Supporting Information

Enantioselective Aminocatalytic Synthesis of Tetrahydropyrano [2,3-c] Pyrazoles via Domino Michael-Hemiacetalization Reaction with Alkylidene Pyrazolones

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1. General information

For the experiments, all starting materials and reagents were purchased from standard commercial sources or were prepared in the laboratory. All reactions were carried out in oven dried glassware under an argon atmosphere with magnetic stirring. Dichloromethane was distillation over CaH_2 under argon and stored over 4A° molecular sieves. All other solvents and reagents were purified according to standard procedures. Organic solvents were dried over anhydrous Na_2SO_4 and concentrated in a rotary evaporator under reduced pressure. Reactions were monitored by TLC on silica gel GF-254 using a combination of hexane and ethyl acetate as eluents. For column chromatography silica gel (60-120 mesh size) were used.

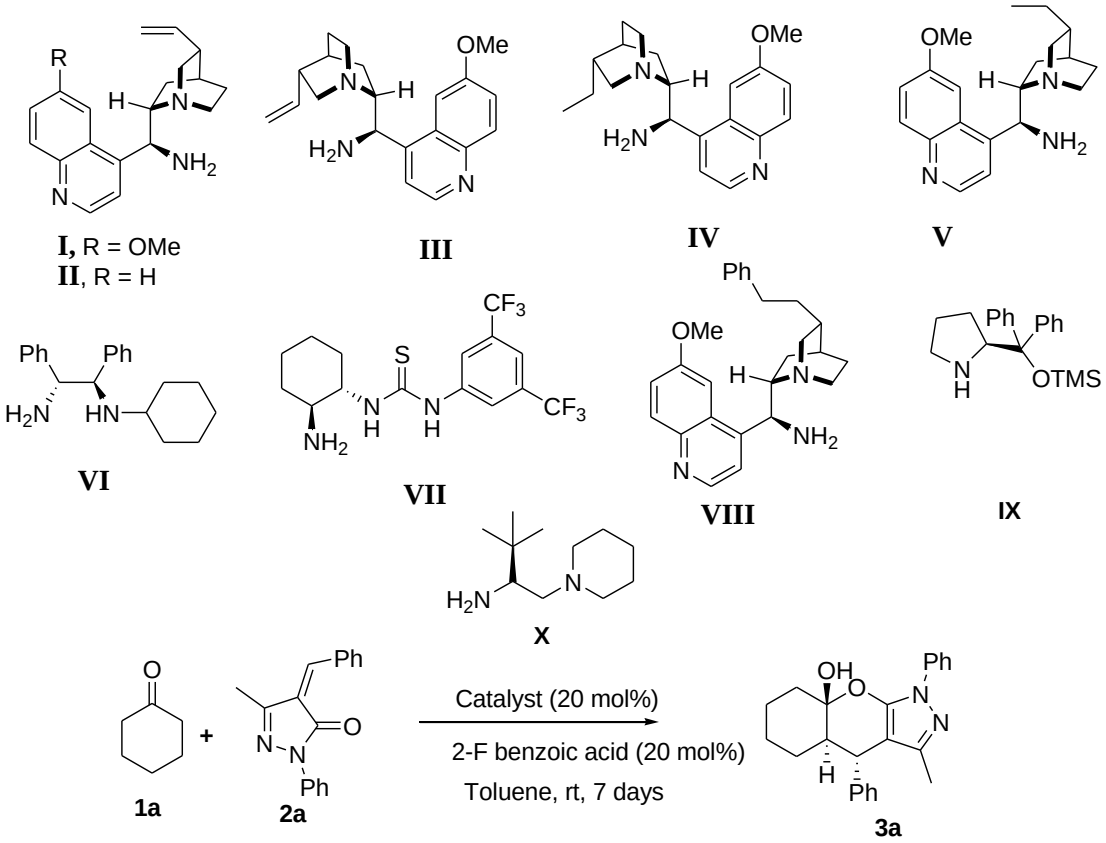
^{1}H NMR spectra were recorded on a 400 MHz and 600 MHz spectrometer at 295 K in $CDCl_3$; chemical shift values (δ, ppm) and coupling constants (Hz) are reported in the standard fashion with reference to either tetramethylsilane (TMS) (δ(H) 0.00 ppm) or CHCl3 (δ(H) 7.26 ppm). ^{13}C NMR spectra were recorded on a 100 MHz and 150 MHz spectrometer at 298 K in $CDCl_3$; chemical shifts (δ, ppm) are reported relative to $CHCl_3$ (δ(C) 77.23 ppm, central line of triplet. In ^{1}H NMR, the following abbreviations are used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, m = multiplet, and br s = broad singlet. IR: spectra were recorded on an FT-IR Instrument at normal temperature making KBr pellet grinding the sample with KBr (IR Grade). High-resolution mass spectra (HR-MS) were recorded in Q-TOF electron spray ionization (ESI). Melting points were obtained with a Mel-Tem capillary melting point apparatus and are uncorrected.

Single crystal X-ray data were collected using Bruker SMART APEXII CCD diffractometer, which is equipped with 1.75 kW sealed-tube Mo-Kα irradiation (λ = 0.71073 Å) at 298(2) K and the structure was solved by direct methods using SHELXS-2014 (Göttingen, Germany)

2. General procedure for the synthesis of compound 3:

To a solution of unsaturated pyrazolones **1** (0.2 mmol), ketone or aldehyde **2** (0.8 mmol) in 2 ml of toluene were added **V** (20 mol%) and 2-F benzoic acid (20 mol%). The reaction mixture was stirred at rt for 7 days. After completion of reaction, the products were purified by silica gel column chromatography (hexane/ethyl acetate).

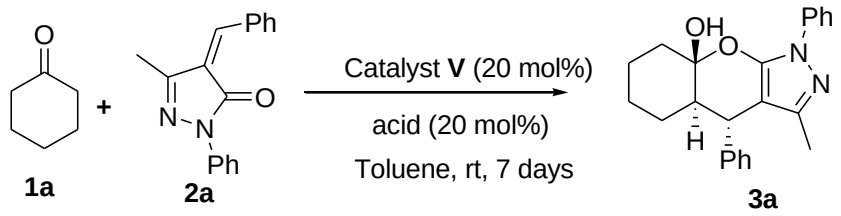
3. **Preliminary catalyst screening (table-1)**:



entry[a]	catalyst	yield (%)[b]	ee(%)[c]
1	**I**	53	72
2	**II**	52	62
3	**III**	50	-50
4	**IV**	52	-85
5	**V**	56	87
6	**VI**	57	34
7	**VII**	45	34
8	**VIII**	55	74
9	**IX**	ND	-
10	**X**	52	-

[a]0.2 mmol of **1a** and 0.05 mmol of **2a** in 0.5 mL solvent using 20 mol% catalyst and 20 mol% acid co-catalyst. [b]Isolated yield after silica gel column chromatography. [c]Determined by HPLC using stationary phase chiral column.

4. **Acid screening (Table 2):**

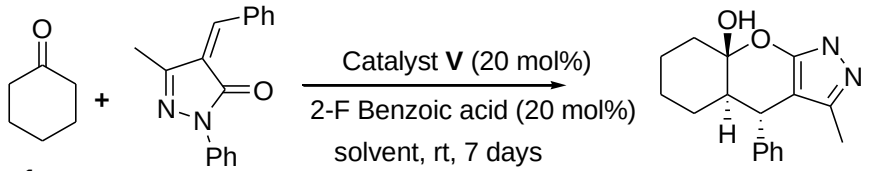


1a **2a** **3a**

entry[a]	acid	yield (%)[b]	ee(%)[c]
1	Benzoic acid	50	56
2	2-F benzoic acid	56	87
3	3-OMe benzoic acid	43	76
4	2,4- dichloro benzoic acid	40	82
5	3-NO_2 benzoic acid	47	48
6	N-Boc l- luicene	50	76
7	TFA	ND	-
8	Acetic acid	45	86

[a]0.2 mmol of **1a** and 0.05 mmol of **2a** in 0.5 mL solvent using 20 mol% catalyst and 20 mol% acid co-catalyst. [b]Isolated yield after silica gel column chromatography. [c]Determined by HPLC using stationary phase chiral column.

5. **Solvent Sceening (Table 3)**:



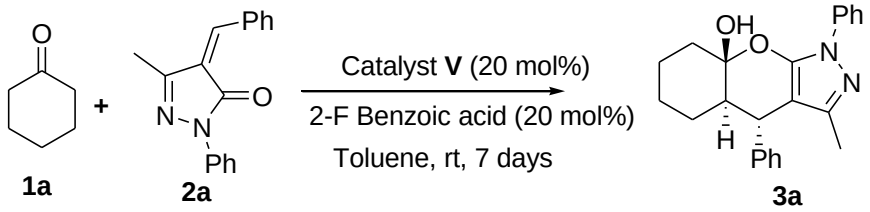
1a **2a** **3a**

entry[a]	solvent	yield (%)[b]	ee(%)[c]
1	Toluene	56	87
2	Benzene	52	86
3	1,4-Dioxane	52	74
4	Trifluorotoluene	50	70
5	Mesitylene	47	76
6	*o*-Xylene	42	78
7	*p*-Xylene	45	80

8	DCM	30	40
9	DCE	35	34
10	MTBE	35	69

[a]0.2 mmol of **1a** and 0.05 mmol of **2a** in 0.5 mL solvent using 20 mol% catalyst and 20 mol% acid co-catalyst. [b]Isolated yield after silica gel column chromatography. [c]Determined by HPLC using stationary phase chiral column.

6. **No of equivalents of Cyclohexanone (1a) used (Table 4)**:



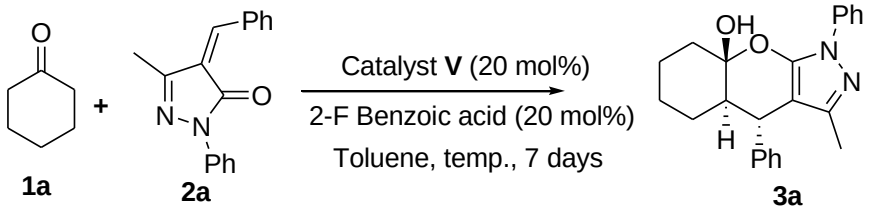
1a **2a** **3a**

x equi. 1 equi.

entry[a]	Cyclohexanone (x eq.)	yield (%)[b]	ee(%)[c]
1	4	56	87
2	5	52	82
3	6	45	78
4	7	42	78
5	2	40	66

[a]x mmol of **1a** and 0.05 mmol of **2a** in 0.5 mL solvent using 20 mol% catalyst and 20 mol% acid co-catalyst. [b]Isolated yield after silica gel column chromatography. [c]Determined by HPLC using stationary phase chiral column.

7. **Temperature screening (Table 5)**:

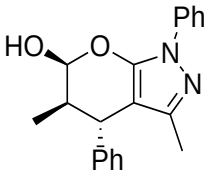


1a **2a** **3a**

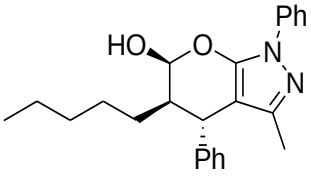
entry[a]	Temp.	yield (%)[b]	ee(%)[c]
1	rt	56	87
2	0 °C	35	87
3	-20 °C	60	60

[a]0.2 mmol of **1a** and 0.05 mmol of **2a** in 0.5 mL solvent using 20 mol% catalyst and 20 mol% acid co-catalyst. [b]Isolated yield after silica gel column chromatography. [c]Determined by HPLC using stationary phase chiral column.

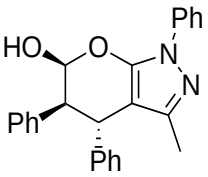
8. **Derivative of different aldehyde substrate (3):**



3za
45 %
72 % ee

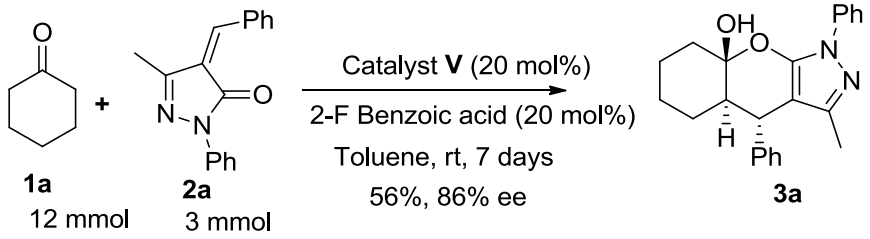


3zb
42 %
30 % ee



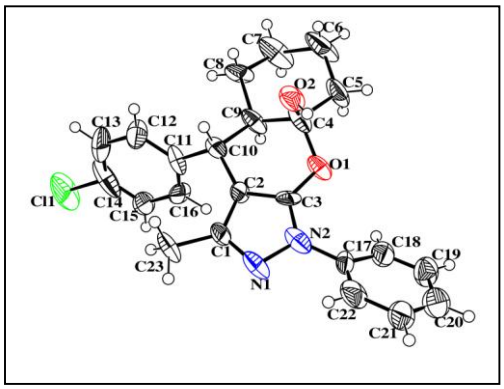
3zc
50 %
40 % ee

9. **Scale up reaction:**

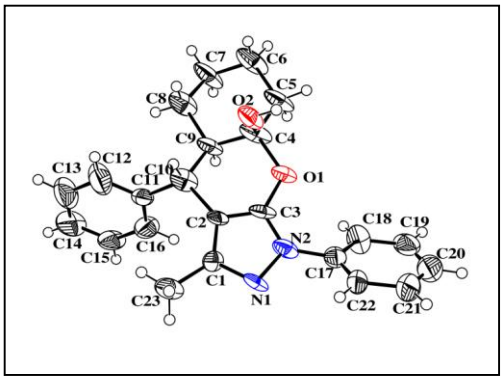


1a 12 mmol
2a 3 mmol
3a

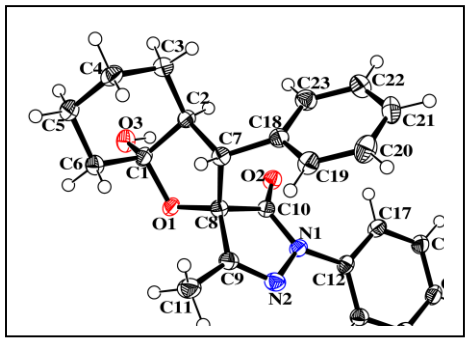
10. **Crystal information**:



Ortep Daigram of compound **3l**



Ortep Daigram of compound **3a**



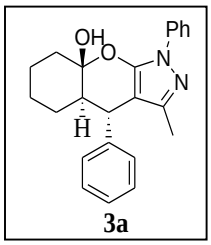
Ortep Daigram of compound 5

Identification code	**3l**	**3a**	**5**
CCDC No	CCDC156055	CCDC1556054	CCDC1556056
Empirical formula	$C_{23}H_{23}ClN_2O_2$	$C_{23}H_{24}N_2O_2$	$C_{23}H_{24}N_2O_3$
Formula weight	394.88	360.44	376.44
Temperature/K	296(2)	296(2)	373.1(2)
Crystal system	orthorhombic	orthorhombic	monoclinic
Space group	$P2_12_12_1$	$P22_12_1$	$P2_1/c$
a/Å	5.9545(7)	5.7189(11)	12.6633(19)
b/Å	12.1404(9)	12.5306(17)	16.796(2)
c/Å	28.388(2)	26.957(3)	9.3590(15)
α/°	90	90	90
β/°	90	90	104.449(16)
γ/°	90	90	90
V/Å^3	2052.2(3)	1931.7(5)	1927.6(5)
Z	4	4	4
D_{calcd} (g m^{-3})	1.278	1.239	1.297
μ (mm^{-1})	0.207	0.079	0.086
F(000)	832.0	768.0	800.0
Reflections collected	4400	4405	22212
Independent reflections	2939[R(int) = 0.1165]	2871[R(int) = 0.0928]	6770[R(int) = 0.0906]
Goodness-of-fit (GOF)[a] on F^2	1.081	1.149	1.017
R_1[b], wR_2[c] ($I \geq 2\sigma(I)$)	0.1216, 0.2518	0.1268, 0.2270	0.0698, 0.1432
R_1[b], wR_2[c] (all data)	0.2029, 0.3503	0.2183, 0.3032	0.1554, 0.1859
Flack parameter	-0.09(13)	-2.7(10)	

[a]GOF = $[\sum[w(F_0^2 - F_c^2)^2] / M - N]^{1/2}$ (M =number of reflections, N = number of parameters refined). [b]$R_1 = \sum \| F_0 | - | Fc \| / \sum | F_0$, [c]$wR_2 = [\sum[w(F_0^2 - F_c^2)^2] / \sum[w(F_0^2)^2]]^{1/2}$

11. **Products Characterizing**:

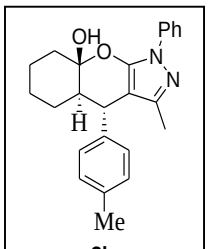
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1,4-diphenylchromeno[2,3-c]pyrazol-8a-ol (**3a**)*



3a

The title was compound according the general procedure. Yellow solid (41 mg, 57% yield); mp 205-206 °C; R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.77 (d, J = 8.5 Hz, 2H), 7.40 (t, J = 7.8 Hz, 2H), 7.31 (dd, J = 16.1, 9.0 Hz, 3H), 7.21 (dd, J = 14.9, 7.3 Hz, 3H), 3.59 (d, J = 10.7 Hz, 1H), 2.72 (s, 1H), 2.12 (d, J = 17.1 Hz, 1H), 1.93 (td, J = 12.9, 3.8 Hz, 2H), 1.85 – 1.63 (m, 4H), 1.61 (s, 3H), 1.33 (dd, J = 21.7, 8.8 Hz, 2H), 1.17 – 1.11 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 148.4, 147.2, 142.0, 138.9, 129.0, 128.6, 126.9, 125.4, 120.5, 103.3, 100.1, 47.9, 40.5, 38.5, 26.9, 25.6, 23.2, 13.5; **ESI-MS**: m/z calcd. for $C_{23}H_{25}N_2O_2^+$ $[M+H]^+$ 361.1911, found 361.1918; **FT-IR**: 3440, 2928, 2855, 1701, 1599, 1515, 1499, 1455, 1387, 1215, 1130, 1051, 1020, 923 cm^{-1}; The ee values 87% (t_{major} = 7.18 min t_{minor} = 9.15 min) and after recrystallization 98% were determined by HPLC using Daicel Chiralpak IB with hexane/i-PrOH (90:10) as the eluent, flow: 1.0 mL/min, 254 nm, 25 °C; **Optical Rotation**: $[\alpha]_D^{28.2}$ = + 16.34 (c 0.355, $CHCl_3$).

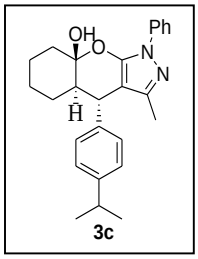
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenyl-4-p-tolylchromeno[2,3-c]pyrazol-8a-ol (**3b**)*



3b

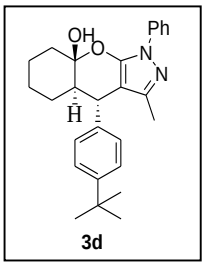
The title was compound according the general procedure. White semi solid (34 mg, 45% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 7.9 Hz, 2H), 7.39 (t, *J* = 7.8 Hz, 2H), 7.19 (t, *J* = 7.4 Hz, 1H), 7.11 (q, *J* = 8.2 Hz, 4H), 3.54 (d, *J* = 10.7 Hz, 1H), 2.94 (s, 1H), 2.35 (s, 3H), 2.06 (dd, *J* = 44.0, 15.7 Hz, 2H), 1.90 (t, *J* = 11.6 Hz, 1H), 1.75 (dd, *J* = 23.4, 11.6 Hz, 3H), 1.62 (s, 3H), 1.57 (d, *J* = 14.4 Hz, 1H), 1.31 (dd, *J* = 24.2, 11.4 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 148.4, 147.4, 138.8, 136.4, 129.3, 129.1, 128.8, 125.4, 120.5, 119.5, 103.3, 102.0, 47.9, 40.1, 38.7, 34.6, 26.9, 25.7, 23.2, 21.3, 13.6; **ESI-MS**: m/z calcd. for $C_{24}H_{27}N_2O_2^+$ $[M+H]^+$ 375.2067, found 375.2069; **FT-IR**: 3440, 2924, 2853, 1701, 1599, 1515, 1496, 1452, 1387, 1215, 1125, 1051, 1020, 921 cm^{-1}; The ee values 80% (t_{major} = 6.45 min, t_{minor} = 26.70 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IC with hexane/i-PrOH (90:10) as the eluent, flow: 1.0 mL/min, 254 nm, 25 °C; **Optical Rotation**: $[\alpha]_D^{29}$ = + 35.18 (c 0.520, $CHCl_3$).

*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-4-(4-isopropylphenyl)-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3c**)*



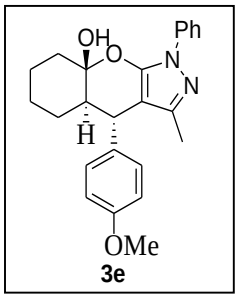
The title was compound according the general procedure. Yellow sticky (50 mg, 62% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, CDCl$_3$)** δ 7.77 (d, *J* = 8.0 Hz, 2H), 7.39 (t, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 7.7 Hz, 1H), 7.14 (dd, *J* = 20.2, 8.0 Hz, 4H), 3.72 (q, *J* = 7.0 Hz, 1H), 3.55 (d, *J* = 10.7 Hz, 1H), 2.88 (ddd, *J* = 22.4, 14.7, 5.9 Hz, 2H), 2.11 (d, *J* = 13.5 Hz, 1H), 1.94 – 1.88 (m, 1H), 1.81 – 1.75 (m, 2H), 1.74 – 1.68 (m, 2H), 1.60 (s, 3H), 1.35 – 1.28 (m, 2H), 1.25 (d, *J* = 6.5 Hz, 6H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 148.4, 147.5, 139.0, 129.2, 128.9, 127.6, 127.4, 126.9, 126.6, 125.5, 125.3, 120.5, 119.7, 103.3, 100.3, 47.8, 42.1, 40.1, 38.5, 33.9, 26.9, 25.7, 24.2, 13.5; **ESI-MS**: m/z calcd. for $C_{26}H_{31}N_2O_2^+$ $[M+H]^+$ 403.2380, found 403.2379; **FT-IR**: 3434, 2920, 2853, 2357, 1725, 1630, 1599, 1511, 1502, 1444, 1380, 1127, 925 cm $^{-1}$; The ee values 84% (t_{major} = 5.86 min, t_{minor} = 23.50 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IC with hexane/i-PrOH (90:10) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{29.5}$ = + 40.38 (c 0.520, $CHCl_3$).

*(4S,4aR,8aS)-4-(4-tert-butylphenyl)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3d**)*



The title was compound according the general procedure. White solid (51 mg, 61% yield); mp- 210-211 ^{o}C; R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, CDCl$_3$)** δ 7.77 (d, *J* = 7.8 Hz, 2H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.13 (d, *J* = 8.2 Hz, 2H), 3.55 (d, *J* = 10.8 Hz, 1H), 2.89 (s, 1H), 2.10 (d, *J* = 15.1 Hz, 1H), 1.91 (td, *J* = 13.7, 4.2 Hz, 1H), 1.82 – 1.67 (m, 4H), 1.59 (s, 3H), 1.32 (s, 9H), 1.27 (d, *J* = 10.7 Hz, 1H), 1.24 (d, *J* = 16.2 Hz, 1H), 1.15 (dt, *J* = 13.2, 3.6 Hz, 1H); **^{13}C NMR (151 MHz, CDCl$_3$)** δ 149.84, 148.30, 147.37, 139.06, 138.60, 129.08, 126.98, 126.38, 125.41, 120.52, 103.22, 100.29, 47.71, 40.02, 38.65, 34.66, 31.64, 27.01, 25.68, 23.23, 13.63; **ESI-MS**: m/z calcd. for $C_{27}H_{33}N_2O_2^+$ $[M+H]^+$ 417.2537, found 417.2542; **FT-IR**: 3440, 2926, 2855, 2357, 1725, 1625, 1599, 1511, 1509, 1452, 1380, 1127, 921 cm $^{-1}$; The ee values 86% (t_{major} = 7.24 min, t_{minor} = 36.64 min) was determined by HPLC using Daicel Chiralpak IC with hexane/i-PrOH (90:10) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{29.2}$ = + 45.22 (c 0.575, $CHCl_3$).

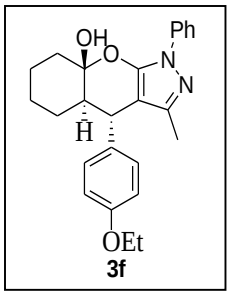
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-4-(4-methoxyphenyl)-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3e**)*



3e

The title was compound according the general procedure. White semi solid (41 mg, 52% yield); R_f value 0.20 (8:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 7.8 Hz, 2H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.13 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 3.81 (s, 3H), 3.54 (d, *J* = 10.7 Hz, 1H), 2.89 (d, *J* = 15.9 Hz, 1H), 2.10 (d, *J* = 13.6 Hz, 1H), 1.91 (td, *J* = 13.7, 4.1 Hz, 1H), 1.77 – 1.70 (m, 3H), 1.63 (s, 3H), 1.57 (d, *J* = 13.9 Hz, 2H), 1.31 (dd, *J* = 12.8, 3.1 Hz, 2H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 158.6, 148.4, 147.3, 138.9, 133.9, 129.0, 125.4, 120.5, 119.5, 113.9, 103.3, 100.3, 55.4, 47.9, 39.6, 38.5, 26.9, 25.7, 23.2, 13.6; **ESI-MS**: m/z calcd. for $C_{24}H_{27}N_2O_3^+$ $[M+H]^+$ 391.2016, found 391.2016; **FT-IR**: 3439, 2923, 2853, 2357, 1723, 1651, 1590, 1517, 1502, 1455, 1390, 1127, 925 cm $^{-1}$; The ee values 82% (t_{major} = 6.31 min, t_{minor} = 20.88 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IC with hexane/i-PrOH (85:15) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.3}$ = + 40.62 (c 0.650, $CHCl_3$).

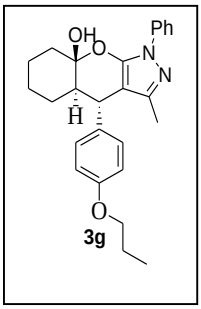
*(4S,4aR,8aS)-4-(4-ethoxyphenyl)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3f**)*



3f

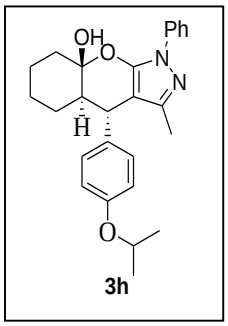
The title was compound according the general procedure. White semi solid (33 mg, 41% yield); R_f value 0.20 (8:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.77 (d, *J* = 7.8 Hz, 2H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.12 (d, *J* = 8.3 Hz, 2H), 6.85 (d, *J* = 8.5 Hz, 2H), 4.03 (dd, *J* = 14.0, 7.0 Hz, 2H), 3.53 (d, *J* = 10.7 Hz, 1H), 2.73 (s, 1H), 2.11 (d, *J* = 13.5 Hz, 1H), 1.91 (td, *J* = 13.6, 3.5 Hz, 1H), 1.79 – 1.66 (m, 4H), 1.64 (s, 3H), 1.42 (t, *J* = 6.9 Hz, 3H), 1.35 – 1.28 (m, 2H), 1.17 – 1.13 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 158.0, 147.4, 138.8, 133.7, 129.9, 129.1, 125.7, 125.6, 120.6, 119.1, 114.6, 114.3, 63.6, 47.9, 39.7, 32.2, 32.0, 25.7, 22.9, 15.1, 13.5; **ESI-MS**: m/z calcd. for $C_{25}H_{29}N_2O_3^+$ $[M+H]^+$ 405.2173, found 405.2185; **FT-IR**: 3443, 2929, 2855, 1597, 1514, 1498, 1454, 1381, 1244, 1123, 1050, 1018, 923 cm $^{-1}$; The ee values 88% (t_{major} = 42.70 min, t_{minor} = 51.46 min) and after recrystallization 98% were determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (99:1) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{29.6}$ = + 28.57 (c 0.210, $CHCl_3$).

*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenyl-4-(4-propoxyphenyl)chromeno[2,3-c]pyrazol-8a-ol (**3g**)*



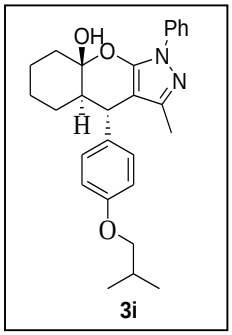
The title was compound according the general procedure. White solid (35 mg, 42% yield); mp 200-201°C; R_f value 0.20 (8:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.77 (d, *J* = 7.9 Hz, 2H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.12 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.5 Hz, 2H), 3.92 (t, *J* = 6.6 Hz, 2H), 3.53 (d, *J* = 10.7 Hz, 1H), 2.81 (s, 1H), 2.10 (d, *J* = 16.8 Hz, 1H), 1.91 (td, *J* = 13.7, 4.2 Hz, 1H), 1.81 (dd, *J* = 14.0, 6.9 Hz, 2H), 1.74 (ddd, *J* = 23.0, 20.4, 13.3 Hz, 4H), 1.64 (s, 3H), 1.57 (s, 1H), 1.34 – 1.27 (m, 1H), 1.14 (ddd, *J* = 16.7, 13.1, 9.7 Hz, 1H), 1.05 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 158.1, 148.4, 147.4, 138.9, 133.7, 129.7, 129.0, 125.4, 120.5, 114.5, 103.3, 100.4, 69.6, 47.9, 39.6, 38.5, 26.9, 25.7, 23.2, 22.8, 13.53, 10.8; **ESI-MS**: m/z calcd. for $C_{26}H_{31}N_2O_3^+$ $[M+H]^+$ 419.2329, found 419.2319; **FT-IR**: 3115, 2922, 2853, 1705, 1602, 1520, 1500, 1453, 1389, 1260, 1217, 1124, 1051, 925 cm $^{-1}$; The ee values 82% (t_{major} = 7.09 min, t_{minor} = 8.30 min) and was determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 °C; **Optical Rotation**: $[\alpha]_D^{29.8}$ = + 42.26 (c 0.530, $CHCl_3$).

*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-4-(4-isopropoxyphenyl)-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3h**)*



The title was compound according the general procedure. White semi solid (29 mg, 35% yield); R_f value 0.20 (8:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 7.6 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.21 – 7.18 (m, 1H), 7.11 (d, *J* = 7.8 Hz, 2H), 6.83 (d, *J* = 8.2 Hz, 2H), 4.56 – 4.52 (m, 1H), 3.52 (d, *J* = 10.7 Hz, 1H), 2.34 – 2.31 (m, 1H), 2.11 (dd, *J* = 26.1, 9.4 Hz, 2H), 2.02 (t, *J* = 9.4 Hz, 1H), 1.92 (dd, *J* = 24.8, 11.9 Hz, 2H), 1.87 – 1.80 (m, 2H), 1.64 (s, 3H), 1.33 (d, *J* = 3.4 Hz, 6H), 1.15 (d, *J* = 10.8 Hz, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 156.9, 148.3, 133.6, 129.9, 129.1, 129.0, 125.4, 120.5, 119.1, 116.0, 103.2, 100.3, 70.0, 47.9, 39.7, 38.6, 32.1, 26.9, 25.7, 22.3, 13.6; **ESI-MS**: m/z calcd. for $C_{26}H_{31}N_2O_2^+$ $[M+H]^+$ 419.2329, found 419.2328; **FT-IR:** 3443, 2925, 2852, 1715, 1603, 1508, 1454, 1384, 1266, 1238, 1028, 955 cm $^{-1}$; The ee values 84% (t_{major} = 10.70 min, t_{minor} = 13.59 min) and after recrystallization 96% were determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (97:3) as the eluent, flow: 1.0 mL/min, 254 nm, 25 °C; **Optical Rotation**: $[\alpha]_D^{29.4}$ = + 22.86 (c 0.210, $CHCl_3$).

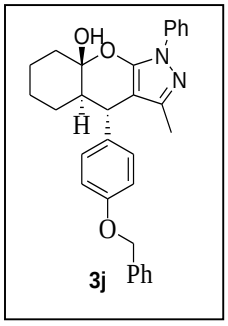
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-4-(4-isobutoxyphenyl)-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3i**)*



3i

The title was compound according the general procedure. White solid (36 mg, 42% yield); mp 199-200°C; R_f value 0.20 (8:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.79 (d, *J* = 8.0 Hz, 2H), 7.42 (t, *J* = 7.8 Hz, 2H), 7.22 (t, *J* = 7.4 Hz, 1H), 7.14 (d, *J* = 8.3 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 3.74 (d, *J* = 6.5 Hz, 2H), 3.55 (d, *J* = 10.7 Hz, 1H), 2.87 (s, 1H), 2.11 (dt, *J* = 13.1, 10.1 Hz, 2H), 1.93 (td, *J* = 13.6, 4.0 Hz, 1H), 1.80 – 1.73 (m, 3H), 1.67 (s, 3H), 1.60 (dd, *J* = 13.0, 2.3 Hz, 1H), 1.35 (dd, *J* = 12.6, 3.4 Hz, 1H), 1.31 (d, *J* = 4.3 Hz, 1H), 1.18 – 1.14 (m, 1H), 1.06 (d, *J* = 6.7 Hz, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 158.3, 148.4, 147.3, 139.0, 133.6, 129.8, 129.0, 125.3, 120.5, 114.6, 103.3, 100.4, 74.6, 48.0, 39.5, 38.5, 28.5, 26.9, 25.7, 23.2, 19.52, 13.7; **ESI-MS**: m/z calcd. for $C_{27}H_{33}N_2O_3^+$ $[M+H]^+$ 433.2484, found 433.2484; **FT-IR**: 3424, 2924, 2855, 1693, 1603, 1570, 1521, 1489, 1458, 1384, 1275, 1069, 1025 cm $^{-1}$; The ee values 82% (t_{major} = 15.12 min, t_{minor} = 20.09 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (98:2) as the eluent, flow: 1.0 mL/min, 254 nm, 25 °C; **Optical Rotation**: $[\alpha]_D^{30.2}$ = + 34.69 (c 0.490, $CHCl_3$).

*(4S,4aR,8aS)-4-(4-(benzyloxy)phenyl)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3j**)*

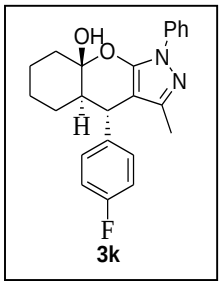


3j

The title was compound according the general procedure. White solid (50 mg, 54% yield); mp 190-191°C; R_f value 0.20 (8:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 7.5 Hz, 2H), 7.45 (d, *J* = 4.3 Hz, 2H), 7.39 (t, *J* = 7.1 Hz, 4H), 7.34 (d, *J* = 7.1 Hz, 1H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.14 (d, *J* = 7.7 Hz, 2H), 6.94 (d, *J* = 8.0 Hz, 2H), 5.05 (s, 2H), 3.54 (d, *J* = 10.8 Hz, 1H), 2.82 (s, 1H), 2.14 – 2.11 (m, 1H), 1.91 (t, *J* = 13.6 Hz, 1H), 1.76 – 1.69 (m, 3H), 1.64 (s, 1H), 1.58 (d, *J* = 13.9 Hz, 2H), 1.33 – 1.28 (m, 1H), 1.14 (d, *J* = 15.6 Hz, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 157.8, 137.2, 129.0, 128.7, 128.2, 127.8, 125.4, 120.5, 118.9, 114.9, 103.3, 100.3, 70.2, 47.9, 39.6, 38.5, 29.9, 25.6, 23.2, 13.6; **ESI-MS**: m/z calcd. for $C_{30}H_{31}N_2O_3^+$ $[M+H]^+$ 467.2329, found 467.2329; **FT-IR**: 3429, 2924, 2855, 1693, 1609, 1575, 1521, 1489, 1458, 1388, 1275, 1069, 1025 cm $^{-1}$; The ee values 84% (t_{major} = 10.08 min, t_{minor} = 14.38 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IC

with hexane/EtOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.9}$ = + 33.09 (c 0.810, $CHCl_3$).

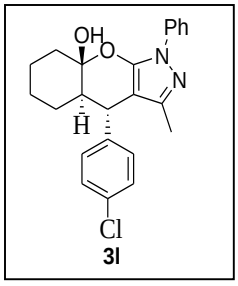
*(4S,4aR,8aS)-4-(4-fluorophenyl)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3k**)*



3k

The title was compound according the general procedure. White semi solid (40 mg, 53% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 7.8 Hz, 2H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.22 – 7.16 (m, 3H), 7.01 (t, *J* = 8.5 Hz, 2H), 3.58 (d, *J* = 10.7 Hz, 1H), 2.95 (s, 1H), 2.10 (d, *J* = 11.7 Hz, 1H), 1.91 (td, *J* = 13.7, 4.1 Hz, 1H), 1.79 – 1.65 (m, 4H), 1.61 (s, 3H), 1.53 (d, *J* = 12.7 Hz, 1H), 1.32 (dd, *J* = 27.2, 14.5 Hz, 1H), 1.14 (dd, *J* = 24.7, 11.6 Hz, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 163.2, 160.7, 147.3, 139.0, 137.8, 135.1, 132.9, 130.5, 129.1, 128.9, 125.6, 120.7, 119.5, 115.6, 115.4, 103.2, 99.9, 48.2, 43.9, 39.9, 26.9, 25.5, 23.2, 13.1; **ESI-MS**: m/z calcd. for $C_{23}H_{24}FN_2O_2^+$ $[M+H]^+$ 379.1816, found 379.1816; **FT-IR**: 3121, 2923, 2853, 1700, 1610, 1520, 1496, 1456, 1389, 1260, 1217, 1124, 1051, 948 cm $^{-1}$; The ee values 84% (t_{major} = 39.727 min, t_{minor} = 54.54 min) .was determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (99:1) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.0}$ = + 21.29 (c 0.565, $CHCl_3$).

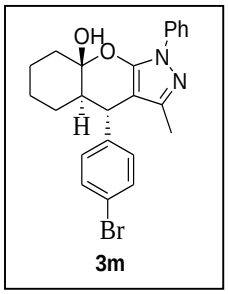
*(4S,4aR,8aS)-4-(4-chlorophenyl)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3l**)*



3l

The title was compound according the general procedure. White semi solid (35 mg, 44% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 8.0 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.29 (d, *J* = 7.4 Hz, 2H), 7.21 (d, *J* = 7.2 Hz, 1H), 7.18 – 7.15 (m, 2H), 3.58 (d, *J* = 10.5 Hz, 1H), 2.19 – 2.11 (m, 1H), 1.95 – 1.84 (m, 1H), 1.75 (s, 2H), 1.62 (s, 3H), 1.52 (d, *J* = 11.4 Hz, 1H), 1.16 – 1.03 (m, 4H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 148.4, 146.9, 140.7, 132.7, 131.6, 129.1, 128.8, 125.6, 120.6, 119.5, 103.2, 99.8, 47.9, 40.1, 38.6, 32.1, 26.9, 22.9, 14.3; **ESI-MS**: m/z calcd. for $C_{23}H_{24}ClN_2O_2^+$ $[M+H]^+$ 395.1521, found 395.1520; **FT-IR**: 3464, 2922, 2855, 1708, 1597, 1517, 1492, 1451, 1390, 1273, 1216, 1120, 1095, 917 cm $^{-1}$; The ee values 82% (t_{major} = 6.38 min, t_{minor} = 20.04 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IC with hexane/i-PrOH (90:10) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C.

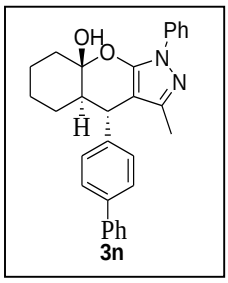
*(4S,4aR,8aS)-4-(4-bromophenyl)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3m**)*



3m

The title was compound according the general procedure. White solid (47 mg, 41% yield); mp 205-206 ^{o}C; R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 7.7 Hz, 2H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.11 (d, *J* = 8.3 Hz, 2H), 3.57 (d, *J* = 10.7 Hz, 1H), 2.87 (s, 1H), 2.12 – 2.07 (m, 1H), 1.91 (td, *J* = 13.7, 4.2 Hz, 1H), 1.77 – 1.69 (m, 3H), 1.63 (s, 3H), 1.52 (d, *J* = 11.8 Hz, 1H), 1.38 – 1.22 (m, 2H), 1.13 (dt, *J* = 13.2, 3.6 Hz, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** 148.7, 147.0, 141.2, 138.9, 131.8, 130.6, 129.1, 125.6, 120.7, 120.5, 103.2, 99.7, 47.8, 40.1, 38.6, 26.9, 25.6, 23.1, 13.7; **ESI-MS**: m/z calcd. for $C_{23}H_{24}BrN_2O_2^+$ $[M+H]^+$ 439.1016, found 439.1016; **FT-IR:** 3439, 2929, 2855, 1603, 1521, 1483, 1454, 1387, 1371, 1273, 1212, 1123, 1069, 923 cm $^{-1}$; The ee values 78% (t_{major} = 8.40 min, t_{minor} = 9.95 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.4}$ = + 71.95 (c 0.397, $CHCl_3$).

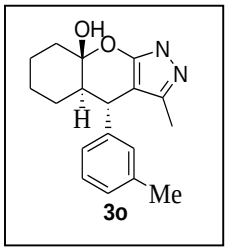
*(4S,4aR,8aS)-4-(biphenyl)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3n**)*



3n

The title was compound according the general procedure. White semi solid (54 mg, 62% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.77 (t, *J* = 8.6 Hz, 3H), 7.41 (dt, *J* = 15.8, 7.8 Hz, 4H), 7.20 (dd, *J* = 16.5, 9.2 Hz, 2H), 7.04 – 7.00 (m, 1H), 6.99 (s, 2H), 6.96 (s, 1H), 6.92 (dd, *J* = 17.3, 10.2 Hz, 1H), 3.94 (d, *J* = 10.8 Hz, 1H), 2.85 (s, 1H), 2.11 (d, *J* = 13.3 Hz, 2H), 1.92 (td, *J* = 13.8, 4.0 Hz, 2H), 1.88 – 1.81 (m, 2H), 1.75 (d, *J* = 14.3 Hz, 1H), 1.57 (s, 3H), 1.39 – 1.36 (m, 1H), 1.16 – 1.13 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 148.4, 147.3, 141.1, 140.9, 139.8, 139.0, 129.0, 128.9, 127.4, 127.3, 127.2, 125.4, 120.5, 119.5, 103.2, 100.2, 47.9, 40.2, 38.7, 27.0, 25.7, 23.2, 13.7; **ESI-MS**: m/z calcd. for $C_{29}H_{29}N_2O_2^+$ $[M+H]^+$ 437.2224, found 437.2216; **FT-IR:** 3455, 2933, 2851, 1601, 1518, 1497, 1454, 1391, 1274, 1211, 1024, 919 cm^{-1}; The ee values 80% (t_{major} = 8.55 min, t_{minor} = 11.25 min) and after recrystallization 98% were determined by HPLC using Daicel Chiralpak IB with hexane/i-PrOH (90:10) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.8}$ = + 39.05 (c 0.210, $CHCl_3$).

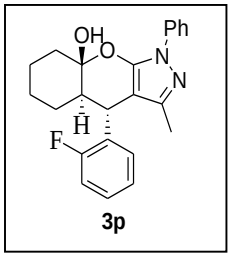
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenyl-4-m-tolylchromeno[2,3-c]pyrazol-8a-ol (**3o**)*



3o

The title was compound according the general procedure. White semi solid (51 mg, 65% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 7.8 Hz, 2H), 7.38 (t, *J* = 7.9 Hz, 2H), 7.19 (t, *J* = 7.3 Hz, 2H), 7.06 (d, *J* = 7.7 Hz, 1H), 7.01 (s, 2H), 3.53 (d, *J* = 10.7 Hz, 1H), 3.22 (s, 1H), 2.33 (s, 3H), 2.09 (d, *J* = 13.4 Hz, 1H), 1.89 (td, *J* = 13.6, 3.9 Hz, 1H), 1.81 – 1.73 (m, 2H), 1.69 (d, *J* = 17.0 Hz, 2H), 1.60 (s, 3H), 1.56 (d, *J* = 13.2 Hz, 1H), 1.31 (dd, *J* = 12.8, 3.1 Hz, 1H), 1.15 – 1.11 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.5, 147.4, 141.9, 138.9, 138.1, 129.0, 128.9, 128.4, 127.7, 125.4, 120.5, 119.6, 103.3, 100.2, 47.8, 40.4, 38.5, 26.9, 25.6, 24.9, 23.2, 13.5; **ESI-MS**: m/z calcd. for $C_{24}H_{27}N_2O_2^+$ $[M+H]^+$ 375.2067, found 375.2060; **FT-IR**: 3431, 2929, 2852, 1711, 1597, 1514, 1492, 1463, 1394, 1371, 1273, 1209, 1022, 917 cm $^{-1}$; The ee values 80% (t_{major} = 9.17 min, t_{minor} = 13.64 min) and after recrystallization 96% were determined by HPLC using Daicel Chiralpak IB with hexane/i-PrOH (94:6) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.8}$ = + 22.79 (c 0.825, $CHCl_3$).

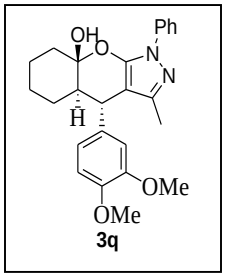
*(4S,4aR,8aS)-4-(3-fluorophenyl)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3p**)*



3p

The title was compound according the general procedure. White semi solid (33 mg, 44% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 7.8 Hz, 2H), 7.40 (t, *J* = 7.9 Hz, 2H), 7.21 (td, *J* = 12.3, 6.5 Hz, 3H), 7.10 (t, *J* = 7.5 Hz, 2H), 4.17 (d, *J* = 10.4 Hz, 1H), 2.85 (s, 1H), 2.13 – 2.08 (m, 1H), 1.96 – 1.88 (m, 1H), 1.74 (t, *J* = 15.8 Hz, 3H), 1.61 (s, 3H), 1.55 (s, 2H), 1.49 – 1.41 (m, 1H), 1.18 – 1.14 (m, 1H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 148.9, 146.8, 139.0, 129.1, 128.3, 125.5, 124.7, 124.3, 120.6, 120.4, 119.7, 115.8, 115.6, 115.4, 115.3, 103.3, 99.3, 49.5, 38.5, 29.5, 27.9, 26.9, 25.6, 23.1, 13.1; **ESI-MS**: m/z calcd. for $C_{23}H_{24}FN_2O_2^+$ $[M+H]^+$ 379.1816, found 379.1816; **FT-IR**: 3119, 2923, 2853, 1700, 1602, 1520, 1496, 1453, 1389, 1265, 1217, 1124, 1051, 948 cm $^{-1}$; The ee values 78% (t_{major} = 4.99 min, t_{minor} = 7.20 min) and after recrystallization 98% were determined by HPLC using Daicel Chiralpak IC with hexane/EtOH (90:10) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.0}$ = + 30.98 (c 0.510, $CHCl_3$).

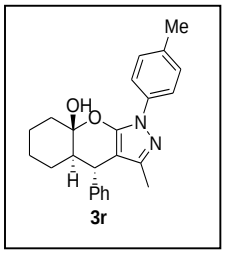
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-4-(3,4-dimethoxyphenyl)-3-methyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3q**)*



3q

The title was compound according the general procedure. Yellow semi solid (35 mg, 42% yield); R_f value 0.20 (5:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.78 (d, *J* = 7.7 Hz, 2H), 7.39 (t, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 7.4 Hz, 1H), 6.80 (d, *J* = 8.2 Hz, 2H), 6.69 (s, 1H), 3.88 (s, 3H), 3.83 (s, 3H), 3.53 (d, *J* = 10.8 Hz, 1H), 2.91 (s, 1H), 2.13 – 2.08 (m, 1H), 1.92 (td, *J* = 13.7, 4.1 Hz, 1H), 1.78 – 1.68 (m, 4H), 1.66 (s, 3H), 1.58 (d, *J* = 12.5 Hz, 1H), 1.31 (ddd, *J* = 16.0, 13.2, 3.4 Hz, 1H), 1.15 (dt, *J* = 13.3, 3.5 Hz, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 149.2, 148.5, 147.9, 147.4, 138.9, 134.4, 129.0, 125.3, 120.4, 119.0, 114.2, 110.9, 103.4, 100.2, 56.1, 55.9, 47.8, 42.1, 27.1, 25.7, 25.1, 23.2, 13.5; **ESI-MS**: m/z calcd. for $C_{25}H_{29}N_2O_4^+$ $[M+H]^+$ 421.2122, found 421.2113; **FT-IR**: 3464, 2925, 2852, 1702, 1594, 1514, 1454, 1387, 1263, 1235, 1142, 1025, 914 cm $^{-1}$; The ee values 82% (t_{major} = 7.70 min, t_{minor} = 10.07 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IB with hexane/i-PrOH (85:15) as the eluent, flow: 1.0 mL/min, 254 nm, 25 °C; **Optical Rotation**: $[\alpha]_D^{31.1}$ = + 37.60 (c 0.250, $CHCl_3$).

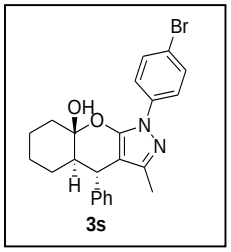
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-4-phenyl-1-p-tolylchromeno[2,3-c]pyrazol-8a-ol (**3r**)*



3r

The title was compound according the general procedure. White solid (75 mg, 56%); mp 188-189 °C; R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.63 (d, *J* = 8.3 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.25 (s, 1H), 7.23 (d, *J* = 7.2 Hz, 2H), 7.20 (d, *J* = 8.2 Hz, 2H), 3.58 (d, *J* = 10.8 Hz, 1H), 2.78 (s, 1H), 2.36 (s, 3H), 2.10 (d, *J* = 13.5 Hz, 1H), 1.91 (td, *J* = 13.7, 4.2 Hz, 1H), 1.82 – 1.77 (m, 1H), 1.72 (dd, *J* = 27.1, 13.2 Hz, 3H), 1.60 (s, 3H), 1.56 (s, 1H), 1.33 (dd, *J* = 24.0, 11.2 Hz, 1H), 1.13 (dd, *J* = 26.3, 13.0 Hz, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 142.1, 135.2, 135.1, 132.8, 129.6, 128.9, 128.6, 126.9, 124.1, 120.8, 119.6, 117.1, 49.5, 42.9, 40.6, 34.5, 25.6, 22.8, 21.1, 13.4; **ESI-MS**: m/z calcd. for $C_{24}H_{27}N_2O_2^+$ $[M+H]^+$ 375.2067, found 375.2067; **FT-IR**: 3435, 2925, 2850, 1721, 1705, 1599, 1518, 1505, 1455, 1388, 1129, 923 cm $^{-1}$; The ee values 78% (t_{major} = 6.96 min, t_{minor} = 11.63 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 °C; **Optical Rotation**: $[\alpha]_D^{30.5}$ = + 14.63 (c 0.410, $CHCl_3$).

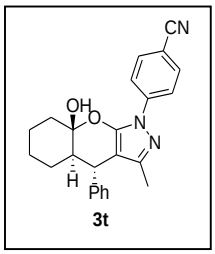
*(4S,4aR,8aS)-1-(4-bromophenyl)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-4-phenylchromeno[2,3-c]pyrazol-8a-ol (**3s**)*



3s

The title was compound according the general procedure. White solid (63 mg, 72% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.67 (d, *J* = 8.6 Hz, 2H), 7.50 (d, *J* = 8.6 Hz, 2H), 7.32 (t, *J* = 7.3 Hz, 2H), 7.27 (s, 1H), 7.21 (d, *J* = 7.1 Hz, 2H), 3.57 (d, *J* = 10.7 Hz, 1H), 2.91 (s, 1H), 2.14 – 2.07 (m, 1H), 1.92 (td, *J* = 13.7, 4.0 Hz, 1H), 1.83 – 1.66 (m, 4H), 1.64 (d, *J* = 3.6 Hz, 1H), 1.58 (s, 3H), 1.33 (dd, *J* = 25.5, 12.9 Hz, 1H), 1.17 – 1.12 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.9, 141.7, 137.8, 135.5, 132.9, 132.2, 128.7, 127.1, 124.3, 121.8, 120.7, 117.2, 49.8, 40.5, 34.4, 32.1, 25.9, 25.6, 13.5; **ESI-MS:** m/z calcd. for $C_{23}H_{24}BrN_2O_2^+$ $[M+H]^+$ 439.1016, found 439.1016; **FT-IR:** 3439, 2925, 2855, 1605, 1525, 1483, 1453, 1386, 1371, 1272, 1212, 1123, 1069, 923 cm^{-1}; The ee values 84% (t_{major} = 6.48 min, t_{minor} = 7.31 min) was determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 °C; **Optical Rotation**: $[\alpha]_D^{31.0}$ = + 19.88 (c 0.835, $CHCl_3$).

