

Enantioselective synthesis of novel pyranopyrazoles and pyranocoumarins using a quinine-derived primary amine salt catalyst

Sai Yang, Liu-lan Shen, Yoon-Jung Kim and Jin-Hyun Jeong*

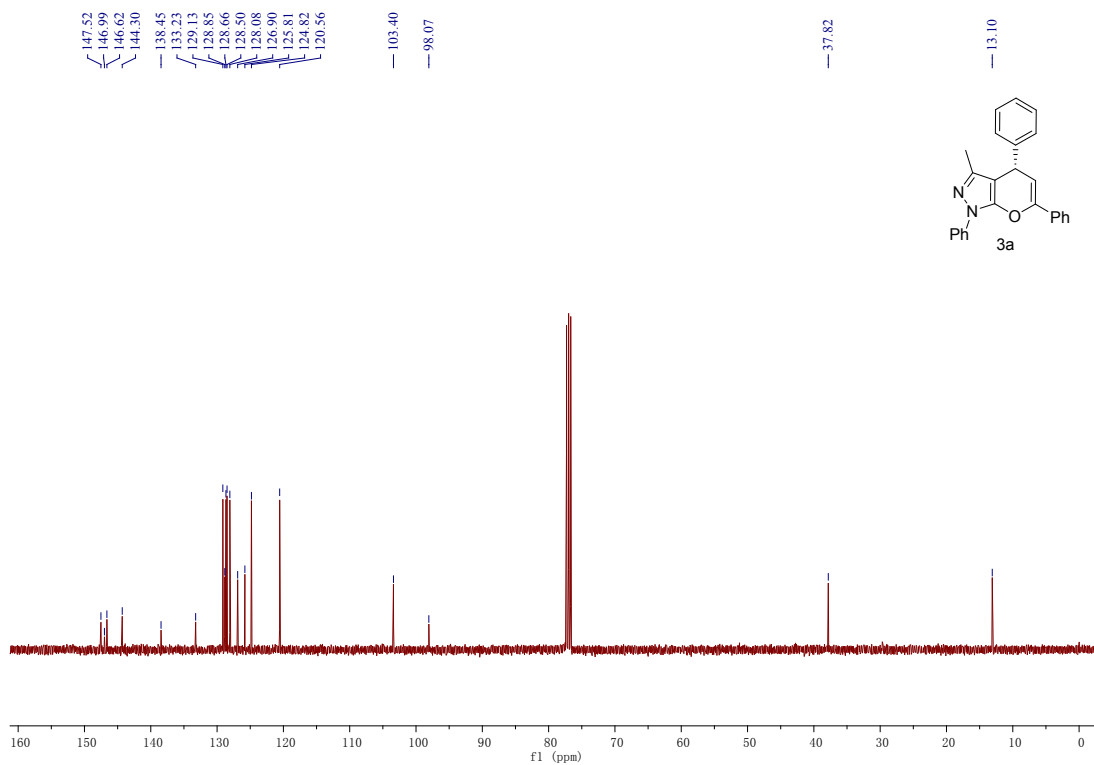
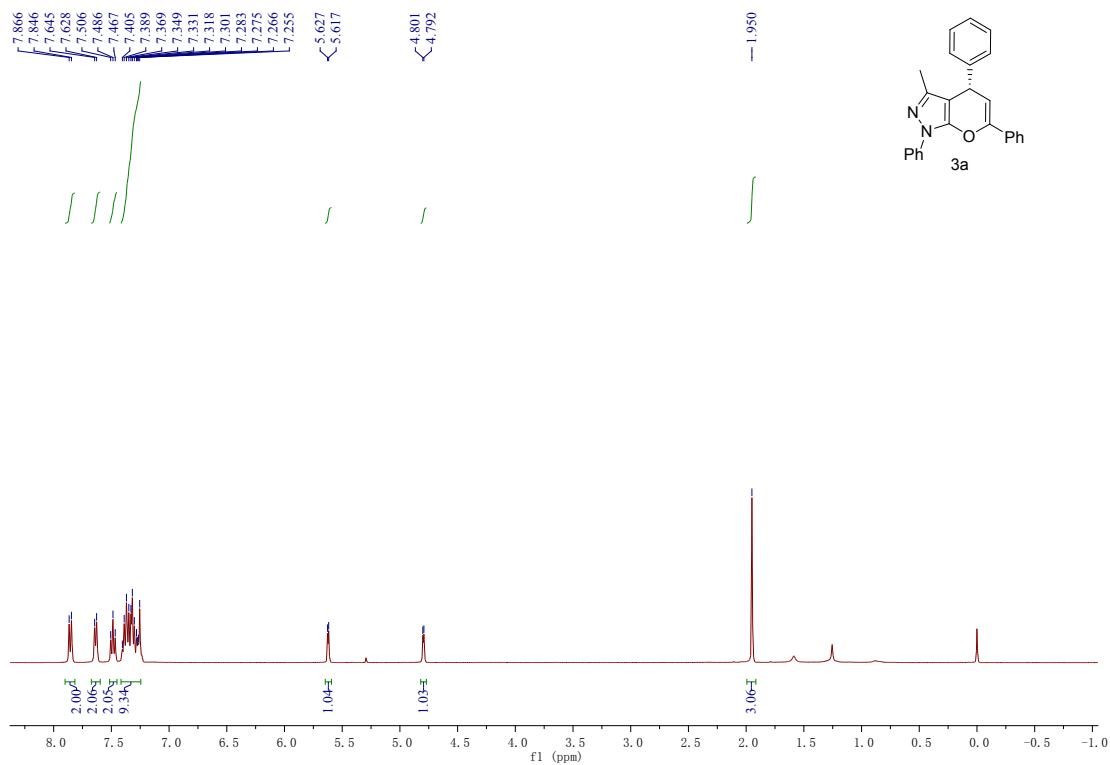
*College of Pharmacy and Yonsei Institute of Pharmaceutical Sciences, Yonsei University
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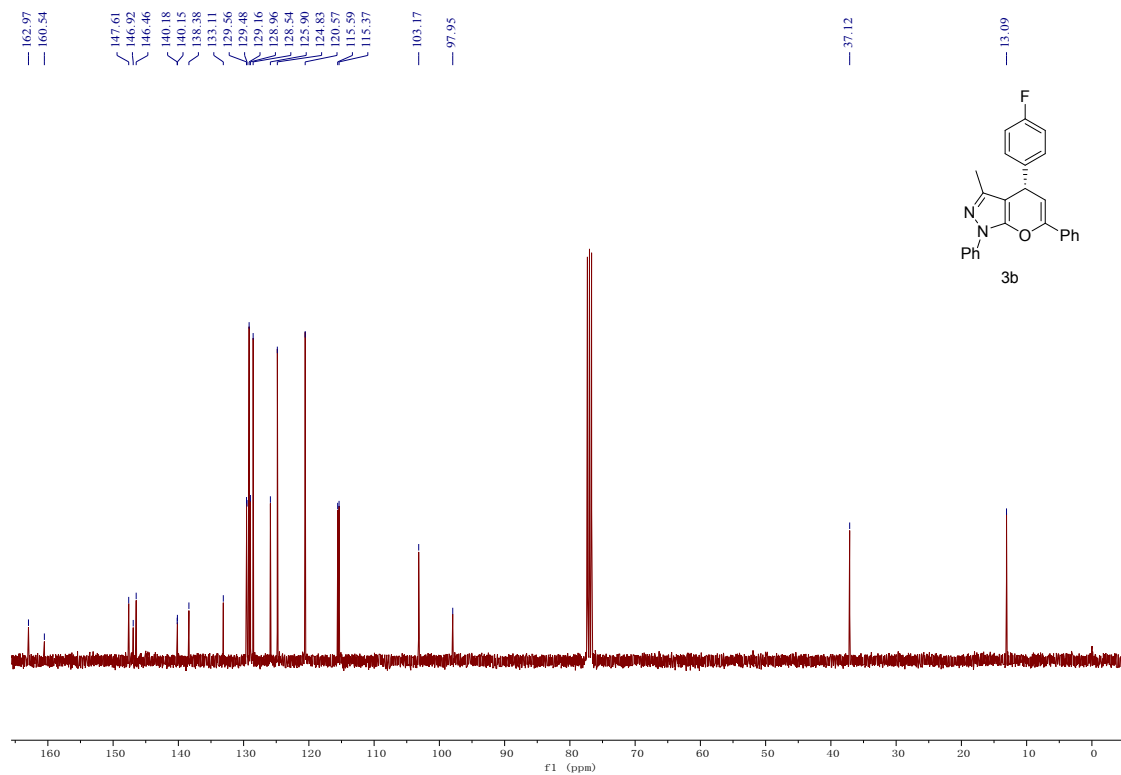
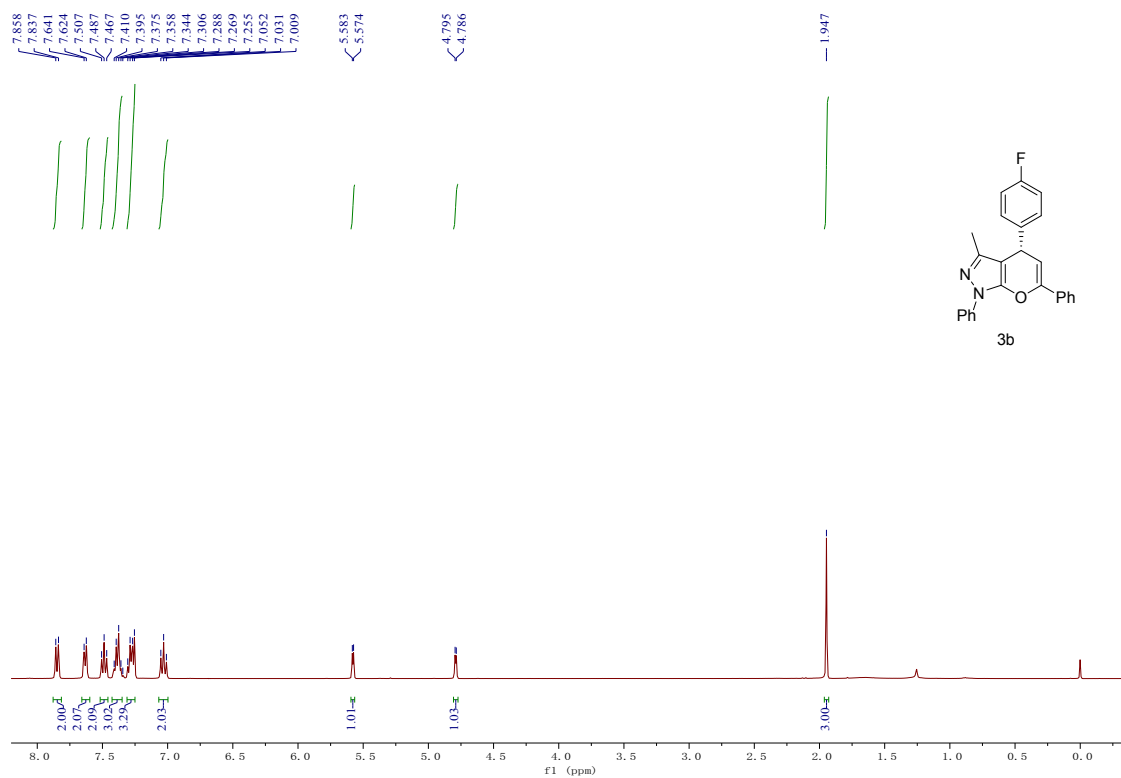
Supporting Information

