

Asymmetric synthesis of atropisomeric arylpyrazoles via direct arylation of 5-aminopyrazoles with naphthoquinones

Jiamin Liu, Xingfu Wei, Yue Wang, Jingping Qu and Baomin Wang*

State Key Laboratory of Fine Chemicals, Department of Pharmaceutical Engineering, School of Chemical Engineering, Dalian University of Technology, Dalian 116024, People's Republic of China

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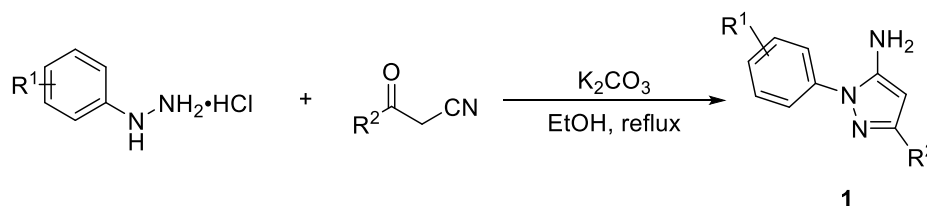
1. General information

Unless otherwise noted, materials were purchased from commercial suppliers and used without further purification. Column chromatography was performed on silica gel (200~300 mesh). Enantiomeric excesses (ee) were determined by HPLC using corresponding commercial chiral columns as stated at 30 °C with UV detector at 254 nm. Optical rotations were reported as follows: $[\alpha]_D^{25}$ (c g/100 mL, solvent). All ^1H NMR spectra were recorded on a Bruker Avance II 400 MHz and Bruker Avance III 600 MHz, ^{13}C NMR spectra were recorded on a Bruker Avance II 101 MHz and Bruker Avance III 151 MHz, ^{19}F NMR spectra were recorded on a Bruker Avance II 376 MHz and Bruker Avance III 377 MHz with chemical shifts reported as ppm (in CDCl_3 , TMS as an internal standard). Data for ^1H NMR are recorded as follows: chemical shift (δ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, br = broad singlet, dd = double doublet, coupling constants in Hz, integration). HRMS (ESI) was obtained with a HRMS/MS instrument (LTQ Orbitrap XL TM). The absolute configuration of **3ha** was assigned by the X-ray analysis.

Catalyst **C6** was synthesized according to the literature procedure.¹ The racemic products were synthesized using diphenylphosphinic acid as the catalyst.

2. Procedures for the preparation of substrate 1

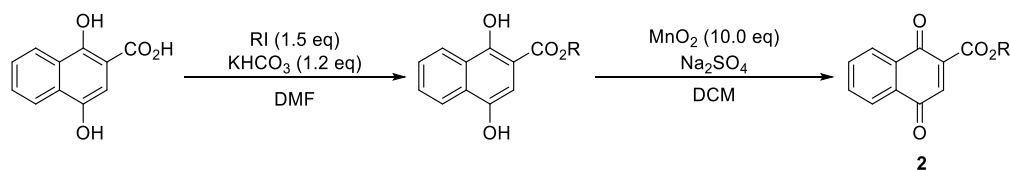
5-Aminopyrazoles **1** were prepared according to the following procedure:²



A solution of arylhydrazine hydrochloride (10 mmol, 1.0 equiv), potassium carbonate (11 mmol, 1.1 equiv) and alkyl formyl acetonitrile (11 mmol, 1.1 equiv) in EtOH (0.5 M relative to arylhydrazine hydrochloride) was heated to reflux for 6 h. After cooling to room temperature, the solution was concentrated under reduced pressure. The residue was dissolved in CH_2Cl_2 and saturated NaHCO_3 (aq.). The layers were separated, and the aqueous layer was extracted twice with CH_2Cl_2 . The combined organic layers were washed with brine and dried with anhydrous Na_2SO_4 . The salts were removed via gravity filtration, and the resulting solution was concentrated under reduced pressure. The crude mixture was purified by flash column chromatography on silica gel. In addition, substrates **1s** and **1t** were synthesized according to the literature procedure.³

3. Procedures for the preparation of substrate 2

1,4-Naphthoquinone derivatives **2** were prepared according to the following procedure:⁴



Solid KHCO_3 (0.92 g, 11 mmol) was added to a stirred mixture of 1,4-dihydroxy-2-naphthoic acid (2.04 g, 10 mmol) and iodoalkane derivatives (11 mmol) in DMF (20 mL). The mixture was heated to

70 °C in an oil bath and stirred for 24 h. The reaction mixture was cooled down to room temperature, diluted with 1 M HCl (50 mL), and extracted with DCM (30 mL x 2). The organic phases were washed with water (20 mL x 2) and dried over MgSO₄. After evaporation of the solvent, the residue was subjected to column chromatography on silica gel with petroleum ether/ethyl acetate 10:1 as eluting solvent to yield 1,4-dihydroxy-2-naphthoate.

Manganese dioxide (6.0 mmol) and magnesium sulfate (3.0 mmol) were added to the solution of 1,4-dihydroxy-2-naphthoate (1.0 mmol) and DCM (20 mL). The reaction mixture was stirred at 25 °C for 5 h, and the filtrate filtered by diatomite was evaporated in a vacuum to obtain the required quinone **2**.

4. The racemization experiment

The enantiomerisation barrier was obtained by kinetic of racemization of an enantiomer. The slope of the first order kinetic line gives the racemization ($k_{ent} = \frac{1}{2} k_{rac}$). Eyring equation gives the enantiomerisation barrier (ΔG_T^\ddagger) from enantiomerisation constant (k_{ent}), $R = 8.31451 \text{ J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$, $h = 6.62608 \times 10^{-34} \text{ J}\cdot\text{s}$, $k_B = 1.38066 \times 10^{-23} \text{ J}\cdot\text{K}^{-1}$. Enantiomeric excess values were determined by HPLC.

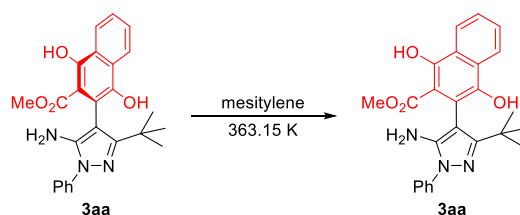


Table S1. Racemization of **3aa** in mesitylene at 90 °C.

p/m	(1+(p/m))/(1-(p/m))	t/s	ln[(1+(p/m))/(1-(p/m))]
0.065	1.138	0	0.129
0.068	1.145	1800	0.136
0.072	1.155	3600	0.144
0.074	1.161	5400	0.149
0.077	1.168	7200	0.155
0.080	1.175	9000	0.161
0.082	1.179	10800	0.165
0.086	1.188	12600	0.172
0.089	1.196	14400	0.179
0.093	1.205	16200	0.187

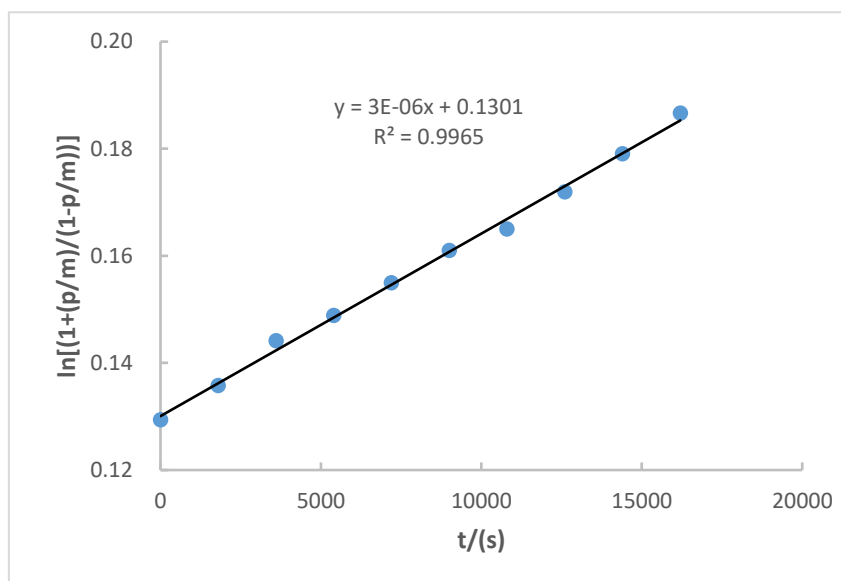


Figure S1. The plot of $\ln[(1+(p/m))/(1-(p/m))]$ vs time of **3aa** at 90 °C

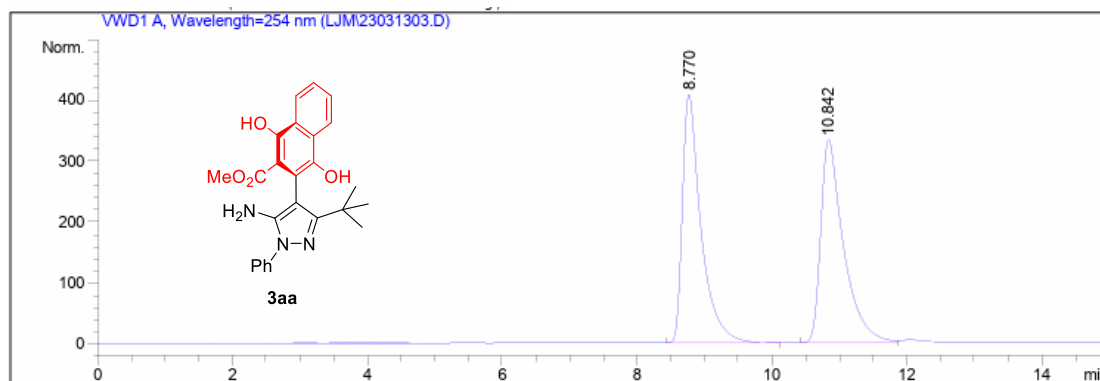
T = 90 °C (mesitylene); $k_{ent} = \frac{1}{2} k_{rac} = \frac{1}{2} slope = 1.5 \times 10^{-6} \text{ s}^{-1}$; $t_{1/2} = \ln 2/k_{rac} = 64.2 \text{ h}$;

$\Delta G_T^\ddagger = \ln\left(\frac{k_B T}{h \times k_{ent}}\right) RT = 130.03 \text{ kJ}\cdot\text{mol}^{-1} = 31.1 \text{ kcal}\cdot\text{mol}^{-1}$

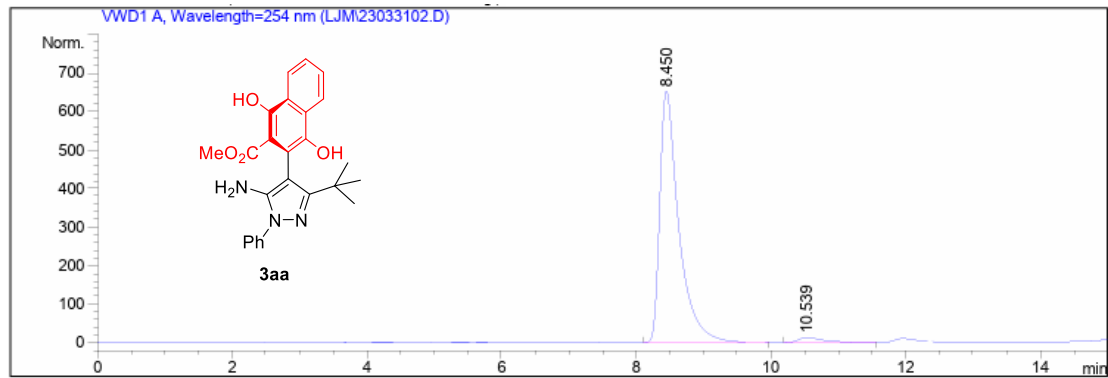
T = 25 °C; $k_{rac} = 2.06 \times 10^{-10} \text{ s}^{-1}$; $t_{1/2} = \ln 2/k_{rac} = 107 \text{ years}$;

5. HPLC traces

Compound 3aa

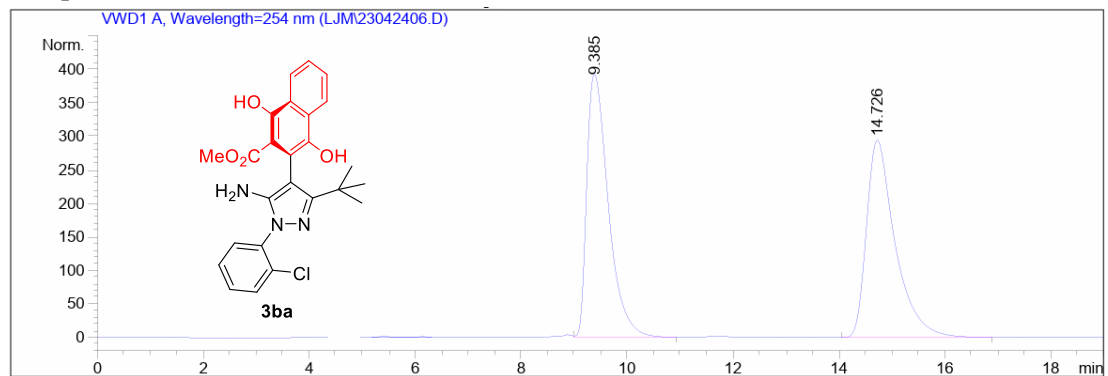


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	8.770	PB	0.2797	7782.17090		407.22647	50.0535
2	10.842	BV	0.3382	7765.54541		332.29099	49.9465

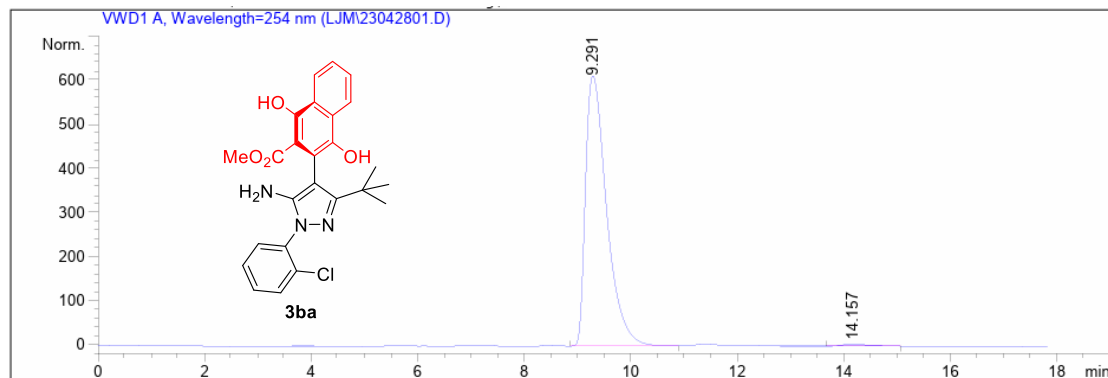


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.450	PB	0.2872	1.27107e4	651.93805	97.5408
2	10.539	PB	0.3487	320.45697	13.20418	2.4592

Compound 3ba

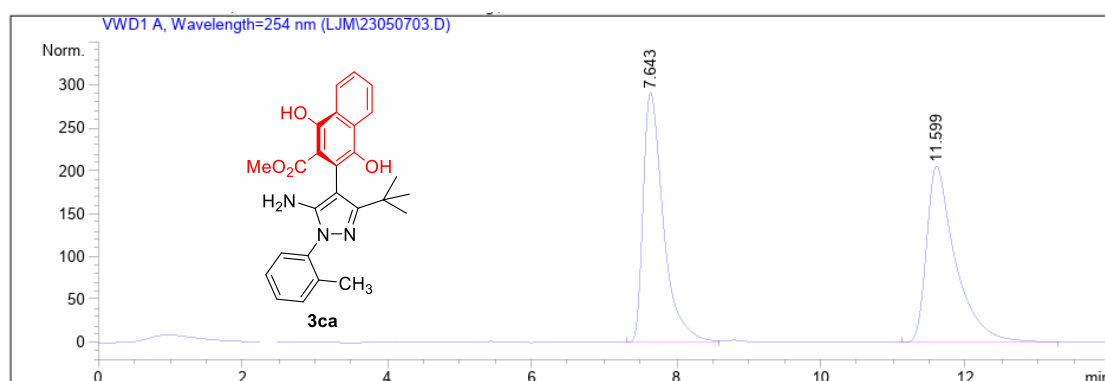


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.385	VB	0.4397	1.10865e4	393.07452	50.0192
2	14.726	BB	0.5582	1.10780e4	294.55469	49.9808

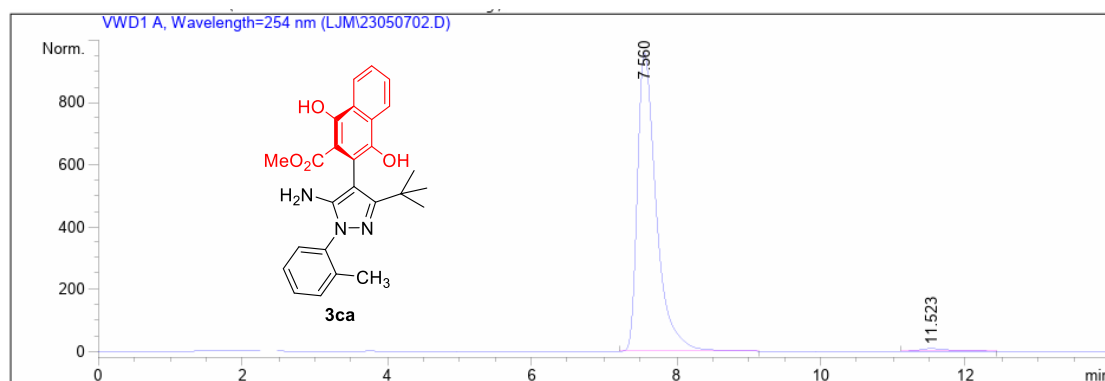


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.291	BB	0.4232	1.68656e4	612.52307	99.1009
2	14.157	BB	0.4670	153.01498	4.87020	0.8991

Compound 3ca

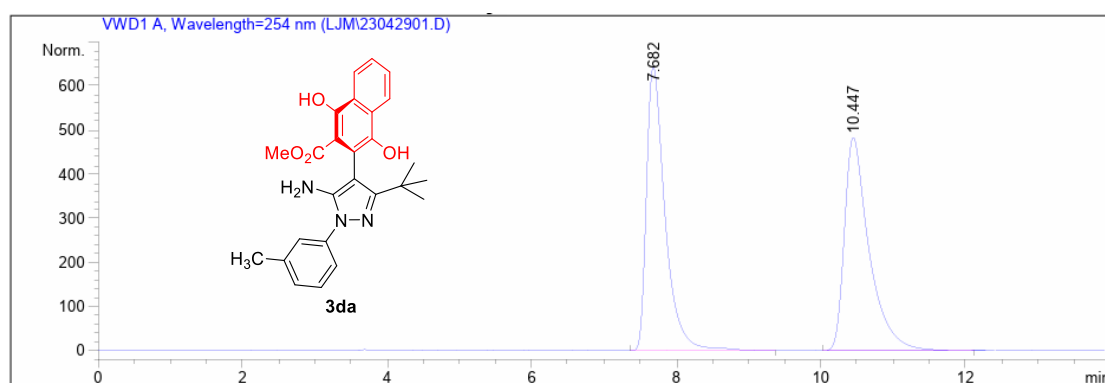


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	7.643	PV	0.2972	5663.50244		290.66556	49.9211
2	11.599	BB	0.4001	5681.41602		205.25821	50.0789

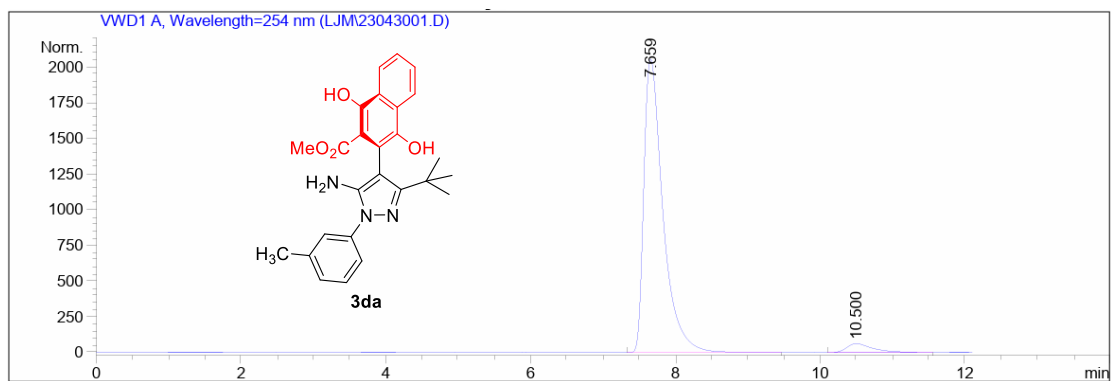


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	7.560	BB	0.2740	1.73701e4		958.64136	98.7197
2	11.523	BP	0.3984	225.27467		8.44999	1.2803

Compound 3da

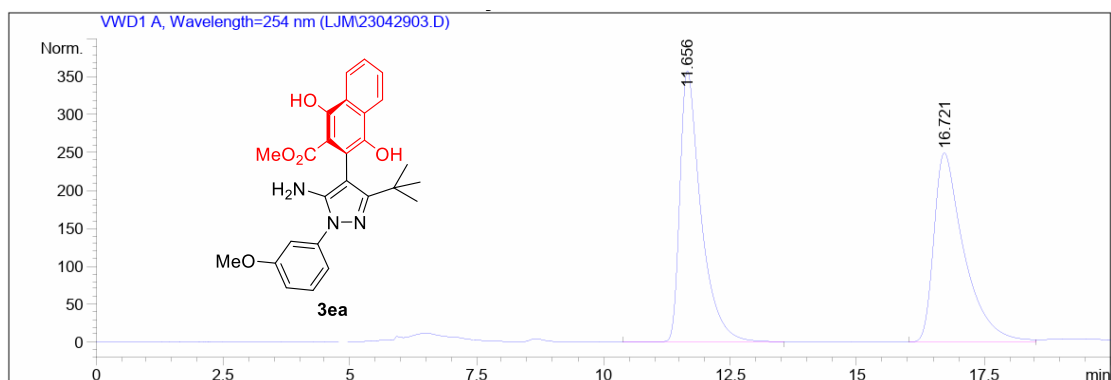


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	7.682	PB	0.2680	1.14761e4		642.88098	50.0791
2	10.447	BB	0.3464	1.14399e4		482.71222	49.9209

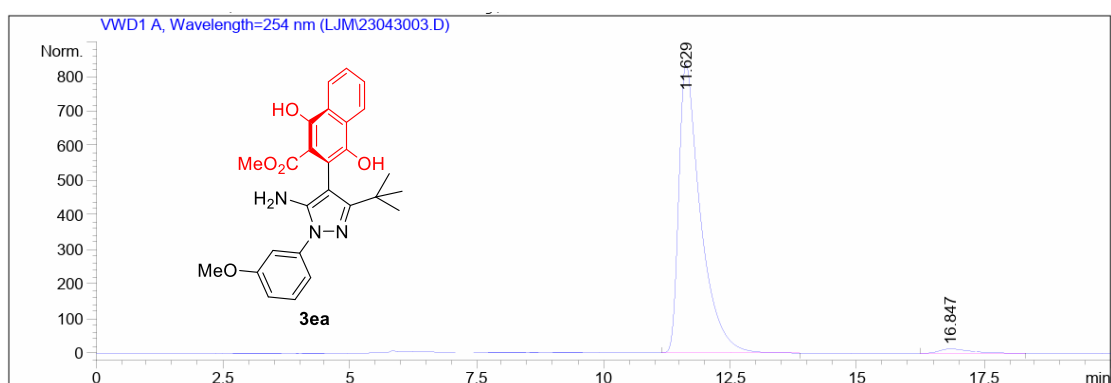


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.659	PB	0.2774	3.75162e4	2051.58301	96.0438
2	10.500	PB	0.3643	1545.34241	62.15542	3.9562

Compound 3ea

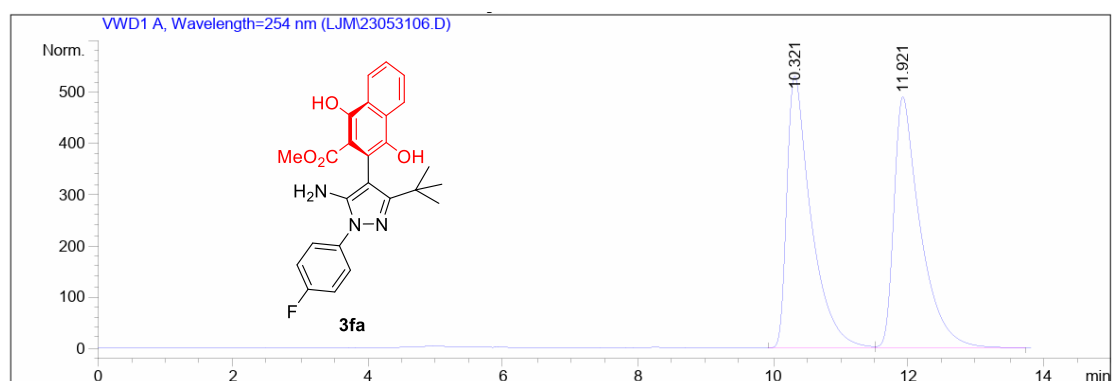


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.656	BB	0.4254	1.03263e4	357.91409	49.9805
2	16.721	BB	0.6009	1.03343e4	249.62901	50.0195

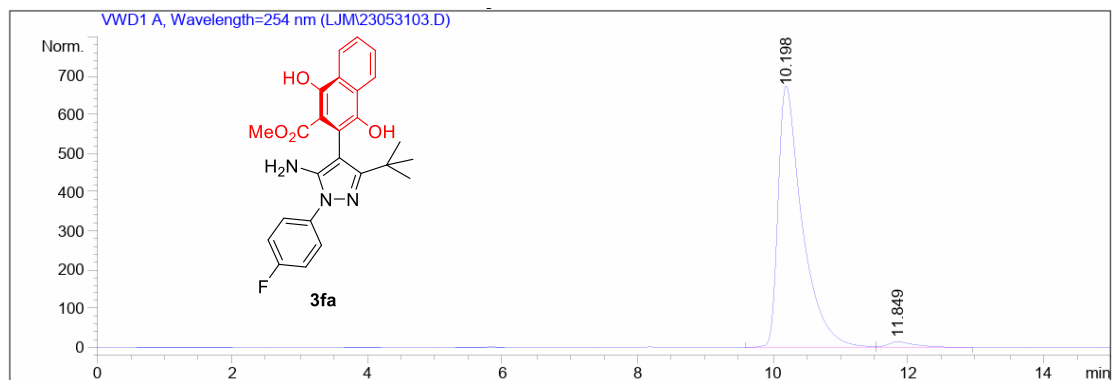


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.629	PB	0.4326	2.47935e4	834.29657	97.8797
2	16.847	BB	0.6100	537.08673	12.77207	2.1203

Compound 3fa

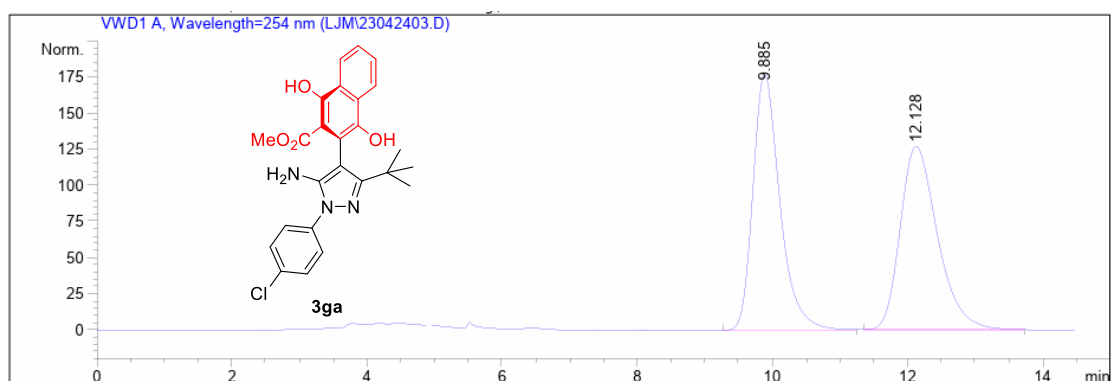


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.321	BV	0.3618	1.32058e4	530.50122	49.8582
2	11.921	VB	0.3903	1.32809e4	490.25571	50.1418

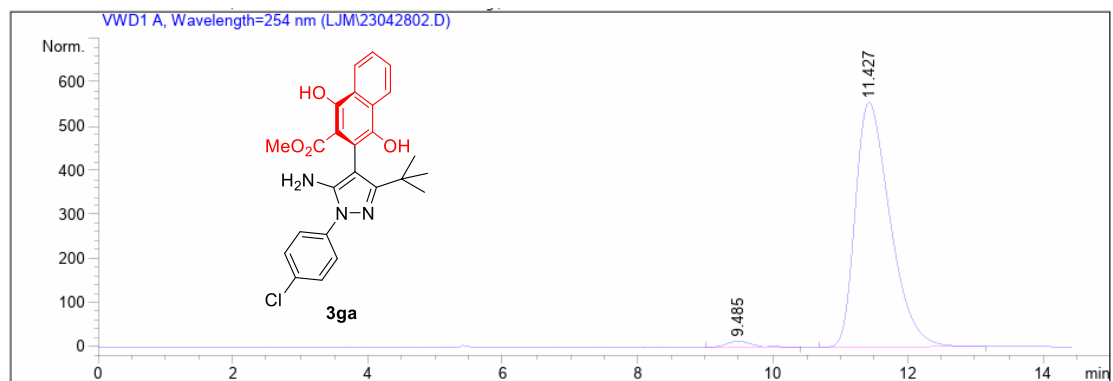


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.198	BV	0.3632	1.67727e4	673.93170	97.5583
2	11.849	VB	0.4005	419.78558	14.81282	2.4417

Compound 3ga

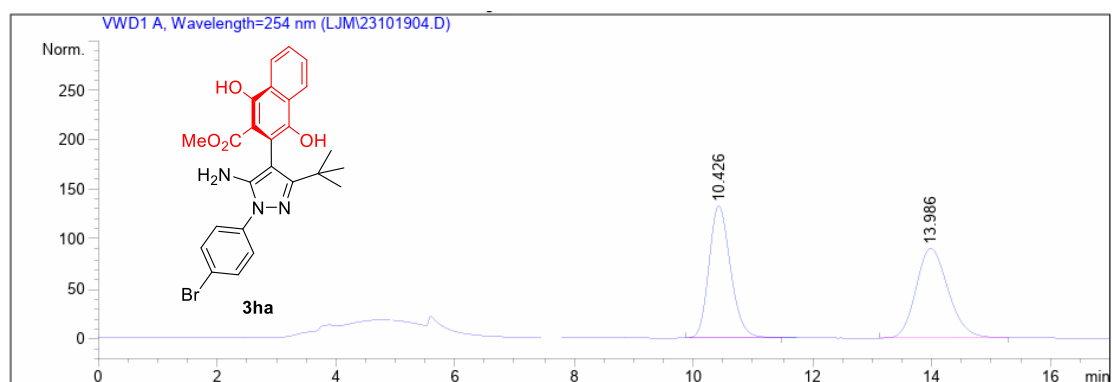


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.885	BB	0.4234	4925.13574	177.13052	50.1538
2	12.128	BB	0.5915	4894.93848	127.24306	49.8462

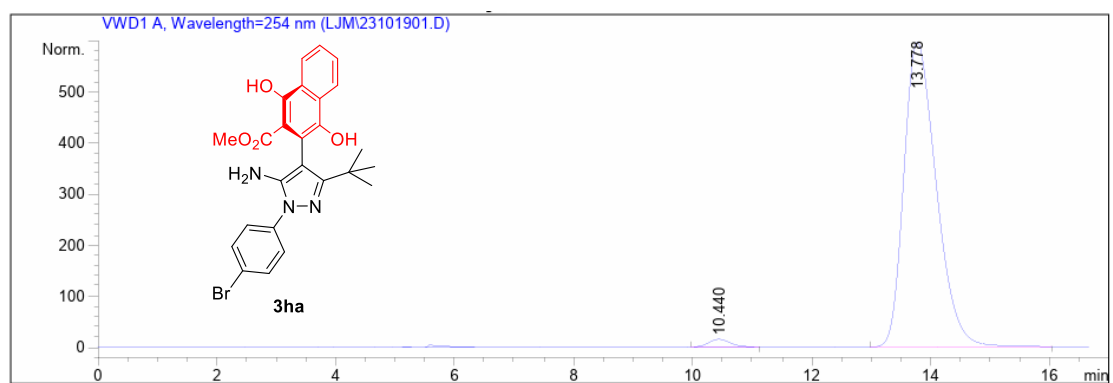


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.485	BB	0.4087	370.25998	13.69192	1.8379
2	11.427	PB	0.5514	1.97756e4	554.60211	98.1621

Compound 3ha

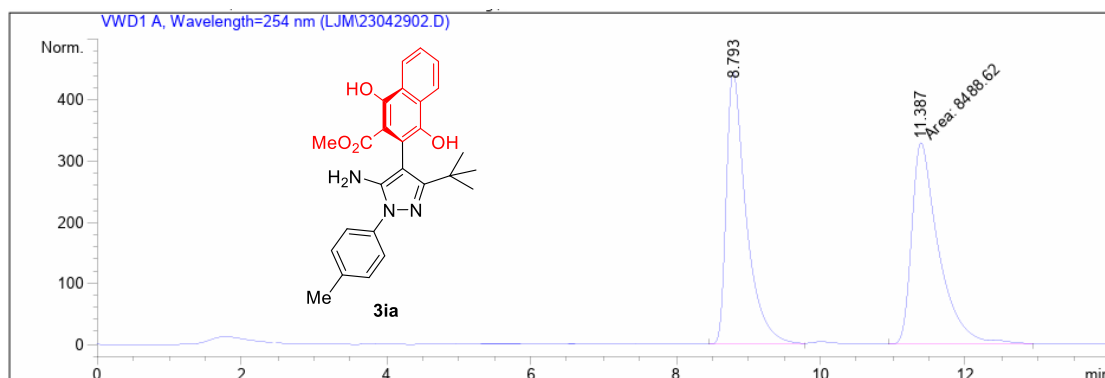


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.426	BB	0.3875	3336.92041	132.95291	49.7320
2	13.986	BB	0.5791	3372.88892	90.16001	50.2680

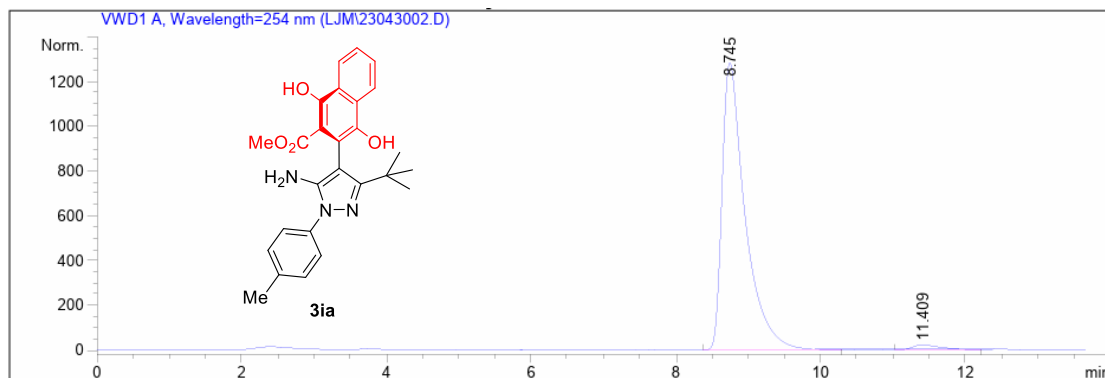


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.440	PB	0.3788	362.06198	14.78962	1.5810
2	13.778	BB	0.5922	2.25381e4	596.58563	98.4190

