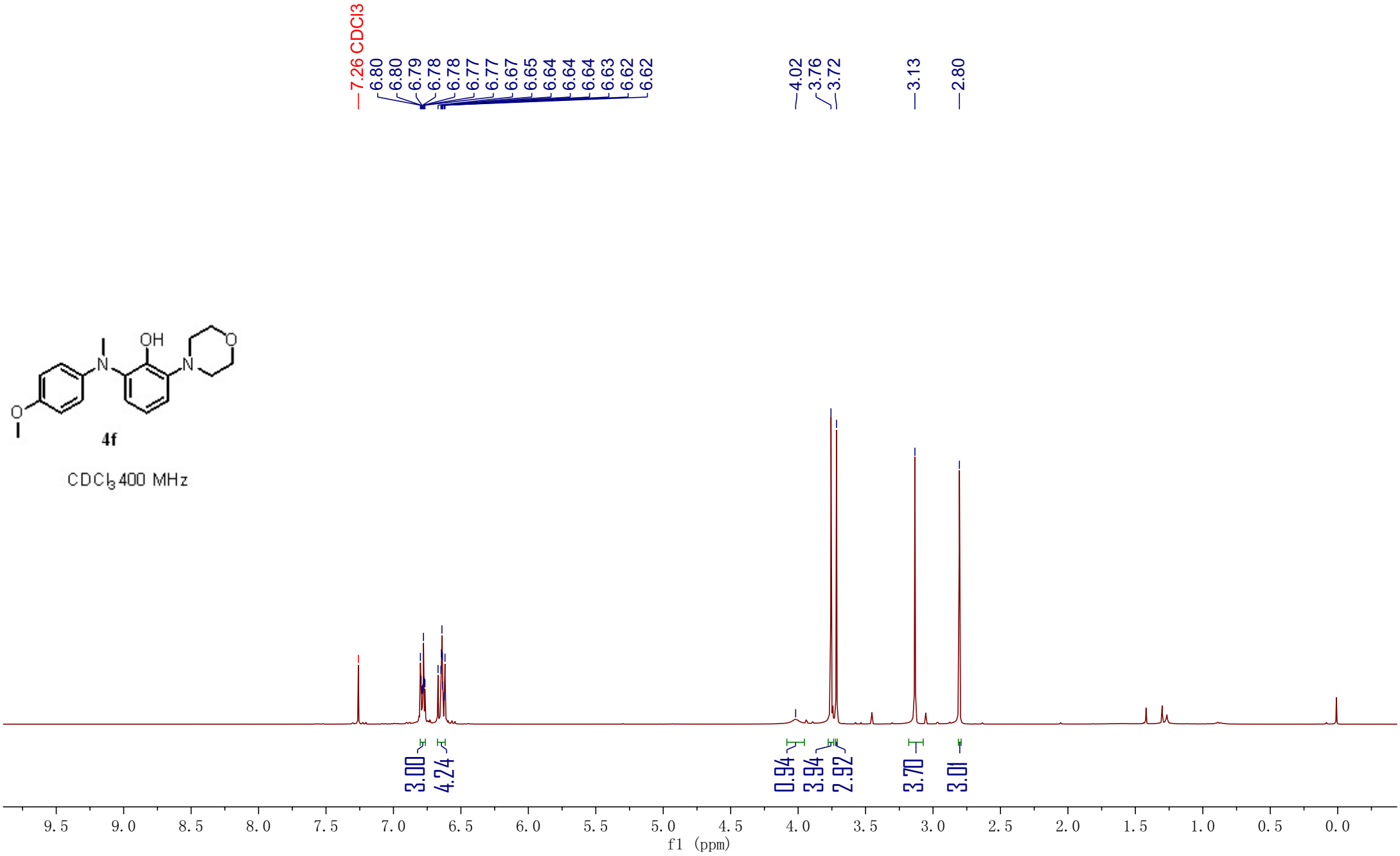
4e

500 MHz CD_3OD

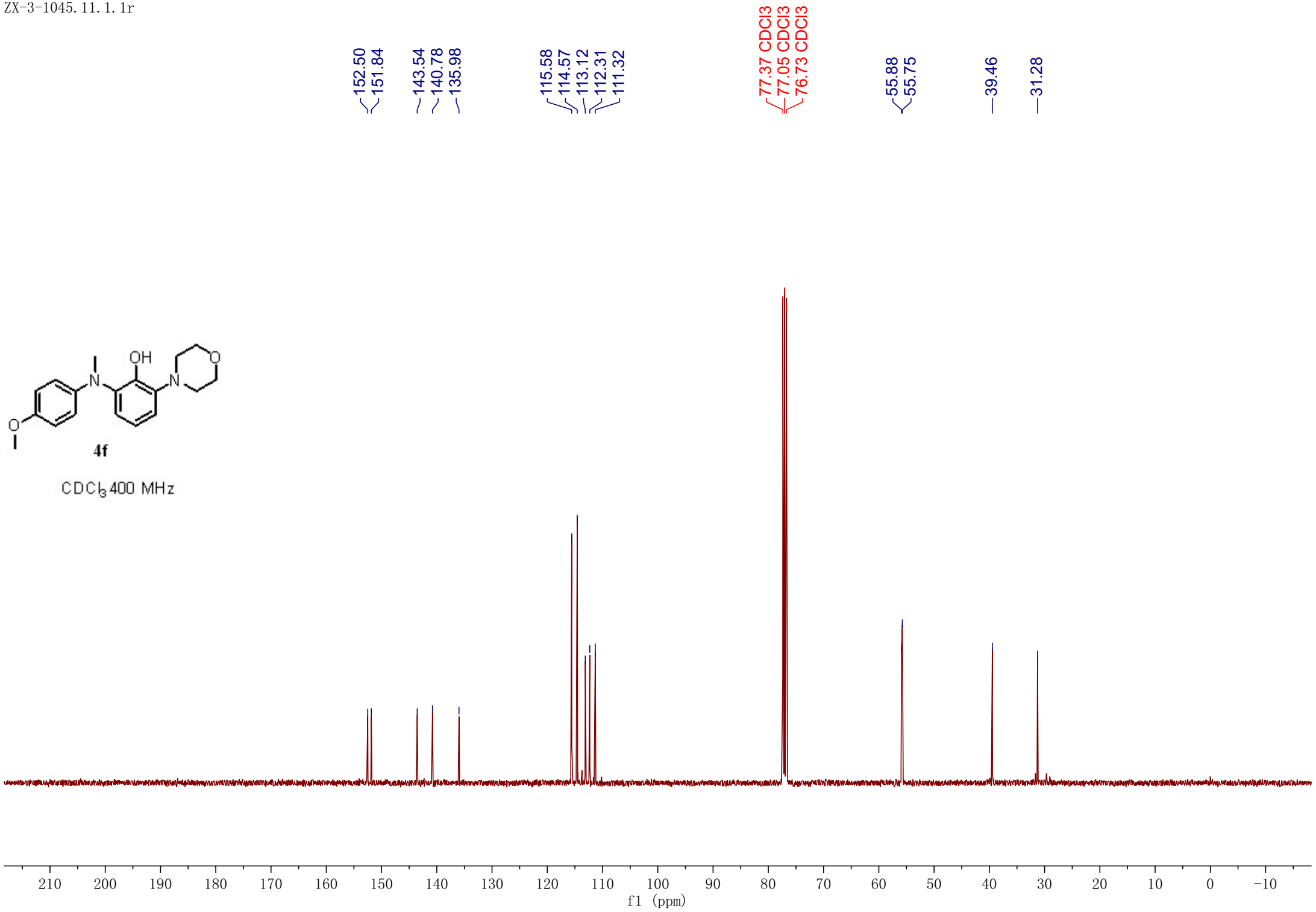
JKG-20210323-ZX-3A-84-3/11
1



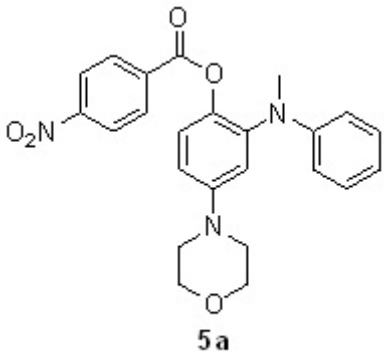
ZX-3-1045.10.1.1r



ZX-3-1045.11.1.1r



ZX-3A-87-6.22.1.1r



5a

400 MHz, $CDCl_3$

ZX-3A-87-6.23.1.1r