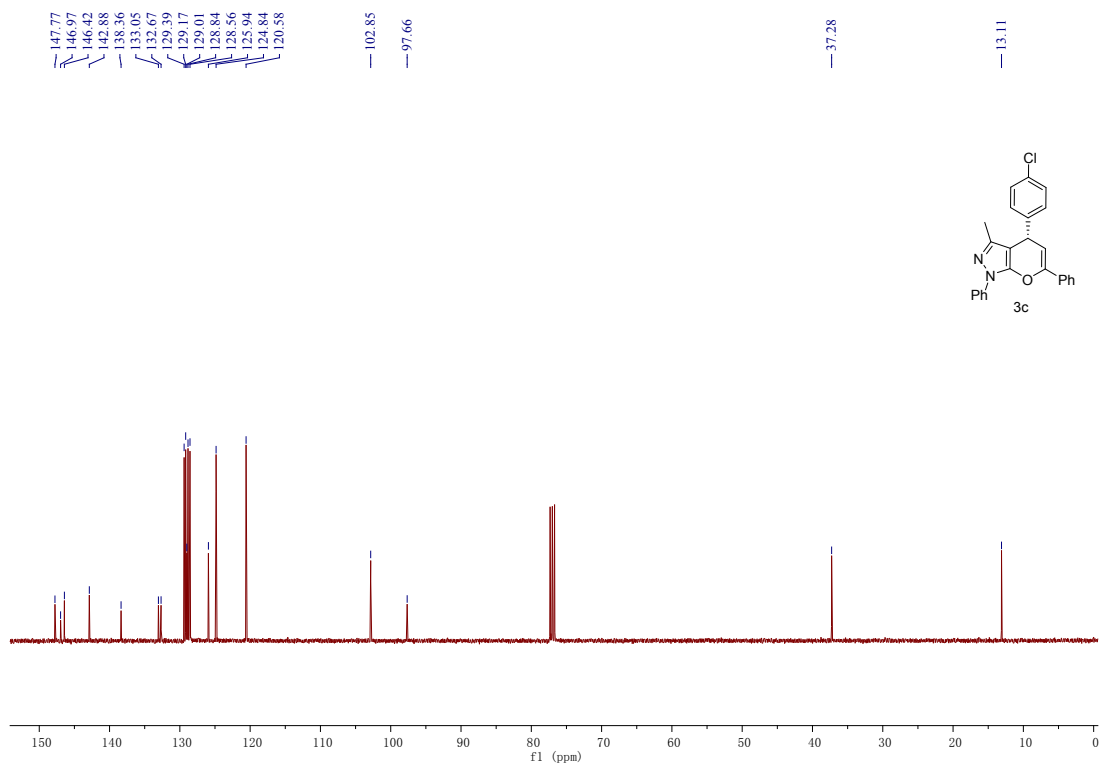
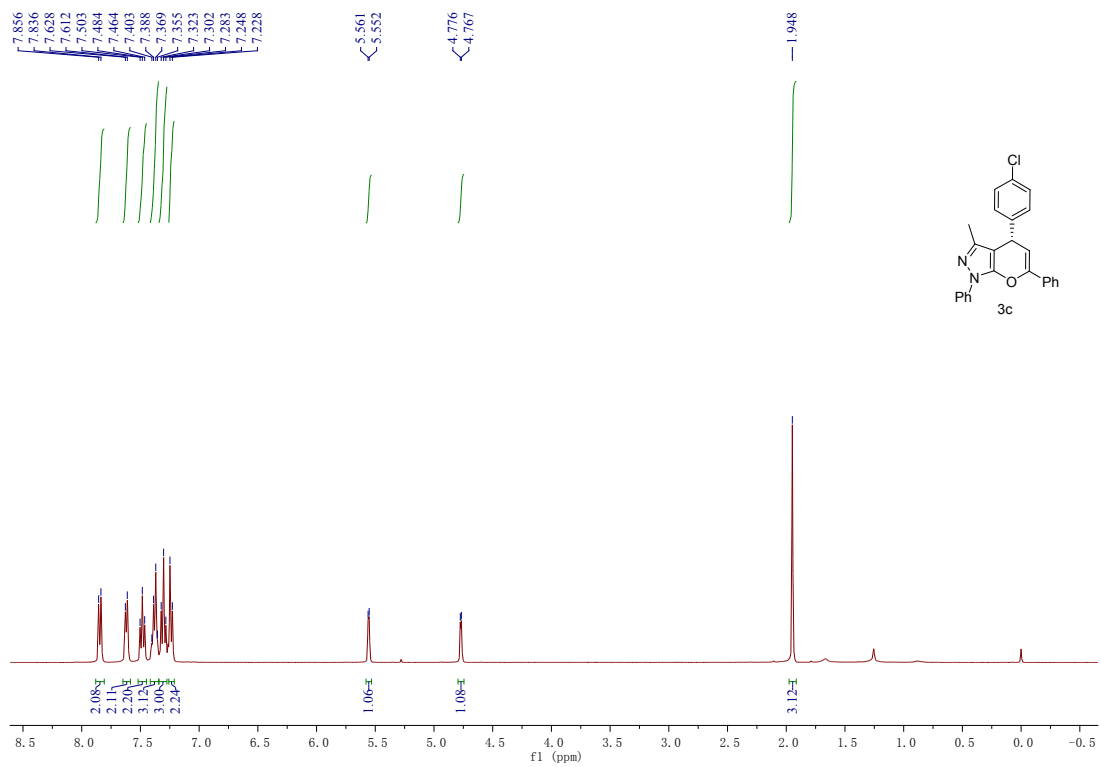
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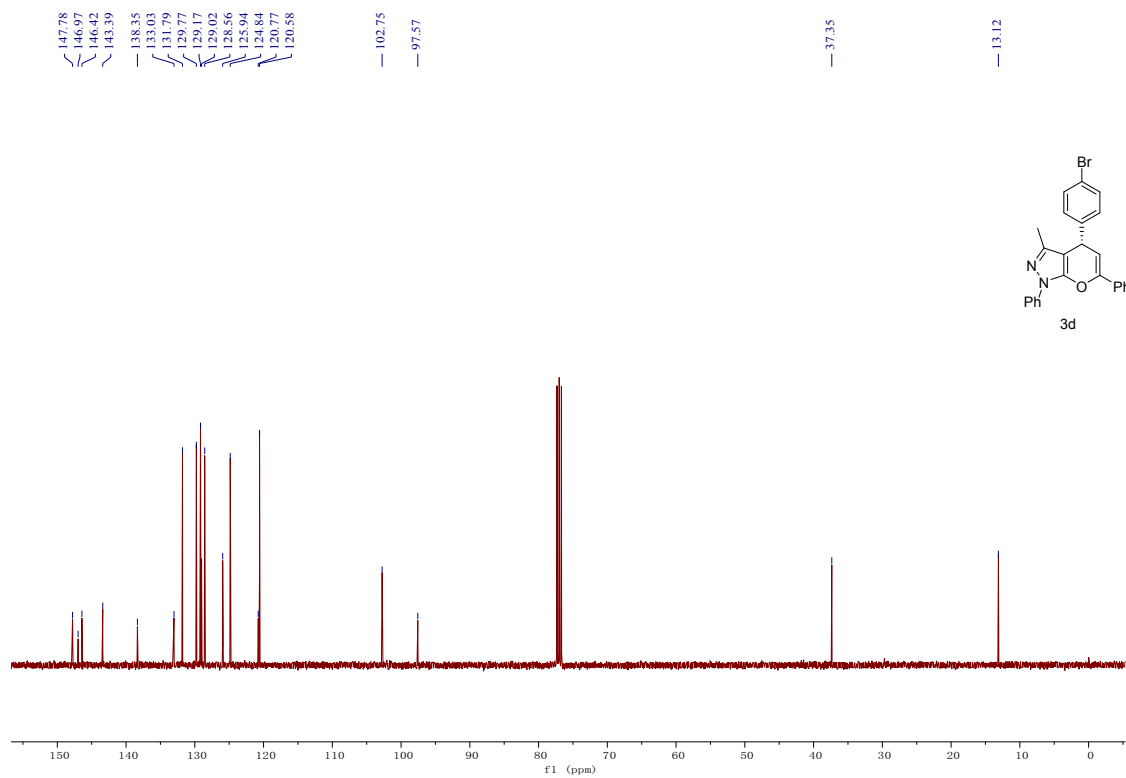
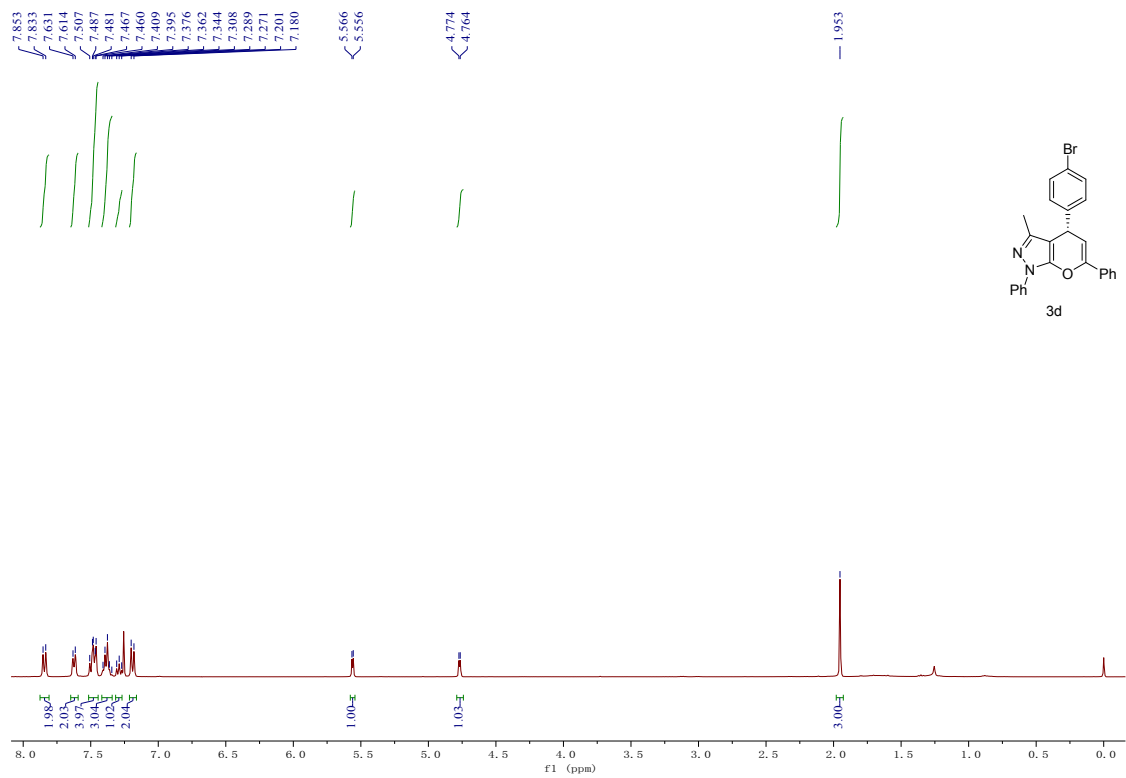
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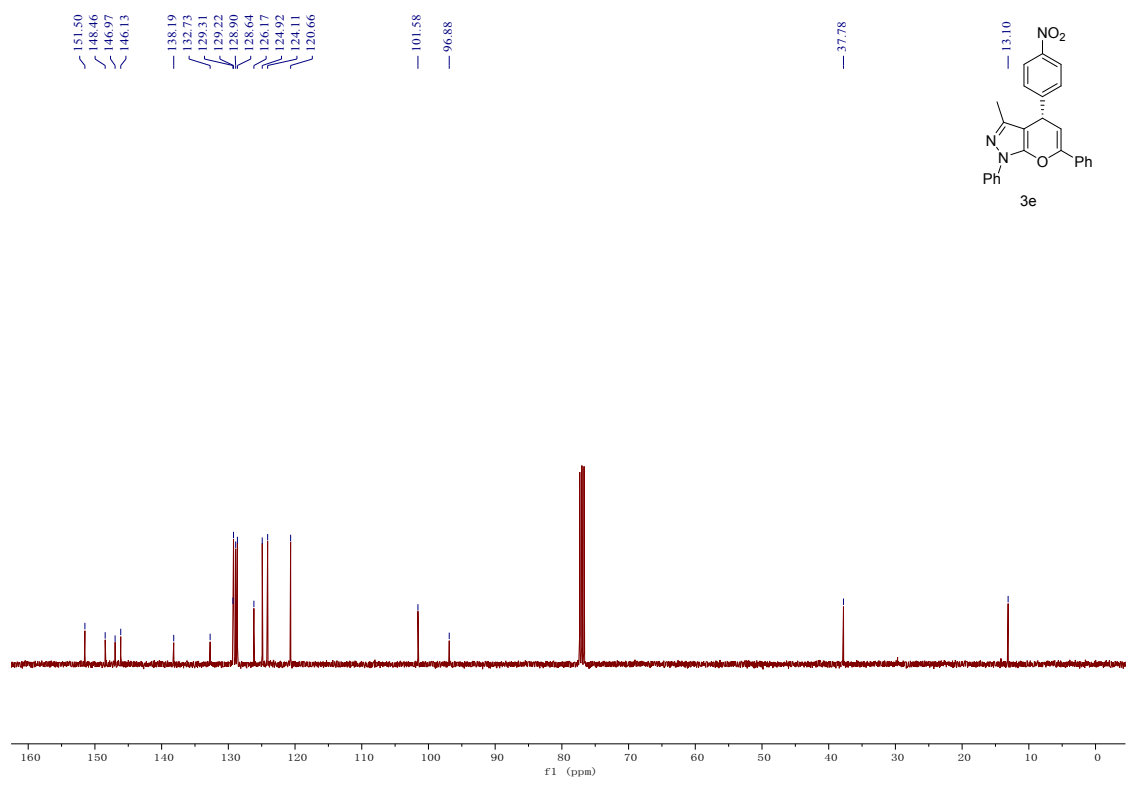
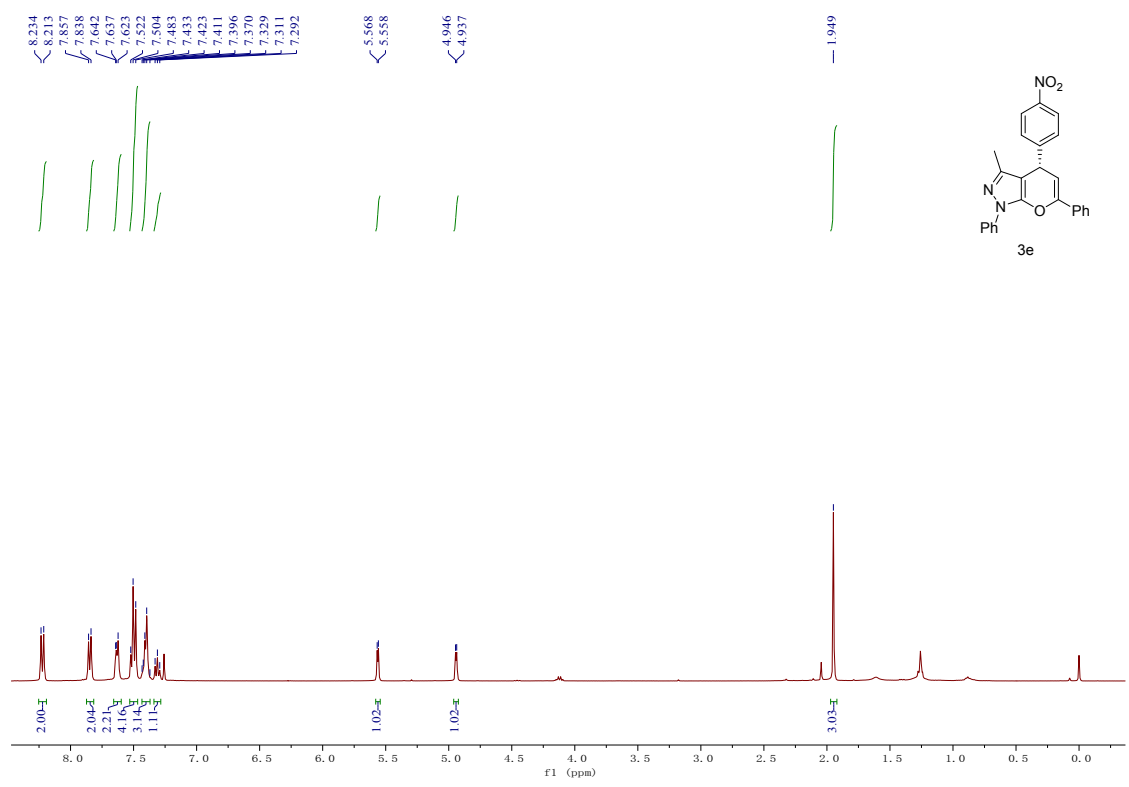
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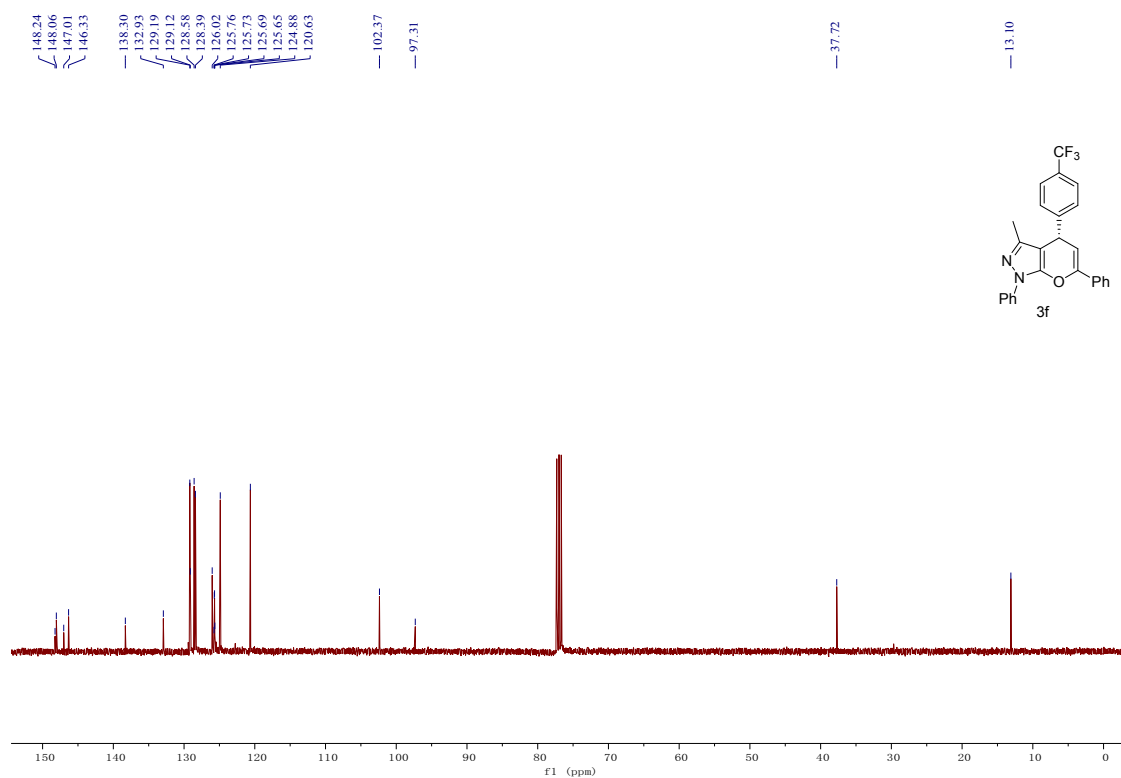
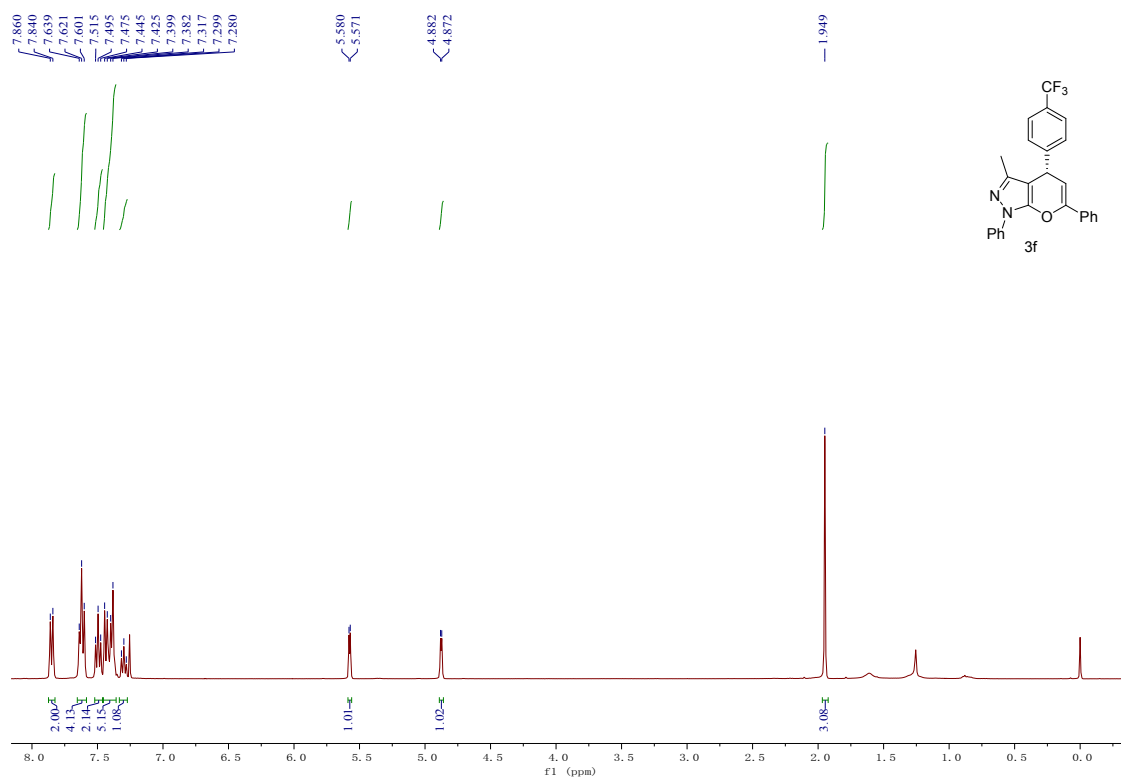


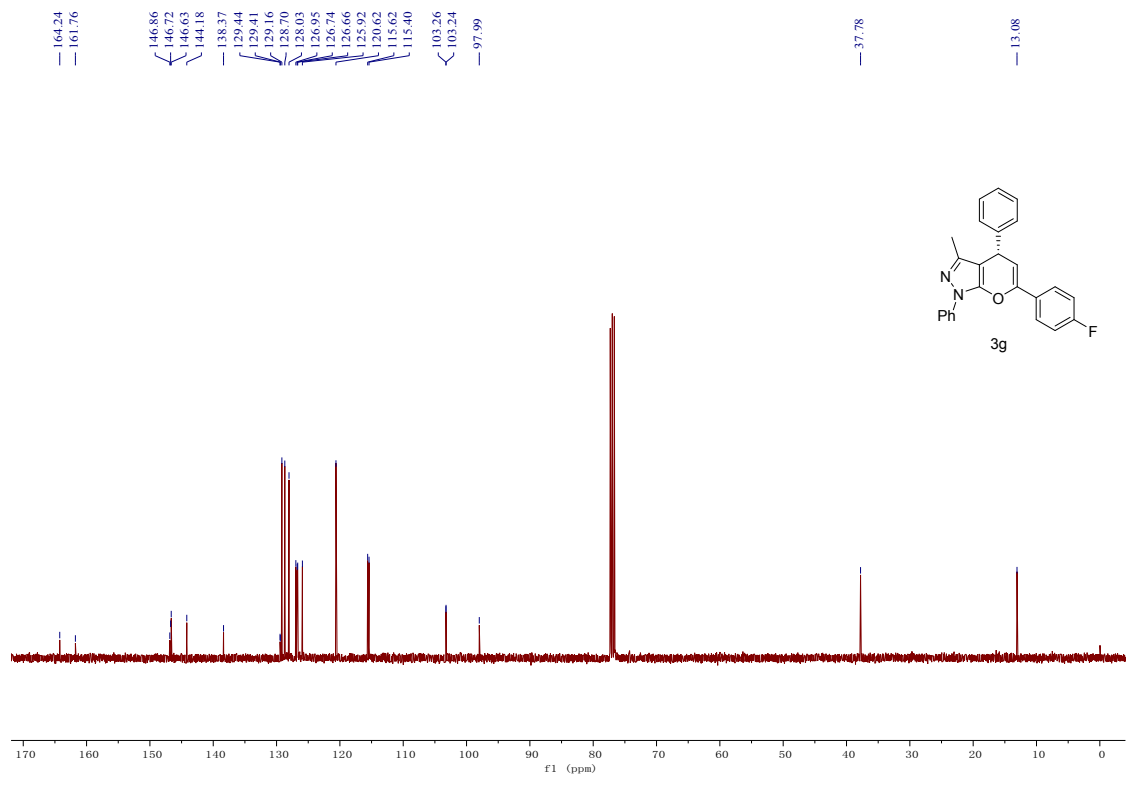
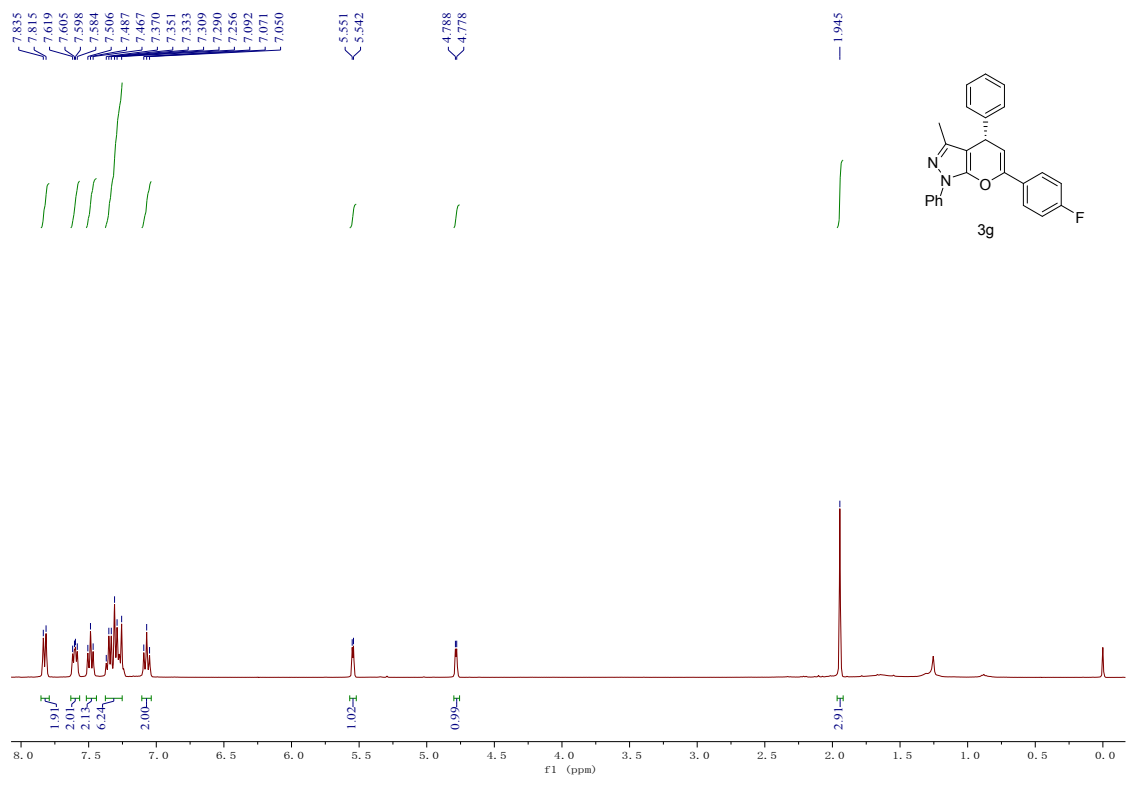