Compound 3ia

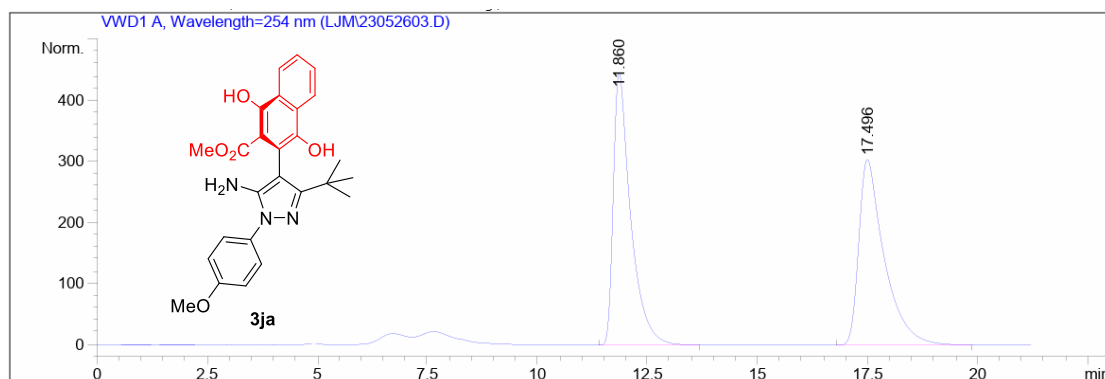


Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	8.793	PV	0.2800	8475.60254		445.90482	49.9616
2	11.387	MM	0.4297	8488.61621		329.21371	50.0384

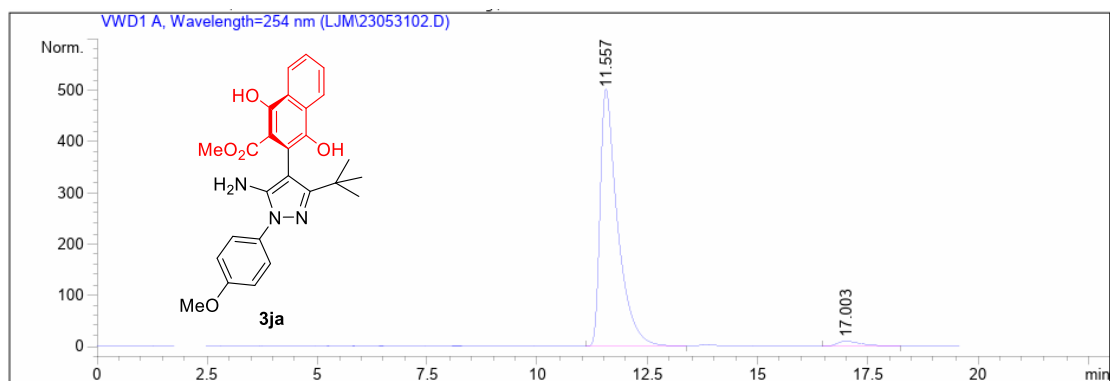


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1	8.745	PB	0.3214	2.83127e4		1283.47046	97.8048
2	11.409	VV	0.3994	635.46954		22.80053	2.1952

Compound 3ja

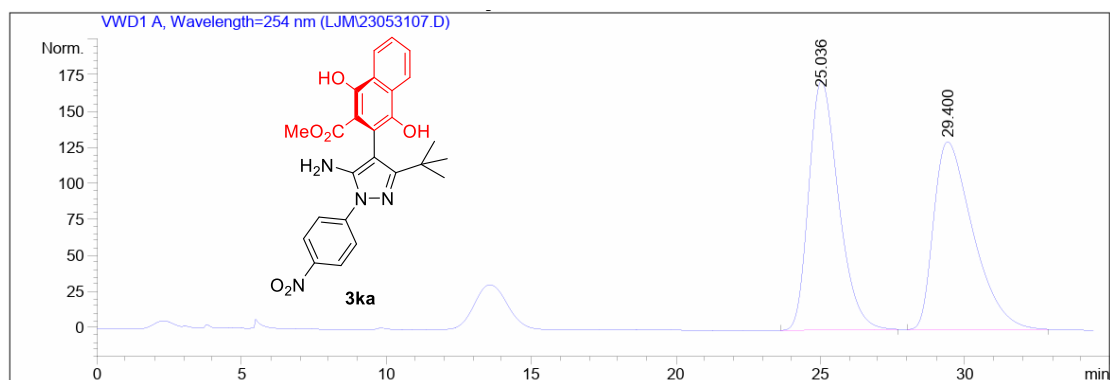


Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	11.860	BB	0.3979	1.22208e4		446.51791	49.9967
2	17.496	BB	0.5767	1.22224e4		304.08835	50.0033

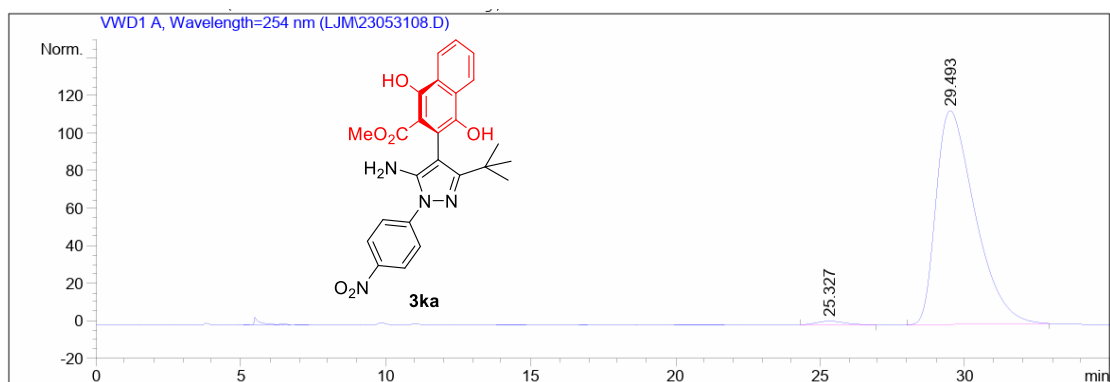


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.557	BB	0.3975	1.37811e4	501.90637	97.3789
2	17.003	BB	0.5495	370.94406	9.86231	2.6211

Compound 3ka

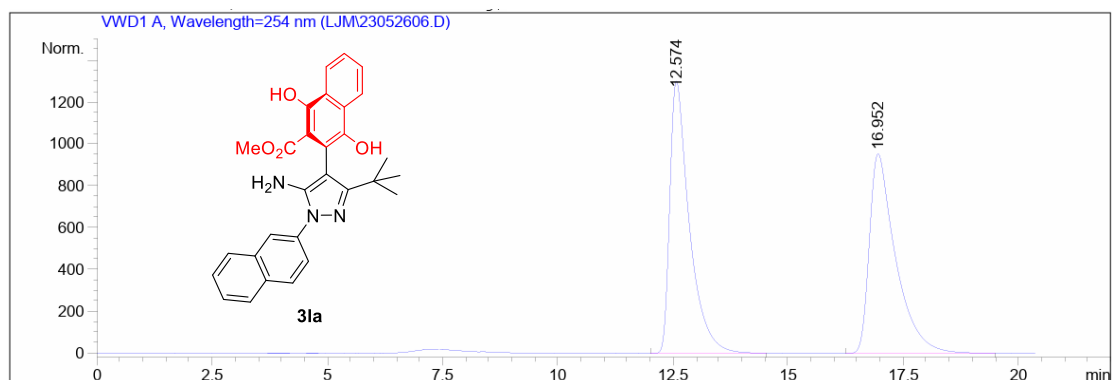


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	25.036	BB	1.1021	1.23472e4	172.68776	50.0983
2	29.400	BB	1.4265	1.22988e4	130.05869	49.9017

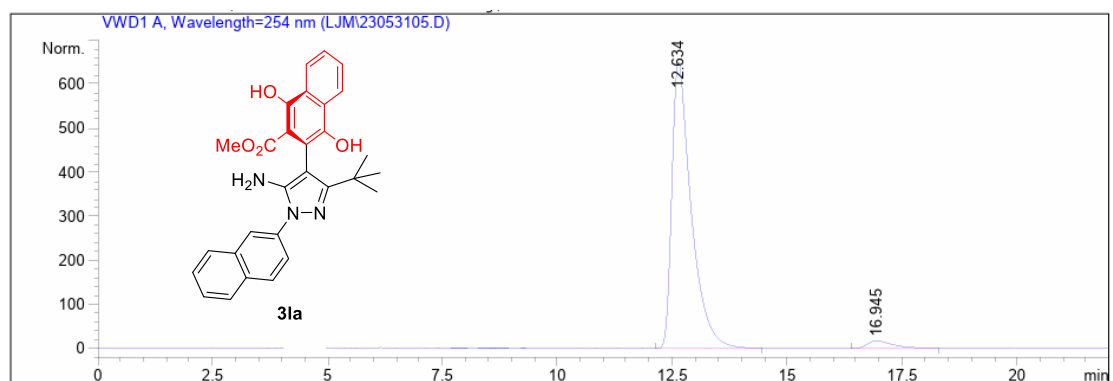


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	25.327	BB	0.8015	130.10416	1.93230	1.2022
2	29.493	BB	1.3892	1.06920e4	114.21236	98.7978

Compound 3la

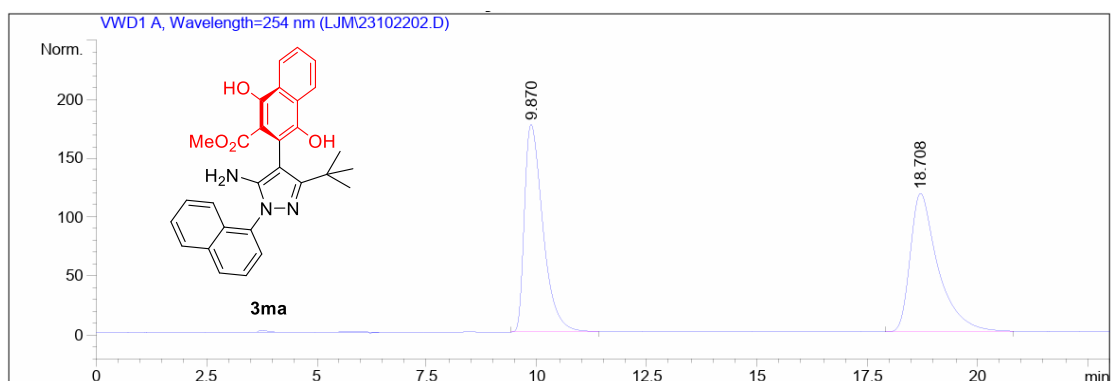


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	12.574	VB	0.4339	3.82935e4		1300.10034	50.3948
2	16.952	PB	0.5665	3.76935e4		952.59644	49.6052

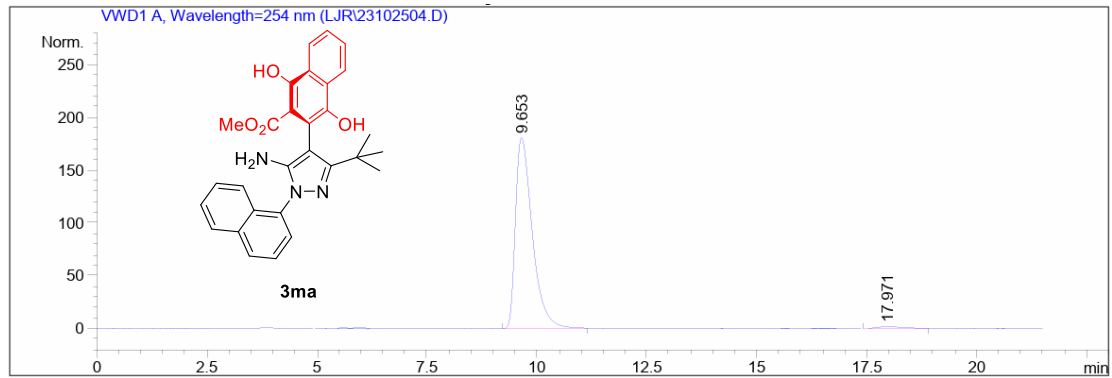


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	12.634	PB	0.4280	1.89490e4		648.98395	96.7296
2	16.945	BB	0.5534	640.66058		16.99238	3.2704

Compound 3ma

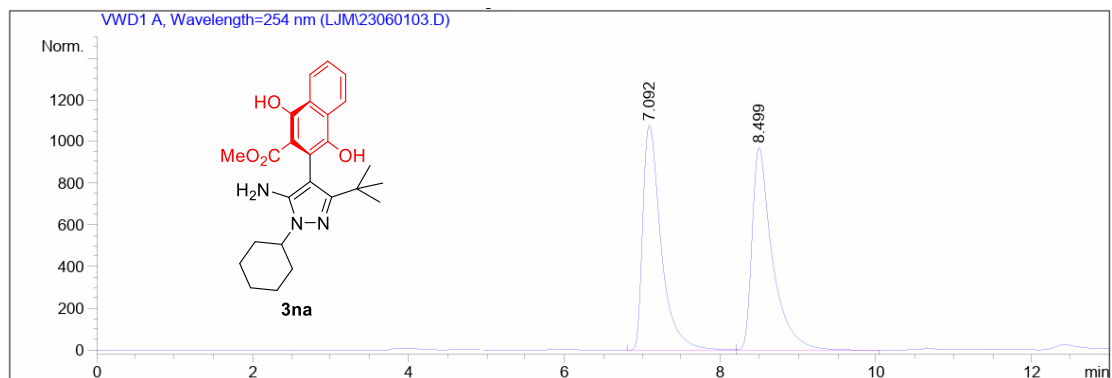


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	9.870	PB	0.4537	5129.75830		175.93369	49.9791
2	18.708	PB	0.6441	5134.05420		117.40109	50.0209

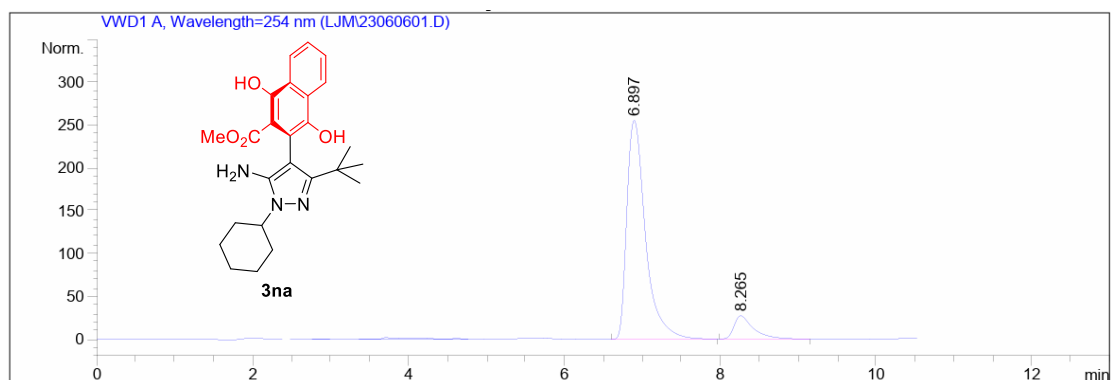


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1	9.653	BB	0.4106	4805.28613		180.75287	98.6464
2	17.971	BB	0.4658	65.93560		1.73846	1.3536

Compound 3na

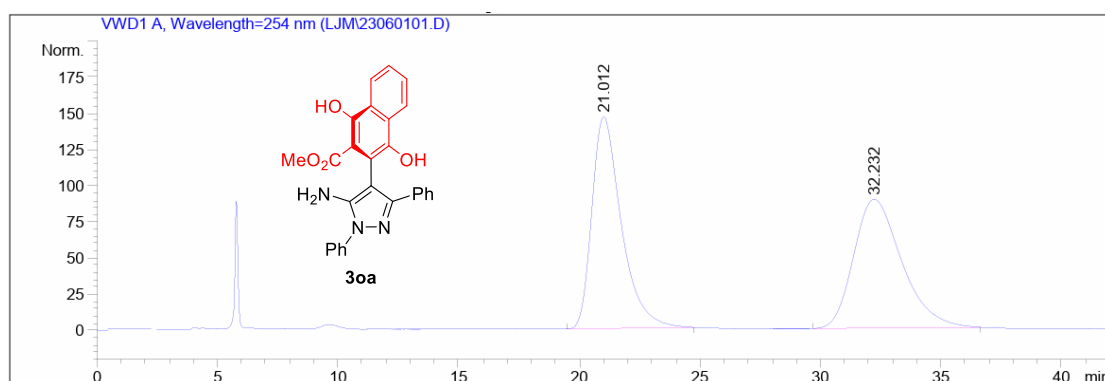


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	7.092	BV	0.2458	1.75855e4		1077.07288	49.9738
2	8.499	VB	0.2626	1.76040e4		970.44611	50.0262

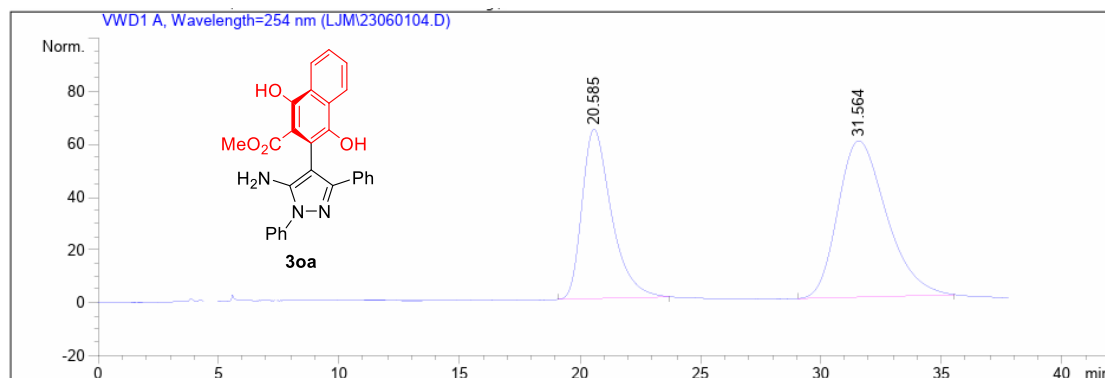


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	6.897	VB	0.2555	4246.07227		254.91765	89.9347
2	8.265	BB	0.2519	475.20877		27.19509	10.0653

Compound 3oa

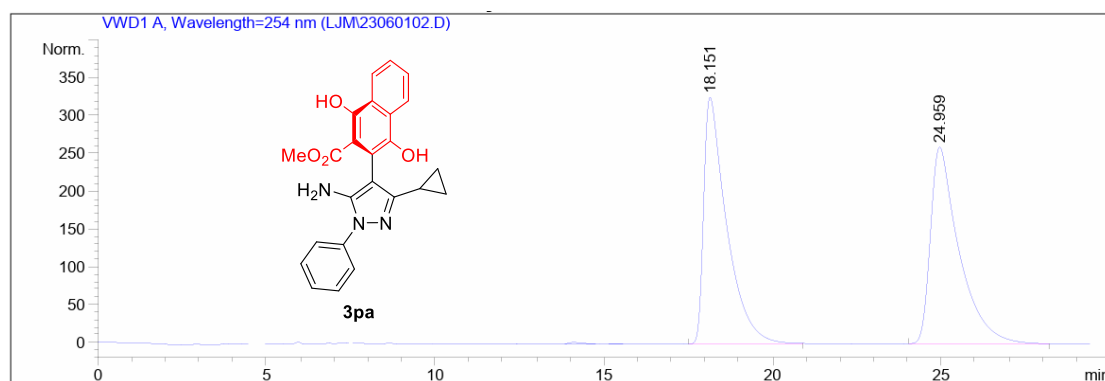


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	21.012	BB	1.2832	1.24291e4	146.39575	50.1288
2	32.232	BB	2.0489	1.23652e4	89.08466	49.8712

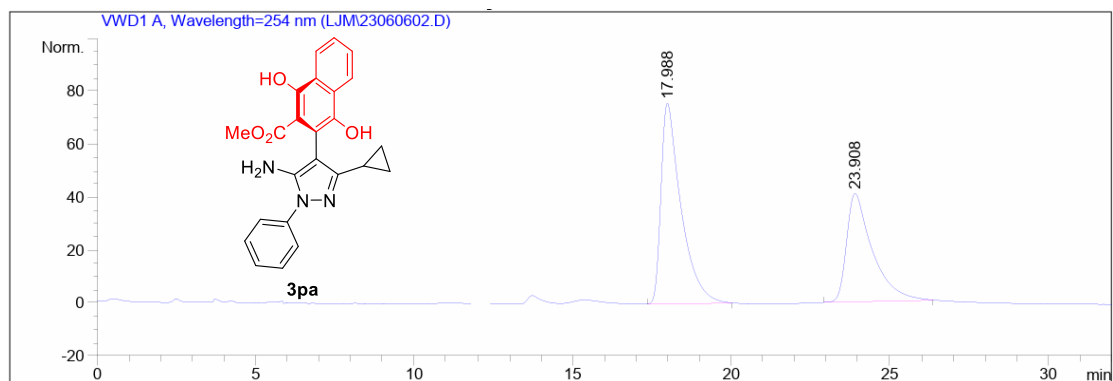


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.585	BB	1.2680	5428.79150	63.98292	39.5949
2	31.564	BB	2.0285	8282.05469	59.08276	60.4051

Compound 3pa

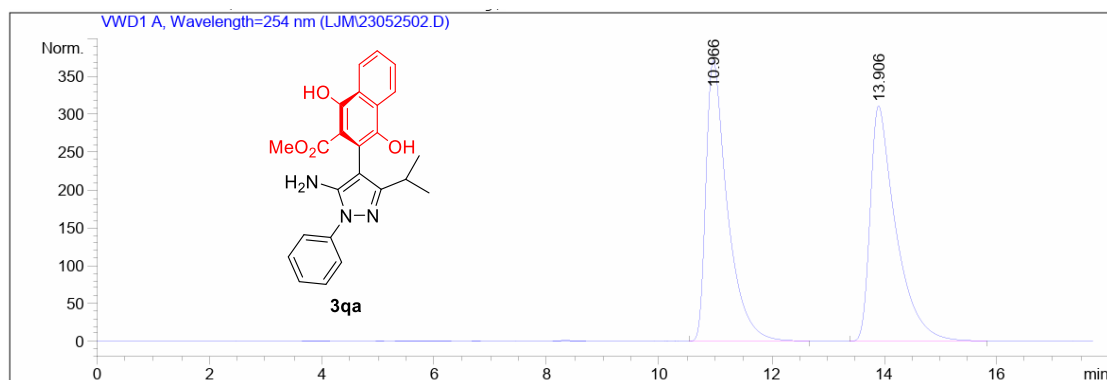


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.151	BB	0.6671	1.53599e4	326.29727	50.0600
2	24.959	BB	0.8386	1.53231e4	260.15469	49.9400

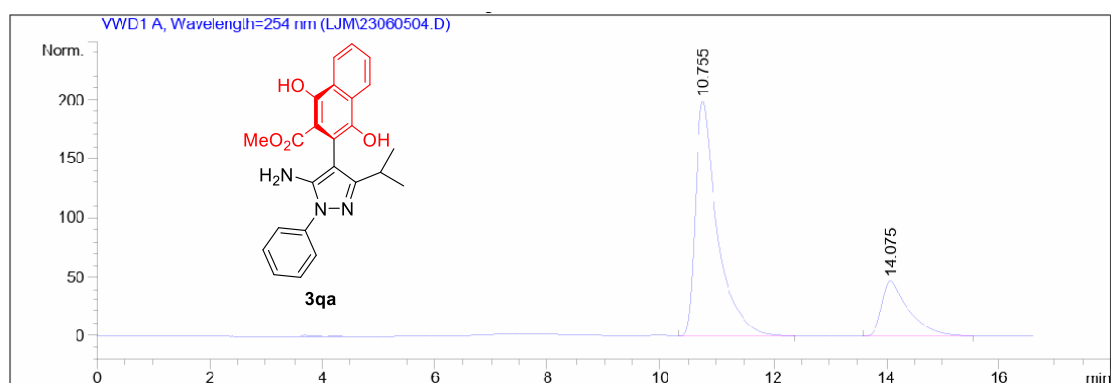


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.988	BB	0.6272	3312.70630	75.67870	58.8438
2	23.908	BB	0.8103	2316.95435	40.84604	41.1562

Compound 3qa

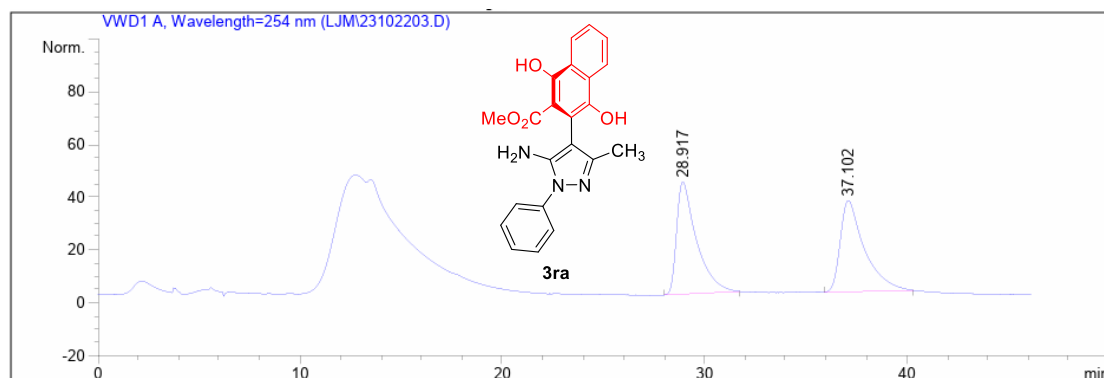


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.966	BB	0.3880	9845.97852	371.28793	49.9730
2	13.906	BB	0.4525	9856.59961	311.18057	50.0270

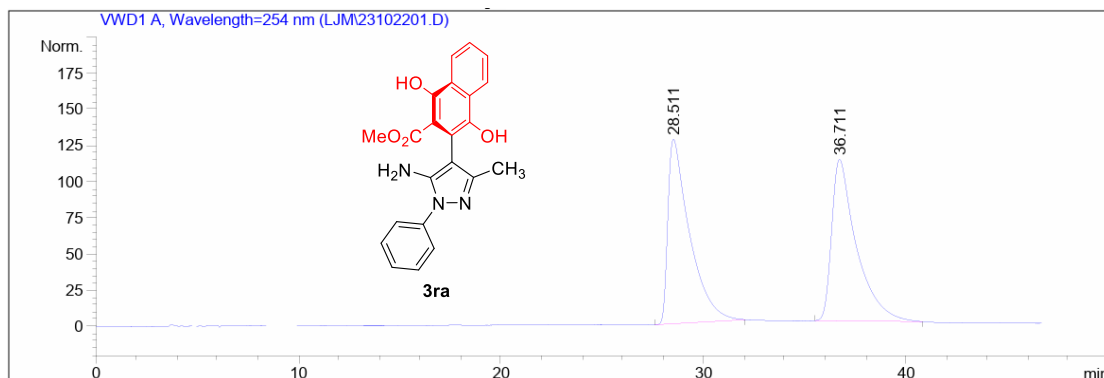


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.755	VB	0.3894	5311.96729	198.49724	77.6779
2	14.075	BB	0.4661	1526.48621	46.48724	22.3221

Compound 3ra

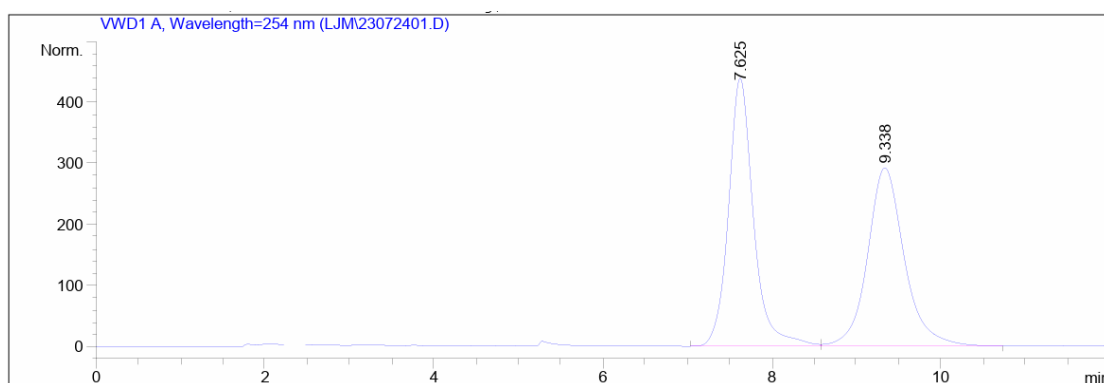


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	28.917	BB	0.9530	2915.38232	42.50959	50.2288
2	37.102	BB	1.1706	2888.82153	34.60181	49.7712

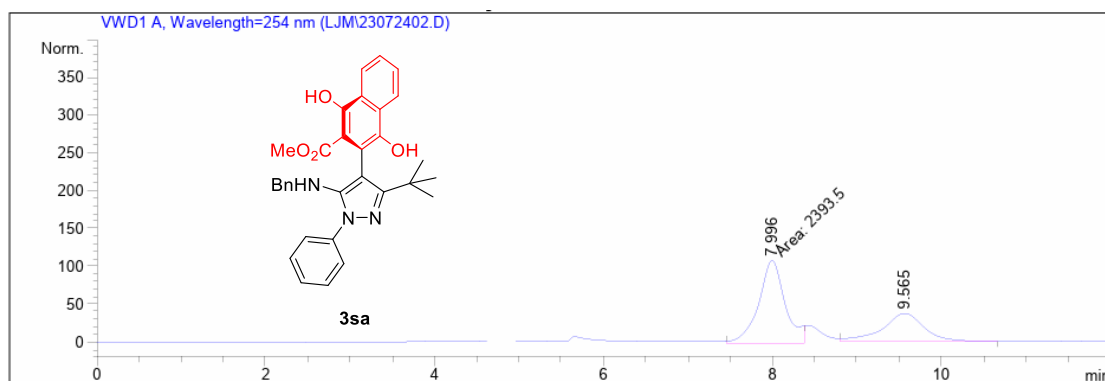


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	28.511	PB	1.0547	9494.67285	127.57211	50.0572
2	36.711	BB	1.1873	9472.96387	111.54948	49.9428

Compound 3sa

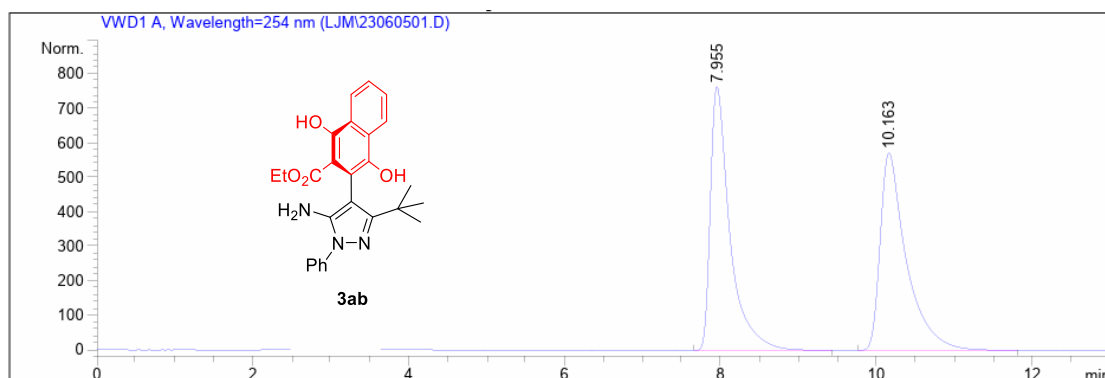


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.625	PV	0.3075	9026.89160	437.84744	50.6440
2	9.338	VB	0.4543	8797.30664	291.30371	49.3560

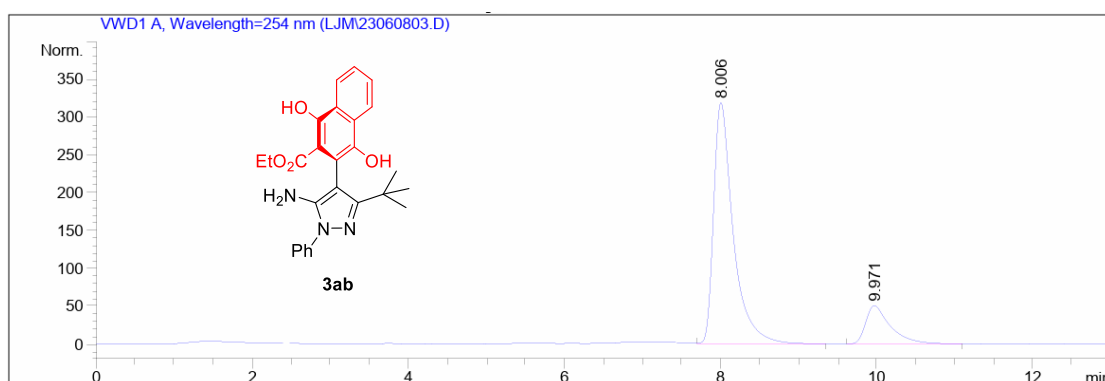


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	7.996	MM	0.3642	2393.50049		109.53661	64.7209
2	9.565	VB	0.5071	1304.68726		37.43550	35.2791

Compound 3ab

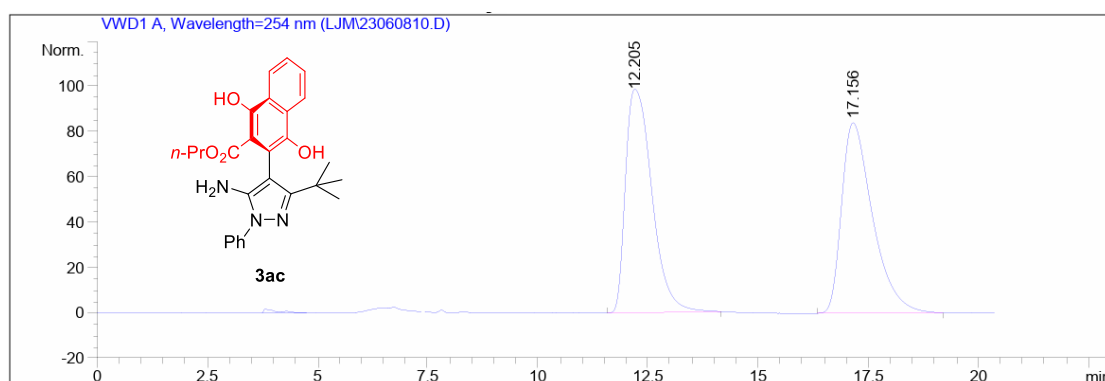


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	7.955	BB	0.2580	1.32643e4		763.48193	49.9153
2	10.163	PB	0.3355	1.33093e4		572.03174	50.0847