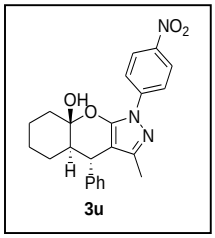
*4-((4S,4aR,8aS)-4a,5,6,7,8,8a-hexahydro-8a-hydroxy-3-methyl-4-phenylchromeno[2,3-c]pyrazol-1(4H)-yl)benzonitrile (**3t**)*



3t

The title was compound according the general procedure. Yellow solid (45 mg, 59% yield); mp 195-200 °C; R_f value 0.20 (8:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.96 (d, *J* = 7.7 Hz, 2H), 7.65 (d, *J* = 7.6 Hz, 2H), 7.32 (d, *J* = 6.6 Hz, 2H), 7.27 (d, *J* = 6.8 Hz, 1H), 7.21 (d, *J* = 5.4 Hz, 2H), 3.58 (d, *J* = 10.4 Hz, 1H), 3.02 (s, 1H), 2.16 (d, *J* = 12.4 Hz, 1H), 1.97 (dd, *J* = 22.8, 12.5 Hz, 1H), 1.79 (d, *J* = 14.7 Hz, 3H), 1.70 (dd, *J* = 23.1, 15.9 Hz, 2H), 1.61 (s, 3H), 1.34 (dd, *J* = 24.8, 12.8 Hz, 1H), 1.19 – 1.12 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 149.4, 149.2, 142.4, 141.4, 133.2, 128.7, 127.1, 119.5, 119.1, 118.5, 107.6, 104.1, 101.5, 47.7, 40.3, 38.5, 26.9, 25.6, 23.2, 13.6; **ESI-MS**: m/z calcd. for $C_{24}H_{24}N_3O_3^+$ $[M+H]^+$ 386.1863, found 386.1863; **FT-IR**: 3453, 2922, 2855, 2229, 1603, 1517, 1495, 1444, 1409, 1394, 1270, 1123, 1098, 917 cm^{-1}; The ee values 84% (t_{major} = 11.14 min, t_{minor} = 13.17 min) was determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 °C; **Optical Rotation**: $[\alpha]_D^{30.6}$ = + 39.74 (c 0.765, $CHCl_3$).

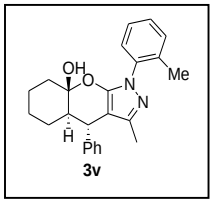
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1-(4-nitrophenyl)-4-phenylchromeno[2,3-c]pyrazol-8a-ol (**3u**)*



3u

The title was compound according the general procedure. Yellow solid (26 mg, 35% yield); R_f value 0.20 (6:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.27 (d, *J* = 9.2 Hz, 3H), 8.03 (d, *J* = 9.1 Hz, 2H), 7.33 (t, *J* = 7.7 Hz, 2H), 7.22 (d, *J* = 7.1 Hz, 2H), 3.60 (d, *J* = 10.8 Hz, 1H), 2.85 (s, 1H), 2.38 – 2.32 (m, 1H), 2.02 – 1.96 (m, 2H), 1.82 – 1.76 (m, 2H), 1.75 – 1.69 (m, 2H), 1.61 (s, 3H), 1.36 – 1.34 (m, 1H), 1.16 (dd, *J* = 10.7, 6.7 Hz, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 148.6, 144.1, 134.0, 133.9, 129.1, 128.8, 125.1, 125.0, 119.1, 118.0, 104.2, 101.7, 47.7, 40.3, 38.7, 26.9, 25.5, 22.9, 14.4; **ESI-MS**: m/z calcd. for $C_{23}H_{24}N_3O_4^+$ $[M+H]^+$ 406.1761, found 406.1761; **FT-IR**: 3453, 2923, 1701, 1614, 1599, 1567, 1521, 1339, 1110, 1092, 916 cm $^{-1}$; The ee values 82% (t_{major} = 10.44 min, t_{minor} = 11.86 min) was determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.3}$ = + 51.43 (c 0.105, $CHCl_3$).

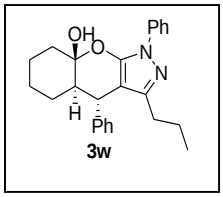
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-4-phenyl-1-o-tolylchromeno[2,3-c]pyrazol-8a-ol (**3v**)*



3v

The title was compound according the general procedure. White solid (56 mg, 75% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.33 (t, *J* = 7.4 Hz, 4H), 7.28 (s, 2H), 7.24 (d, *J* = 7.2 Hz, 3H), 3.61 (d, *J* = 10.7 Hz, 1H), 2.83 (s, 1H), 2.24 (s, 2H), 1.95 (d, *J* = 13.4 Hz, 1H), 1.79 – 1.73 (m, 3H), 1.67 (d, *J* = 11.2 Hz, 3H), 1.59 (s, 3H), 1.31 (dd, *J* = 19.2, 9.1 Hz, 1H), 1.10 (d, *J* = 13.3 Hz, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 142.3, 135.6, 134.8, 132.7, 130.9, 128.6, 127.8, 126.9, 126.4, 124.0, 117.2, 116.9, 49.6, 42.1, 40.6, 29.9, 25.6, 25.1, 18.1, 13.2; **ESI-MS**: m/z calcd. for $C_{24}H_{27}N_2O_2^+$ $[M+H]^+$ 375.2067, found 375.2067; **FT-IR**: 3439, 2923, 2853, 1723, 1701, 1599, 1515, 1500, 1451, 1388, 1127, 923 cm $^{-1}$; The ee values 88% (t_{major} = 19.26 min, t_{minor} = 21.46 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IC with hexane/EtOH (98:2) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.9}$ = + 54.13 (c 0.248, $CHCl_3$).

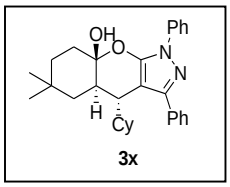
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-1,4-diphenyl-3-propylchromeno[2,3-c]pyrazol-8a-ol (**3w**)*



3w

The title was compound according the general procedure. White sticky (30 mg, 38% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, CDCl$_3$)** δ 7.91 (d, *J* = 7.7 Hz, 2H), 7.45 (dd, *J* = 14.8, 7.2 Hz, 3H), 7.35 (d, *J* = 4.4 Hz, 2H), 7.09 (t, *J* = 7.9 Hz, 2H), 7.03 (s, 1H), 3.89 (d, *J* = 10.6 Hz, 1H), 3.72 (dd, *J* = 14.1, 7.0 Hz, 1H), 2.86 (s, 1H), 2.16 (d, *J* = 13.8 Hz, 2H), 1.95 (d, *J* = 13.8 Hz, 1H), 1.78 – 1.70 (m, 4H), 1.60 (s, 4H), 1.40 (dd, *J* = 23.6, 10.9 Hz, 2H), 1.13 (dd, *J* = 24.6, 14.1 Hz, 2H), 0.88 (t, *J* = 6 Hz, 3H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 149.4, 141.9, 139.0, 133.4, 129.1, 128.0, 127.8, 127.5, 125.9, 121.0, 102.9, 99.4, 48.4, 42.9, 41.4, 38.6, 32.1, 27.2, 25.6, 23.2, 14.3; **ESI-MS**: m/z calcd. for $C_{25}H_{29}N_2O_4^+$ $[M+H]^+$ 389.2224, found 389.2219; **FT-IR**: 3424, 2925, 2855, 1699, 1600, 1575, 1511, 1489, 1457, 1384, 1276, 1069, 1025 cm $^{-1}$; The ee values 84% (t_{major} = 7.52 min, t_{minor} = 11.52 min) and after recrystallization 98% were determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.2}$ = + 27.83 (c 0.115, $CHCl_3$).

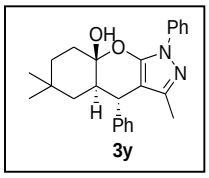
*(4R,4aR,8aS)-4-cyclohexyl-1,4,4a,5,6,7,8,8a-octahydro-1,3-diphenylchromeno[2,3-c]pyrazol-8a-ol (**3x**)*



3x

The title was compound according the general procedure. White solid (31 mg, 36% yield); R_f value 0.20 (12:1 hex/EA); **^{1}H NMR (600 MHz, CDCl$_3$)** δ 7.76 (d, *J* = 7.4 Hz, 2H), 7.41 (t, *J* = 7.8 Hz, 3H), 7.29 (d, *J* = 6.9 Hz, 1H), 7.24 (d, *J* = 7.4 Hz, 1H), 7.18 (s, 1H), 7.15 (t, *J* = 7.4 Hz, 2H), 3.75 – 3.69 (m, 1H), 3.51 (d, *J* = 11.0 Hz, 1H), 2.61 – 2.51 (m, 1H), 2.03 (dd, *J* = 14.3, 6.7 Hz, 1H), 1.69 – 1.62 (m, 5H), 1.58 (s, 5H), 1.08 – 1.01 (m, 2H), 0.88 (t, *J* = 6.9 Hz, 2H), 0.84 – 0.78 (m, 2H), 0.66 (dd, *J* = 22.0, 9.1 Hz, 2H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 150.5, 139.4, 137.4, 128.9, 128.8, 128.6, 127.3, 126.3, 121.5, 119.0, 114.3, 91.0, 42.2, 36.9, 36.1, 34.0, 32.7, 32.1, 29.6, 27.2, 26.6, 25.2, 22.9, 14.3; **ESI-MS**: m/z calcd. for $C_{28}H_{33}N_2O_2^+$ $[M+H]^+$ 429.2537, found 429.2537; **FT-IR**: 3506, 2924, 2853, 1699, 1600, 1515, 1448, 1392, 1364, 1275, 1123, 1020, 916 cm $^{-1}$; The ee values 86% (t_{major} = 8.84, min t_{minor} = 11.54 min) and after recrystallization 98% were determined by HPLC using Daicel Chiralpak IB with hexane/EtOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.2}$ = + 6.00 (c 0.100, $CHCl_3$).

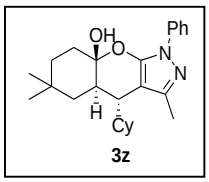
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-3,6,6-trimethyl-1,4-diphenylchromeno[2,3-c]pyrazol-8a-ol (**3y**)*



3y

The title was compound according the general procedure. Yellow sticky (57 mg, 44% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.69 (d, *J* = 7.7 Hz, 2H), 7.54 (d, *J* = 7.7 Hz, 1H), 7.39 (t, *J* = 7.8 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 1H), 7.29 (s, 2H), 7.15 (d, *J* = 7.3 Hz, 1H), 7.11 (d, *J* = 2.5 Hz, 1H), 4.24 (d, *J* = 5.2 Hz, 1H), 2.69 (s, 1H), 2.61 (d, *J* = 12.0 Hz, 1H), 2.39 – 2.34 (m, 1H), 2.28 (d, *J* = 12.0 Hz, 1H), 2.19 – 2.16 (m, 1H), 1.69 – 1.65 (m, 2H), 1.59 (s, 3H), 1.47 (s, 1H), 1.44 (dd, *J* = 4.5, 1.3 Hz, 2H), 1.25 (s, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 159.9, 138.0, 129.0, 128.9, 128.9, 128.7, 127.8, 125.3, 119.5, 114.3, 102.1, 86.6, 55.9, 48.8, 44.5, 38.6, 32.1, 25.0, 22.9, 15.9, 14.3; **ESI-MS**: m/z calcd. for $C_{25}H_{29}N_2O_2^+$ $[M+H]^+$ 389.2224, found 389.2223; **FT-IR**: 3428, 2922, 2852, 1702, 1597, 1502, 1454, 1368, 1400, 1308, 1127, 1028, 910 cm $^{-1}$; The ee values 80% (t_{major} = 9.31 min, t_{minor} = 14.24 min) was determined by HPLC using Daicel Chiralpak IB with hexane/i-PrOH (93:7) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.1}$ = + 110.81 (c 0.675, $CHCl_3$).

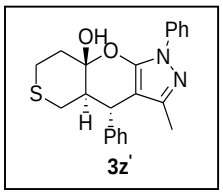
*(4R,4aR,8aS)-4-cyclohexyl-1,4,4a,5,6,7,8,8a-octahydro-3,6,6-trimethyl-1-phenylchromeno[2,3-c]pyrazol-8a-ol (**3z**)*



3z

The title was compound according the general procedure. Yellow semi solid (32 mg, 40% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.87 (d, *J* = 7.7 Hz, 1H), 7.81 (d, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.1 Hz, 1H), 7.39 (t, *J* = 7.9 Hz, 1H), 7.21 – 7.16 (m, 1H), 2.78 (s, 1H), 2.37 – 2.29 (m, 1H), 2.31 – 2.25 (m, 1H), 1.87 – 1.77 (m, 3H), 1.77 (d, *J* = 17.0 Hz, 2H), 1.66 (d, *J* = 9.9 Hz, 4H), 1.58 (s, 3H), 1.46 (d, *J* = 6.5 Hz, 1H), 1.38 (d, *J* = 18.6 Hz, 2H), 1.25 (s, 6H), 0.97 (d, *J* = 4.7 Hz, 2H), 0.87 (d, *J* = 5.4 Hz, 5H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 137.8, 129.2, 128.9, 126.4, 125.5, 121.5, 121.2, 119.4, 72.2, 68.9, 55.1, 37.7, 35.2, 32.1, 29.6, 26.7, 25.7, 22.9, 21.5, 14.4, 12.4; **ESI-MS**: m/z calcd. for $C_{25}H_{35}N_2O_2^+$ $[M+H]^+$ 395.2693, found 395.2698; **FT-IR**: 3434, 2922, 2852, 1708, 1629, 1597, 1559, 1495, 1457, 1371, 1298, 1028, 901 cm $^{-1}$; The ee values 88% (t_{major} = 9.76, min t_{minor} = 16.88 min) was determined by HPLC using Daicel Chiralpak IC with hexane/EtOH (94:6) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{30.1}$ = + 15.79 (c 0.380, $CHCl_3$).

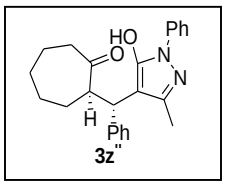
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-3-methyl-1,4-diphenylthiochromeno[2,3-c]pyrazol-8a-ol (**3z'**)*



3z'

The title was compound according the general procedure. Yellow semi solid (30 mg, 40% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 7.9 Hz, 2H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.34 (t, *J* = 7.4 Hz, 2H), 7.29 (d, *J* = 7.2 Hz, 1H), 7.23 (dd, *J* = 18.4, 7.3 Hz, 3H), 3.62 (d, *J* = 10.7 Hz, 1H), 3.05 (d, *J* = 11.0 Hz, 1H), 2.99 – 2.94 (m, 1H), 2.86 (s, 1H), 2.79 – 2.72 (m, 1H), 2.72 – 2.67 (m, 1H), 2.41 (dd, *J* = 28.7, 15.4 Hz, 2H), 2.18 (t, *J* = 11.1 Hz, 1H), 1.60 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.42, 147.27, 140.88, 138.80, 129.20, 128.94, 127.47, 125.68, 120.63, 120.56, 101.96, 99.73, 49.35, 40.70, 40.40, 29.08, 25.81, 13.47; **ESI-MS**: m/z calcd. for $C_{22}H_{23}N_2O_2S^+$ $[M+H]^+$ 379.1475, found 379.1469; **FT-IR**: 3439, 2925, 2860, 1708, 1629, 1600, 1555, 1490, 1457, 1371, 1298, 1028, 910 cm $^{-1}$; The ee values 88% (t_{major} = 16.13 min, t_{minor} = 26.48 min) was determined by HPLC using Daicel Chiralpak IB with hexane/i-PrOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C.

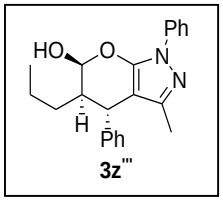
*(R)-2-((S)-(5-hydroxy-3-methyl-1-phenyl-1H-pyrazol-4-yl)(phenyl)methyl)cycloheptanone (**3z''**)*



3z''

The title was compound according the general procedure. Yellow sticky (36 mg, 48% yield); R_f value 0.25 (5:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 7.8 Hz, 2H), 7.43 – 7.38 (m, 3H), 7.33 (d, *J* = 7.9 Hz, 2H), 7.22 – 7.18 (m, 3H), 4.12 (t, *J* = 10.5 Hz, 1H), 3.81 (d, *J* = 5.1 Hz, 1H), 3.57 (t, *J* = 12.5 Hz, 1H), 3.07 (d, *J* = 16.4 Hz, 1H), 2.54 (s, 3H), 2.10 (s, 3H), 1.93 (s, 2H), 1.76 (s, 4H), 1.13 (d, *J* = 13.9 Hz, 1H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 217.0, 173.0, 159.8, 138.2, 137.9, 129.0, 128.9, 128.2, 127.9, 125.4, 119.6, 53.8, 50.4, 45.7, 44.3, 44.1, 27.8, 24.5, 22.9, 15.9; **ESI-MS**: m/z calcd. for $C_{24}H_{27}N_2O_2^+$ $[M+H]^+$ 375.2067, found 375.2070; **FT-IR**: 3440, 2924, 2850, 1804, 1723, 1650, 1599, 1545, 1520, 1451, 1396, 1302, 1154, 1074, 906 cm^{-1}; The ee values 90% (t_{major} = 30.55 min, t_{minor} = 58.55 min) was determined by HPLC using phenomenex cellulose-4 with hexane/i-PrOH (90:10) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C.

*(4S,5R,6S)-1,4,5,6-tetrahydro-3-methyl-1,4-diphenyl-5-propylpyrano[2,3-c]pyrazol-6-ol (**3z'''**)*

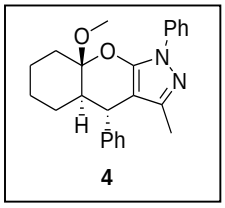


3z'''

The title was compound according the general procedure. Yellow semi solid (28 mg, 40% yield); R_f value 0.20 (15:1 hex/EA); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 7.9 Hz, 2H), 7.38 (t, *J* = 7.9 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 2H), 7.21 (dt, *J* = 15.0, 7.8 Hz, 4H), 5.73 (s, 1H), 3.70 (d, *J* = 9.9 Hz, 1H), 1.99 (s,

1H), 1.86 (s, 1H), 1.60 (s, 3H), 1.51 – 1.43 (m, 2H), 1.39 – 1.33 (m, 1H), 1.29 (dd, *J* = 16.9, 6.4 Hz, 1H), 0.79 (t, *J* = 7.2 Hz, 8H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 147.4, 142.3, 138.7, 129.4, 129.1, 128.9, 128.7, 128.0, 127.0, 125.6, 120.7, 119.4, 117.9, 100.8, 99.8, 53.4, 45.1, 39.7, 30.2, 20.5, 14.3, 13.3; **ESI-MS**: m/z calcd. for $C_{22}H_{25}N_2O_2^+$ $[M+H]^+$ 349.1911, found 349.1916; **FT-IR**: 3440, 2924, 2853, 1804, 1723, 1599, 1549, 1518, 1451, 1396, 1302, 1154, 1074, 906 cm^{-1}; The ee values 80% (t_{major} = 18.07 min, t_{minor} = 14.83 min) and after recrystallization >99% were determined by HPLC using Daicel Chiralpak IA with hexane/i-PrOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ºC; **Optical Rotation**: $[\alpha]_D^{30.4}$ = + 41.40 (c 0.570, $CHCl_3$).

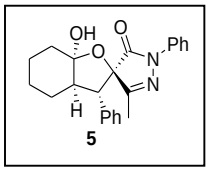
*(4S,4aR,8aS)-1,4,4a,5,6,7,8,8a-octahydro-8a-methoxy-3-methyl-1,4-diphenylchromeno[2,3-c]pyrazole (**4**)*



4

Colour less sticky (56 mg, 74% yield); R_f value 0.20 (10:1 hex/EA); **^{1}H NMR (600 MHz, CDCl$_3$)** δ 7.82 (d, J = 7.7 Hz, 2H), 7.43 – 7.39 (m, 2H), 7.30 (t, J = 7.4 Hz, 2H), 7.20 (dd, J = 14.5, 7.2 Hz, 4H), 3.60 (d, J = 10.7 Hz, 1H), 3.30 (s, 3H), 2.48 (d, J = 14.0 Hz, 1H), 1.82 – 1.77 (m, 1H), 1.69 (t, J = 11.7 Hz, 3H), 1.60 (s, 3H), 1.44 – 1.41 (m, 1H), 1.40 – 1.34 (m, 2H), 1.14 (dd, J = 9.3, 4.0 Hz, 1H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 148.2, 147.2, 142.3, 139.2, 129.1, 128.6, 126.9, 125.2, 120.0, 105.6, 100.9, 49.0, 48.7, 40.4, 31.8, 26.9, 25.6, 22.8, 13.6; **ESI-MS**: m/z calcd. for $C_{24}H_{27}N_2O_2^+$ $[M+H]^+$ 375.2067, found 375.2069; **FT-IR**: 3431, 2925, 2852, 1600, 1514, 1489, 1444, 1397, 1273, 1254, 1209, 1149, 1076, 993 cm^{-1}; The ee values 84% (t_{major} = 5.40 min, t_{minor} = 4.50 min) was determined by HPLC using Daicel Chiralpak IA with hexane/i-PrOH (98:3) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ºC.

*(3S,3aR,7aR)-(octahydro-3-phenylbenzofuran-7a-ol) 3-methyl-1-phenyl-1H-pyrazol-5-one (**5**)*

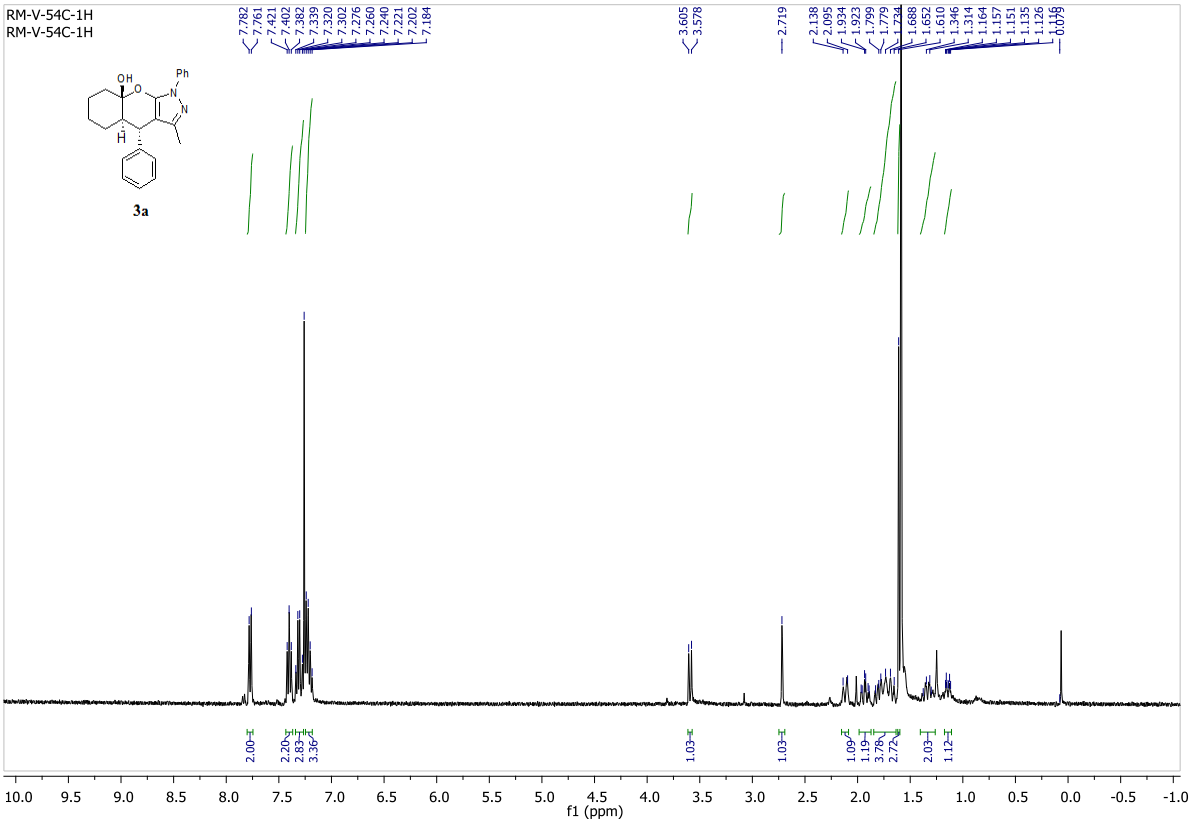


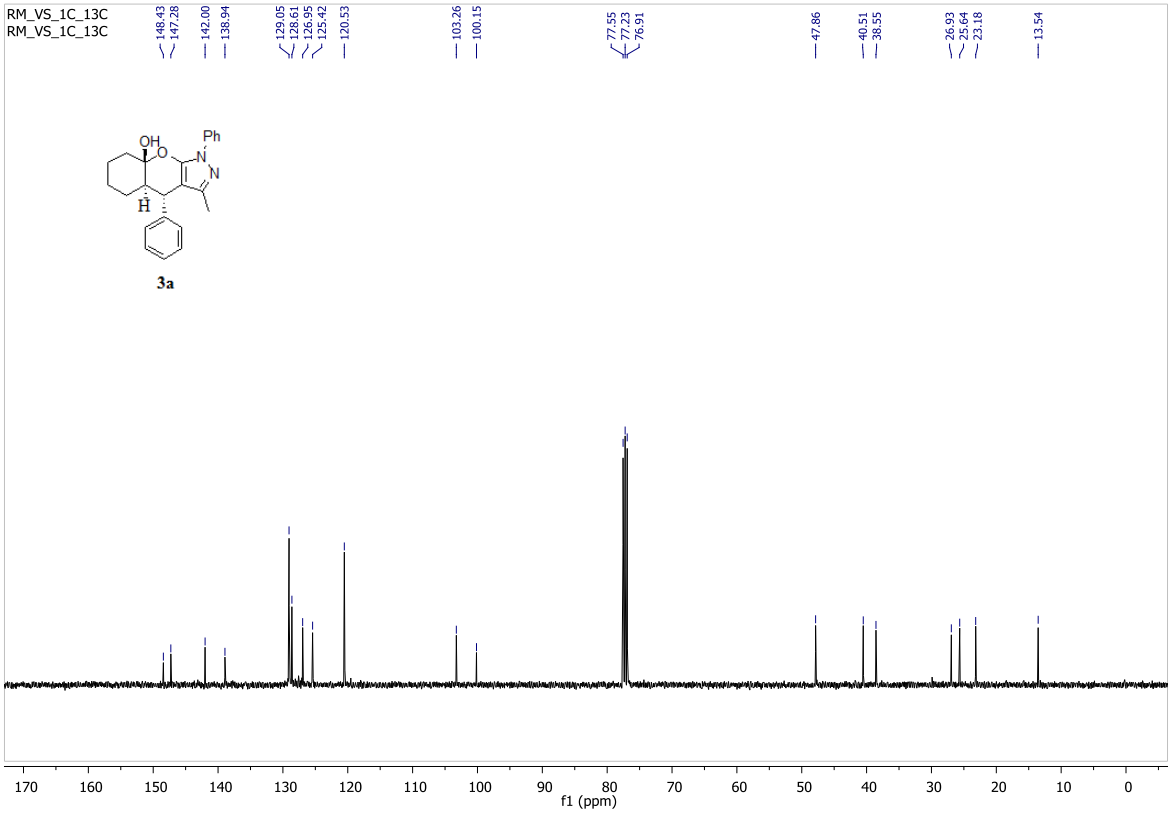
5

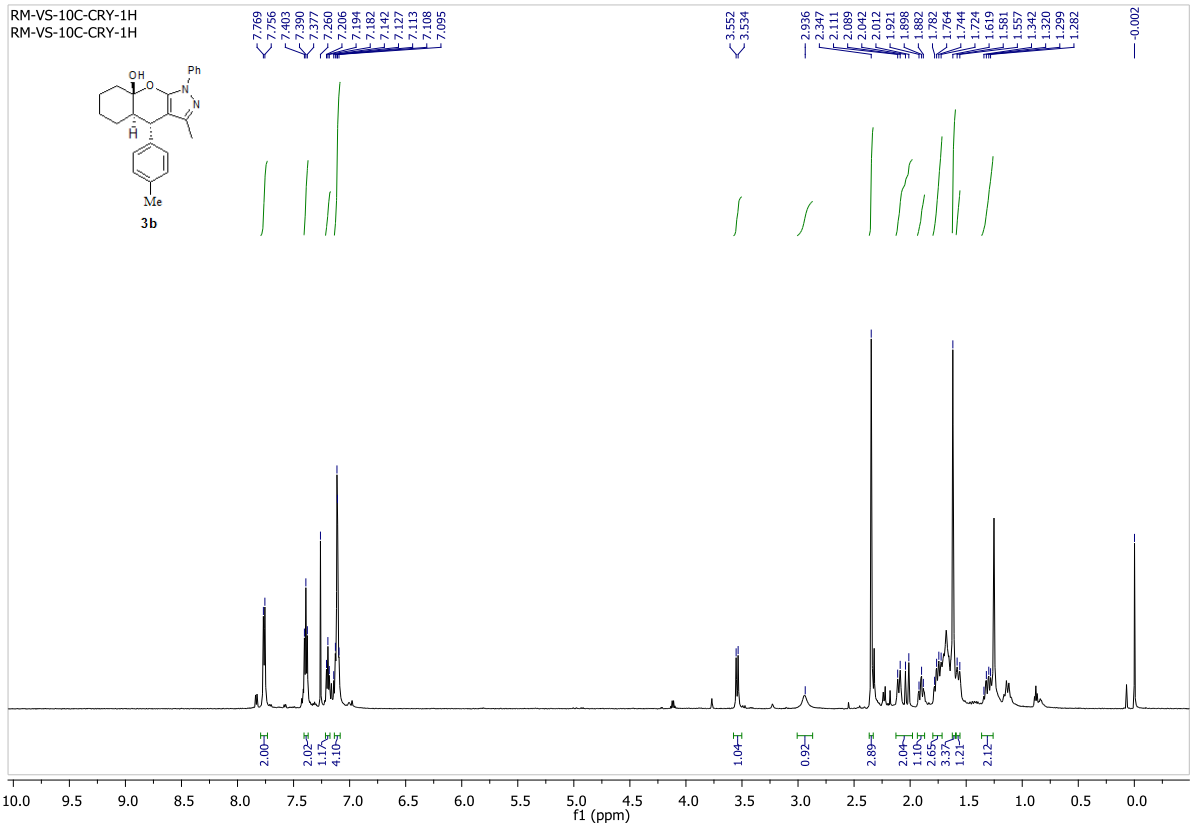
Yellow sticky oil (57 mg, 75% yield); R_f value 0.30 (10:1 hex/EA); **^{1}H NMR (600 MHz, CDCl$_3$)** δ 7.35 (d, *J* = 8.1 Hz, 2H), 7.28 (dd, *J* = 7.3, 3.1 Hz, 4H), 7.23 (t, *J* = 6.3 Hz, 3H), 7.13 (t, *J* = 7.3 Hz, 1H), 3.56 (d, *J* = 12.4 Hz, 1H), 3.35 – 3.28 (m, 1H), 2.32 (s, 3H), 2.17 (dt, *J* = 13.6, 4.7 Hz, 1H), 2.06 (dd, *J* = 14.6, 6.9 Hz, 1H), 1.93 – 1.83 (m, 3H), 1.74 (dd, *J* = 12.5, 6.9 Hz, 2H), 1.69 (dd, *J* = 17.5, 7.0 Hz, 2H), 1.56 (d, *J* = 4.3 Hz, 1H), 1.51 – 1.47 (m, 1H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 173.1, 159.7, 136.9, 132.2, 128.9, 128.8, 128.2, 125.8, 119.7, 108.7, 89.2, 55.2, 45.6, 42.1, 34.7, 27.2, 23.2, 19.3, 12.9; **ESI-MS**: m/z calcd. for $C_{23}H_{23}N_2O_2^+$ $[M-OH]^+$ 359.1754, found 359.1757; **FT-IR**: 3439, 2929, 2860, 1710, 1640, 1629, 1600, 1555, 1490, 1457, 1375, 1298, 1028, 911 cm^{-1}; The ee values 80%

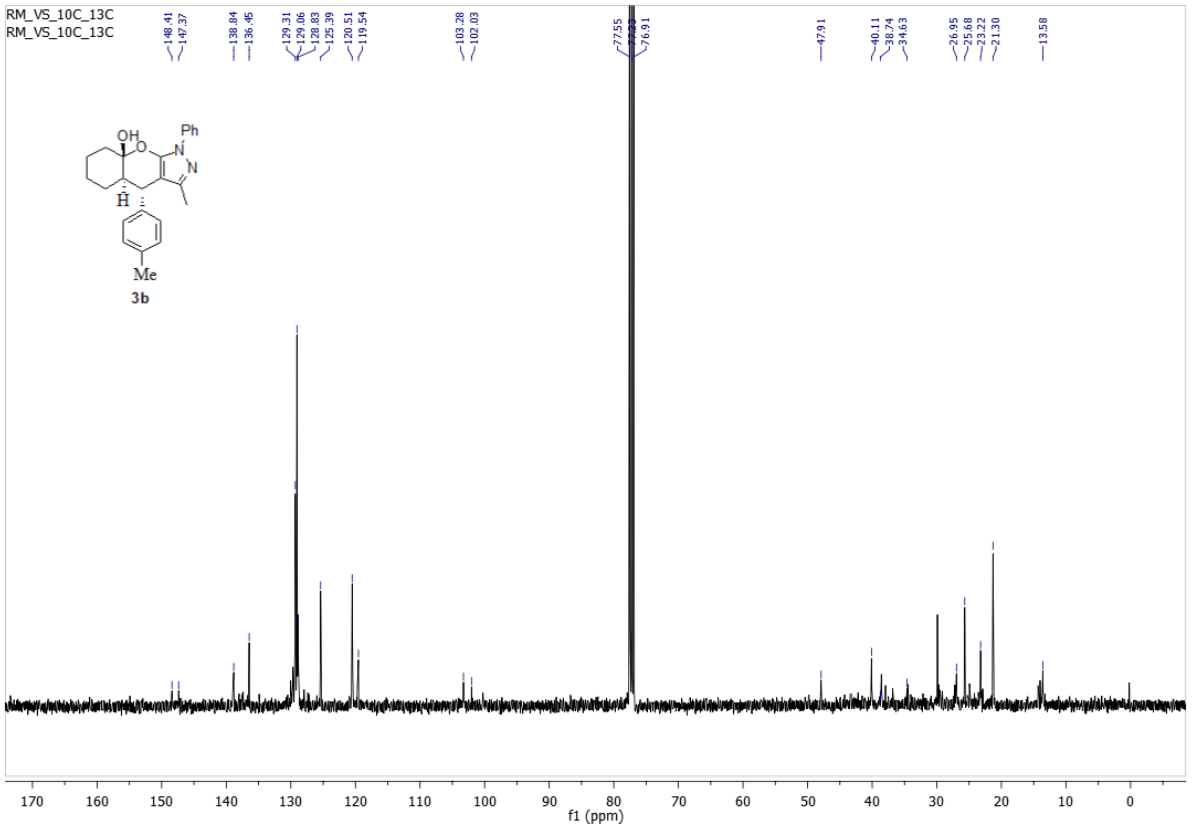
(t_{major} = 25.92 min, t_{minor} = 23.79 min) was determined by HPLC using Daicel Chiralpak IC with hexane/i-PrOH (95:5) as the eluent, flow: 1.0 mL/min, 254 nm, 25 ^{o}C; **Optical Rotation**: $[\alpha]_D^{31.0}$ = + 154.17 (c 0.515, $CHCl_3$).

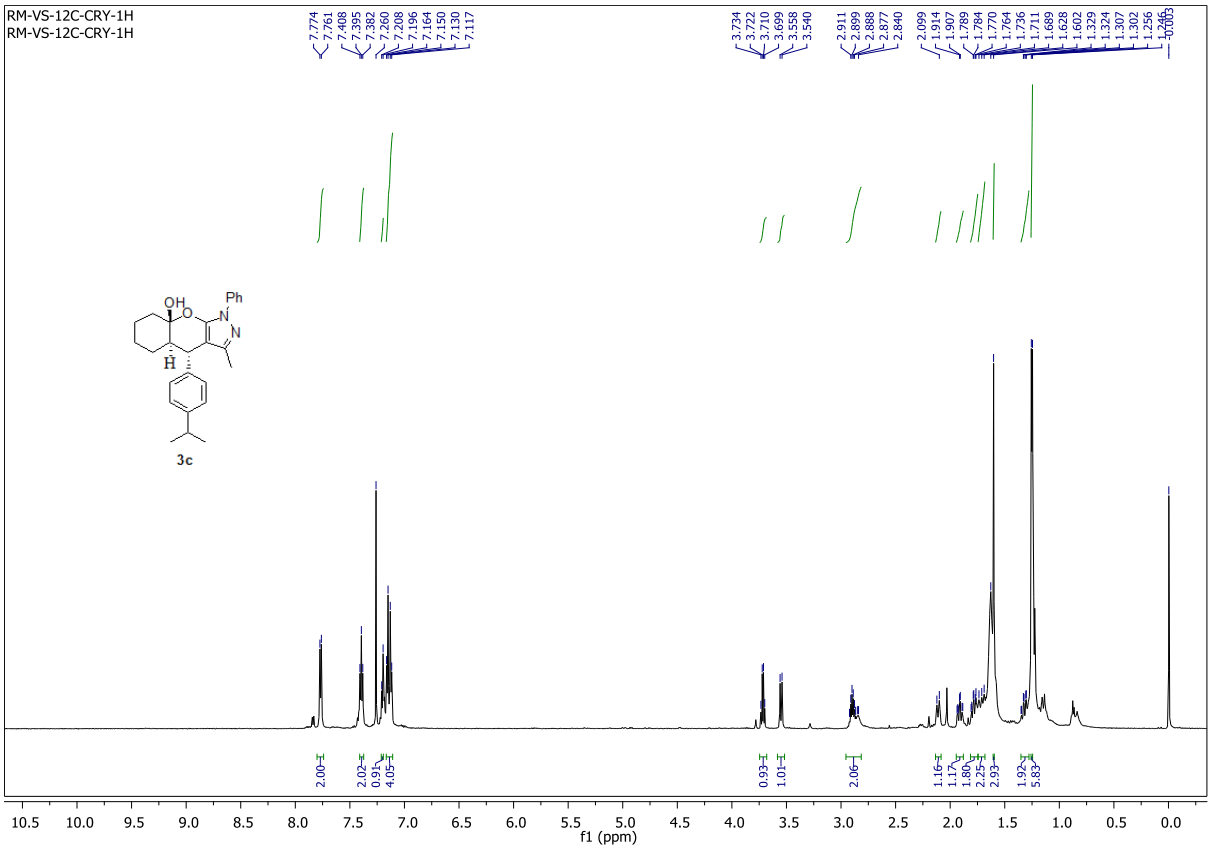
12. **NMR Spectra**:

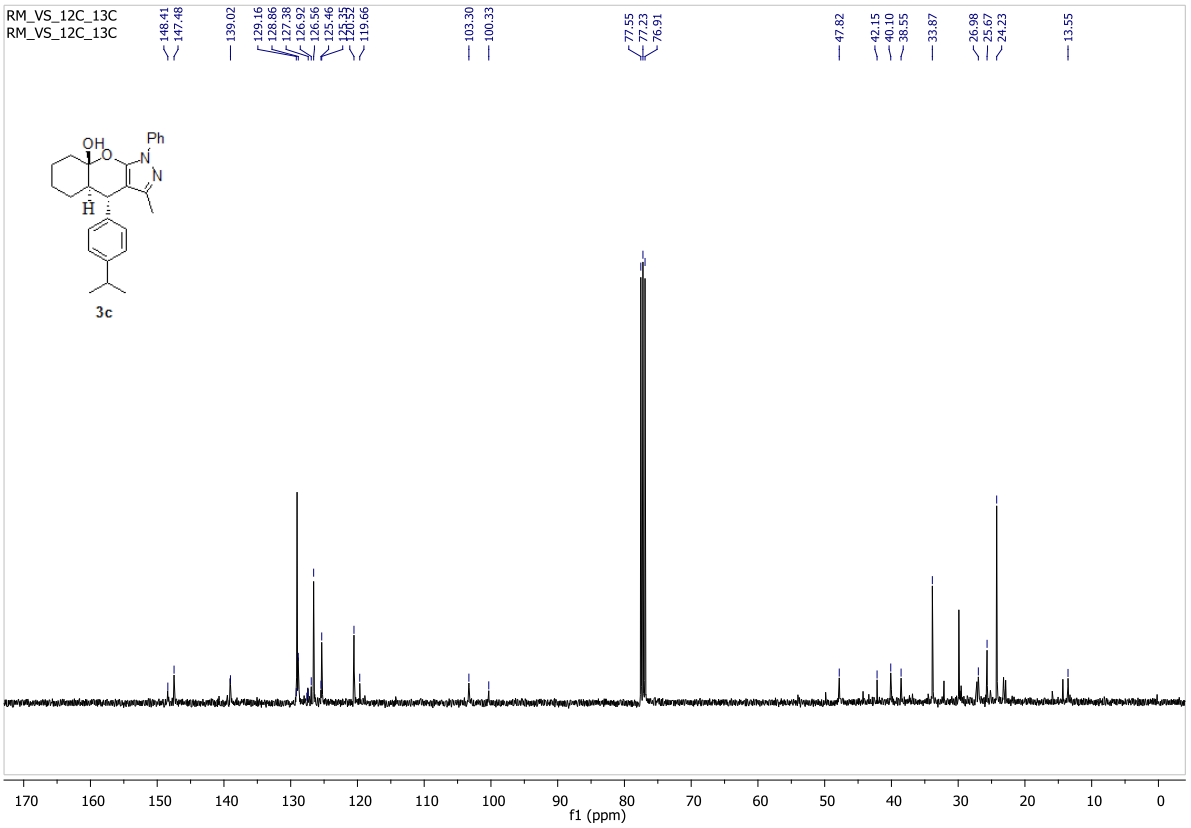


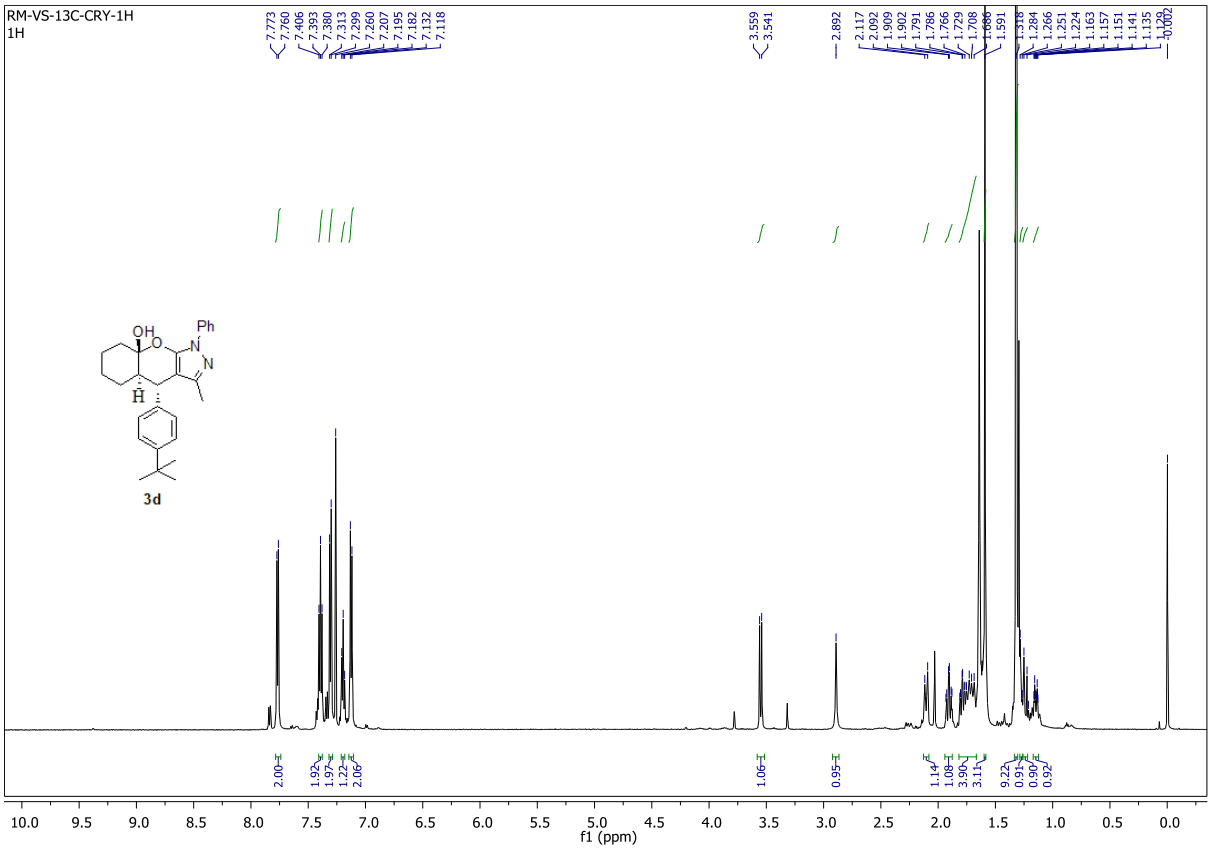


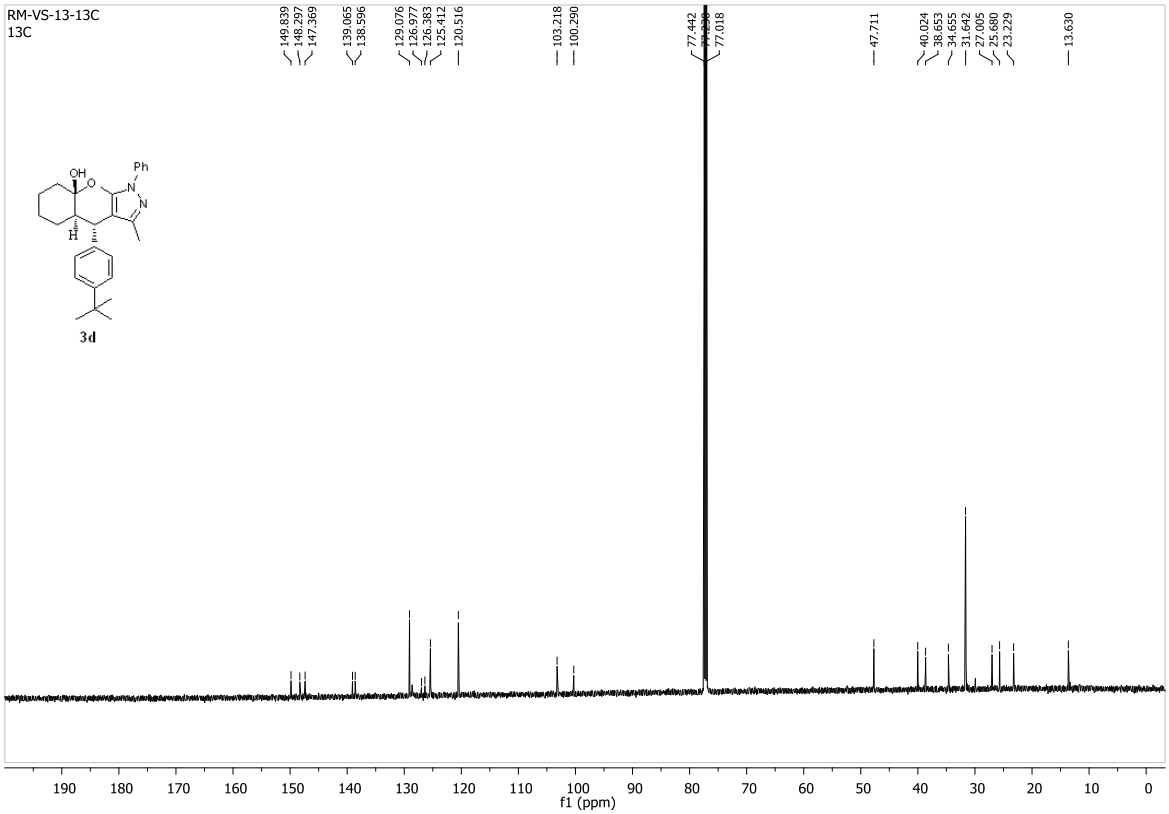

RM-VS-10C-CRY-1H
RM-VS-10C-CRY-1H
3b
f1 (ppm)

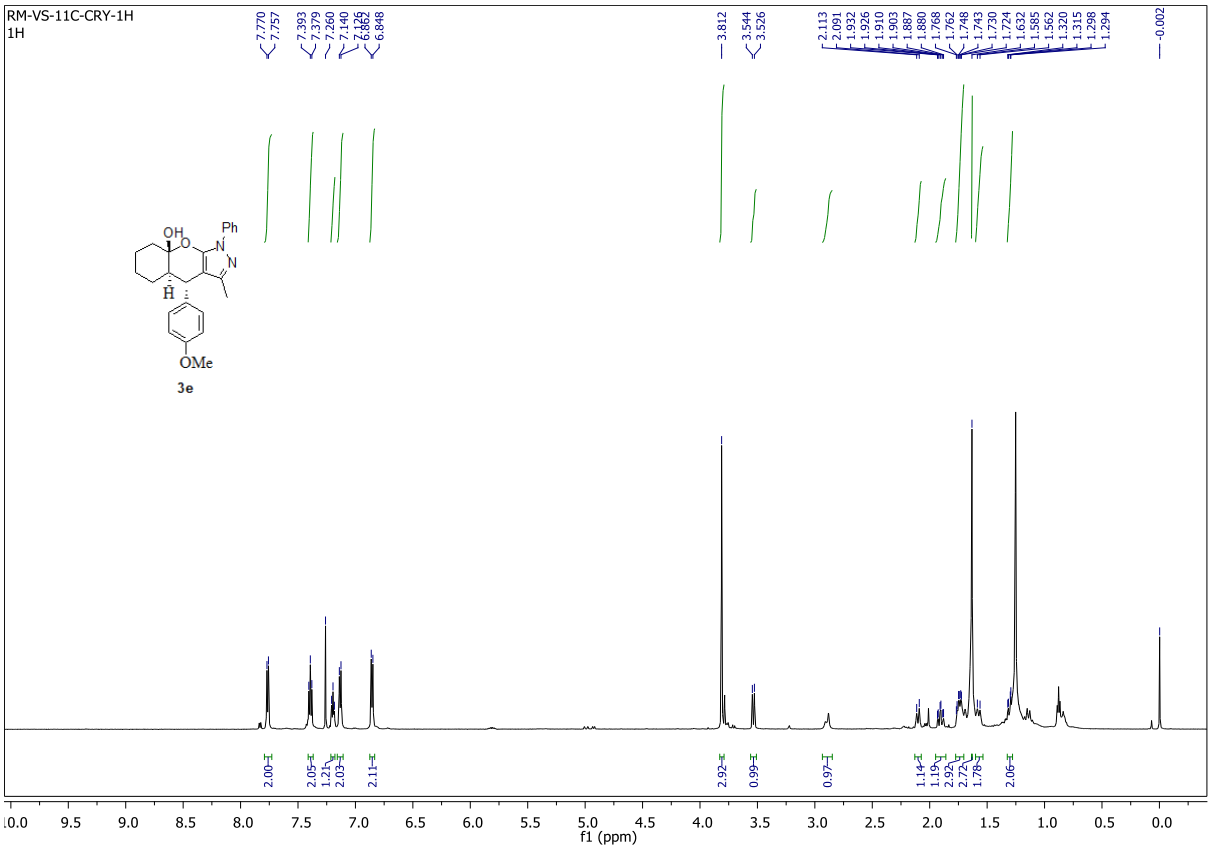

RM_VS_10C_13C
RM_VS_10C_13C
3b
f1 (ppm)