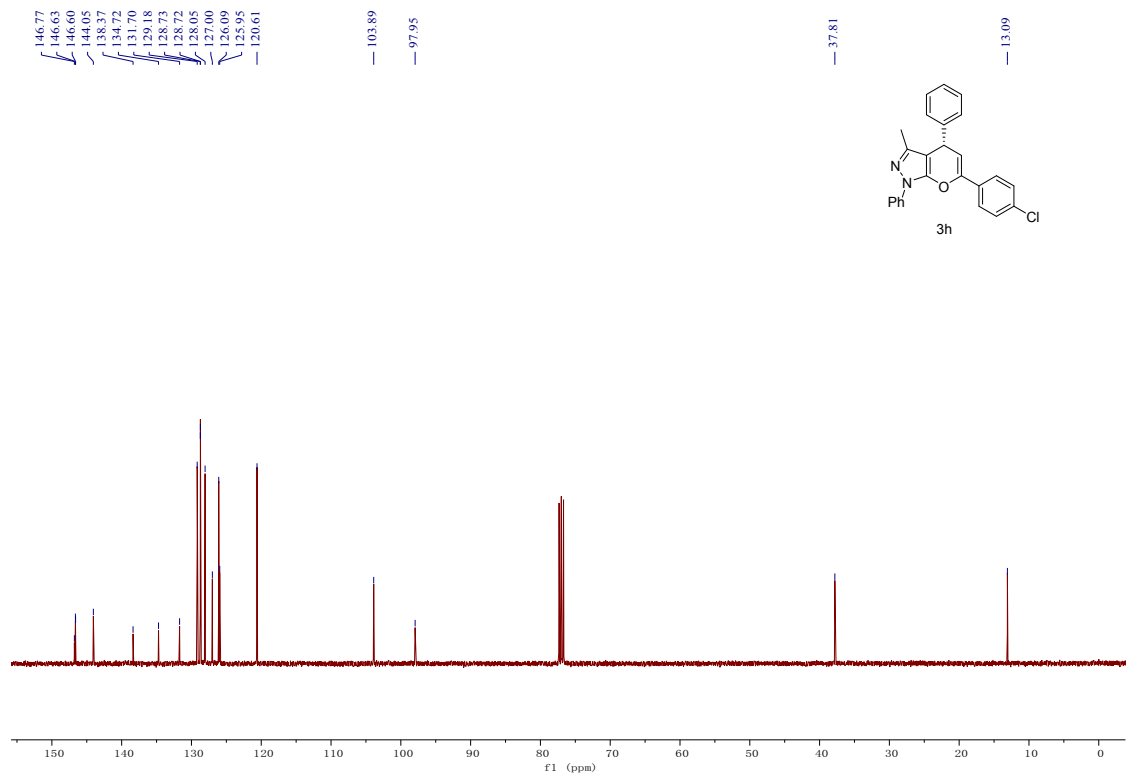
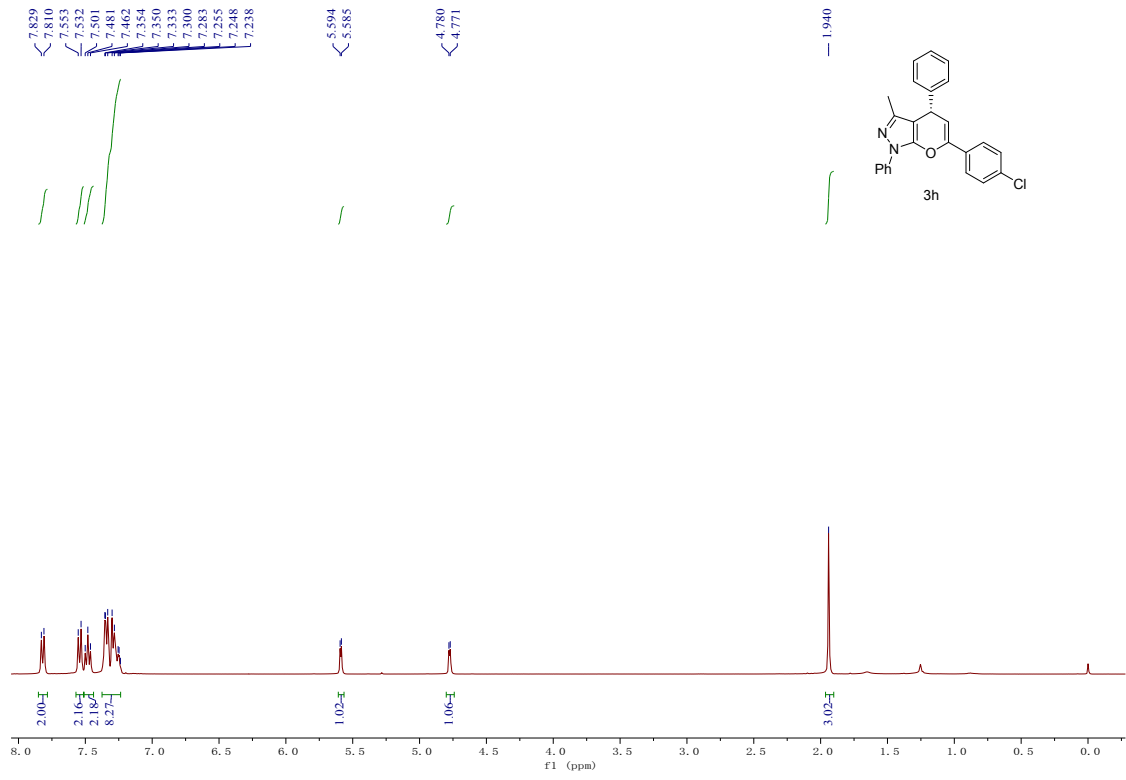


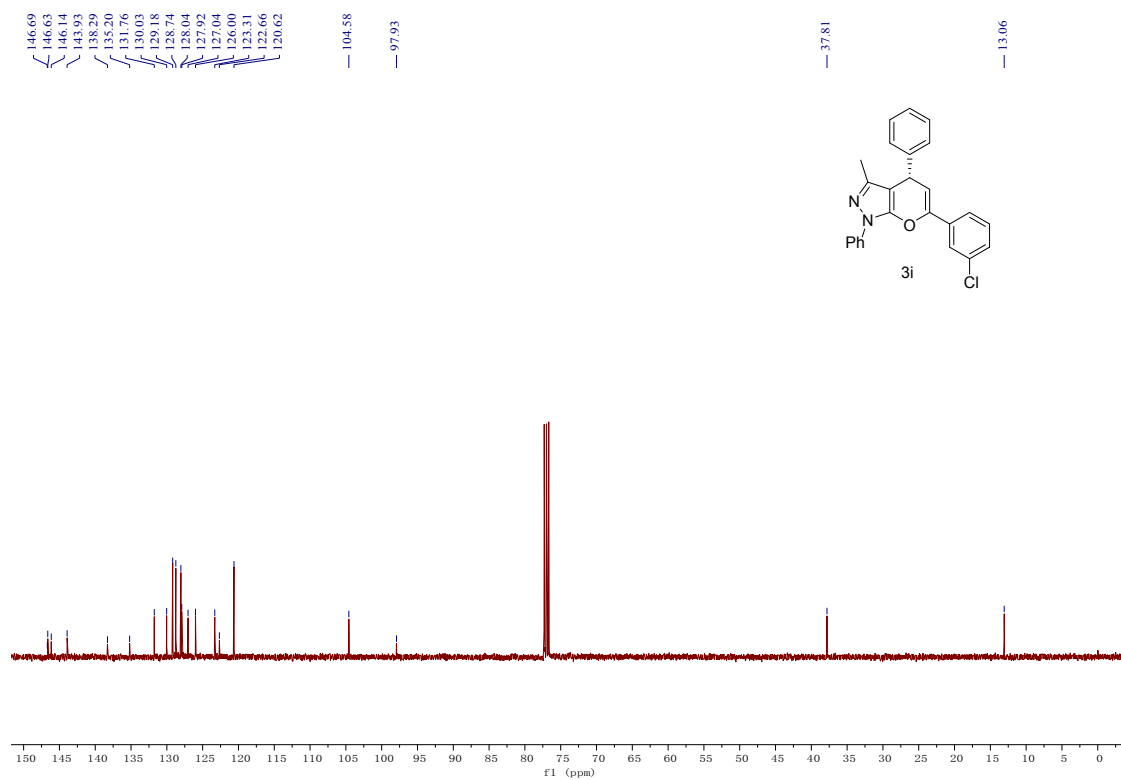
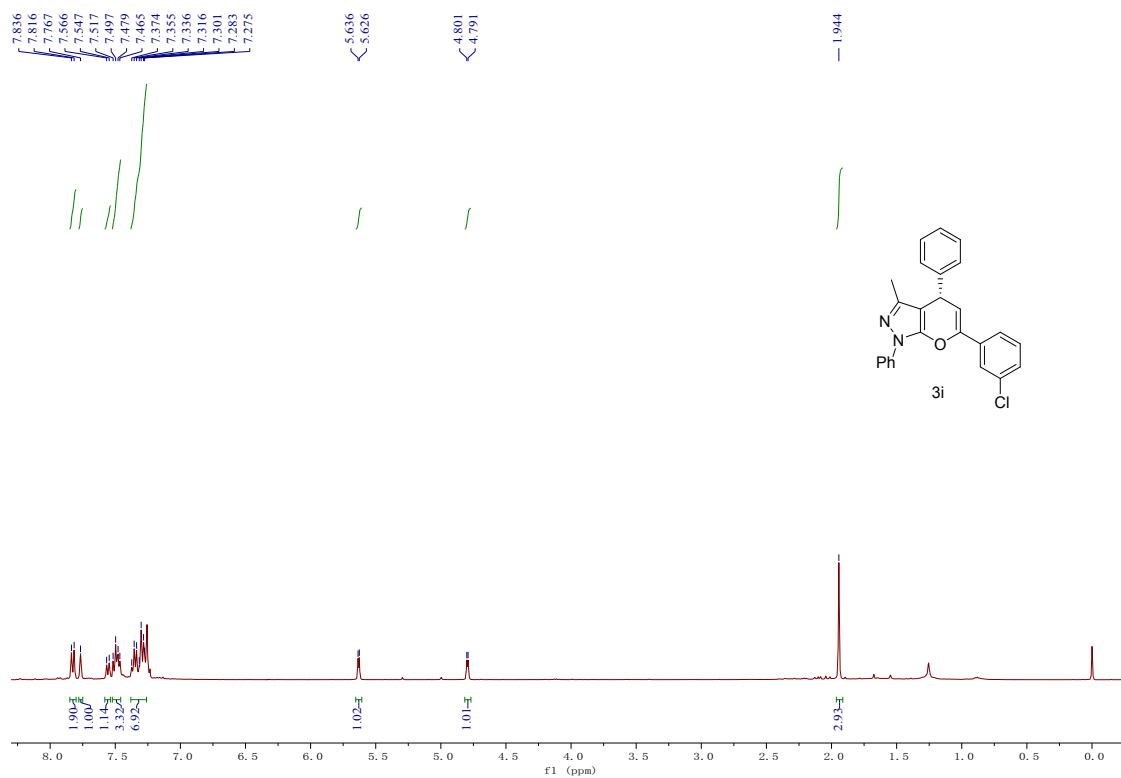


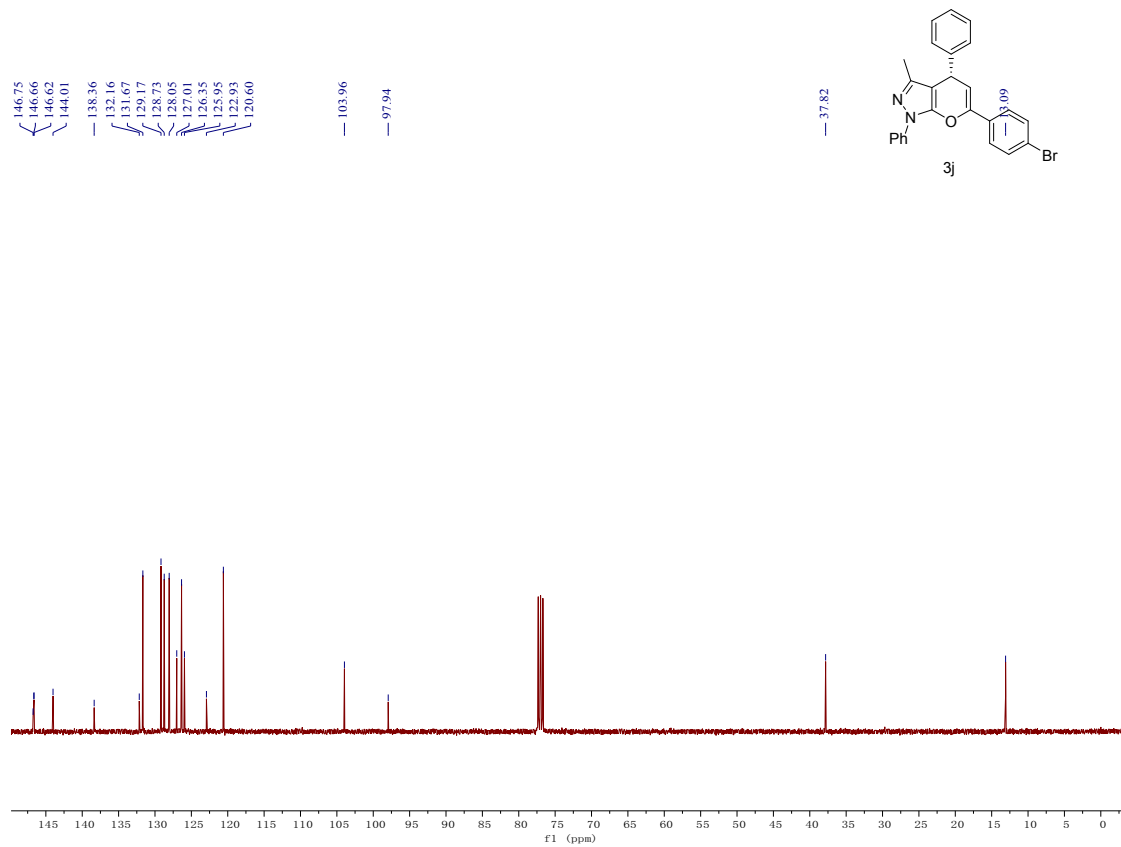
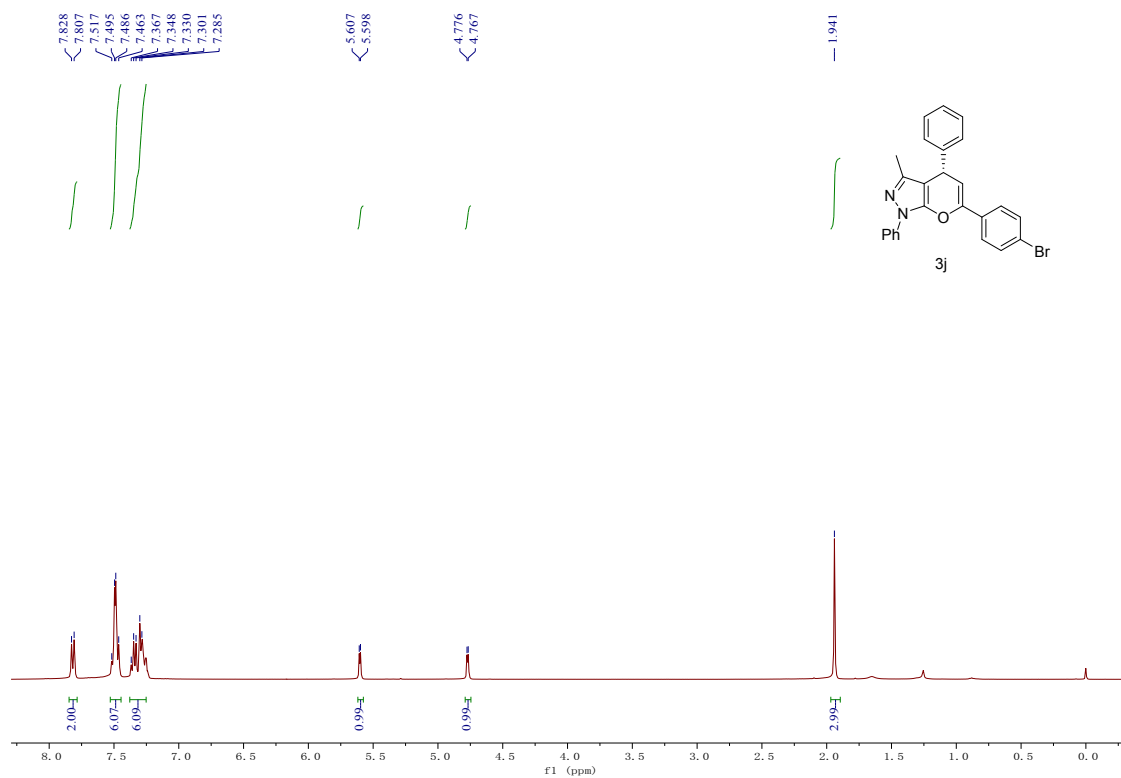