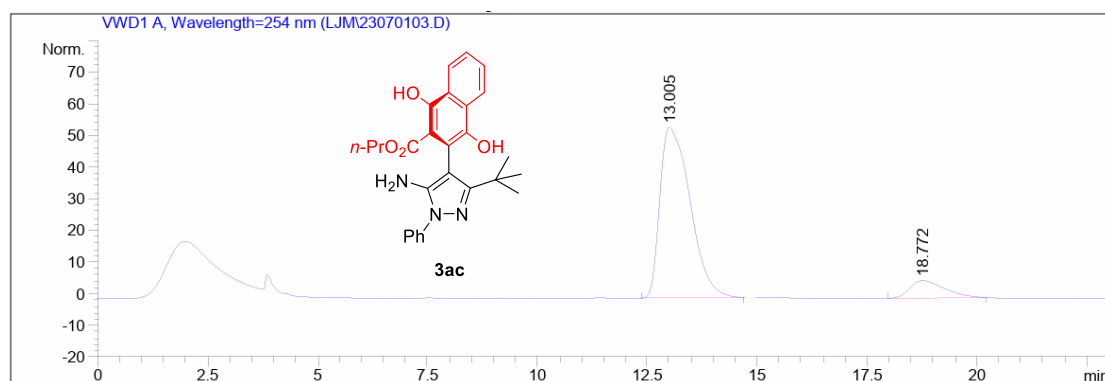


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	8.006	VB	0.2602	5548.29639		318.20441	83.2331
2	9.971	PB	0.3213	1117.68066		50.40136	16.7669

Compound 3ac

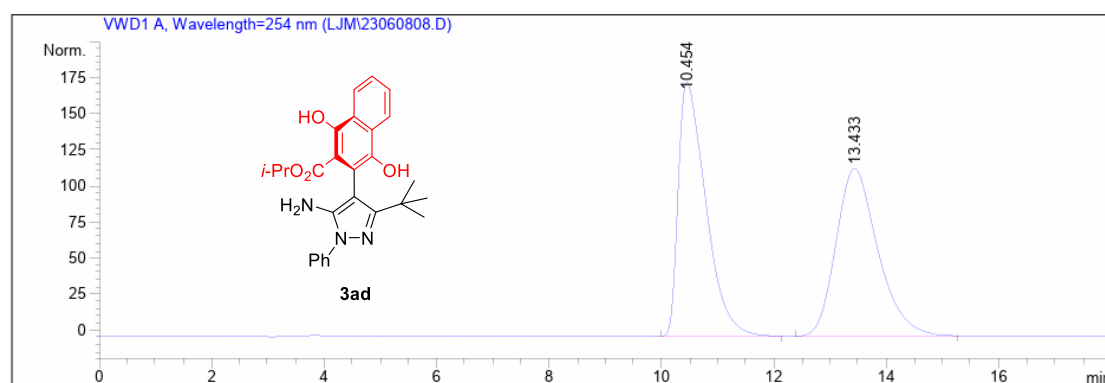


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.205	PB	0.6615	4122.77441	98.51266	50.0468
2	17.156	BB	0.7463	4115.06152	83.99258	49.9532

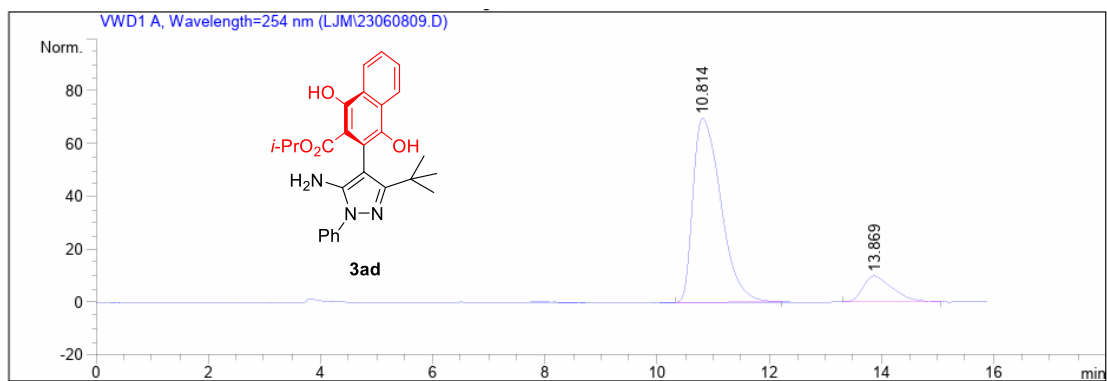


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.005	BB	0.6634	2569.74731	53.81838	89.4147
2	18.772	BB	0.7462	304.21674	5.53345	10.5853

Compound 3ad

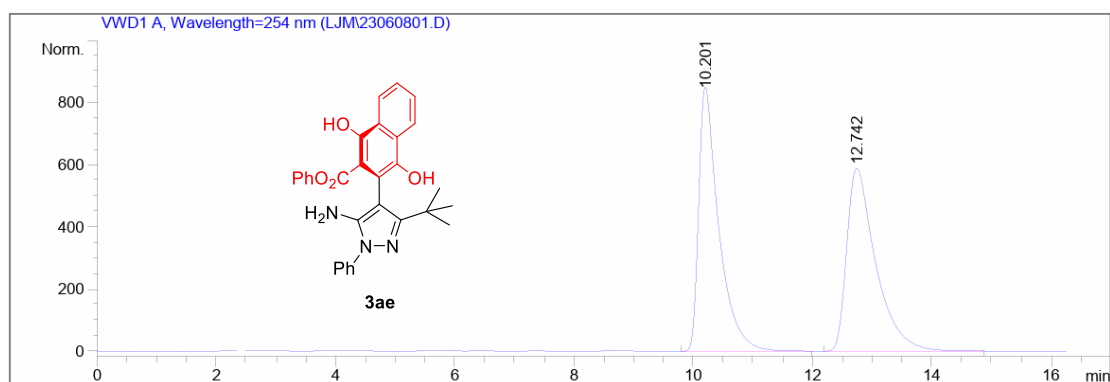


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.454	BB	0.5450	5970.06982	175.66077	49.9479
2	13.433	PB	0.7840	5982.51758	115.93729	50.0521

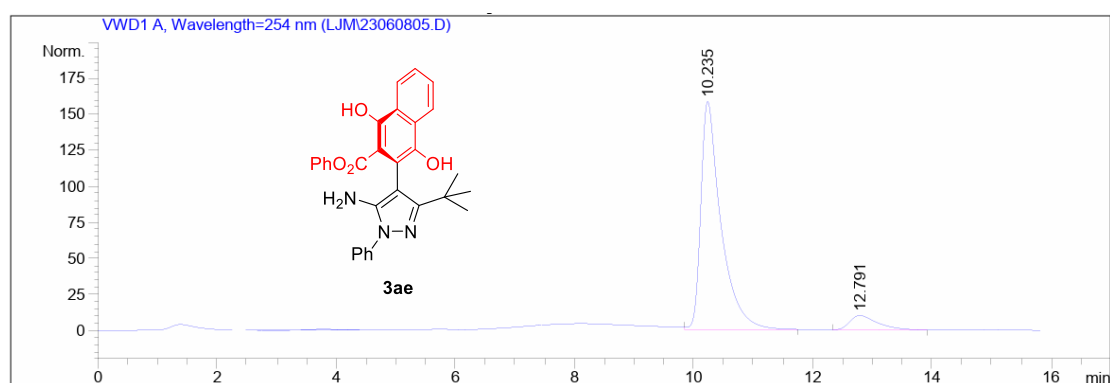


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.814	BB	0.5506	2402.86743	69.71330	86.9466
2	13.869	BB	0.5306	360.74530	9.88536	13.0534

Compound 3ae

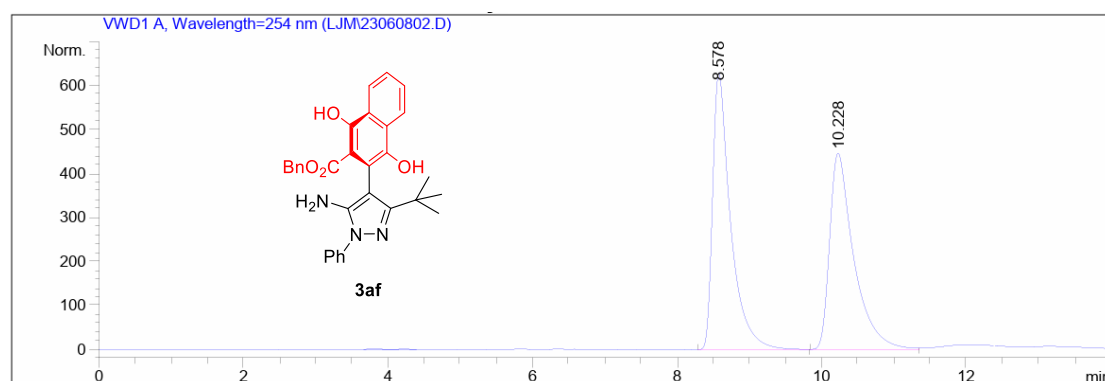


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.201	BB	0.3357	1.97630e4	848.84637	49.9922
2	12.742	PB	0.4959	1.97692e4	587.57397	50.0078

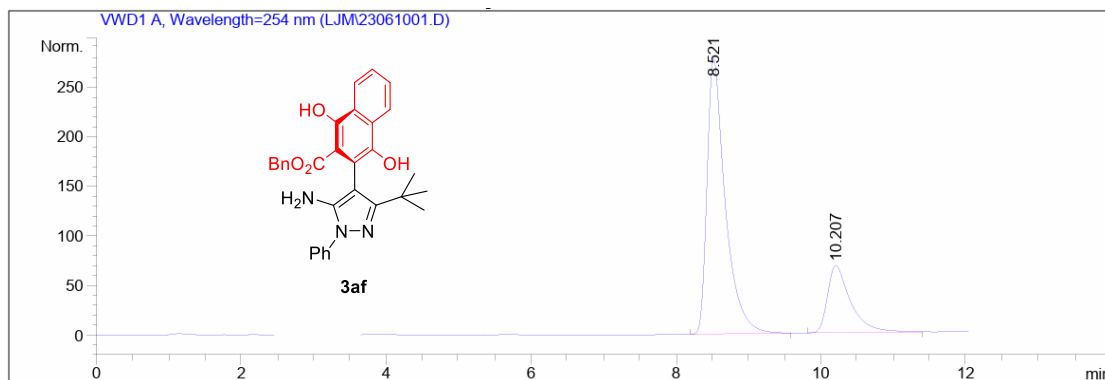


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.235	VB	0.3358	3705.90820	158.25160	91.9957
2	12.791	PB	0.4780	322.44257	9.92463	8.0043

Compound 3af

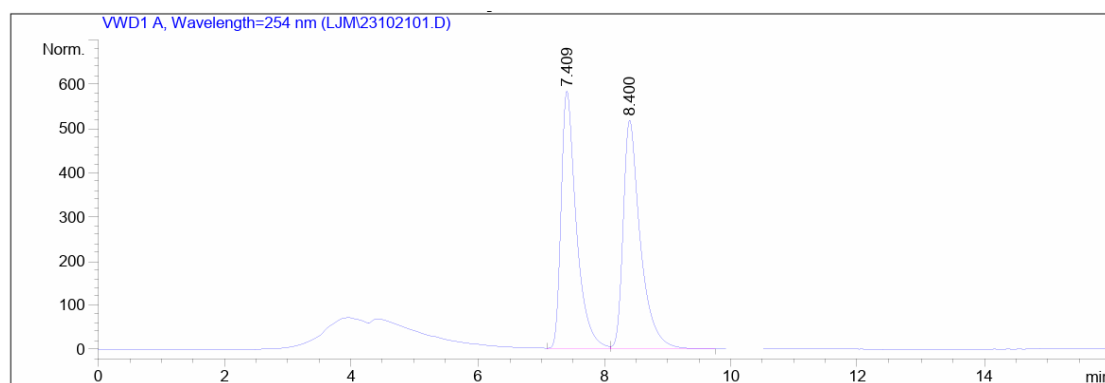


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	8.578	VB	0.2519	1.07886e4		626.43762	51.0825
2	10.228	BV	0.3377	1.03313e4		445.28378	48.9175

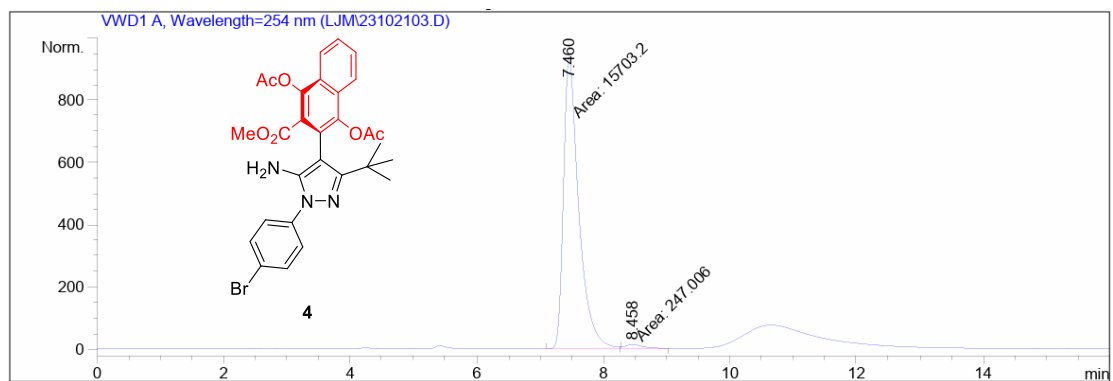


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	8.521	VB	0.2574	4829.89893		276.92062	76.5379
2	10.207	PB	0.3216	1480.56946		67.45082	23.4621

Compound 4

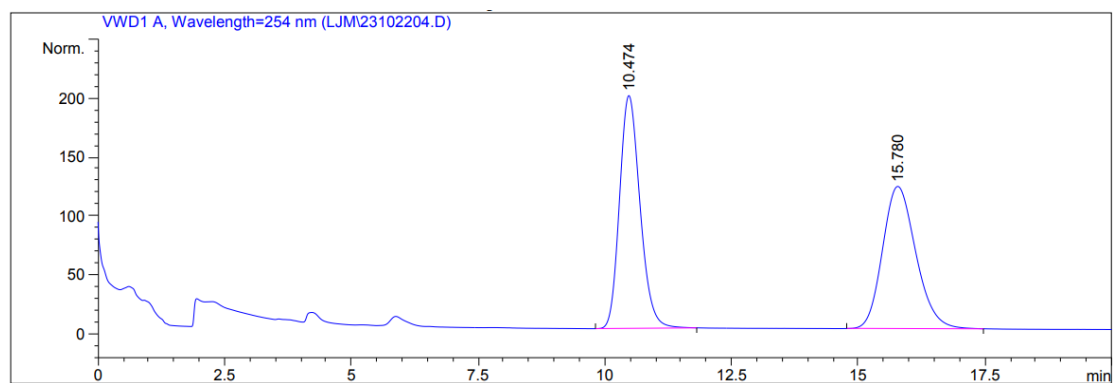


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	7.409	VV	0.2465	9783.51563		583.60919	49.9251
2	8.400	VB	0.2780	9812.86133		517.62292	50.0749

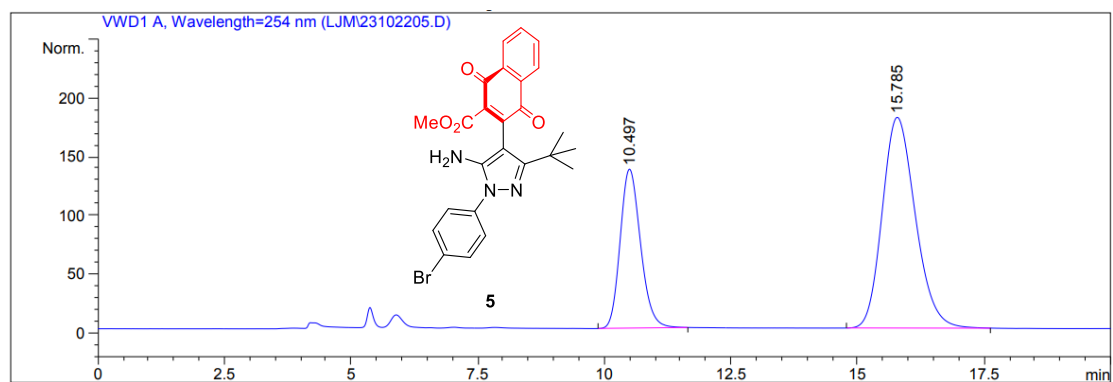


Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	7.460	MM	0.2791	1.57032e4		937.85052	98.4514
2	8.458	MM	0.3153	247.00552		13.05557	1.5486

Compound 5



Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	10.474	PB	0.4347	5561.59863		197.58485	50.1911
2	15.780	BB	0.7124	5519.24268		120.74728	49.8089

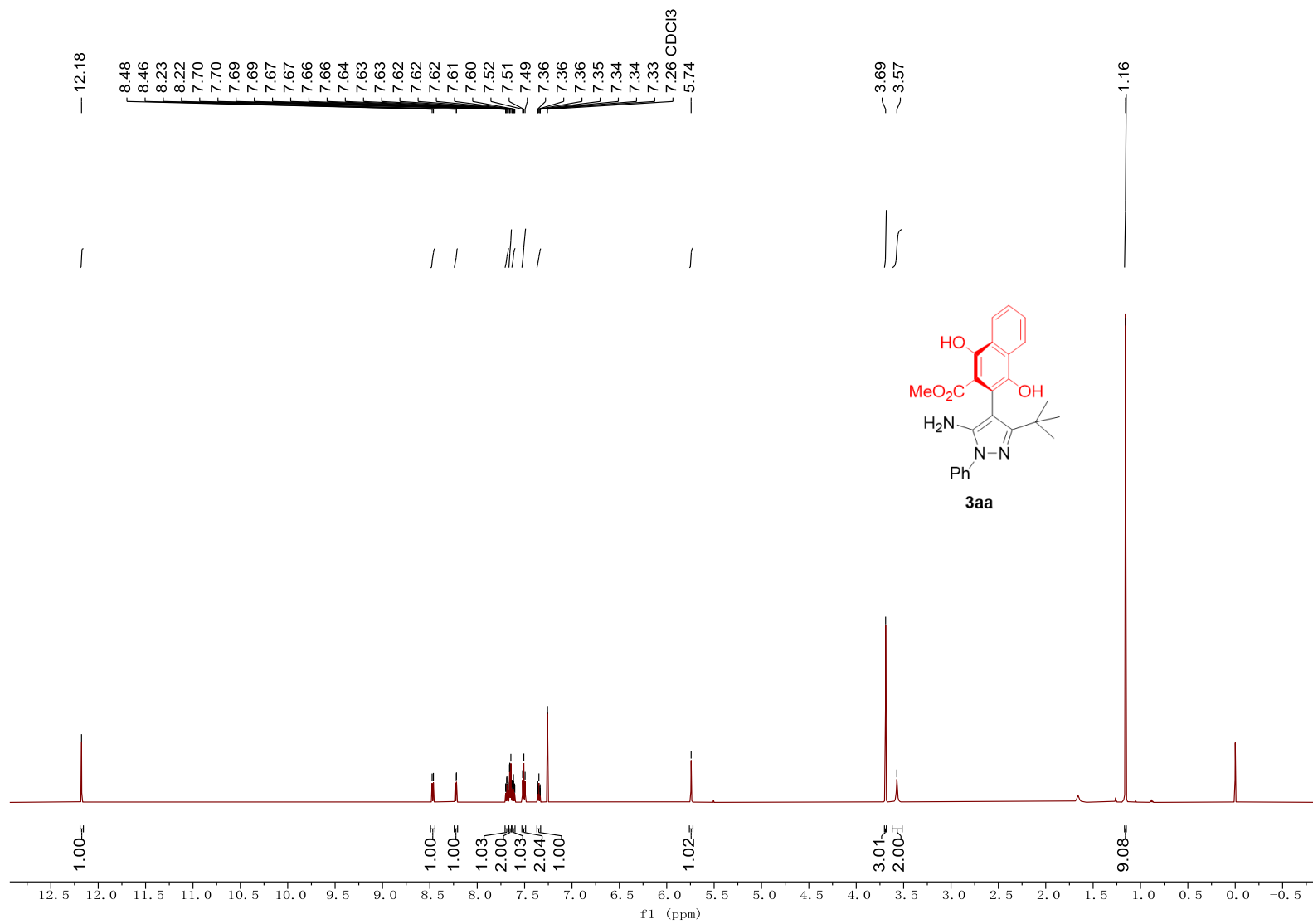


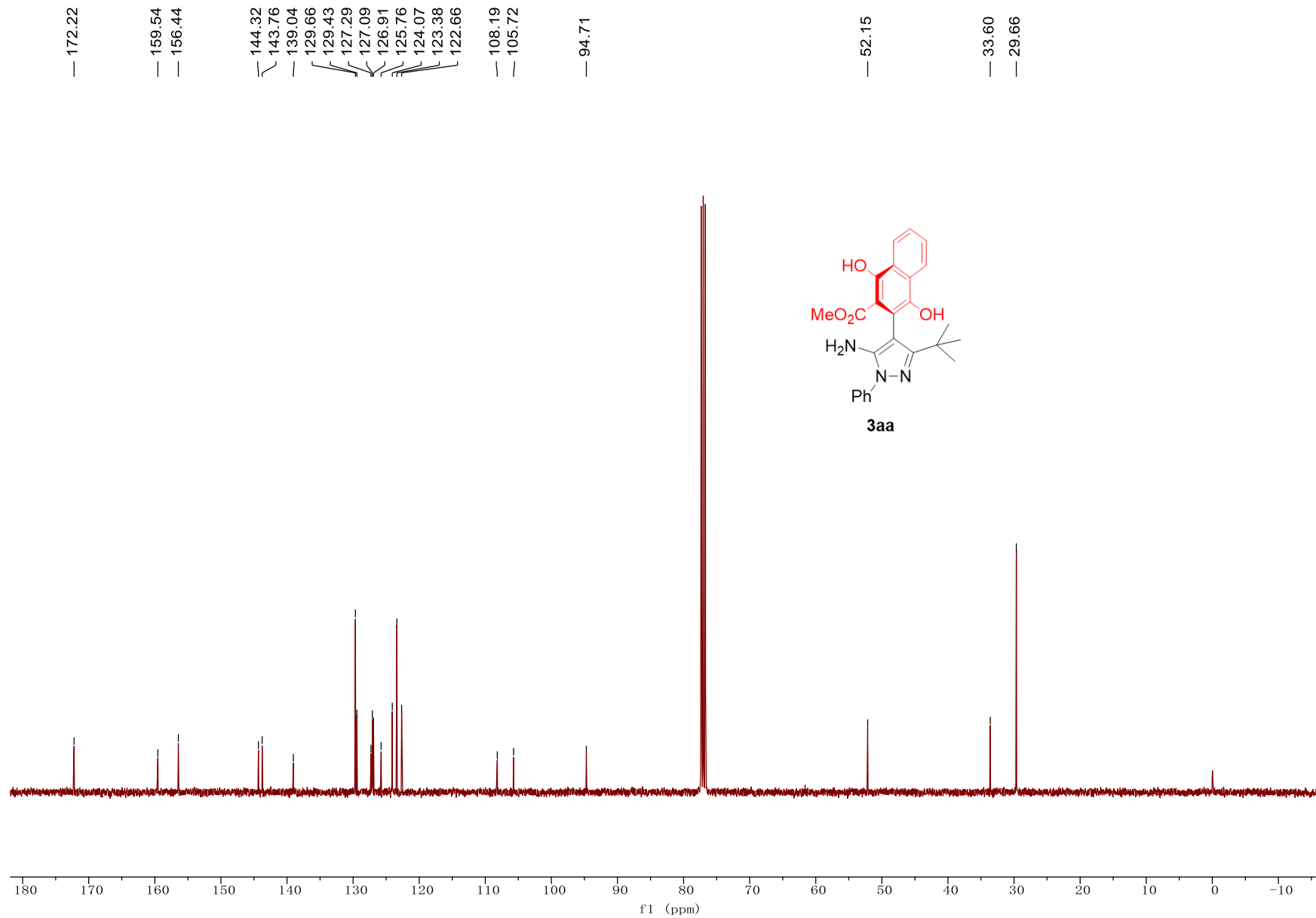
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1	10.497	PB	0.4345	3789.64478		135.32426	31.7146
2	15.785	BB	0.7025	8159.56592		179.36205	68.2854

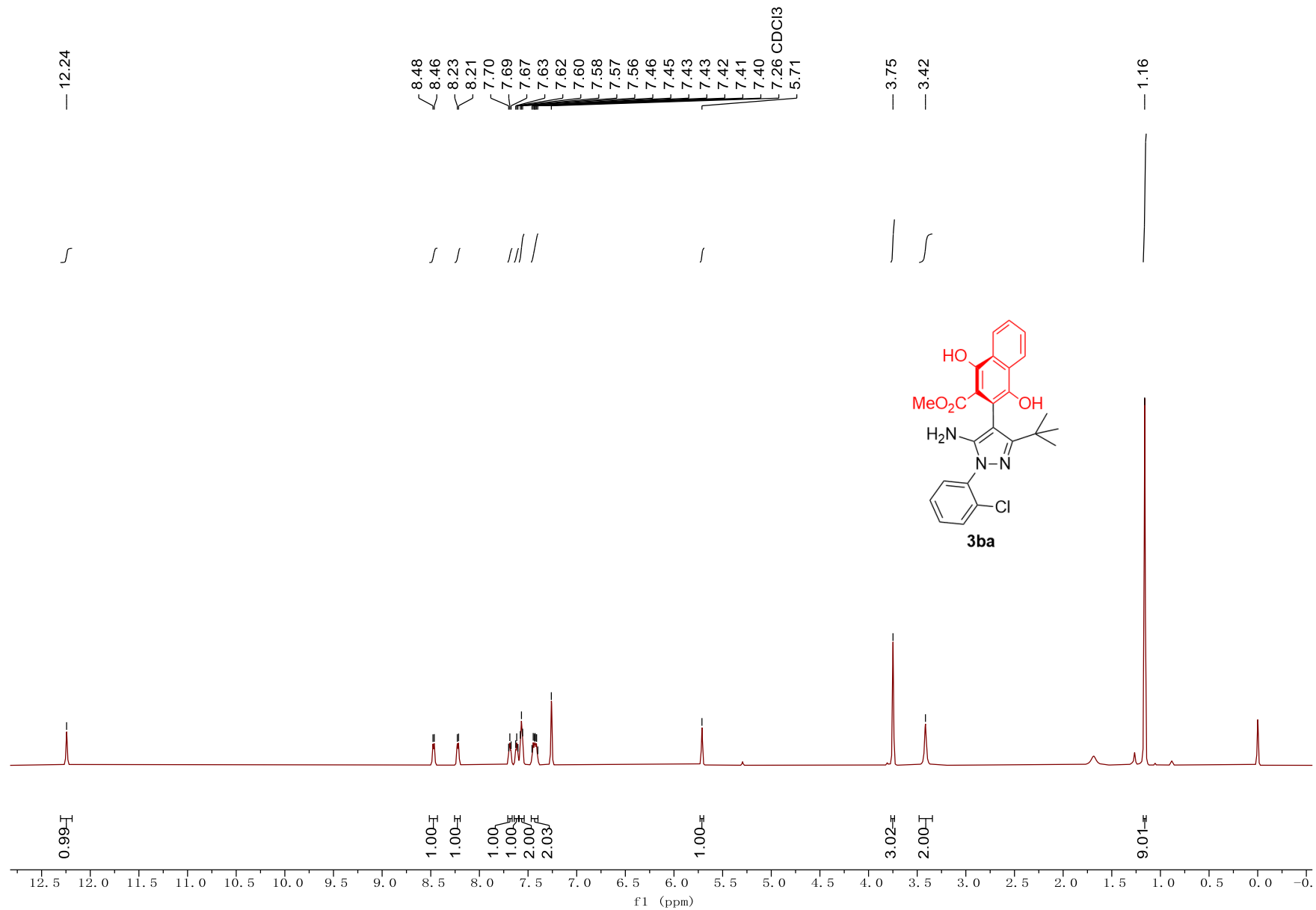
6. References

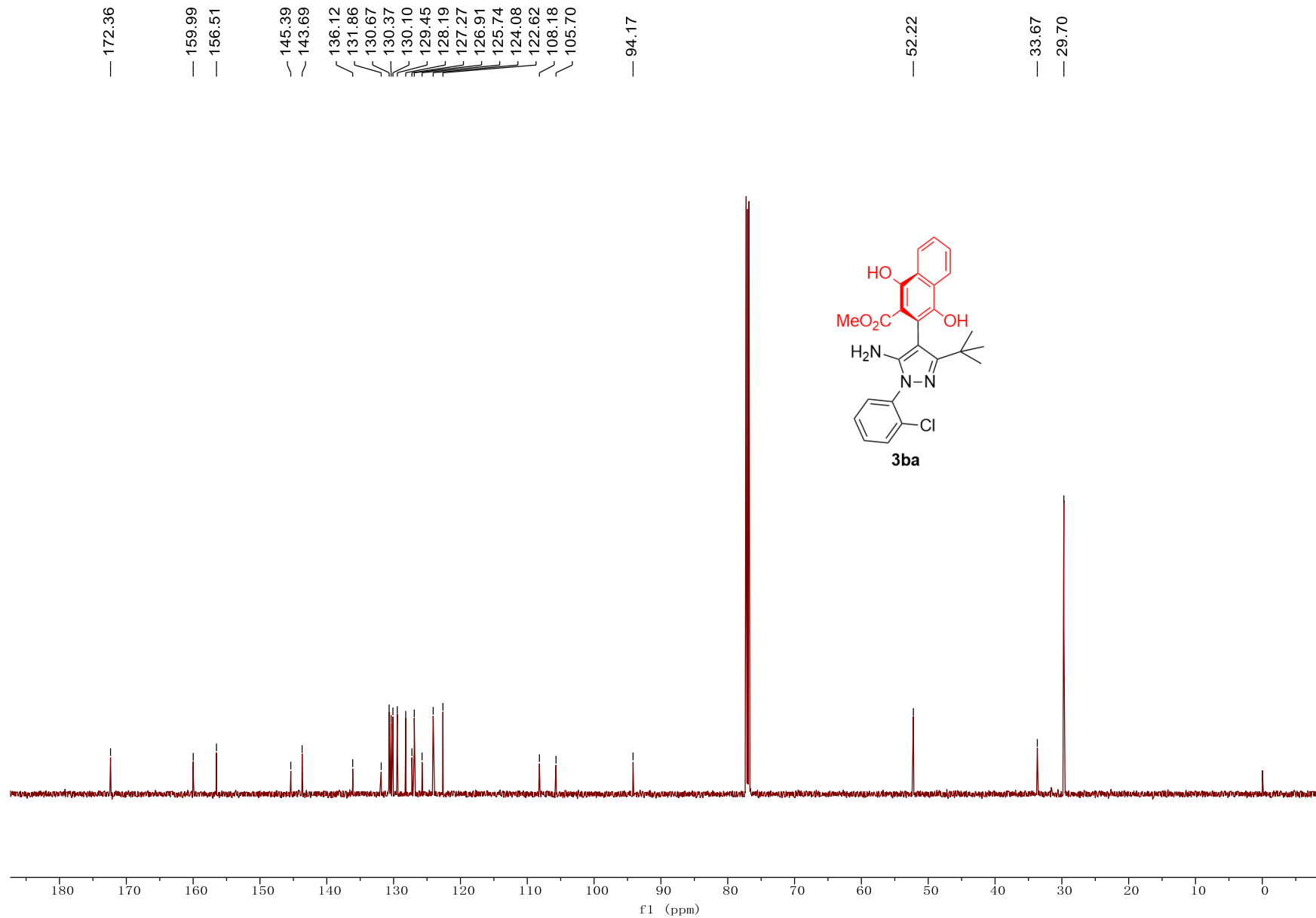
1. M. Bartoszek, M. Beller, J. Deutsch, M. Klawonn, A. Köckritz, N. Nemati and A. Pews-Davtyan, *Tetrahedron*, 2008, **64**, 1316-1322.
2. D. S. Huang, E. V. LeBlanc, T. Shekhar-Guturja, N. Robbins, D. J. Krysan, J. Pizarro, L. Whitesell, L. E. Cowen and L. E. Brown, *J. Med. Chem.*, 2019, **63**, 2139-2180.
3. R. Abonia, J. Castillo, B. Insuasty, J. Quiroga, M. Nogueras and J. Cobo, *Eur. J. Org. Chem.*, 2010, 6454-6463.
4. M. A. Brimble, C. Burgess, R. Halim, M. Petersson and J. Ray, *Tetrahedron*, 2004, **60**, 5751-5758.