RM-VS-12C-CRY-1H
RM-VS-12C-CRY-1H
3c
f1 (ppm)


RM_VS_12C_13C
RM_VS_12C_13C
3c
f1 (ppm)


RM-VS-13C-CRY-1H
1H
3d
f1 (ppm)


RM-VS-13-13C
13C
3d
f1 (ppm)


RM-VS-11C-CRY-1H
1H
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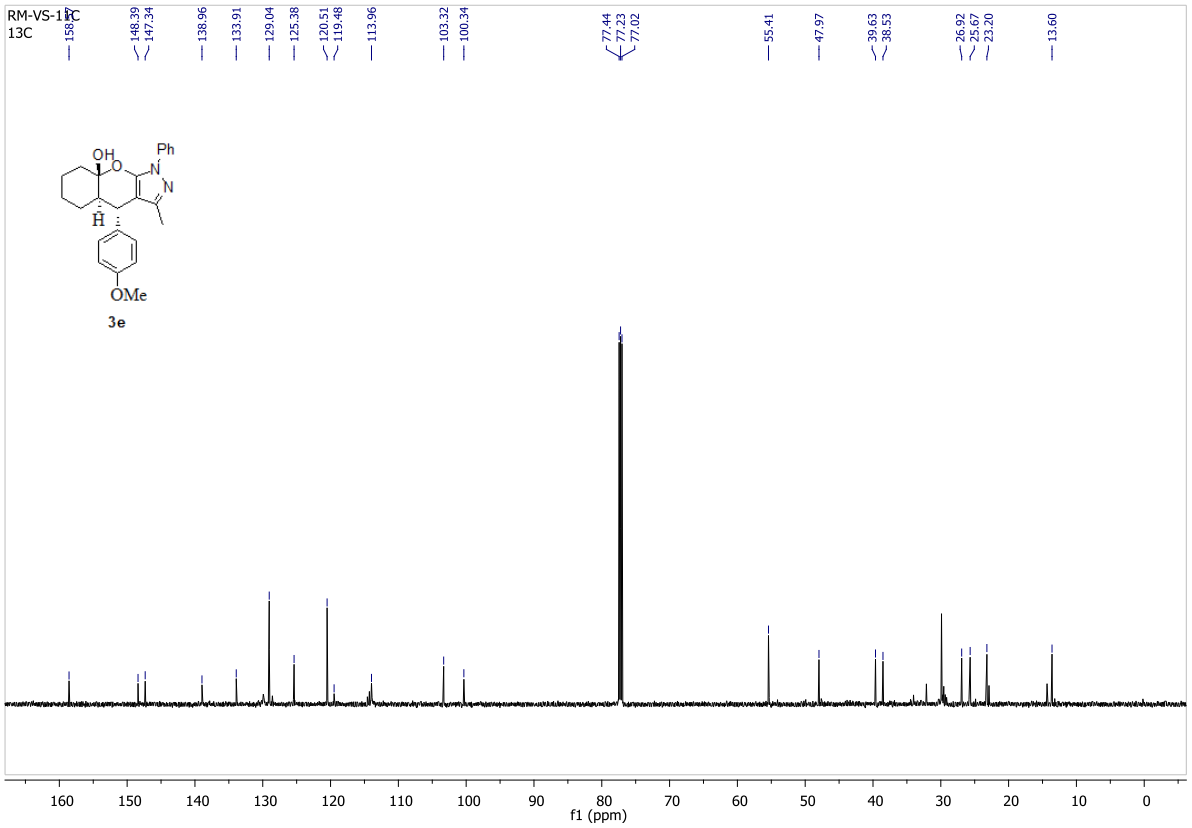
7.757
7.393
7.379
7.260
7.140
7.126
6.862
6.848
3.812
3.544
3.526
2.113
2.091
1.932
1.926
1.910
1.903
1.887
1.880
1.768
1.762
1.748
1.743
1.730
1.724
1.632
1.585
1.562
1.320
1.315
1.298
1.294
-0.002
OH
O
Ph
N
N
H
OMe
3e
2.00
2.05
1.21
2.03
2.11
2.92
0.99
0.97
1.14
1.19
2.92
2.72
1.78
2.06
f1 (ppm)


RM-VS-11C
13C
158.87
148.39
147.34
138.96
133.91
129.04
125.38
120.51
119.48
113.96
103.32
100.34
77.44
77.23
77.02
55.41
47.97
39.63
38.53
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23.20
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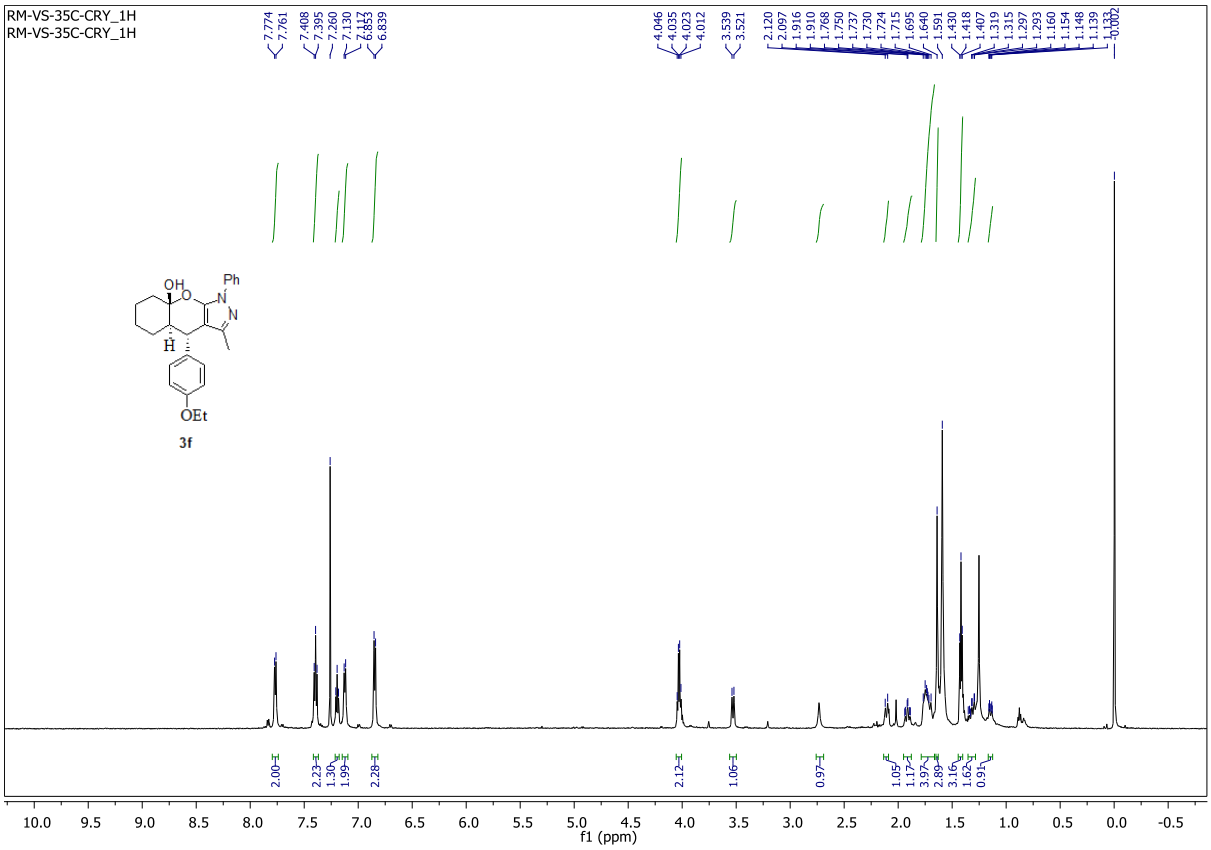
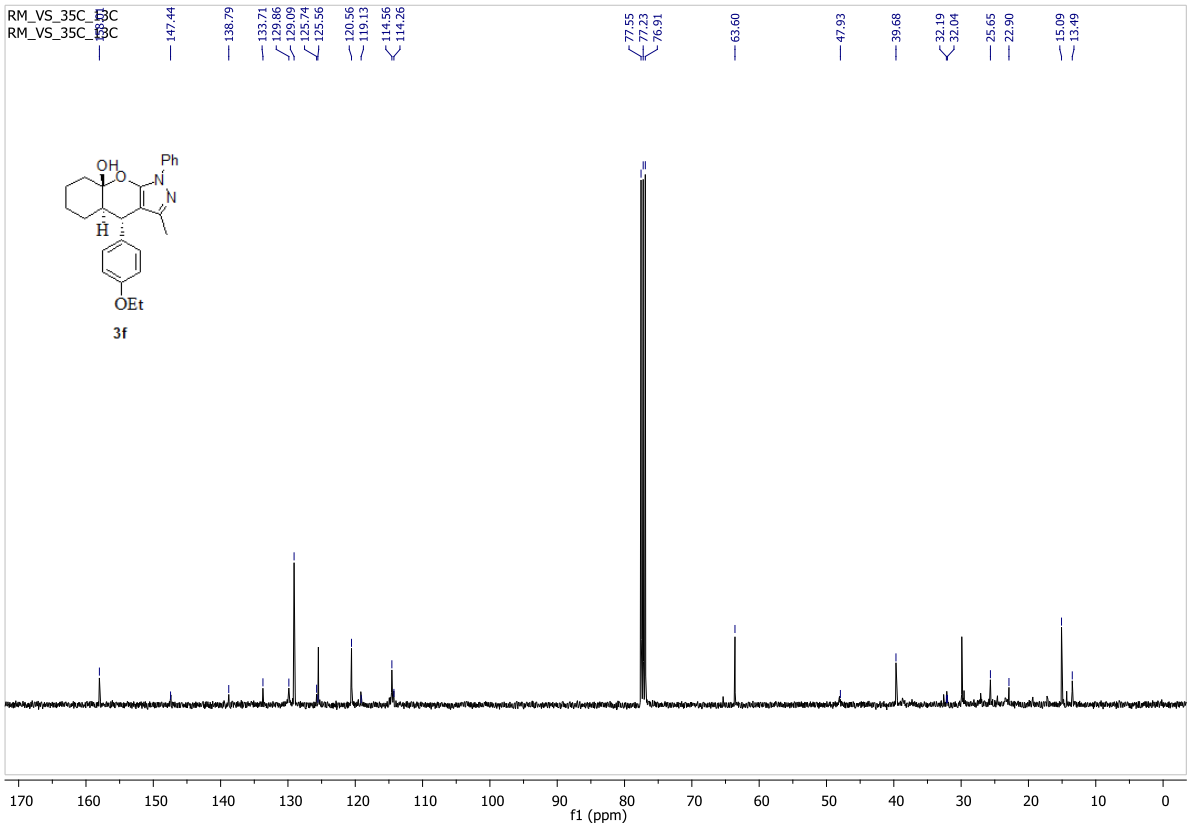
OH
O
Ph
N
N
H
OMe
3e
f1 (ppm)


RM-VS-35C-CRY_1H
RM-VS-35C-CRY_1H
7.774
7.761
7.408
7.395
7.260
7.130
7.117
6.853
6.839
4.046
4.035
4.023
4.012
3.539
3.521
2.120
2.097
1.916
1.910
1.768
1.750
1.737
1.730
1.724
1.715
1.695
1.640
1.591
1.430
1.418
1.407
1.319
1.315
1.297
1.293
1.160
1.154
1.148
1.139
1.133
0.002
OH
O
Ph
N
N
H
OEt
3f
2.00
2.23
1.30
1.99
2.28
2.12
1.06
0.97
1.05
1.17
3.97
2.89
3.16
1.62
0.91
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


RM_VS_35C_13C
RM_VS_35C_13C
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133.71
129.86
129.09
125.74
125.56
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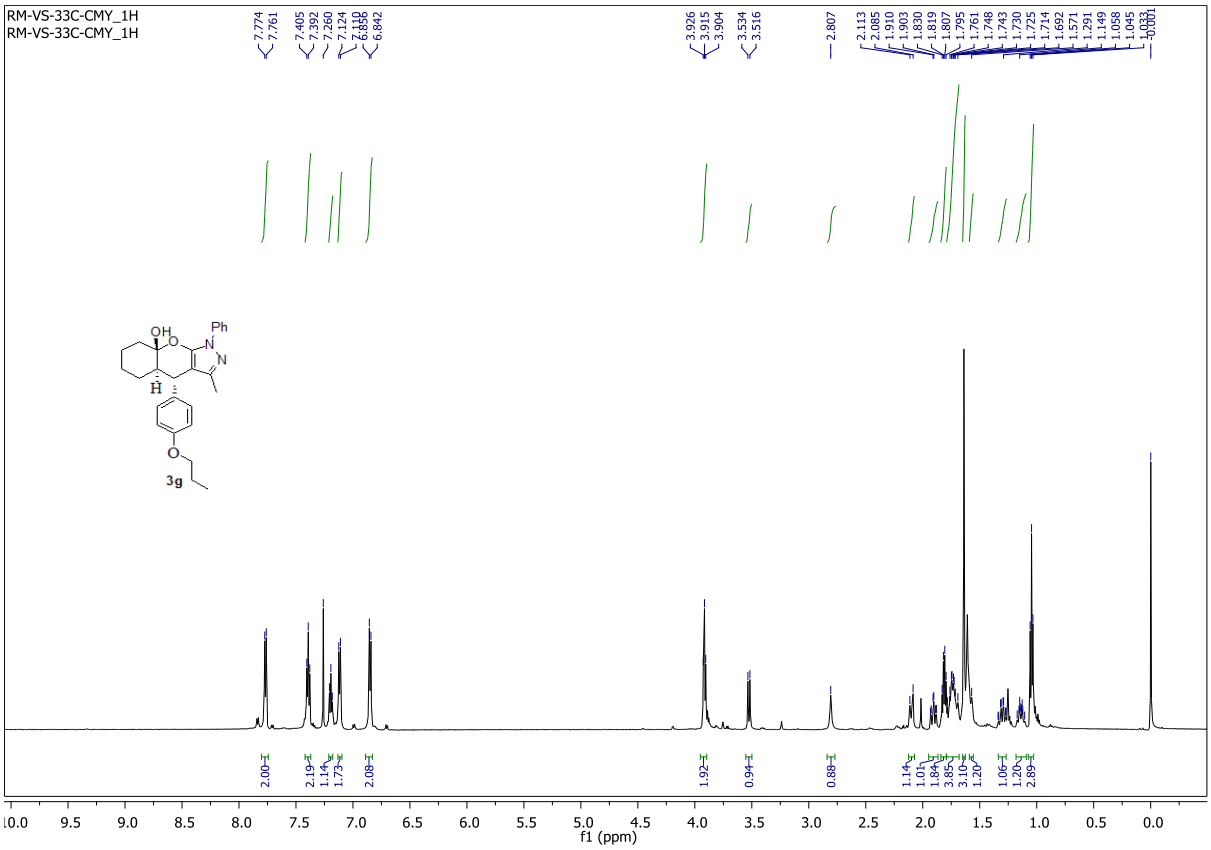
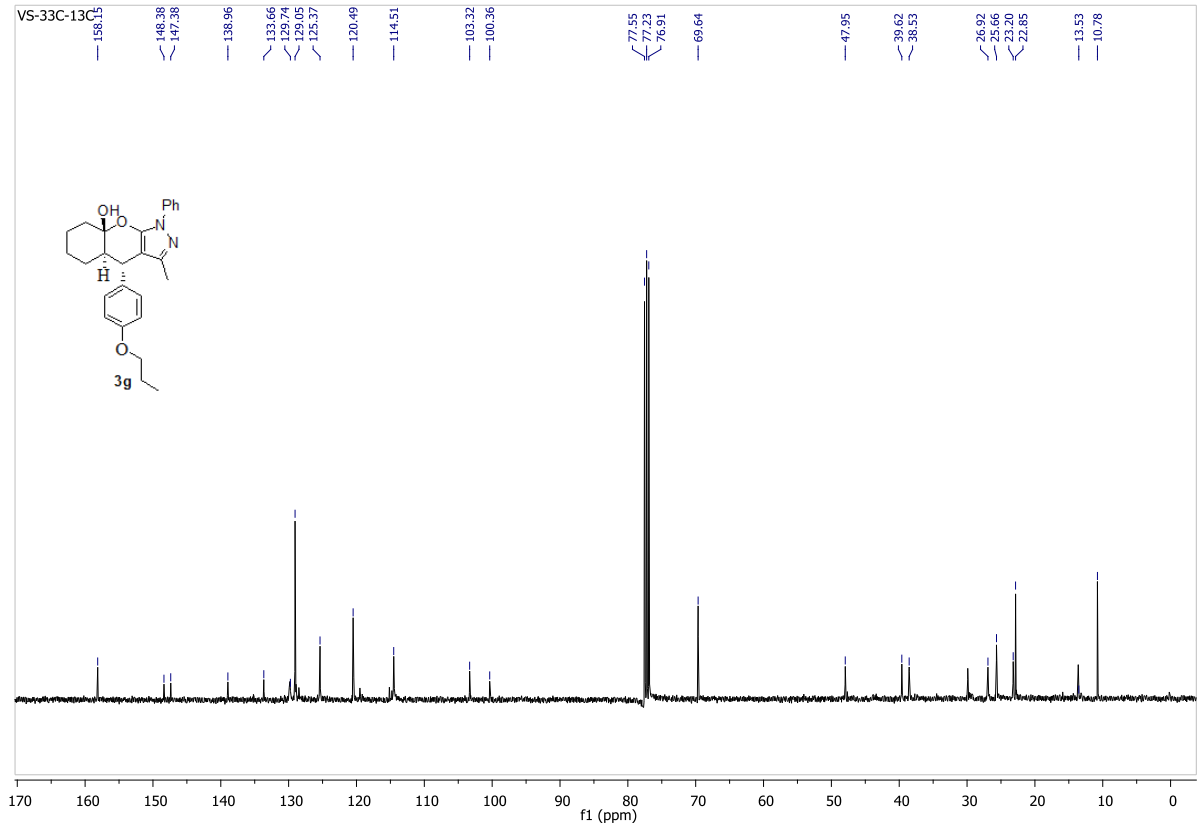
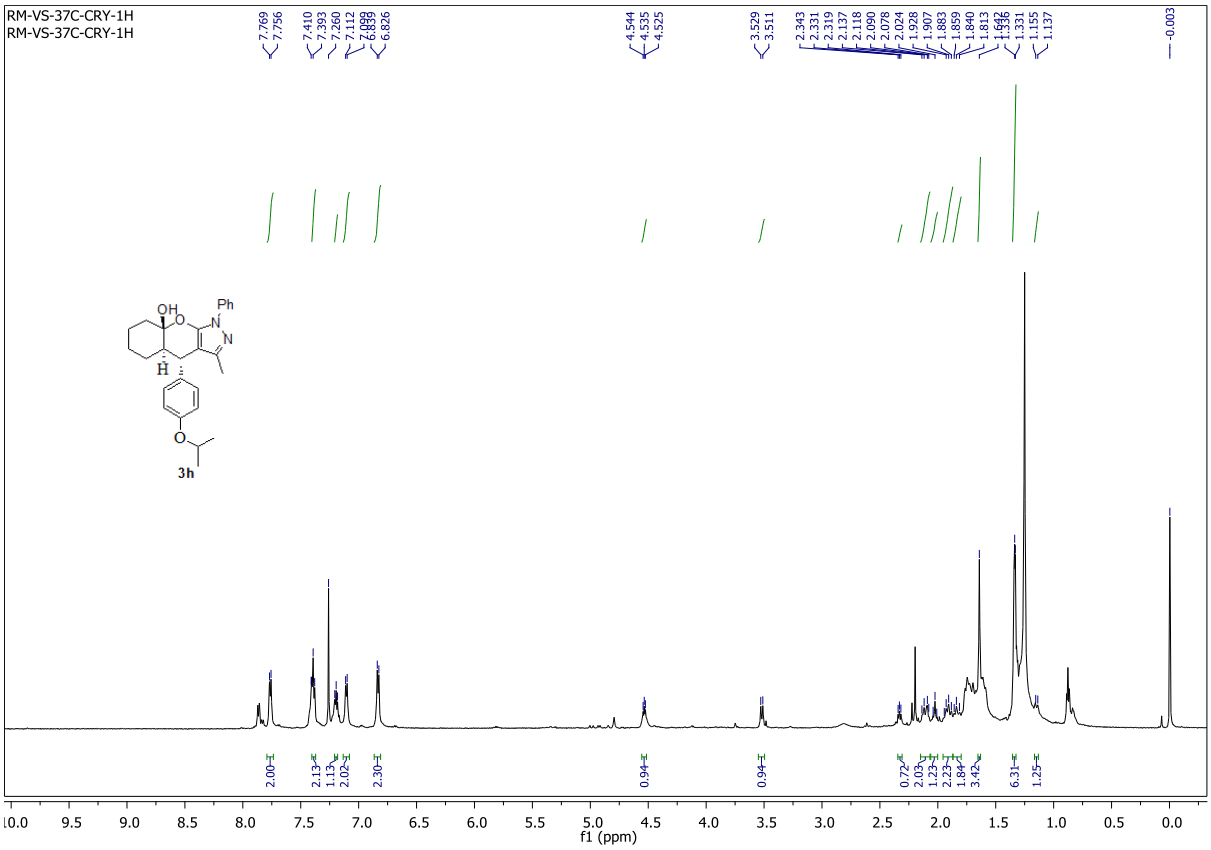
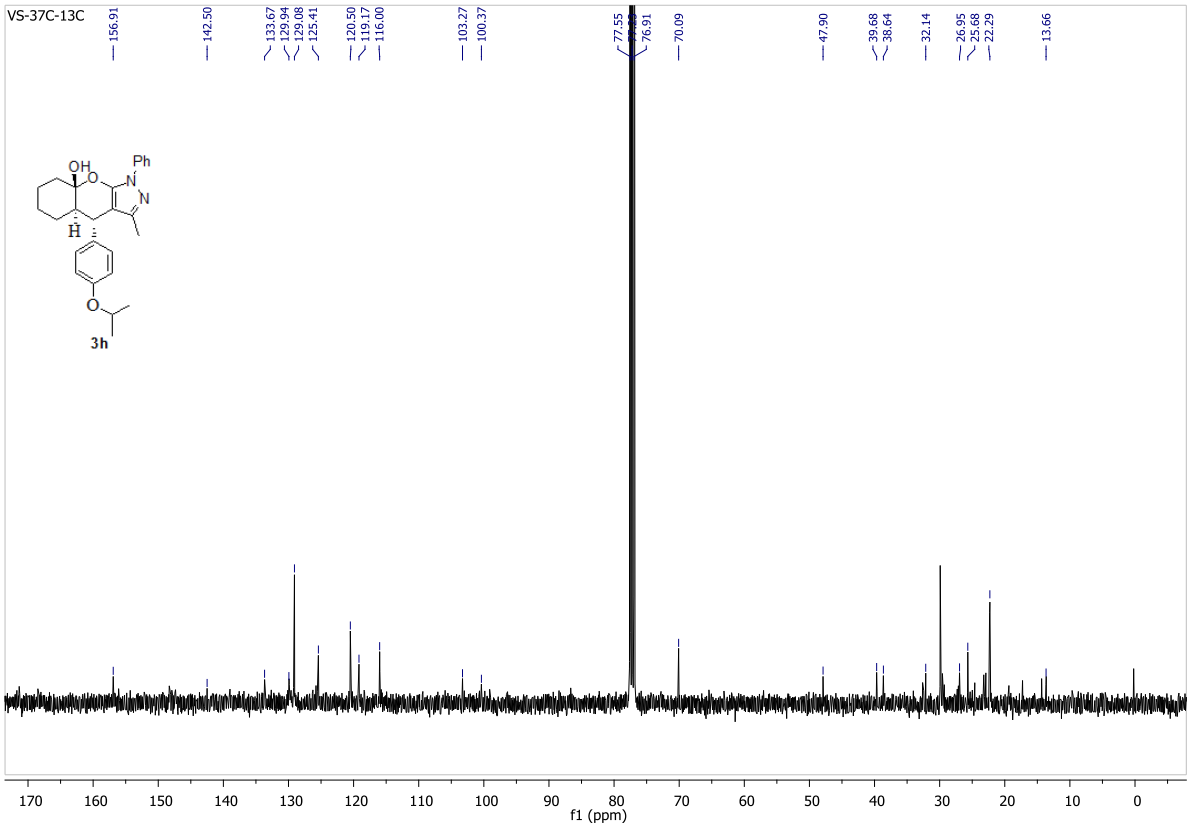
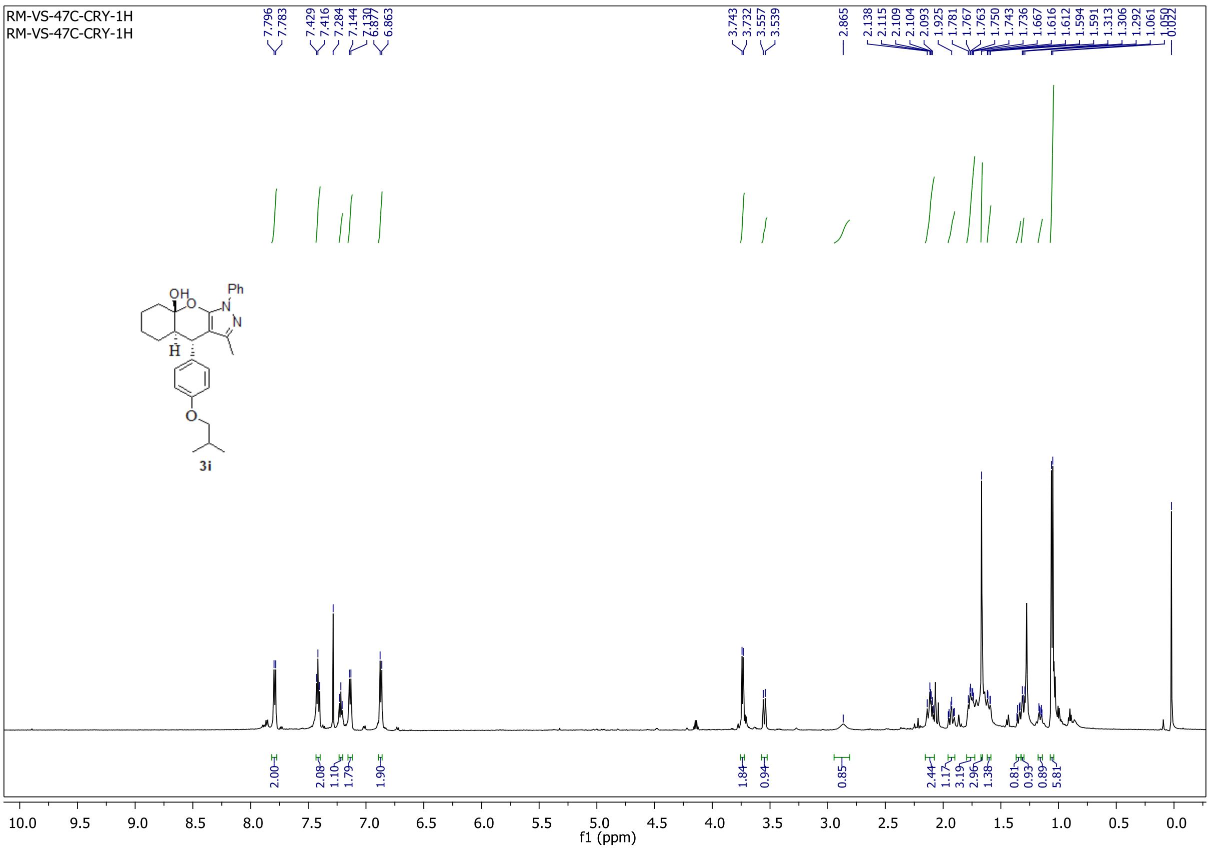
119.13
114.56
114.26
77.55
77.23
76.91
63.60
47.93
39.68
32.19
32.04
25.65
22.90
15.09
13.49
OH
O
Ph
N
N
H
OEt
3f
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

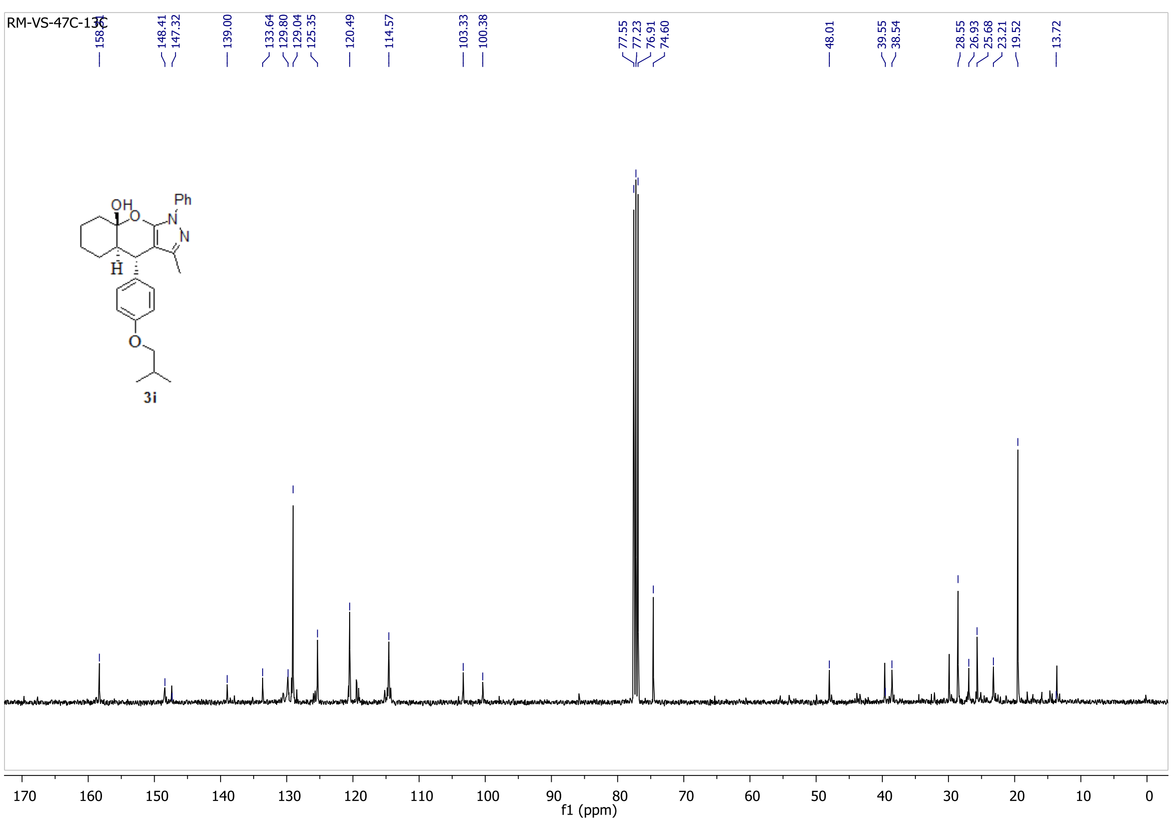

RM-VS-33C-CMY_1H
RM-VS-33C-CMY_1H
7.774
7.761
7.405
7.392
7.260
7.124
6.842
3.926
3.915
3.904
3.534
3.516
2.807
2.113
2.085
1.910
1.903
1.830
1.819
1.807
1.795
1.761
1.748
1.743
1.730
1.725
1.714
1.692
1.571
1.291
1.149
1.058
1.045
1.033
0.001
OH
O
Ph
N
N
H
O
3g
2.00
2.19
1.14
1.73
2.08
1.92
0.94
0.88
1.14
1.01
1.84
3.85
3.10
1.20
1.06
1.20
2.89
f1 (ppm)

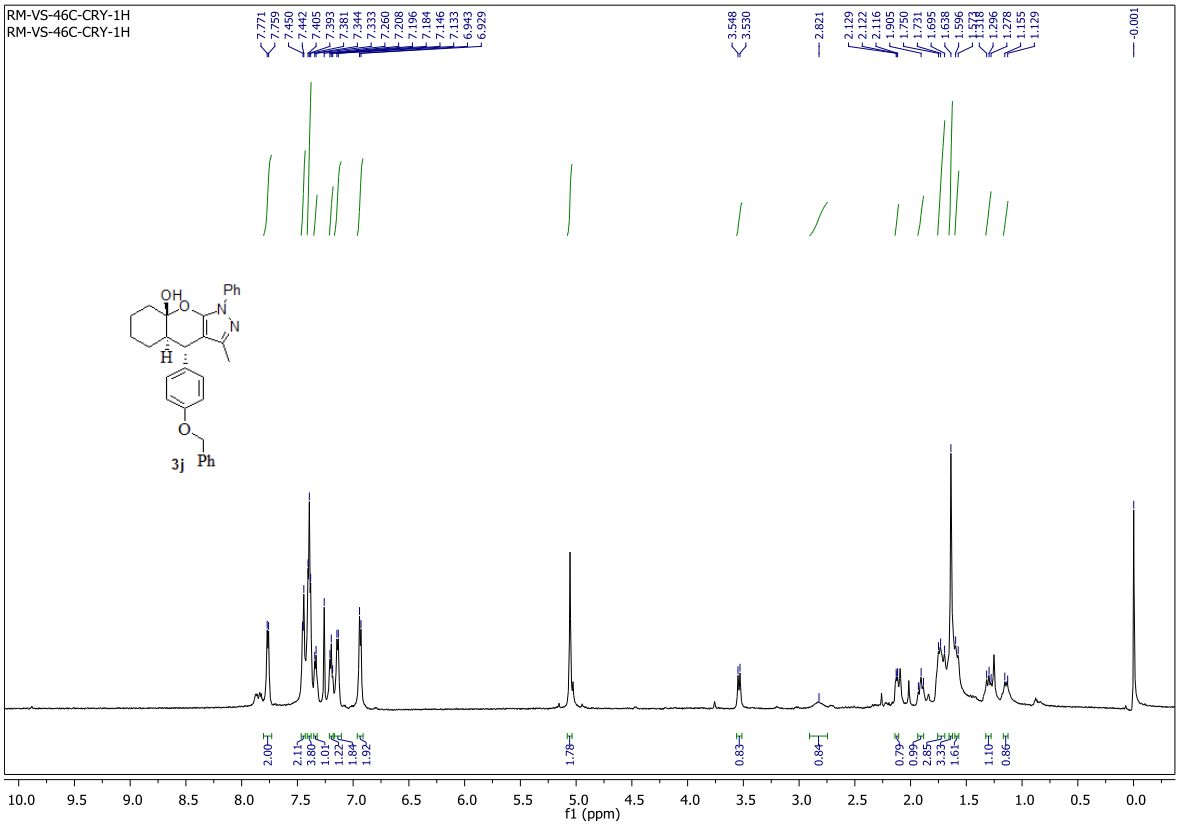

VS-33C-13C
158.15
148.38
147.38
138.96
133.66
129.74
129.05
125.37
120.49
114.51
103.32
100.36
77.55
77.23
76.91
69.64
47.95
39.62
38.53
26.92
25.66
23.20
22.85
13.53
10.78
OH
O
Ph
N
N
H
O
3g
f1 (ppm)

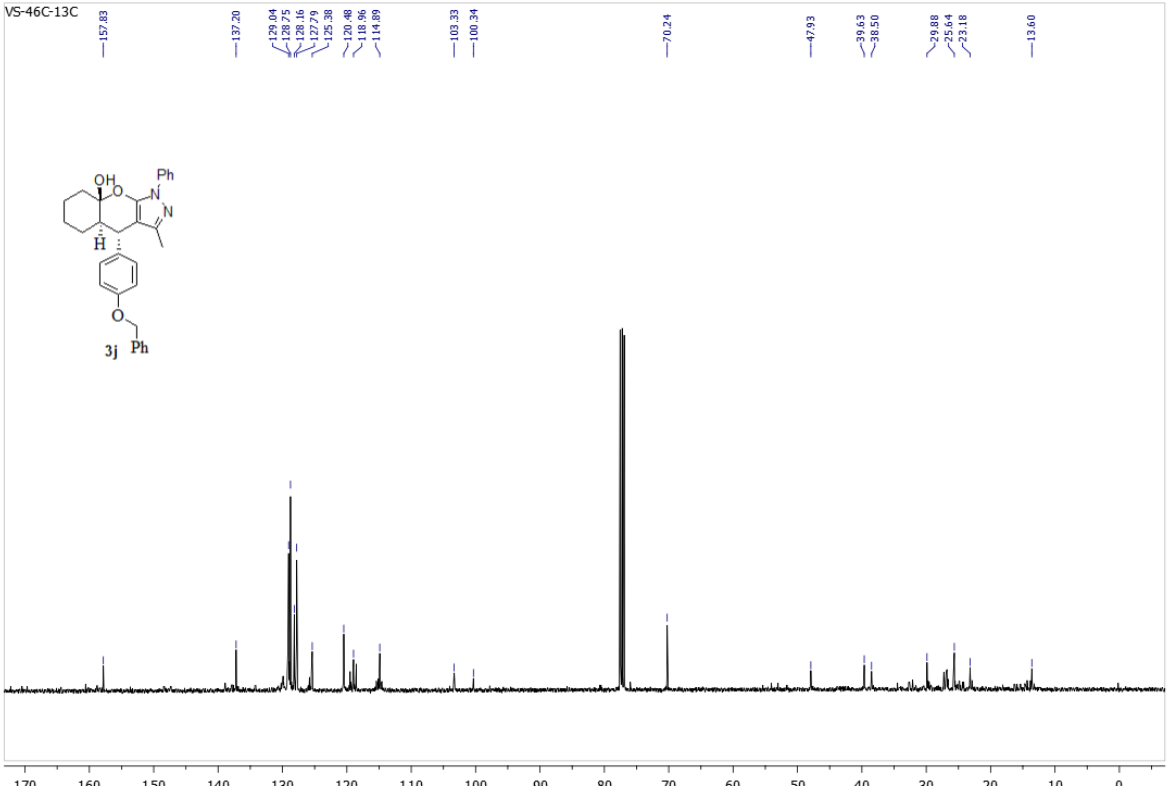

RM-VS-37C-CRY-1H
RM-VS-37C-CRY-1H
7.769
7.756
7.410
7.393
7.260
7.112
6.839
6.826
4.544
4.535
4.525
3.529
3.511
2.343
2.331
2.319
2.137
2.118
2.090
2.078
2.024
1.928
1.907
1.883
1.859
1.840
1.813
1.331
1.155
1.137
-0.003
OH
O
Ph
N
N
H
O
3h
2.00
2.13
1.13
2.02
2.30
0.94
0.94
0.72
2.03
1.23
2.23
1.84
3.42
6.31
1.25
f1 (ppm)

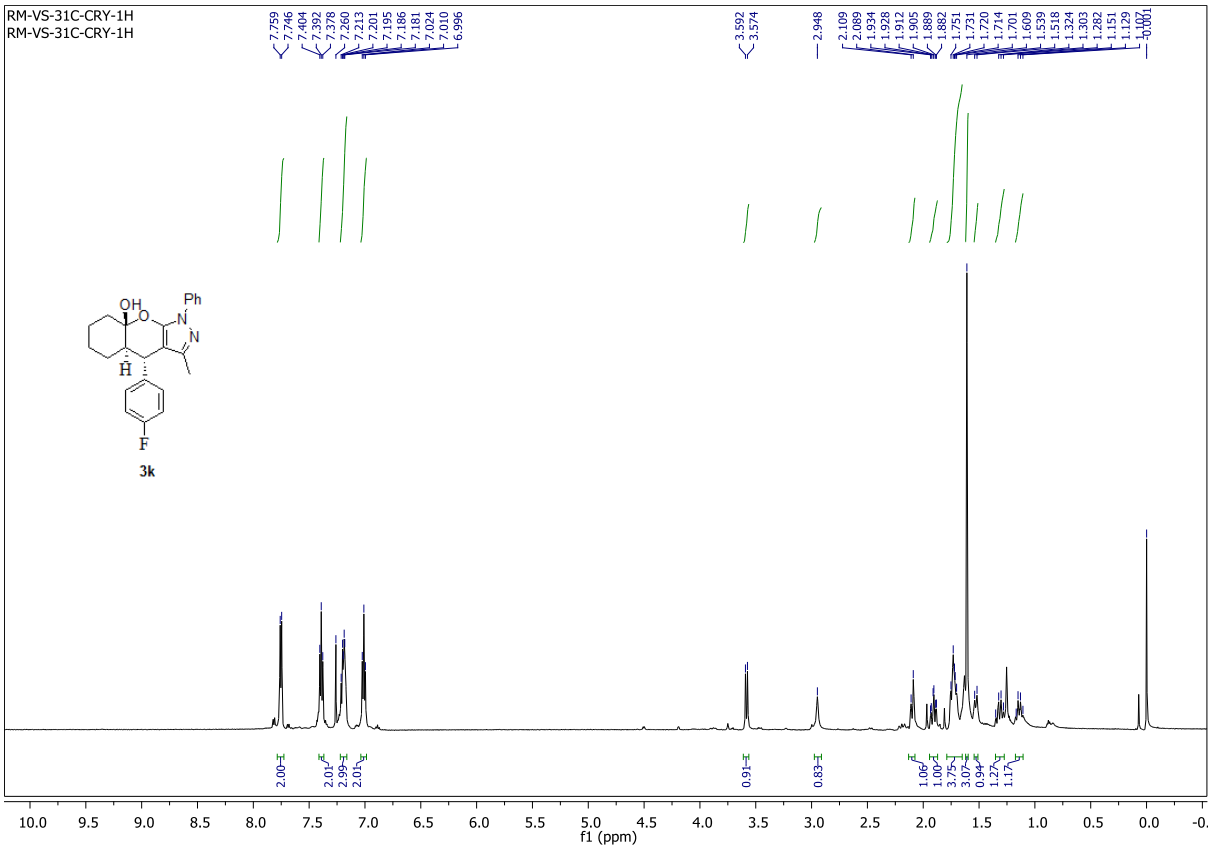

VS-37C-13C
156.91
142.50
133.67
129.94
129.08
125.41
120.50
119.17
116.00
103.27
100.37
77.55
76.91
70.09
47.90
39.68
38.64
32.14
26.95
25.68
22.29
13.66
OH
O
Ph
N
N
H
O
3h
f1 (ppm)

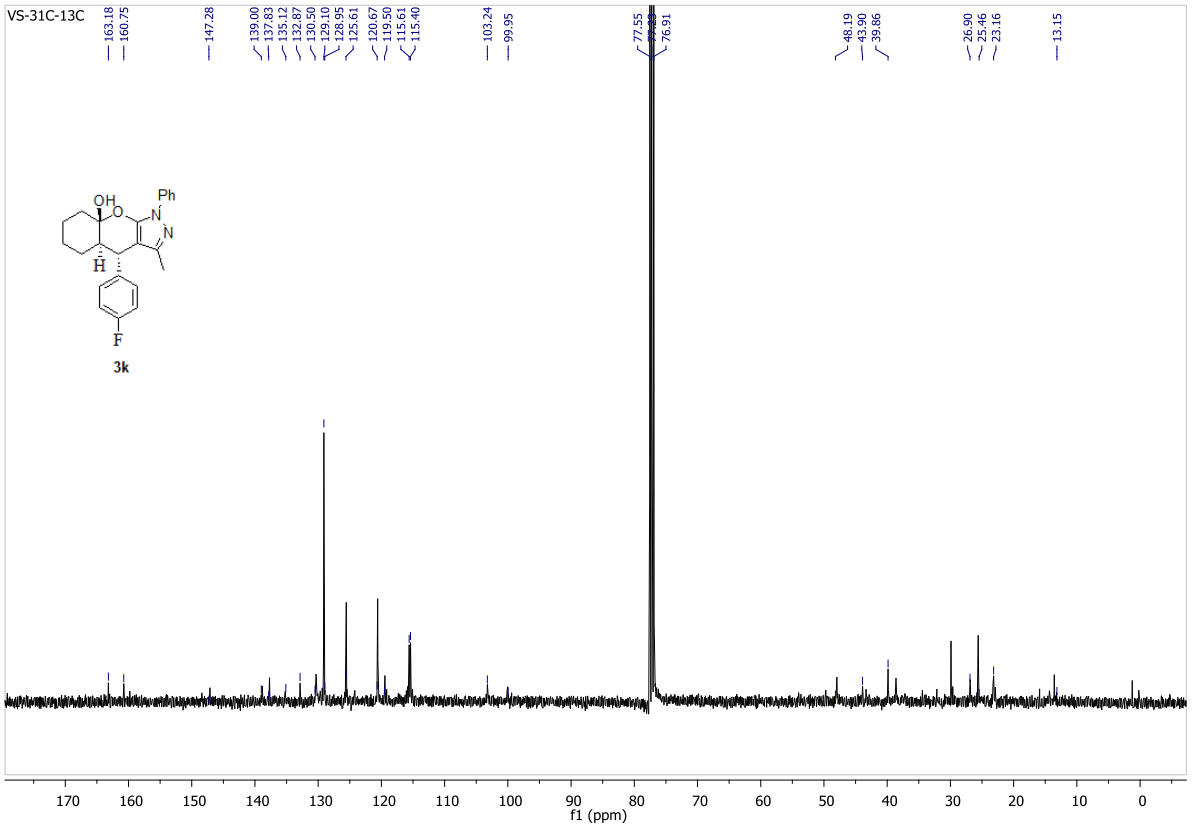

RM-VS-47C-CRY-1H
RM-VS-47C-CRY-1H
7.796 7.783 7.429 7.416 7.284 7.144 7.130 6.877 6.863
3.743 3.732 3.557 3.539
2.865
2.138 2.115 2.109 2.104 2.093 1.925 1.781 1.767 1.763 1.750 1.743 1.736 1.667 1.616 1.612 1.594 1.591 1.313 1.306 1.292 1.061 1.050 0.022
OH
O
N
N
Ph
H
O
3i
2.00 2.08 1.10 1.79 1.90 1.84 0.94 0.85 2.44 1.17 3.19 2.96 1.38 0.81 0.93 0.89 5.81
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

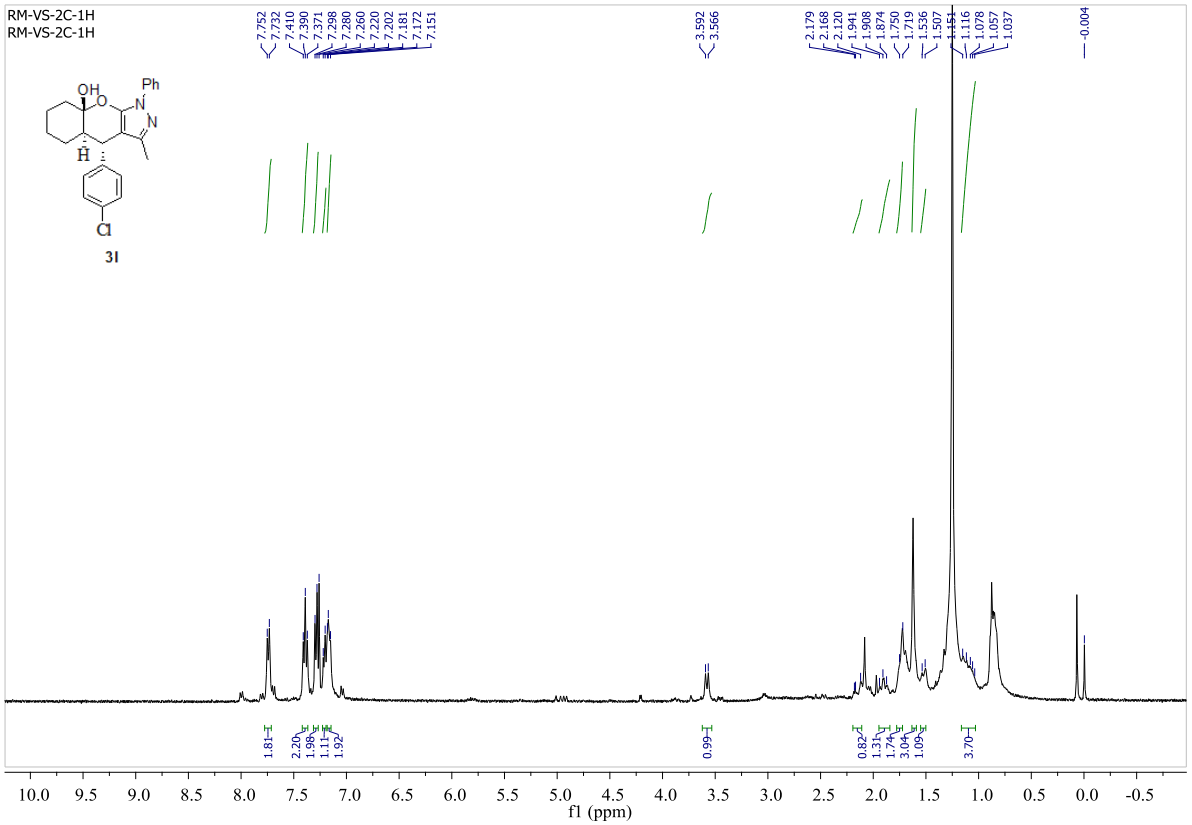

RM-VS-47C-13C
148.41 147.32 139.00 133.64 129.80 129.04 125.35 120.49 114.57 103.33 100.38
77.55 77.23 76.91 74.60
48.01 39.55 38.54 28.55 26.93 25.68 23.21 19.52 13.72
OH
O
N
N
Ph
H
O
3i
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

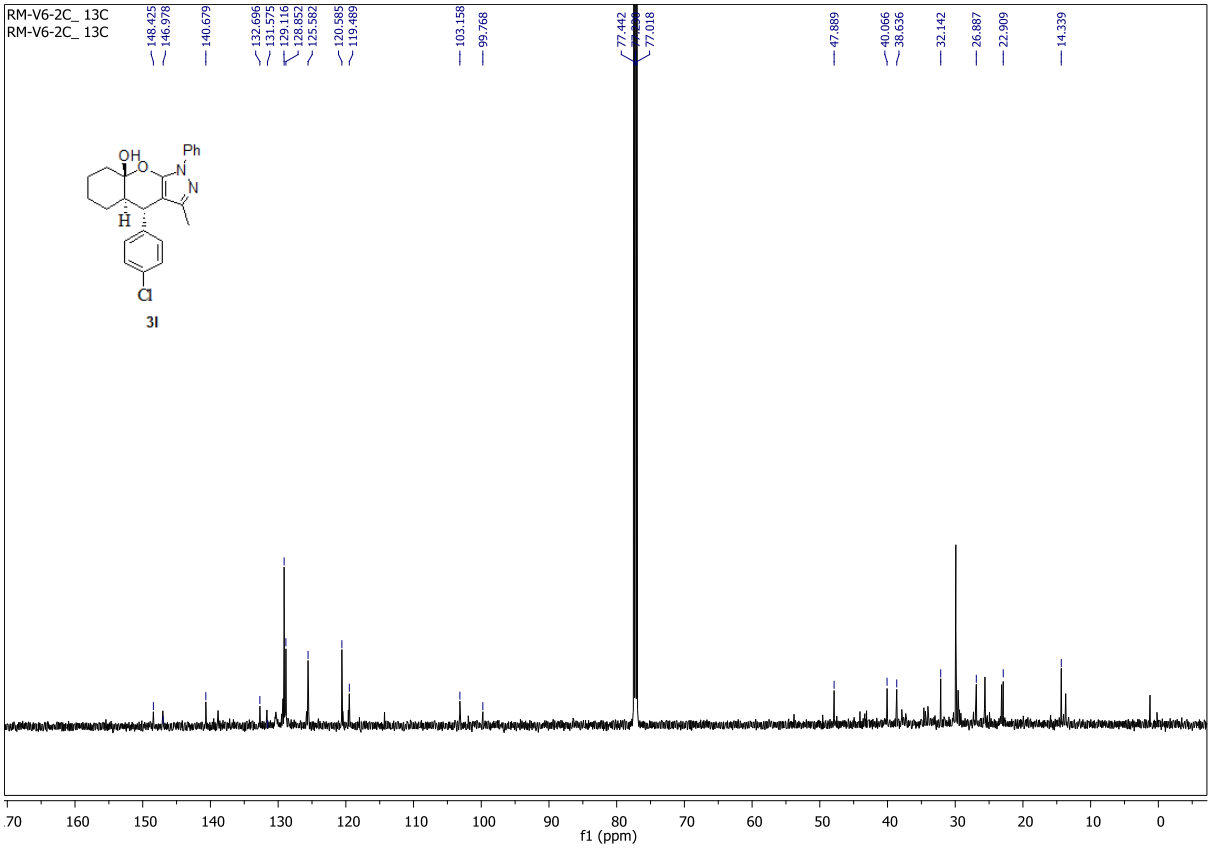

RM-VS-46C-CRY-1H
RM-VS-46C-CRY-1H
7.771
7.759
7.450
7.442
7.405
7.393
7.381
7.344
7.333
7.260
7.208
7.196
7.184
7.146
7.133
6.943
6.929
3.548
3.530
2.821
2.129
2.122
2.116
1.905
1.750
1.731
1.695
1.638
1.596
1.573
1.518
1.296
1.278
1.155
1.129
0.001
OH
O
N
Ph
N
H
O
3j
Ph
2.00
2.11
3.80
1.01
1.22
1.84
1.92
1.79
0.83
0.84
0.79
0.99
2.85
3.33
1.61
1.10
0.86
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