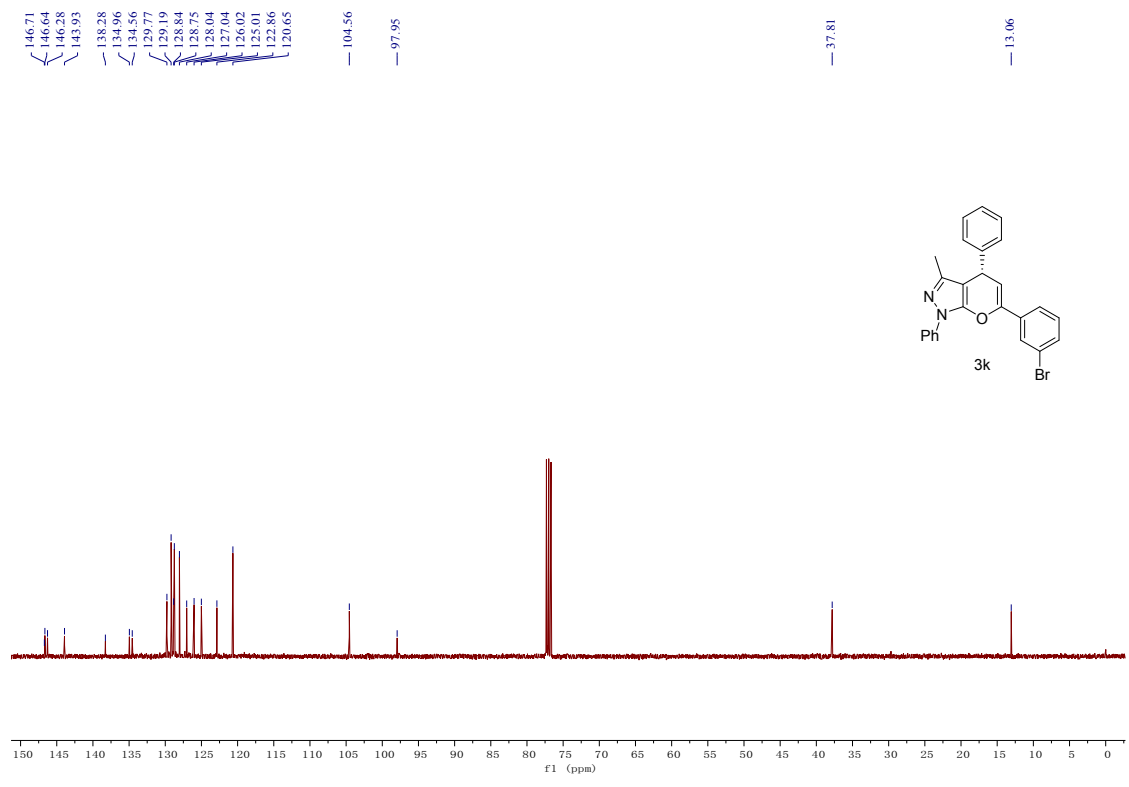
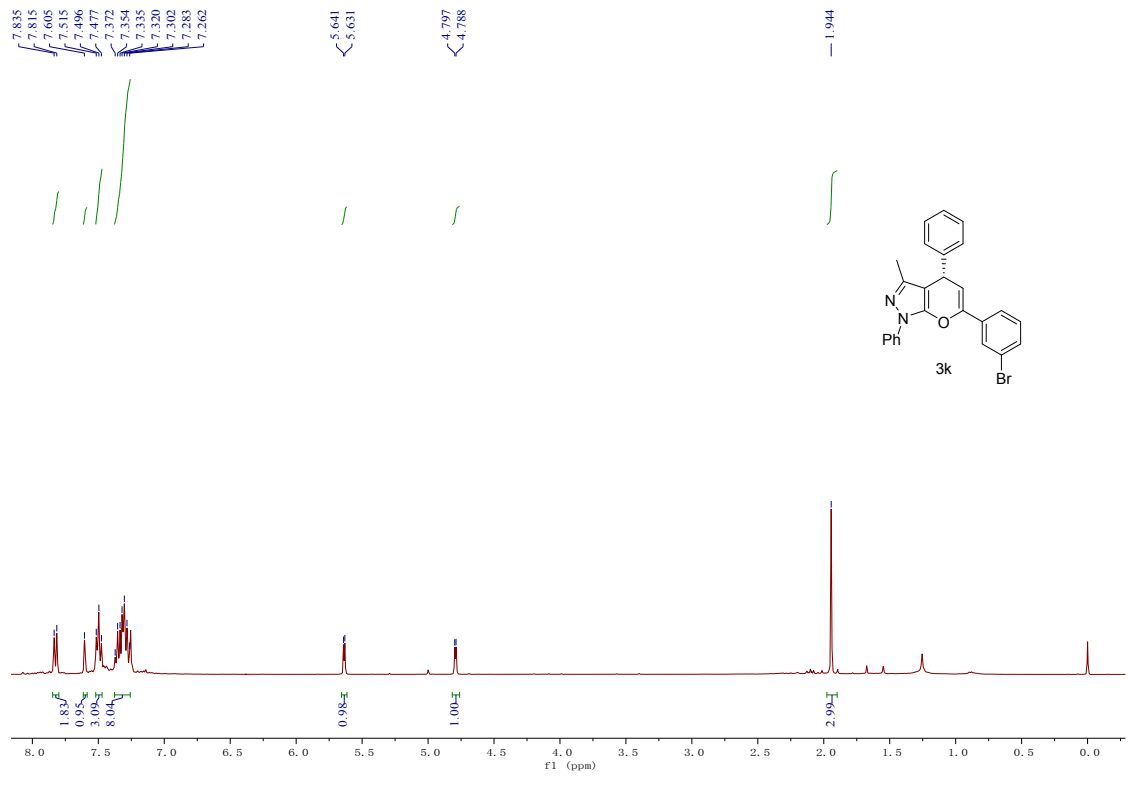


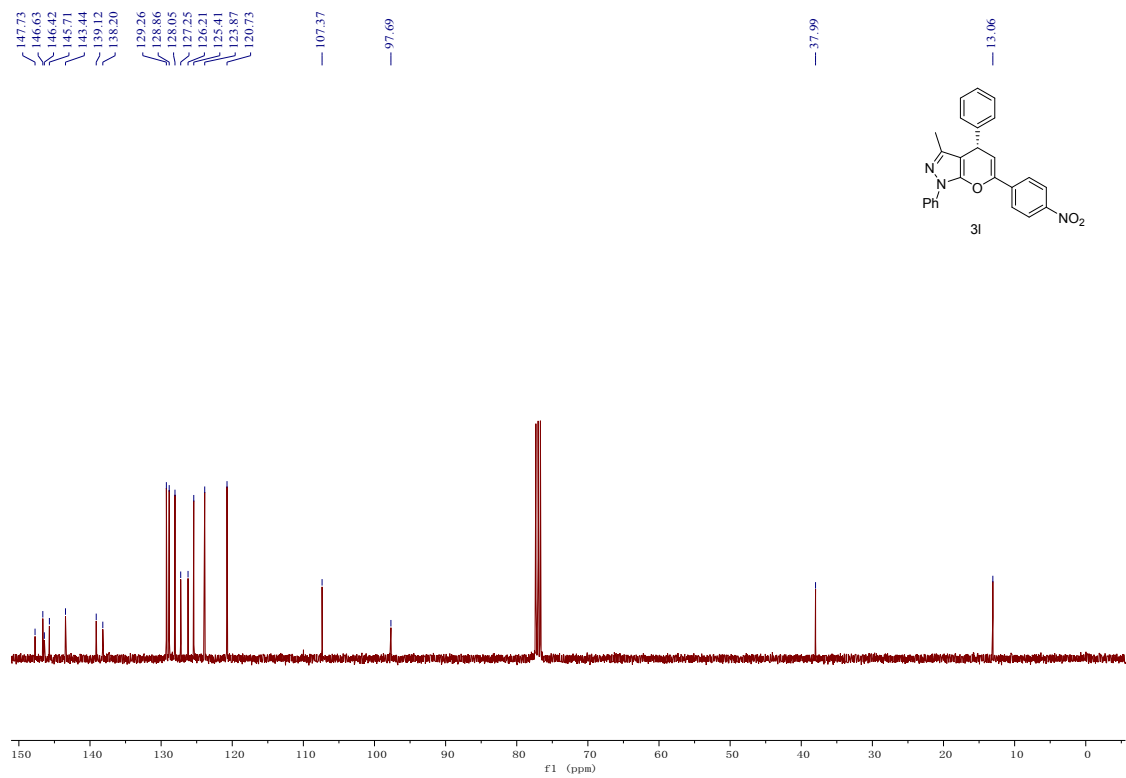
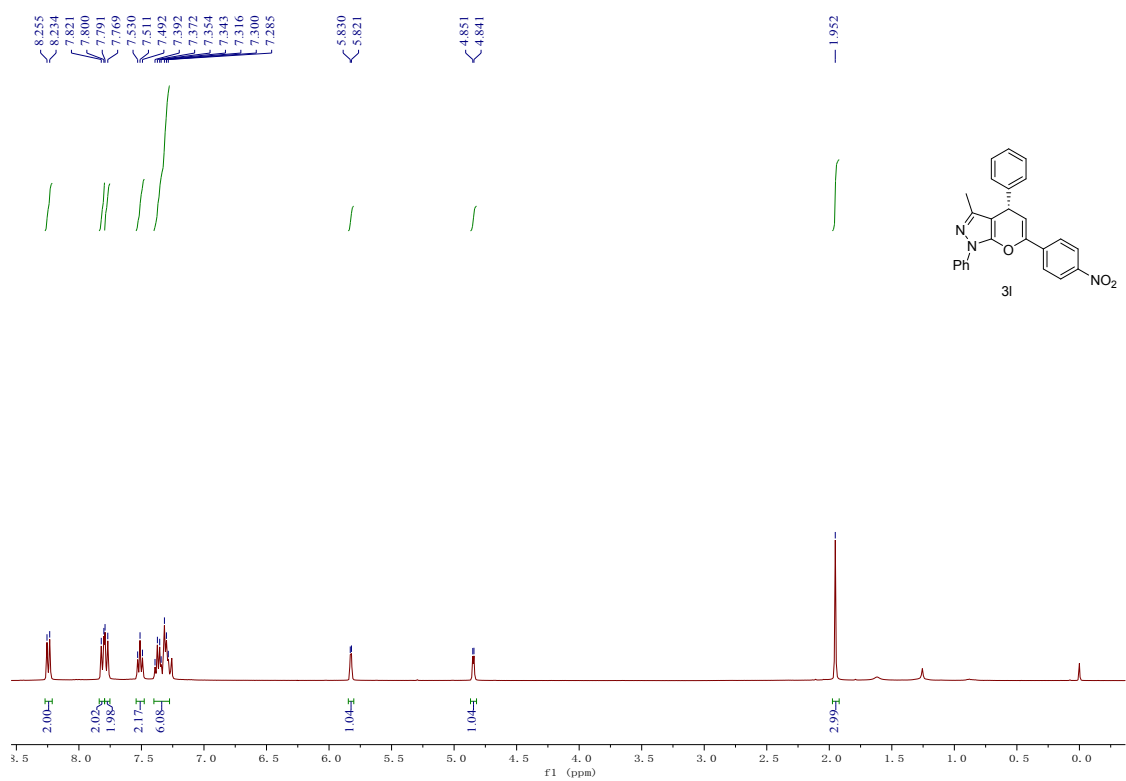


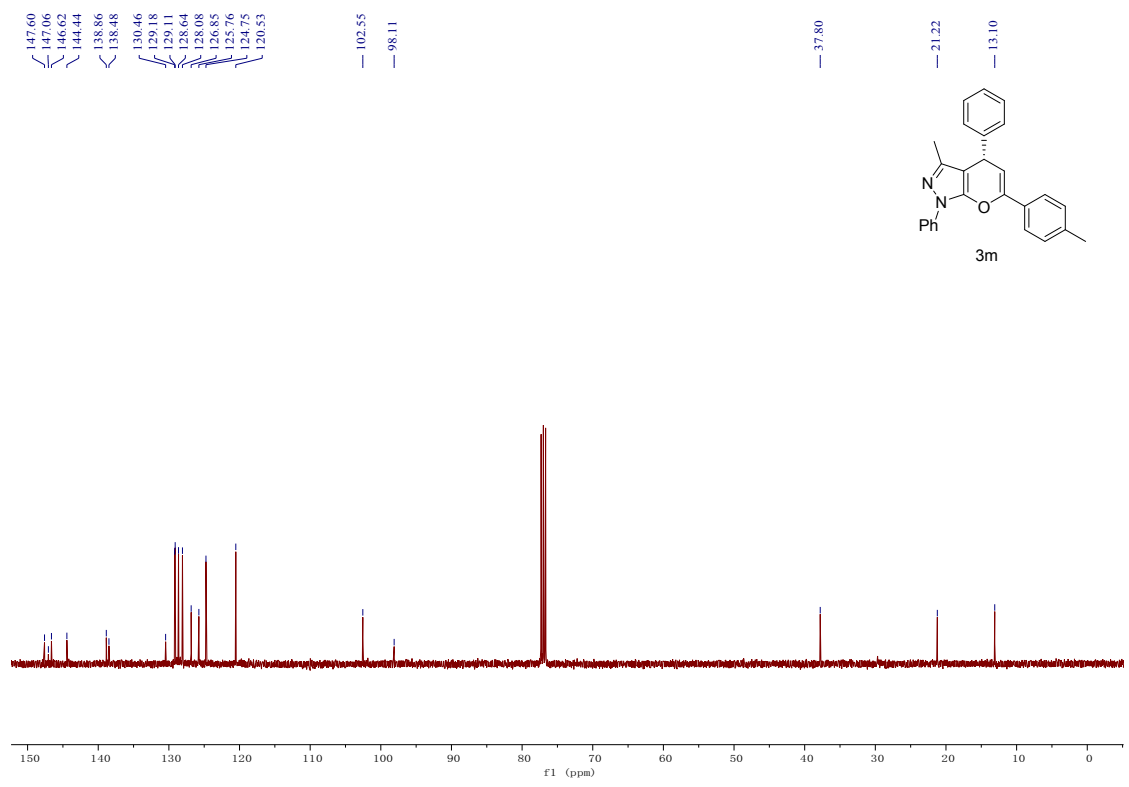
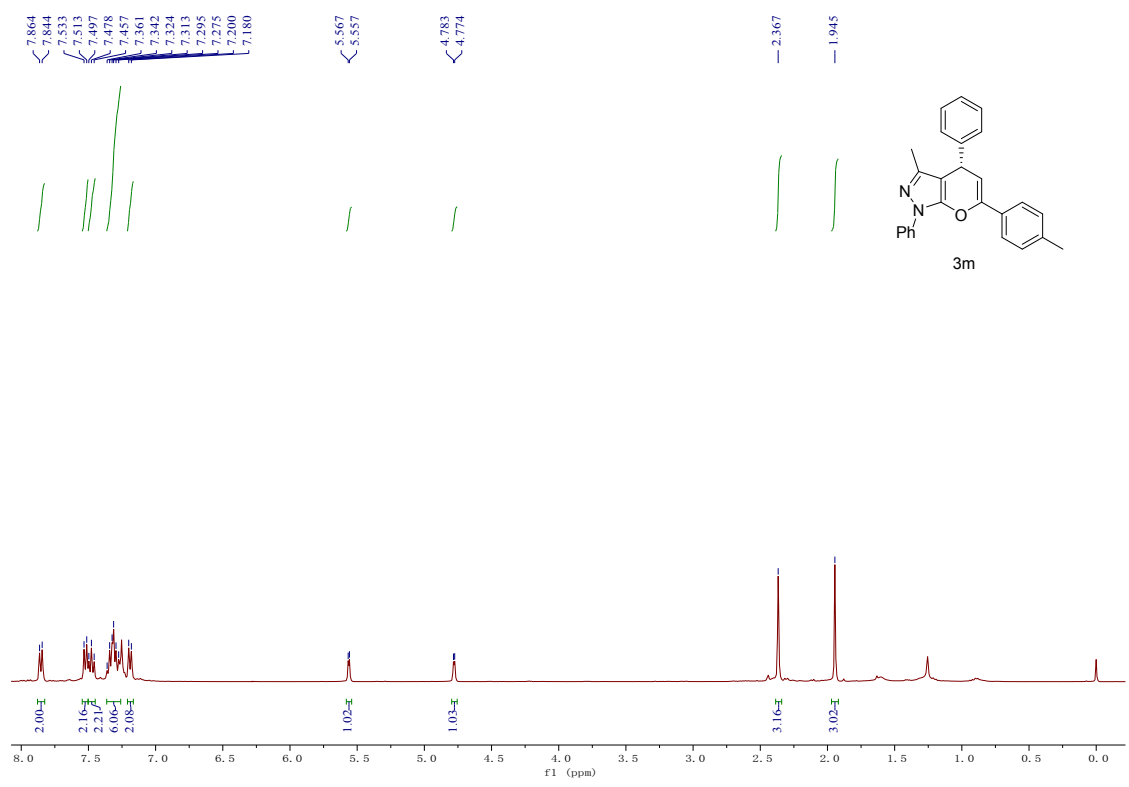


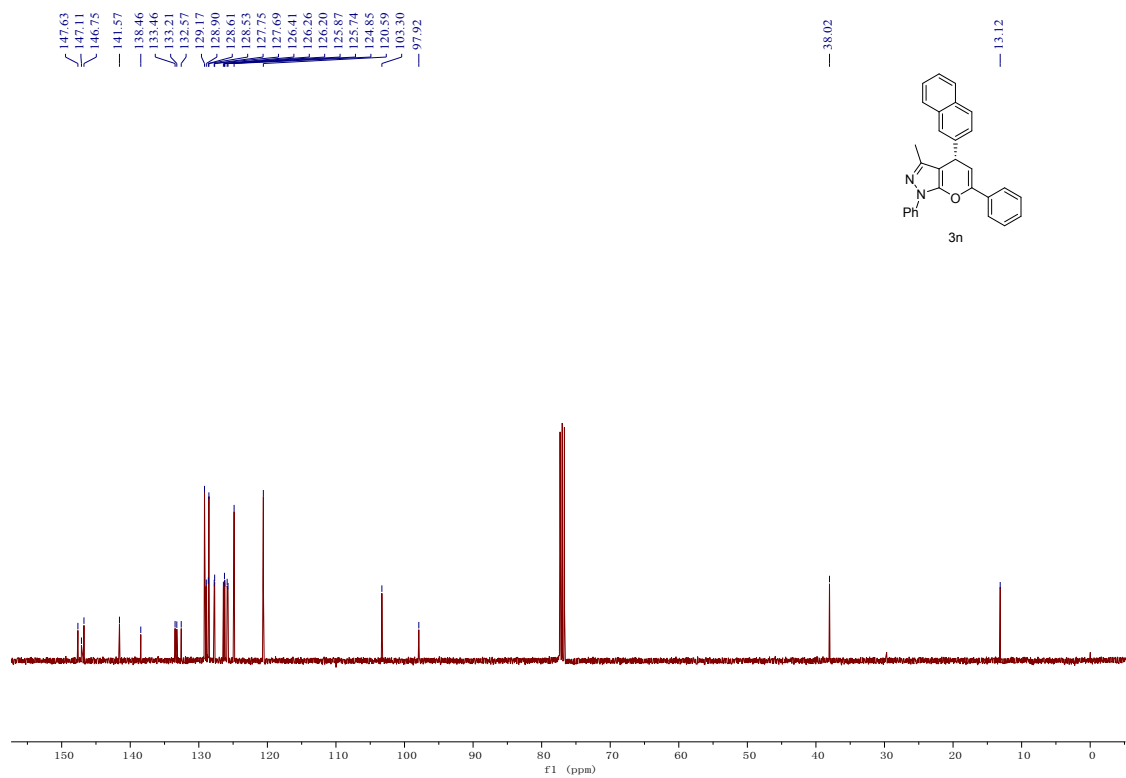
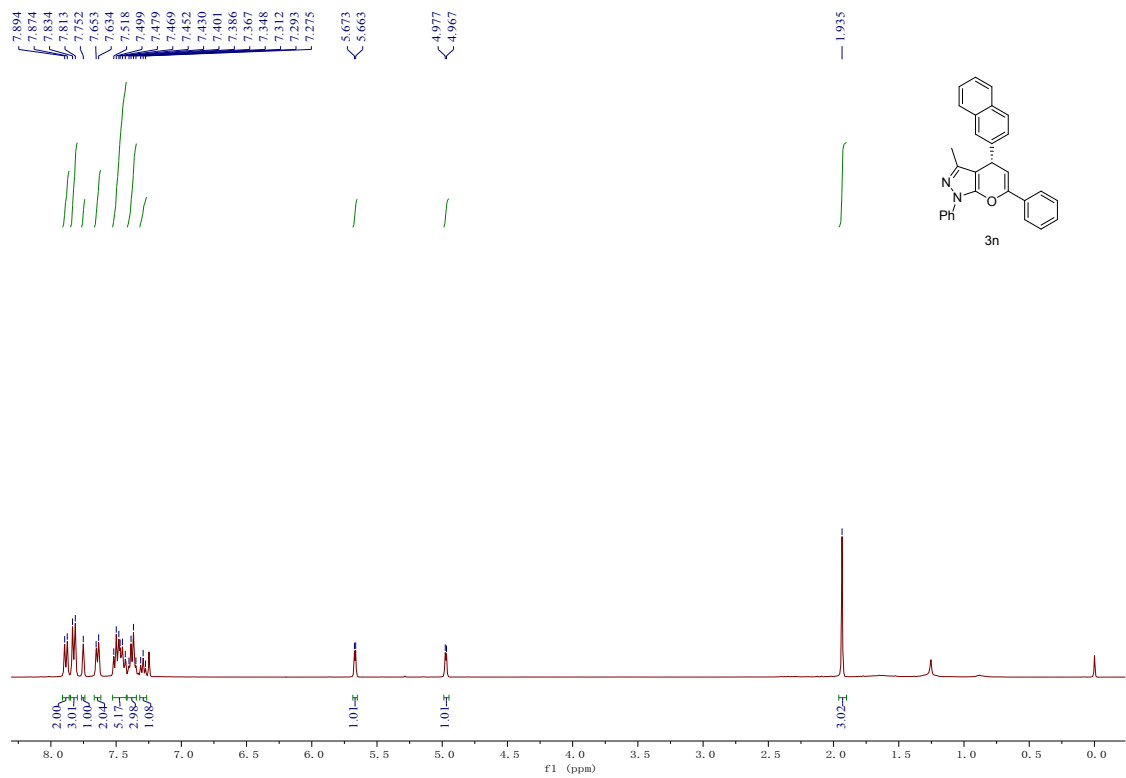