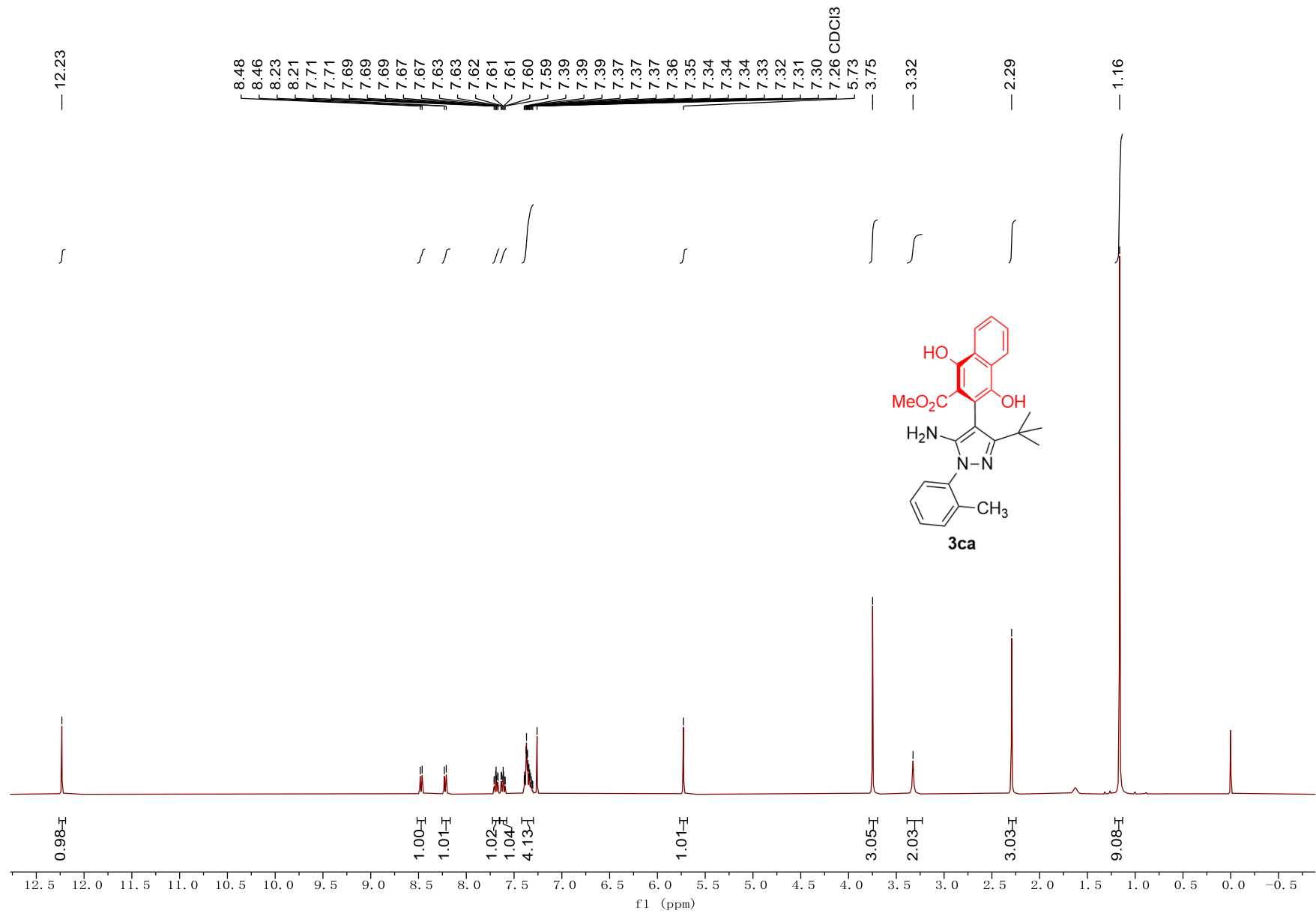
7. NMR spectra for compounds

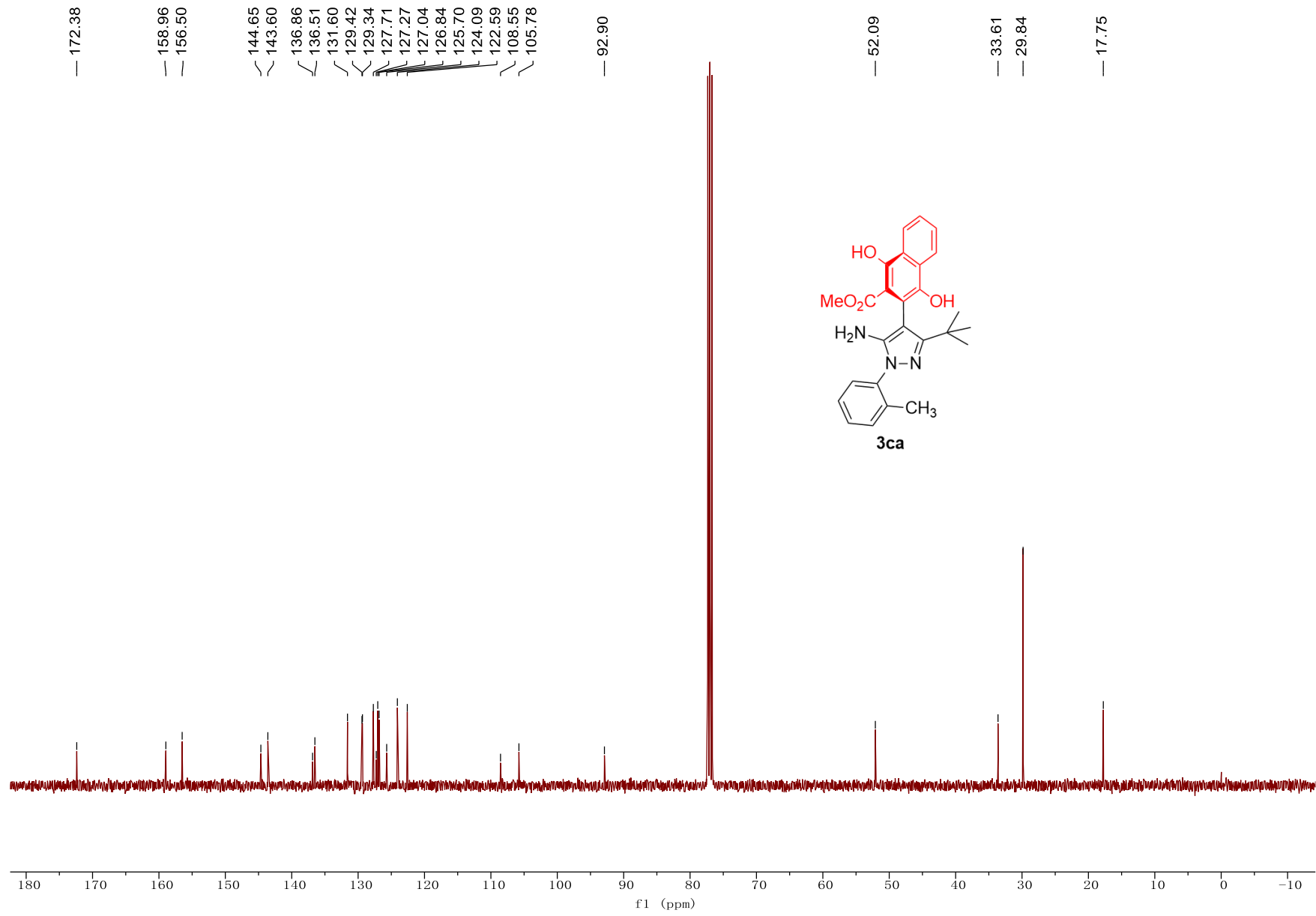


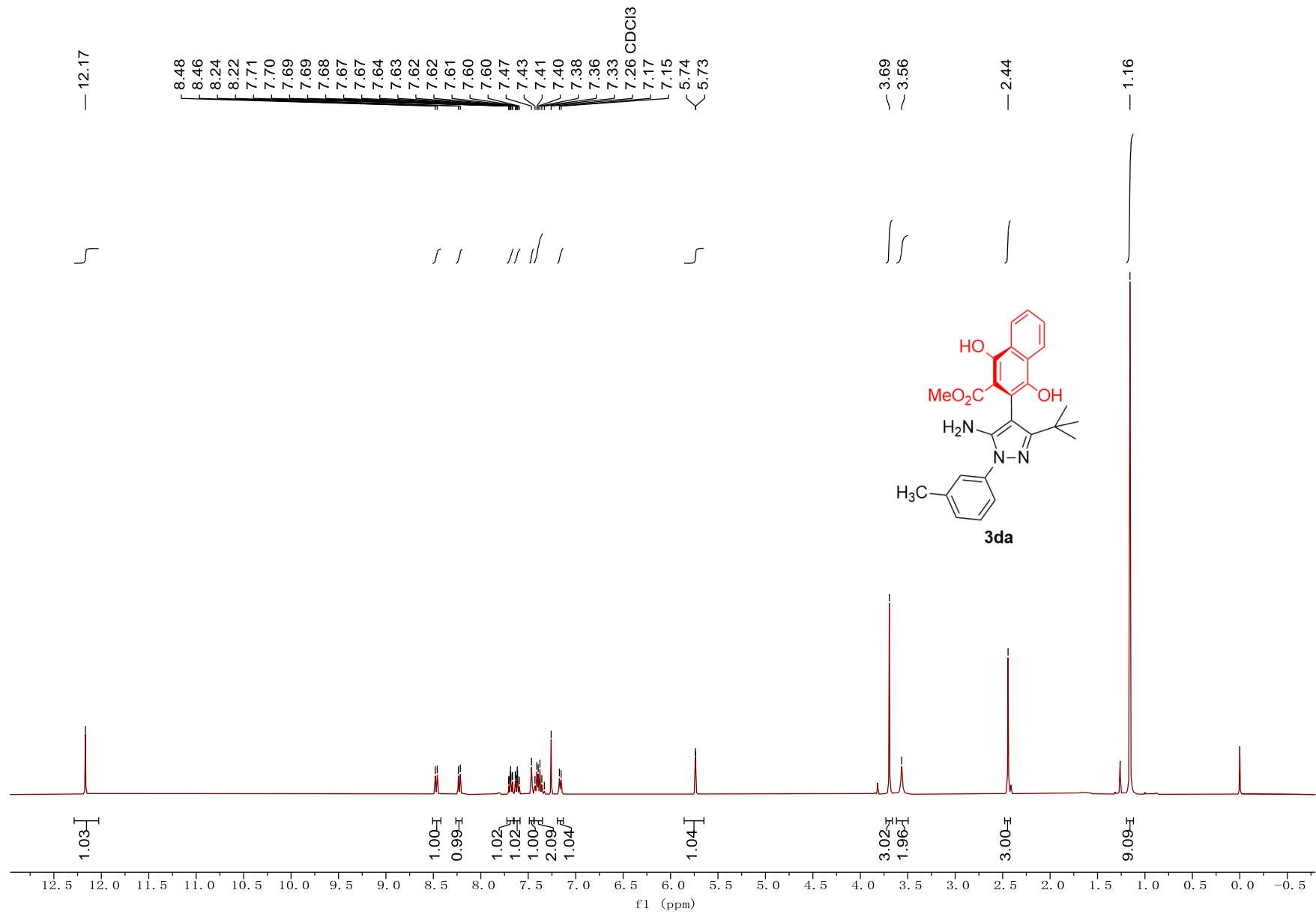


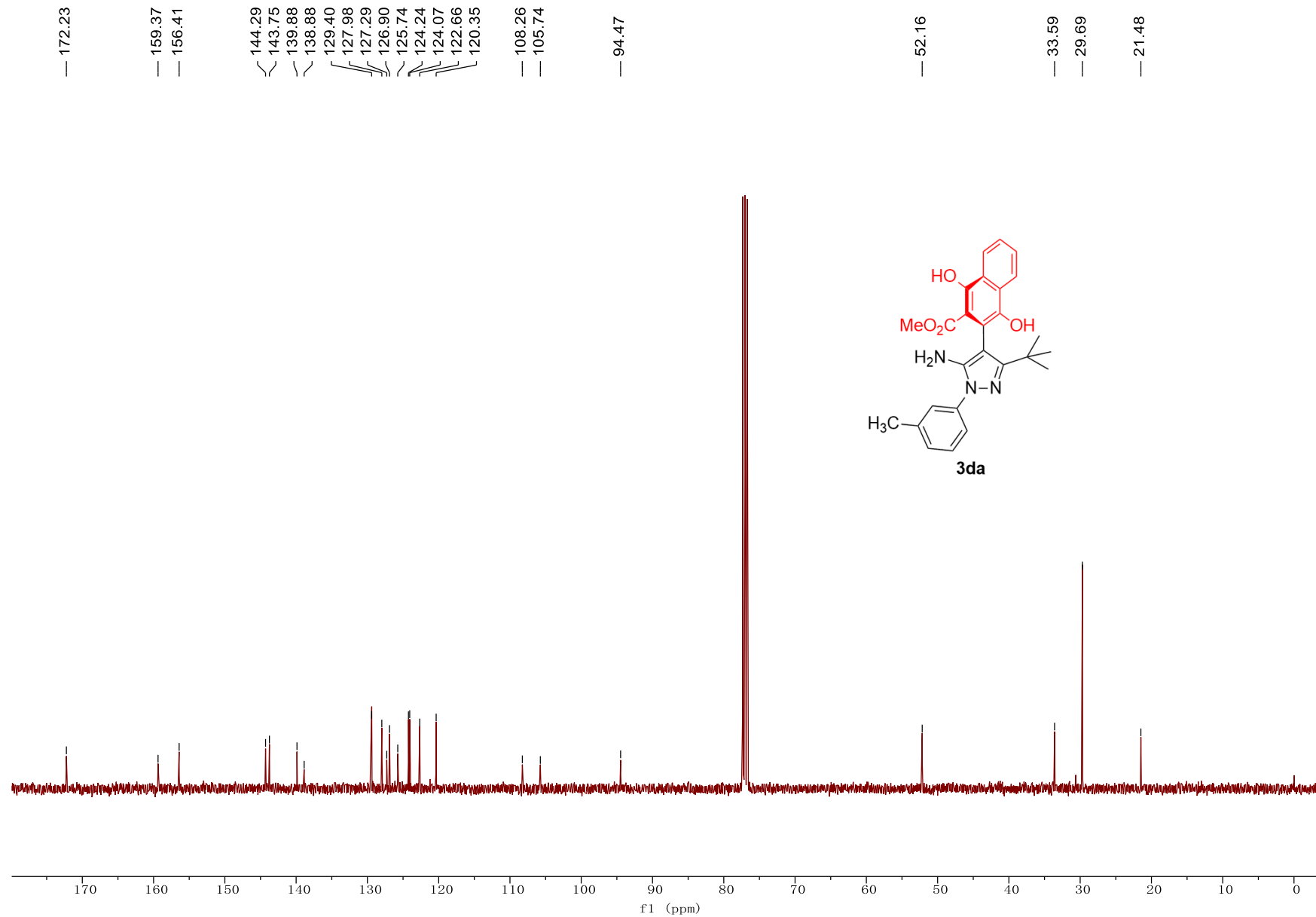


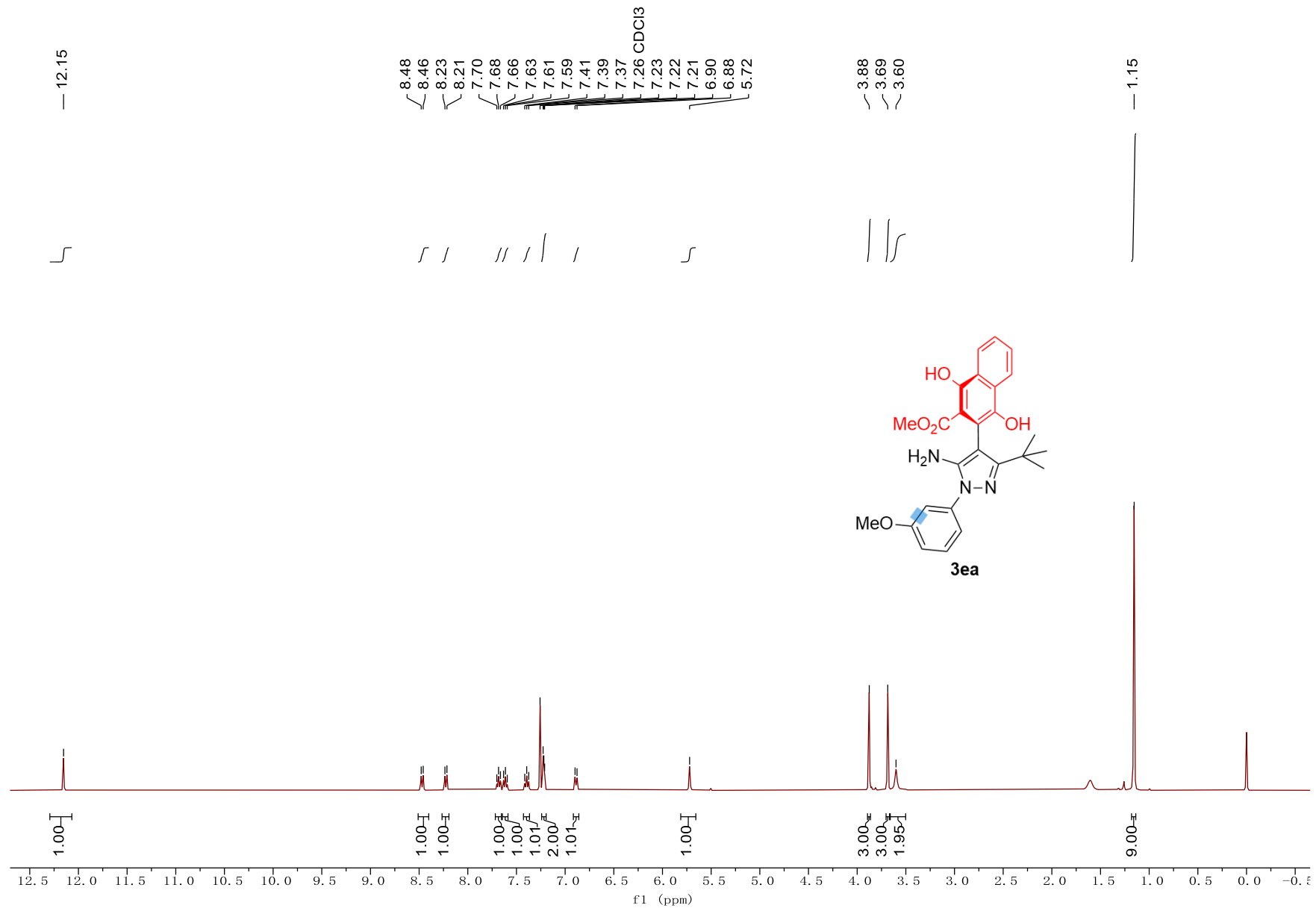


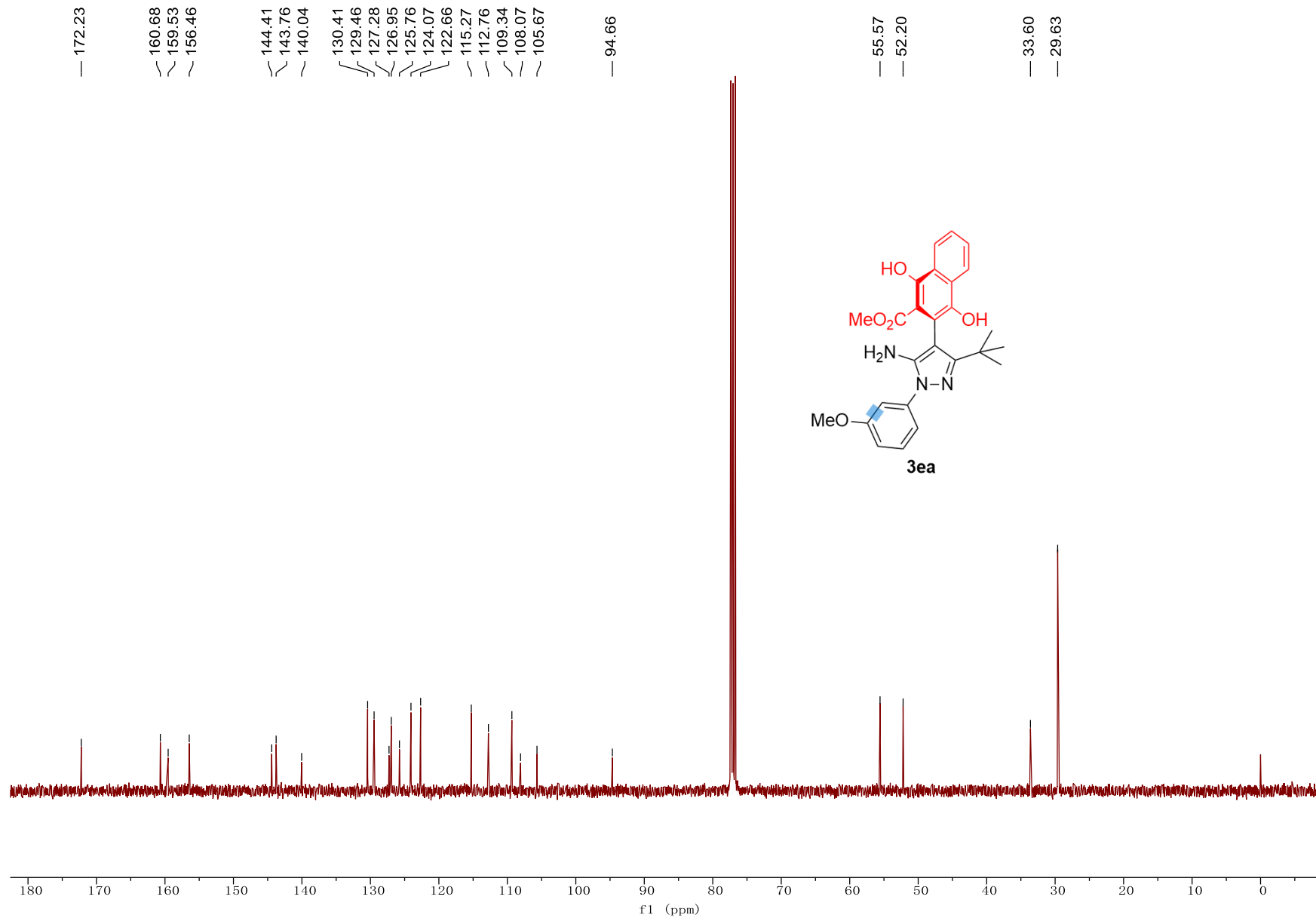


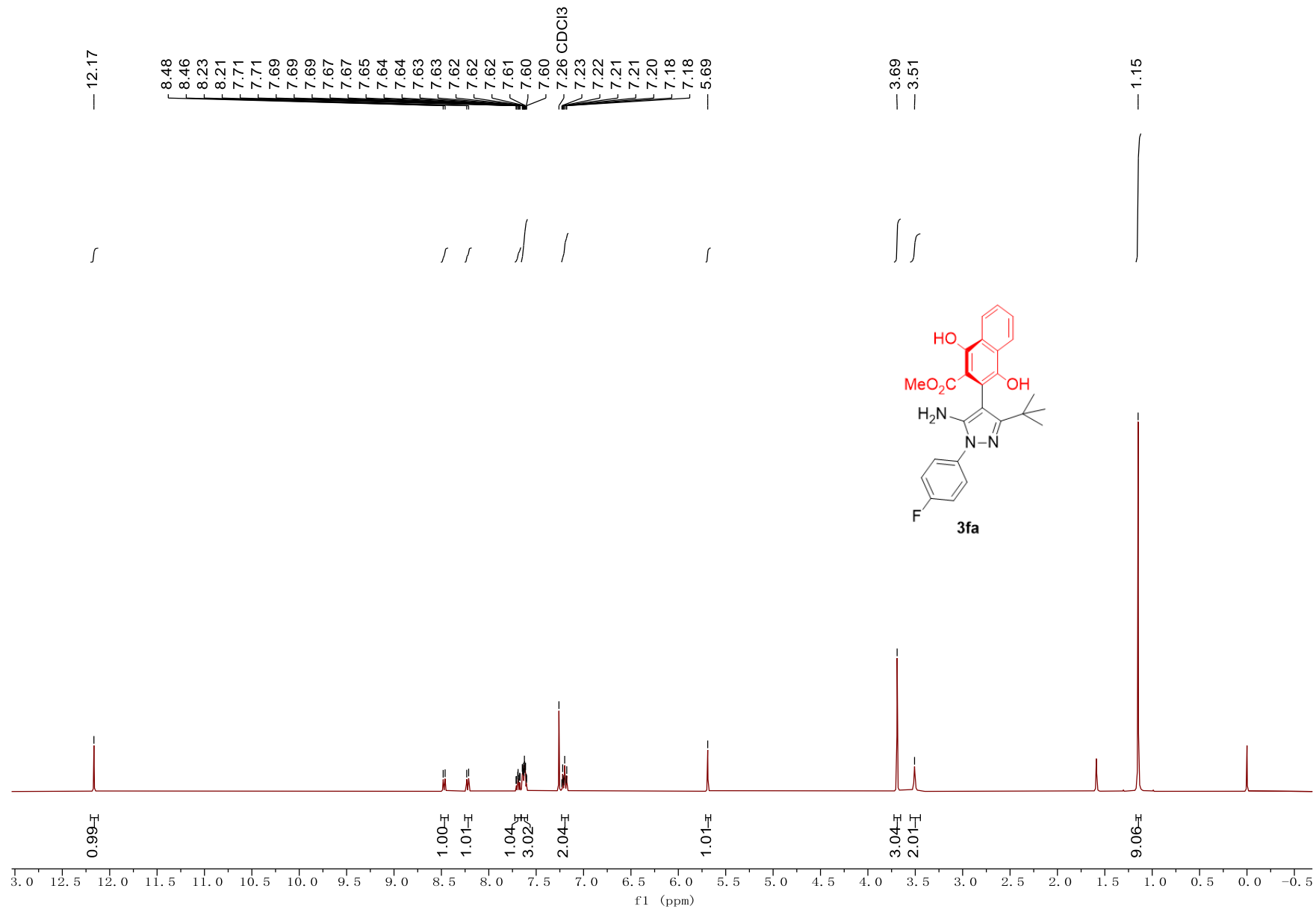


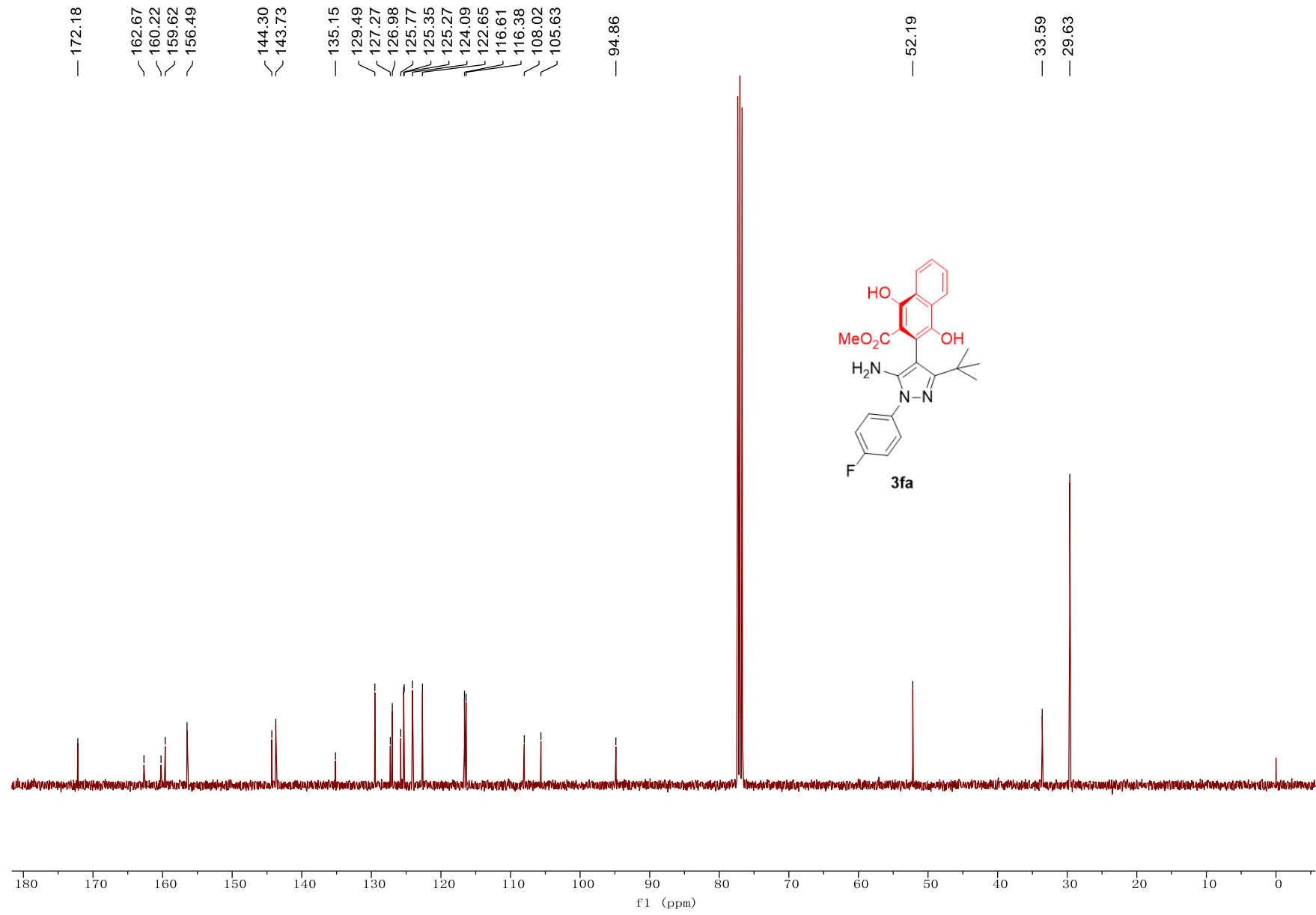




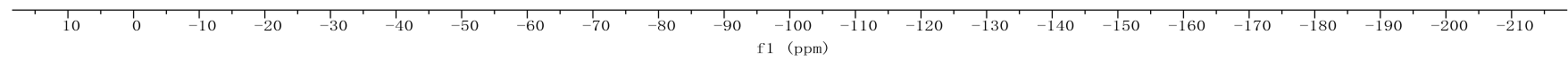
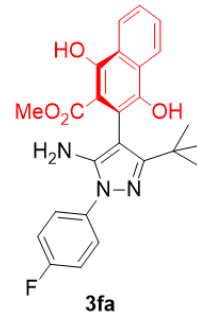