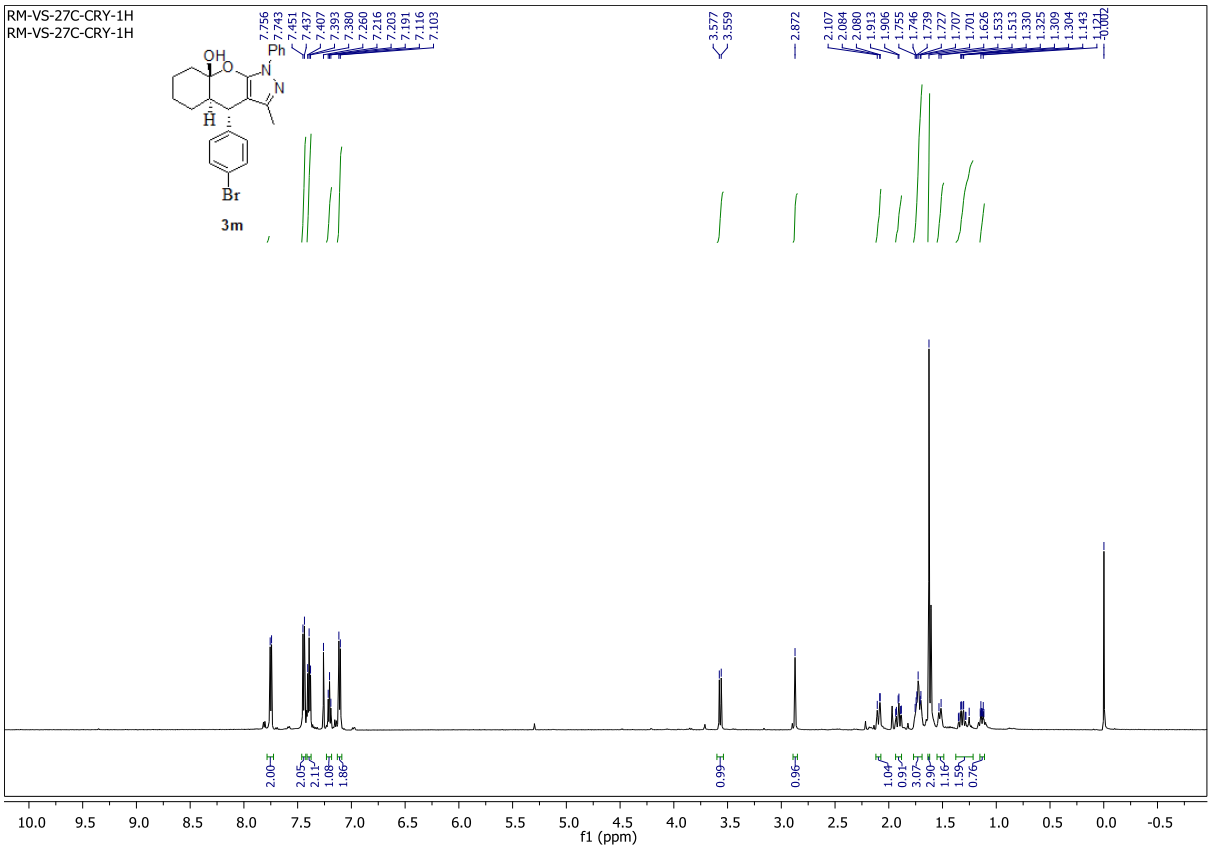

VS-46C-13C
157.83
137.20
129.04
128.75
128.16
127.79
125.38
120.48
118.96
114.89
103.33
100.34
70.24
47.93
39.63
38.50
29.88
25.64
23.18
13.60
OH
O
N
Ph
N
H
O
3j
Ph

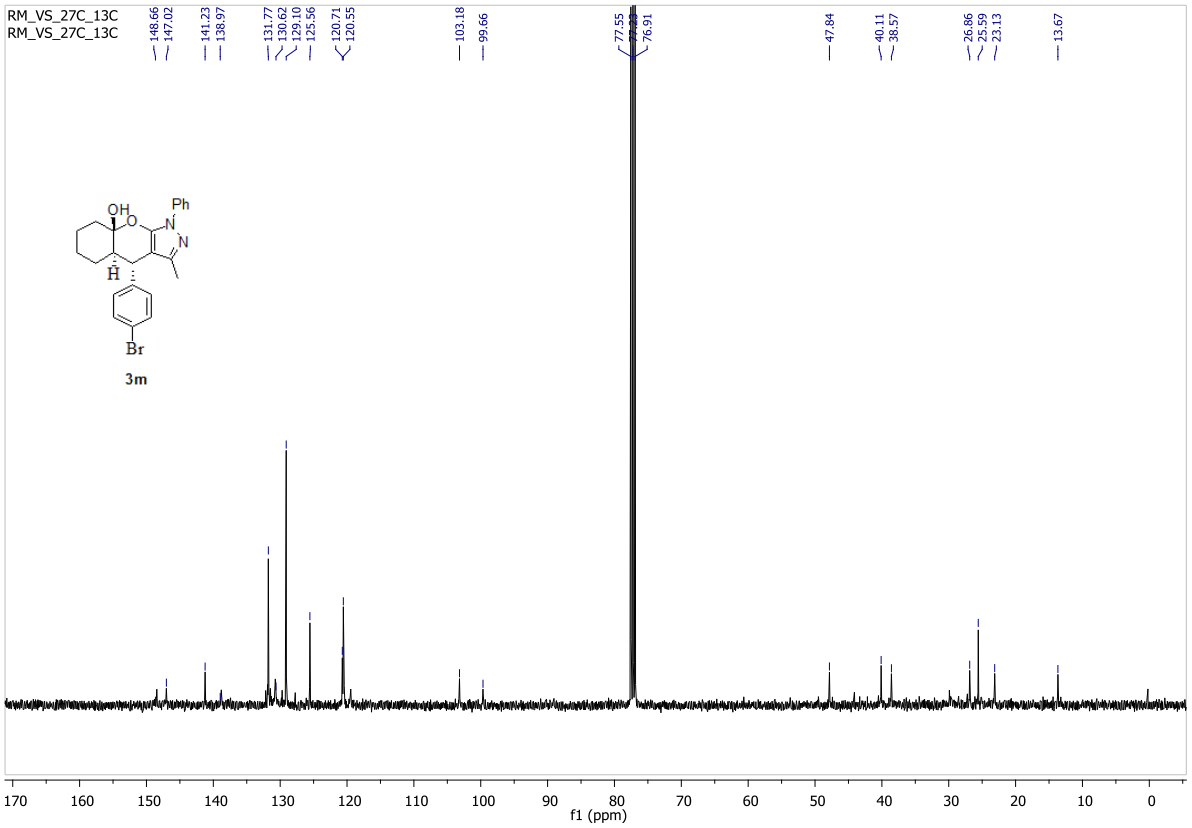

RM-VS-31C-CRY-1H
RM-VS-31C-CRY-1H
7.759
7.746
7.404
7.392
7.378
7.260
7.213
7.201
7.195
7.186
7.181
7.024
7.010
6.996
3.592
3.574
2.948
2.109
2.089
1.934
1.928
1.912
1.905
1.889
1.882
1.751
1.731
1.720
1.714
1.701
1.609
1.539
1.518
1.324
1.303
1.282
1.151
1.129
0.001
OH
O
N
N
Ph
H
F
3k
2.00
2.01
2.99
2.01
0.91
0.83
1.06
1.00
3.75
3.07
0.94
1.22
1.17
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

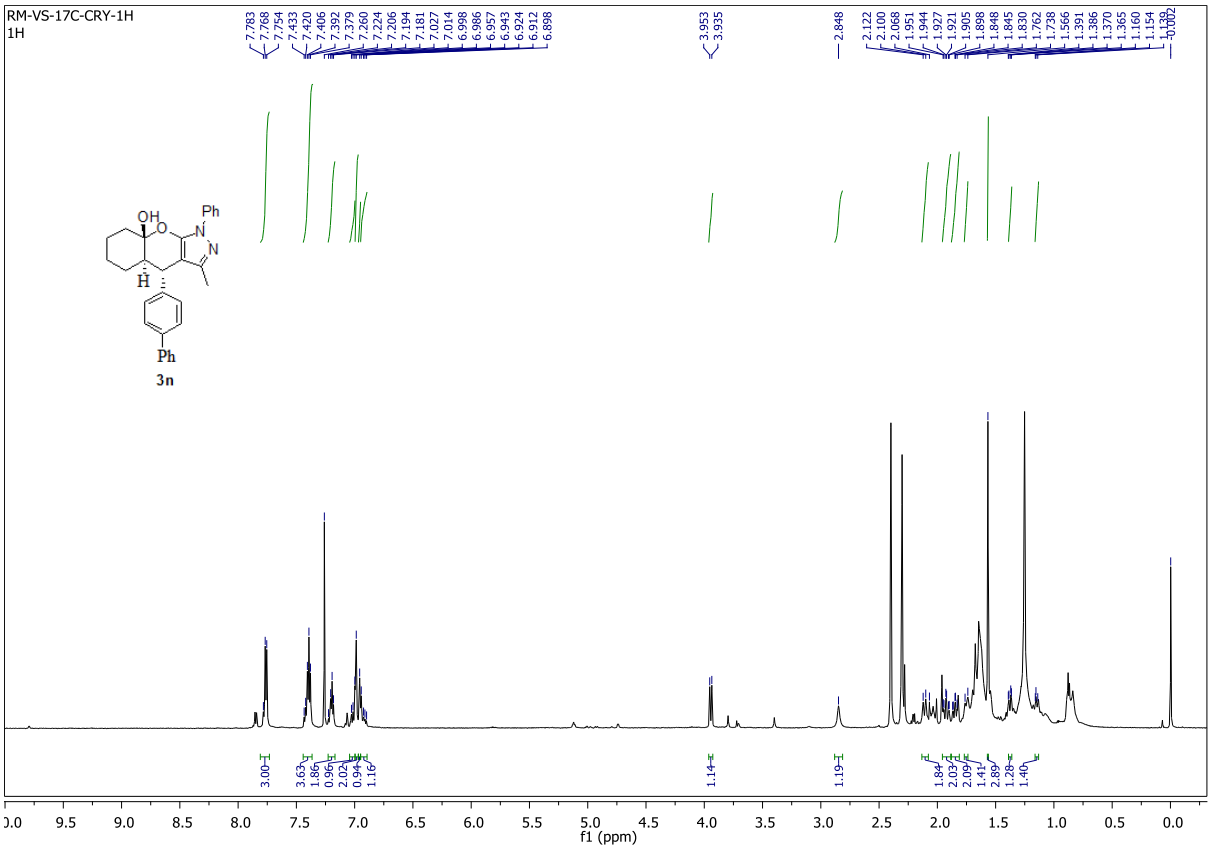

VS-31C-13C
163.18
160.75
147.28
139.00
137.83
135.12
132.87
130.50
129.10
128.95
125.61
120.67
119.50
115.61
115.40
103.24
99.95
77.55
77.23
76.91
48.19
43.90
39.86
26.90
25.46
23.16
13.15
OH
O
N
N
Ph
H
F
3k
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

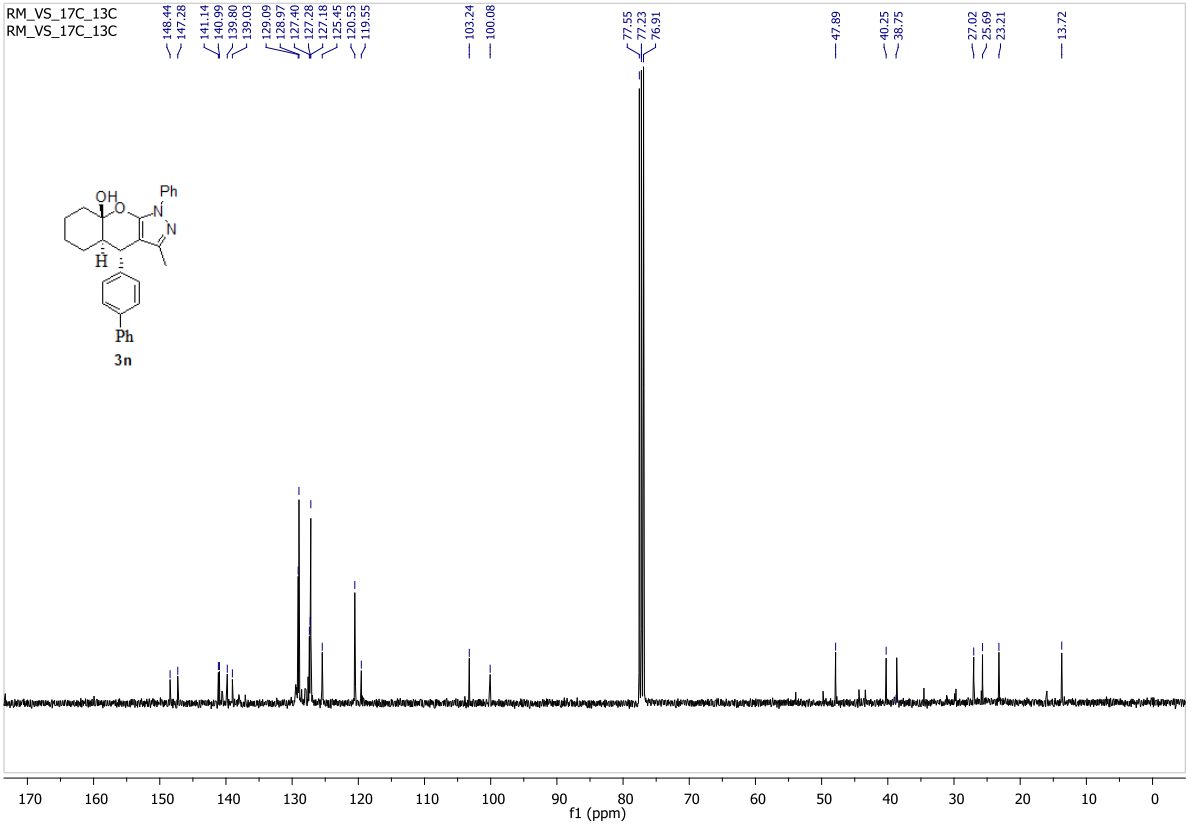

RM-VS-2C-1H
RM-VS-2C-1H
7.752
7.732
7.410
7.390
7.371
7.298
7.280
7.260
7.220
7.202
7.181
7.172
7.151
3.592
3.566
2.179
2.168
2.120
1.941
1.908
1.874
1.750
1.719
1.536
1.507
1.151
1.116
1.078
1.057
1.037
-0.004
OH
O
Ph
N
N
H
Cl
3l
1.81
2.20
1.98
1.11
1.92
0.99
0.82
1.31
1.74
3.04
1.09
3.70
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

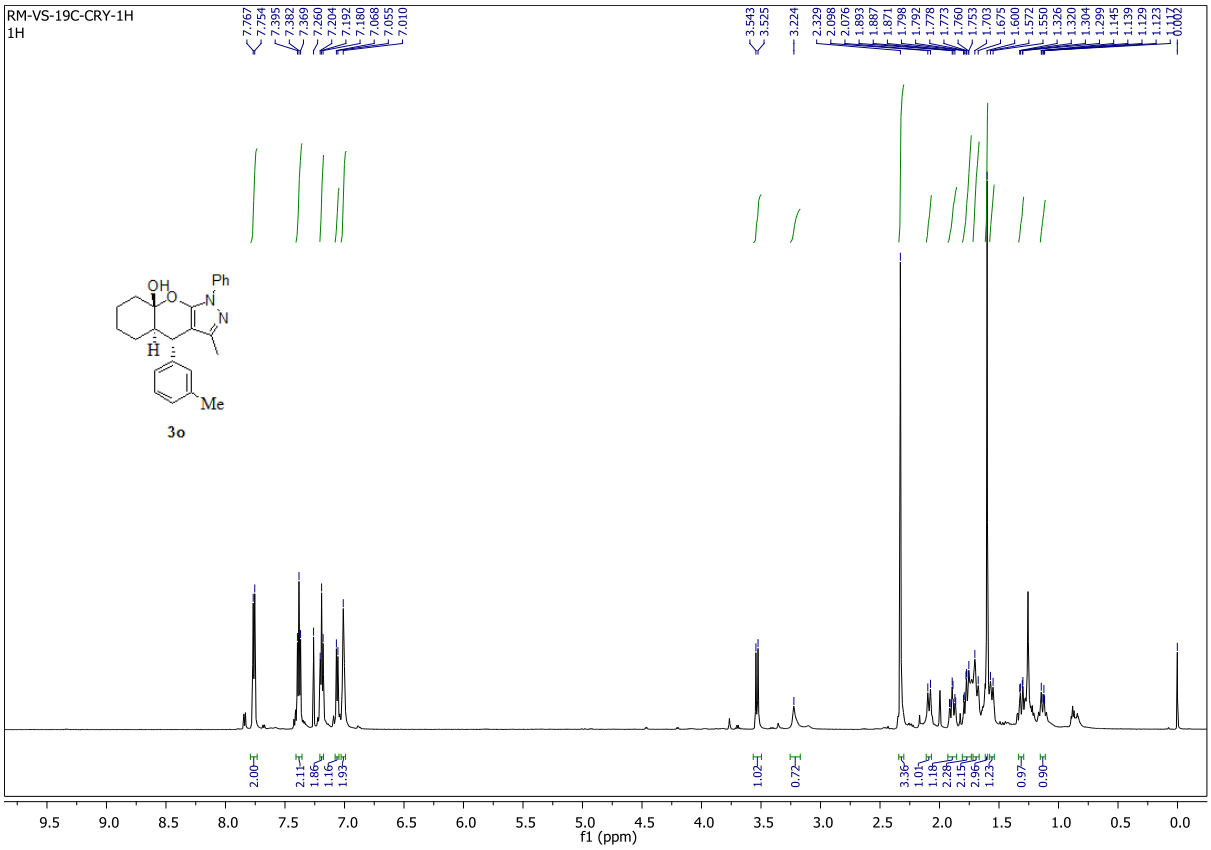

RM-V6-2C_ 13C
RM-V6-2C_ 13C
148.425
146.978
140.679
132.696
131.575
129.116
128.852
125.582
120.585
119.489
103.158
99.768
77.442
77.018
47.889
40.066
38.636
32.142
26.887
22.909
14.339
OH
O
Ph
N
N
H
Cl
3l
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

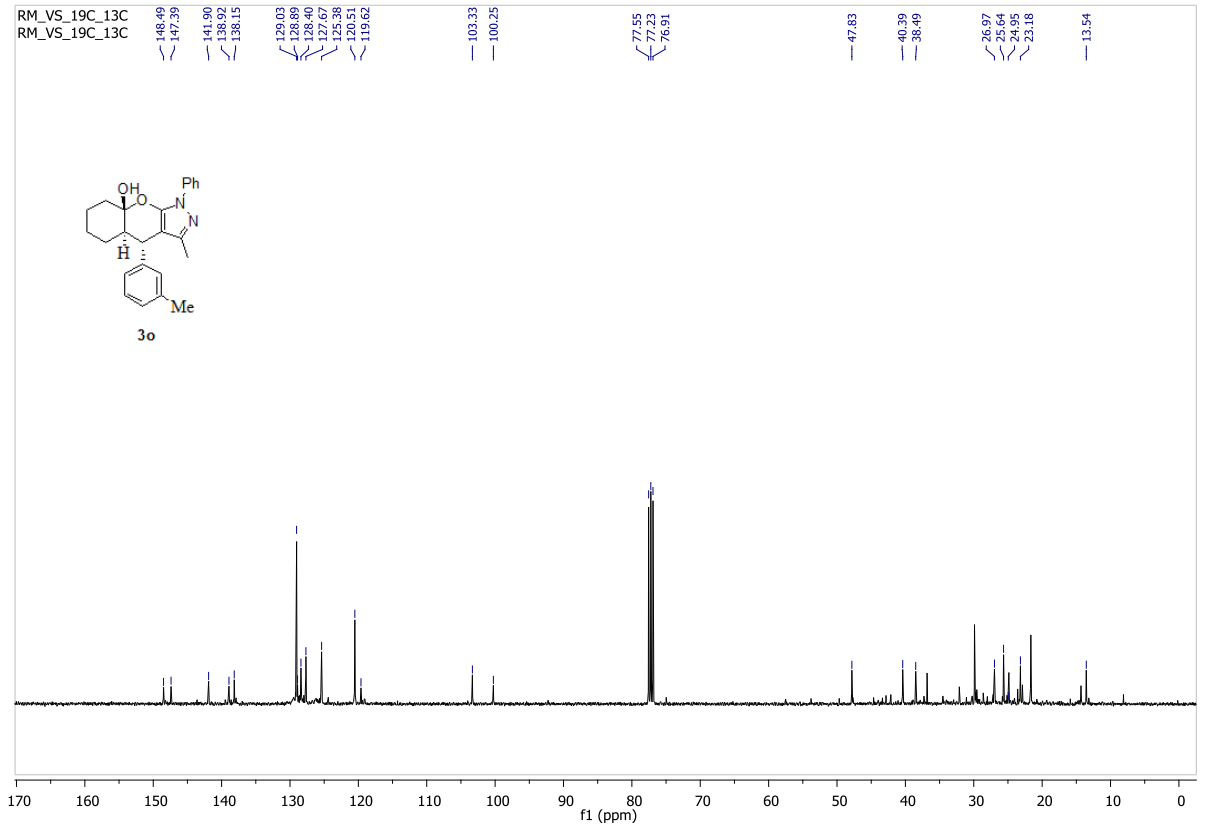

RM-VS-27C-CRY-1H
RM-VS-27C-CRY-1H
3m
f1 (ppm)

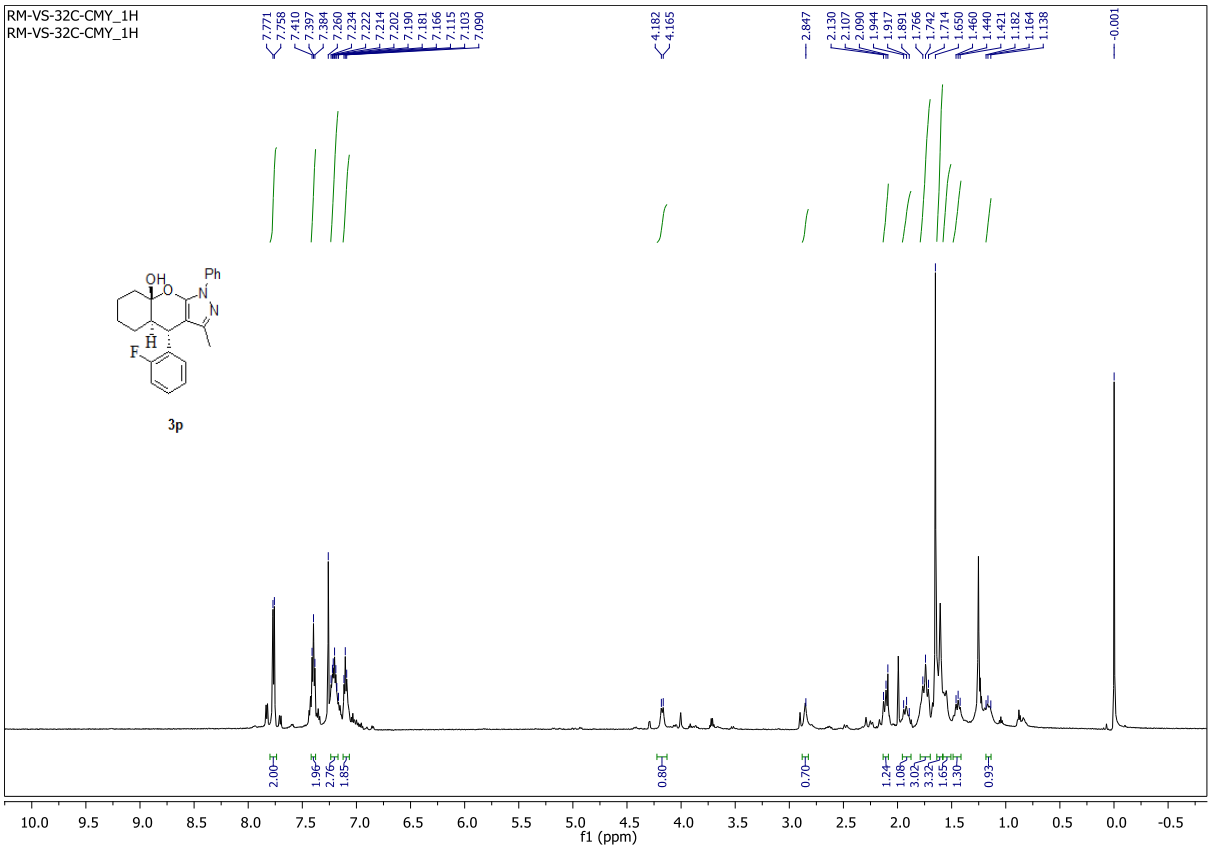

RM_VS_27C_13C
RM_VS_27C_13C
3m
f1 (ppm)

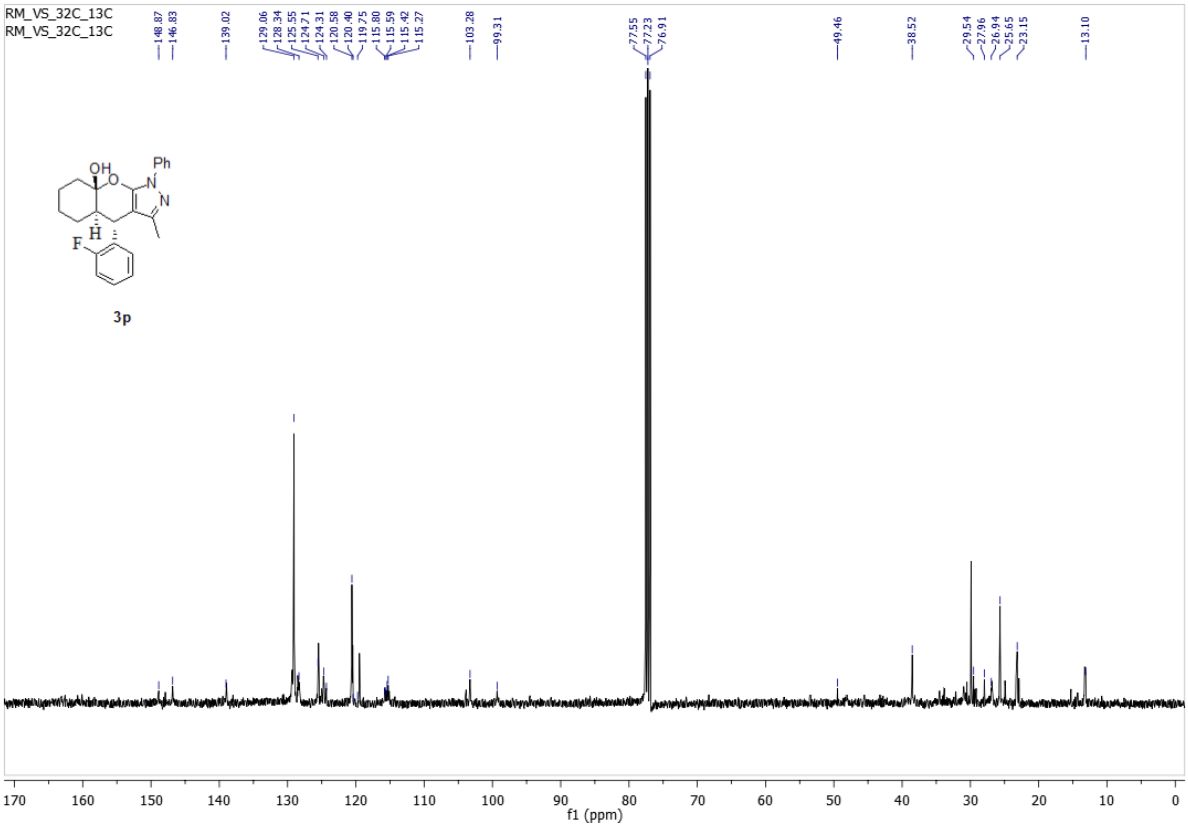

RM-VS-17C-CRY-1H
1H
f1 (ppm)

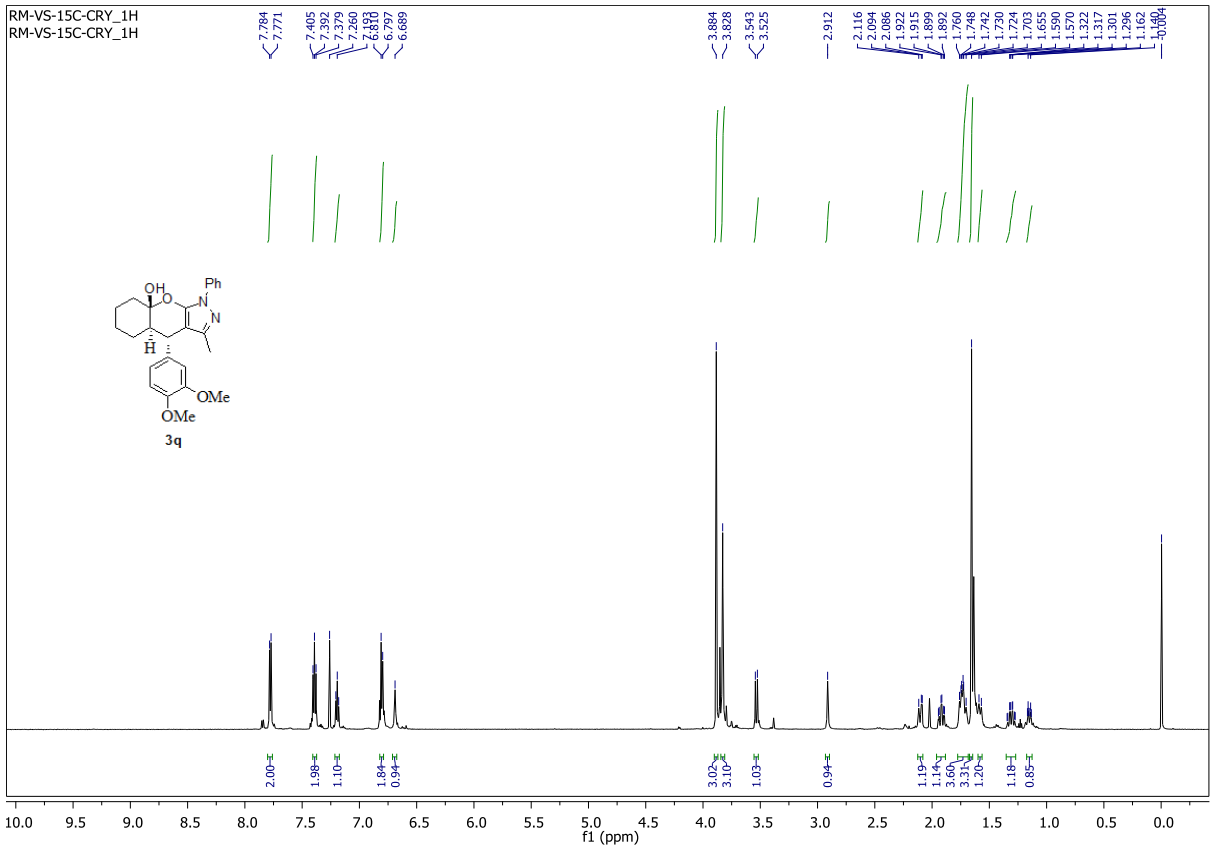

RM_VS_17C_13C
RM_VS_17C_13C
f1 (ppm)

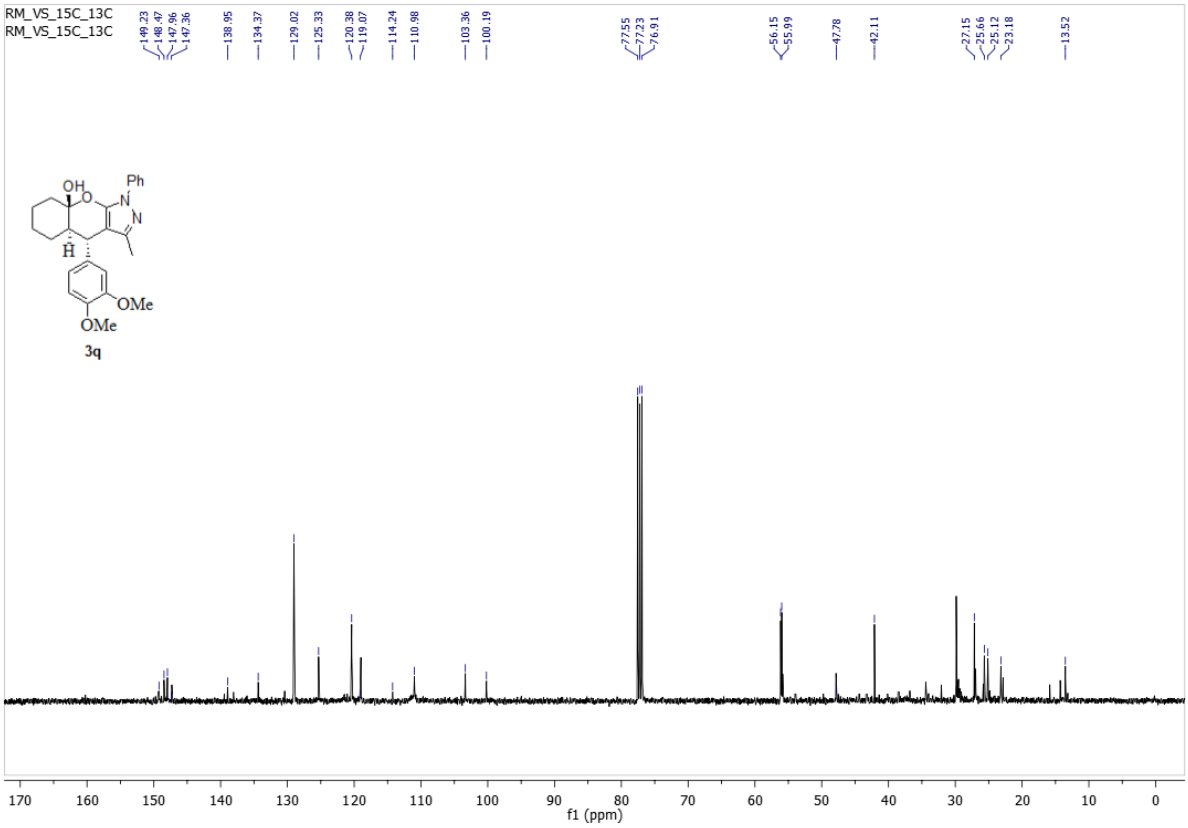

RM-VS-19C-CRY-1H
1H
7.767
7.754
7.395
7.382
7.369
7.260
7.204
7.192
7.180
7.068
7.055
7.010
3.543
3.525
3.224
2.329
2.098
2.076
1.893
1.887
1.871
1.798
1.792
1.778
1.773
1.760
1.753
1.703
1.675
1.600
1.572
1.550
1.326
1.320
1.304
1.299
1.145
1.139
1.129
1.123
1.117
0.002
OH
O
Ph
N
N
H
Me
3o
2.00
2.11
1.86
1.16
1.93
1.02
0.72
3.36
1.01
1.18
2.28
2.15
2.96
1.23
0.97
0.90
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

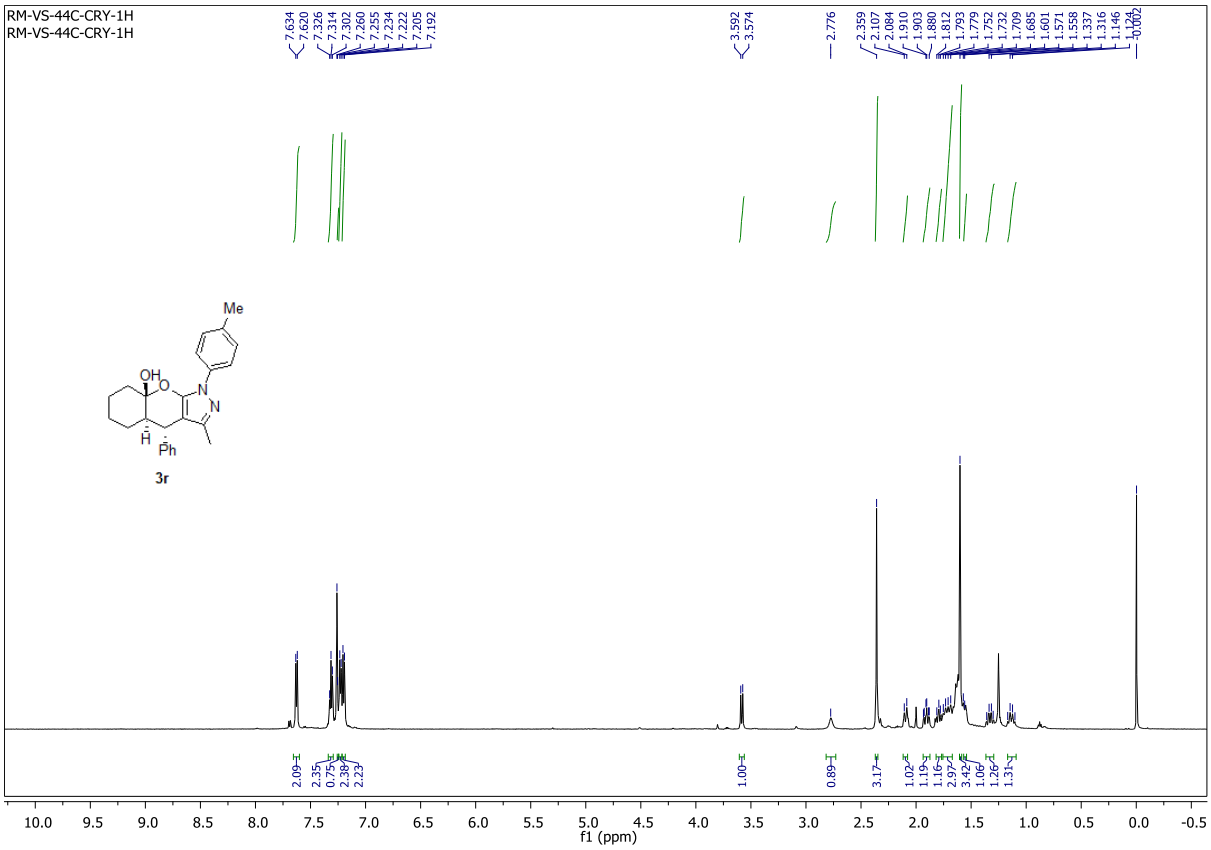

RM_VS_19C_13C
RM_VS_19C_13C
148.49
147.39
141.90
138.92
138.15
129.03
128.89
128.40
127.67
125.38
120.51
119.62
103.33
100.25
77.55
77.23
76.91
47.83
40.39
38.49
26.97
25.64
24.95
23.18
13.54
OH
O
Ph
N
N
H
Me
3o
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

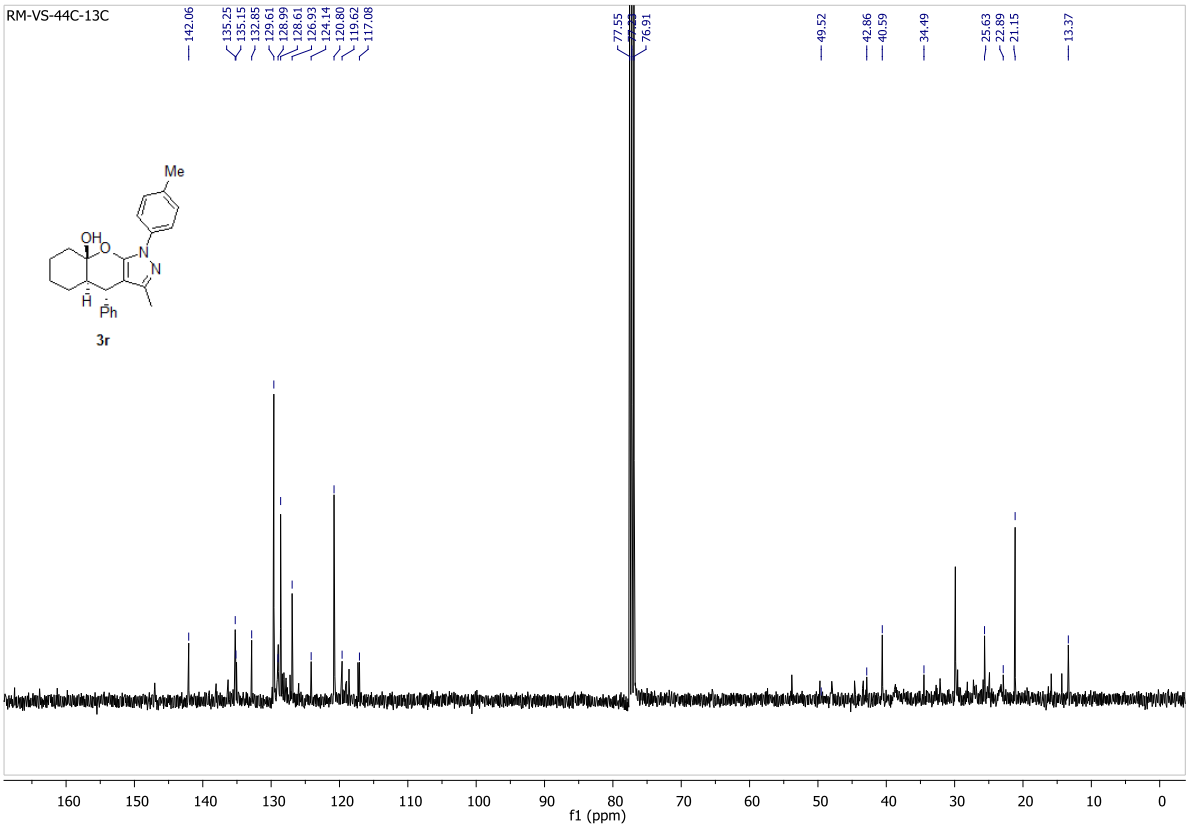

RM-VS-32C-CMY_1H
RM-VS-32C-CMY_1H
7.771
7.758
7.410
7.397
7.384
7.260
7.234
7.222
7.214
7.202
7.190
7.181
7.166
7.115
7.103
7.090
4.182
4.165
2.847
2.130
2.107
2.090
1.944
1.917
1.891
1.766
1.742
1.714
1.650
1.460
1.440
1.421
1.182
1.164
1.138
-0.001
OH
O
Ph
N
N
H
F
3p
2.00
1.96
2.76
1.85
0.80
0.70
1.24
1.08
3.02
3.32
1.65
1.30
0.93
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

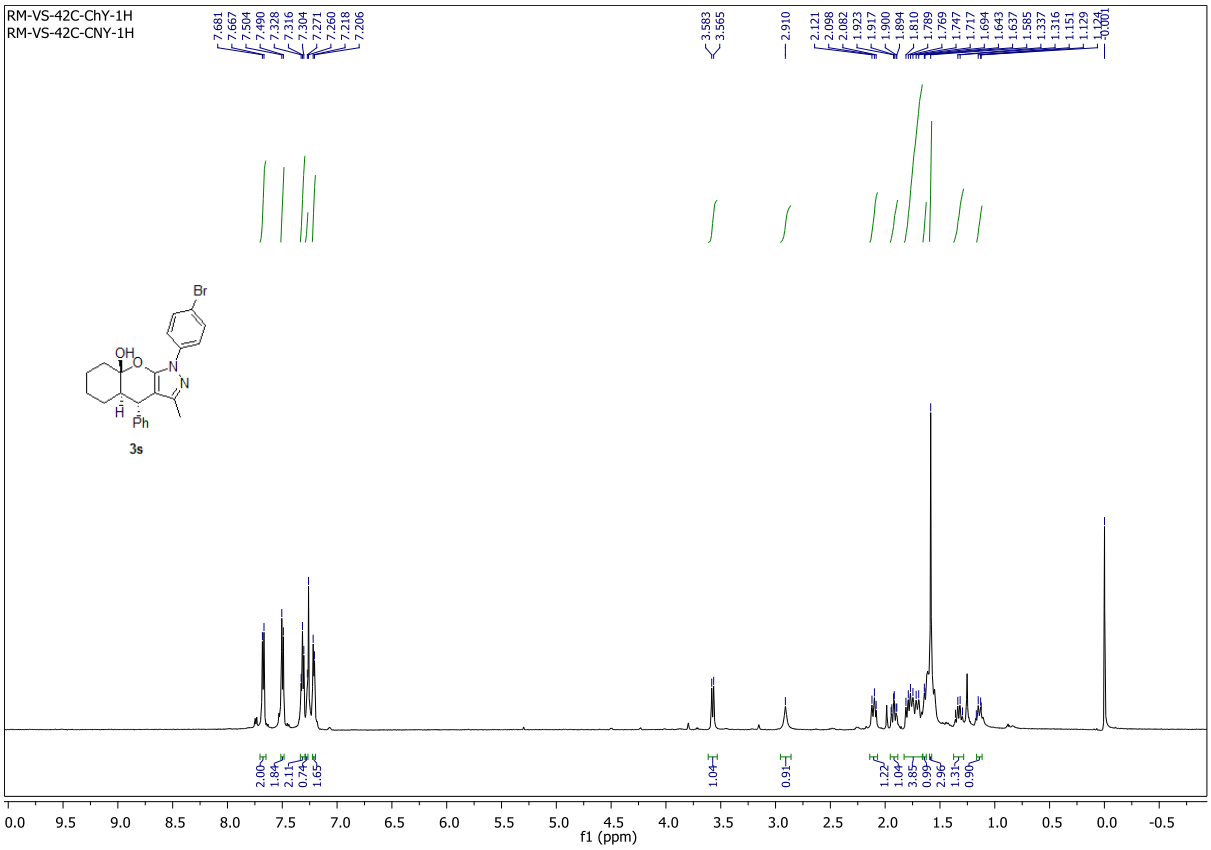

RM_VS_32C_13C
RM_VS_32C_13C
148.87
146.83
139.02
129.06
128.34
125.55
124.71
124.31
120.58
120.40
119.75
115.80
115.59
115.42
115.27
103.28
99.31
77.55
77.23
76.91
49.46
38.52
29.54
27.96
26.94
25.65
23.15
13.10
OH
O
Ph
N
N
H
F
3p
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

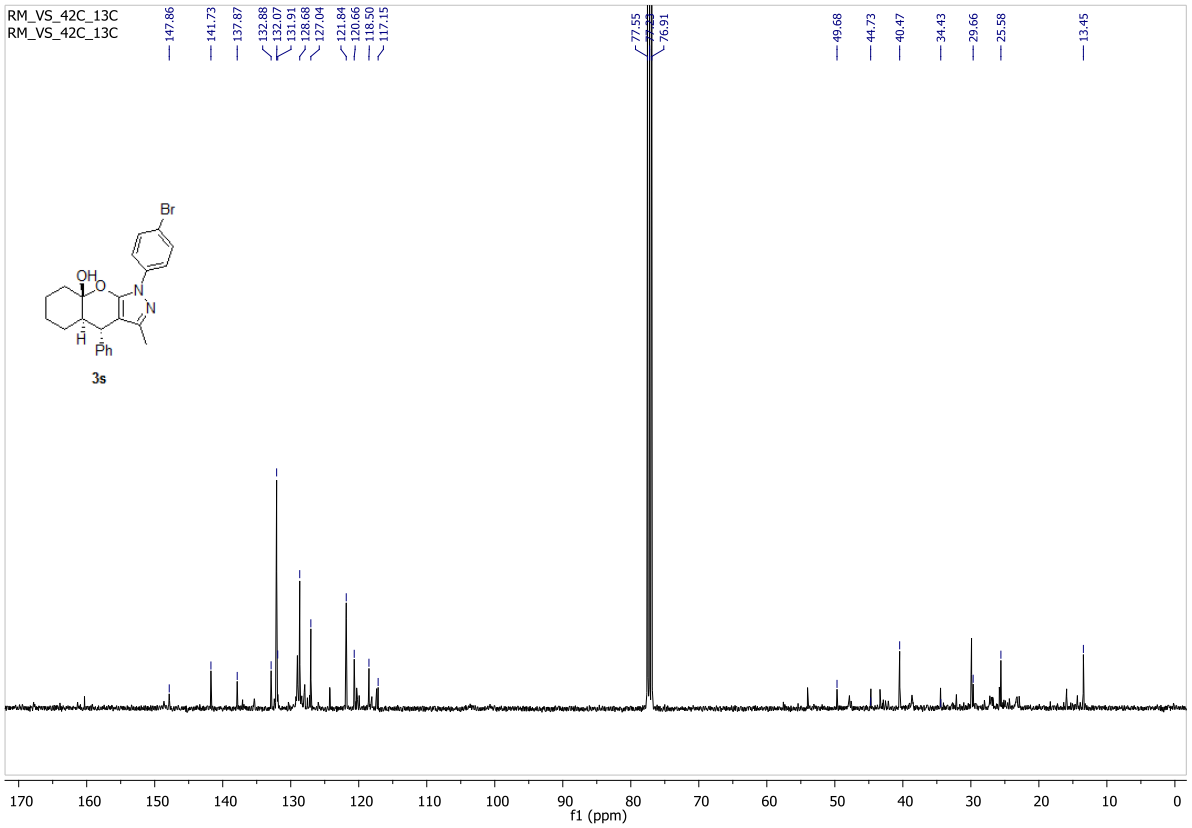

RM-VS-15C-CRY_1H
RM-VS-15C-CRY_1H
f1 (ppm)

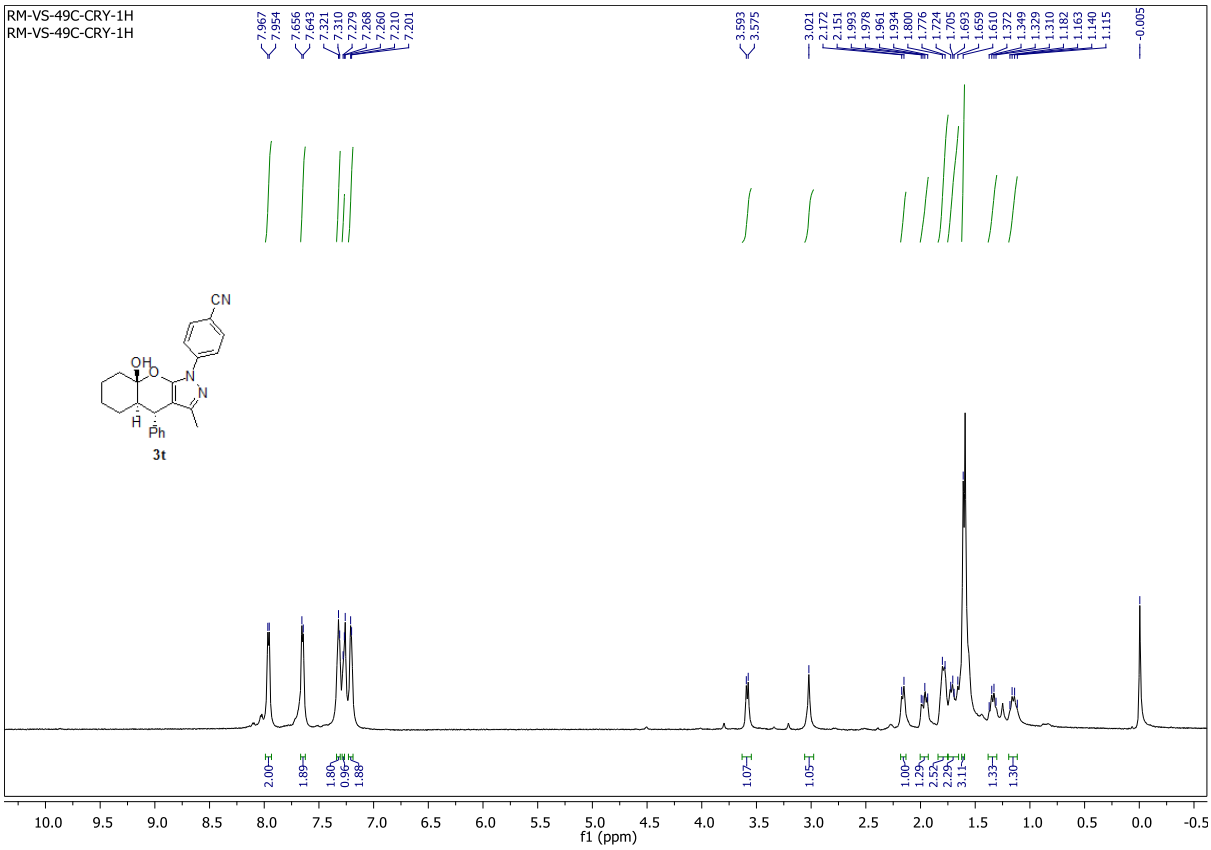

RM_VS_15C_13C
RM_VS_15C_13C
f1 (ppm)