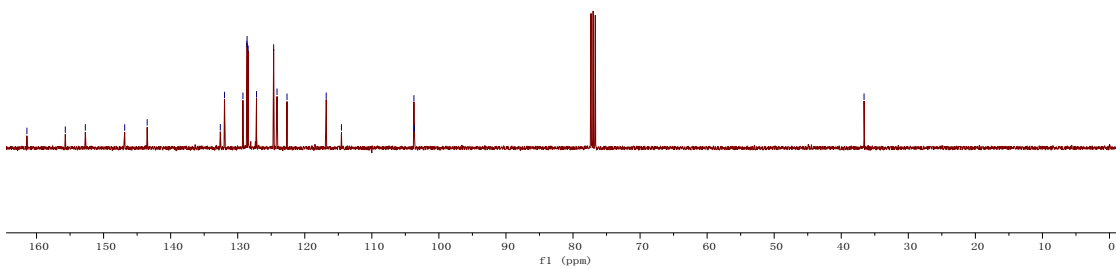
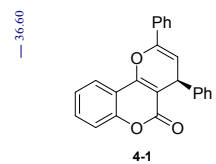
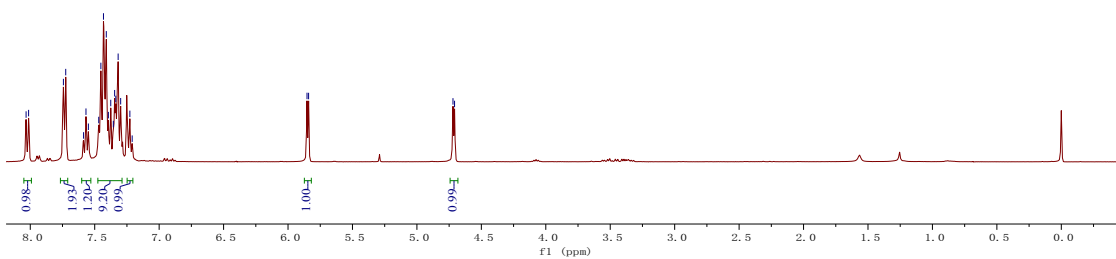
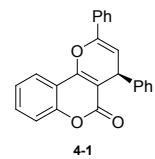
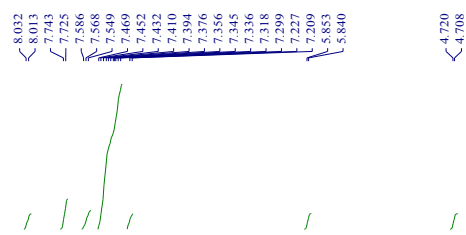


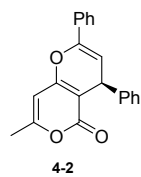
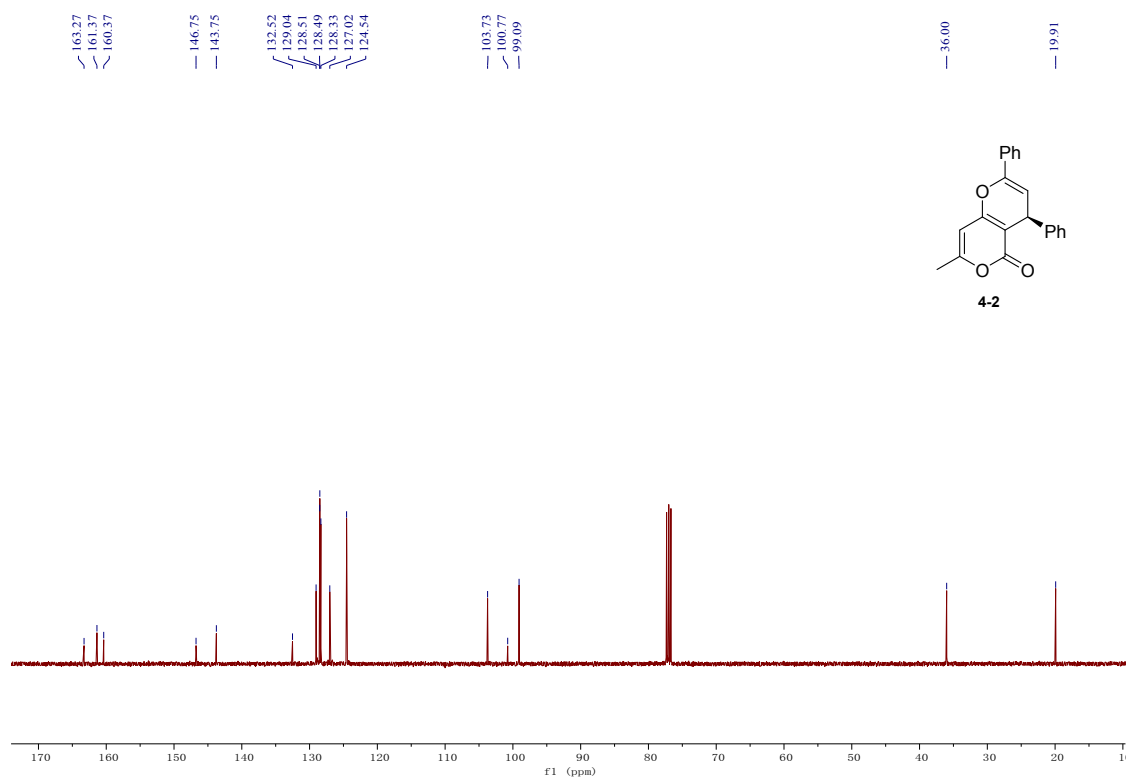
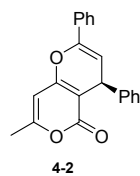
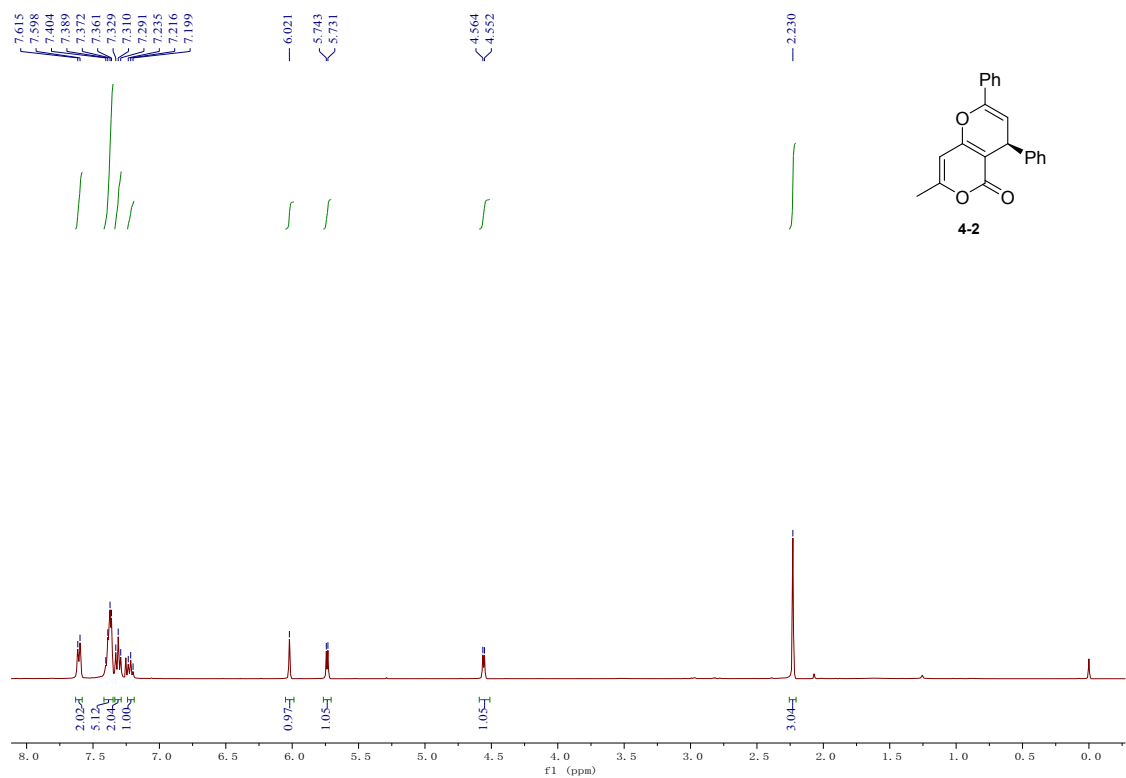




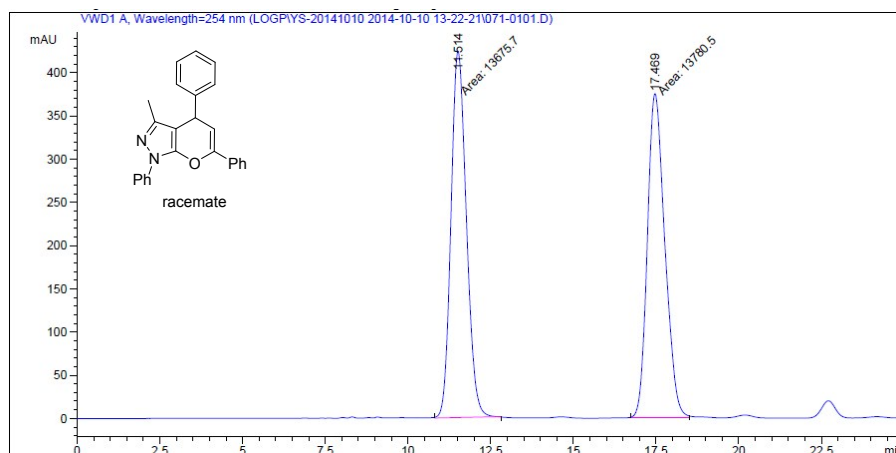






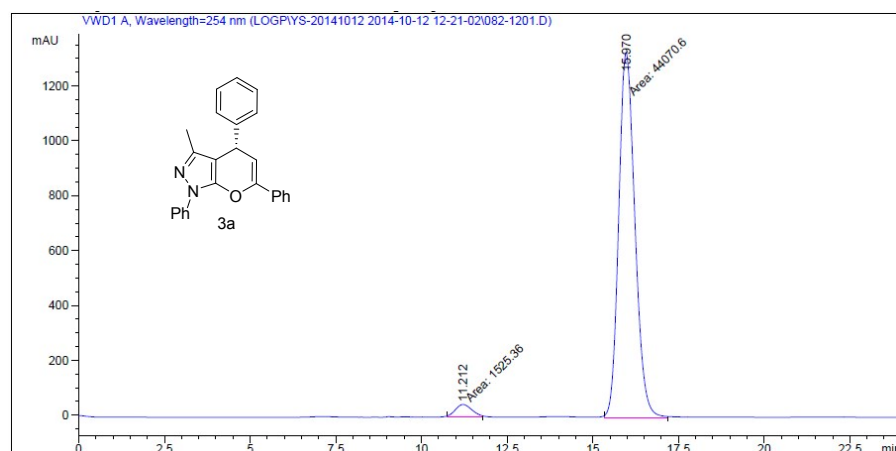


Copies of HPLC chromatograms of new compounds



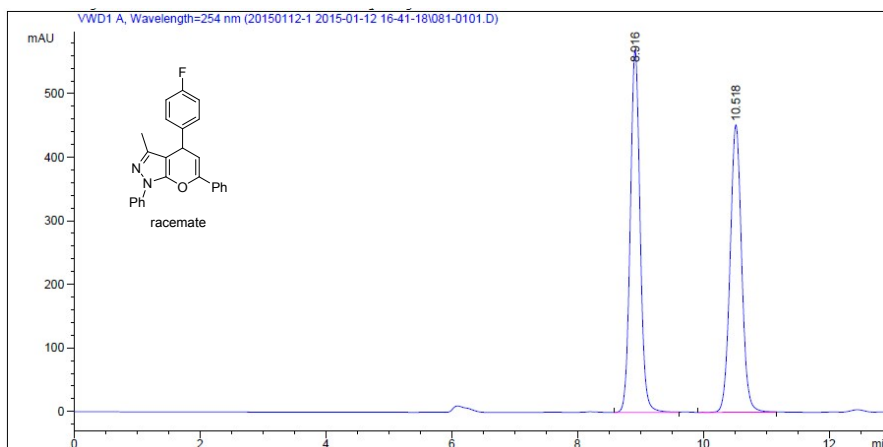
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2	17.469	MM	0.6123	1.37805e4	375.08987	50.1908

Totals : 2.74562e4 798.87738



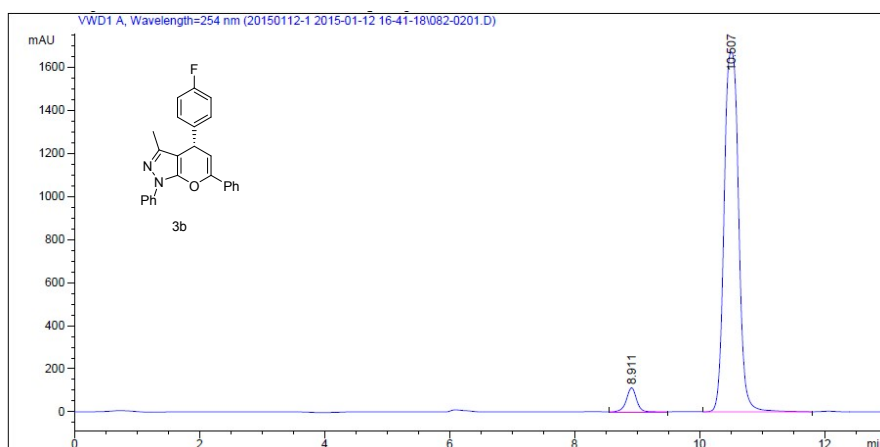
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1	11.212	MM	0.5620	1525.35840	45.23414	3.3454
2	15.970	MM	0.5507	4.40706e4	1333.73535	96.6546

Totals : 4.55960e4 1378.96949



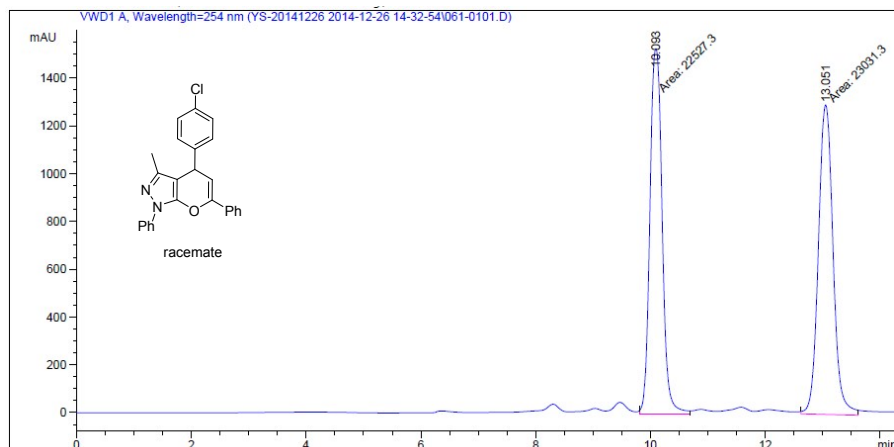
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1	8.916	VB	0.1655	6168.44727	570.31036	51.6884
2	10.518	VB	0.1967	5765.47217	452.34433	48.3116

Totals : 1.19339e4 1022.65469



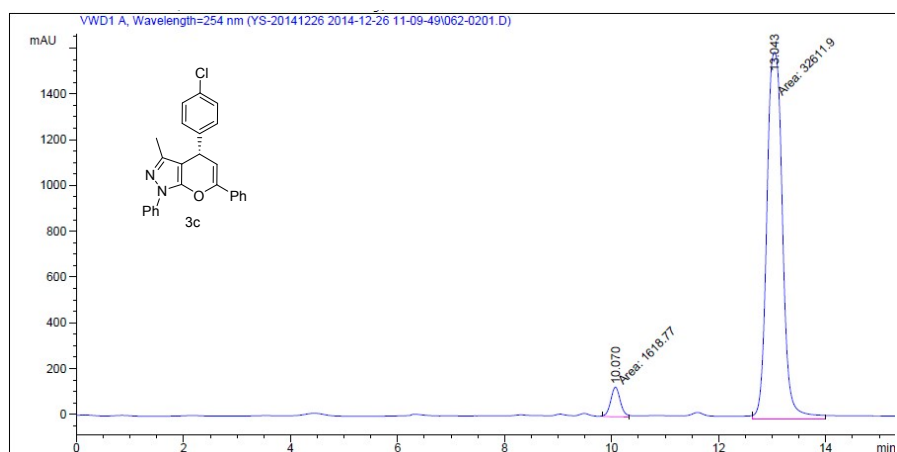
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1	8.911	VB	0.1709	1262.13257	111.93694	4.4876
2	10.507	VB	0.2559	2.68624e4	1672.10815	95.5124

Totals : 2.81246e4 1784.04510



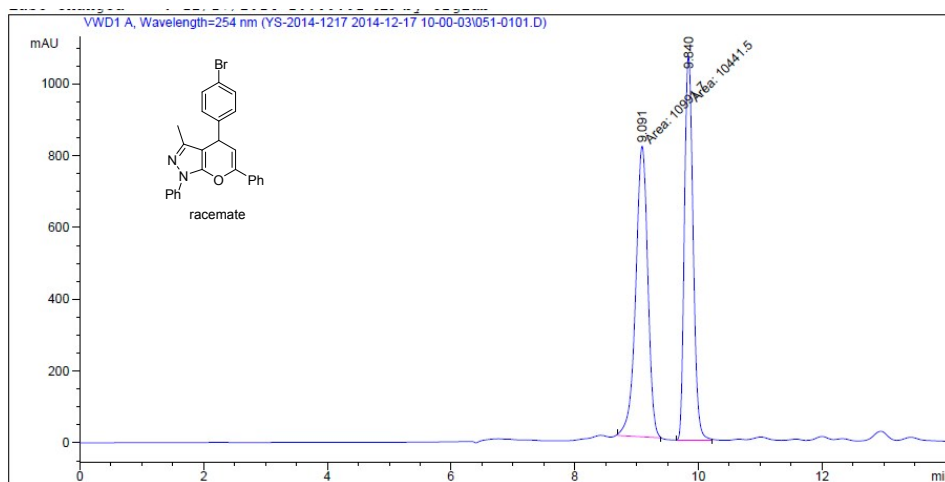
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1	10.093	MM	0.2457	2.25273e4	1528.36890	49.4468
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Totals : 4.55587e4 2824.27637



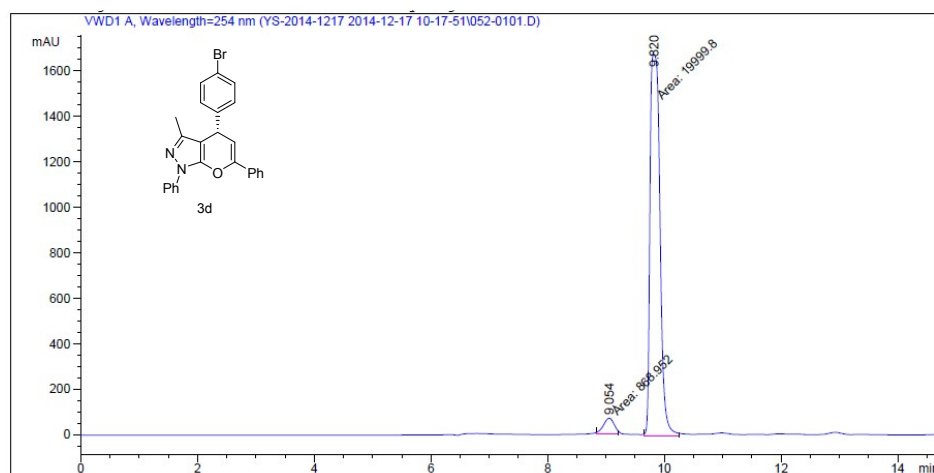
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2	13.043	MM	0.3393	3.26119e4	1602.07434	95.2710