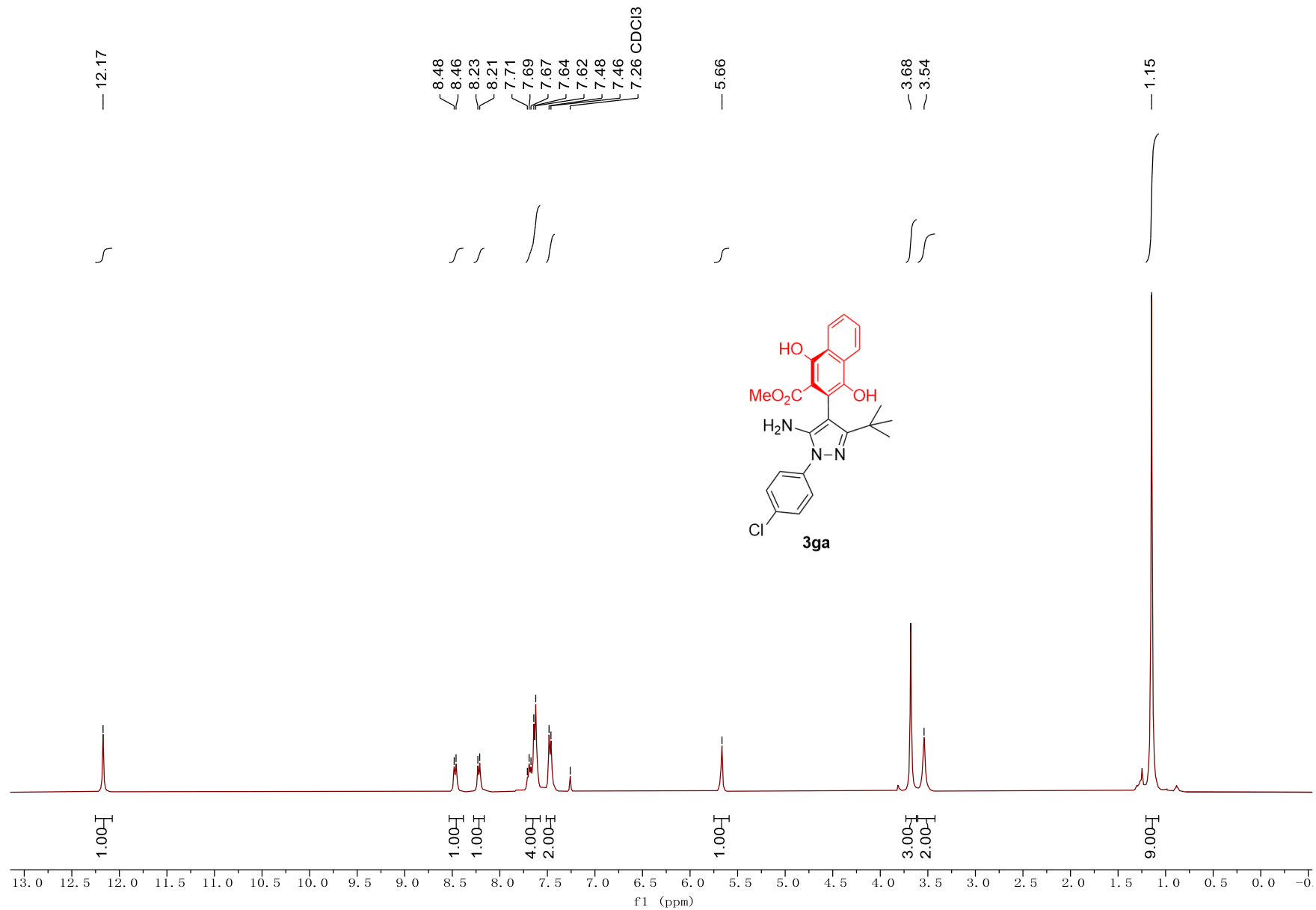


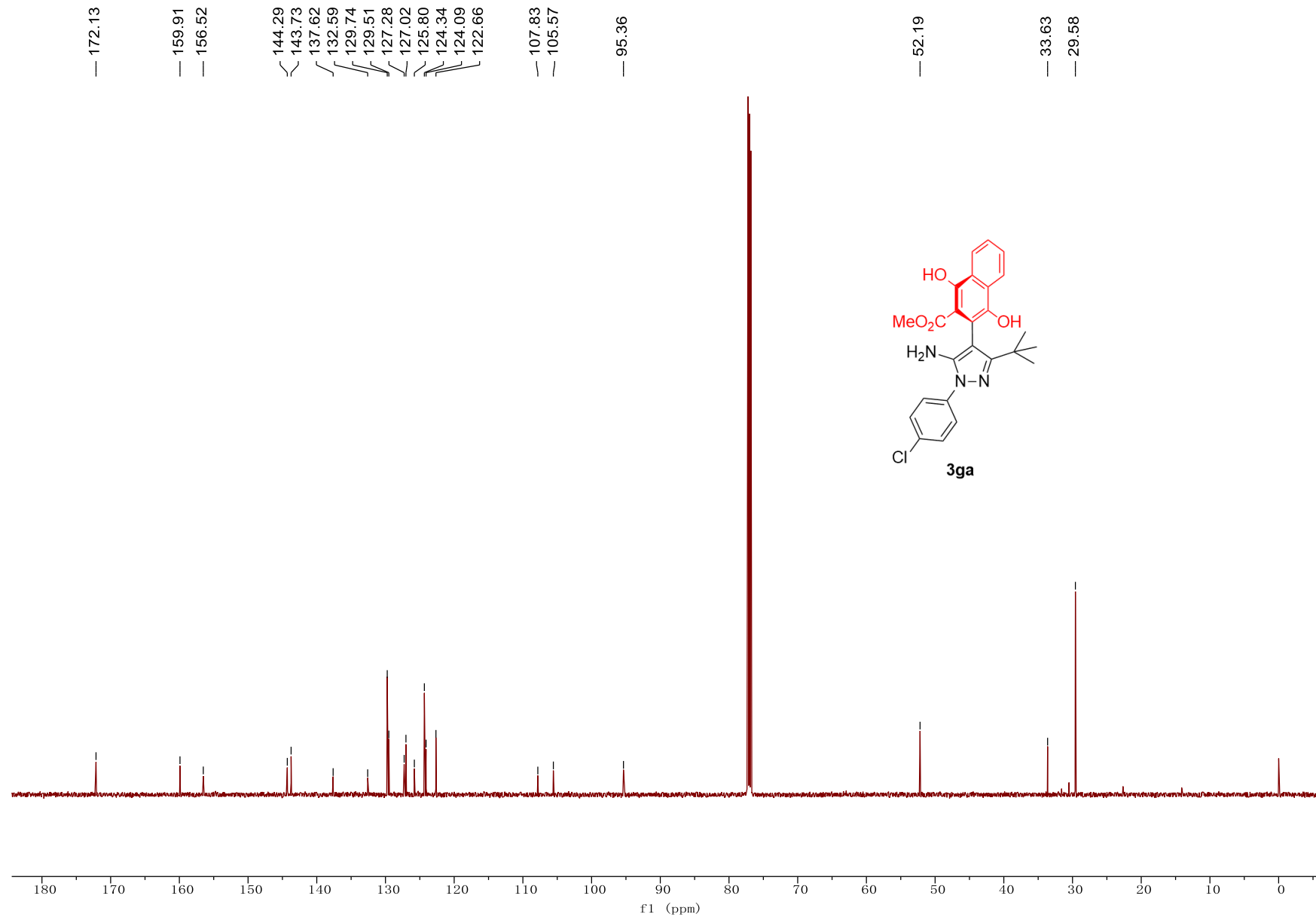


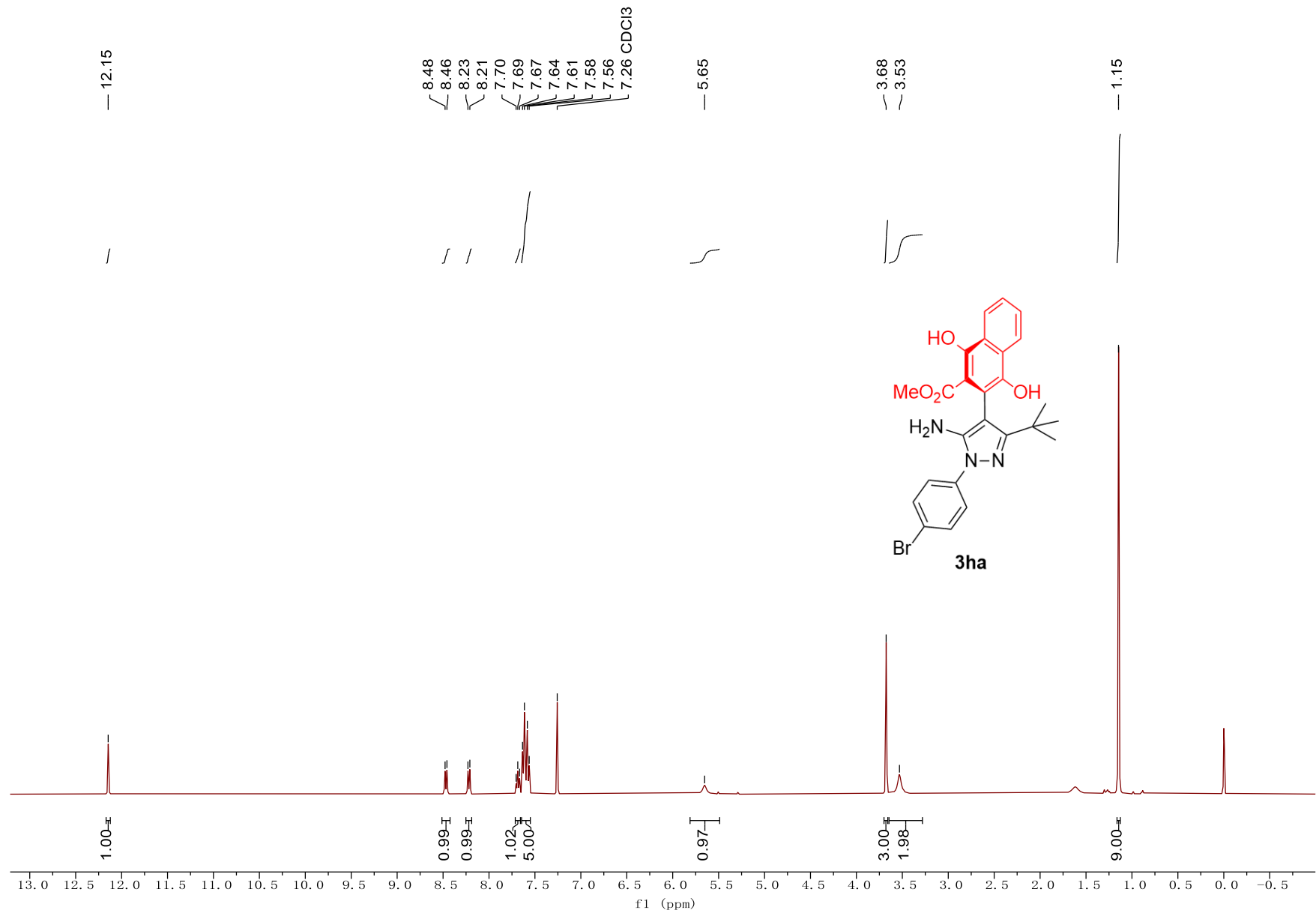


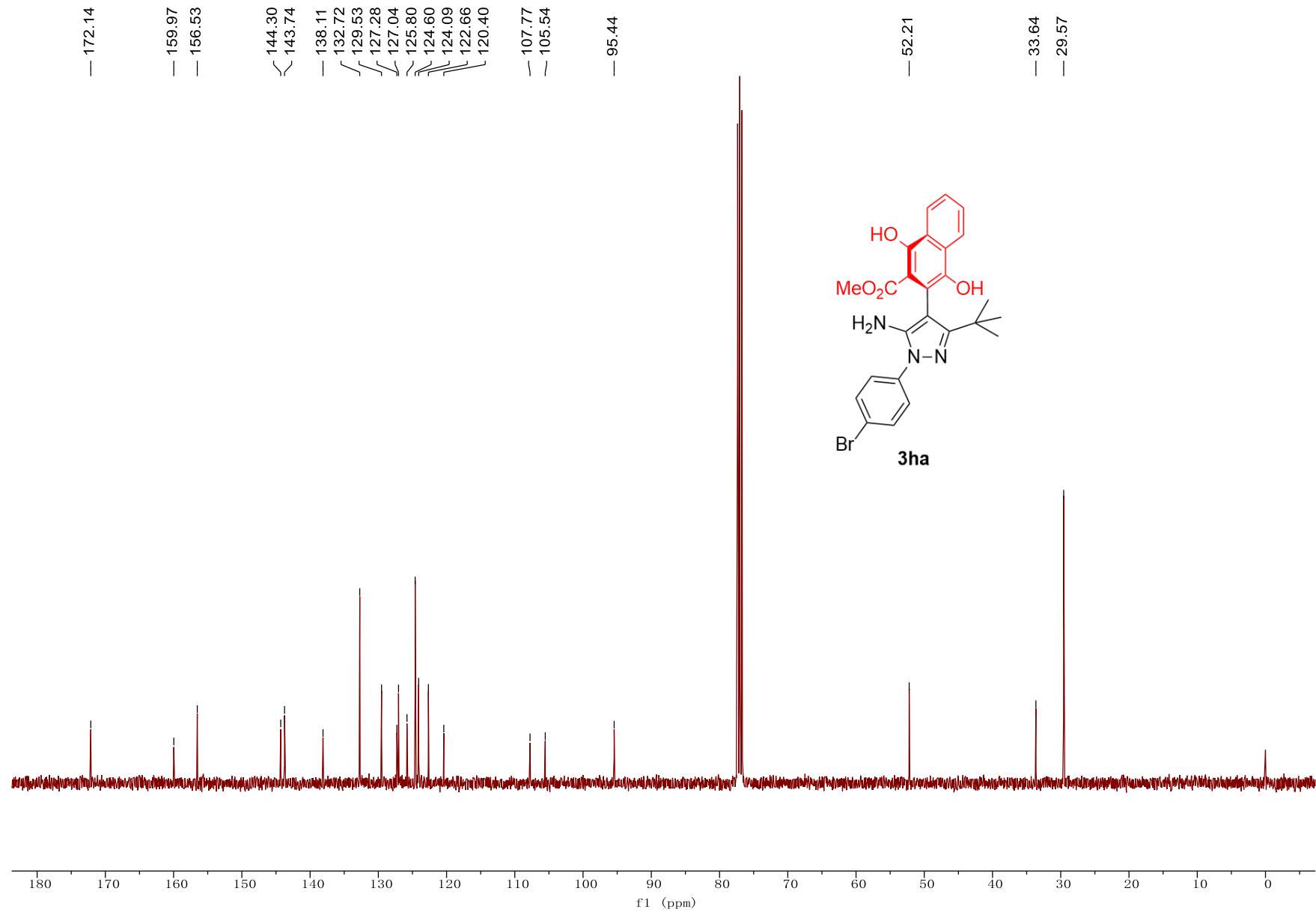
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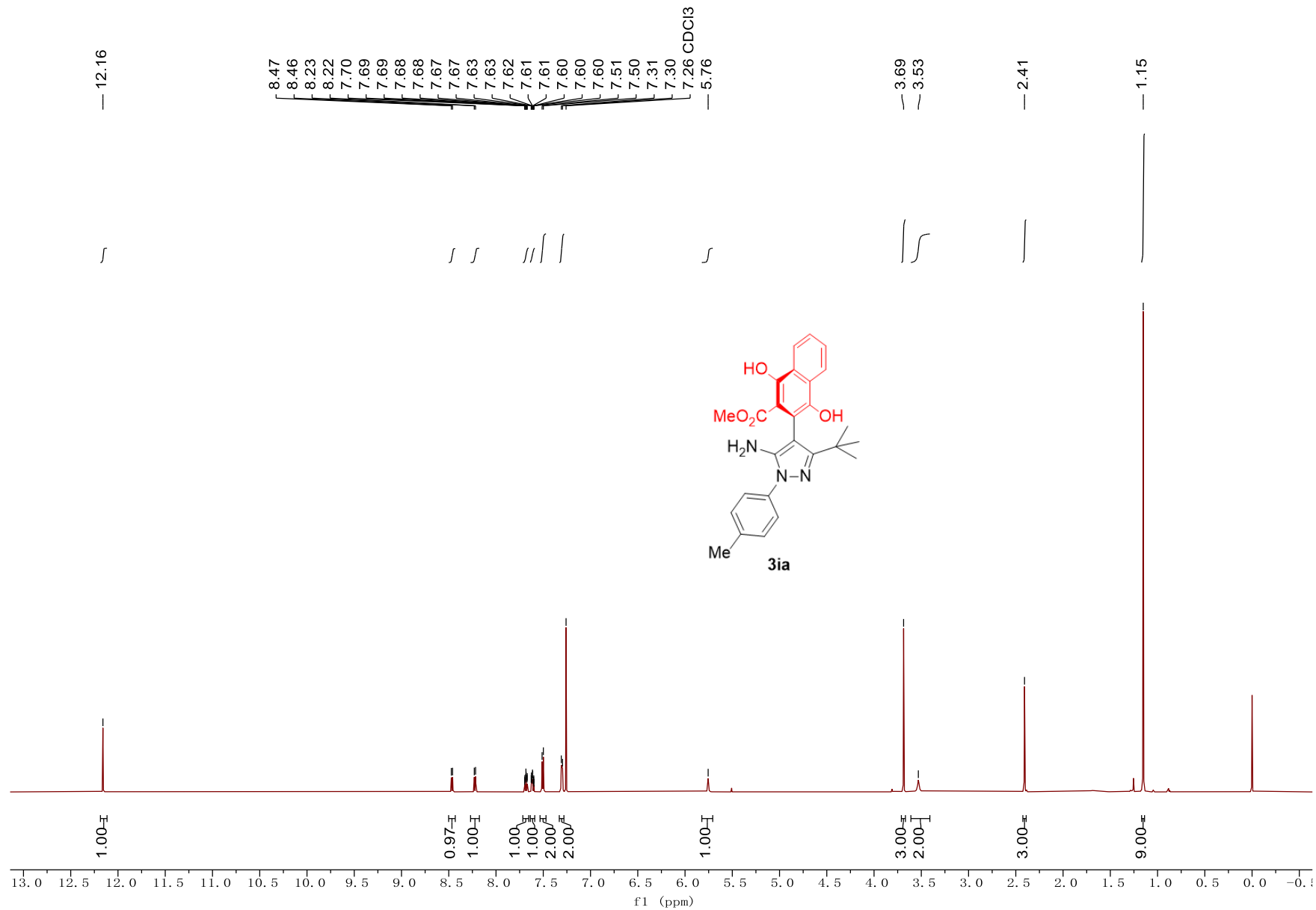