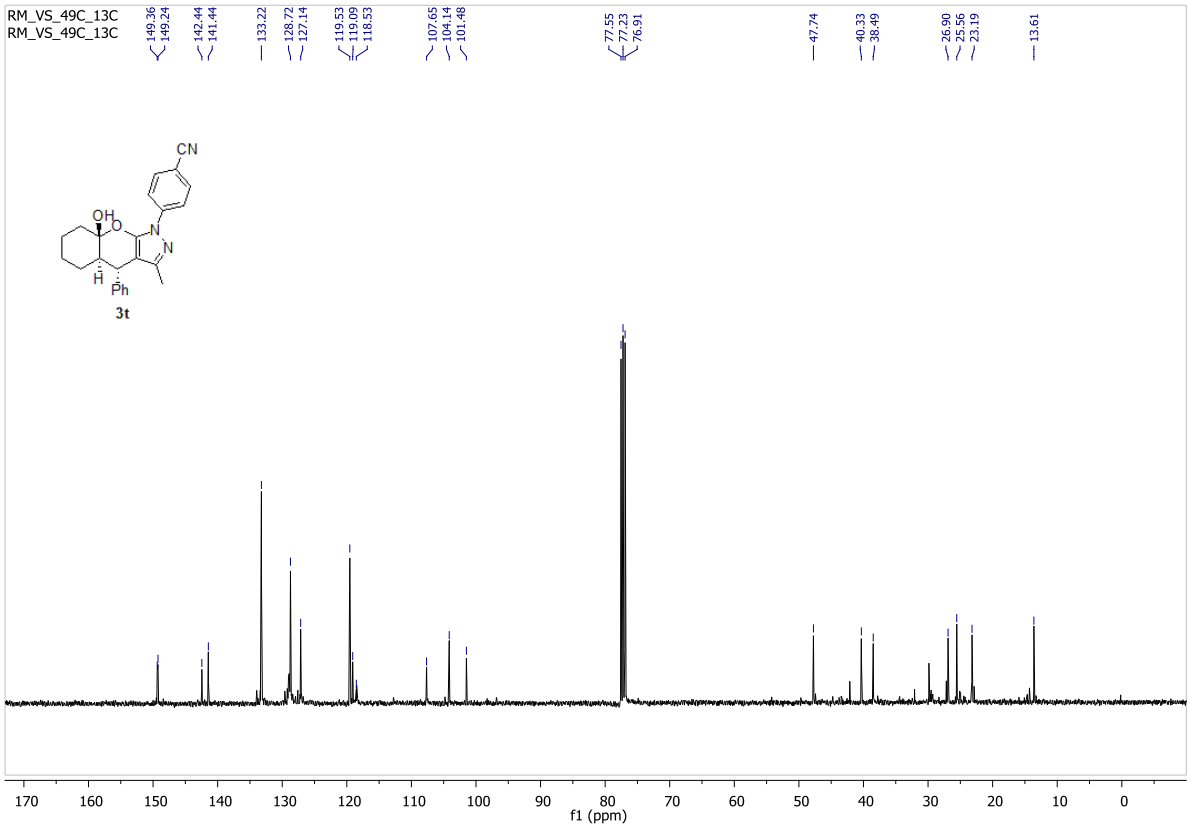

RM-VS-44C-CRY-1H
RM-VS-44C-CRY-1H
Me
OH
O
N
N
H
Ph
3r
f1 (ppm)

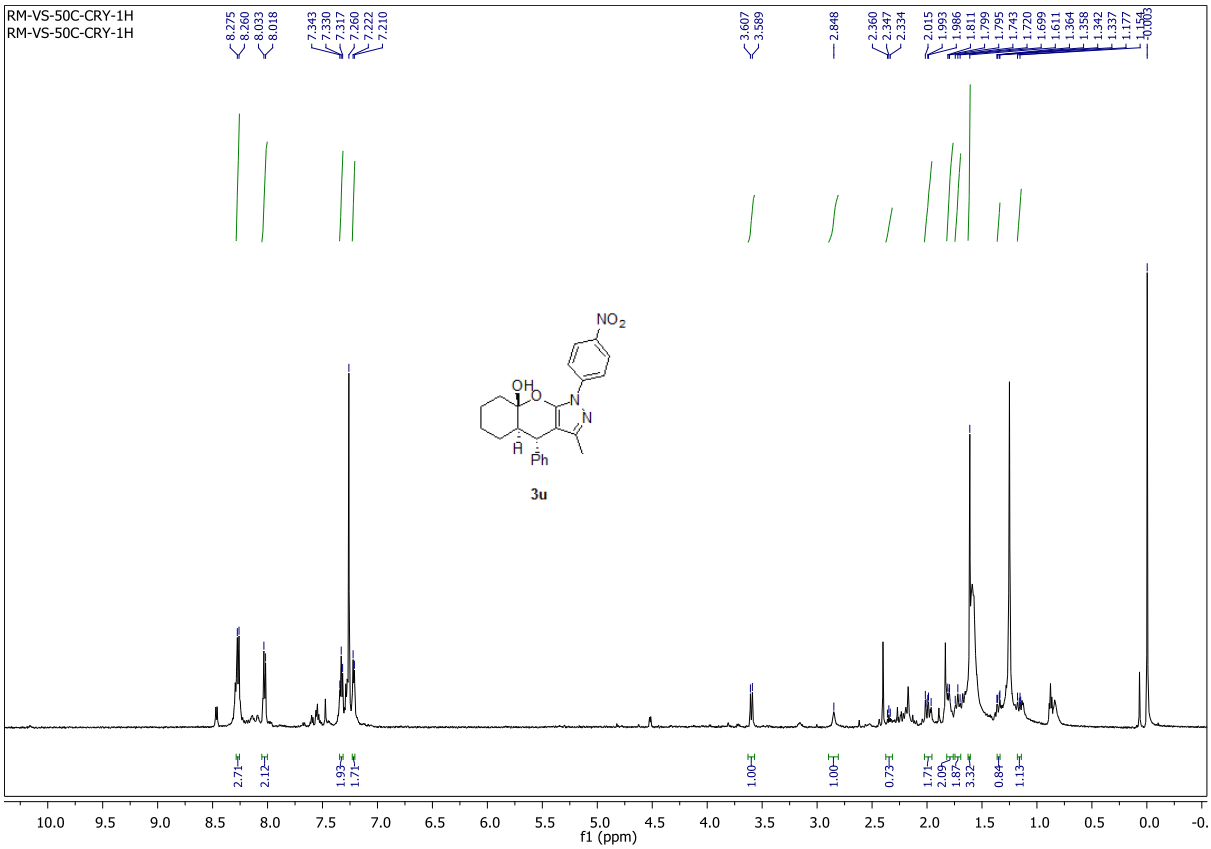

RM-VS-44C-13C
Me
OH
O
N
N
H
Ph
3r
f1 (ppm)

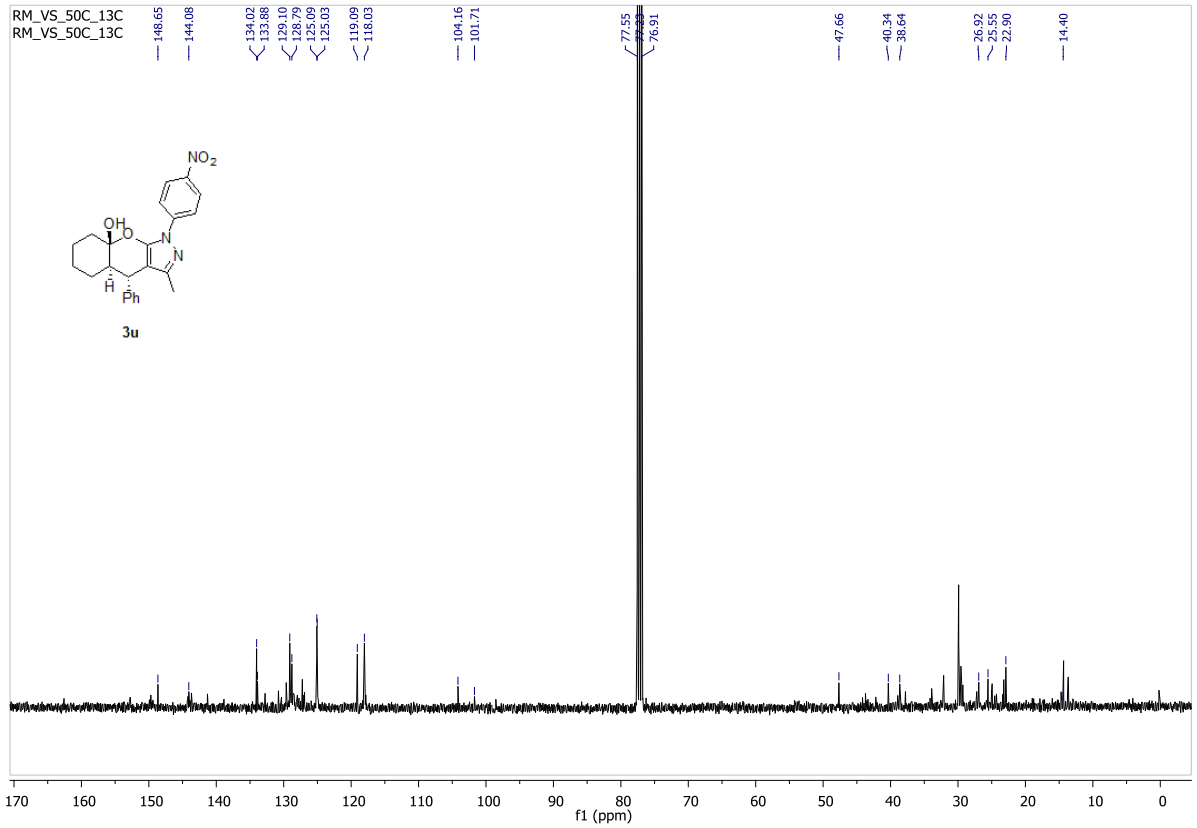

RM-VS-42C-ChY-1H
RM-VS-42C-CNY-1H
7.681
7.667
7.504
7.490
7.328
7.316
7.304
7.271
7.260
7.218
7.206
3.583
3.565
2.910
2.121
2.098
2.082
2.082
1.923
1.917
1.900
1.894
1.810
1.789
1.769
1.747
1.717
1.694
1.643
1.637
1.585
1.337
1.316
1.151
1.129
1.124
0.001
Br
OH
O
N
N
H
Ph
3s
2.00
1.84
2.11
0.74
1.65
1.04
0.91
1.22
1.04
3.85
0.99
2.96
1.31
0.90
f1 (ppm)

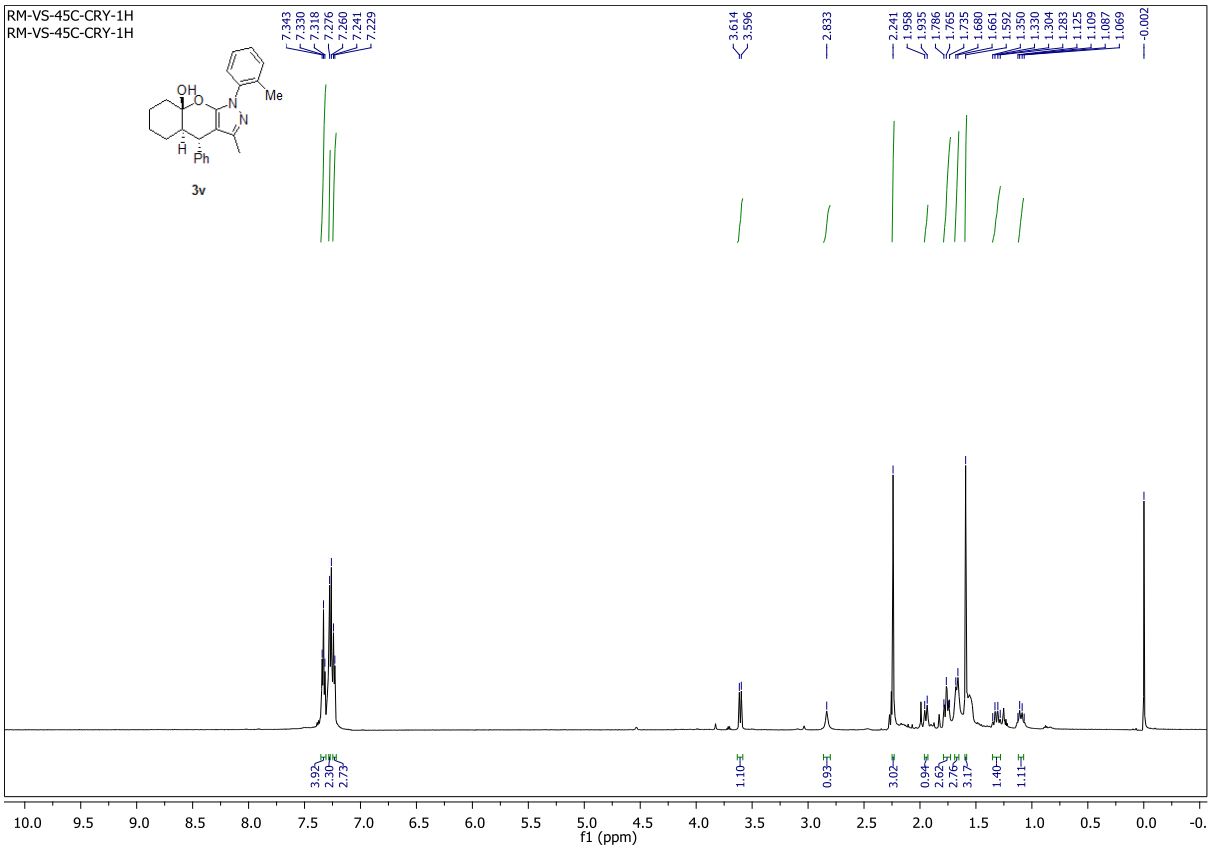

RM_VS_42C_13C
RM_VS_42C_13C
147.86
141.73
137.87
132.88
132.07
131.91
128.68
127.04
121.84
120.66
118.50
117.15
77.55
77.23
76.91
49.68
44.73
40.47
34.43
29.66
25.58
13.45
Br
OH
O
N
N
H
Ph
3s
f1 (ppm)

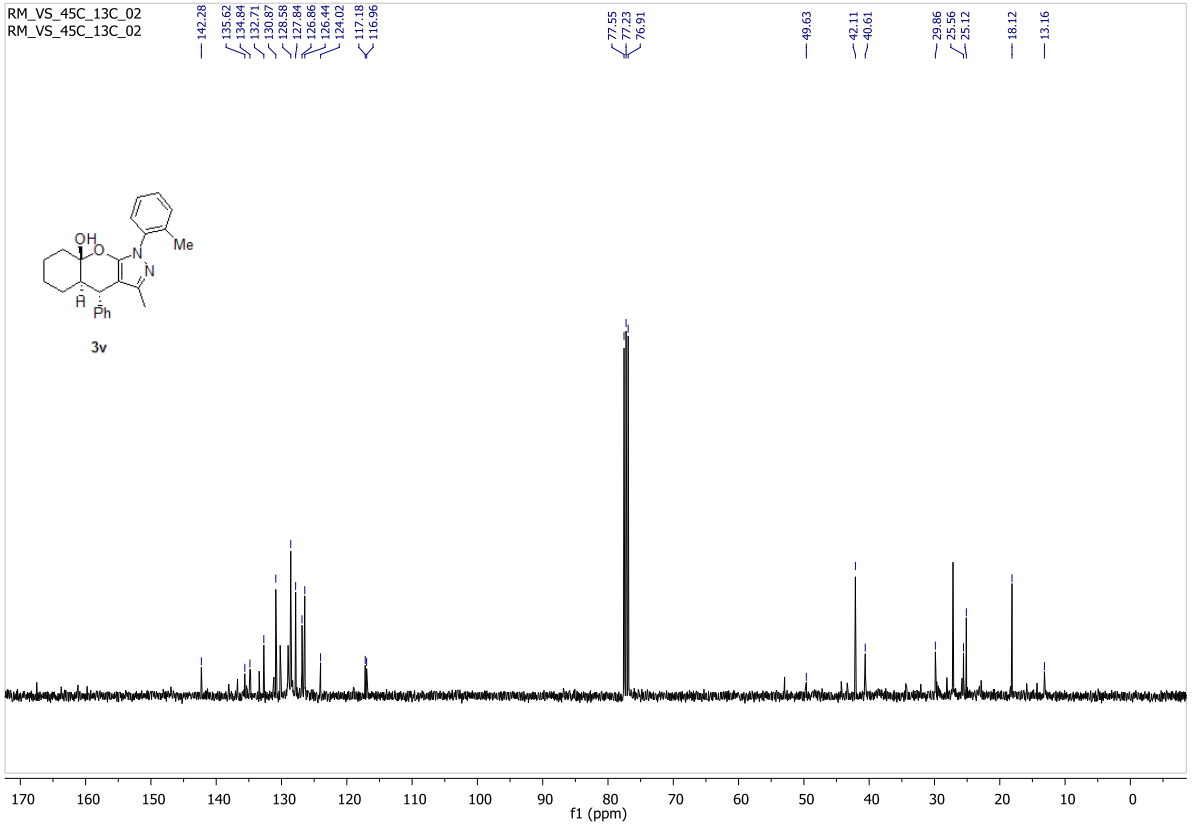

RM-VS-49C-CRY-1H
RM-VS-49C-CRY-1H
7.967
7.954
7.656
7.643
7.321
7.310
7.279
7.268
7.260
7.210
7.201
3.593
3.575
3.021
2.172
2.151
1.993
1.978
1.961
1.934
1.800
1.776
1.724
1.705
1.693
1.659
1.610
1.372
1.349
1.329
1.310
1.182
1.165
1.140
1.115
-0.005
CN
OH
O
N
N
H
Ph
3t
2.00
1.89
1.80
0.96
1.89
1.07
1.05
1.00
1.29
2.52
2.29
3.11
1.33
1.30
f1 (ppm)

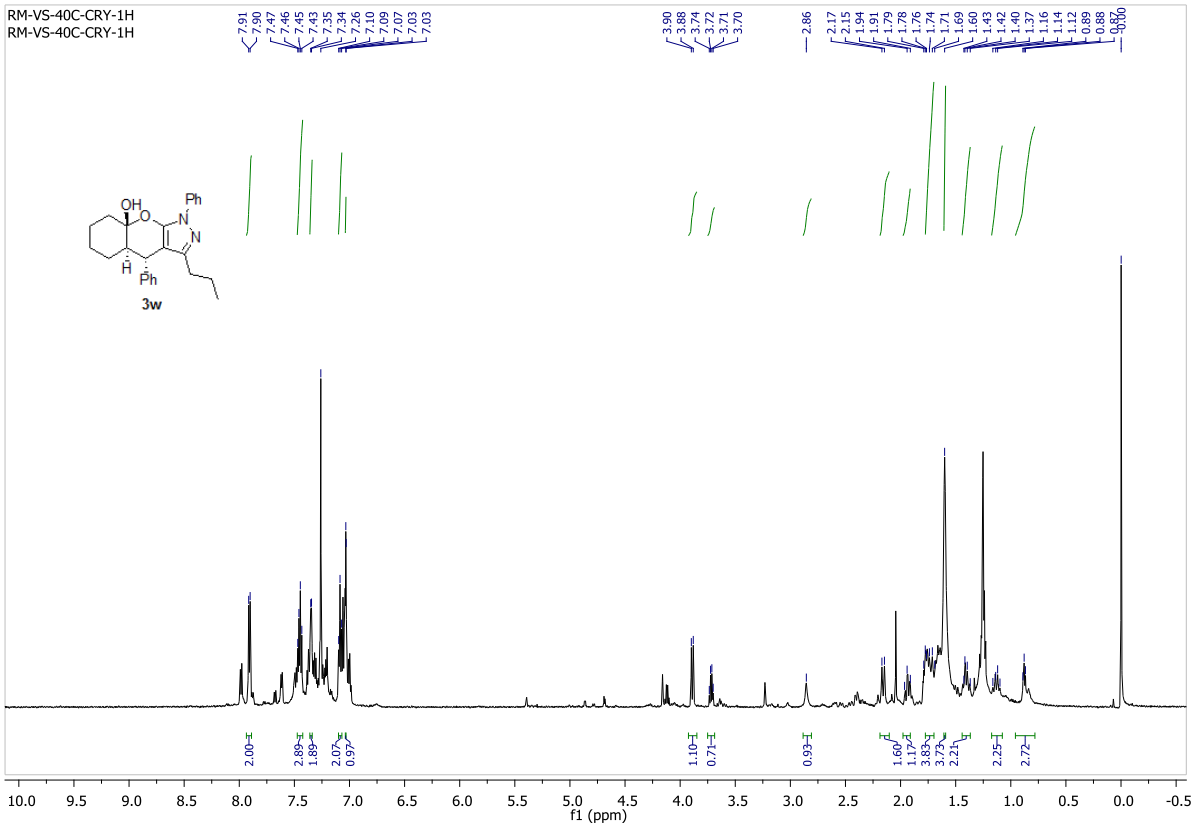

RM_VS_49C_13C
RM_VS_49C_13C
149.36
149.24
142.44
141.44
133.22
128.72
127.14
119.53
119.09
118.53
107.65
104.14
101.48
77.55
77.23
76.91
47.74
40.33
38.49
26.90
25.56
23.19
13.61
CN
OH
O
N
N
H
Ph
3t
f1 (ppm)

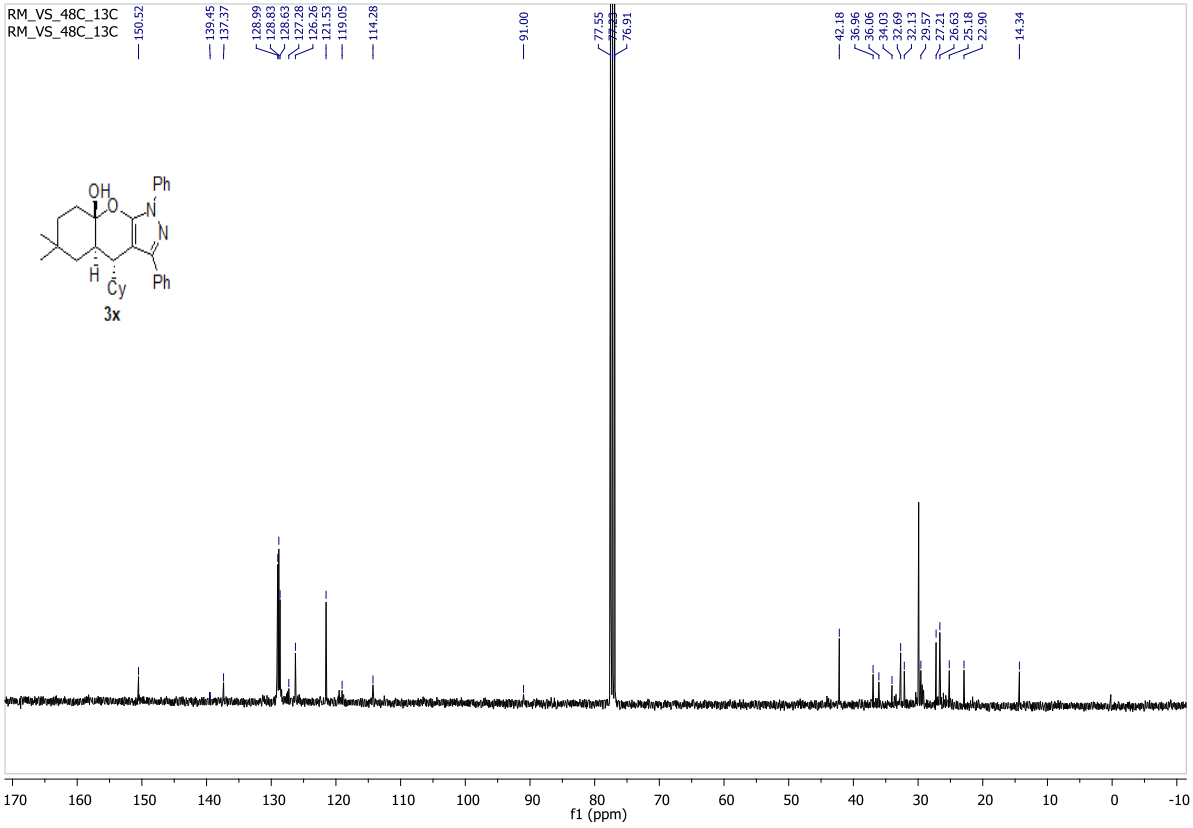

RM-VS-50C-CRY-1H
RM-VS-50C-CRY-1H
8.275
8.260
8.033
8.018
7.343
7.330
7.317
7.260
7.222
7.210
3.607
3.589
2.848
2.360
2.347
2.334
2.015
1.993
1.986
1.811
1.799
1.795
1.743
1.720
1.699
1.611
1.364
1.358
1.342
1.337
1.177
1.156
0.803
NO2
OH
O
N
N
H
Ph
3u
2.71
2.12
1.93
1.71
1.00
1.00
0.73
1.71
2.09
1.87
3.32
0.84
1.13
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

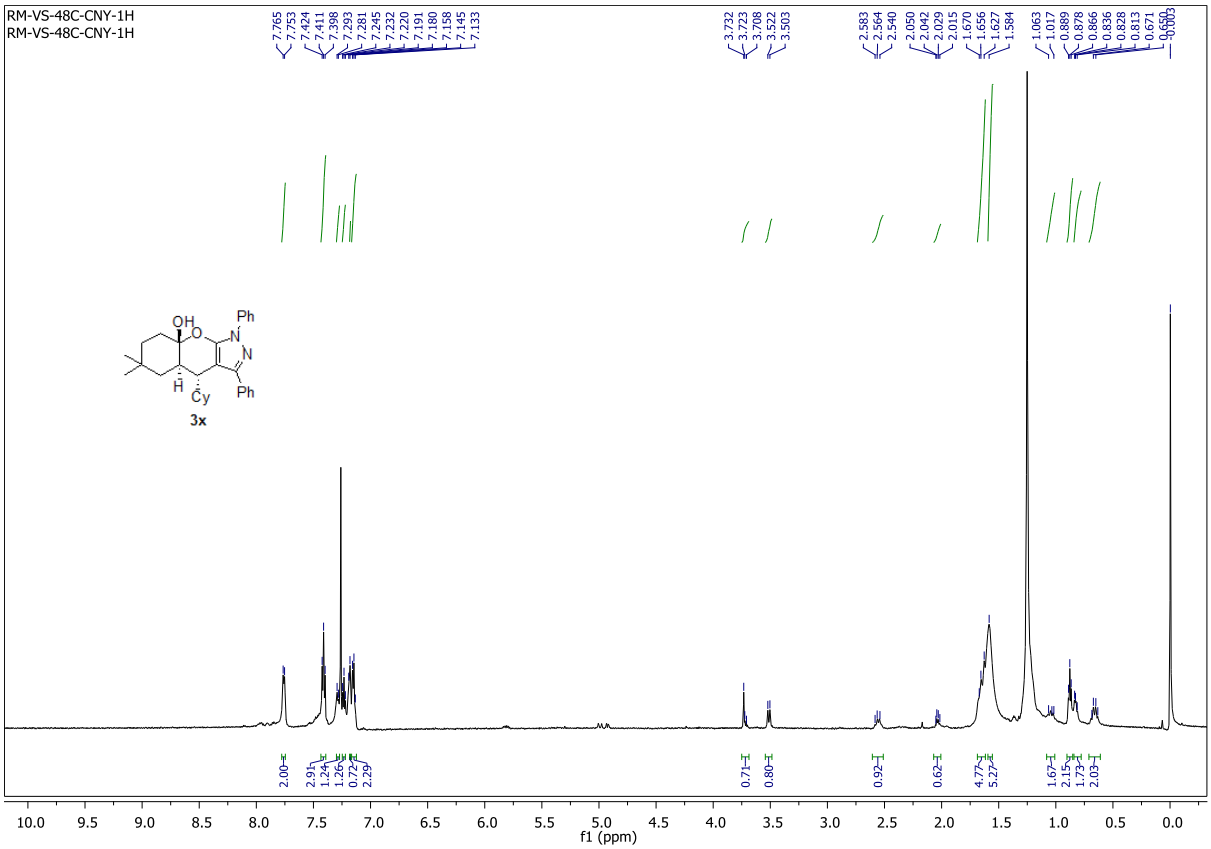

RM_VS_50C_13C
RM_VS_50C_13C
148.65
144.08
134.02
133.88
129.10
128.79
125.09
125.03
119.09
118.03
104.16
101.71
77.55
77.23
76.91
47.66
40.34
38.64
26.92
25.55
22.90
14.40
NO2
OH
O
N
N
H
Ph
3u
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

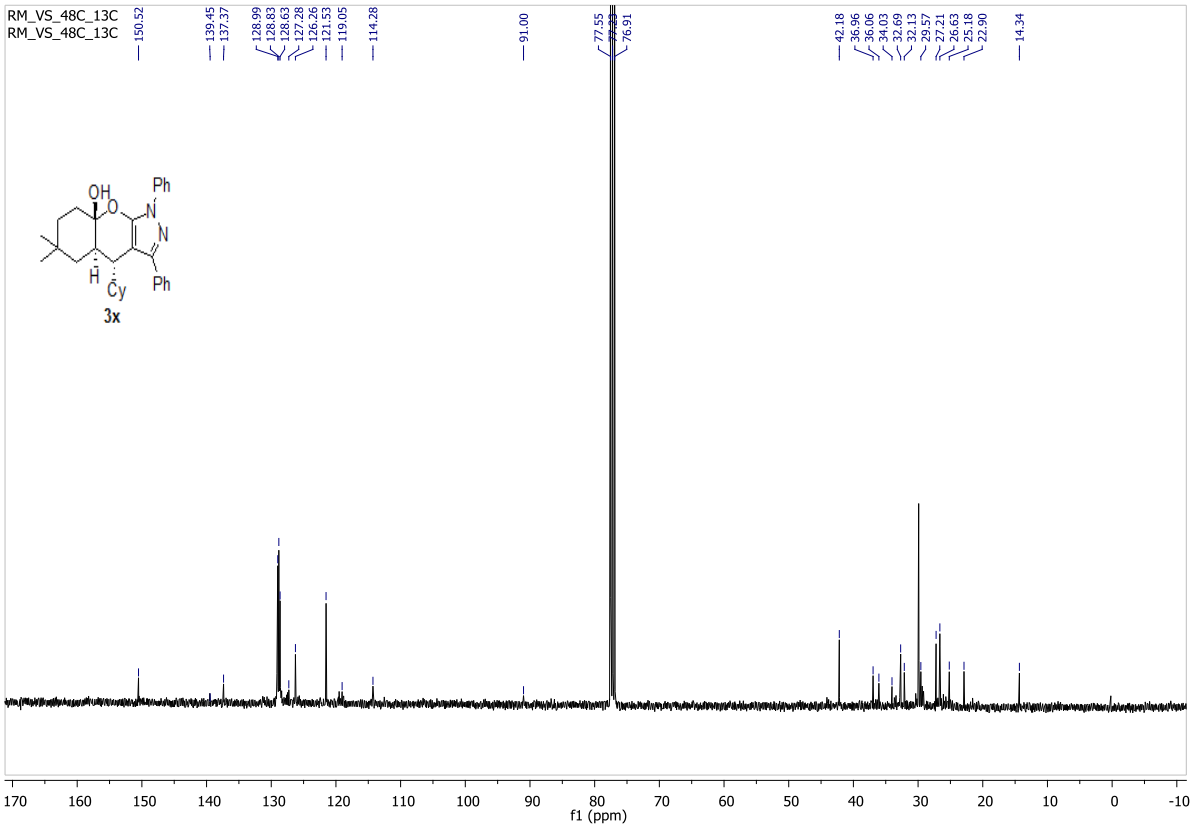

RM-VS-45C-CRY-1H
RM-VS-45C-CRY-1H
7.343
7.330
7.318
7.276
7.260
7.241
7.229
3.614
3.596
2.833
2.241
1.958
1.935
1.786
1.765
1.735
1.680
1.661
1.592
1.350
1.330
1.304
1.283
1.125
1.109
1.087
1.069
-0.002
OH
O
N
N
Me
H
Ph
3v
3.92
2.30
2.73
1.10
0.93
3.02
0.94
2.62
2.76
3.17
1.40
1.11
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

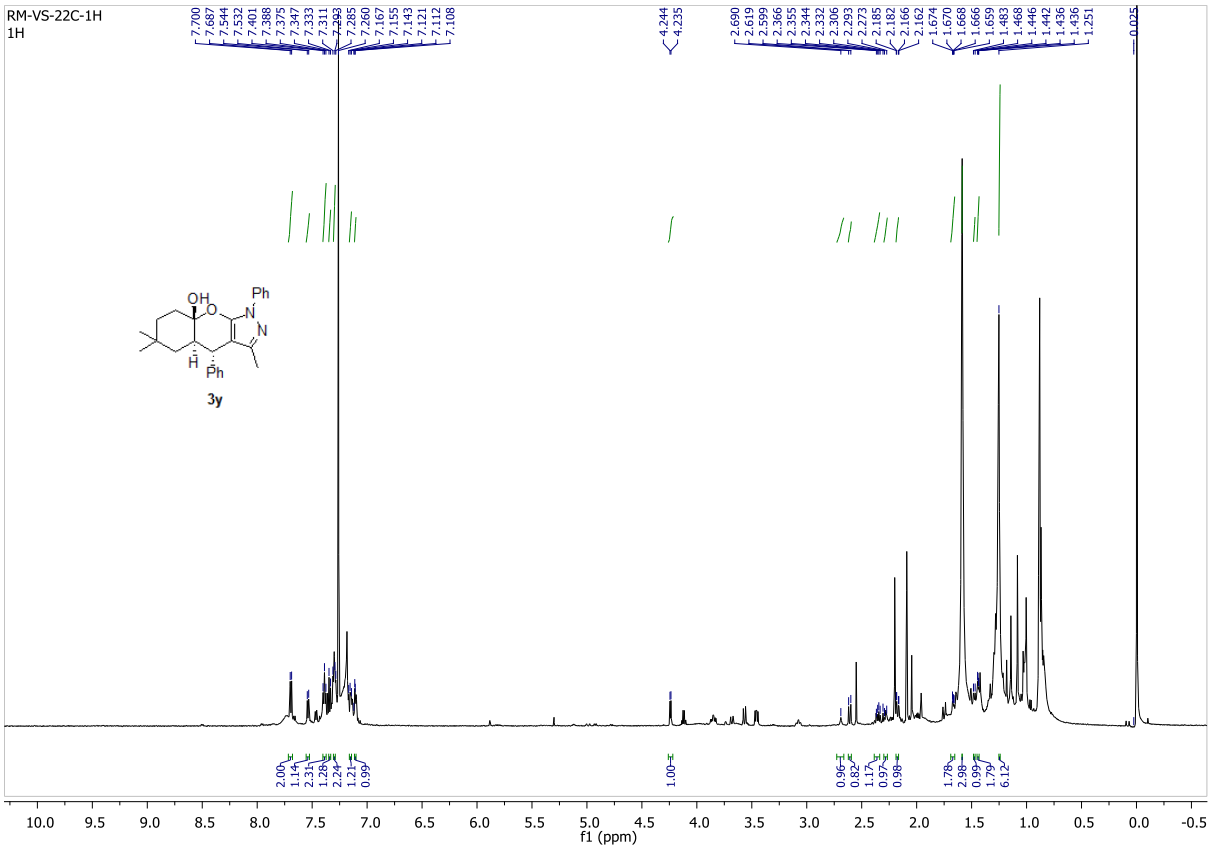

RM_VS_45C_13C_02
RM_VS_45C_13C_02
142.28
135.62
134.84
132.71
130.87
128.58
127.84
126.86
126.44
124.02
117.18
116.96
77.55
77.23
76.91
49.63
42.11
40.61
29.86
25.56
25.12
18.12
13.16
OH
O
N
N
Me
H
Ph
3v
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

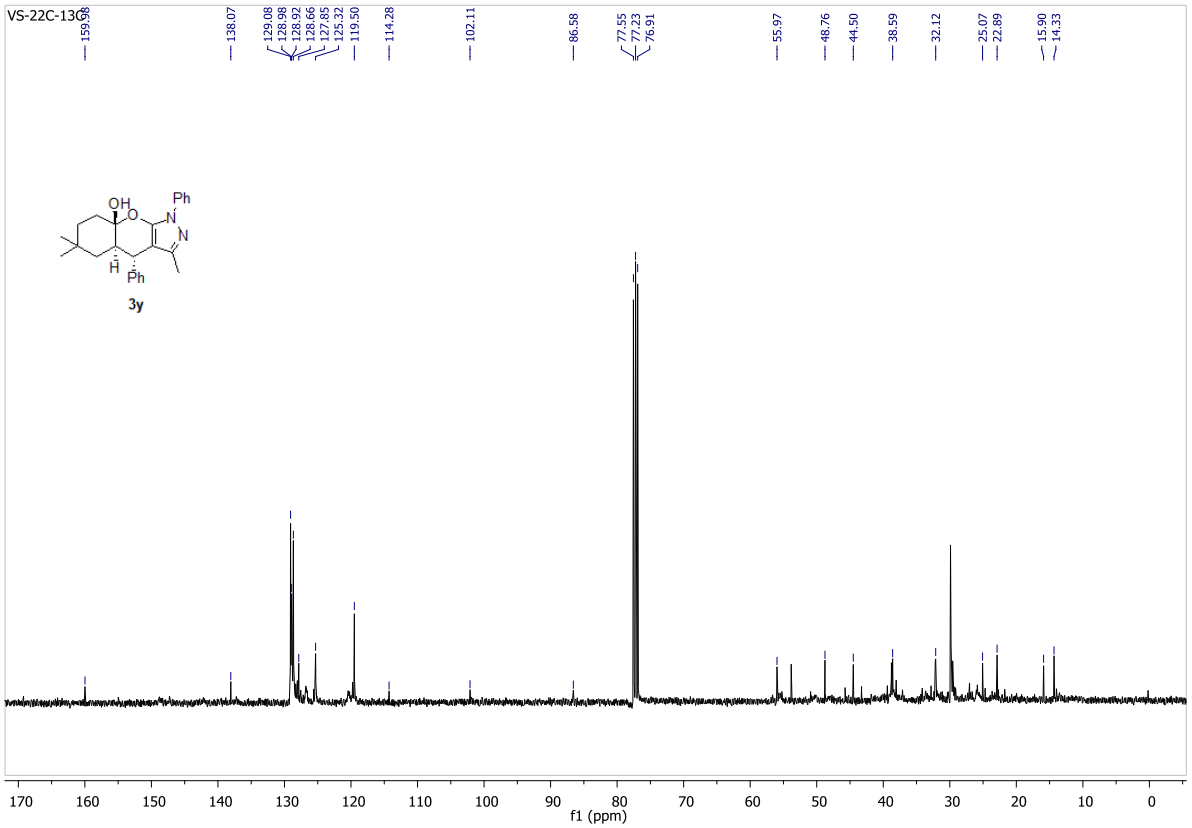

RM-VS-40C-CRY-1H
RM-VS-40C-CRY-1H
3w
f1 (ppm)

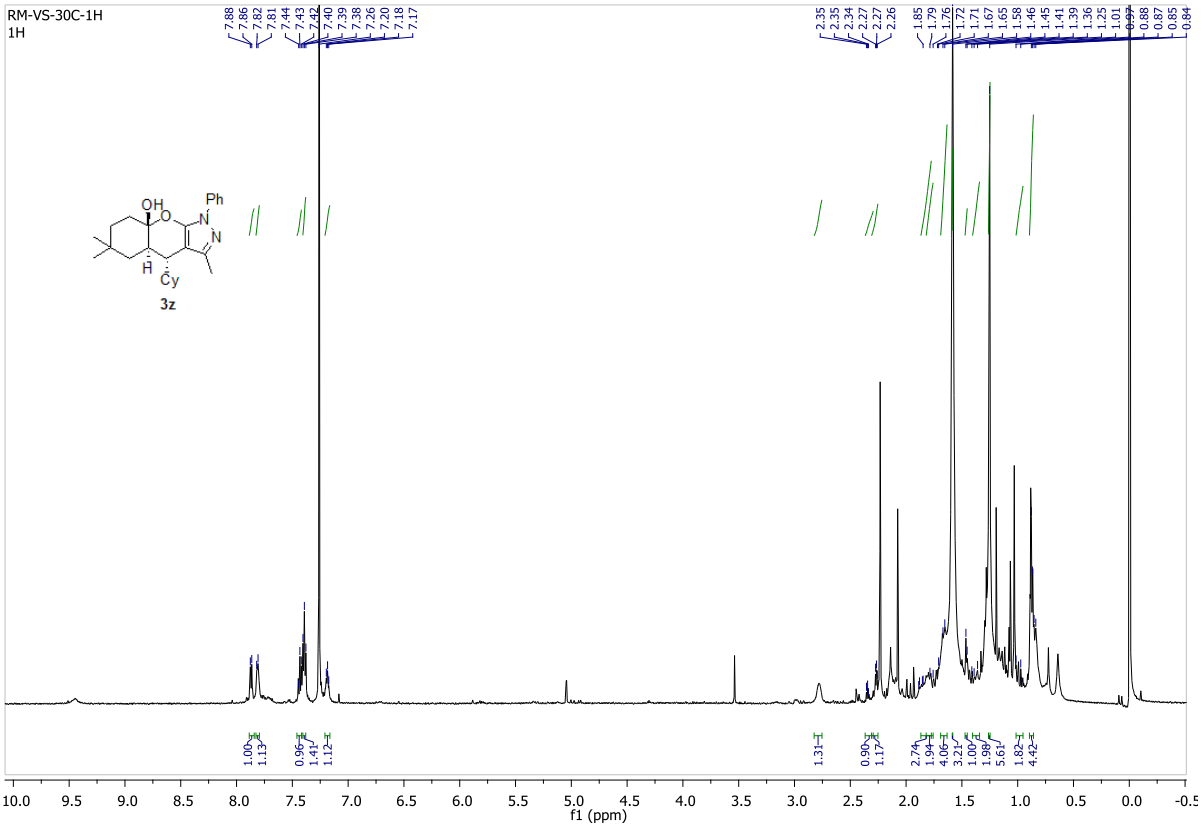

RM_VS_48C_13C
RM_VS_48C_13C
3x
f1 (ppm)

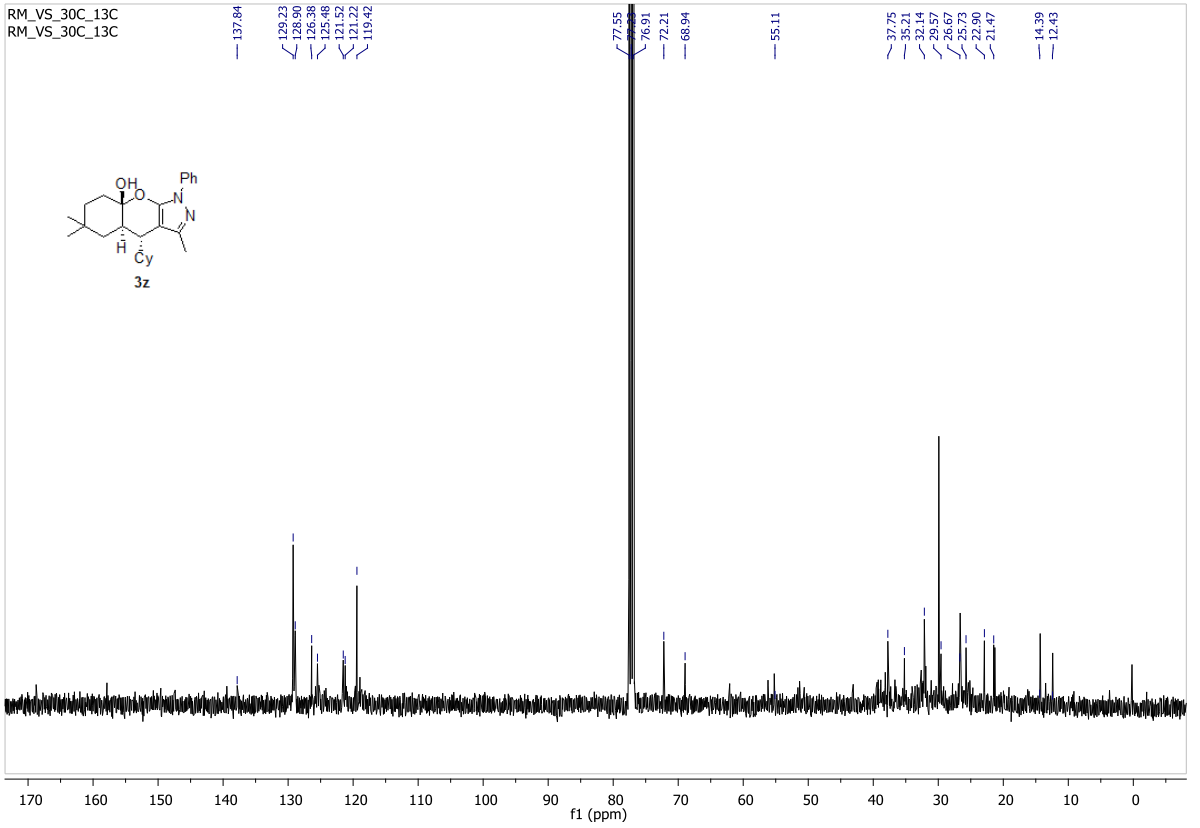

RM-VS-48C-CNY-1H
RM-VS-48C-CNY-1H
7.765
7.753
7.424
7.411
7.398
7.293
7.281
7.245
7.232
7.220
7.191
7.180
7.158
7.145
7.133
3.732
3.723
3.708
3.522
3.503
2.583
2.564
2.540
2.050
2.042
2.029
2.015
1.670
1.656
1.627
1.584
1.063
1.037
0.889
0.878
0.866
0.836
0.828
0.813
0.671
0.660
0.603
OH
O
N
N
Ph
H
Cy
Ph
3x
2.00
2.91
1.24
1.26
0.72
2.29
0.71
0.80
0.92
0.62
4.77
5.27
1.67
2.15
1.73
2.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

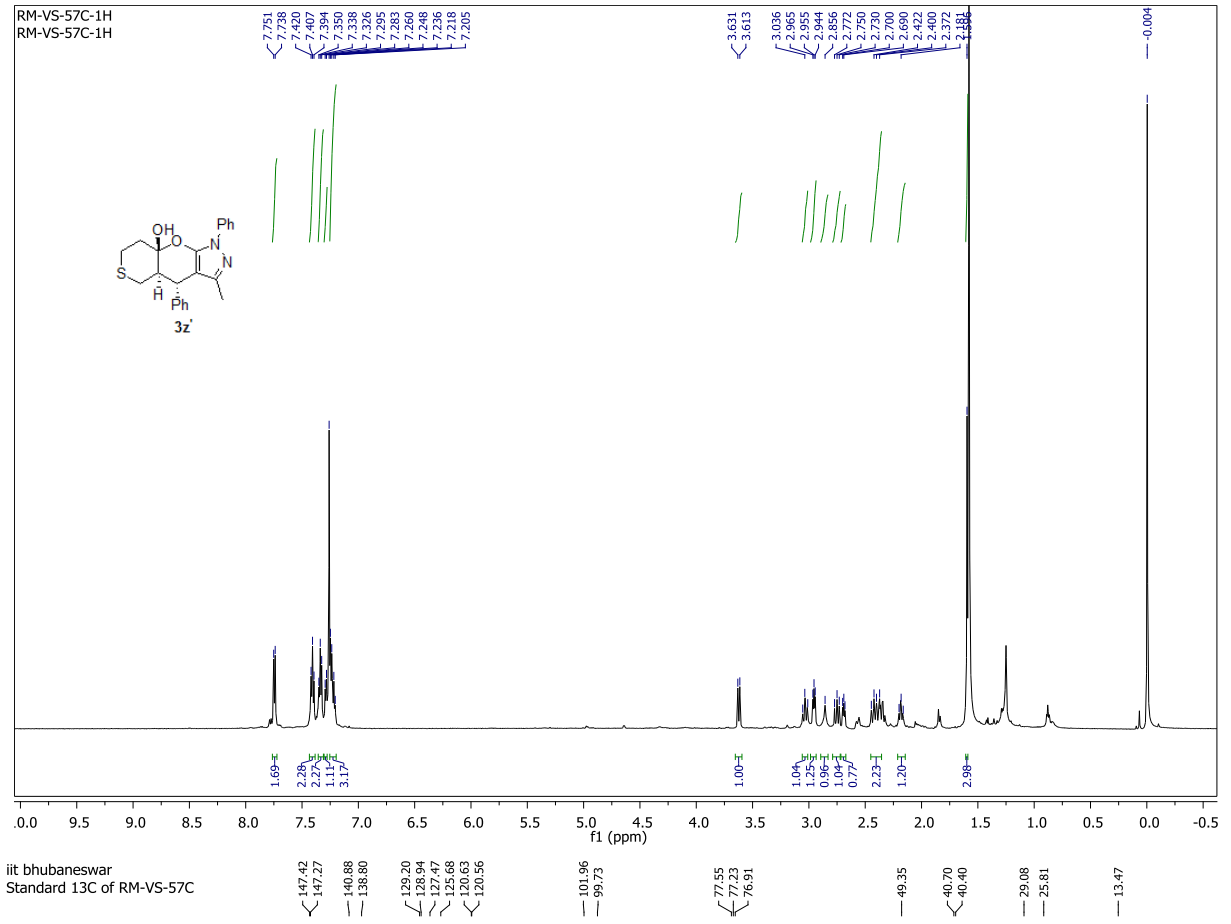

RM_VS_48C_13C
RM_VS_48C_13C
150.52
139.45
137.37
128.99
128.83
128.63
127.28
126.26
121.53
119.05
114.28
91.00
77.55
77.23
76.91
42.18
36.96
36.06
34.03
32.69
32.13
29.57
27.21
26.63
25.18
22.90
14.34
OH
O
N
N
Ph
H
Cy
Ph
3x
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