Totals : 3.42306e4 1731.41264



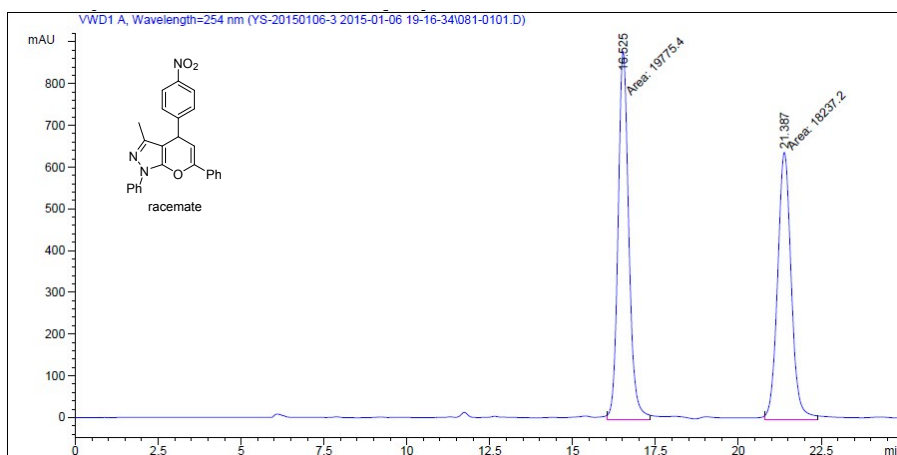
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2	9.840	MM	0.1625	1.04415e4	1071.04590	48.7164

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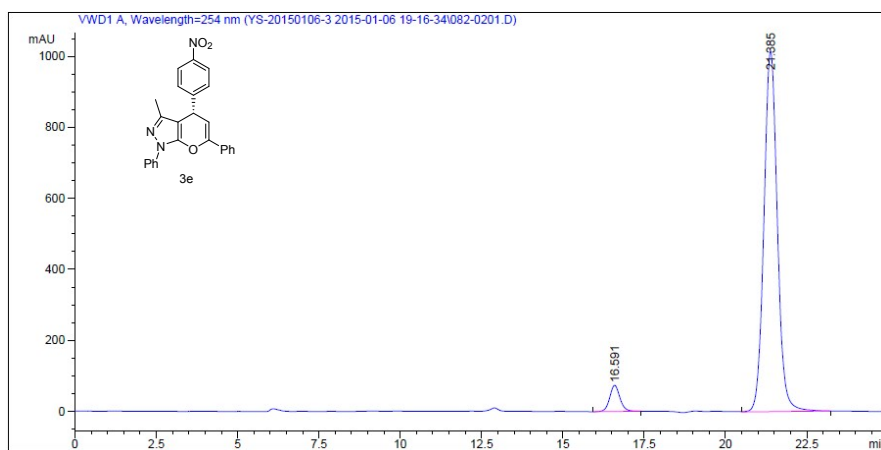
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2	9.820	MM	0.1986	1.99998e4	1678.65979	95.8361

Totals : 2.08687e4 1747.57157



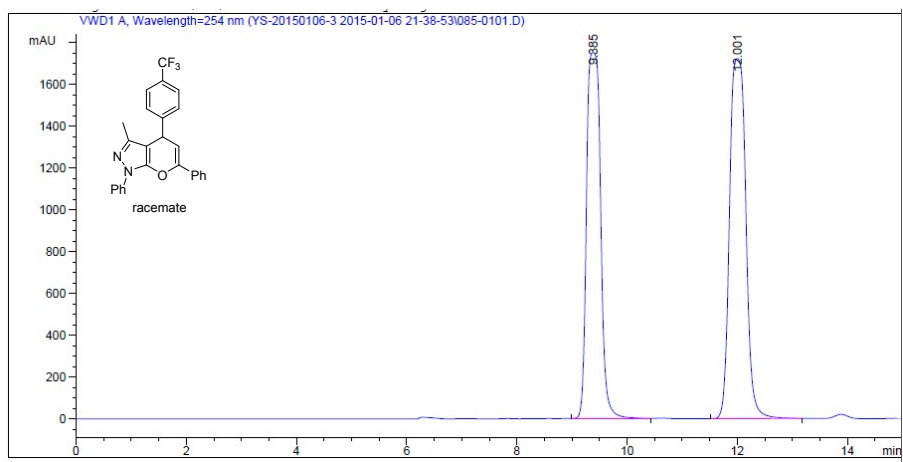
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1	16.525	MM	0.3741	1.97754e4	881.03809	52.0233
2	21.387	MM	0.4753	1.82372e4	639.48157	47.9767

Totals : 3.80125e4 1520.51965

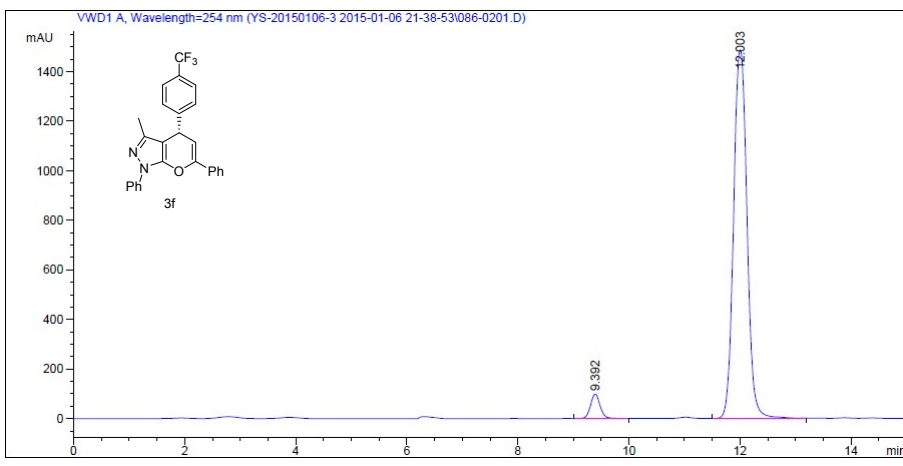


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1	16.591	VB	0.3351	1620.85046	73.72836	5.2853
2	21.385	VB	0.4428	2.90462e4	1015.95203	94.7147

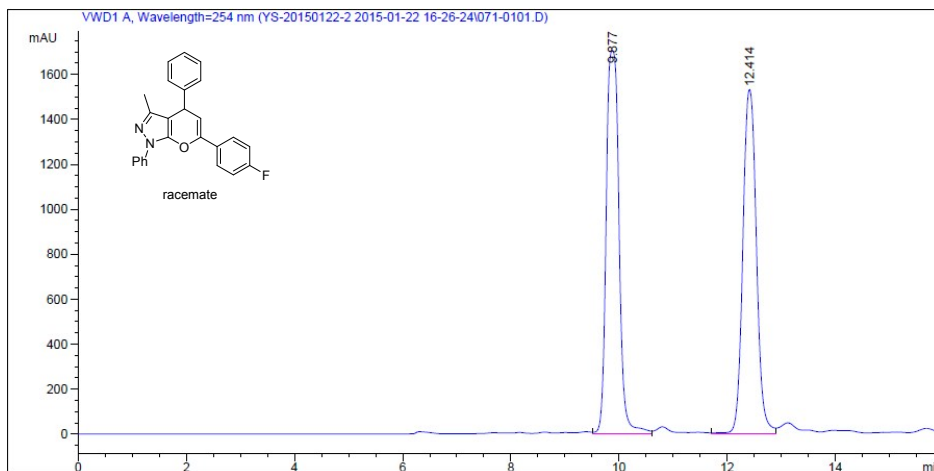
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1	9.385	VV	0.2820	3.09704e4	1751.63013	46.7996
2	12.001	VB	0.3260	3.52062e4	1721.35059	53.2004
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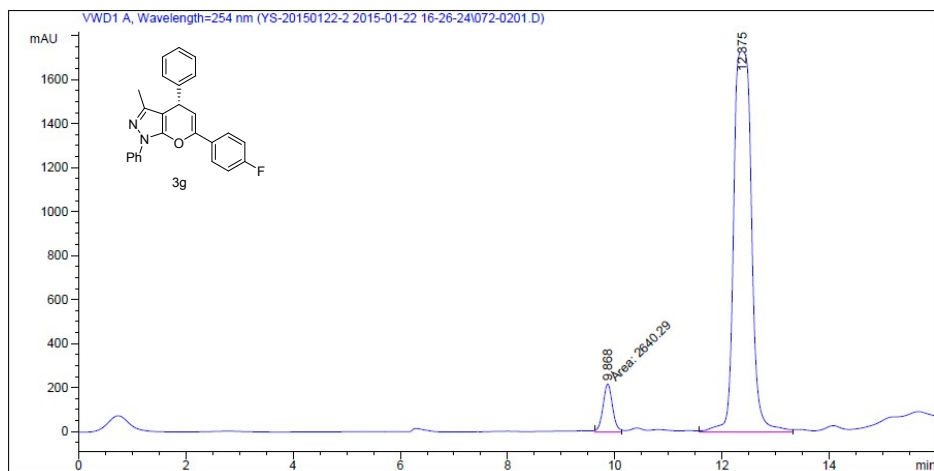


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1	9.392	VB	0.1857	1190.87305	98.87281	4.5685
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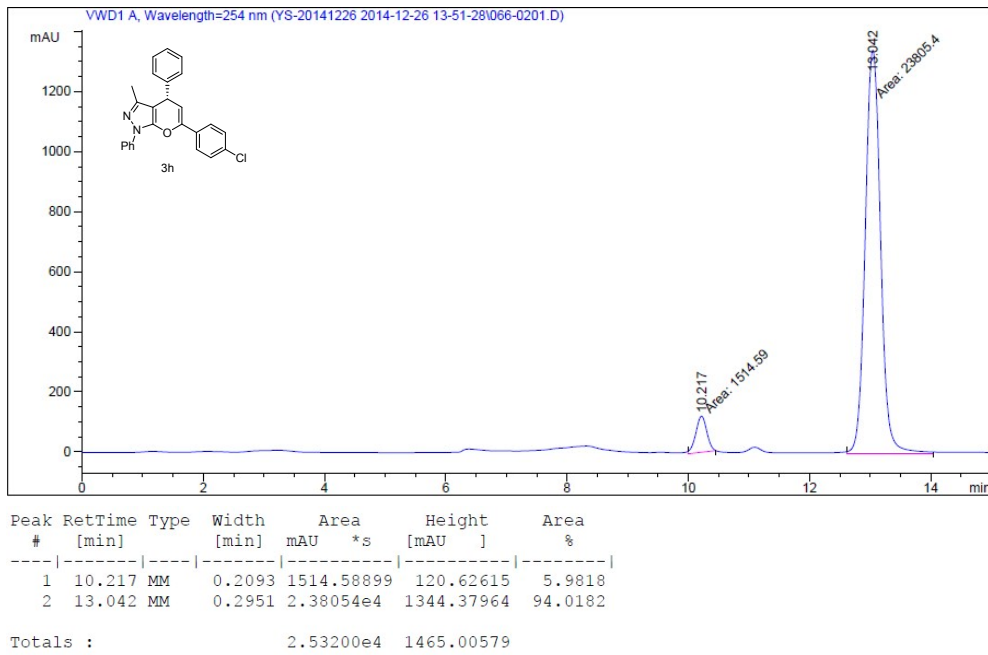
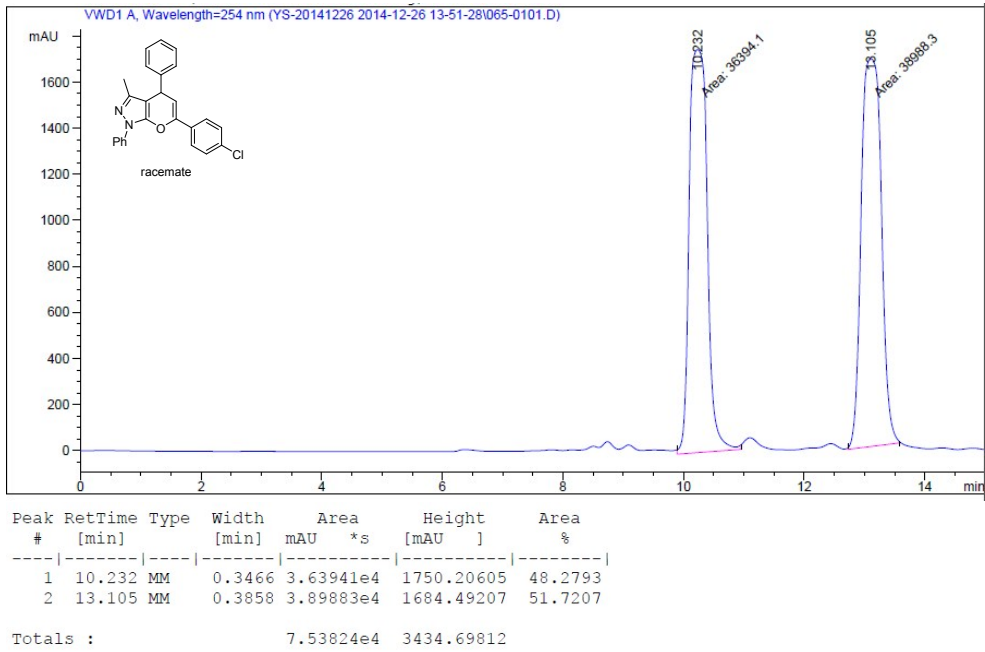
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1	9.877	VV	0.2622	2.81501e4	1707.53662	51.2769
2	12.414	VV	0.2749	2.67481e4	1533.27393	48.7231

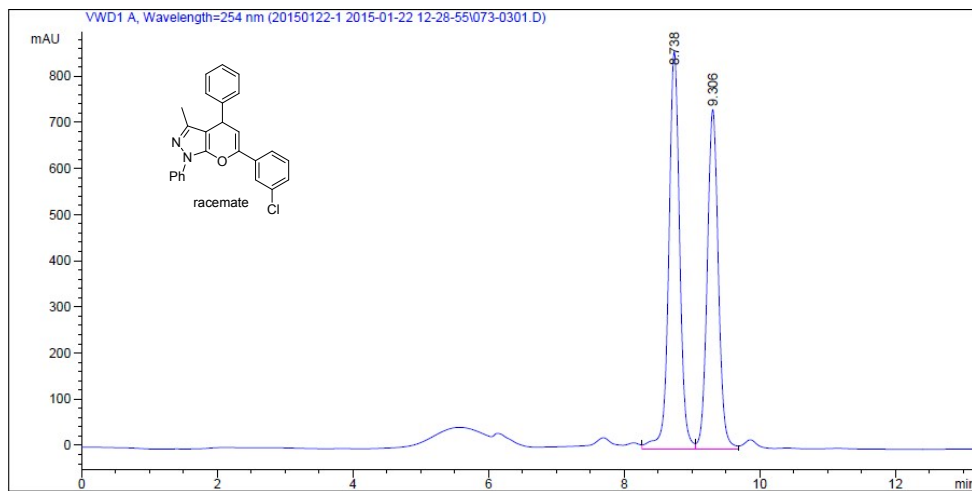
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2	12.375	VV	0.3662	4.01201e4	1733.08203	93.8254

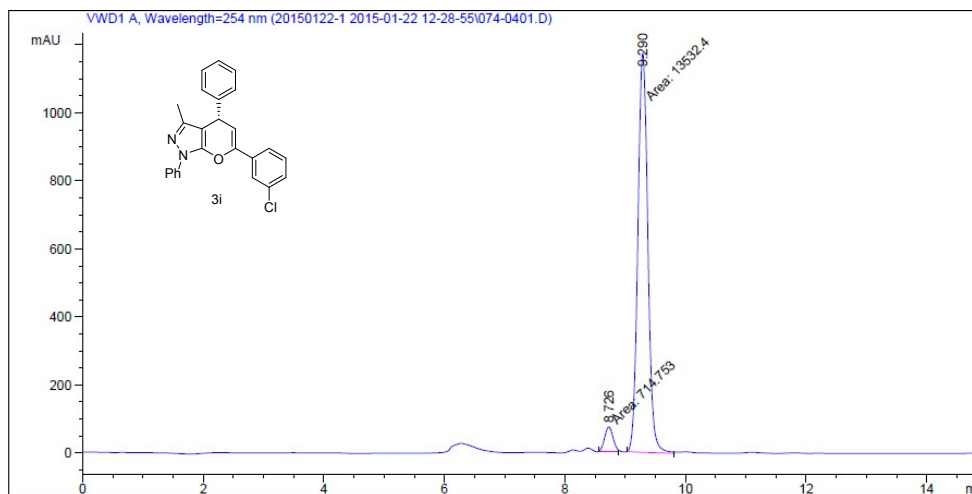
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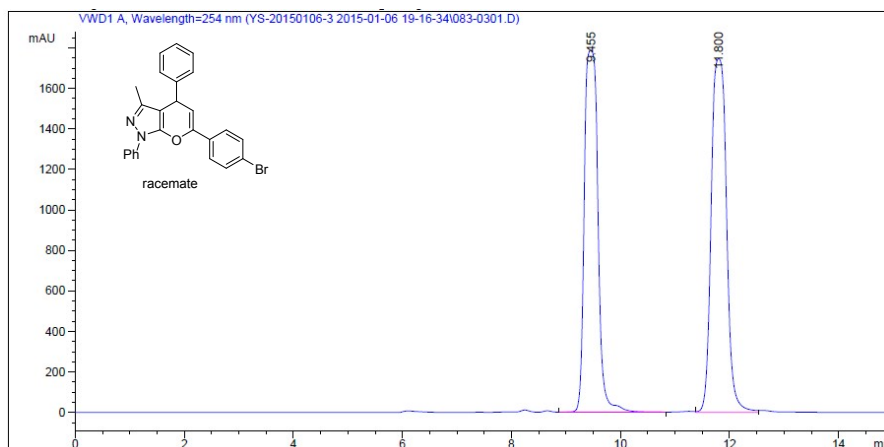
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1	8.738	VV	0.1712	9631.56543	861.62970	53.4944
2	9.306	VV	0.1749	8373.23242	736.26532	46.5056

Totals : 1.80048e4 1597.89502



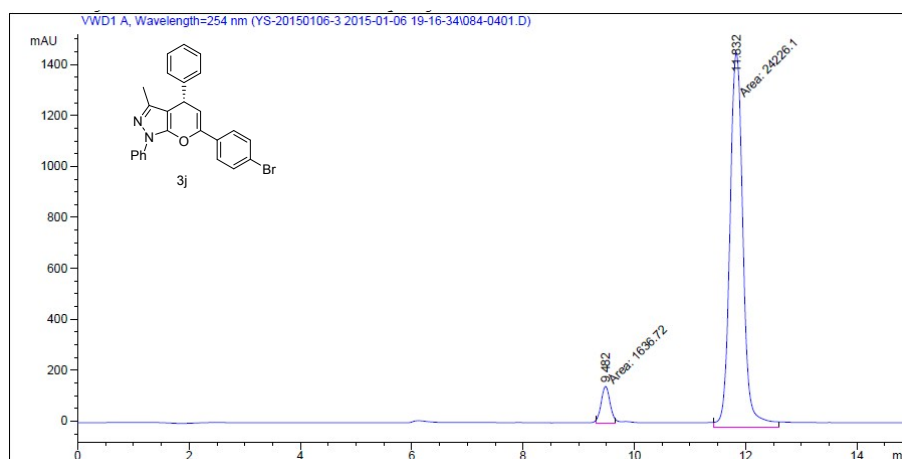
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1	8.726	MM	0.1624	714.75281	73.33372	5.0168
2	9.290	MM	0.1922	1.35324e4	1173.73926	94.9832