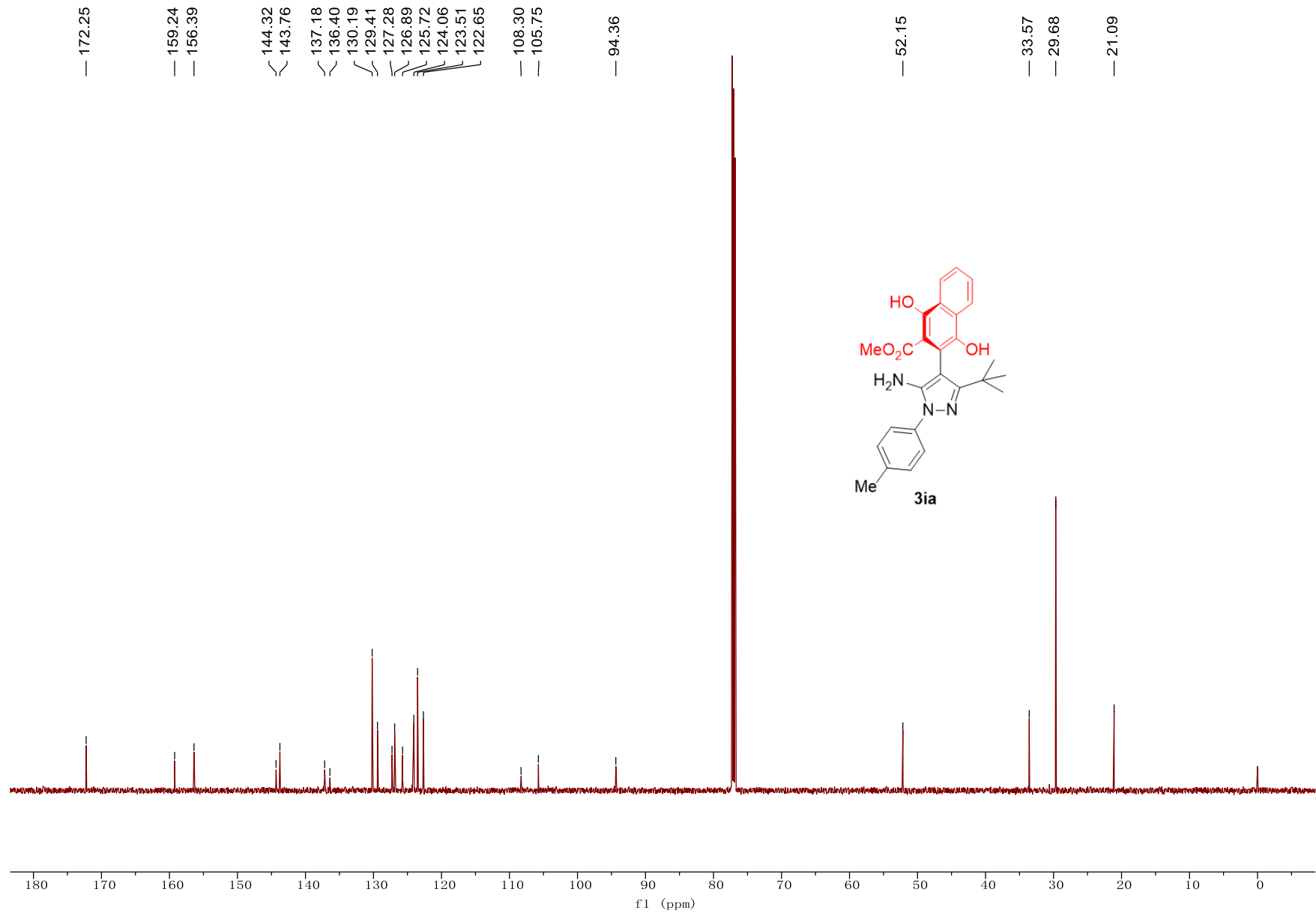


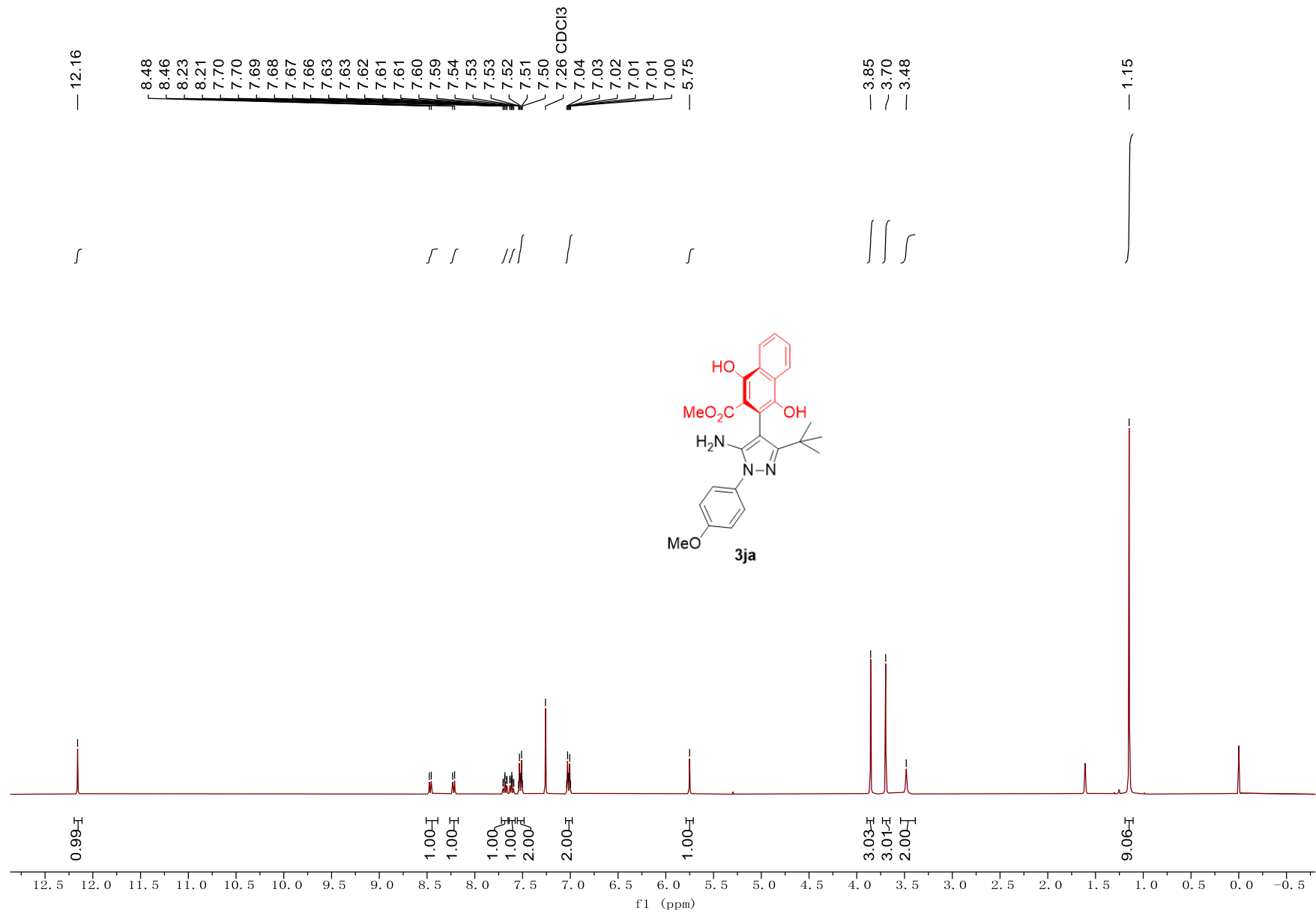


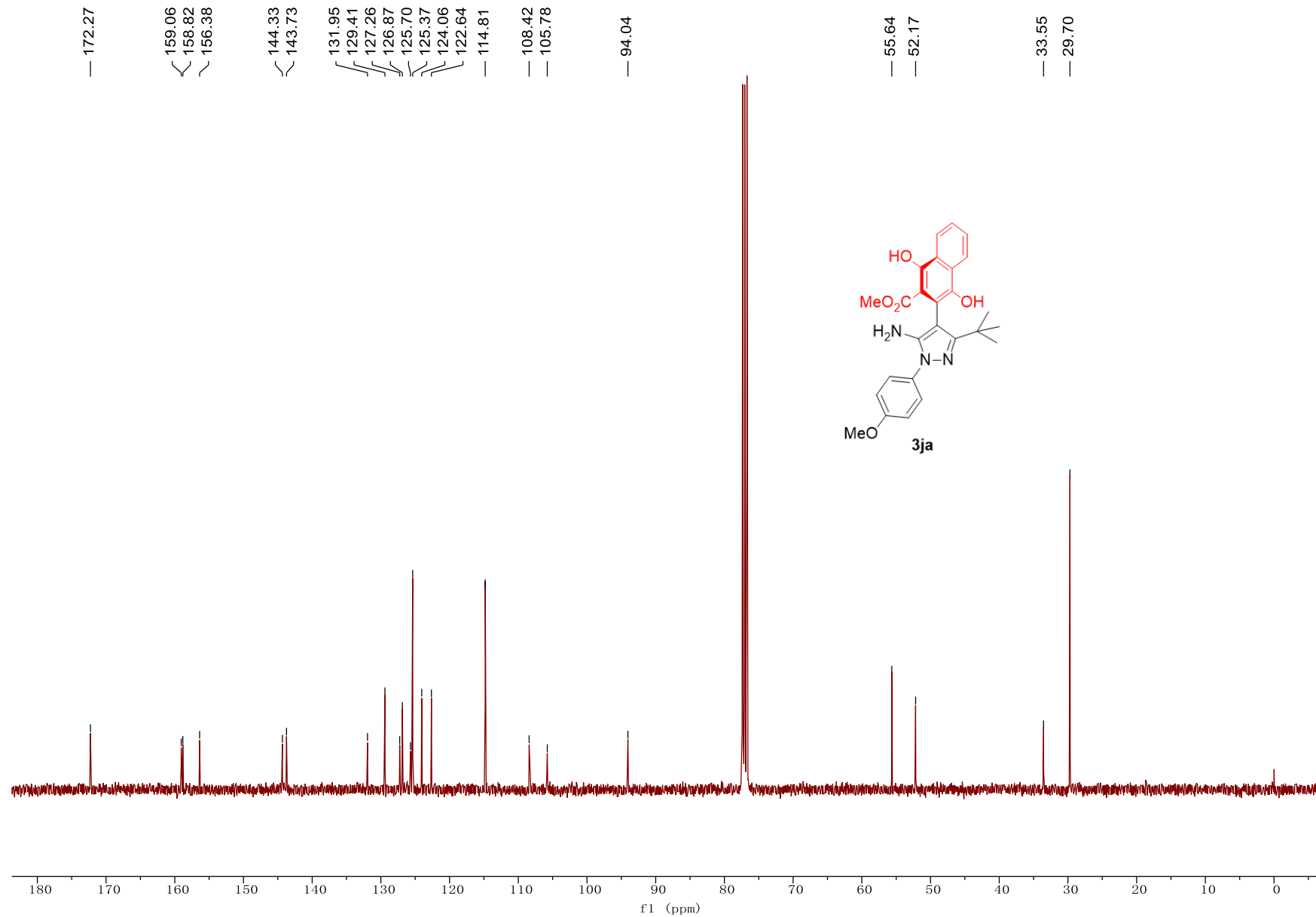


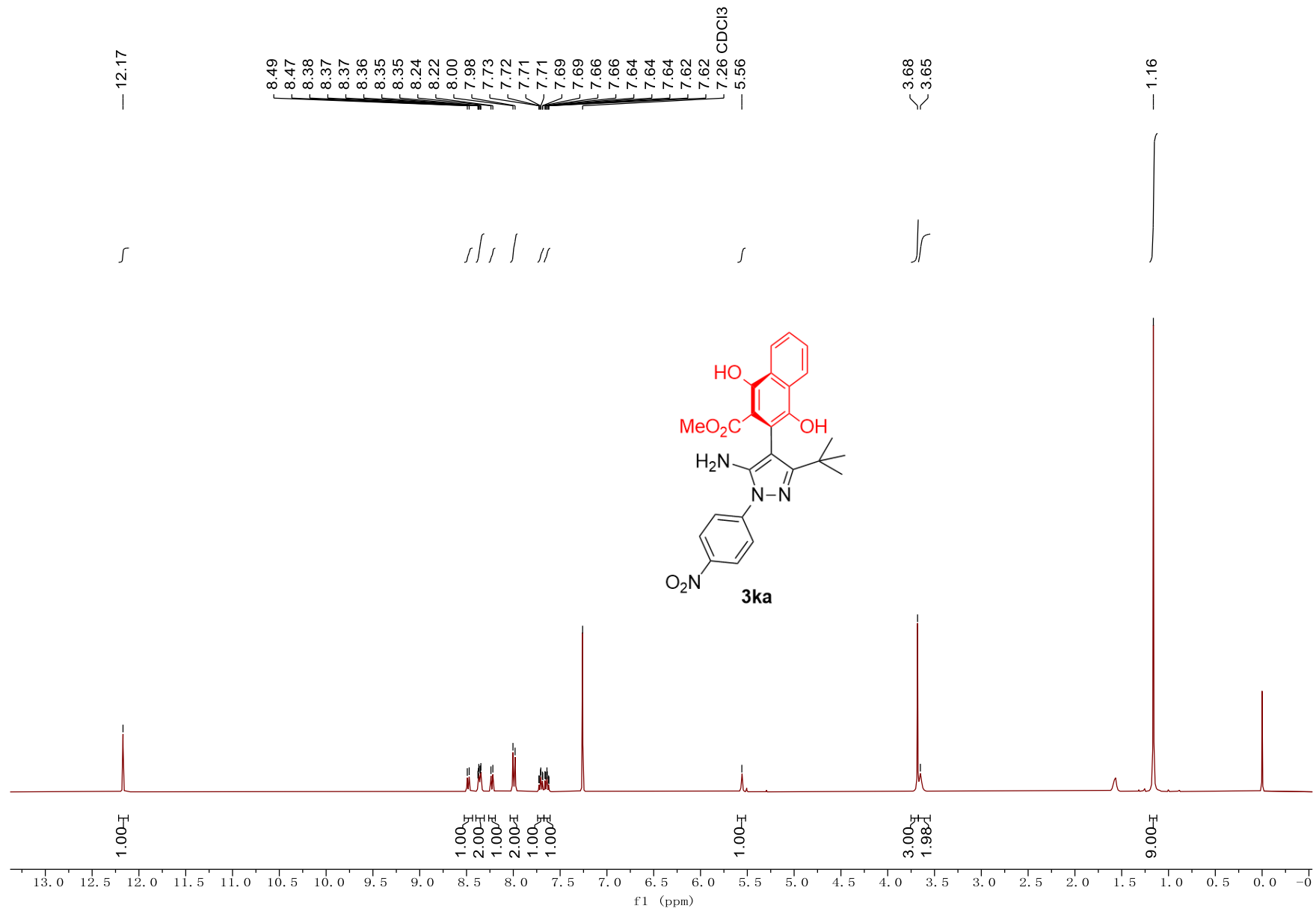


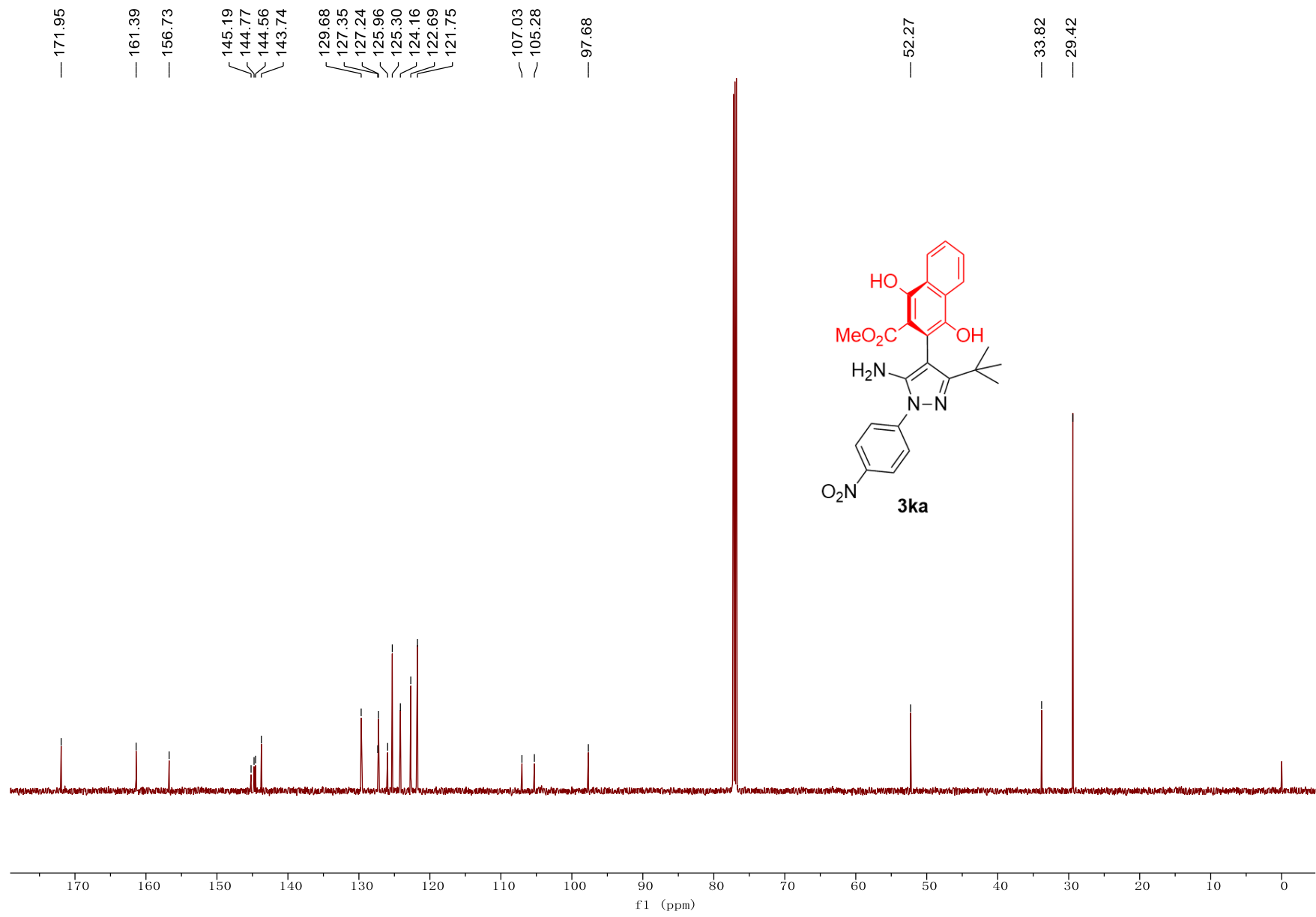


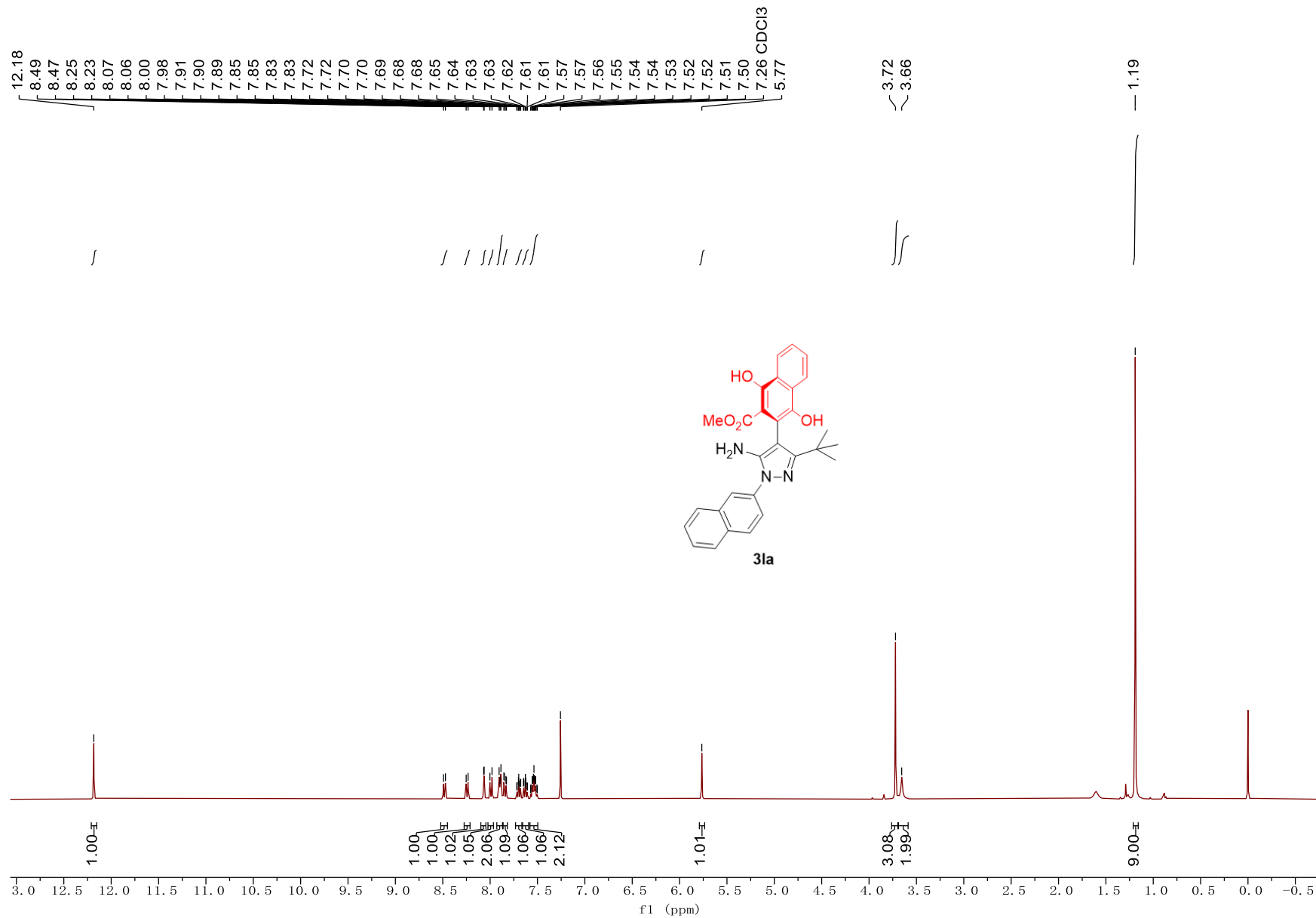


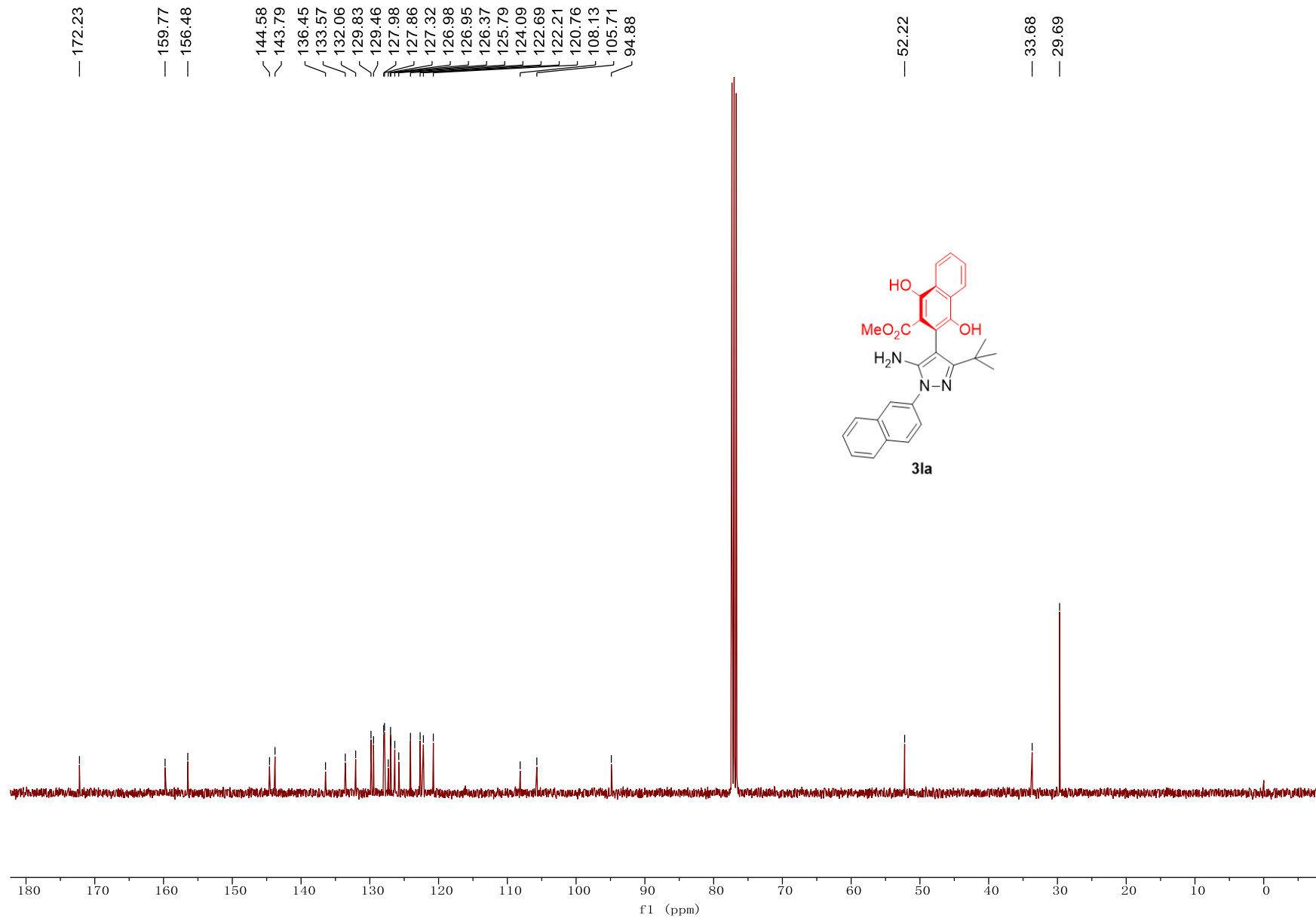


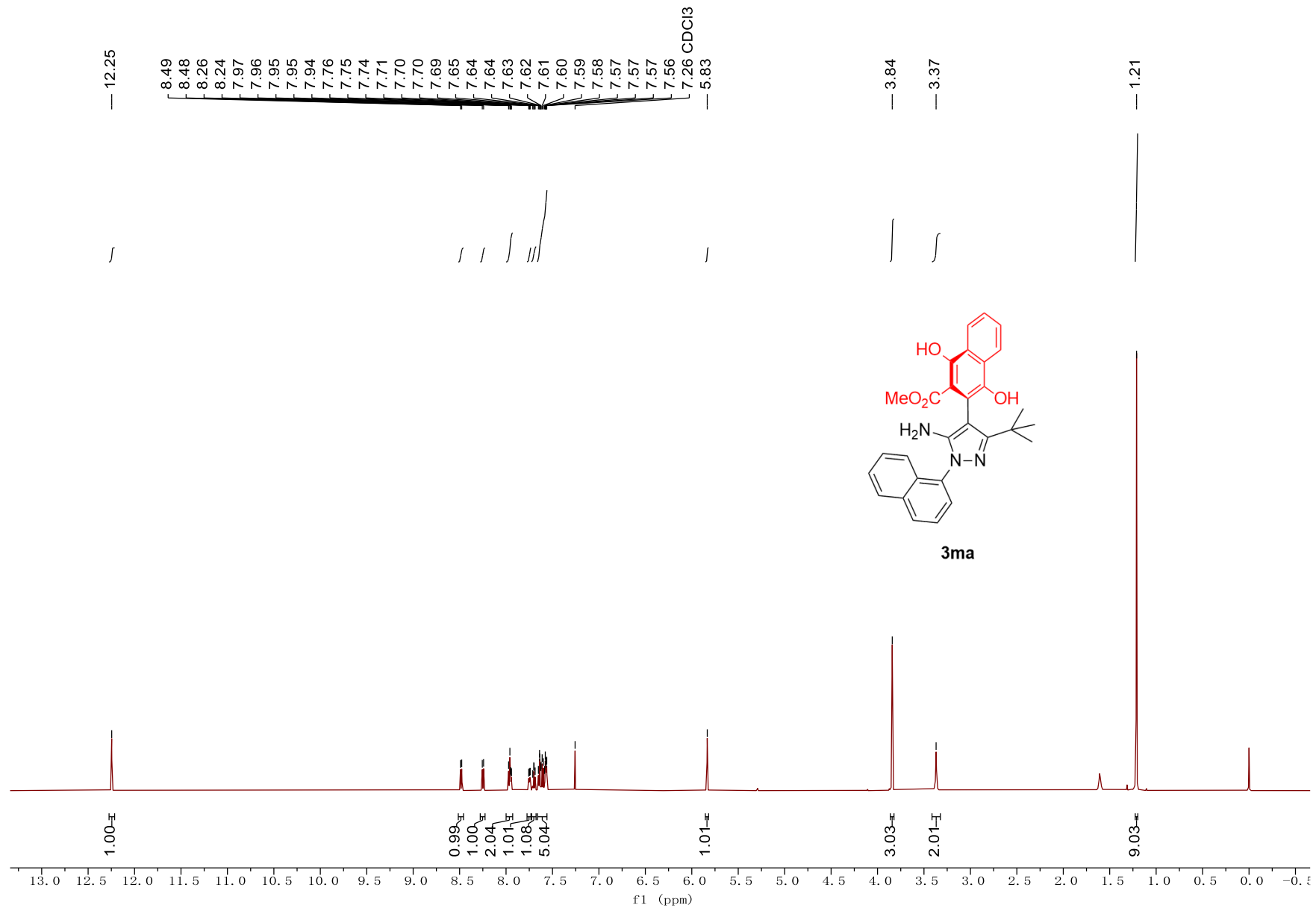


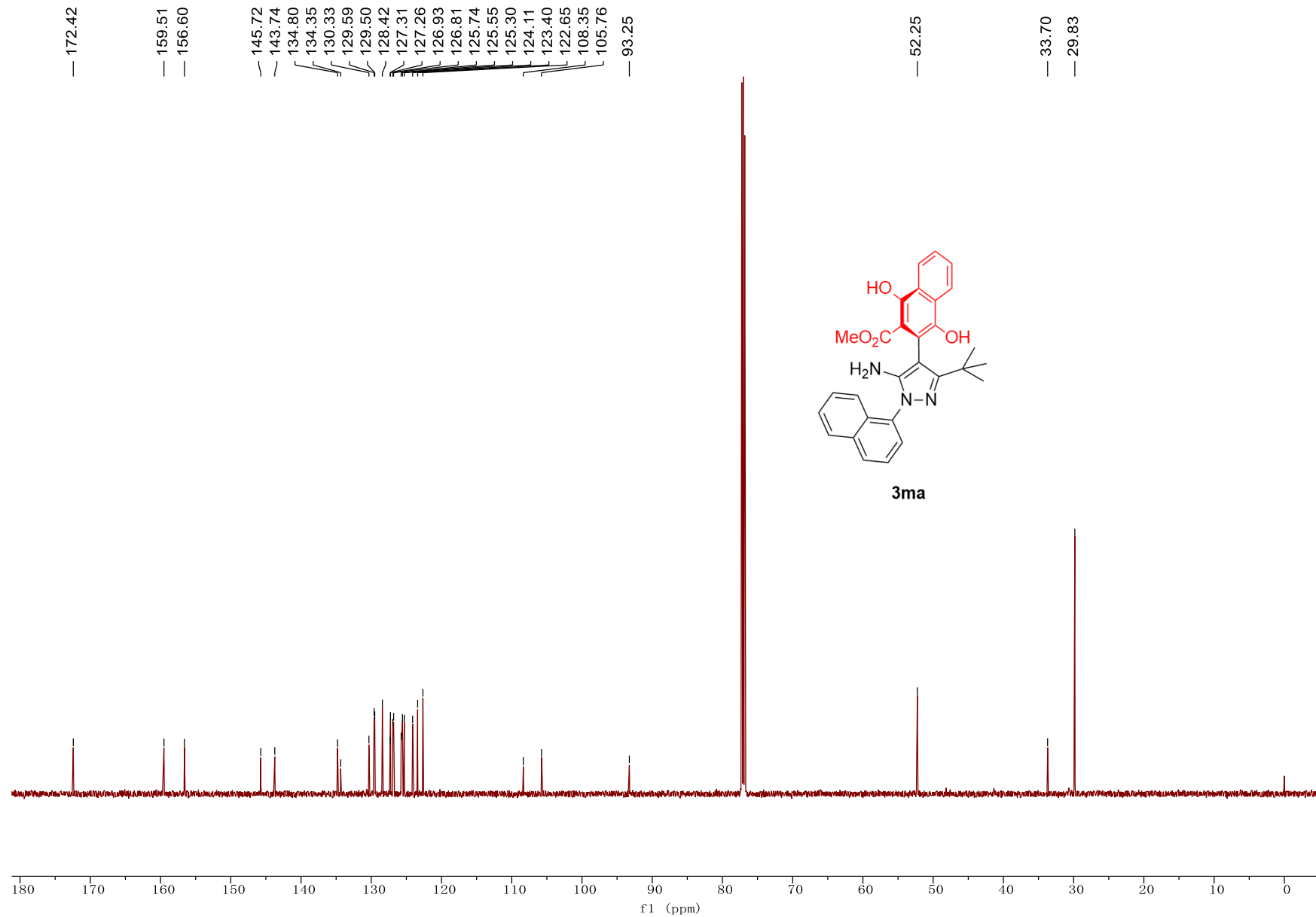


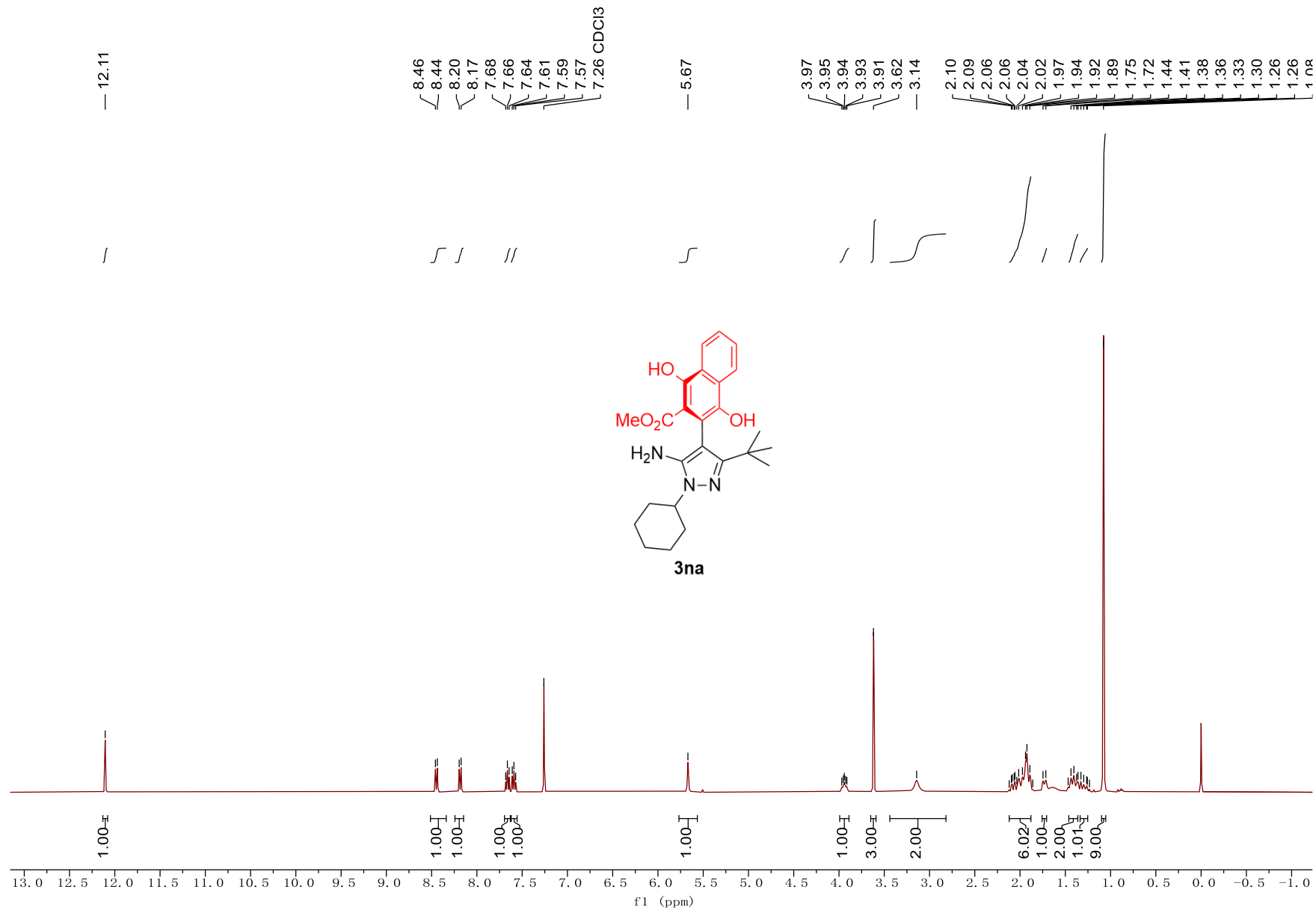


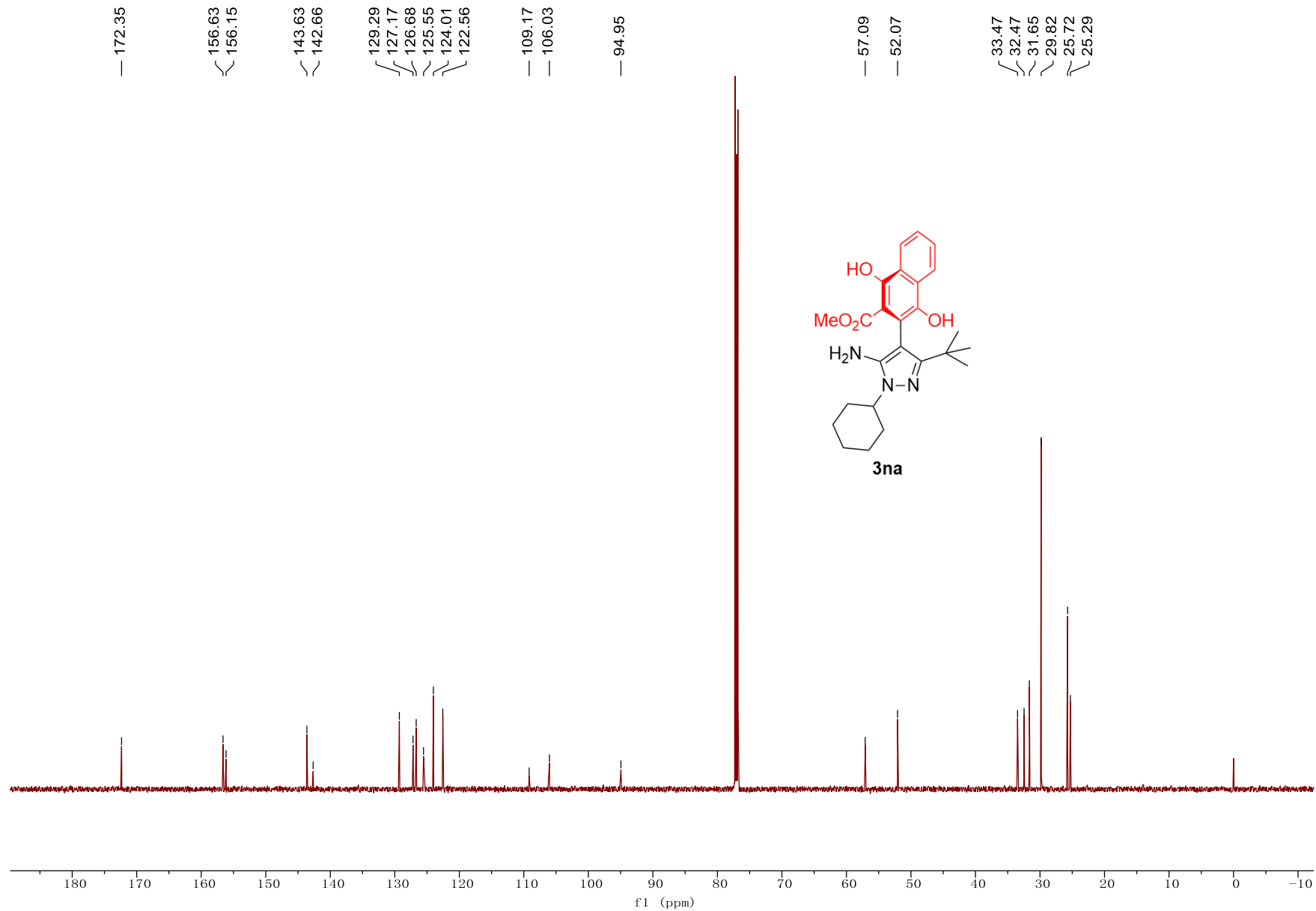


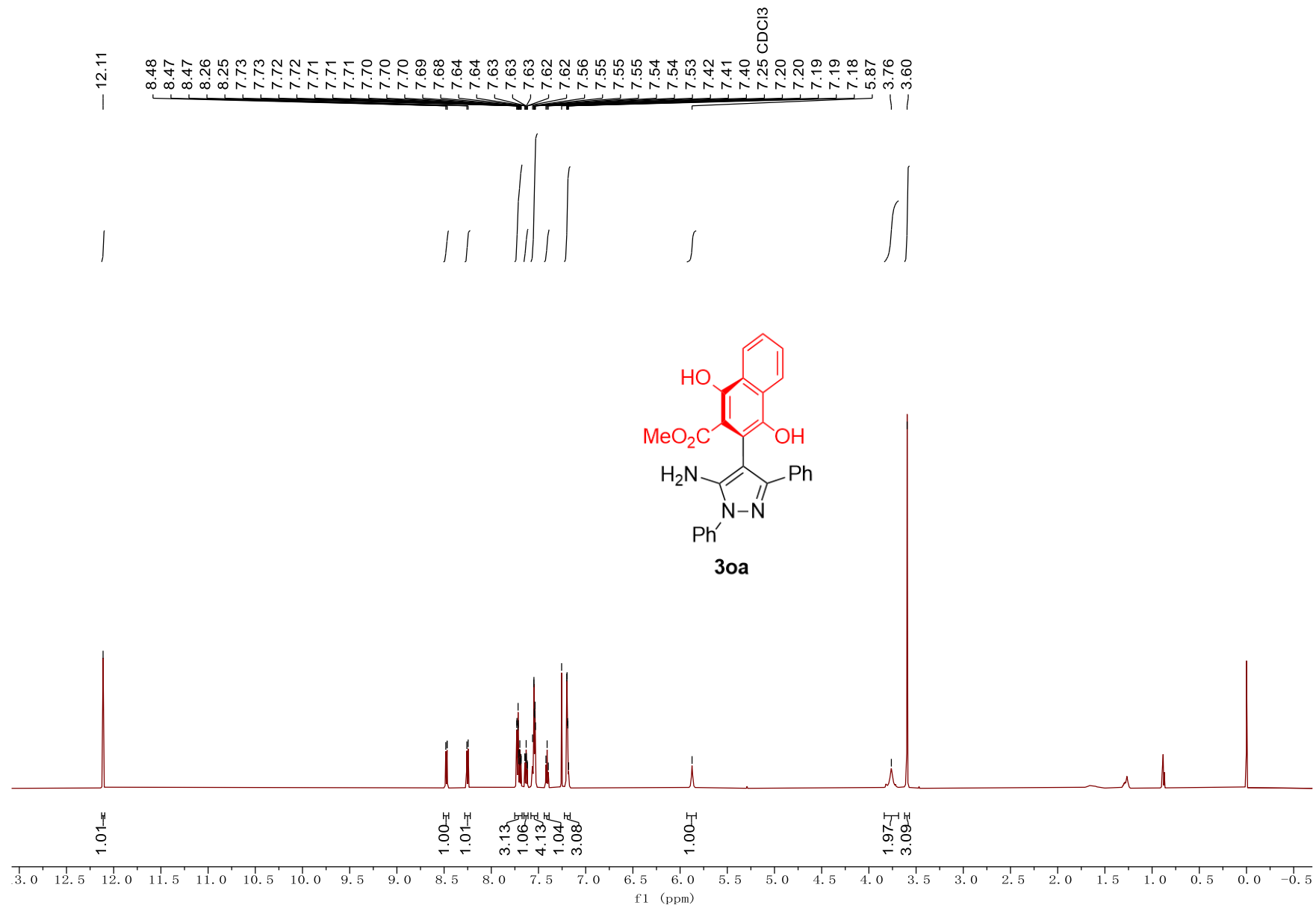


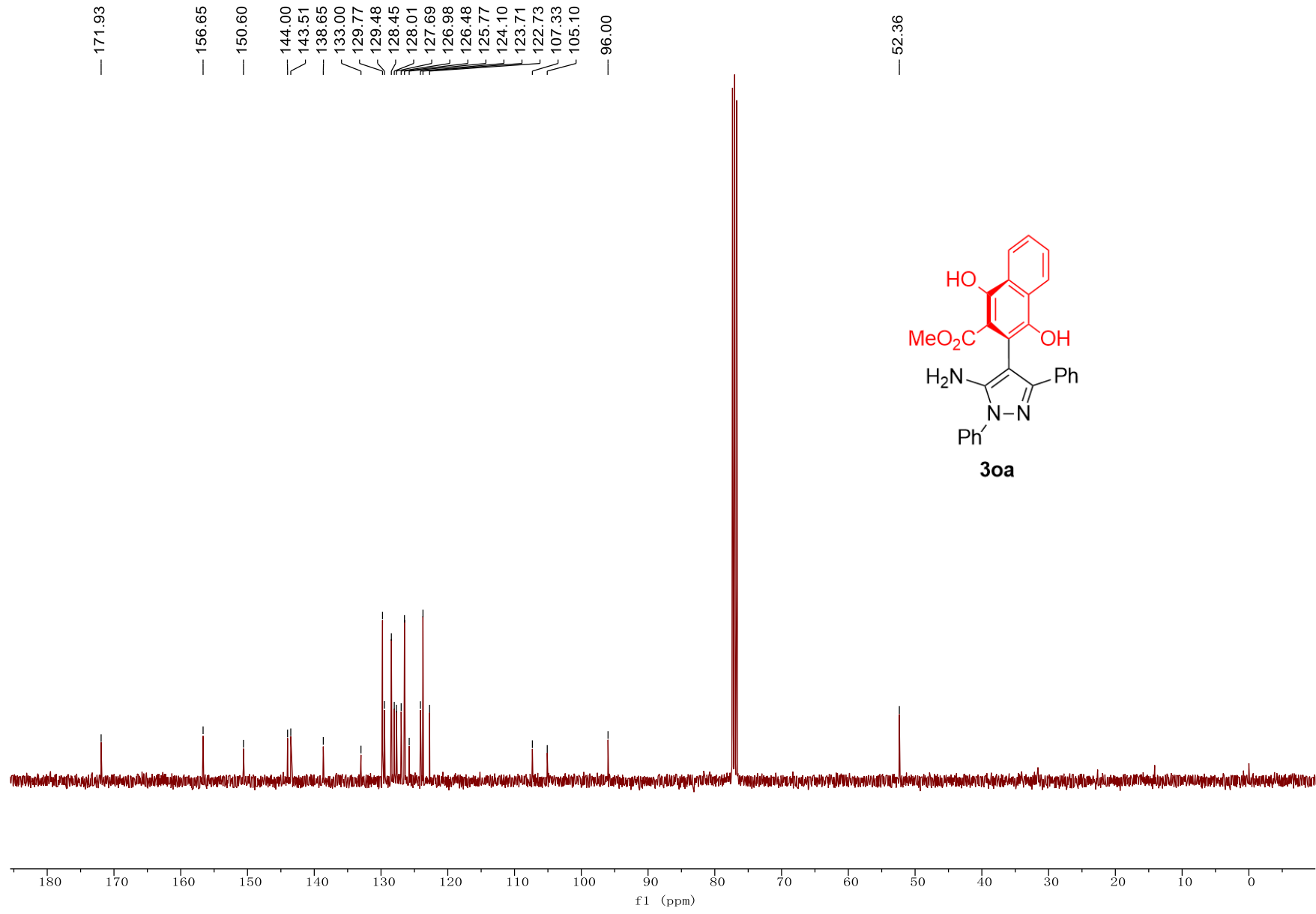


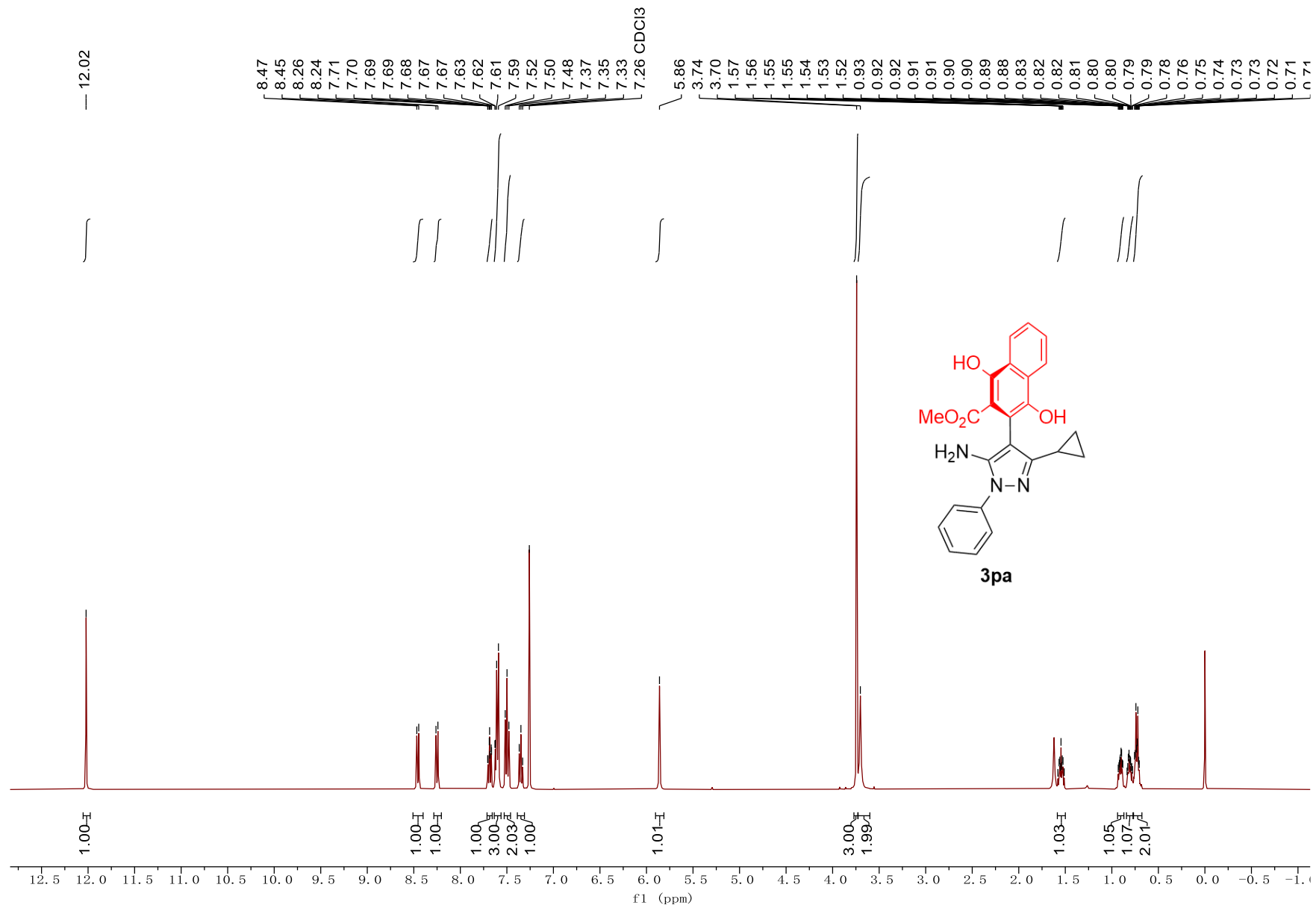


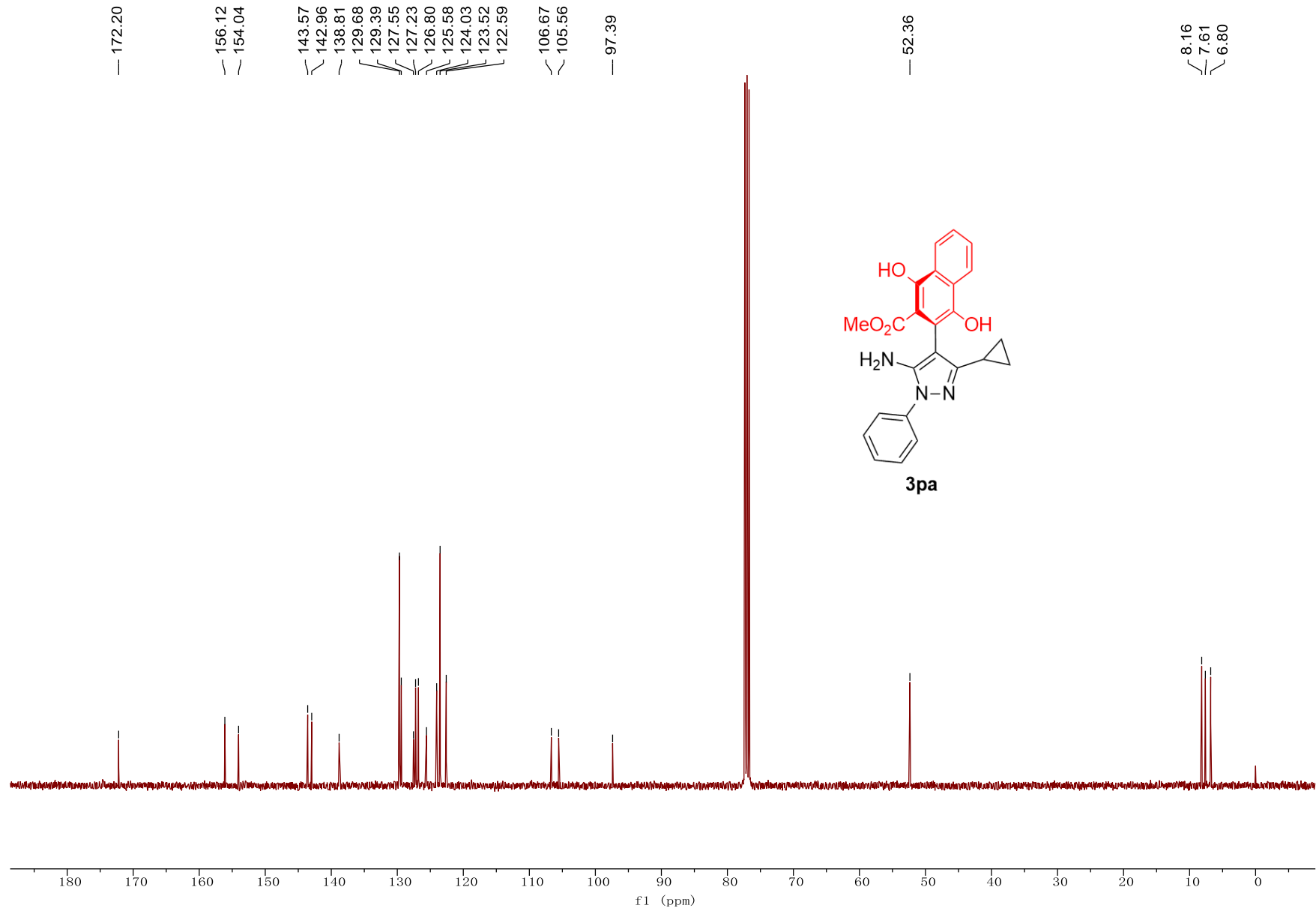


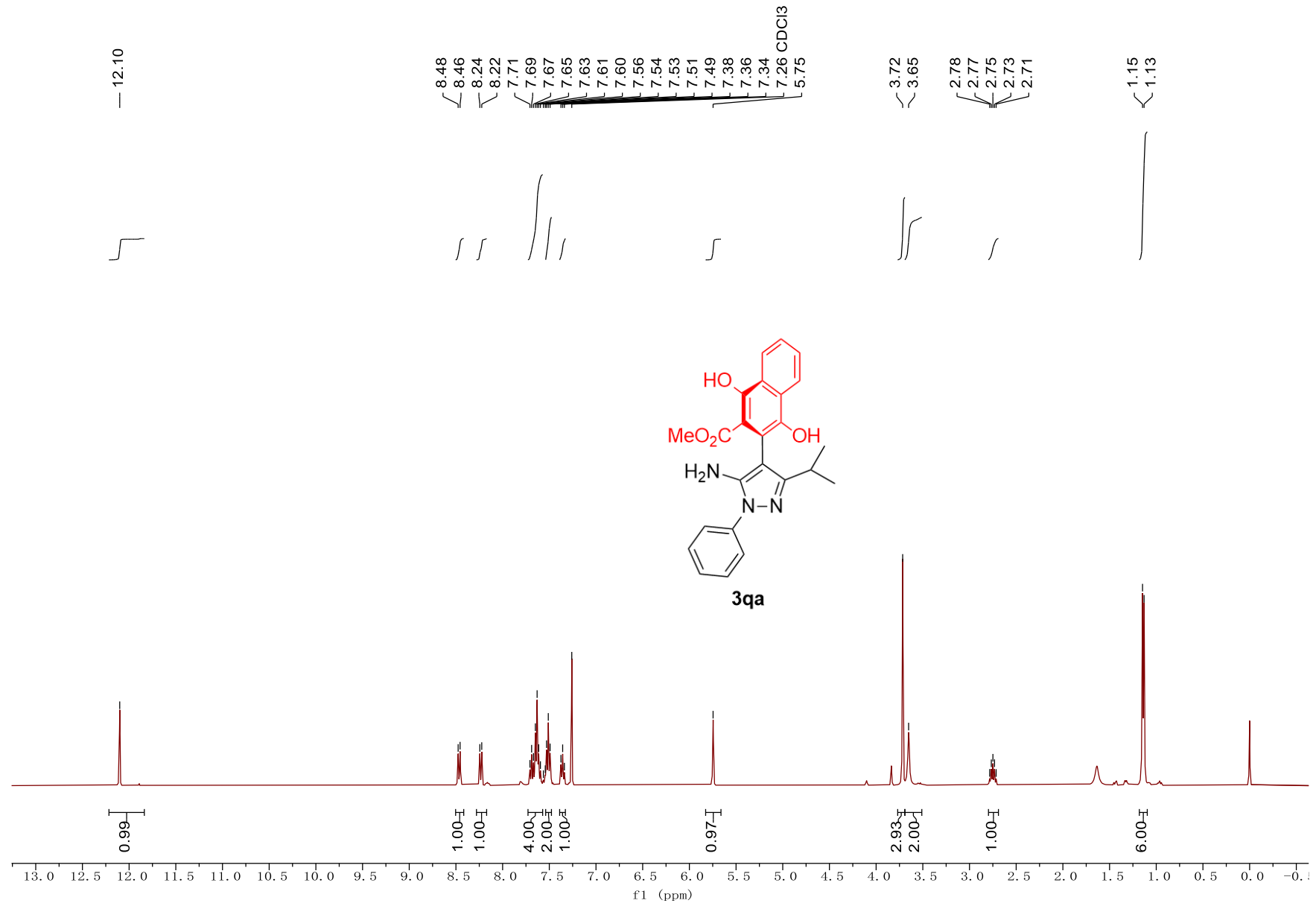


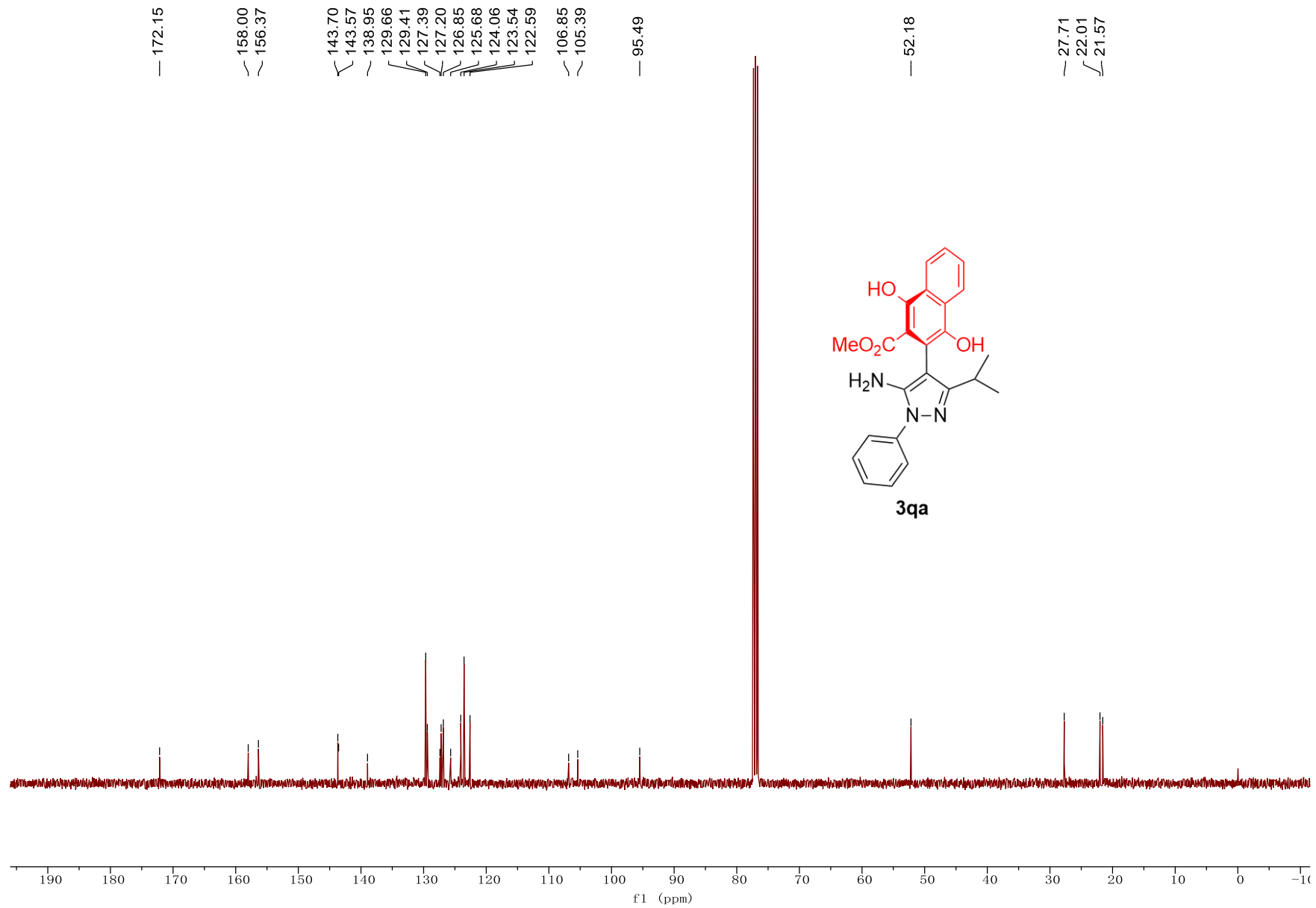