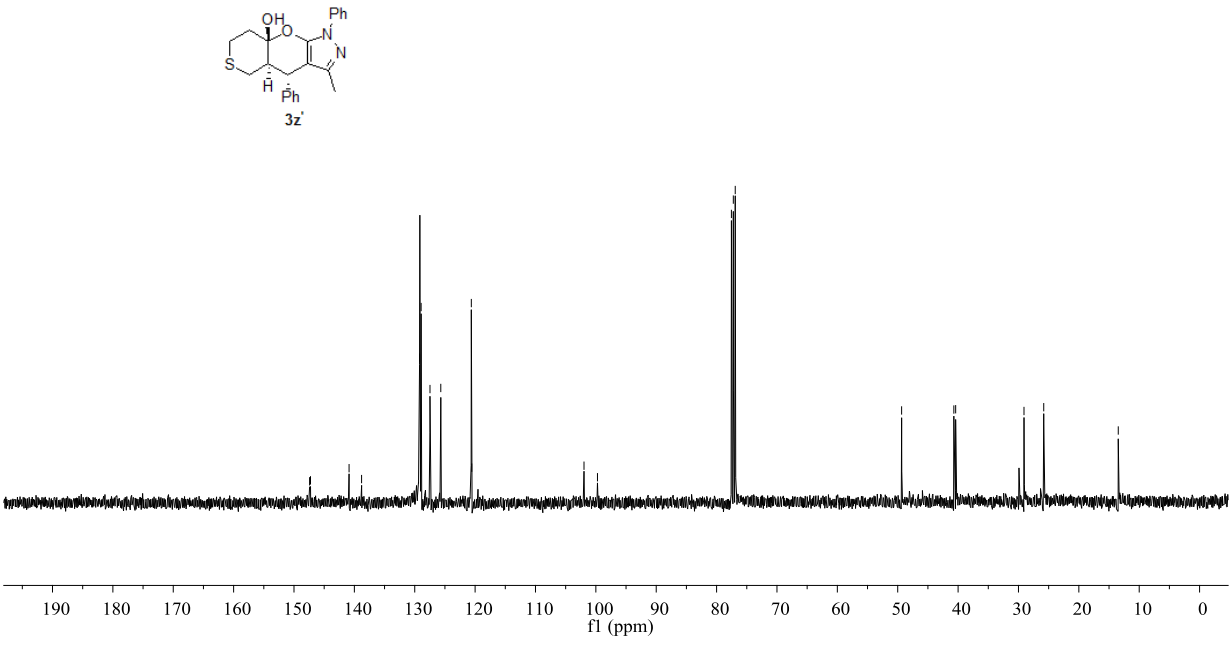

RM-VS-22C-1H
1H
3y
f1 (ppm)

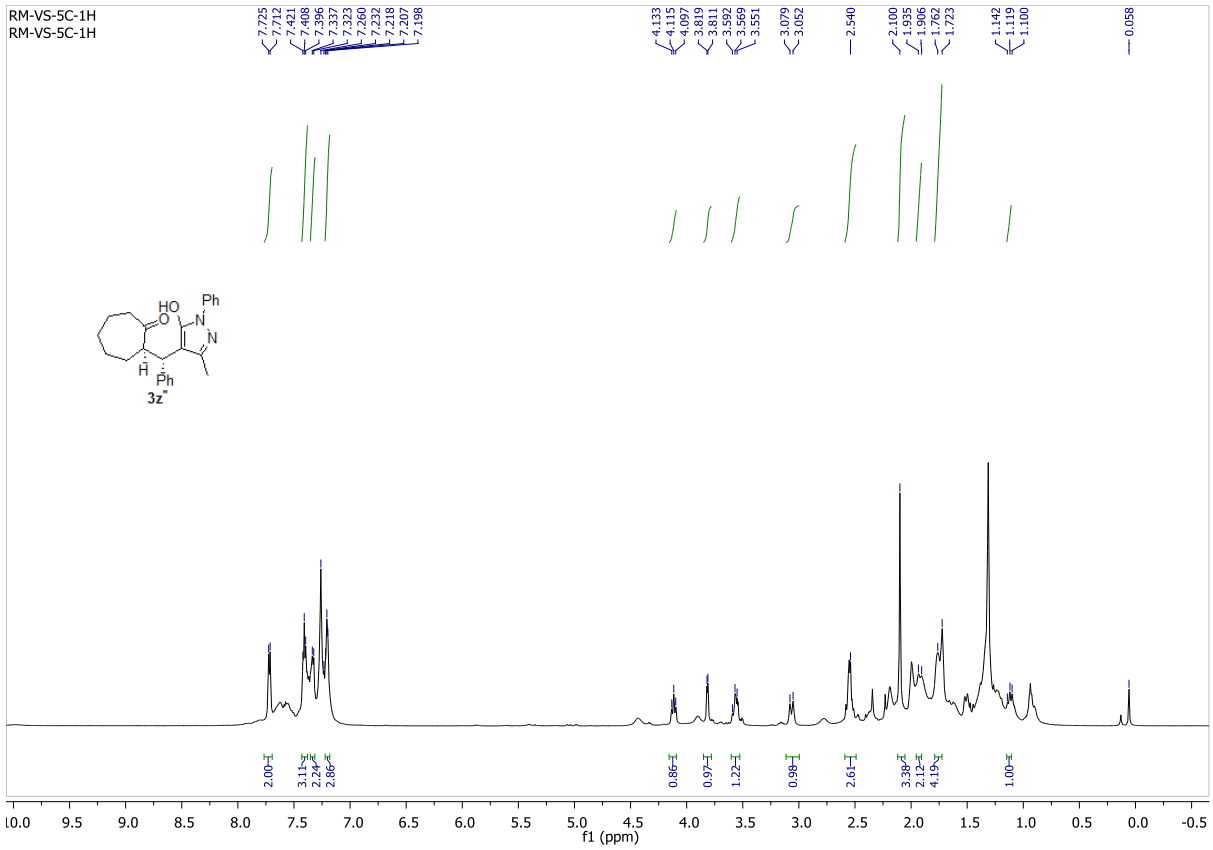

VS-22C-13C
3y
f1 (ppm)

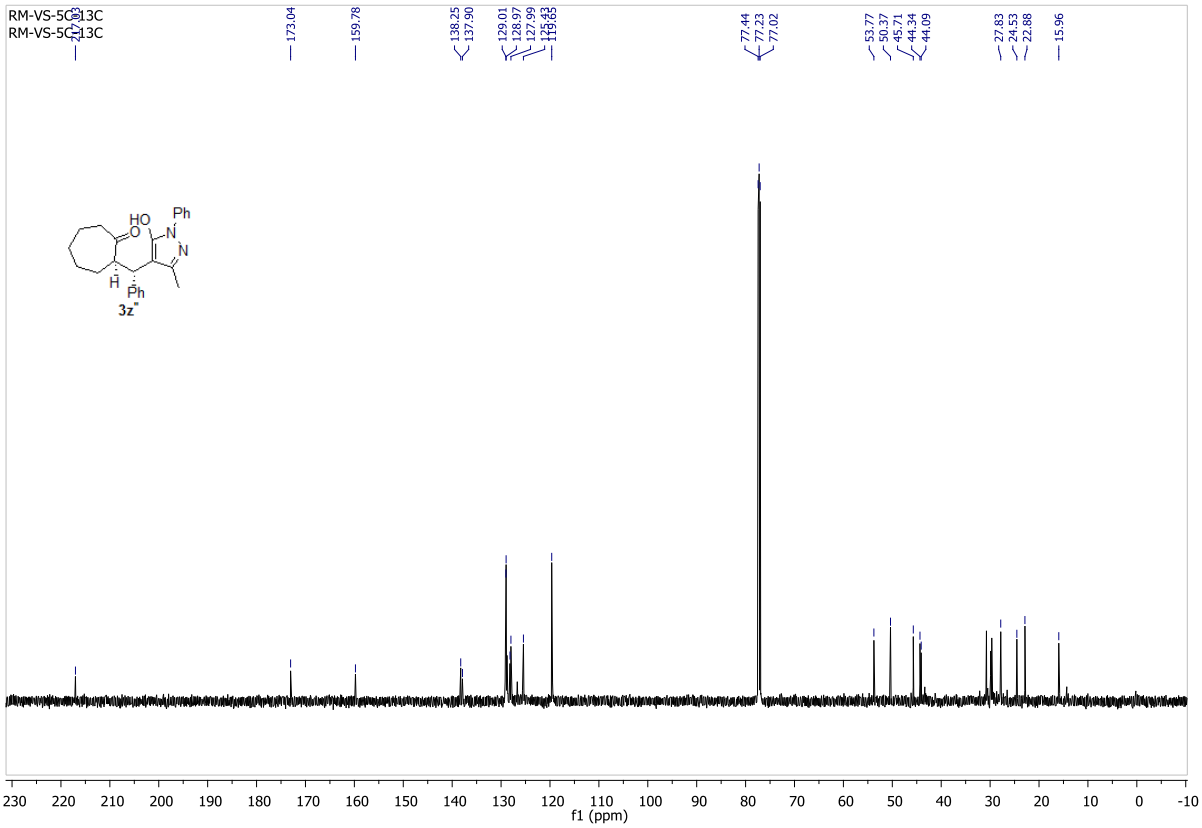

RM-VS-30C-1H
1H
f1 (ppm)

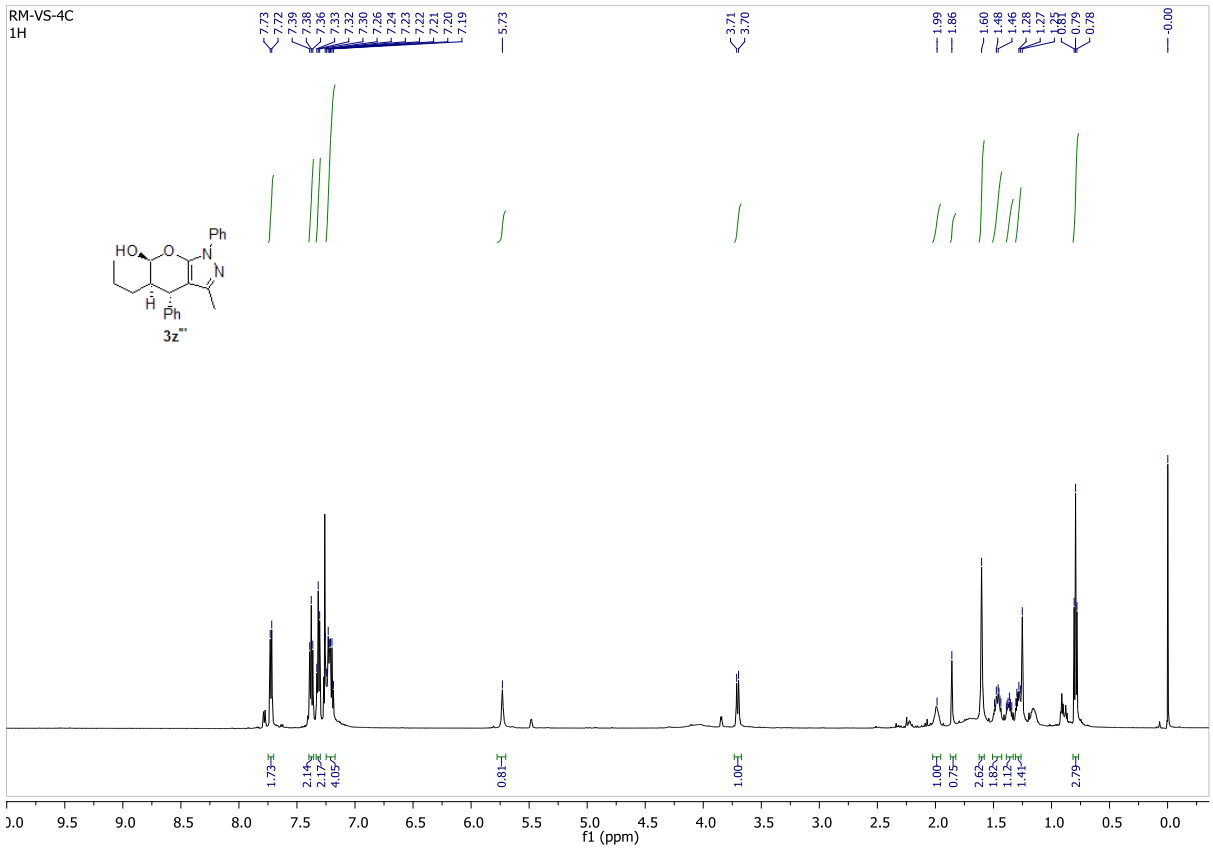

RM_VS_30C_13C
RM_VS_30C_13C
f1 (ppm)

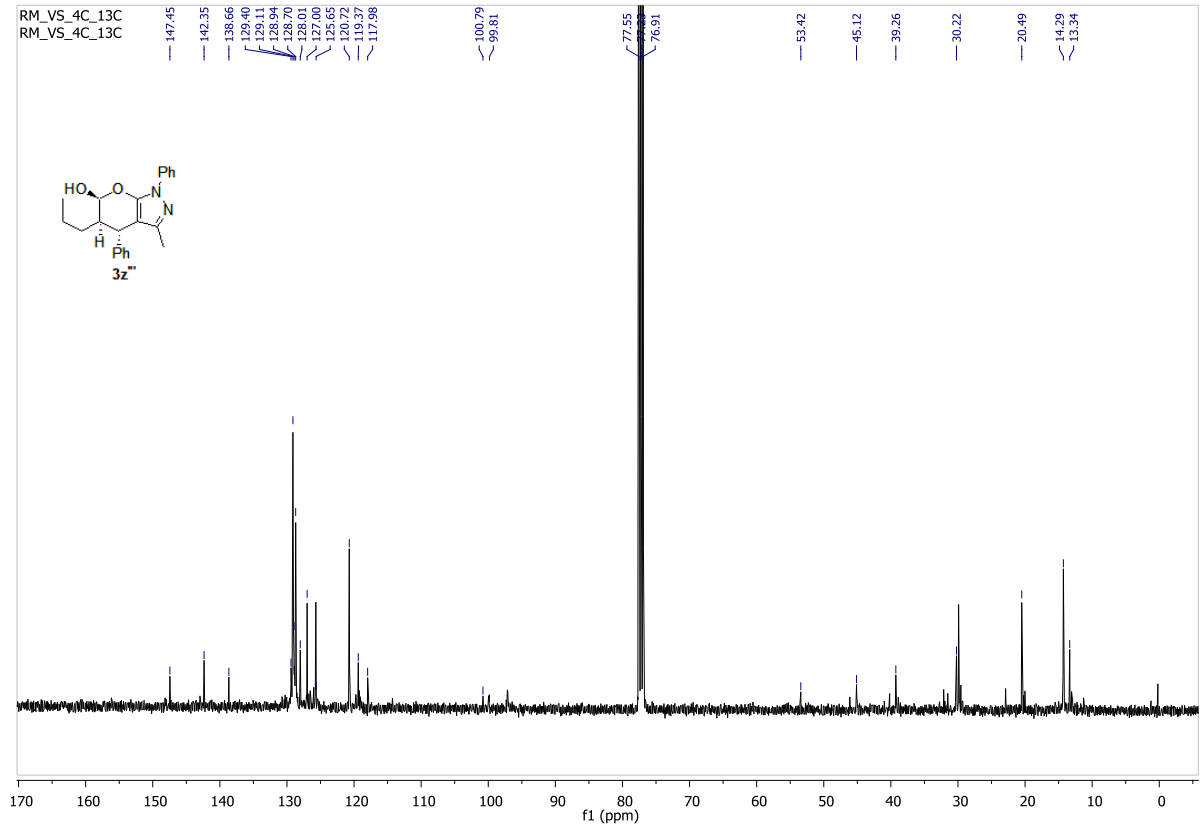

RM-VS-57C-1H
RM-VS-57C-1H
f1 (ppm)

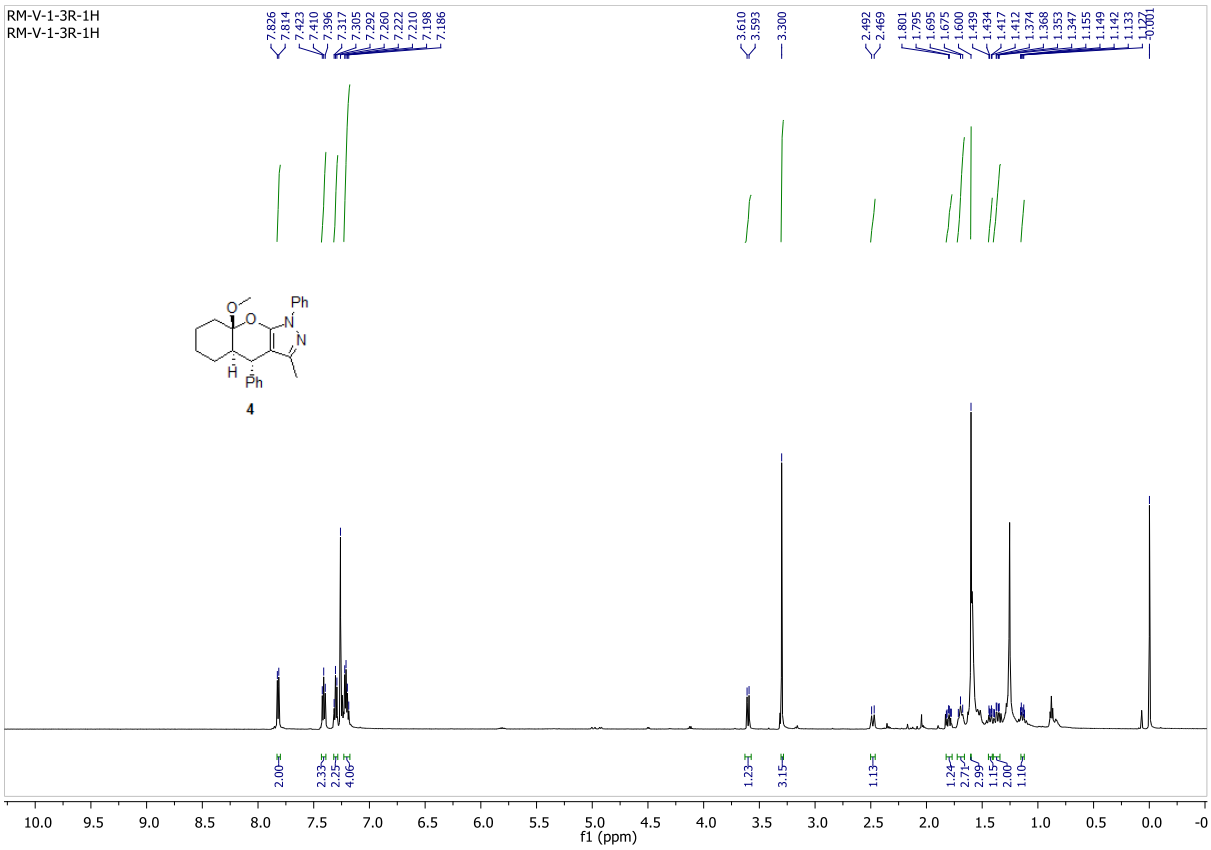

iit bhubaneswar
Standard 13C of RM-VS-57C
f1 (ppm)

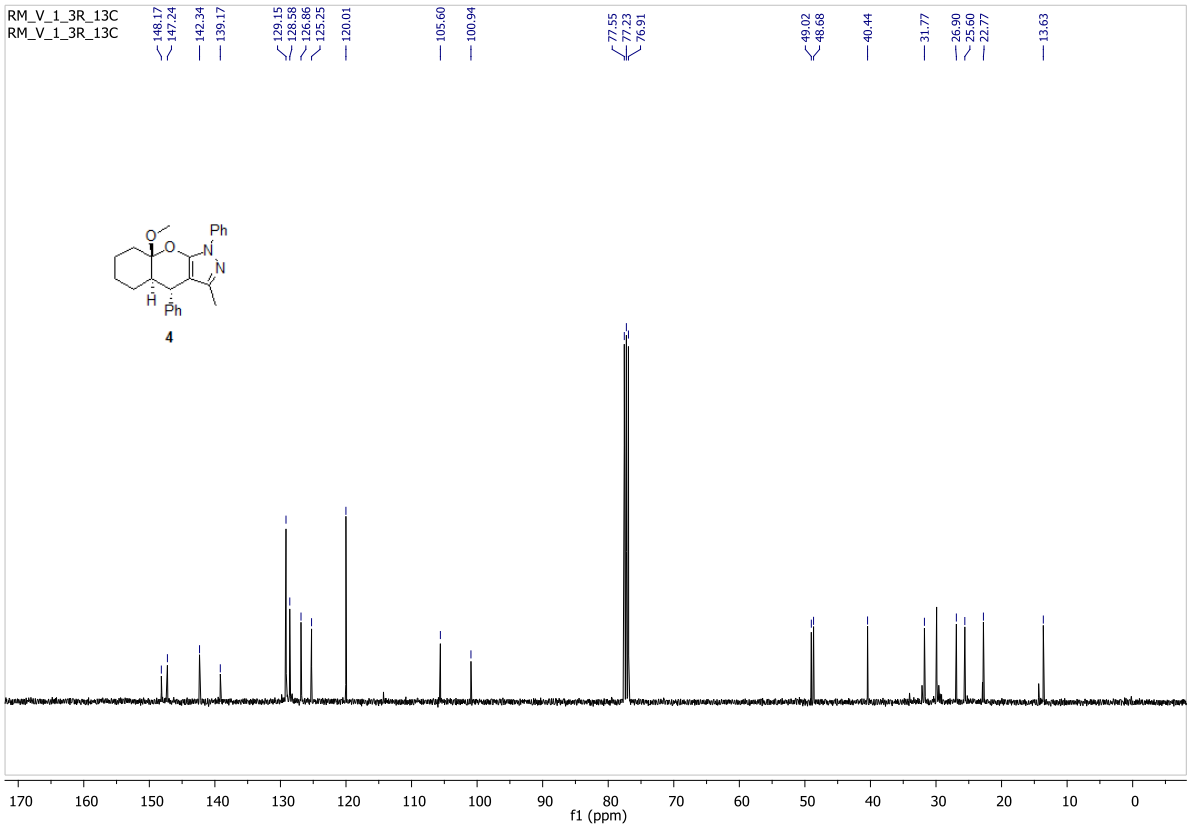

RM-VS-5C-1H
RM-VS-5C-1H
HO
Ph
N
N
H
Ph
3z"
f1 (ppm)

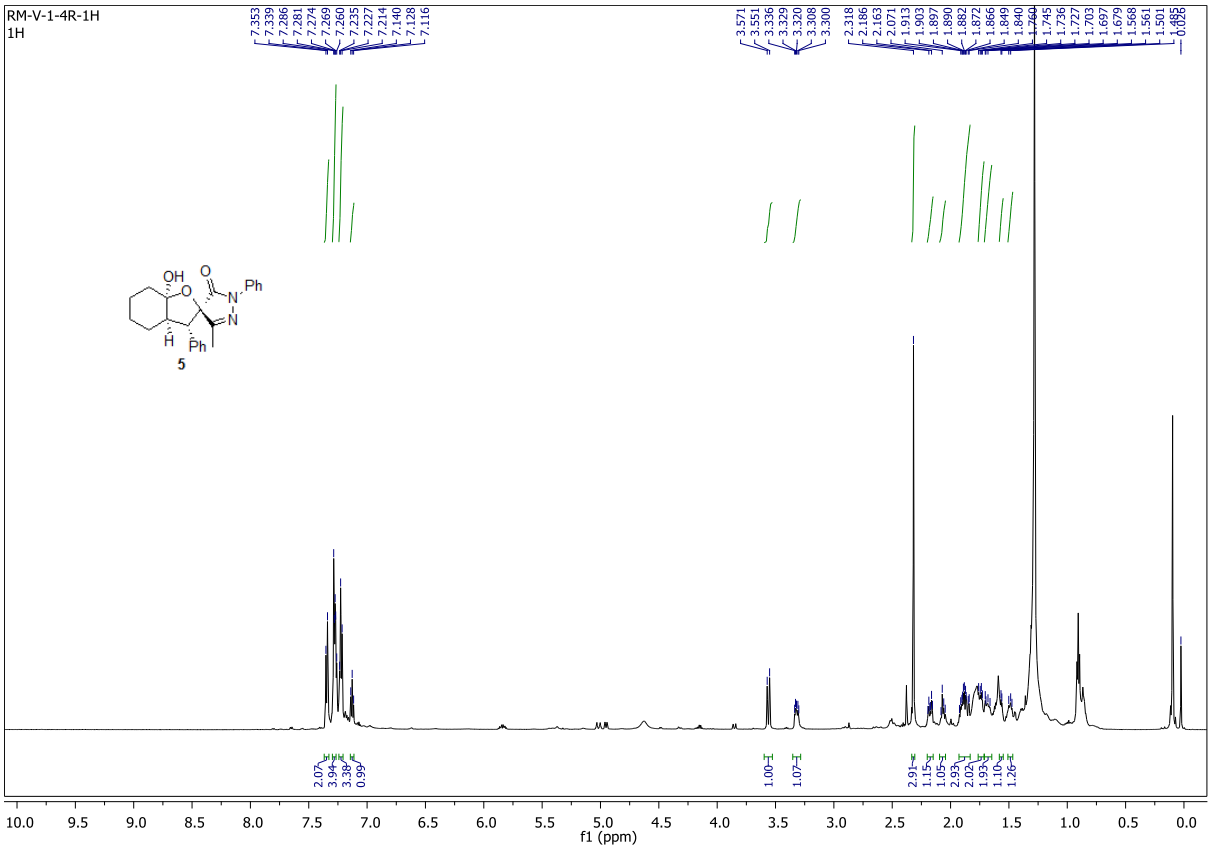

RM-VS-5C-13C
RM-VS-5C-13C
HO
Ph
N
N
H
Ph
3z"
f1 (ppm)

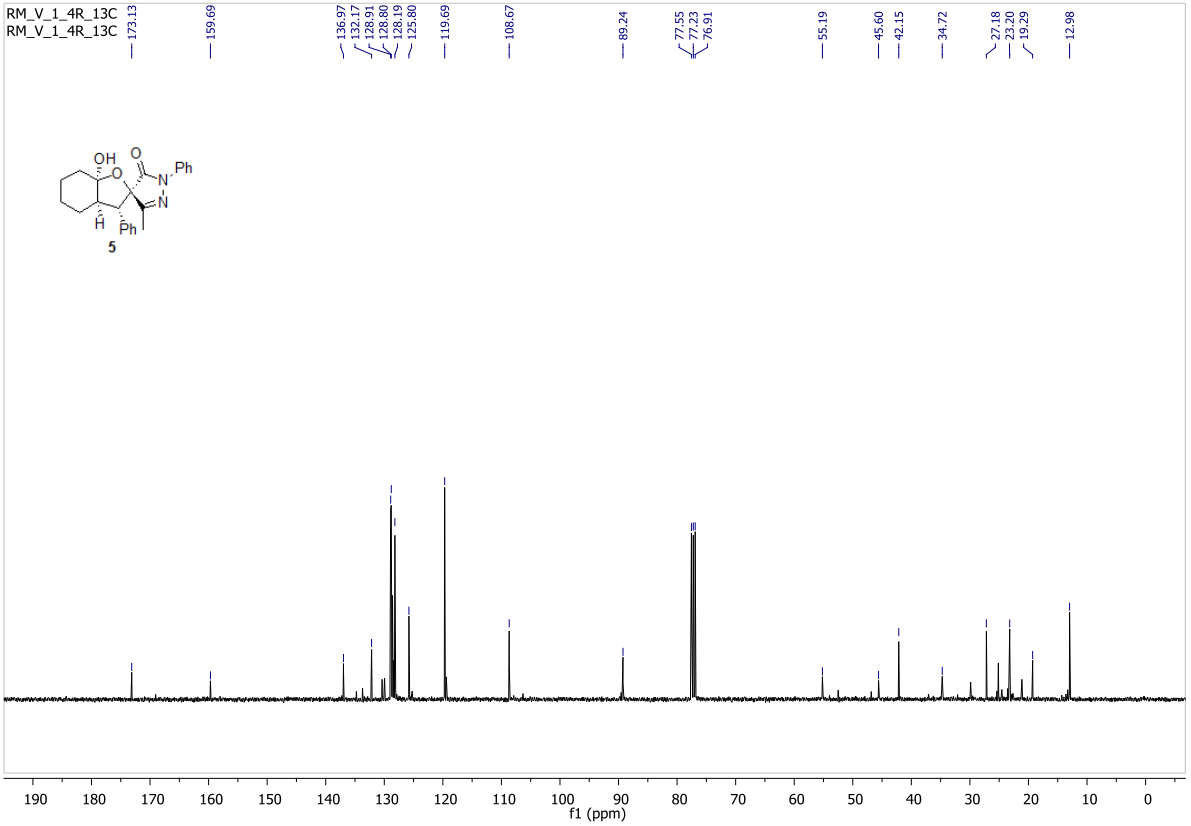

RM-VS-4C
1H
7.73
7.72
7.39
7.38
7.36
7.33
7.32
7.30
7.26
7.24
7.23
7.22
7.21
7.20
7.19
5.73
3.71
3.70
1.99
1.86
1.60
1.48
1.46
1.28
1.27
0.81
0.79
0.78
-0.00
HO
O
N
N
Ph
H
Ph
3z**
1.73
2.14
2.17
4.05
0.81
1.00
1.00
0.75
2.62
1.82
1.12
1.41
2.79
f1 (ppm)


RM_VS_4C_13C
RM_VS_4C_13C
147.45
142.35
138.66
129.40
129.11
128.94
128.70
128.01
127.00
125.65
120.72
119.37
117.98
100.79
99.81
77.55
77.23
76.91
53.42
45.12
39.26
30.22
20.49
14.29
13.34
HO
O
N
N
Ph
H
Ph
3z**
f1 (ppm)

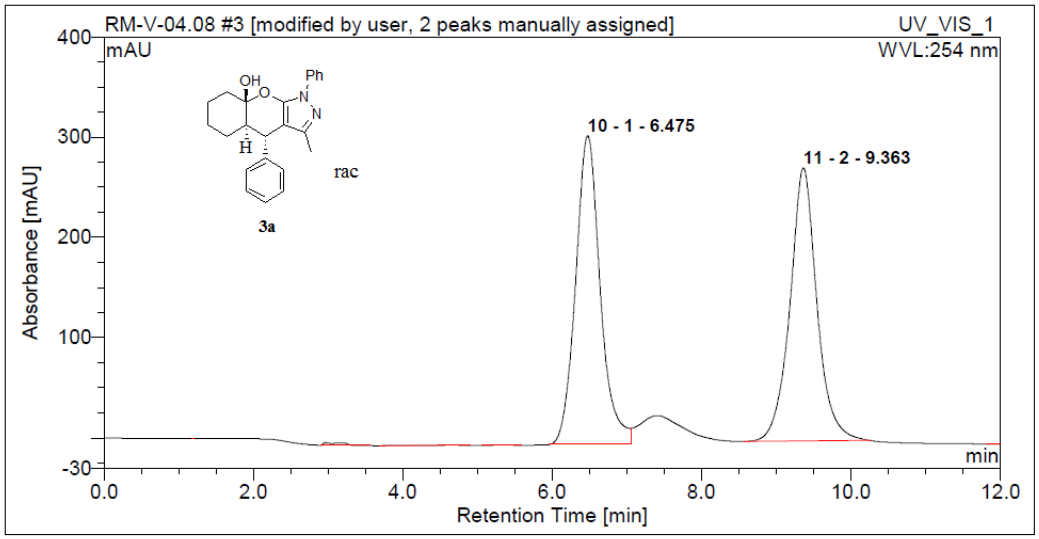

RM-V-1-3R-1H
RM-V-1-3R-1H
7.826
7.814
7.423
7.410
7.396
7.317
7.305
7.292
7.260
7.222
7.210
7.198
7.186
3.610
3.593
3.300
2.492
2.469
1.801
1.795
1.695
1.675
1.600
1.439
1.434
1.417
1.412
1.374
1.368
1.353
1.347
1.155
1.149
1.142
1.133
1.127
0.001
Ph
O
O
N
N
H
Ph
4
2.00
2.33
2.25
4.06
1.23
3.15
1.13
1.24
2.71
2.99
1.15
2.00
1.10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


RM_V_1_3R_13C
RM_V_1_3R_13C
148.17
147.24
142.34
139.17
129.15
128.58
126.86
125.25
120.01
105.60
100.94
77.55
77.23
76.91
49.02
48.68
40.44
31.77
26.90
25.60
22.77
13.63
Ph
O
O
N
N
H
Ph
4
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

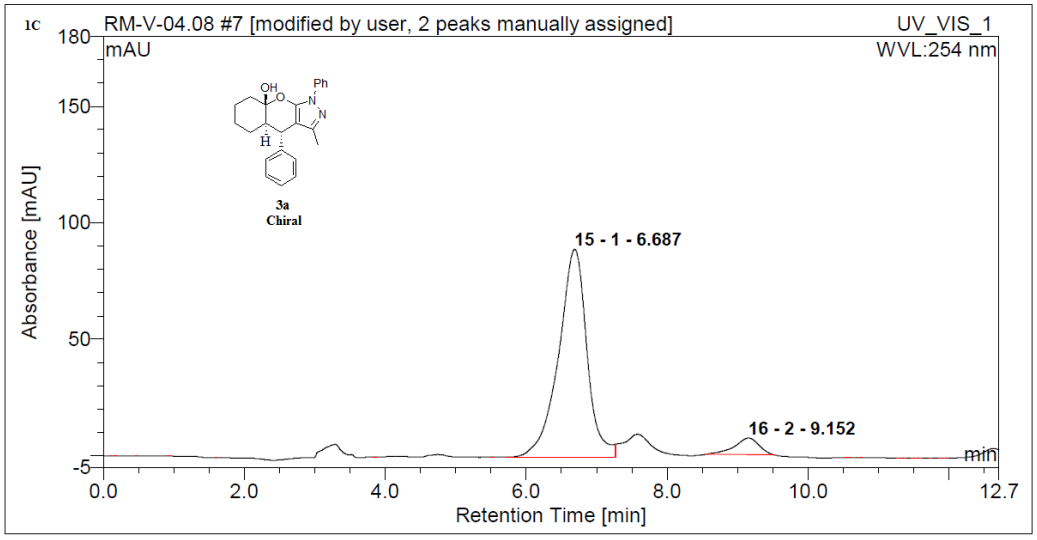

RM-V-1-4R-1H
1H
f1 (ppm)


RM_V_1_4R_13C
RM_V_1_4R_13C
f1 (ppm)

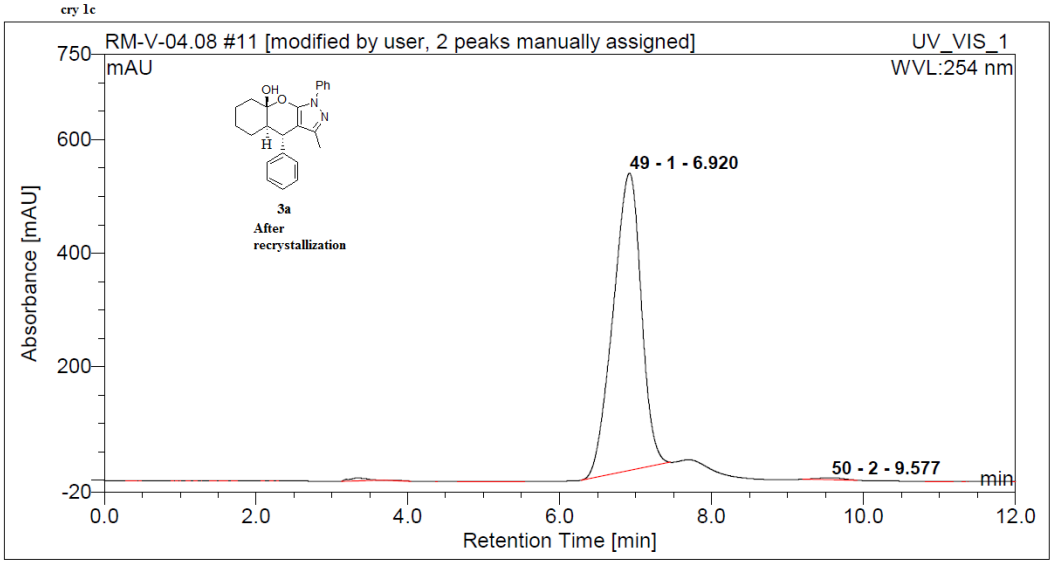
13. **HPLC Spectra**:



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
10	1	6.48	117.5589	50.7342853	308.0702	n.a.
11	2	9.36	114.156	49.2657147	272.572	n.a.

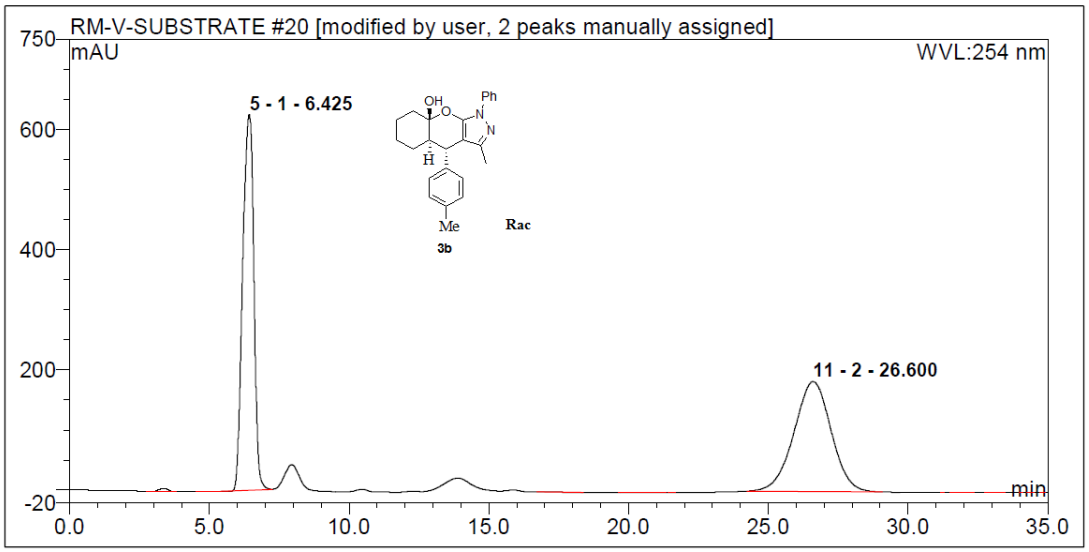


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
15	1	6.69	41.65523	93.51426841	89.29235	n.a.
16	2	9.15	2.889	6.485731593	7.100	n.a.



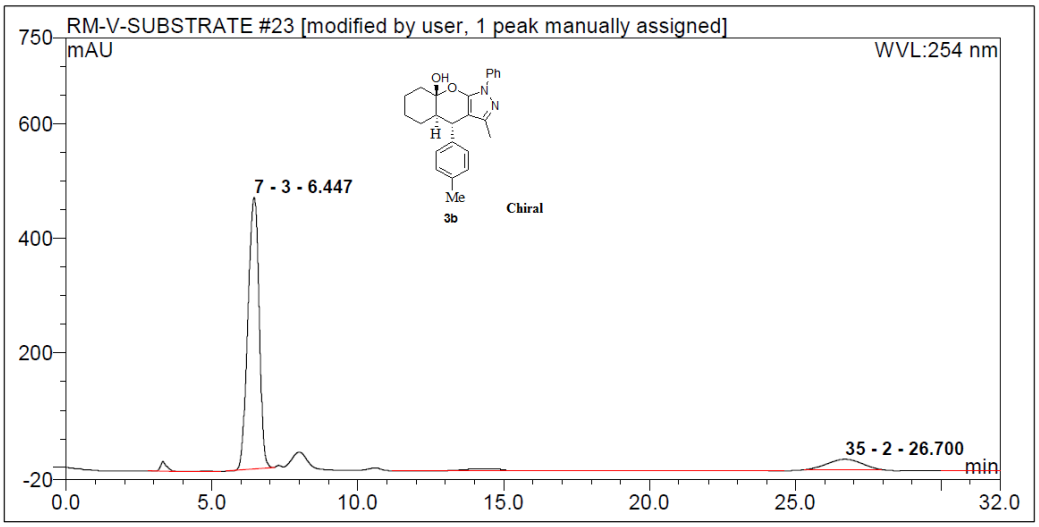
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
49	1	6.92	224.6229	99.40378003	523.4093	n.a.
50	2	9.58	1.347	0.5962199704	3.419	n.a.

10R



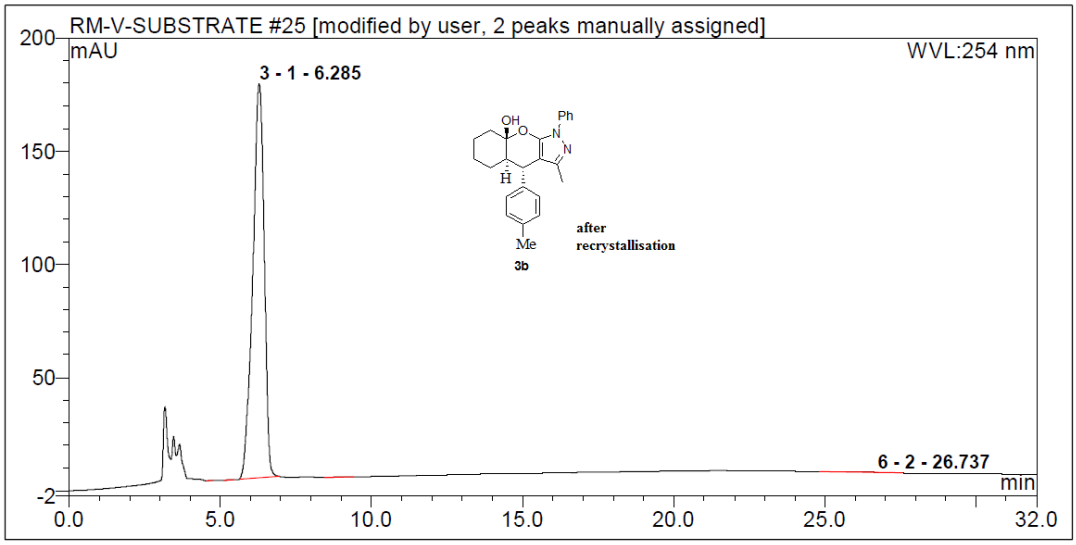
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
5	1	6.43	288.6821	50.59049573	625.3244	n.a.
11	2	26.60	281.943	49.40950427	182.757	n.a.

10C



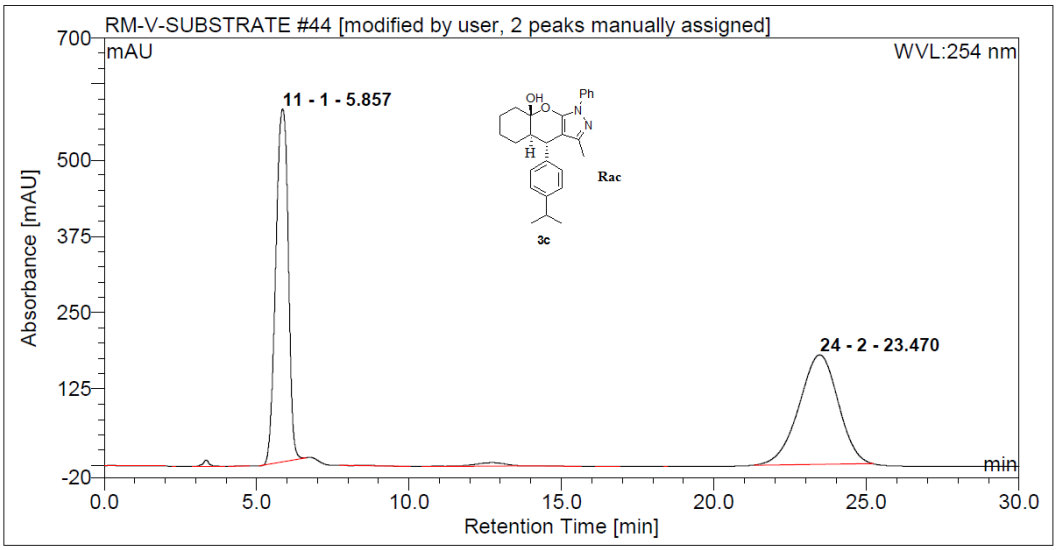
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
7	3	6.45	211.325	89.70364713	473.8624	n.a.
35	2	26.70	24.256	10.29635287	18.079	n.a.

10C-CRY



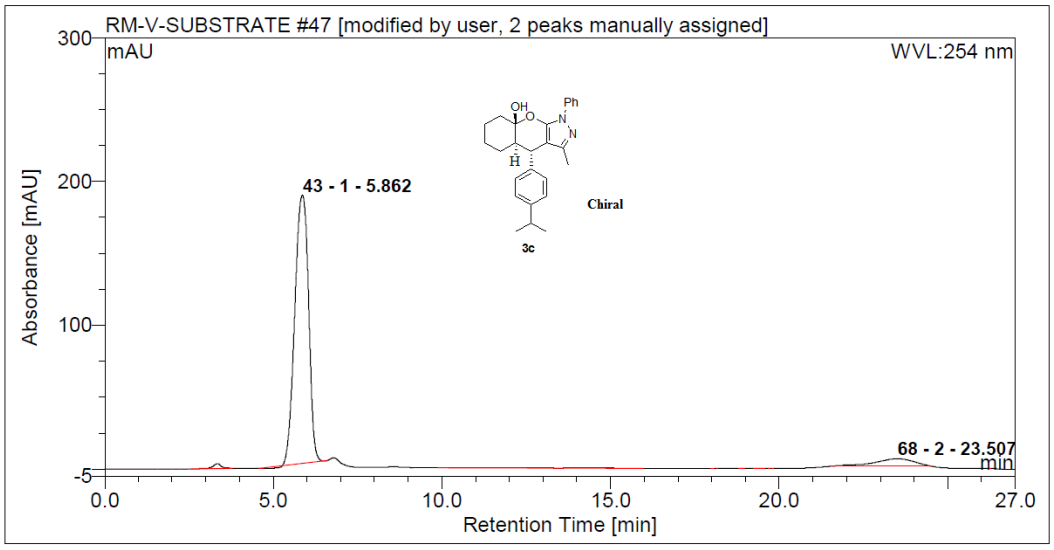
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
3	1	6.29	76.08073	99.93532454	173.9603	n.a.
6	2	26.74	0.049	0.06467546399	0.056	n.a.

12R



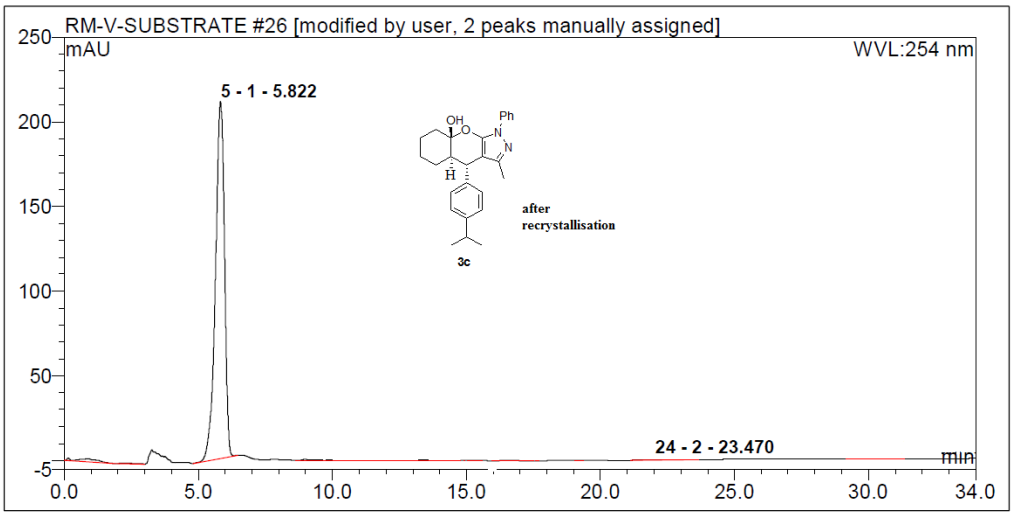
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
11	1	5.86	273.8805	50.61426129	577.7158	n.a.
24	2	23.47	267.233	49.38573871	179.308	n.a.

12C



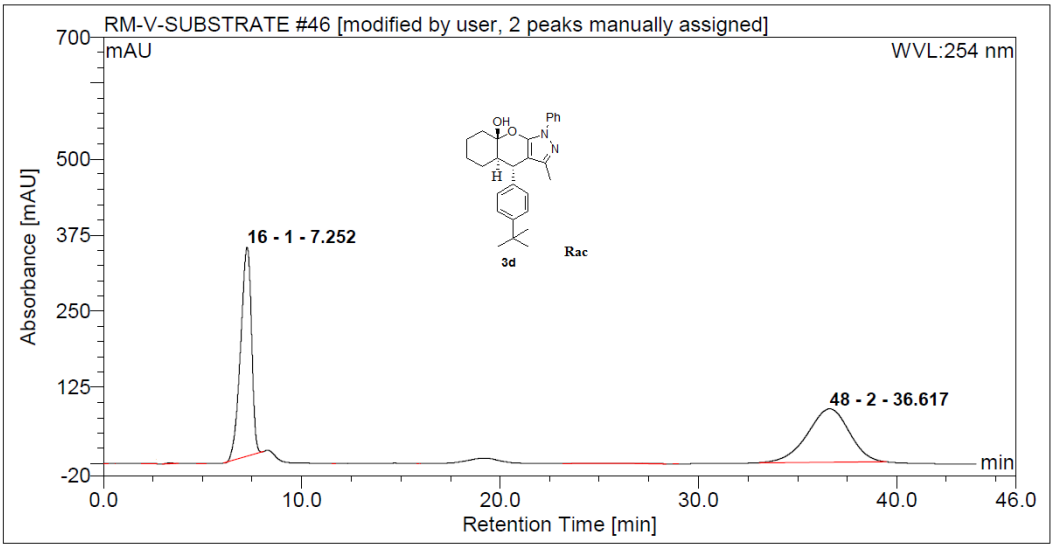
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
43	1	5.86	88.0503	92.30001249	186.7818	n.a.
68	2	23.51	7.345	7.699987506	5.248	n.a.

RM-VS-12C



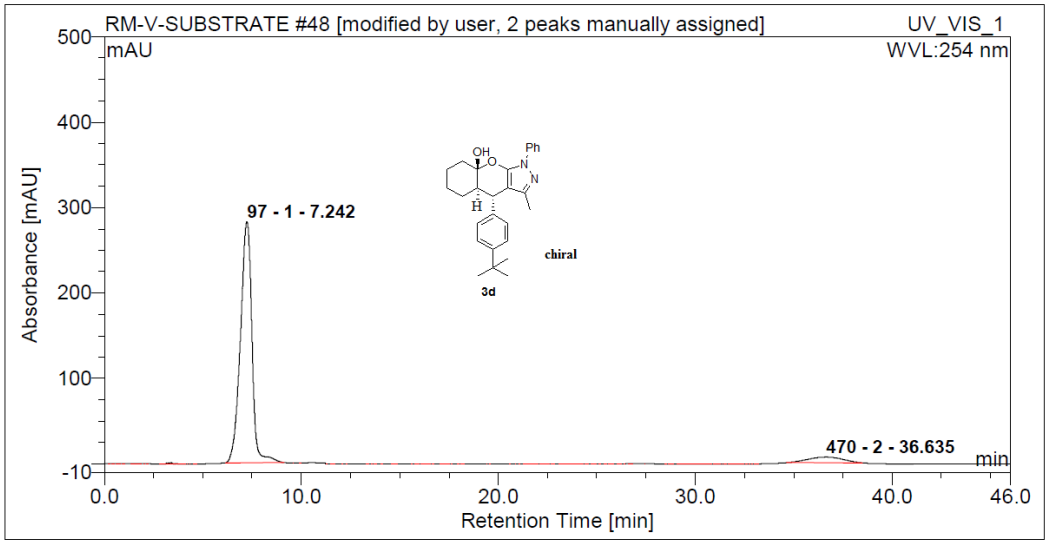
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
5	1	5.82	82.60759	99.98062793	210.9517	n.a.
40	2	23.47	0.016	0.01937206862	0.021	n.a.

13R



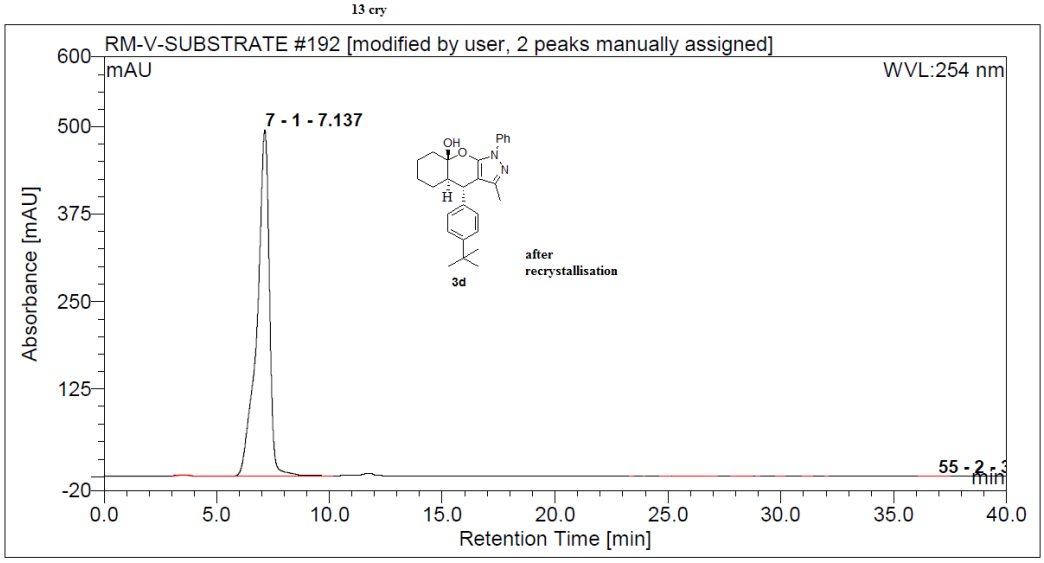
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
16	1	7.25	219.4135	50.62147807	343.2796	n.a.
48	2	36.62	214.026	49.37851683	87.564	n.a.