Totals : 1.42472e4 1247.07298



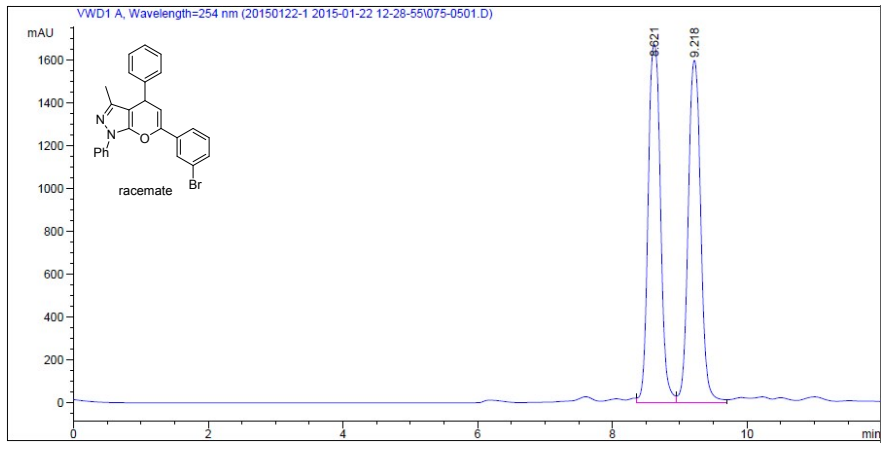
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1	9.455	VV	0.2772	3.11306e4	1789.41003	47.7939
2	11.800	VV	0.3086	3.40045e4	1747.02222	52.2061

Totals : 6.51350e4 3536.43225



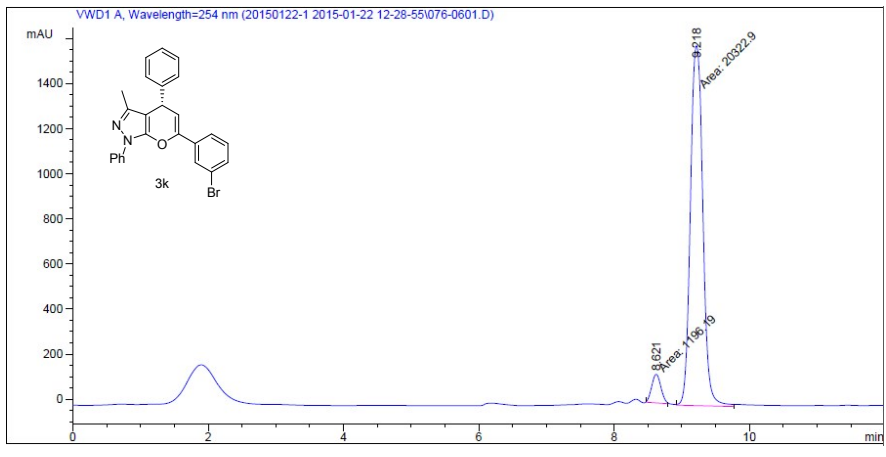
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1	9.482	MM	0.1861	1636.71716	146.58005	6.3285
2	11.832	MM	0.2746	2.42261e4	1470.41772	93.6715

Totals : 2.58628e4 1616.99777



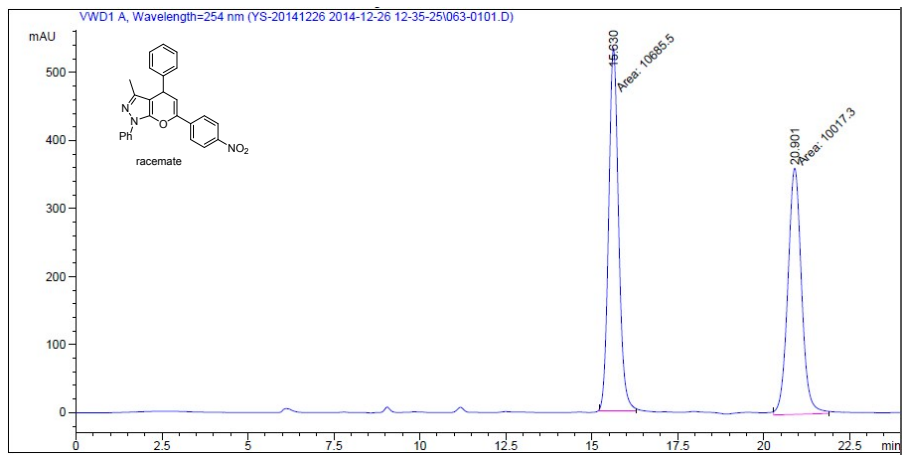
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1	8.621	VV	0.2023	2.12947e4	1673.93591	50.7253
2	9.218	VV	0.2033	2.06858e4	1599.87451	49.2747

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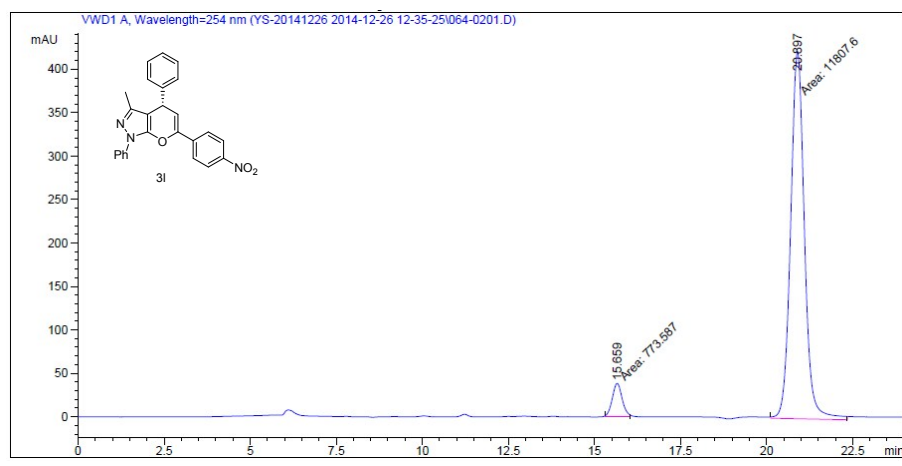


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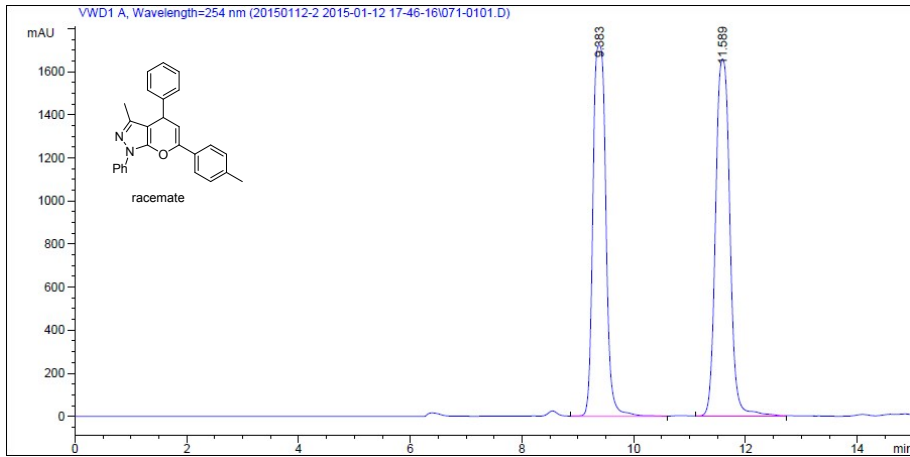
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1	15.630	MM	0.3344	1.06855e4	532.51013	51.6138
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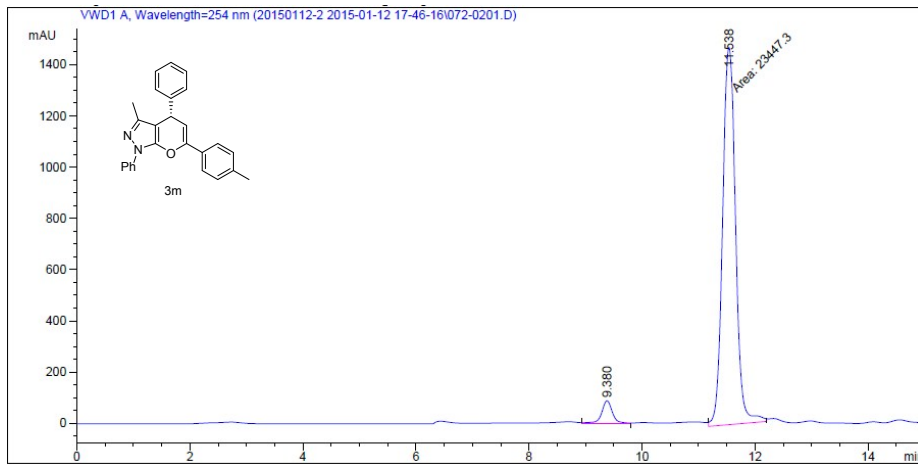


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1	15.659	MM	0.3347	773.58728	38.51691	6.1487
2	20.897	MM	0.4656	1.18076e4	422.70999	93.8513
Totals :				1.25812e4	461.22690	



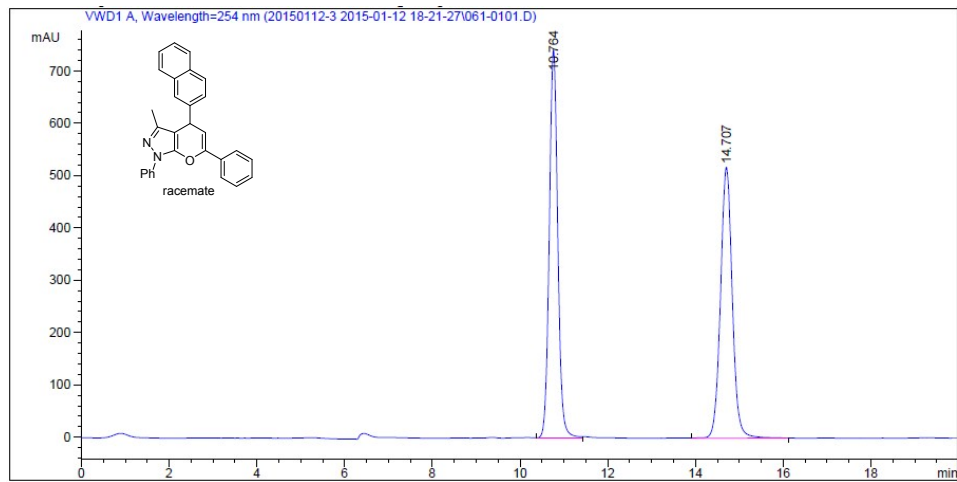
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1	9.383	VB	0.2556	2.76536e4	1724.02222	48.3661
2	11.589	VB	0.2819	2.95220e4	1658.77588	51.6339

Totals : 5.71756e4 3382.79810



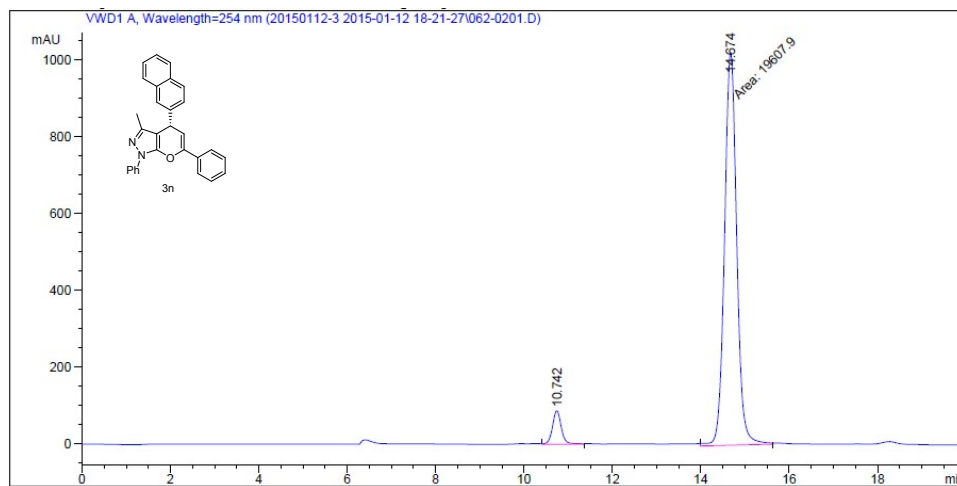
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1	9.380	VV	0.2054	1249.38440	90.13348	5.0589
2	11.538	MM	0.2656	2.34473e4	1471.58655	94.9411

Totals : 2.46967e4 1561.72003



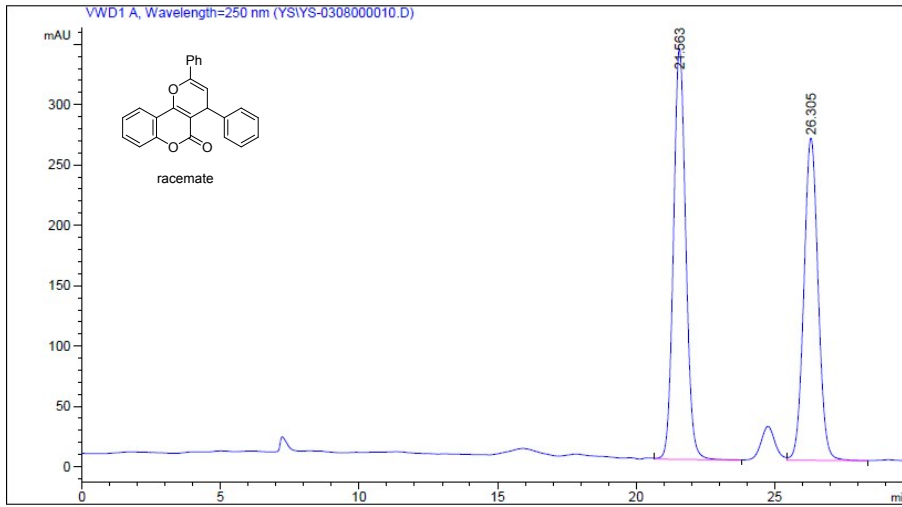
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	10.764	VV	0.2068	9999.89648	741.90961	50.8584	50.8584
2	14.707	VB	0.2861	9662.35449	517.78040	49.1416	49.1416

Totals : 1.96623e4 1259.69000

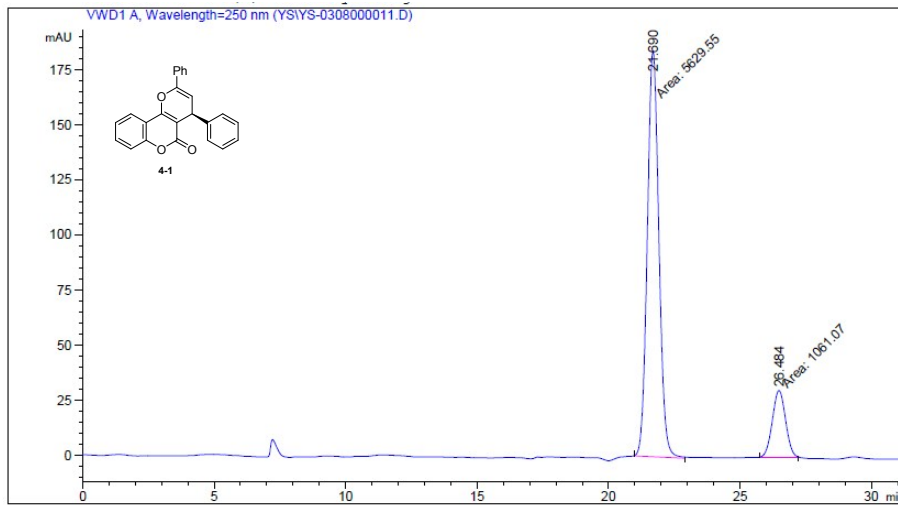


Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	10.742	VV	0.2124	1220.55969	87.44185	5.8601	5.8601
2	14.674	MM	0.3195	1.96079e4	1022.88086	94.1399	94.1399

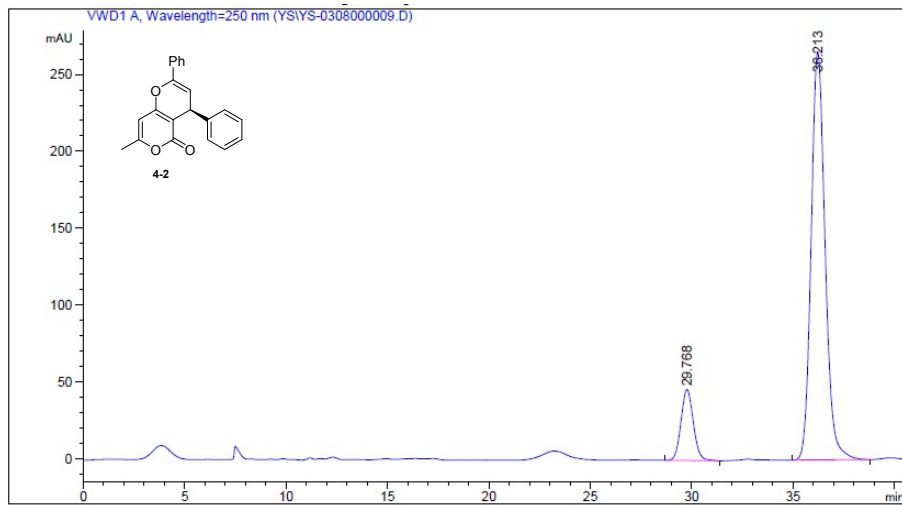
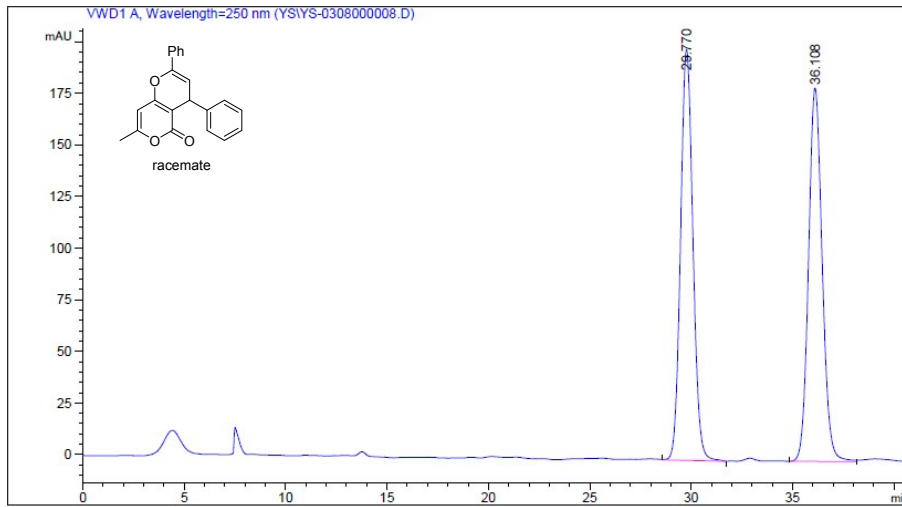
Totals : 2.08284e4 1110.32271



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.563	VB	0.4891	1.05084e4	339.66769	52.4064
2	26.305	VV	0.5688	9543.38086	266.73843	47.5936
Totals :				2.00518e4	606.40613	



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.690	MM	0.5086	5629.55420	184.46404	84.1409
2	26.484	MM	0.5825	1061.07373	30.36044	15.8591
Totals :				6690.62793	214.82447	



3C

Table 1 Crystal data and structure refinement for 3C.