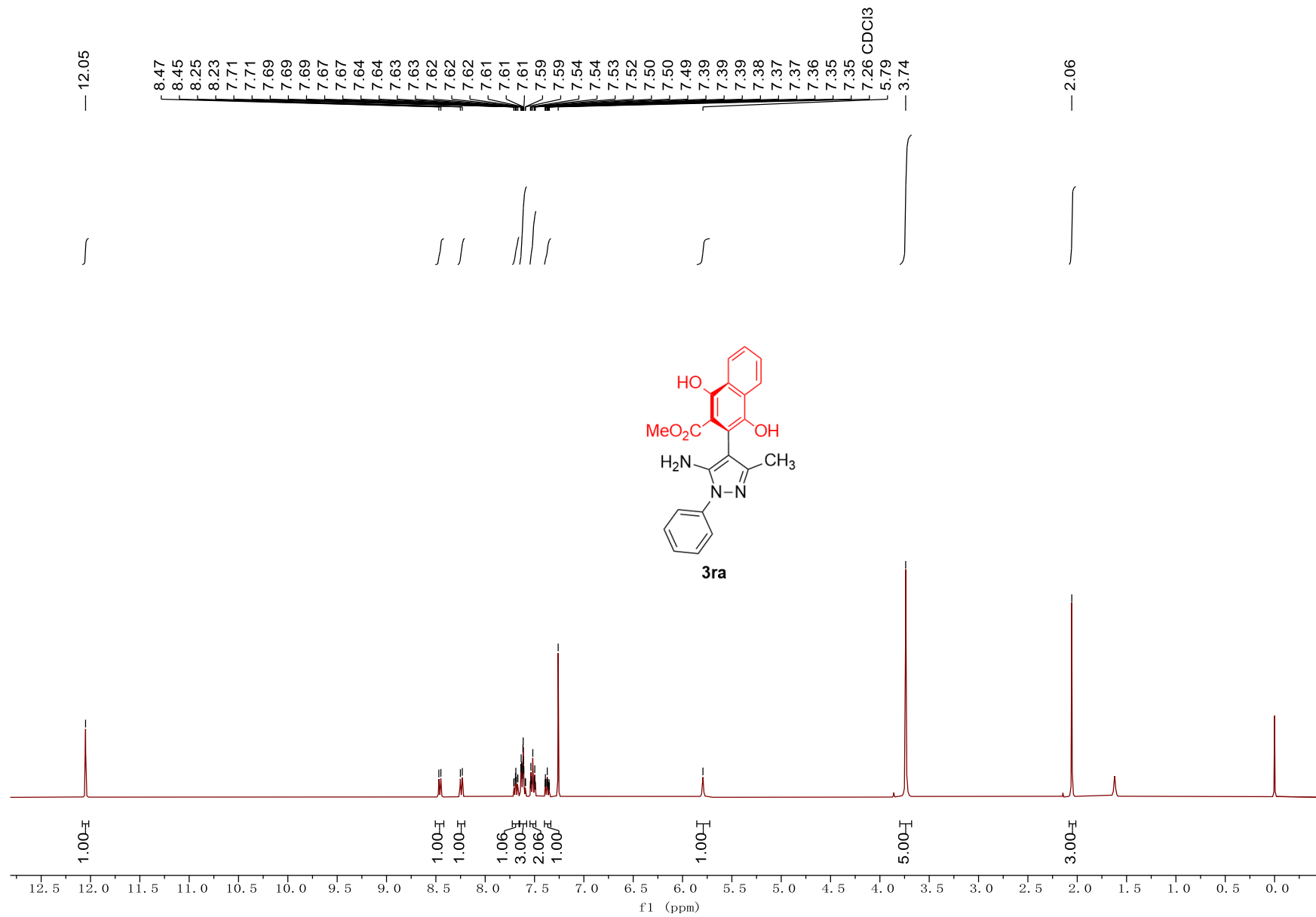


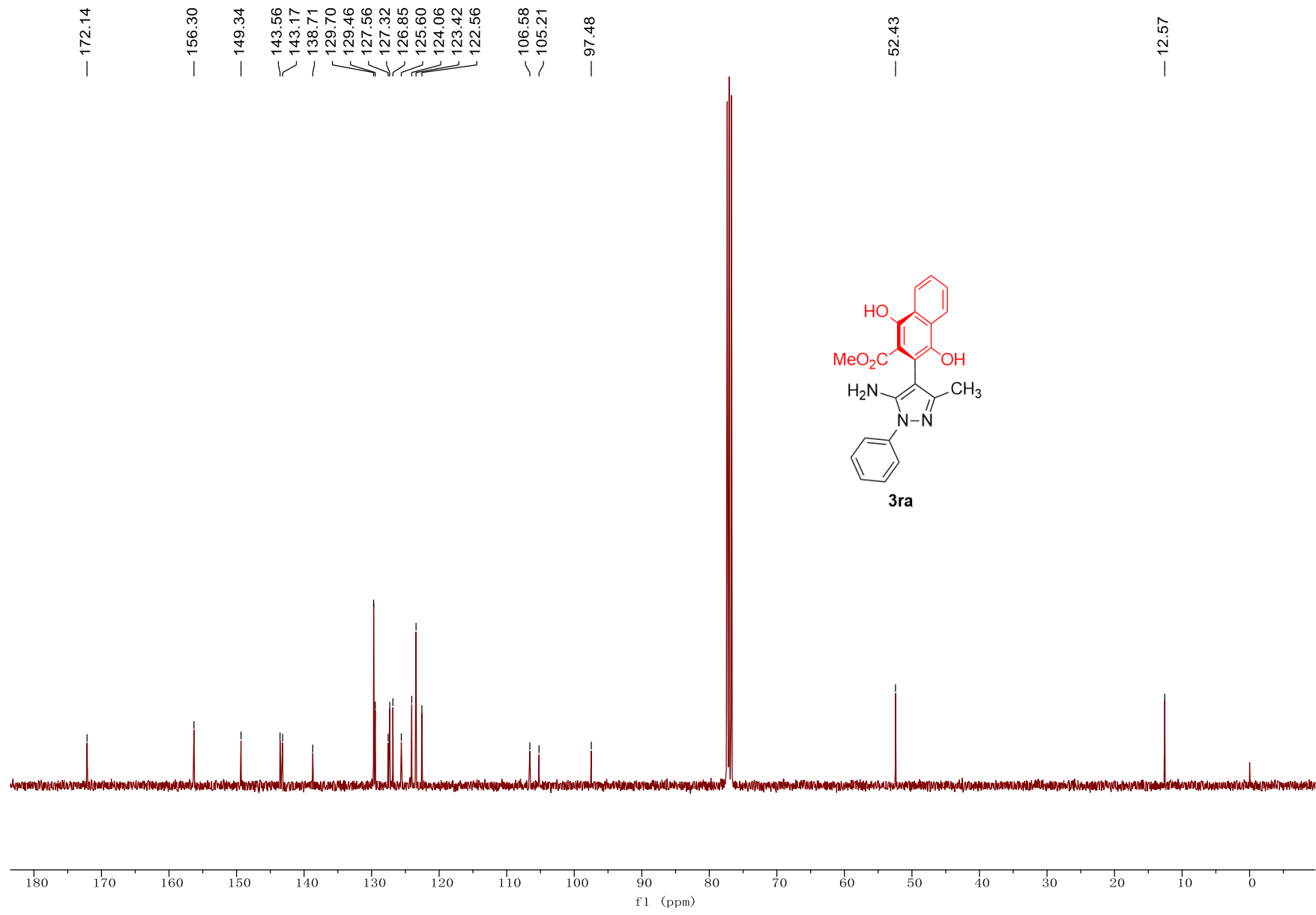












— 172.14

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— 149.34

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143.17

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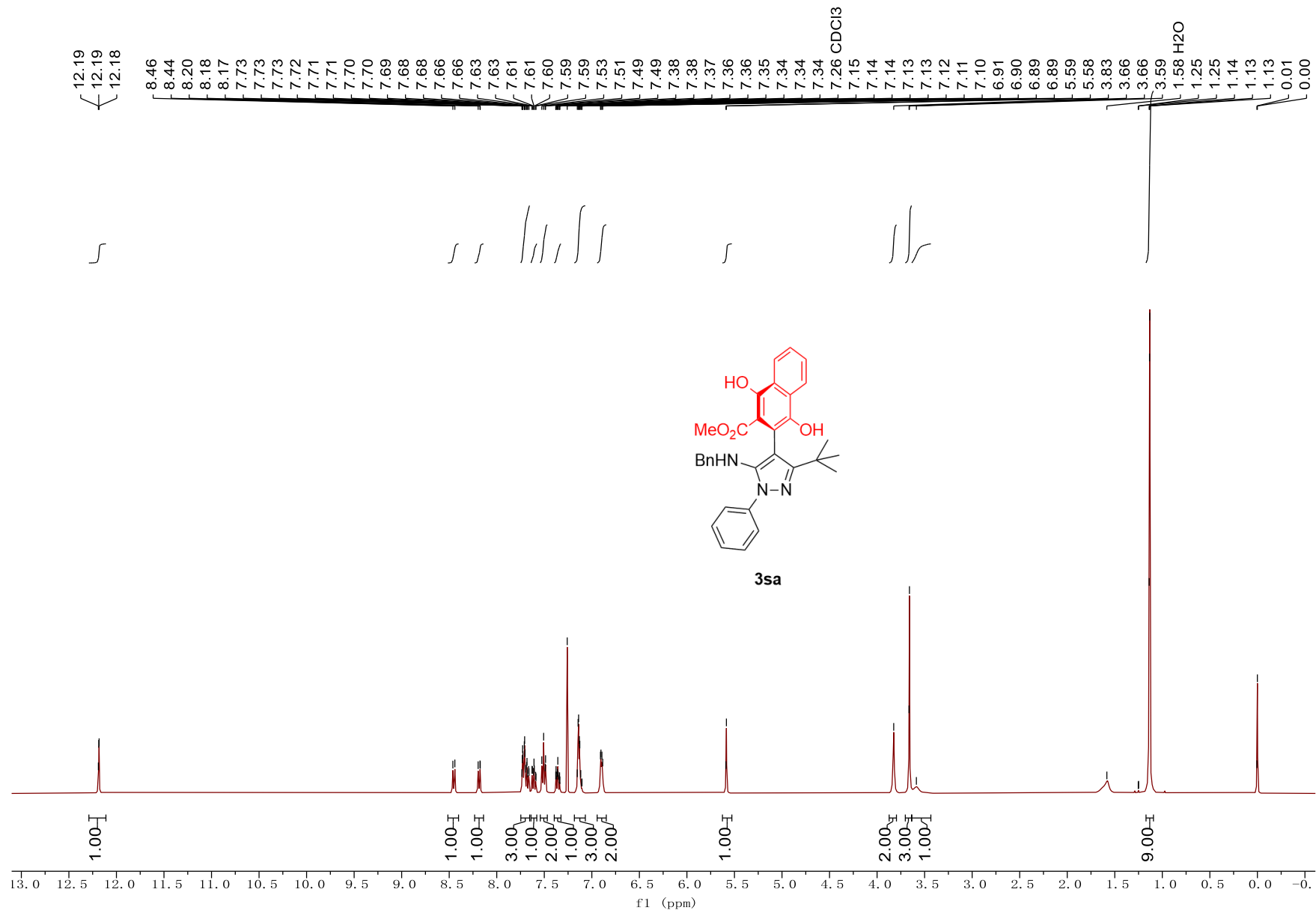
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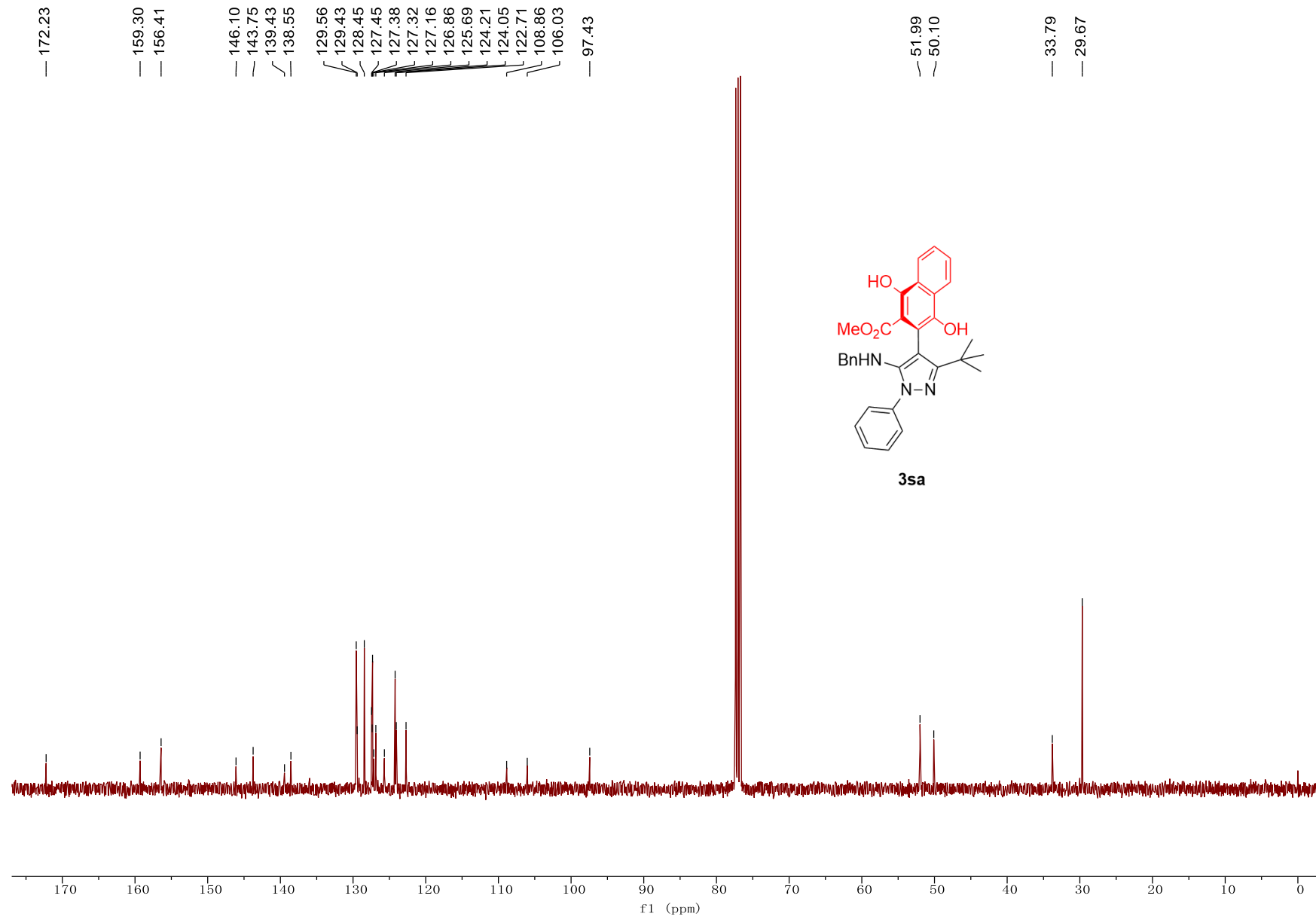
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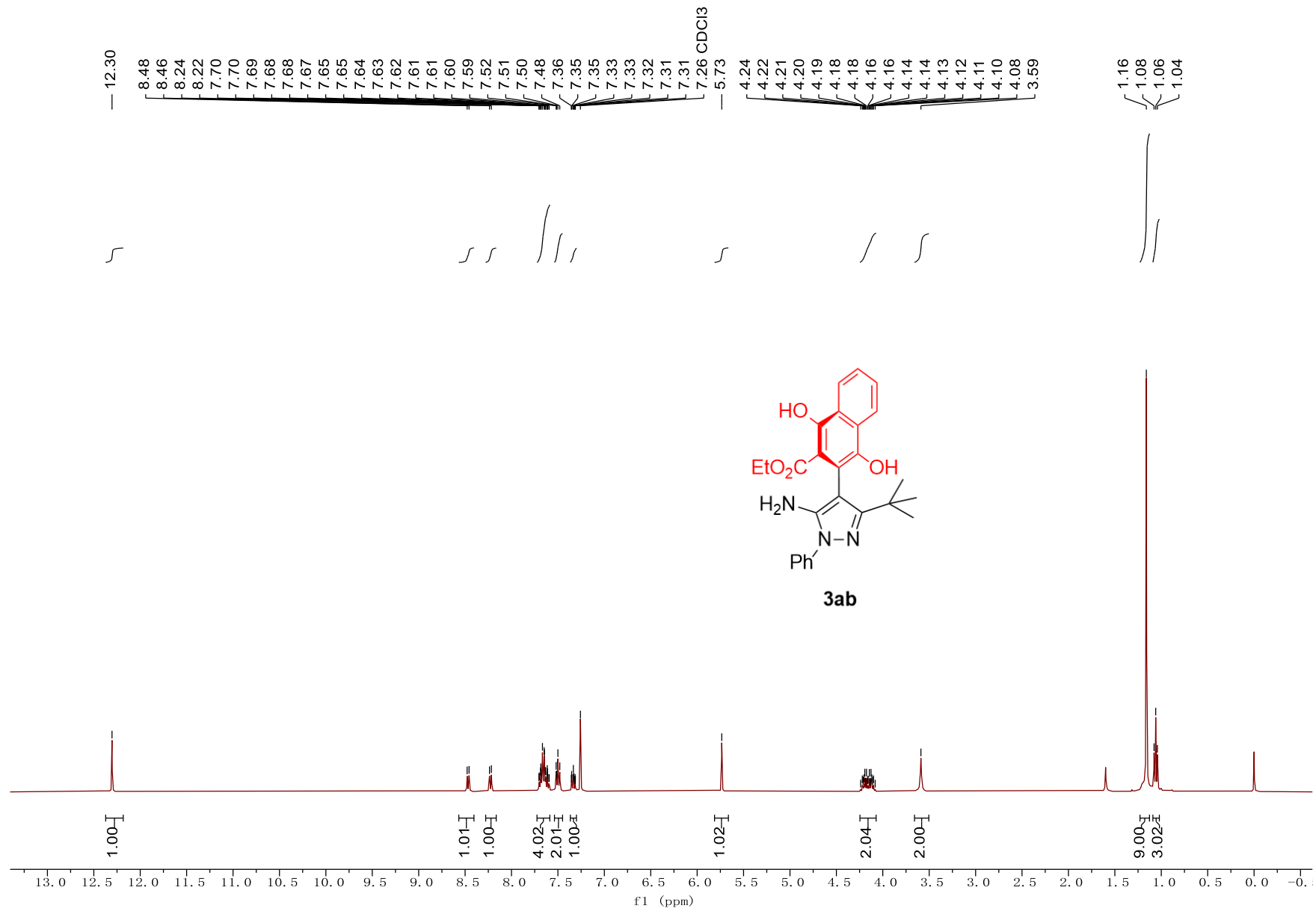
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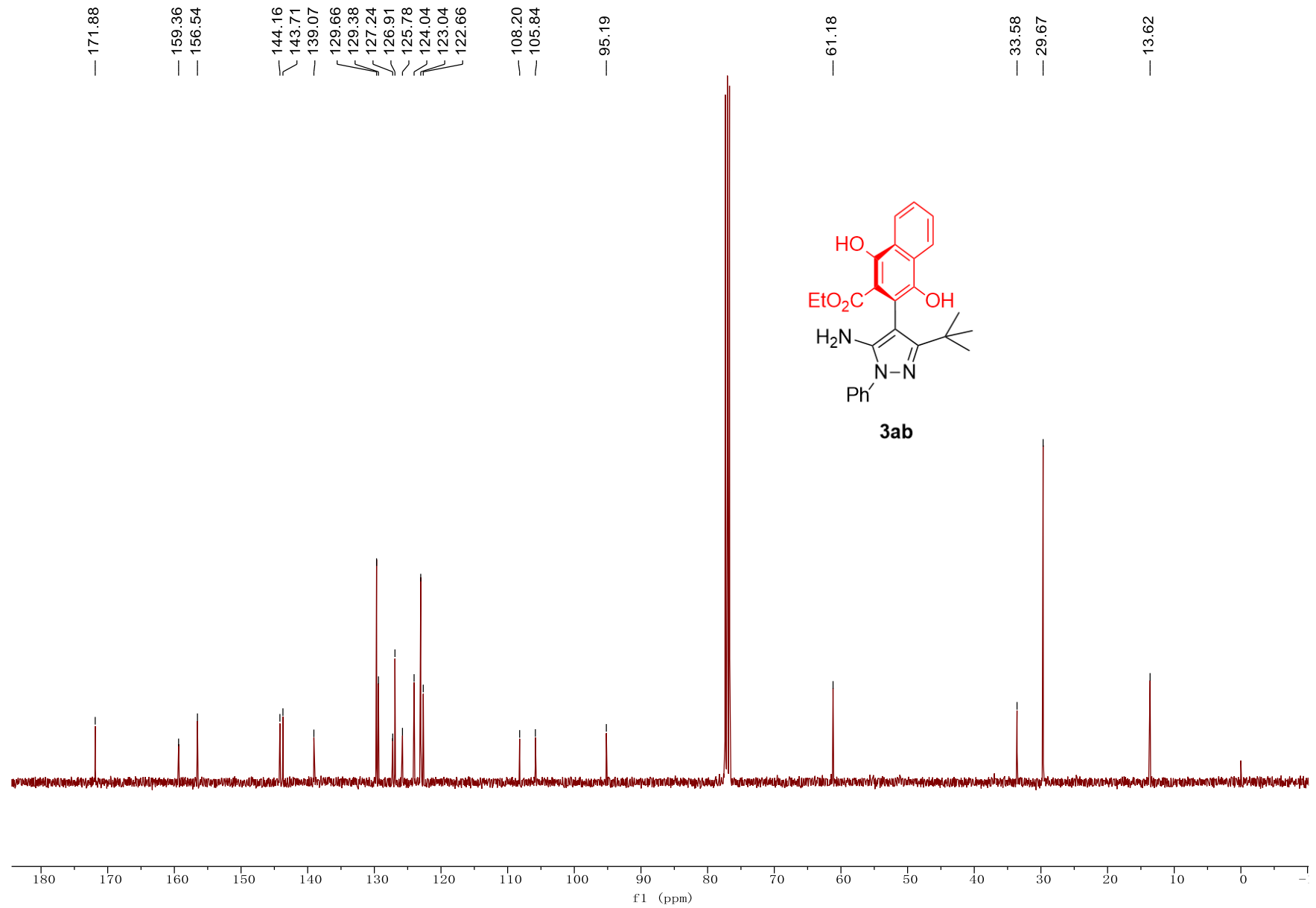
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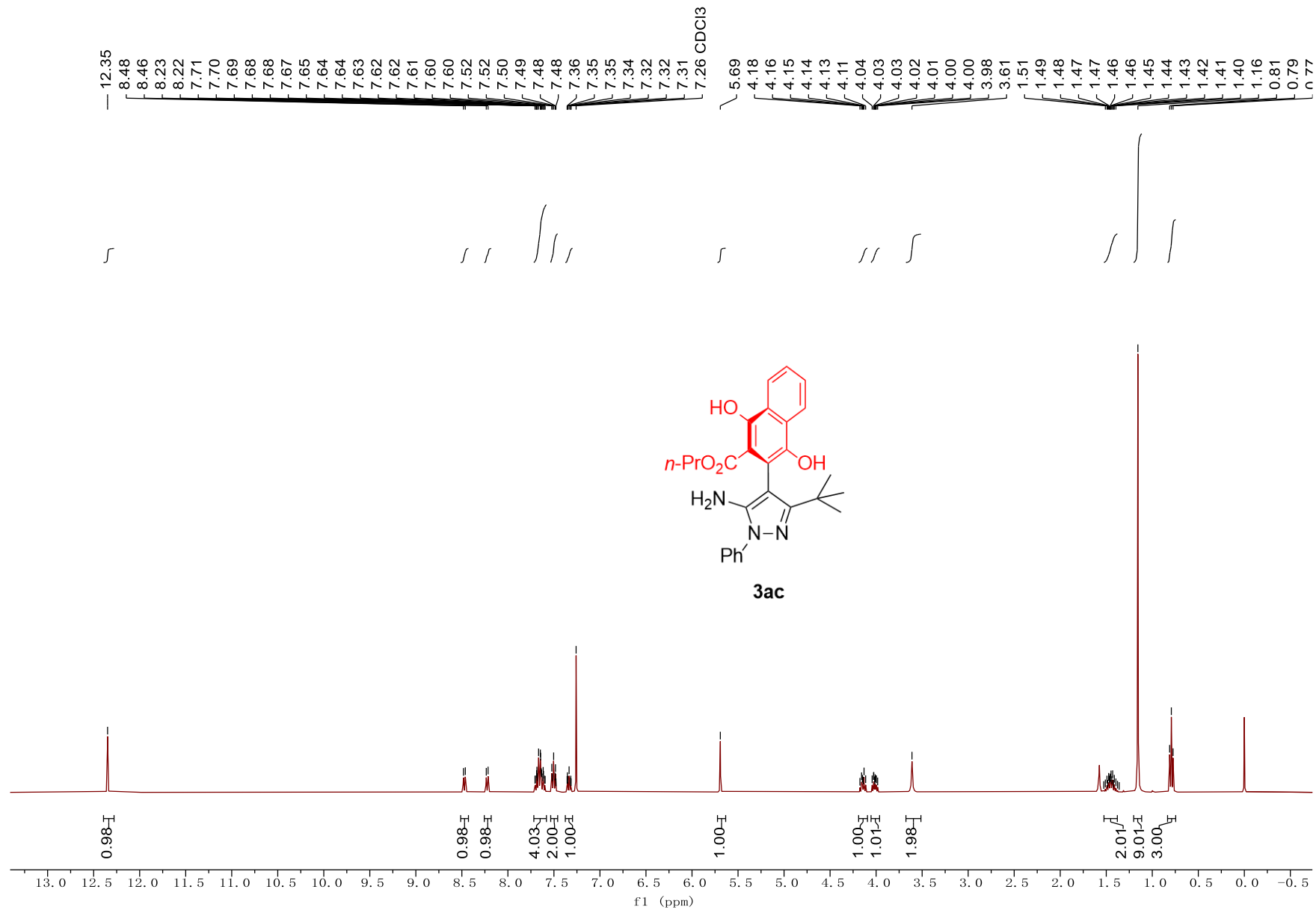
S 58