13 c



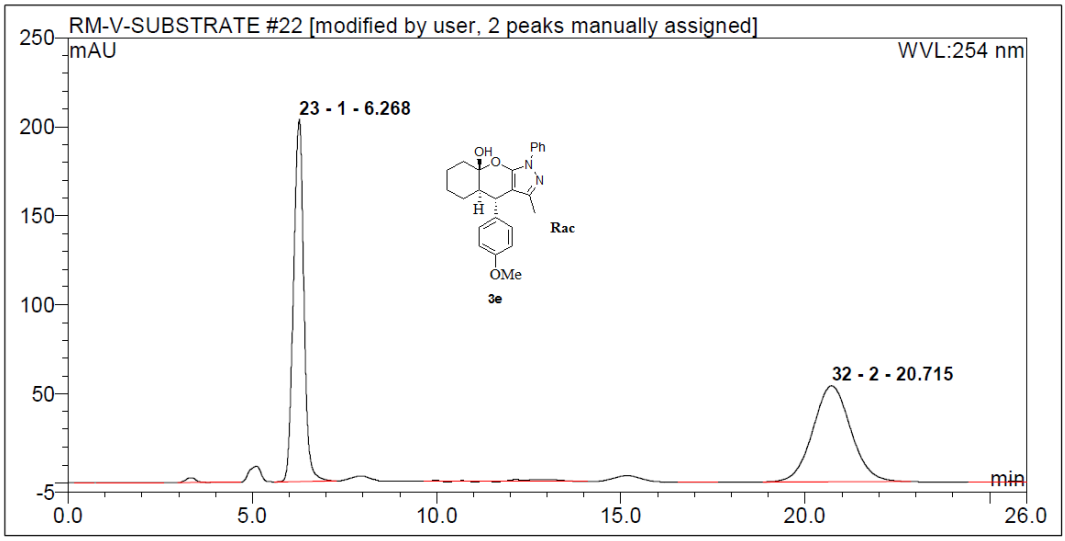
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
97	1	7.24	190.7981	93.33540174	282.2116	n.a.
470	2	36.64	13.624	6.664598258	6.666	n.a.

13 cry



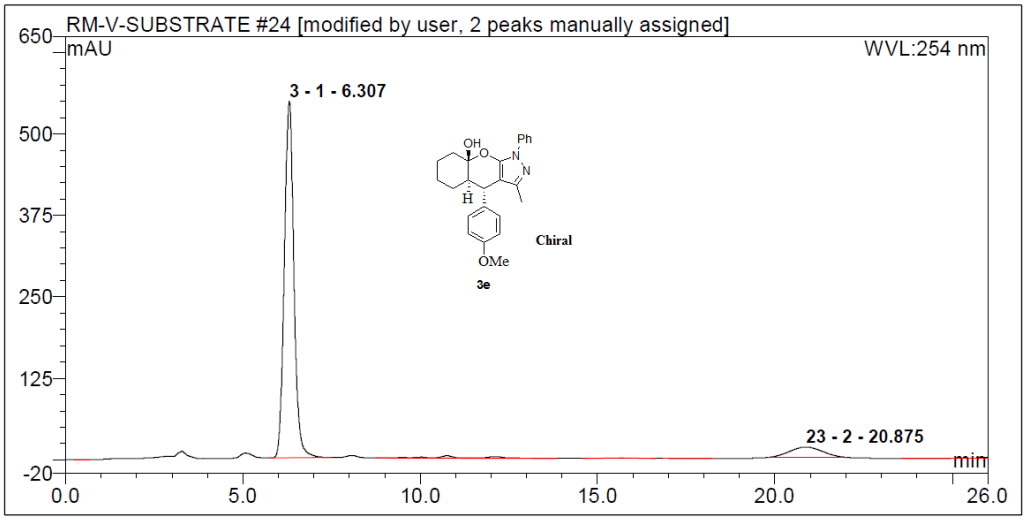
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
7	1	7.14	315.4059	99.99750754	494.9328	n.a.
55	2	36.96	0.008	0.002492459156	0.013	n.a.

11R



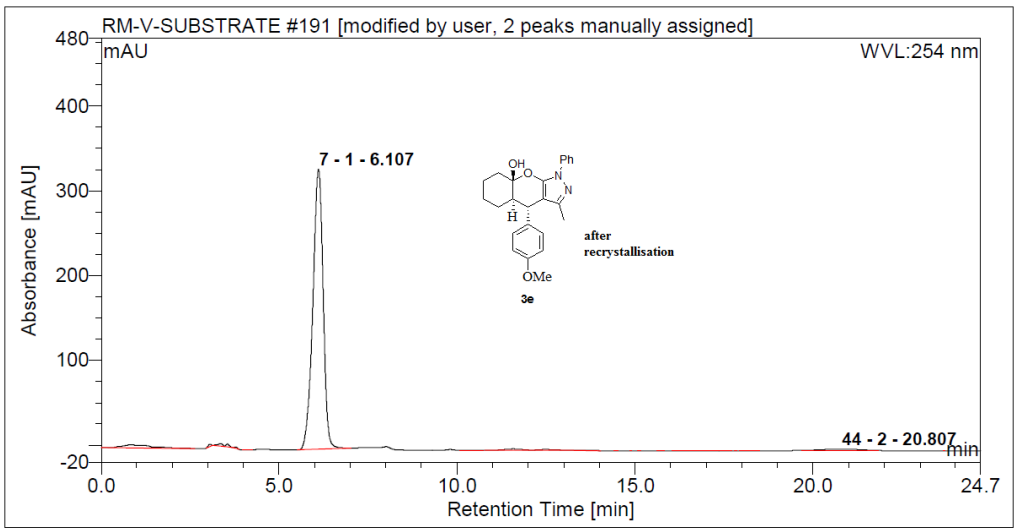
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
23	1	6.27	65.97423	50.63191045	203.5002	n.a.
32	2	20.72	64.327	49.36808955	53.914	n.a.

11C



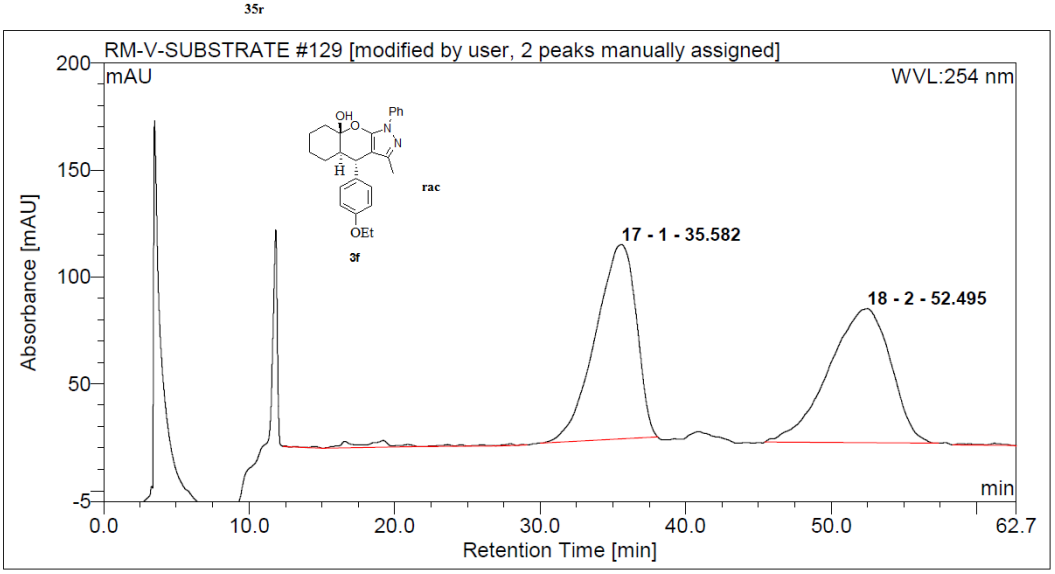
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
3	1	6.31	171.7366	91.01002782	548.0407	n.a.
23	2	20.88	16.964	8.989972184	15.819	n.a.

11C-CRY

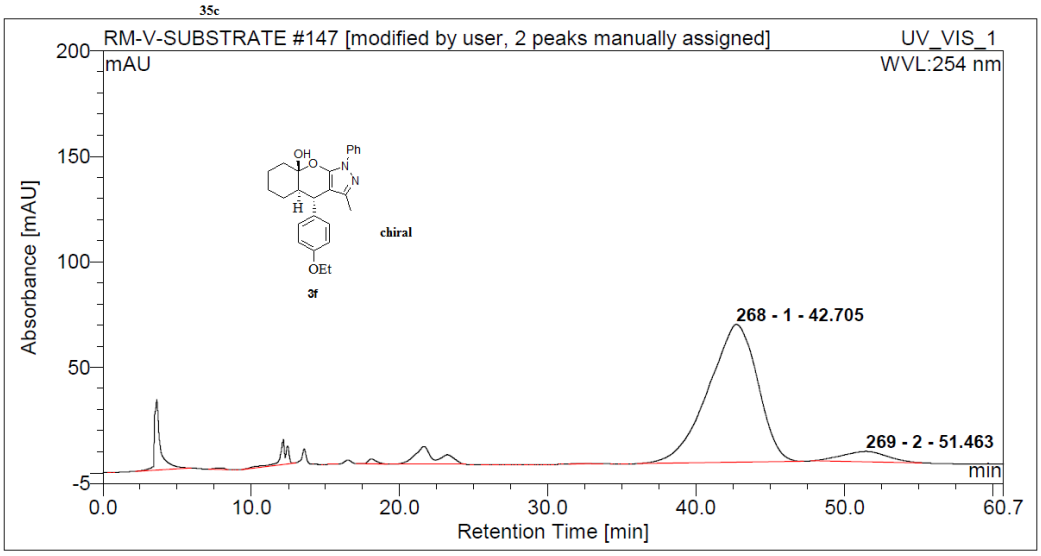


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
7	1	6.11	111.0432	98.19594105	330.4575	n.a.
44	2	20.81	2.040	1.804058949	1.803	n.a.

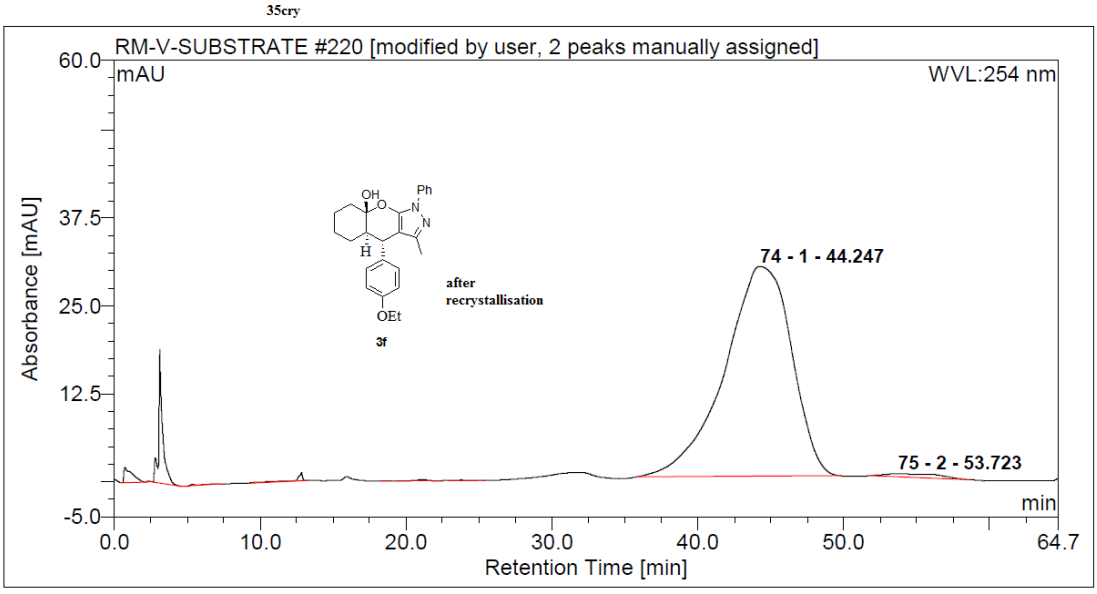
35r



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
17	1	35.58	295.8556	48.95531857	90.80867	n.a.
18	2	52.50	308.482	51.04468143	62.708	n.a.

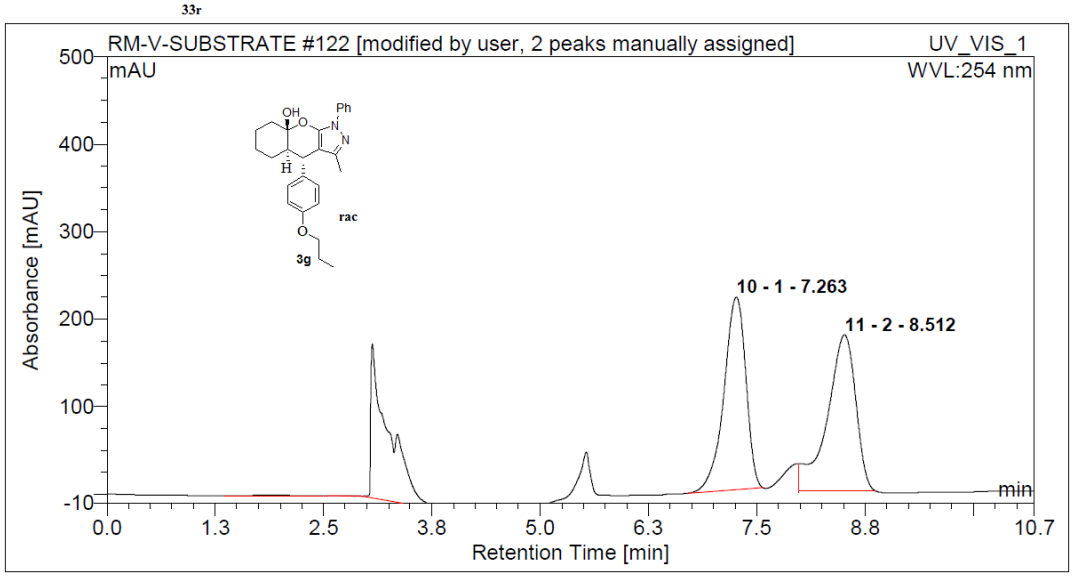


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
268	1	42.71	252.5974	93.6448356	65.32547	n.a.
269	2	51.46	17.142	6.355164403	4.903	n.a.



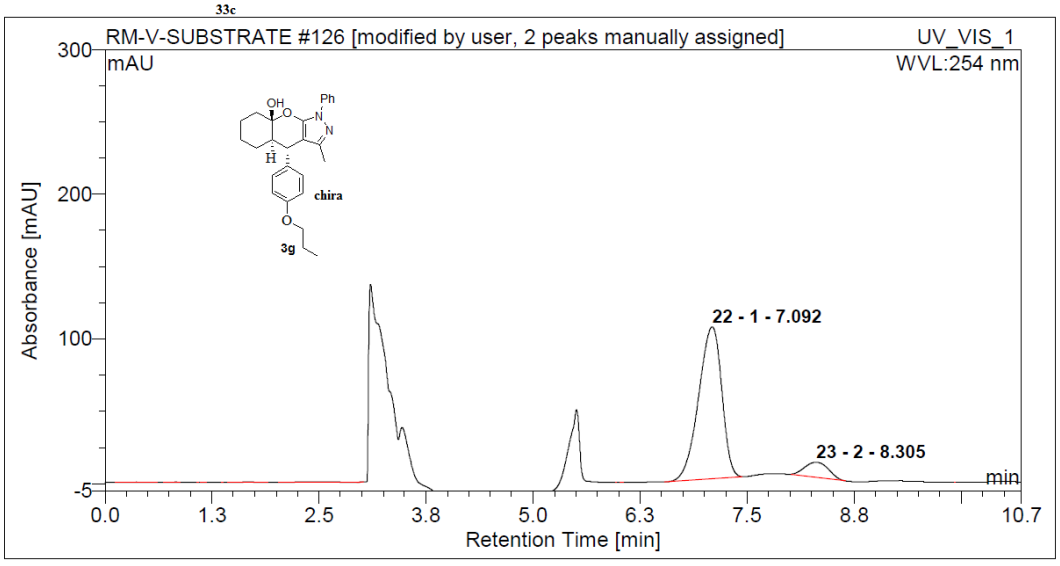
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
74	1	44.25	159.1451	98.67173314	29.88679	n.a.
75	2	53.72	2.142	1.328266864	0.434	n.a.

33r

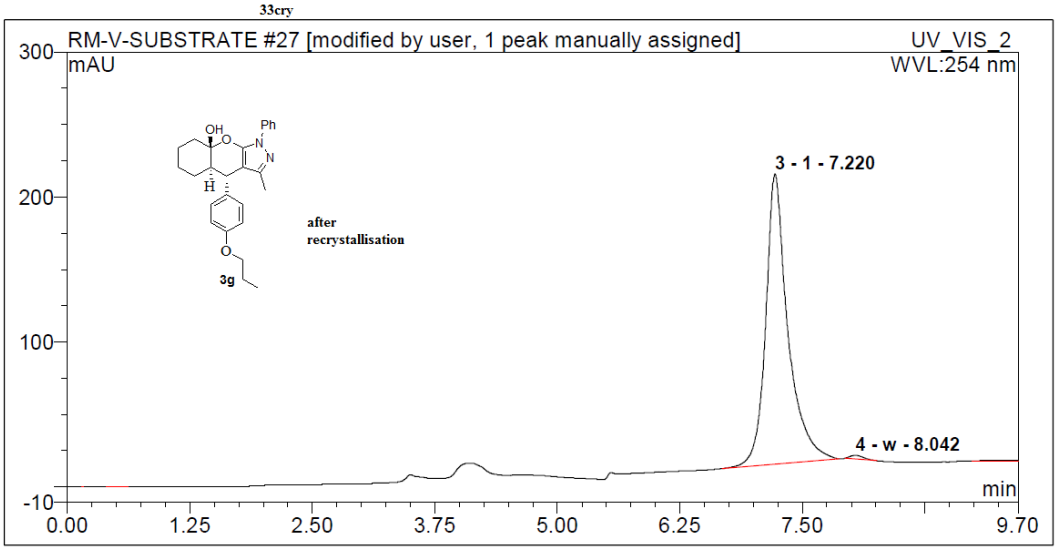


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
10	1	7.26	64.60902	48.76759285	220.1137	n.a.
11	2	8.51	67.874	51.23240715	178.351	n.a.

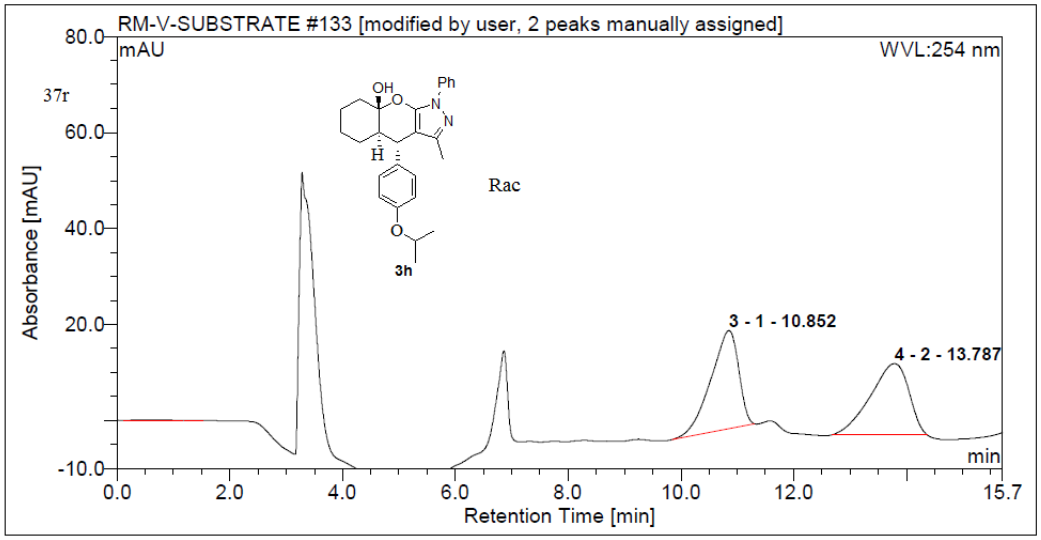
33c



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
22	1	7.09	32.95711	90.83071789	105.2045	n.a.
23	2	8.31	3.327	9.169282107	10.321	n.a.

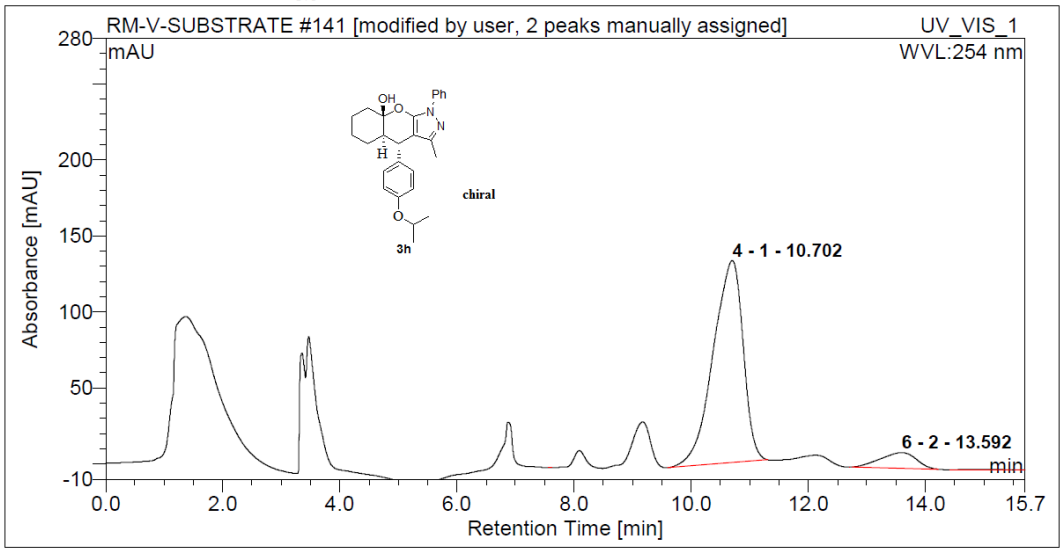


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
3	1	7.22	52.52972	99.22314133	200.2215	n.a.
4	w	8.04	0.411	0.7768586727	2.598	n.a.

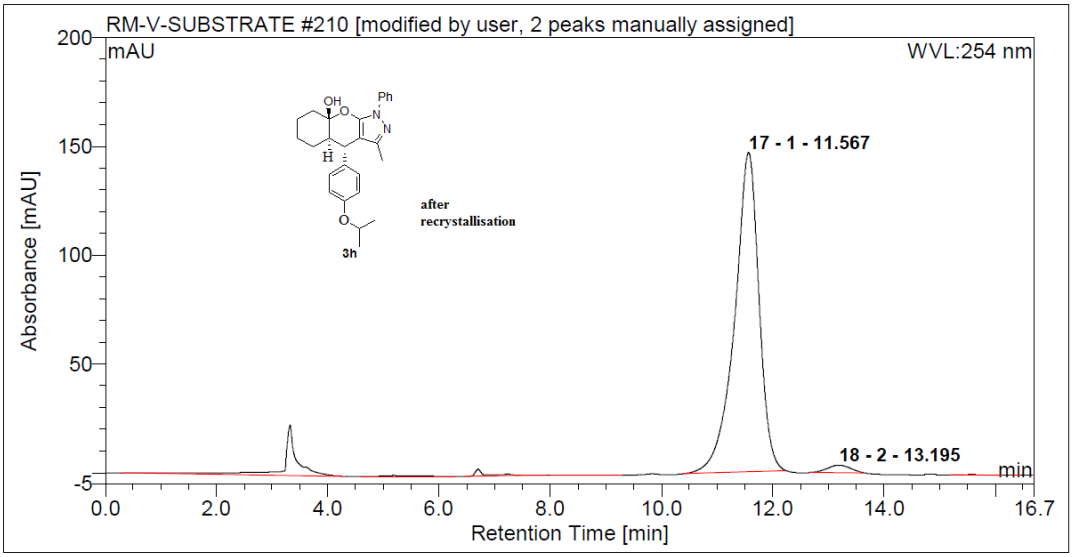


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
3	1	10.85	11.64963	49.83626555	20.40862	n.a.
4	2	13.79	11.726	50.16373445	14.856	n.a.

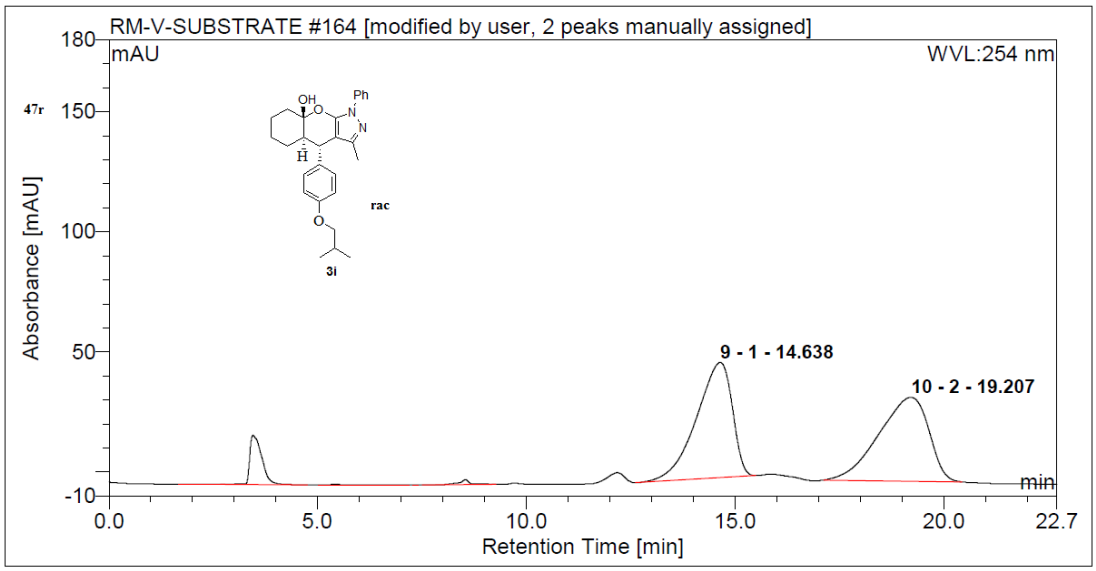
37c



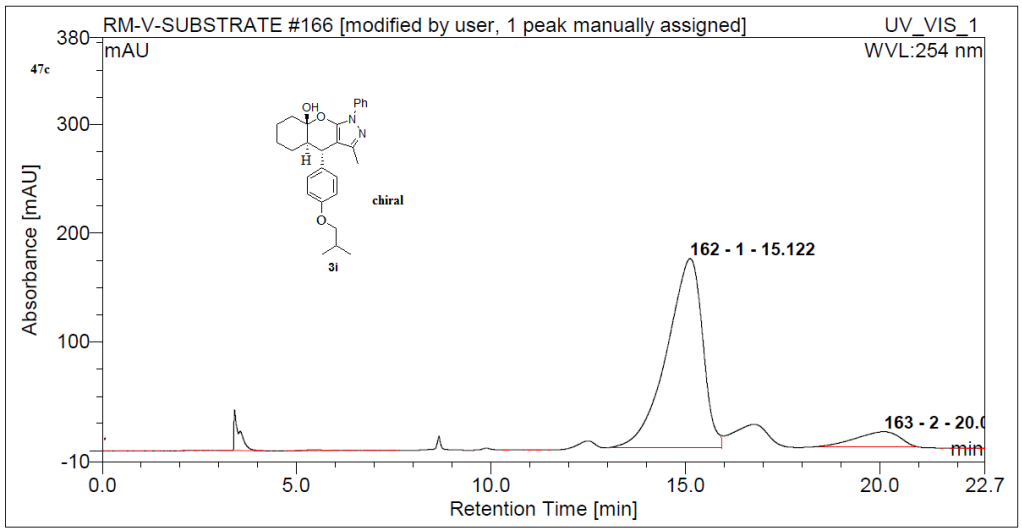
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
4	1	10.70	78.97673	91.54337673	132.943	n.a.
6	2	13.59	7.296	8.45662327	10.299	n.a.



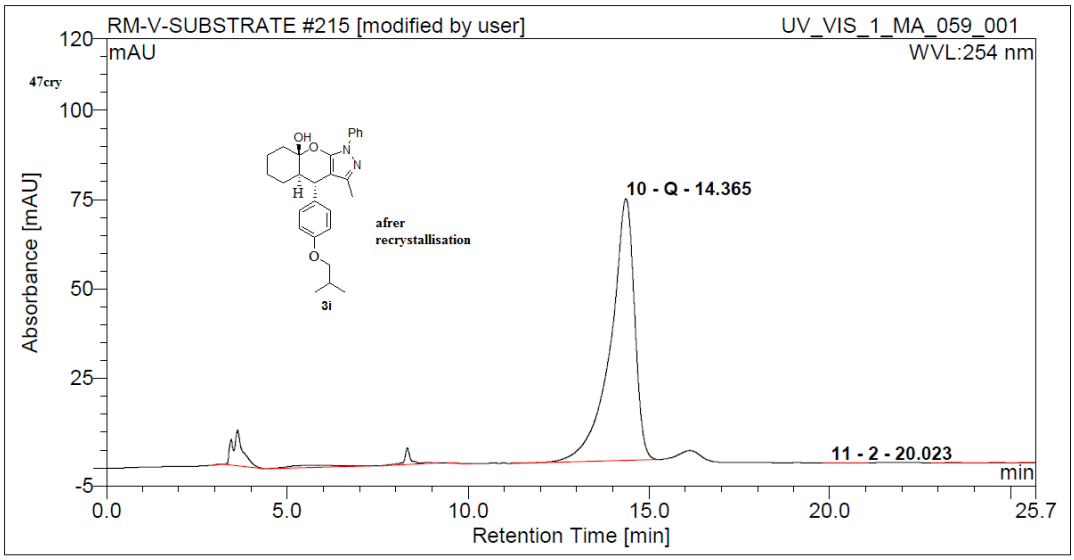
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
17	1	11.57	74.43305	97.81633576	146.6285	n.a.
18	2	13.20	1.662	2.183664237	3.463	n.a.



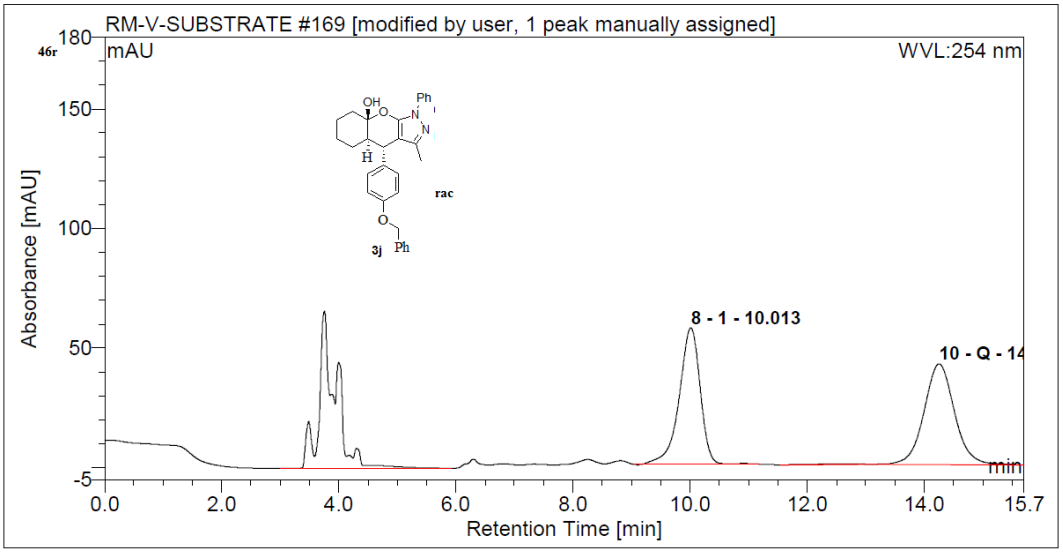
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
9	1	14.64	47.91159	49.69475423	48.05422	n.a.
10	2	19.21	48.500	50.30524577	34.928	n.a.



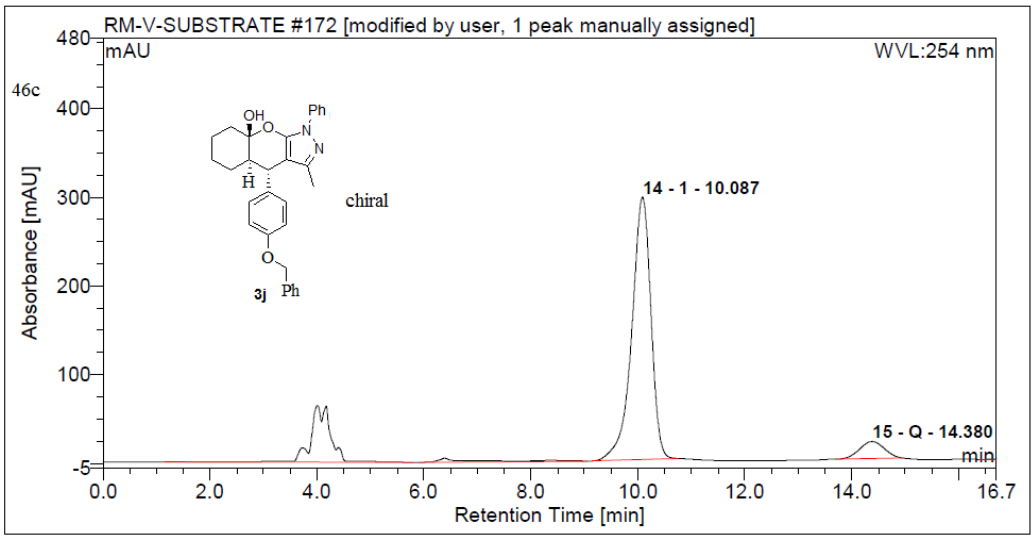
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
162	1	15.12	185.2489	91.40914943	173.8759	n.a.
163	2	20.10	17.410	8.590821825	13.588	n.a.



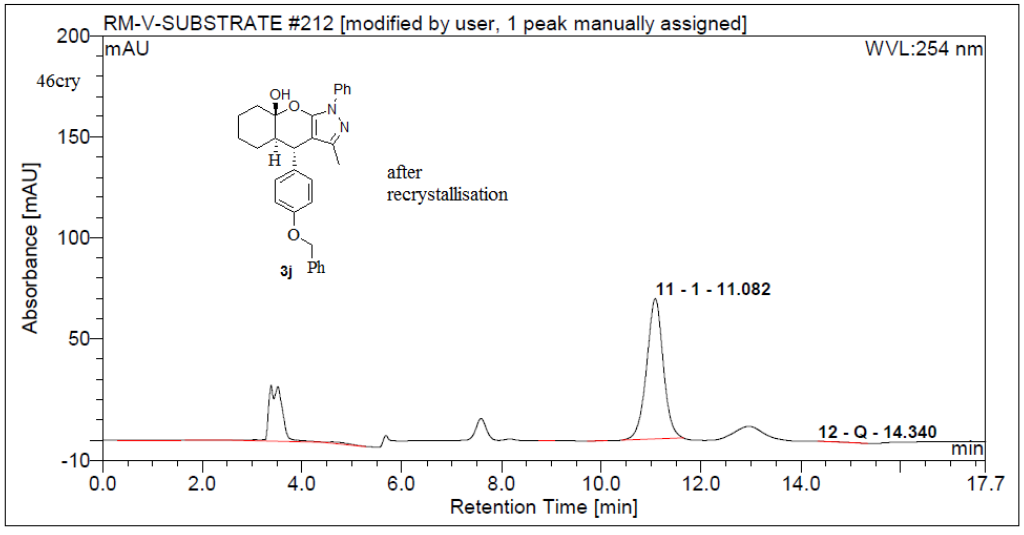
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
10	Q	14.37	56.19517	99.94929402	73.14159	n.a.
11	2	20.02	0.029	0.05070597747	0.038	n.a.



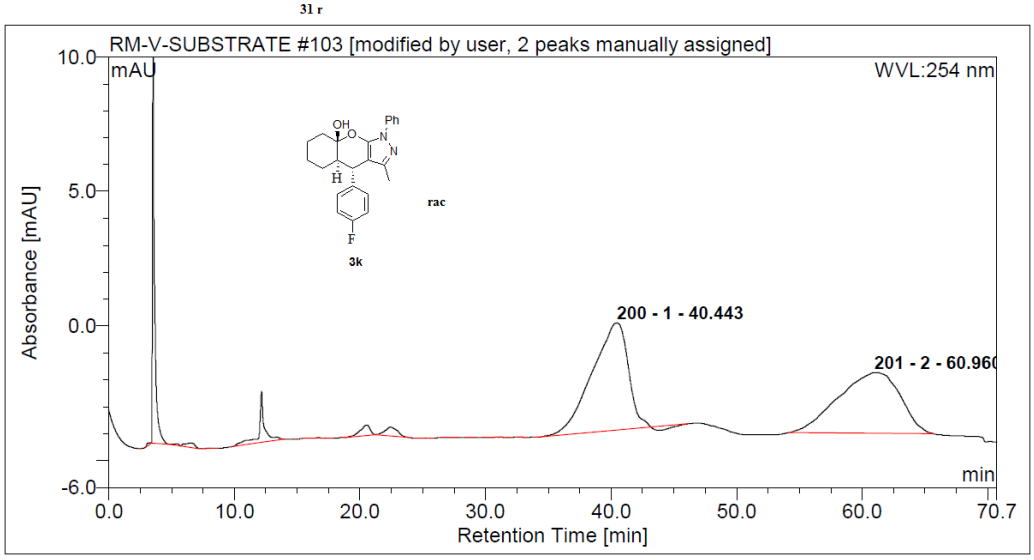
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
8	1	10.01	23.74339	48.5428718	57.04979	n.a.
10	Q	14.25	25.169	51.4571282	42.032	n.a.



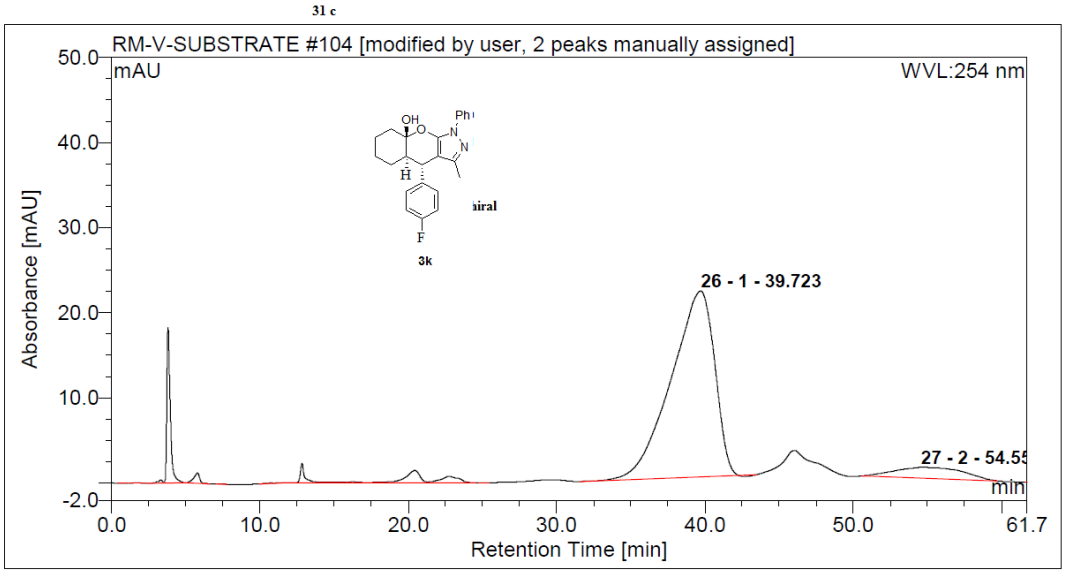
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
14	1	10.09	122.8068	92.17728124	295.8164	n.a.
15	Q	14.38	10.422	7.822718758	19.334	n.a.



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
11	1	11.08	26.81413	99.8139195	69.37472	n.a.
12	Q	14.34	0.050	0.1860805048	0.001	n.a.

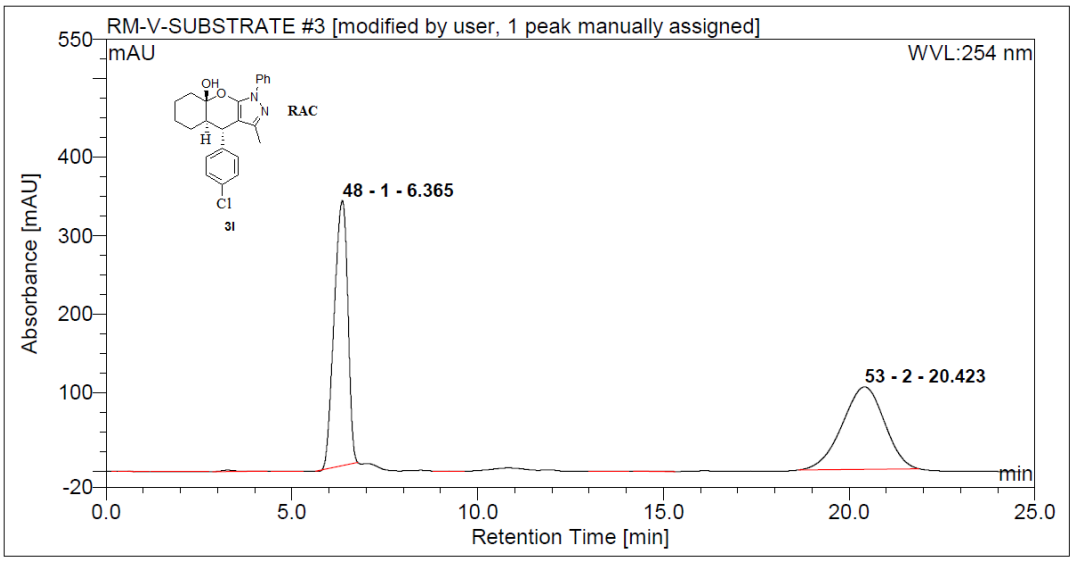


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
200	1	40.44	13.07581	50.29760624	3.98058	n.a.
201	2	60.96	12.921	49.70239376	2.259	n.a.



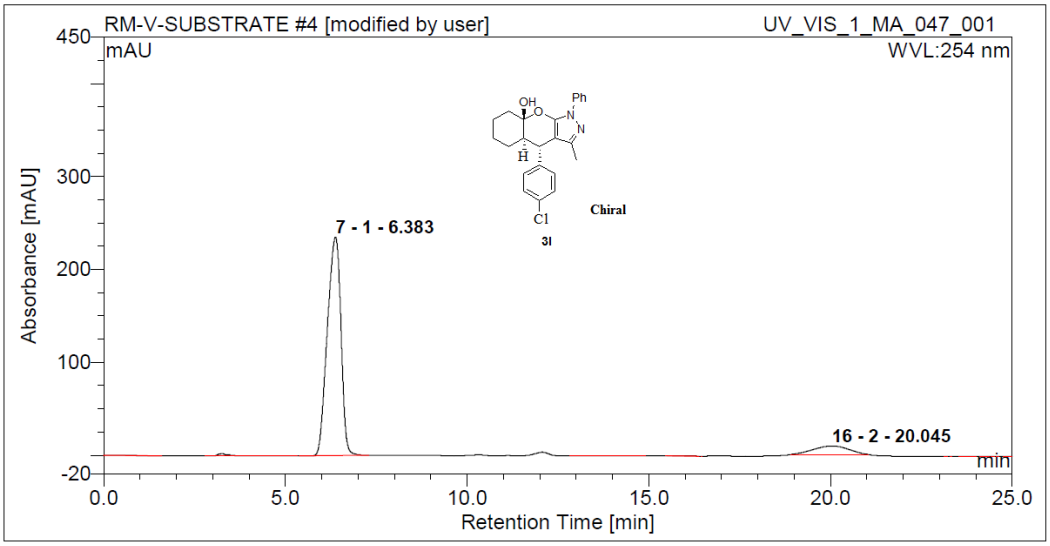
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
26	1	39.72	74.07457	91.55114943	21.80919	n.a.
27	2	54.55	6.836	8.448850568	1.248	n.a.

2R

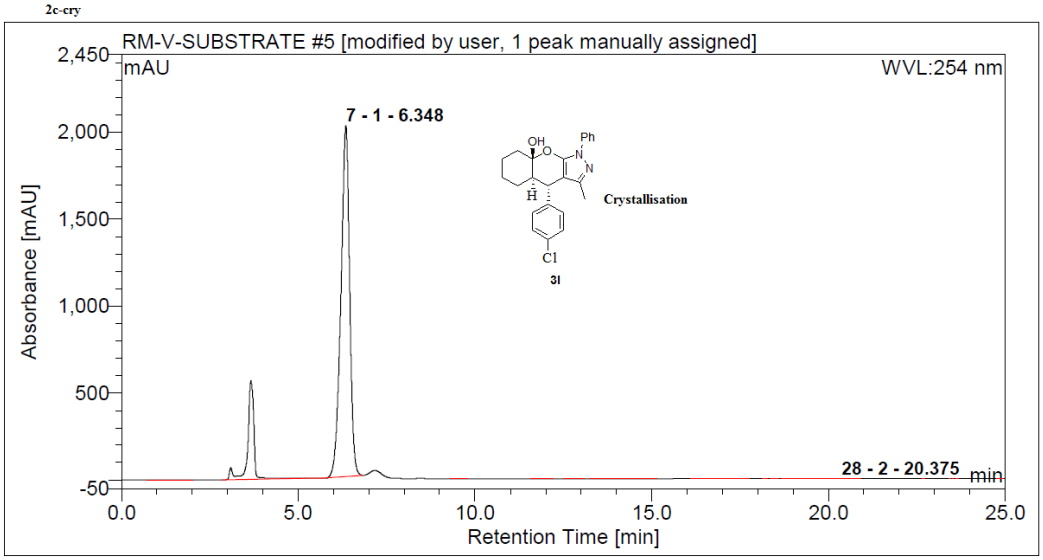


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
48	1	6.37	143.4327	50.97636551	337.4685	n.a.
53	2	20.42	137.938	49.02363449	104.748	n.a.

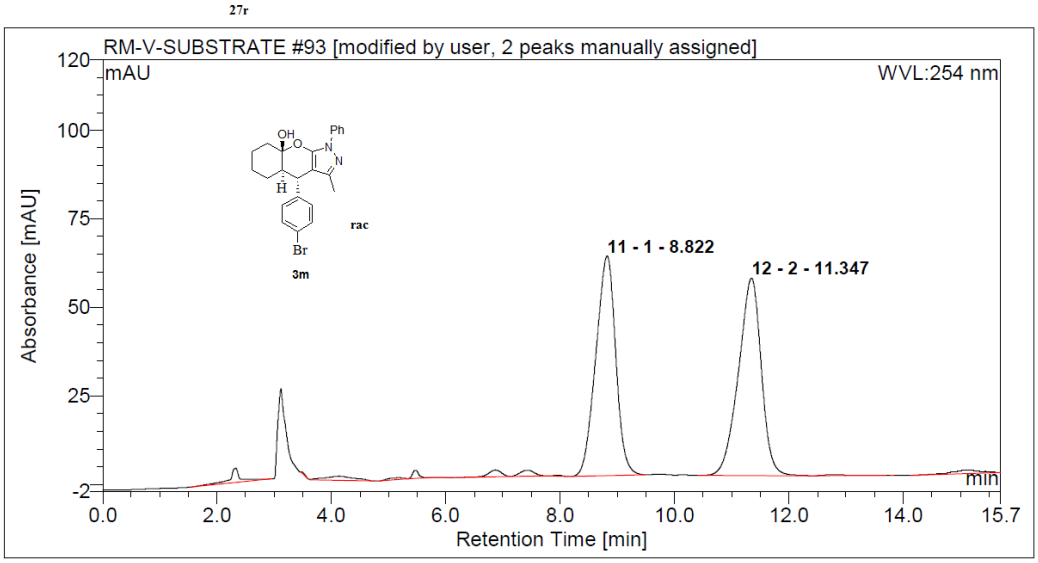
2C



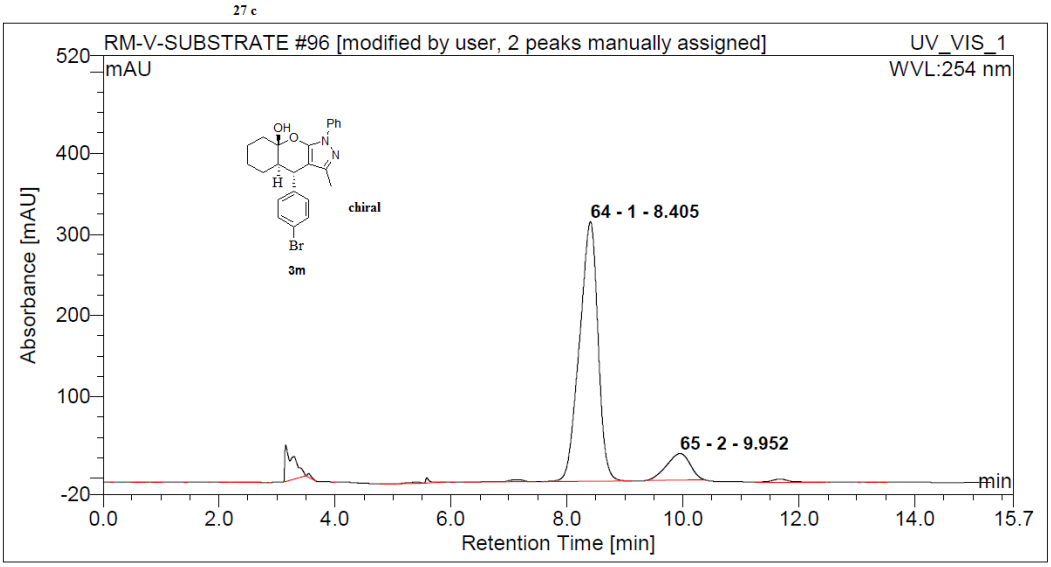
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
7	1	6.38	102.0657	90.60426966	235.2563	n.a.
16	2	20.05	10.584	9.395730337	9.216	n.a.



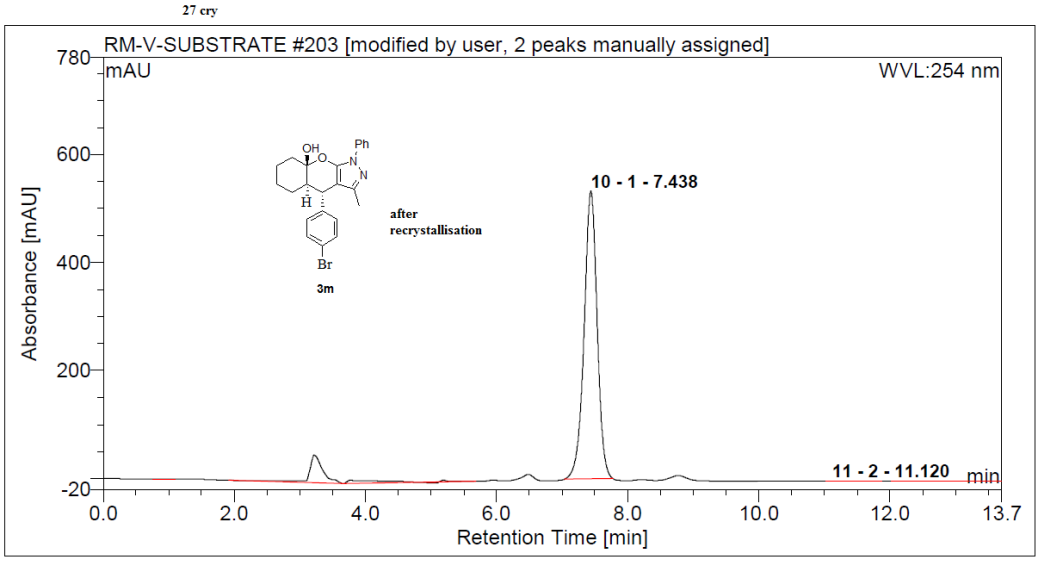
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
7	1	6.35	580.0302	99.82584845	2021.68	n.a.
28	2	20.38	1.012	0.1741515509	1.303	n.a.