Identification code	3C
Empirical formula	C ₂₅ H ₁₉ ClN ₂ O
Formula weight	398.87
Temperature/K	303.9
Crystal system	monoclinic
Space group	C2
a/Å	21.832(2)
b/Å	5.1765(5)
c/Å	18.5849(19)
α/°	90
β/°	103.747(2)
γ/°	90
Volume/Å ³	2040.2(4)
Z	4
ρ _{calc} /g/cm ³	1.299
μ/mm ⁻¹	0.206
F(000)	832.0
Crystal size/mm ³	0.1 × 0.06 × 0.04
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	4.512 to 52.036
Index ranges	-26 ≤ h ≤ 26, -6 ≤ k ≤ 5, -22 ≤ l ≤ 22
Reflections collected	18832
Independent reflections	3766 [R _{int} = 0.0707, R _{sigma} = 0.0606]
Data/restraints/parameters	3766/73/300
Goodness-of-fit on F ²	1.064
Final R indexes [I ≥ 2σ(I)]	R ₁ = 0.0650, wR ₂ = 0.1376
Final R indexes [all data]	R ₁ = 0.1098, wR ₂ = 0.1556
Largest diff. peak/hole / e Å ⁻³	0.18/-0.20
Flack parameter	0.14(5)

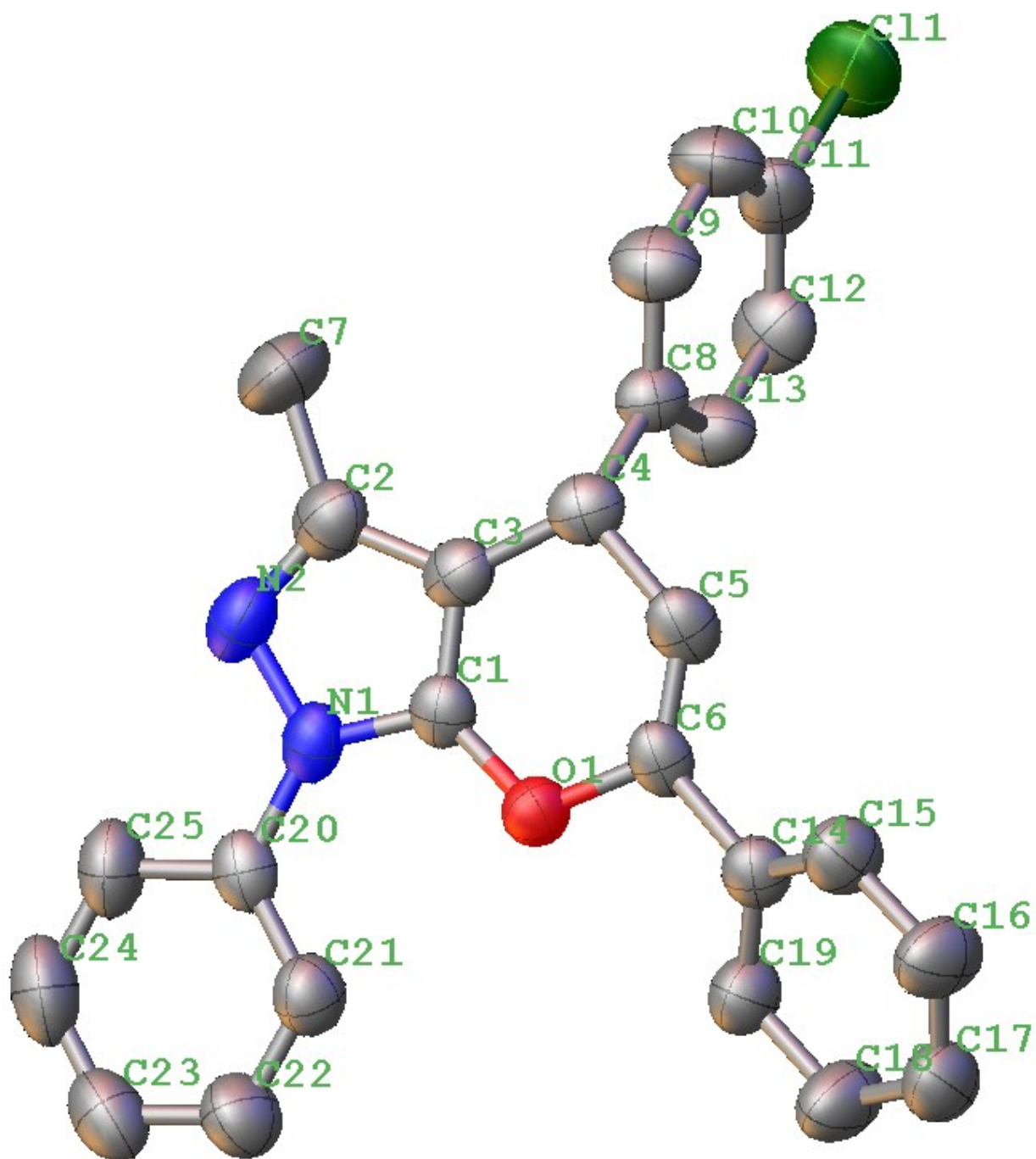


Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 3C. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{ij} tensor.

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Cl1	3533.0 (9)	9493 (6)	6560.2 (8)	111.3 (9)
O1	2766.2 (15)	6258 (8)	2019.9 (17)	51.7 (10)
N1	3773.9 (17)	4284 (11)	2207 (2)	51.5 (13)
N2	4359.8 (19)	4553 (14)	2689 (2)	66.6 (15)
C1	3376 (2)	6034 (12)	2401 (2)	45.6 (14)
C2	4298 (3)	6438 (15)	3149 (3)	64.7 (18)
C3	3681 (2)	7416 (13)	2989 (3)	50.7 (15)
C4	3382 (2)	9628 (13)	3286 (2)	50.4 (13)
C5	2714 (2)	9741 (14)	2839 (2)	54.4 (14)
C6	2444 (2)	8276 (11)	2277 (2)	47.1 (14)
C7	4858 (3)	7290 (20)	3727 (4)	99 (3)
C8	3424 (2)	9553 (12)	4113 (2)	46.0 (13)
C9	3770 (3)	11313 (14)	4577 (3)	66.0 (17)
C10	3809 (3)	11317 (16)	5332 (3)	76.1 (19)
C11	3491 (3)	9505 (17)	5615 (3)	66.0 (18)
C12	3140 (3)	7657 (15)	5174 (3)	67.4 (18)
C13	3105 (3)	7708 (14)	4422 (3)	60.9 (16)
C14	1792 (2)	8410 (12)	1841 (3)	50.3 (13)
C17	559 (3)	8821 (15)	1024 (3)	75.6 (18)
C20	3684 (2)	2386 (12)	1635 (3)	49.2 (14)
C21	3118 (3)	2089 (14)	1134 (3)	61.9 (17)
C22	3050 (3)	221 (15)	601 (4)	74 (2)
C23	3542 (3)	-1362 (15)	554 (4)	77 (2)
C24	4106 (3)	-1065 (16)	1060 (4)	85 (2)
C25	4182 (3)	787 (16)	1600 (4)	74 (2)
C15	1522 (6)	10790 (30)	1725 (7)	65 (3)
C16	899 (7)	11010 (40)	1335 (9)	80 (4)
C18	876 (7)	6420 (40)	1093 (10)	84 (5)
C19	1507 (6)	6190 (40)	1505 (8)	64 (3)
C26	1292 (4)	9290 (30)	2169 (5)	62 (3)
C27	677 (5)	9420 (30)	1762 (6)	70 (4)
C28	990 (5)	7980 (30)	712 (6)	66 (4)
C29	1604 (5)	7800 (30)	1118 (5)	62 (3)

Table 3 Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 3C. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C11	103.0 (13)	185 (3)	48.0 (7)	0.7 (12)	21.6 (8)	14.0 (16)
O1	48.2 (19)	59 (3)	46.4 (17)	1.1 (17)	7.4 (15)	6.9 (19)
N1	44 (2)	65 (4)	48 (2)	15 (2)	17.9 (18)	10 (2)
N2	44 (2)	92 (5)	64 (3)	13 (3)	13 (2)	5 (3)
C1	50 (3)	52 (4)	37 (2)	13 (2)	15 (2)	5 (3)
C2	54 (3)	87 (6)	55 (3)	8 (3)	16 (3)	-1 (3)
C3	48 (3)	61 (5)	44 (3)	8 (3)	13 (2)	-6 (3)
C4	57 (3)	49 (4)	45 (2)	11 (3)	10 (2)	-4 (3)
C5	68 (3)	55 (4)	40 (2)	12 (3)	13 (2)	15 (3)
C6	59 (3)	47 (4)	37 (2)	12 (2)	16 (2)	14 (3)
C7	56 (4)	138 (9)	92 (5)	-15 (5)	-5 (3)	0 (5)
C8	50 (3)	40 (4)	45 (2)	6 (3)	6 (2)	0 (3)
C9	75 (4)	59 (5)	62 (3)	3 (3)	14 (3)	-15 (4)
C10	83 (4)	82 (6)	58 (3)	-12 (4)	4 (3)	-9 (4)
C11	59 (3)	91 (6)	44 (3)	6 (4)	5 (2)	18 (4)
C12	71 (4)	79 (5)	56 (3)	19 (3)	22 (3)	-5 (4)
C13	67 (3)	61 (5)	53 (3)	2 (3)	11 (3)	-13 (3)
C14	54 (2)	56 (4)	41 (2)	8 (2)	11.4 (18)	8 (2)
C17	61 (3)	88 (5)	72 (3)	-7 (3)	5 (2)	17 (3)
C20	59 (3)	43 (4)	52 (3)	13 (3)	26 (2)	6 (3)
C21	61 (4)	62 (5)	63 (3)	4 (3)	16 (3)	11 (3)
C22	78 (4)	66 (6)	78 (4)	-8 (4)	18 (3)	9 (4)
C23	93 (5)	69 (6)	79 (4)	-8 (4)	41 (4)	2 (4)
C24	85 (5)	79 (7)	106 (5)	7 (5)	52 (4)	23 (4)
C25	63 (4)	79 (6)	87 (4)	10 (4)	31 (3)	20 (4)
C15	65 (5)	67 (6)	59 (7)	4 (4)	5 (4)	14 (4)
C16	68 (5)	77 (6)	84 (9)	-3 (5)	-7 (5)	18 (4)
C18	61 (5)	79 (6)	100 (10)	-11 (6)	-5 (5)	10 (4)
C19	53 (5)	67 (6)	68 (7)	0 (4)	10 (4)	9 (4)
C26	57 (4)	75 (9)	54 (4)	-7 (4)	16 (3)	7 (4)
C27	55 (4)	91 (10)	64 (4)	-9 (5)	12 (3)	14 (4)
C28	59 (4)	82 (10)	50 (4)	-4 (4)	1 (3)	19 (4)
C29	57 (4)	83 (10)	44 (4)	-2 (5)	8 (3)	21 (4)

Table 4 Bond Lengths for 3C.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Cl1	C11	1.737 (5)	C12	C13	1.383 (7)
O1	C1	1.356 (5)	C14	C15	1.361 (16)
O1	C6	1.405 (6)	C14	C19	1.384 (18)
N1	N2	1.383 (6)	C14	C26	1.445 (11)
N1	C1	1.362 (7)	C14	C29	1.346 (10)
N1	C20	1.426 (8)	C17	C16	1.403 (19)
N2	C2	1.325 (9)	C17	C18	1.42 (2)
C1	C3	1.342 (7)	C17	C27	1.370 (12)
C2	C3	1.403 (8)	C17	C28	1.293 (12)
C2	C7	1.488 (8)	C20	C21	1.369 (7)
C3	C4	1.489 (9)	C20	C25	1.379 (8)
C4	C5	1.500 (7)	C21	C22	1.367 (9)
C4	C8	1.518 (6)	C22	C23	1.369 (9)
C5	C6	1.312 (8)	C23	C24	1.370 (9)
C6	C14	1.461 (7)	C24	C25	1.370 (10)
C8	C9	1.355 (8)	C15	C16	1.385 (18)
C8	C13	1.384 (8)	C18	C19	1.415 (18)
C9	C10	1.385 (8)	C26	C27	1.377 (13)
C10	C11	1.347 (10)	C28	C29	1.378 (14)
C11	C12	1.369 (10)			

Table 5 Bond Angles for 3C.

Ato m	Ato m	Ato m	Angle/°	Ato m	Ato m	Ato m	Angle/°
C1	O1	C6	113.3 (4)	C12	C11	Cl1	119.4 (6)
N2	N1	C20	119.6 (4)	C11	C12	C13	118.7 (6)
C1	N1	N2	108.1 (5)	C12	C13	C8	121.3 (6)
C1	N1	C20	132.3 (4)	C15	C14	C6	117.4 (7)
C2	N2	N1	105.8 (5)	C15	C14	C19	123.3 (9)
O1	C1	N1	121.8 (5)	C19	C14	C6	118.9 (7)
C3	C1	O1	128.0 (5)	C26	C14	C6	121.0 (5)
C3	C1	N1	110.2 (4)	C29	C14	C6	124.6 (6)
N2	C2	C3	111.6 (5)	C29	C14	C26	114.4 (7)
N2	C2	C7	119.5 (6)	C16	C17	C18	118.2 (10)
C3	C2	C7	128.9 (7)	C28	C17	C27	122.8 (8)
C1	C3	C2	104.3 (5)	C21	C20	N1	121.7 (5)
C1	C3	C4	122.3 (5)	C21	C20	C25	119.6 (6)

C2	C3	C4	133.0 (5)	C25	C20	N1	118.6 (5)
C3	C4	C5	106.2 (5)	C22	C21	C20	119.8 (6)
C3	C4	C8	115.5 (5)	C21	C22	C23	121.2 (6)
C5	C4	C8	112.3 (4)	C22	C23	C24	118.7 (7)
C6	C5	C4	127.8 (6)	C25	C24	C23	120.9 (6)
O1	C6	C14	110.3 (4)	C24	C25	C20	119.7 (6)
C5	C6	O1	122.2 (5)	C14	C15	C16	119.5 (14)
C5	C6	C14	127.4 (5)	C15	C16	C17	120.5 (15)
C9	C8	C4	121.2 (5)	C19	C18	C17	121.1 (15)
C9	C8	C13	117.4 (5)	C14	C19	C18	116.7 (14)
C13	C8	C4	121.5 (5)	C27	C26	C14	121.3 (8)
C8	C9	C10	122.4 (6)	C17	C27	C26	117.8 (9)
C11	C10	C9	118.8 (6)	C17	C28	C29	119.7 (9)
C10	C11	C11	119.3 (6)	C14	C29	C28	123.9 (9)
C10	C11	C12	121.4 (5)				

Table 6 Torsion Angles for 3C.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C11	C11	C12	C13	179.3 (5)	C5	C6	C14	C26	-30.0 (11)
O1	C1	C3	C2	-178.5 (5)	C5	C6	C14	C29	148.8 (9)
O1	C1	C3	C4	-4.3 (9)	C6	O1	C1	N1	-176.2 (5)
O1	C6	C14	C15	-146.4 (8)	C6	O1	C1	C3	2.6 (7)
O1	C6	C14	C19	27.0 (9)	C6	C14	C15	C16	-178.0 (11)
O1	C6	C14	C26	149.0 (8)	C6	C14	C19	C18	179.7 (11)
O1	C6	C14	C29	-32.2 (10)	C6	C14	C26	C27	-179.8 (11)
N1	N2	C2	C3	0.6 (7)	C6	C14	C29	C28	-178.6 (11)
N1	N2	C2	C7	-177.7 (6)	C7	C2	C3	C1	177.4 (7)
N1	C1	C3	C2	0.4 (6)	C7	C2	C3	C4	4.1 (11)
N1	C1	C3	C4	174.7 (5)	C8	C4	C5	C6	-129.6 (6)
N1	C20	C21	C22	-179.1 (6)	C8	C9	C10	C11	-0.1 (10)
N1	C20	C25	C24	179.5 (6)	C9	C8	C13	C12	-0.2 (9)
N2	N1	C1	O1	179.0 (4)	C9	C10	C11	C11	-179.8 (5)
N2	N1	C1	C3	0.0 (6)	C9	C10	C11	C12	0.9 (11)
N2	N1	C20	C21	-178.4 (5)	C10	C11	C12	C13	-1.4 (10)
N2	N1	C20	C25	2.8 (8)	C11	C12	C13	C8	1.0 (9)
N2	C2	C3	C1	-0.6 (7)	C13	C8	C9	C10	-0.3 (9)
N2	C2	C3	C4	-174.0 (6)	C14	C15	C16	C17	-4 (2)
C1	O1	C6	C5	-1.1 (7)	C14	C26	C27	C17	-4 (2)
C1	O1	C6	C14	179.8 (4)	C17	C18	C19	C14	1 (2)

C1 N1 N2 C2	-0.4 (6)	C17C28C29C14	1 (2)
C1 N1 C20C21	1.9 (9)	C20N1 N2 C2	179.8 (5)
C1 N1 C20C25	-176.9 (6)	C20N1 C1 O1	-1.3 (9)
C1 C3 C4 C5	3.6 (7)	C20N1 C1 C3	179.7 (5)
C1 C3 C4 C8	128.8 (5)	C20C21C22C23	-0.4 (10)
C2 C3 C4 C5	176.0 (6)	C21C20C25C24	0.7 (9)
C2 C3 C4 C8	-58.8 (8)	C21C22C23C24	0.7 (10)
C3 C4 C5 C6	-2.5 (7)	C22C23C24C25	-0.4 (10)
C3 C4 C8 C9	113.2 (7)	C23C24C25C20	-0.3 (10)
C3 C4 C8 C13	-67.7 (7)	C25C20C21C22	-0.4 (9)
C4 C5 C6 O1	1.5 (9)	C15C14C19C18	-7.3 (18)
C4 C5 C6 C14	-179.6 (5)	C16C17C18C19	4 (2)
C4 C8 C9 C10	178.9 (6)	C18C17C16C15	-3 (2)
C4 C8 C13C12	-179.4 (5)	C19C14C15C16	8.9 (18)
C5 C4 C8 C9	-124.9 (6)	C26C14C29C28	0.2 (18)
C5 C4 C8 C13	54.3 (8)	C27C17C28C29	-3 (2)
C5 C6 C14C15	34.6 (10)	C28C17C27C26	5 (2)
C5 C6 C14C19	-152.1 (9)	C29C14C26C27	1.3 (17)

Table 7 Hydrogen Atom Coordinates ($\text{\AA} \times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 3C.

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U(eq)
H4	3594	11208	3184	61
H5	2460	11002	2976	65
H7A	5226	6395	3661	149
H7B	4793	6904	4208	149
H7C	4916	9115	3684	149

H9	3990	12565	4382	79
H10	4051	12548	5638	91
H12	2929	6393	5377	81
H13	2863	6477	4117	73
H17A	151	9026	735	91
H17	135	8946	779	91
H21	2780	3155	1156	74
H22	2663	22	263	88
H23	3493	-2615	186	92
H24	4443	-2135	1036	102
H25	4567	964	1942	89
H15	1753	12256	1907	78
H16	706	12623	1279	97
H18	666	4965	862	101
H19	1721	4618	1549	76
H26	1388	9790	2664	74
H27	353	9892	1982	84
H28	886	7513	214	79
H29	1907	7208	879	75

Table 8 Atomic Occupancy for 3C.

Atom	Occupancy	Atom	Occupancy	Atom	Occupancy
H17A	0.5	H17	0.5	C15	0.445 (9)
H15	0.445 (9)	C16	0.445 (9)	H16	0.445 (9)
C18	0.445 (9)	H18	0.445 (9)	C19	0.445 (9)
H19	0.445 (9)	C26	0.555 (9)	H26	0.555 (9)
C27	0.555 (9)	H27	0.555 (9)	C28	0.555 (9)
H28	0.555 (9)	C29	0.555 (9)	H29	0.555 (9)