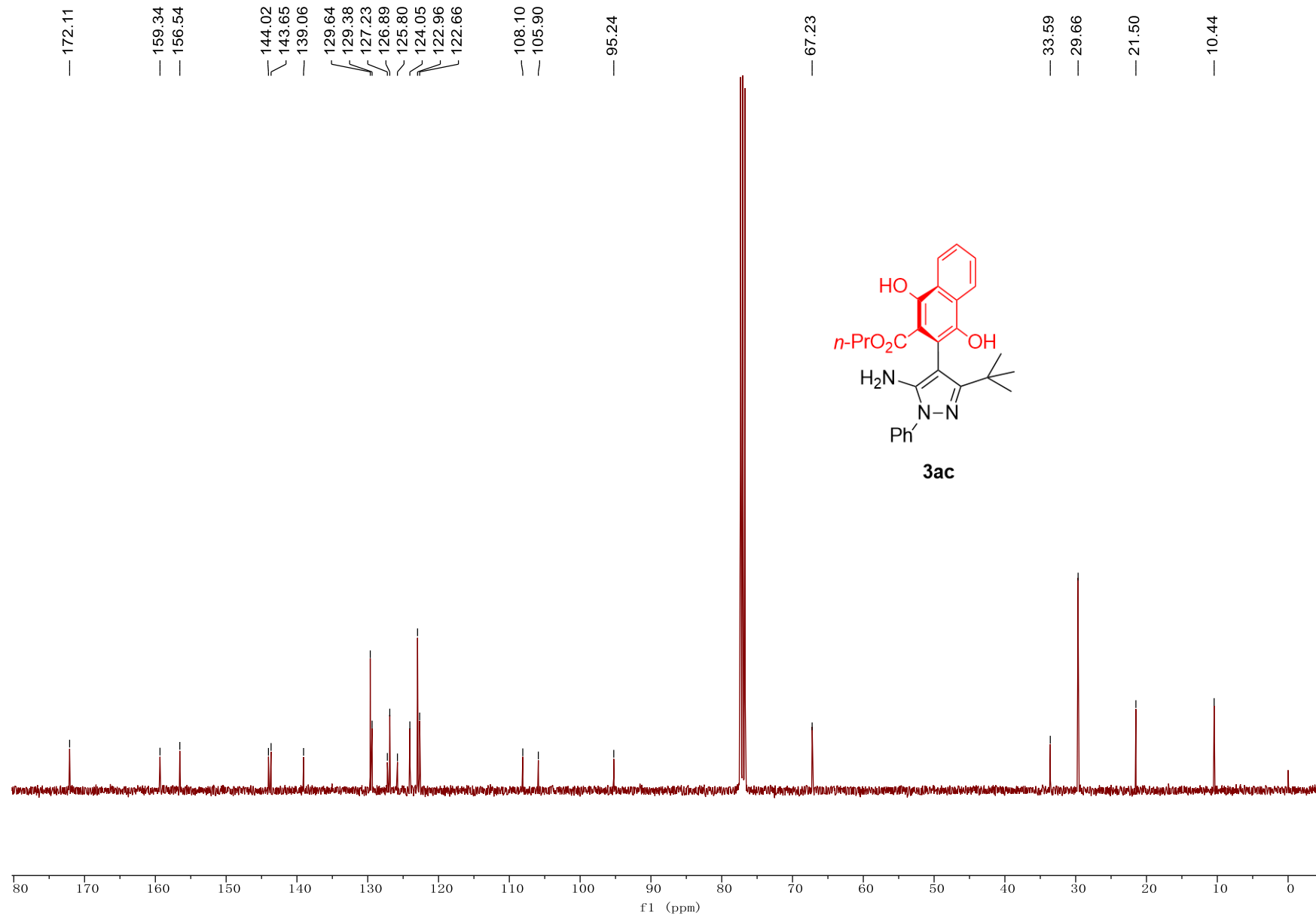


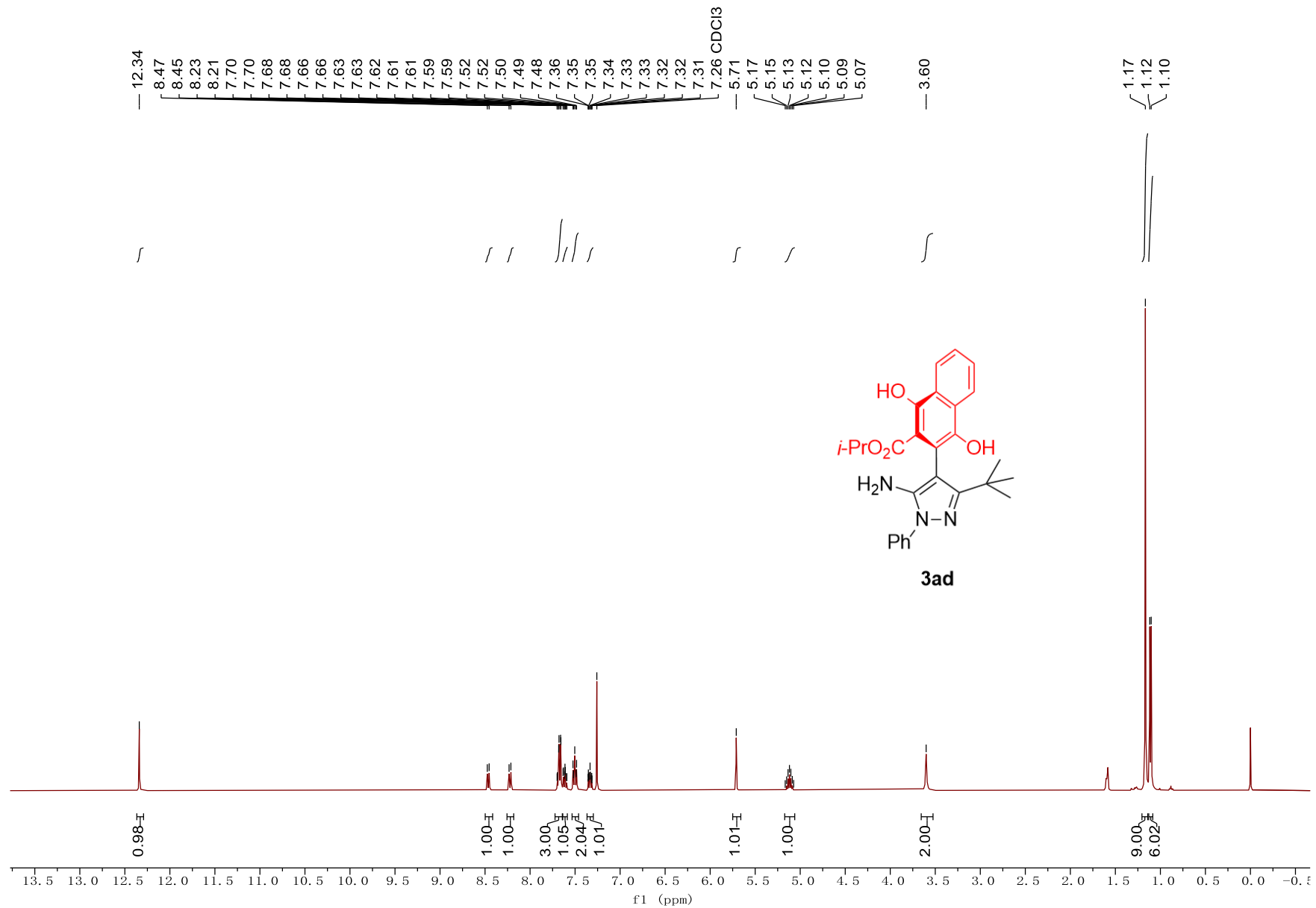


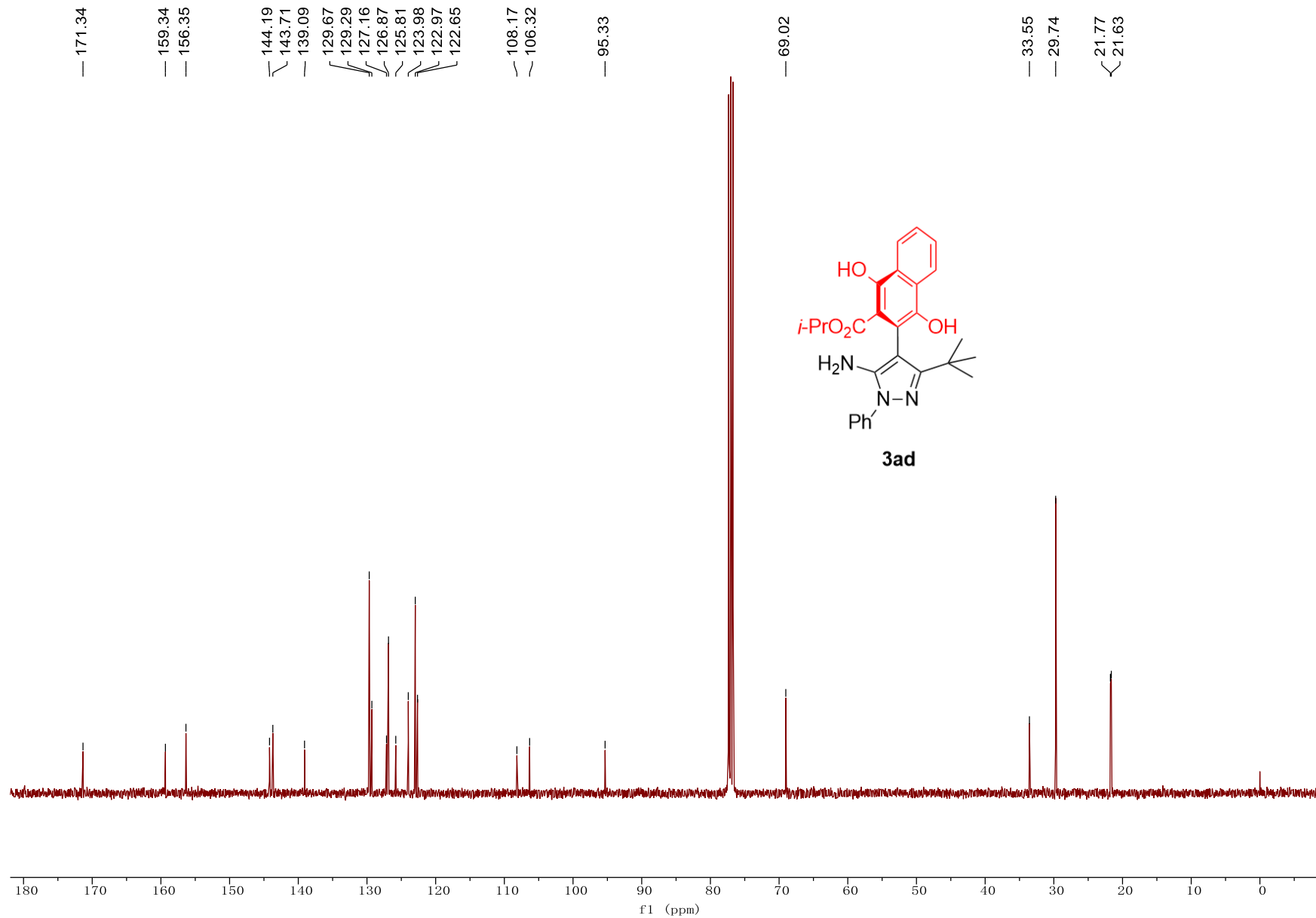


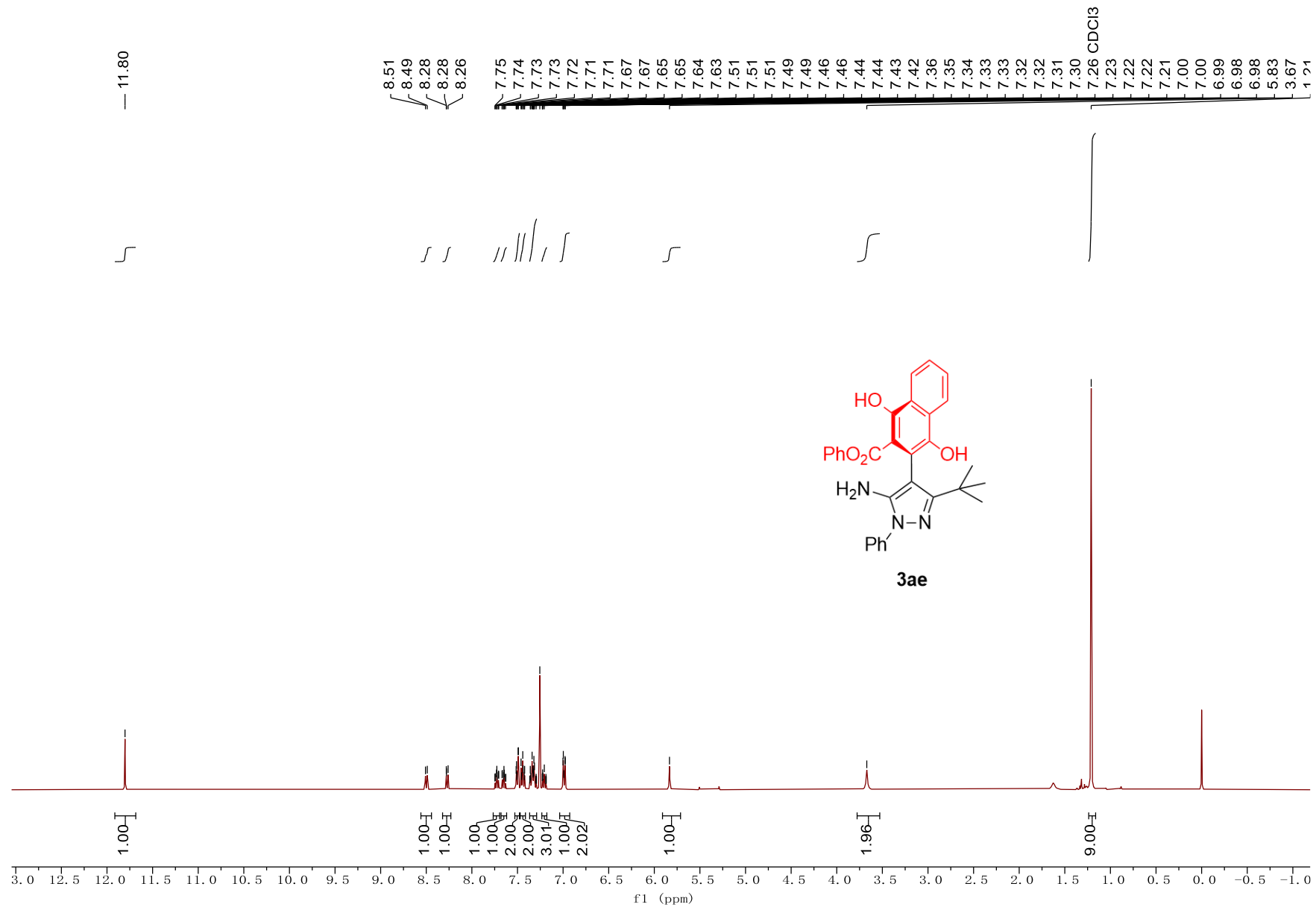


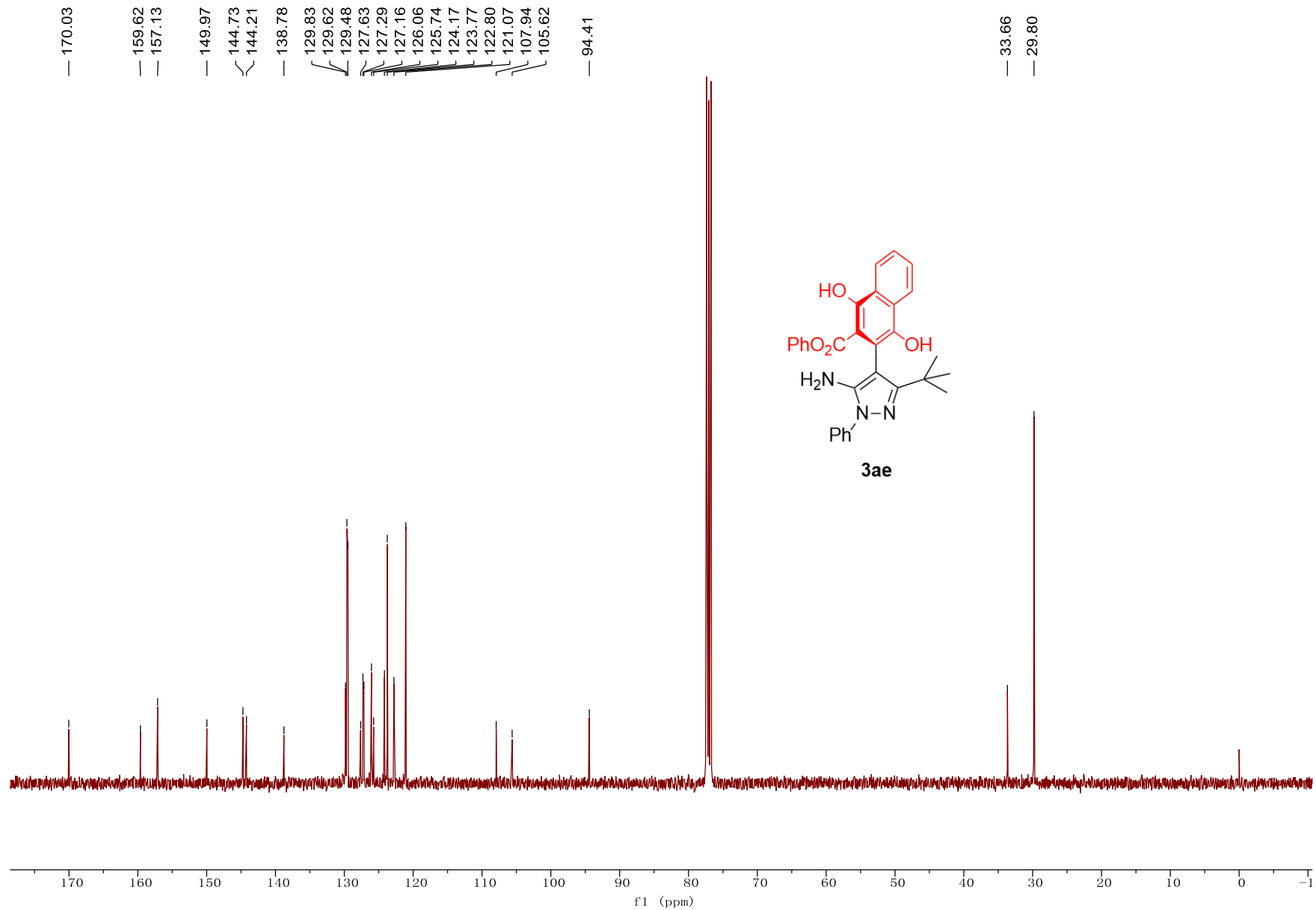


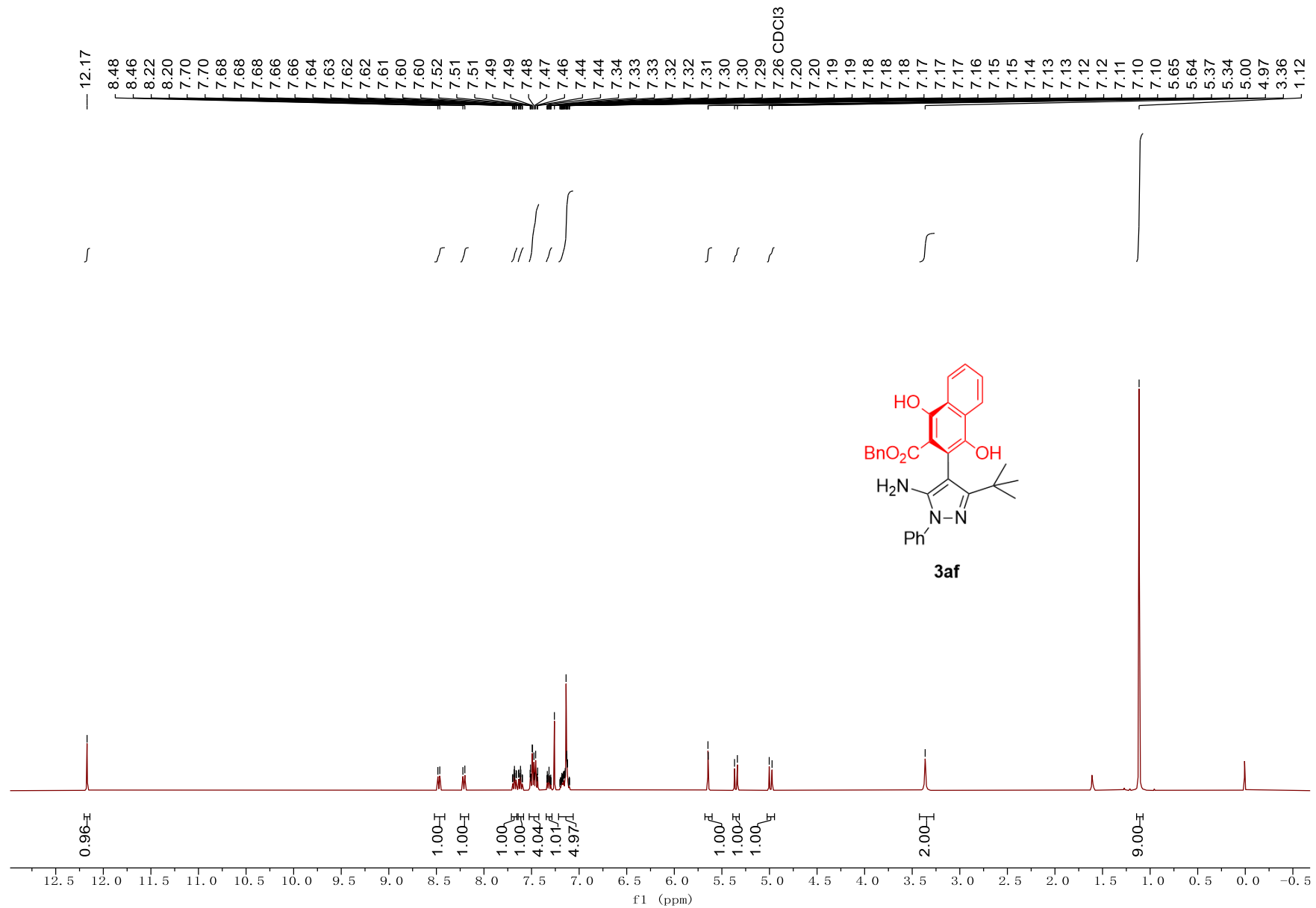


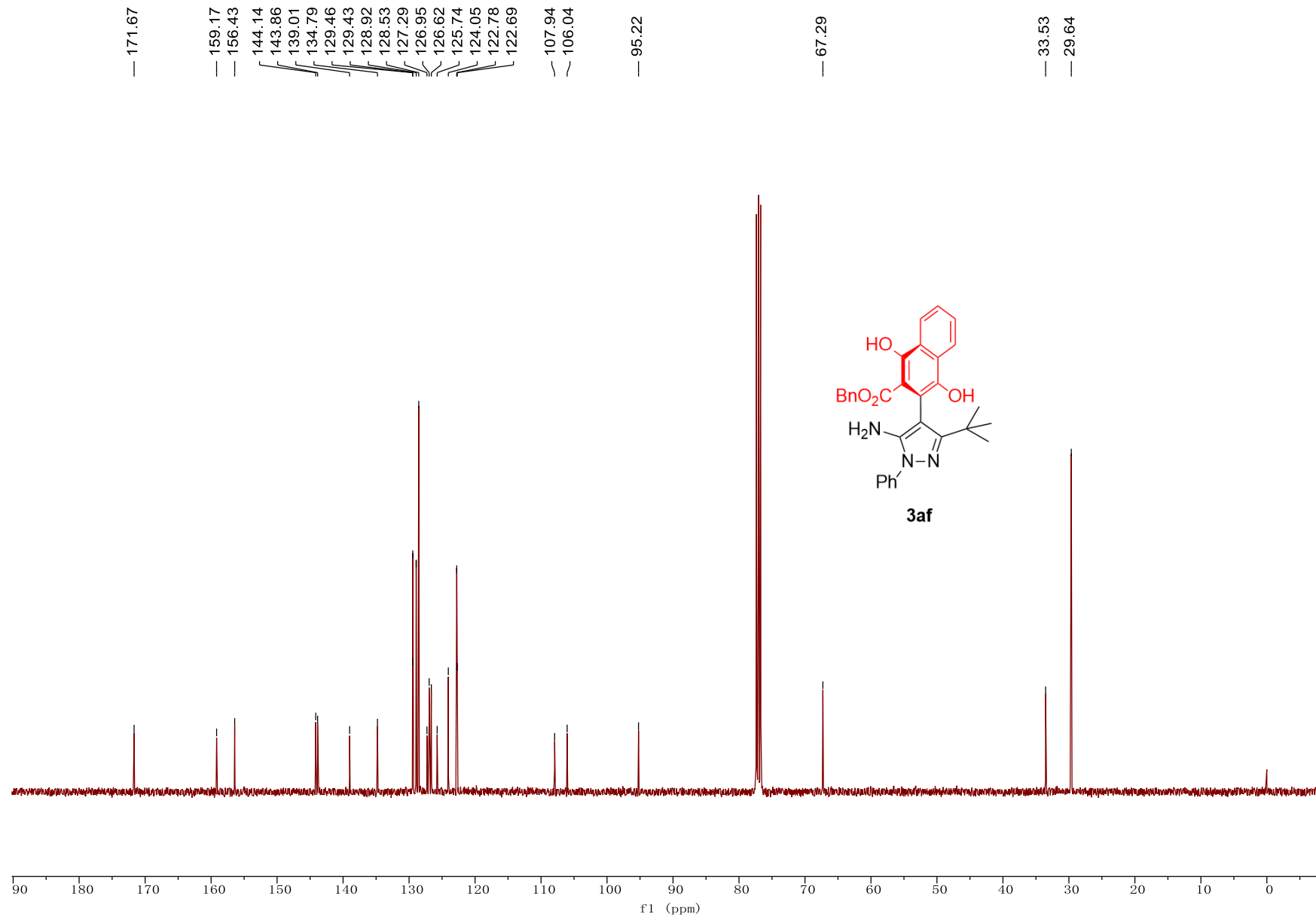












7.91
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7.65
7.64
7.63
7.63
7.62
7.59
7.56
7.54
7.26 CDCl₃



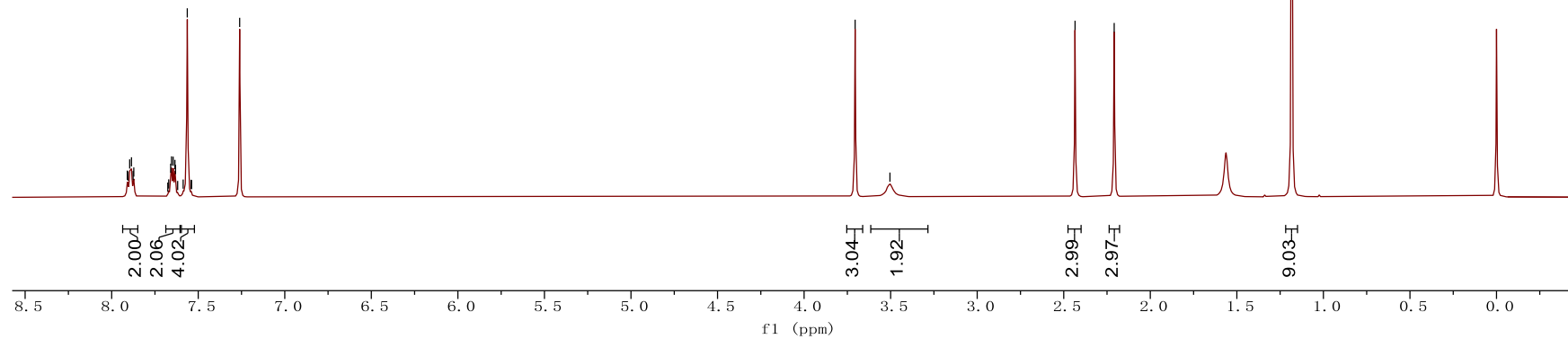
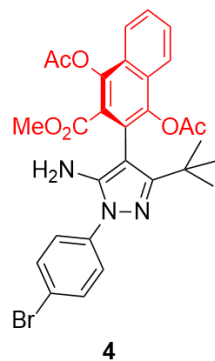
— 3.71
— 3.50

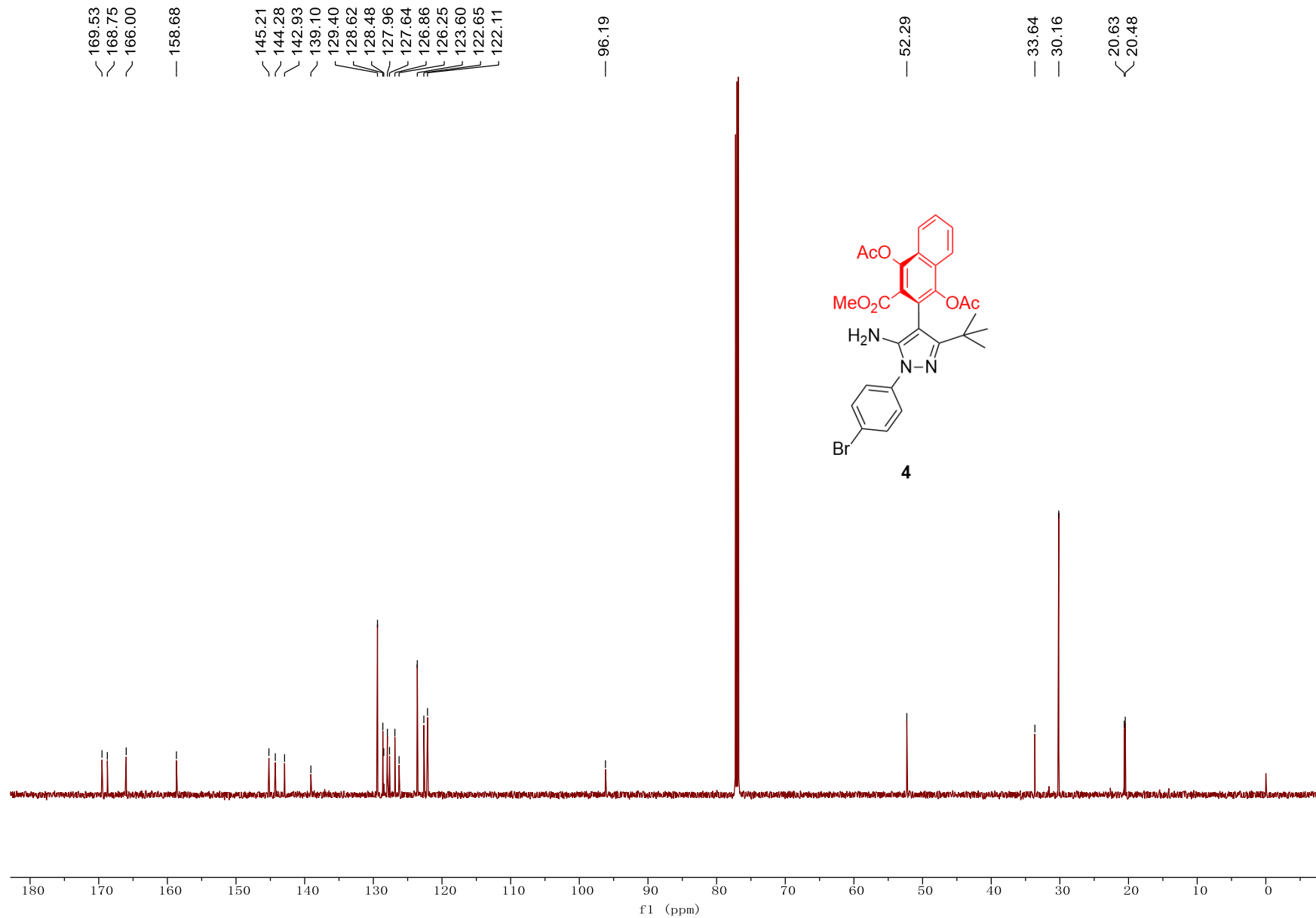


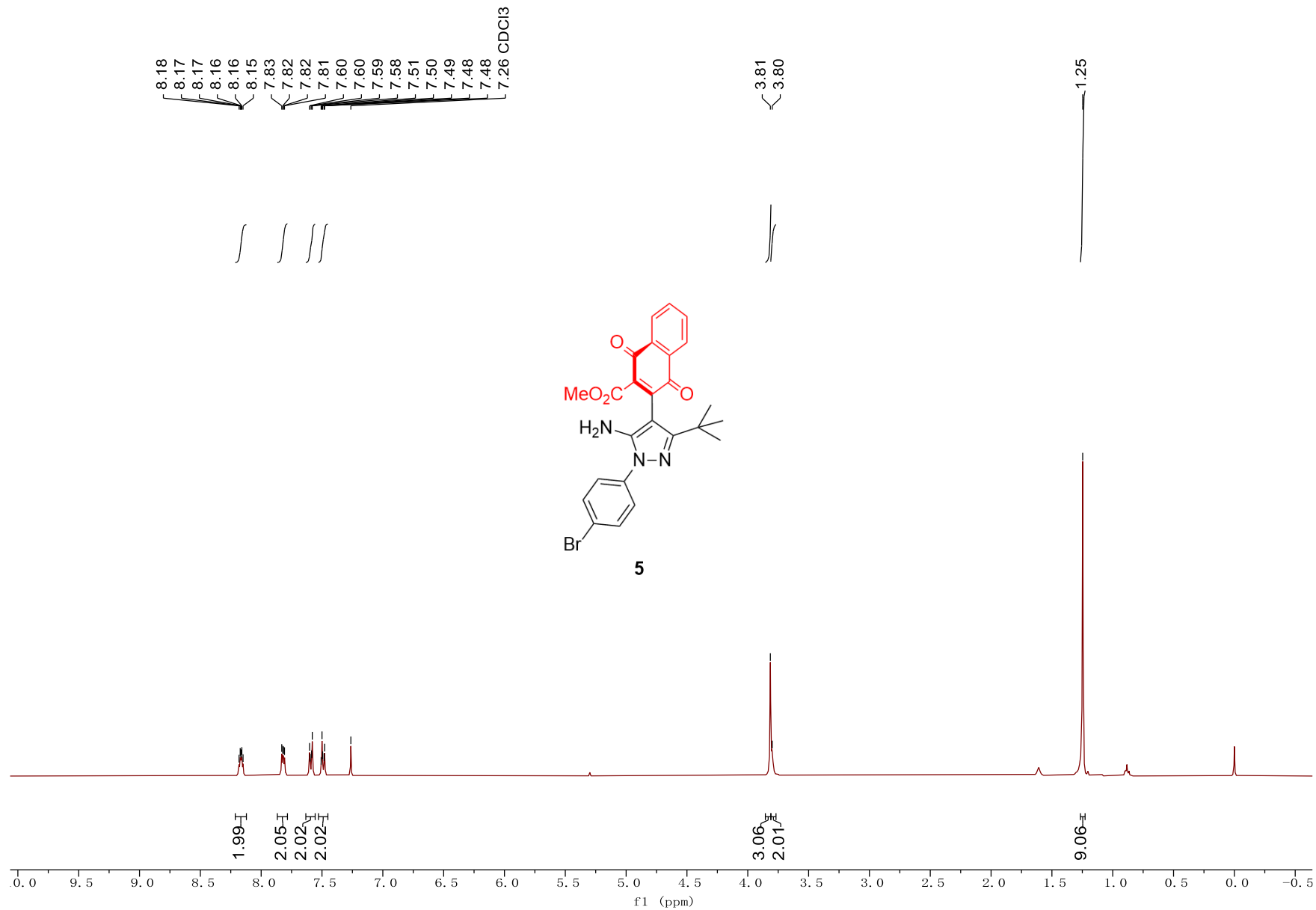
— 2.43
— 2.21

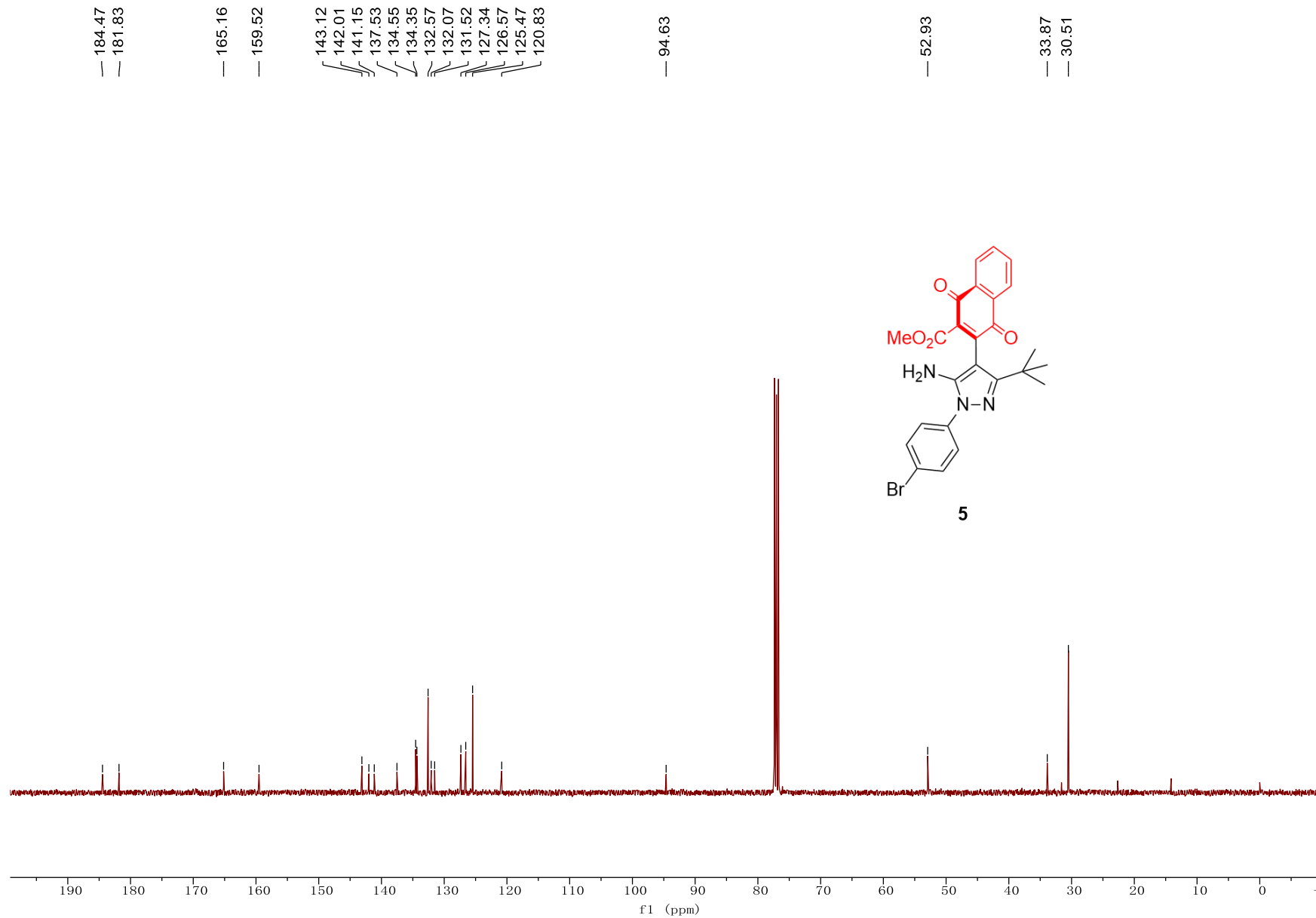


— 1.18









8. X-ray crystal structure of 3ha

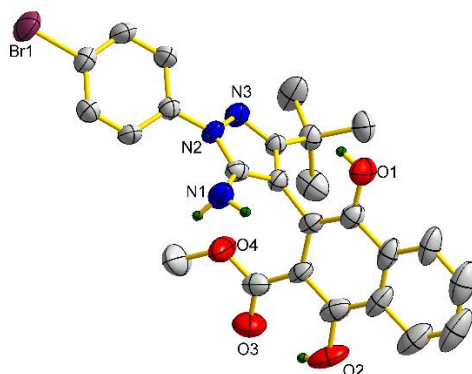


Figure S2. ORTEP diagram of **3ha**. Thermal ellipsoids are shown at 50% probability level. All hydrogen atoms on carbon are omitted for clarity.

CCDC: 2311460

Table 1 Crystal data and structure refinement for 1.

Identification code	1
Empirical formula	C ₂₅ H ₂₄ BrN ₃ O ₄
Formula weight	510.38
Temperature/K	299.0
Crystal system	orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁
a/Å	8.5837(8)
b/Å	16.3187(15)
c/Å	19.3809(17)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	2714.8(4)
Z	4
ρ _{calc} /cm ³	1.249
μ/mm ⁻¹	1.546
F(000)	1048.0
Crystal size/mm ³	0.33 × 0.32 × 0.29
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	4.204 to 52.742
Index ranges	-10 ≤ h ≤ 10, -20 ≤ k ≤ 20, -24 ≤ l ≤ 24
Reflections collected	70426
Independent reflections	5559 [R _{int} = 0.0755, R _{sigma} = 0.0490]
Data/restraints/parameters	5559/0/306
Goodness-of-fit on F ²	1.020
Final R indexes [I > 2σ (I)]	R ₁ = 0.0539, wR ₂ = 0.1205
Final R indexes [all data]	R ₁ = 0.0944, wR ₂ = 0.1364
Largest diff. peak/hole / e Å ⁻³	0.33/-0.44
Flack parameter	0.041(16)