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
11	1	8.82	25.12067	49.26859273	62.09706	n.a.
12	2	11.35	25.867	50.73140727	55.838	n.a.

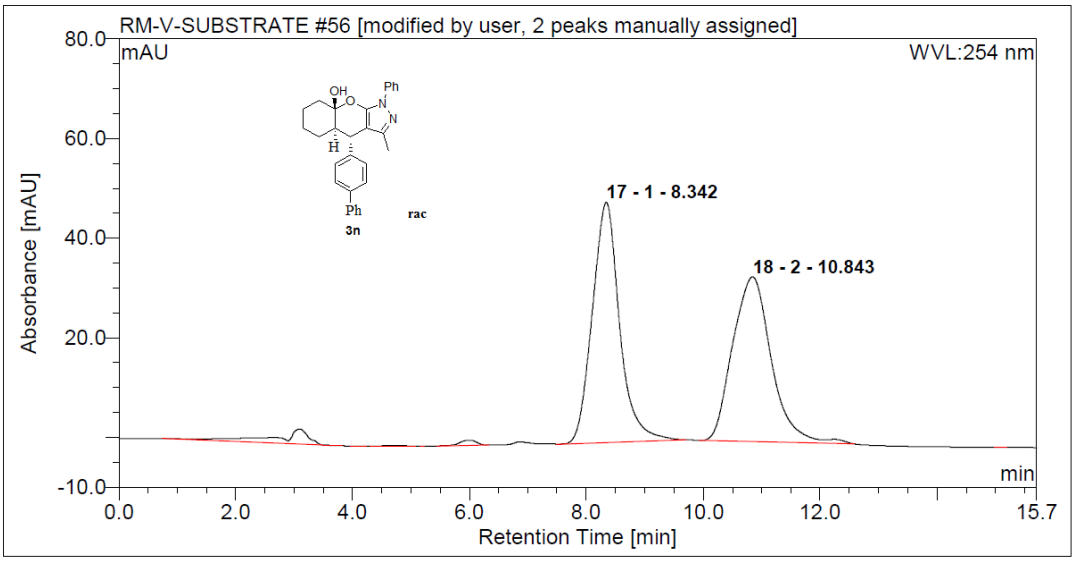


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
64	1	8.41	118.148	88.5557724	319.5478	n.a.
65	2	9.95	15.268	11.4442276	32.521	n.a.



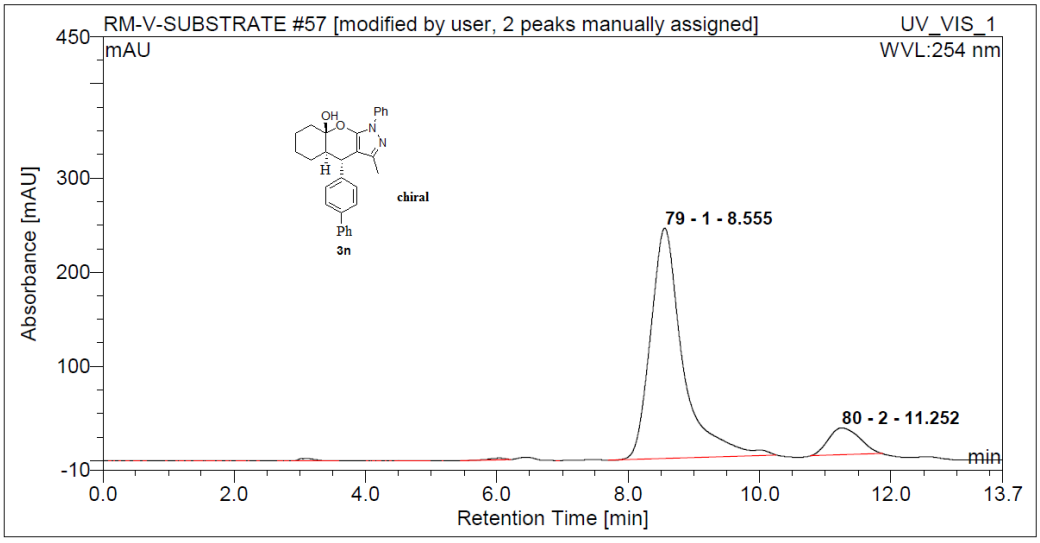
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
10	1	7.44	123.0126	99.92915816	532.6069	n.a.
11	2	11.12	0.087	0.07084184129	0.096	n.a.

17 r

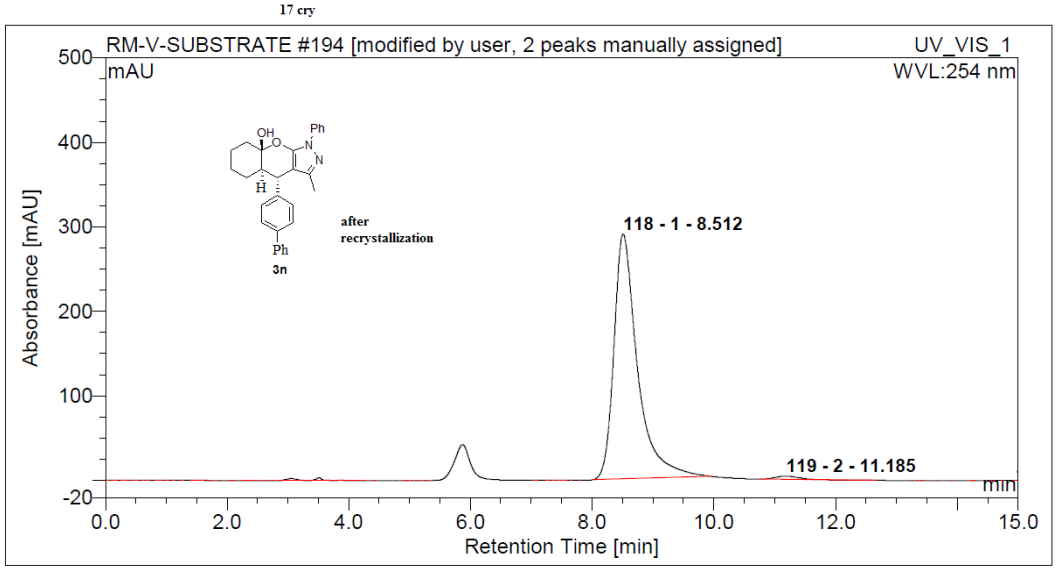


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
17	1	8.34	26.00719	50.5943782	48.1696	n.a.
18	2	10.84	25.396	49.4056218	33.069	n.a.

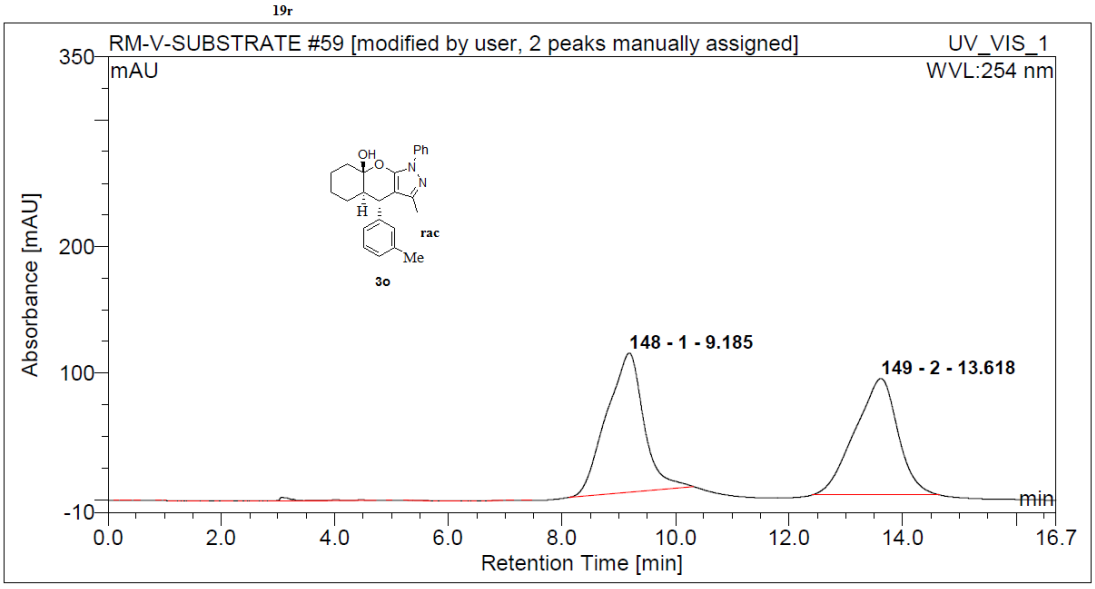
17c



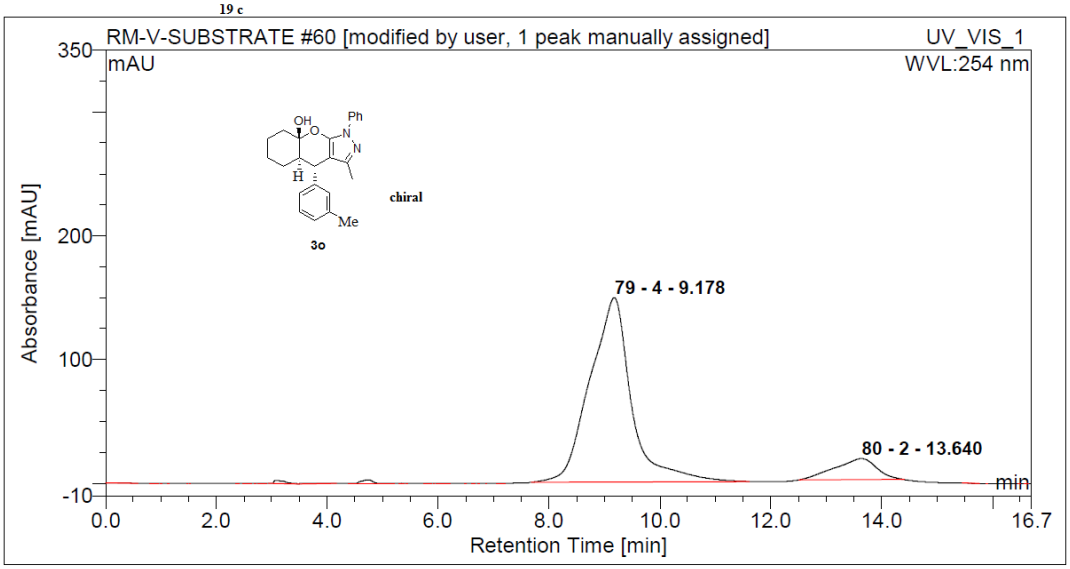
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
79	1	8.56	139.0654	89.78258946	244.5251	n.a.
80	2	11.25	15.826	10.21741054	28.666	n.a.



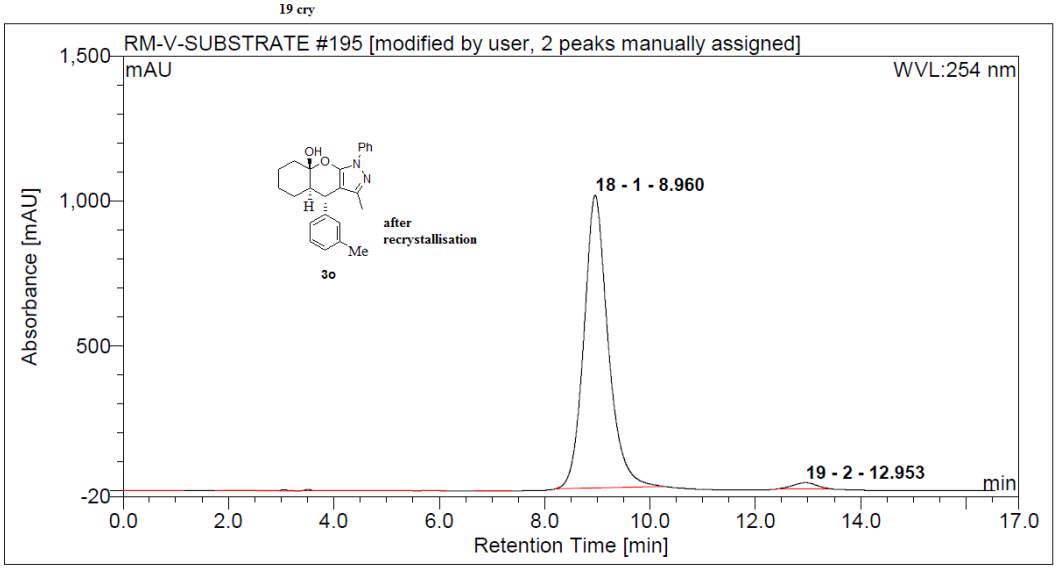
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
118	1	8.51	130.1518	98.77345639	289.4745	n.a.
119	2	11.19	1.616	1.226468399	3.841	n.a.



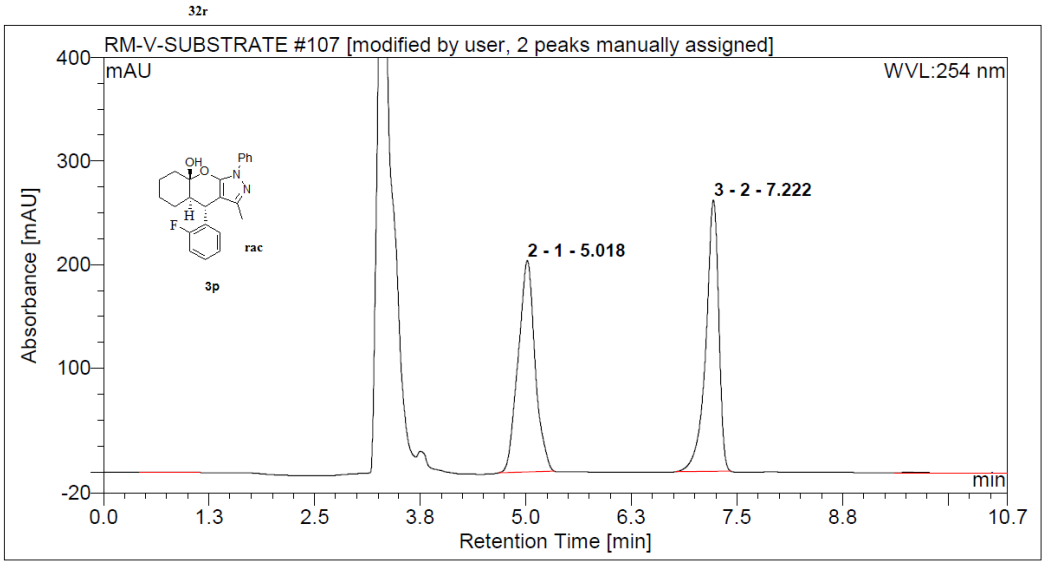
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
148	1	9.19	83.3417	50.83030384	109.8366	n.a.
149	2	13.62	80.619	49.16969616	92.038	n.a.



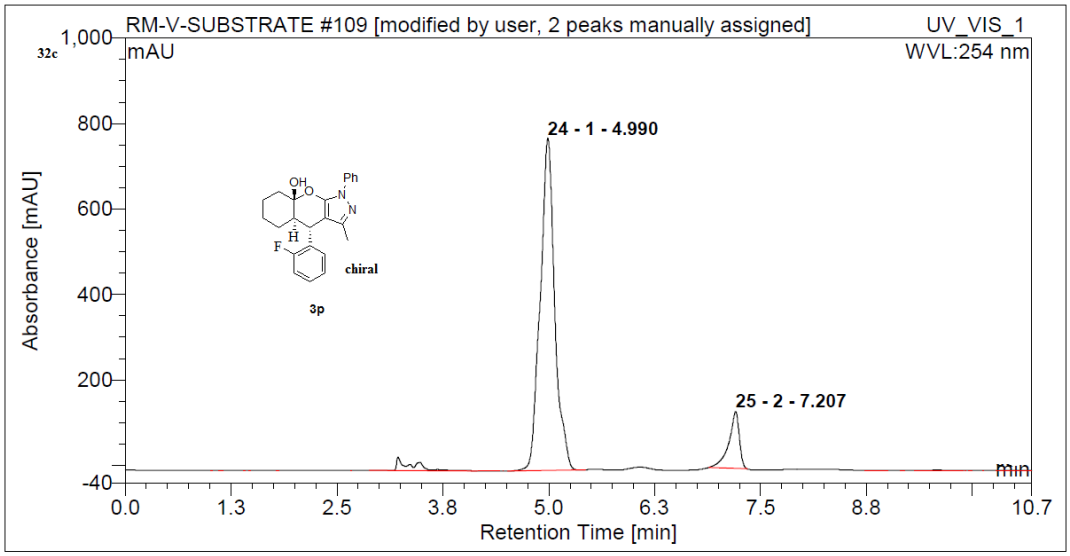
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
79	4	9.18	129.0787	89.61614847	148.9249	n.a.
80	2	13.64	14.956	10.38380771	17.000	n.a.



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
18	1	8.96	537.5597	98.10463902	1011.313	n.a.
19	2	12.95	10.386	1.895360984	20.929	n.a.

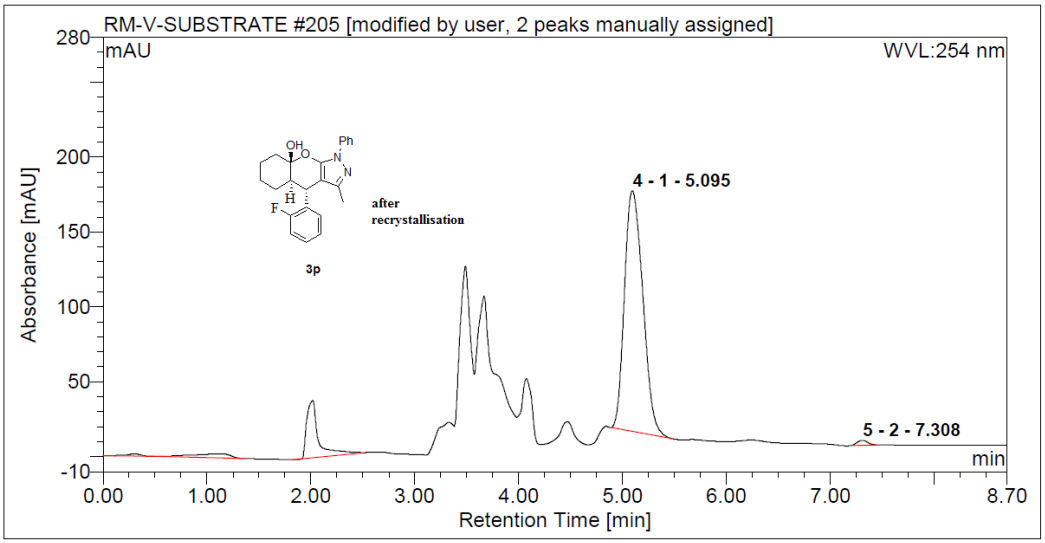


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
2	1	5.02	47.44924	50.6336588	204.016	n.a.
3	2	7.22	46.25371	49.35789532	261.6498	n.a.



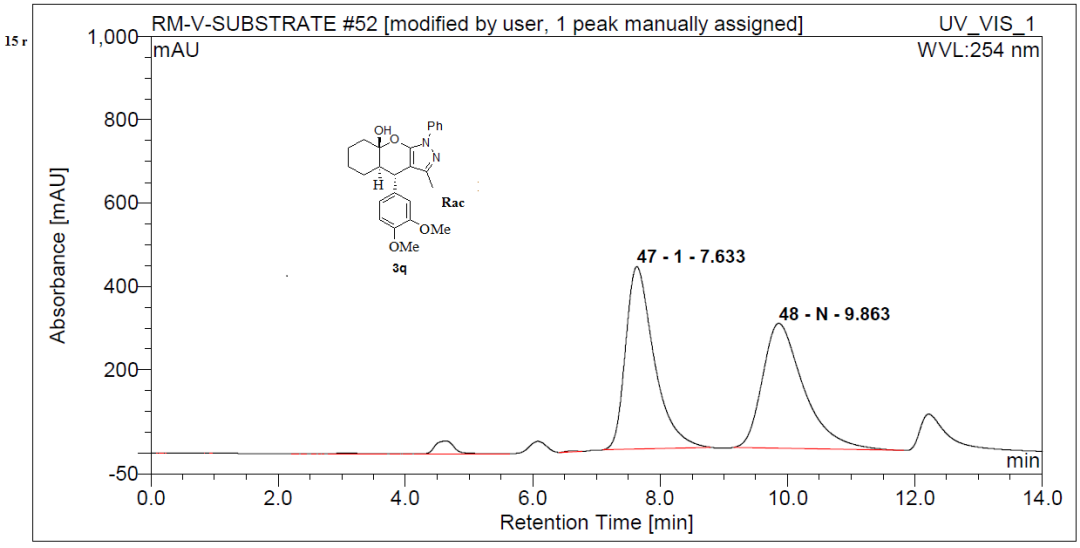
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
24	1	4.99	154.0304	89.00551095	776.7246	n.a.
25	2	7.21	19.027	10.994488	133.507	n.a.

32c

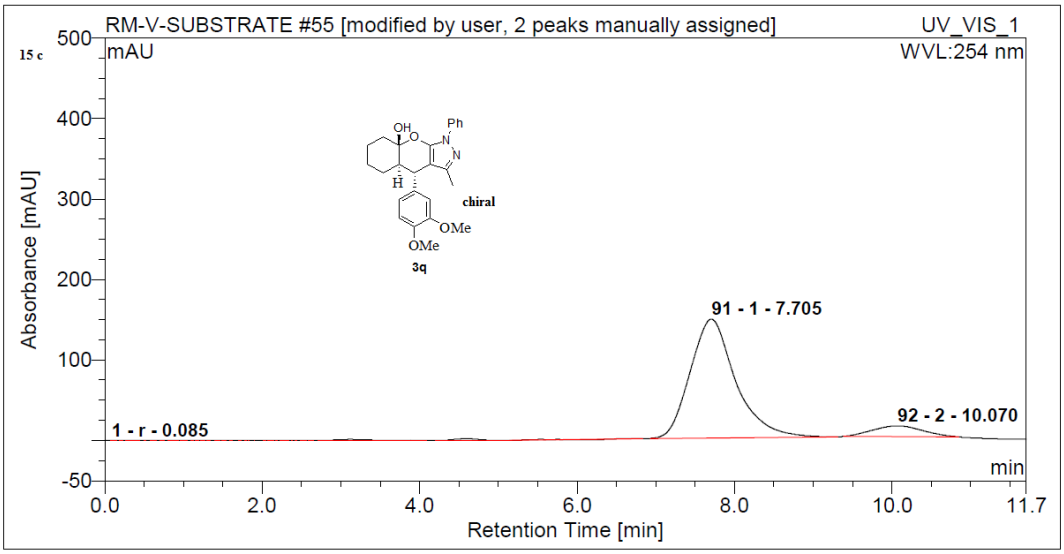


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
4	1	5.10	32.99658	98.90005687	160.7145	n.a.
5	2	7.31	0.367	1.099943134	3.222	n.a.

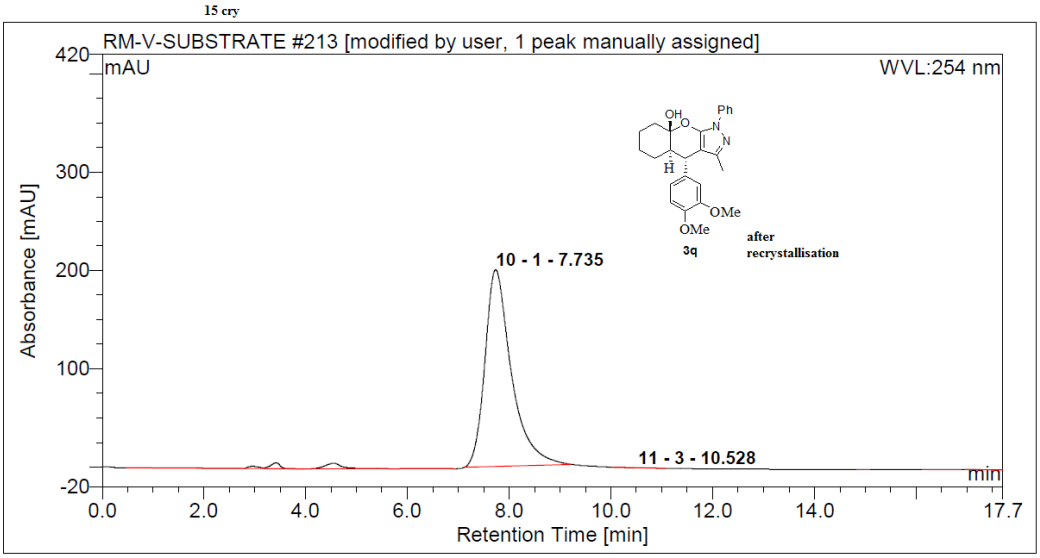
15 r



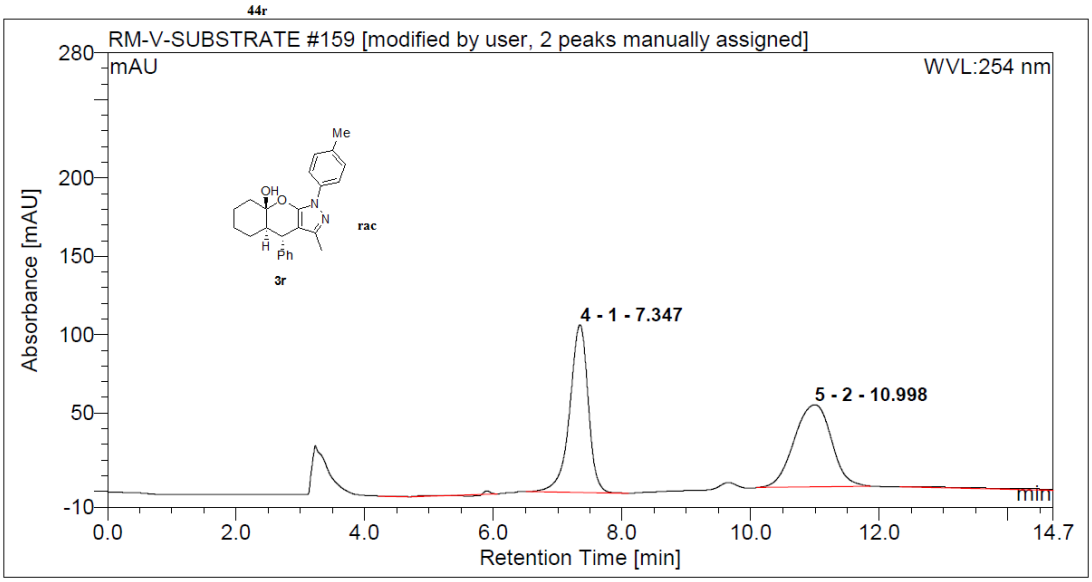
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
47	1	7.63	226.9436	50.21857287	437.6909	n.a.
48	N	9.86	224.968	49.78142713	299.778	n.a.



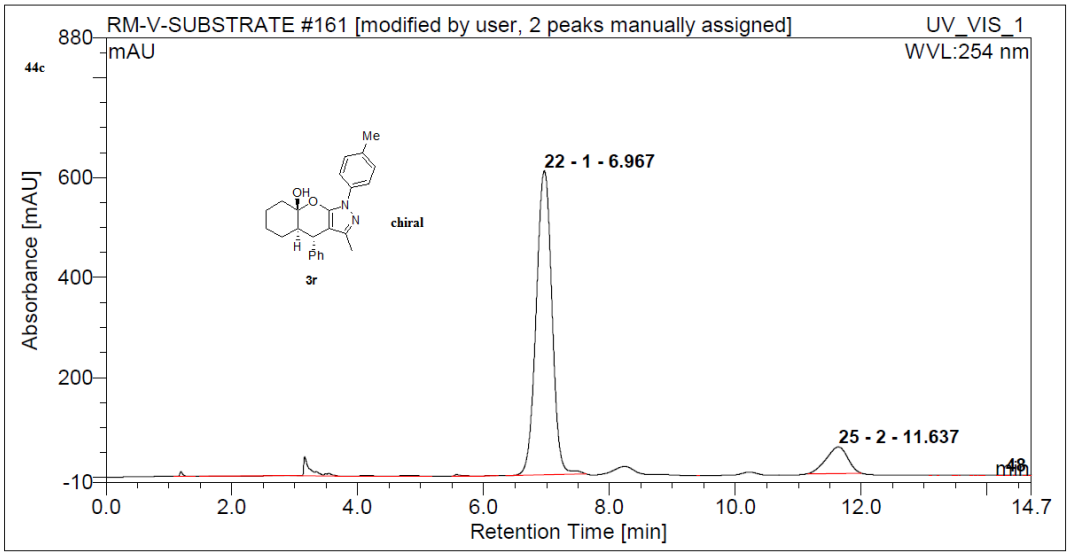
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
91	1	7.71	98.26504	90.79933758	147.5366	n.a.
92	2	10.07	9.957	9.200631487	13.488	n.a.



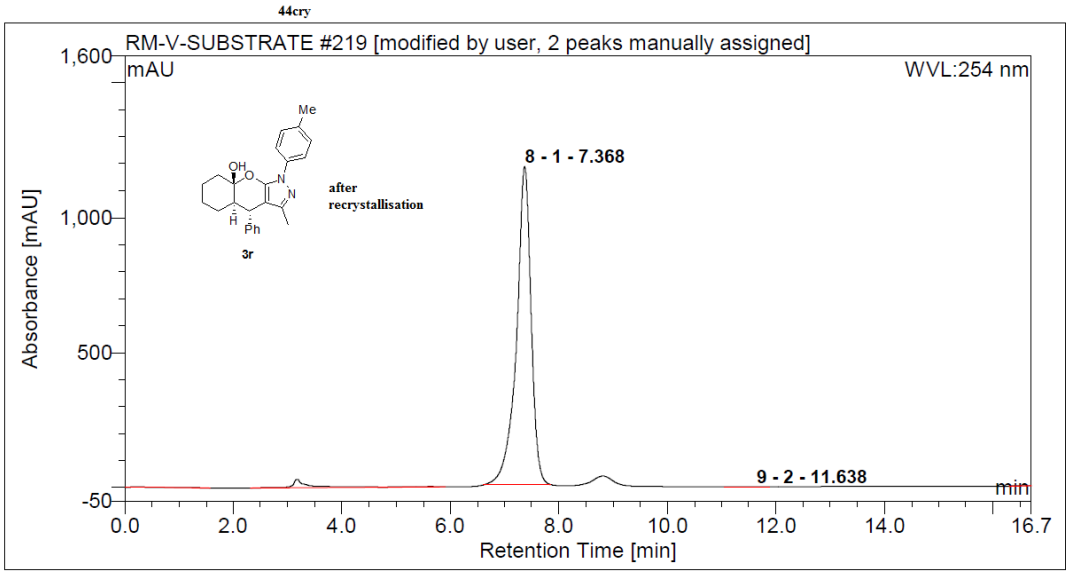
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
10	1	7.74	120.5015	99.94685467	200.3093	n.a.
11	3	10.53	0.064	0.05314533272	0.107	n.a.



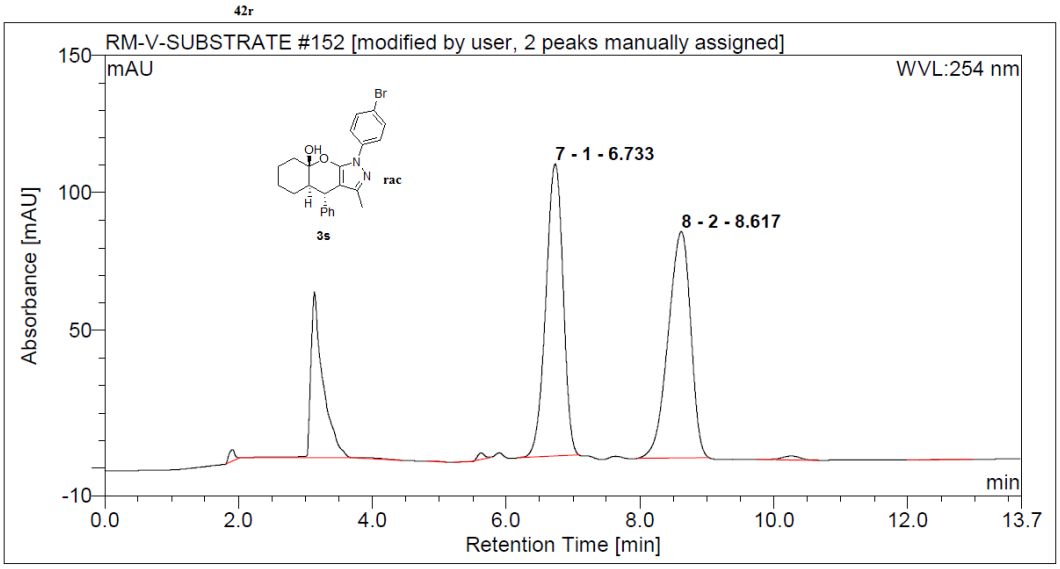
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
4	1	7.35	35.70472	50.415872	107.0007	n.a.
5	2	11.00	35.116	49.584128	52.430	n.a.



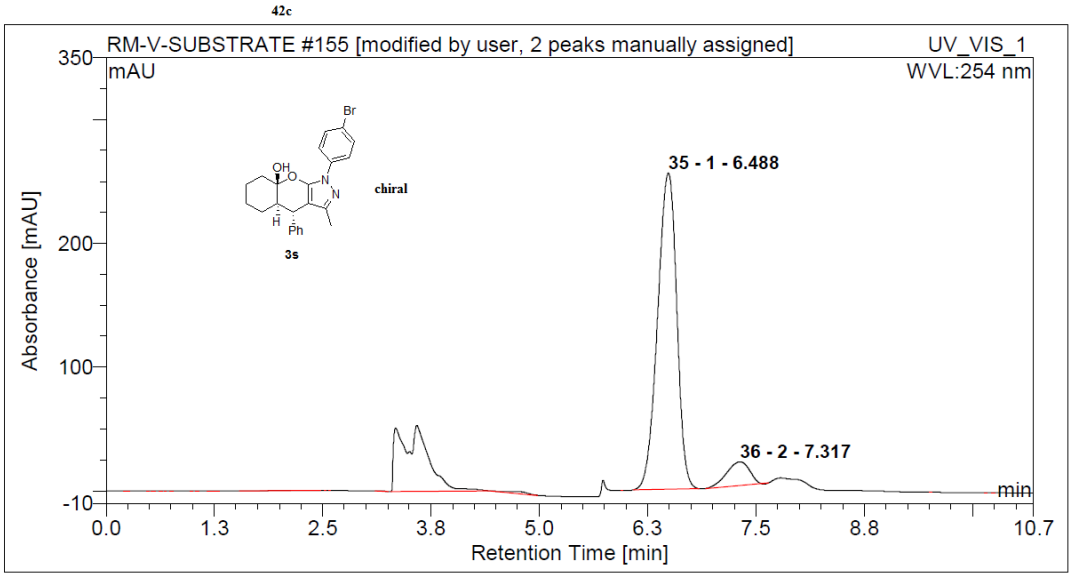
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
22	1	6.97	176.7051	89.47454337	608.8062	n.a.
25	2	11.64	20.787	10.52540101	52.747	n.a.



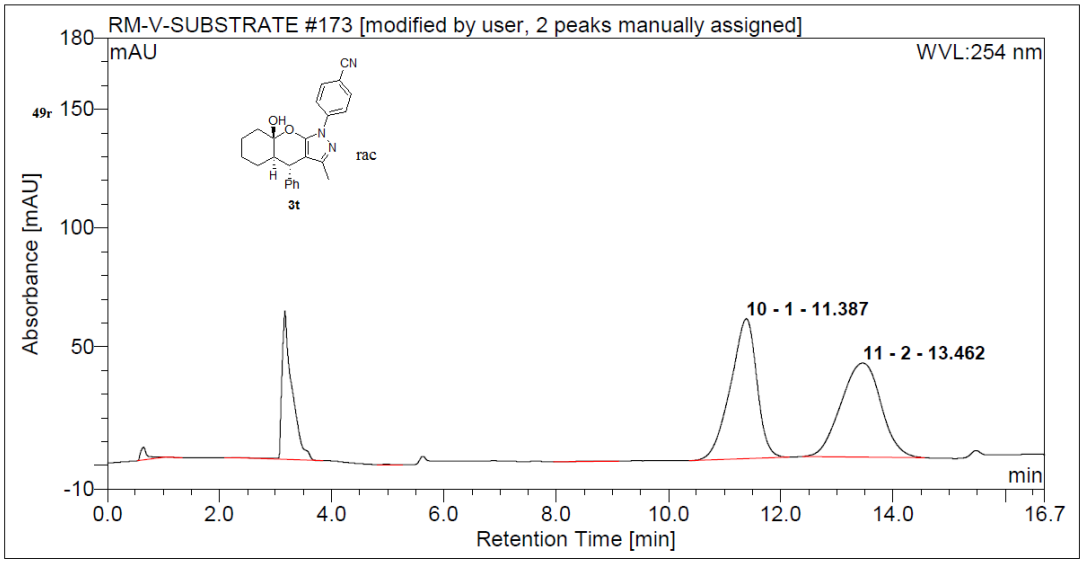
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
8	1	7.37	367.8052	99.9962393	1178.243	n.a.
9	2	11.64	0.014	0.003760699293	0.045	n.a.



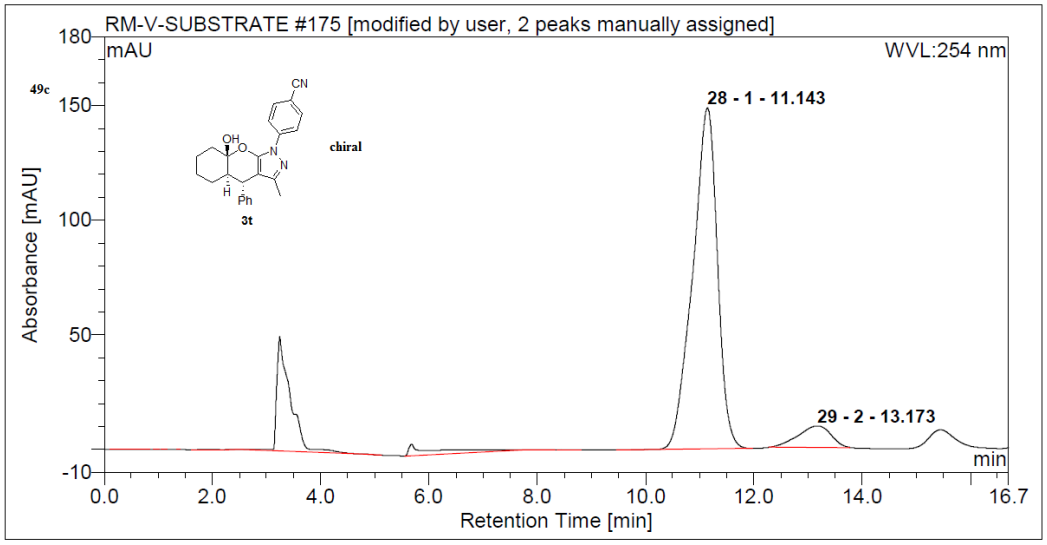
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
7	1	6.73	31.75629	49.78829222	106.1779	n.a.
8	2	8.62	32.026	50.21170778	82.197	n.a.



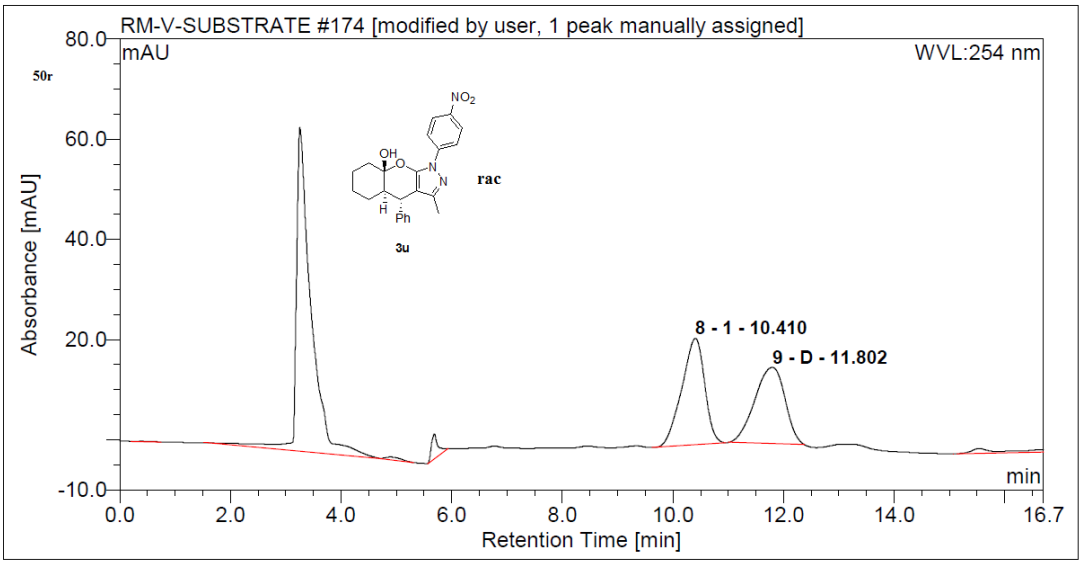
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
35	1	6.49	65.40509	91.84109101	255.3015	n.a.
36	2	7.32	5.810	8.158908986	18.999	n.a.



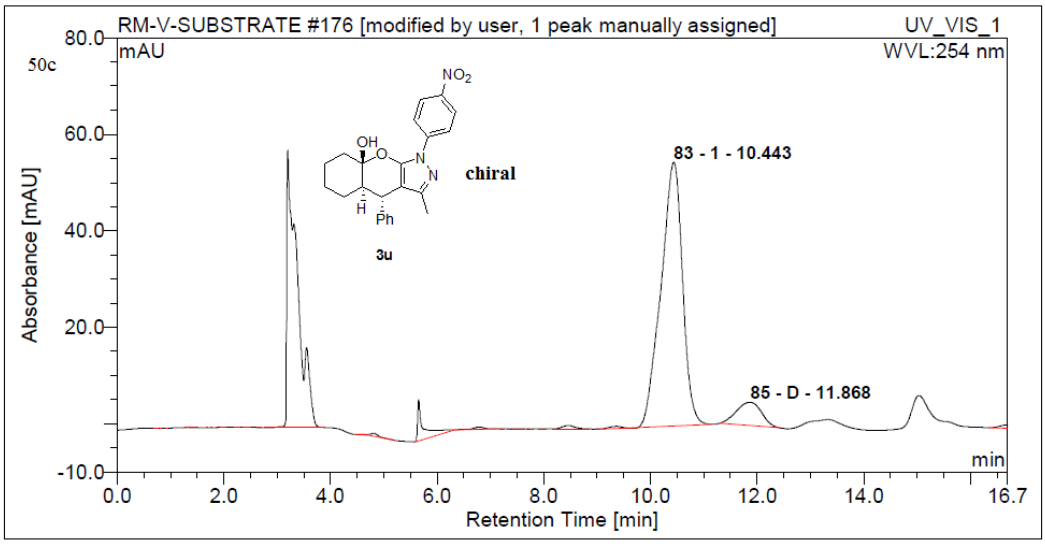
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
10	1	11.39	32.10438	49.46097822	58.95474	n.a.
11	2	13.46	32.804	50.53902178	39.632	n.a.



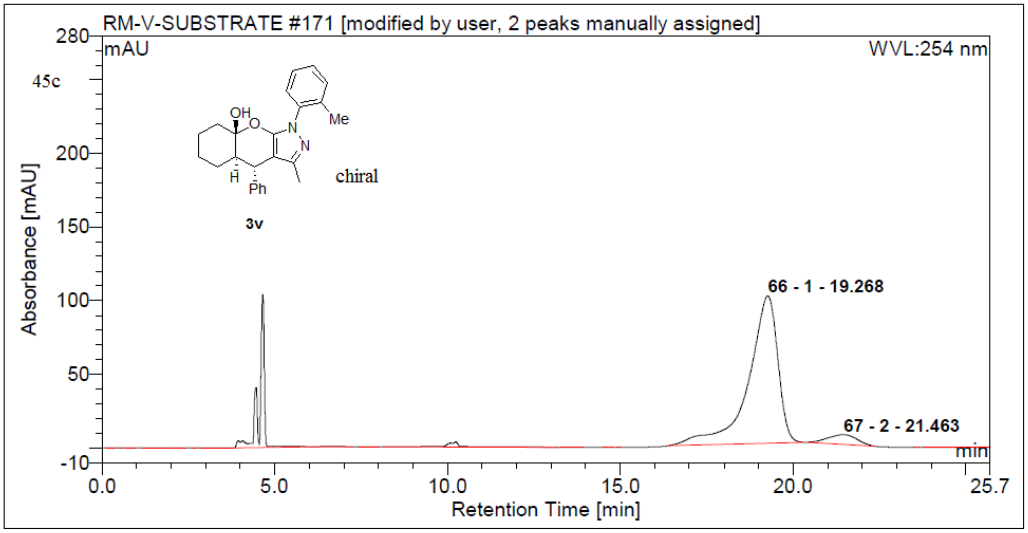
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
28	1	11.14	78.54228	92.30055572	148.8167	n.a.
29	2	13.17	6.552	7.699444278	9.354	n.a.



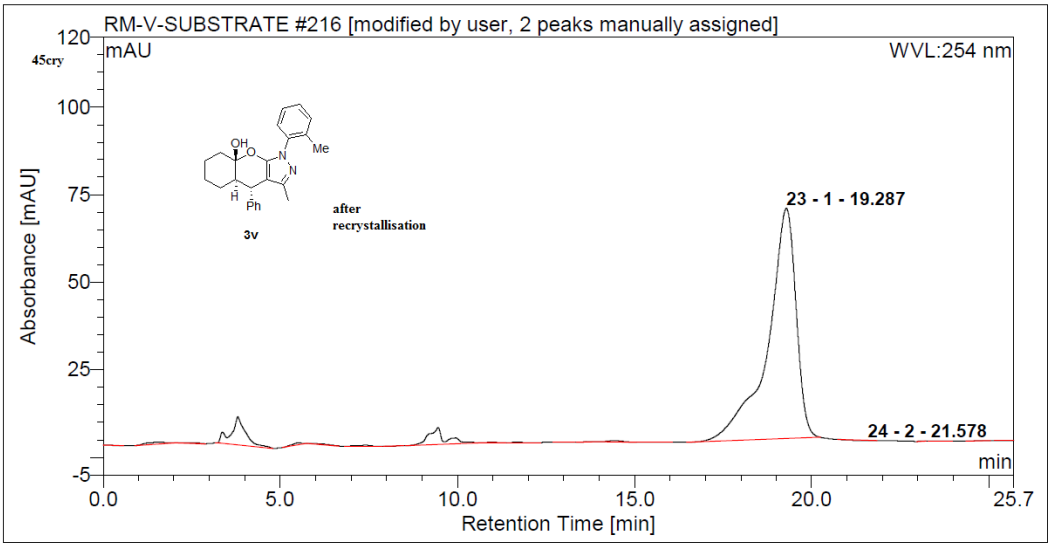
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
8	1	10.41	10.17577	51.37146763	21.20719	n.a.
9	D	11.80	9.632	48.62853237	15.225	n.a.



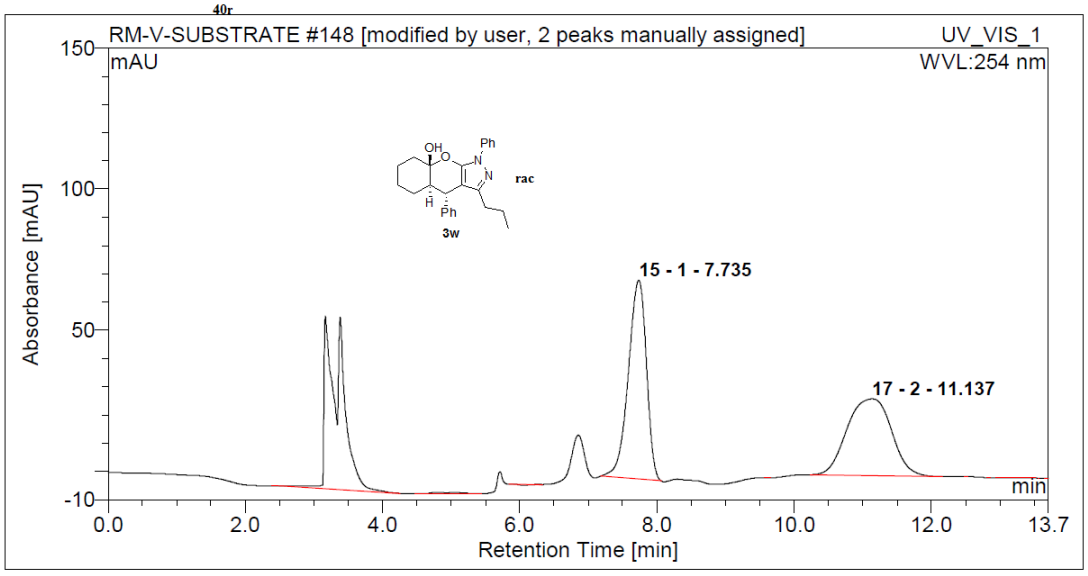
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
83	1	10.44	25.61669	90.63297457	54.68655	n.a.
85	D	11.87	2.648	9.367025427	4.853	n.a.



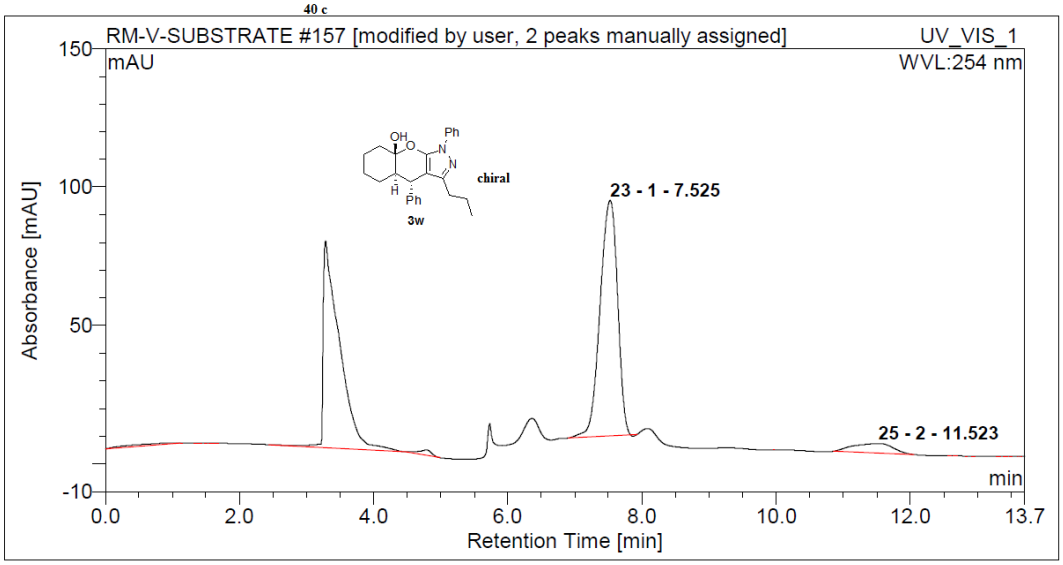
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
66	1	19.27	98.41064	93.9321524	100.0489	n.a.
67	2	21.46	6.357	6.067847599	6.595	n.a.



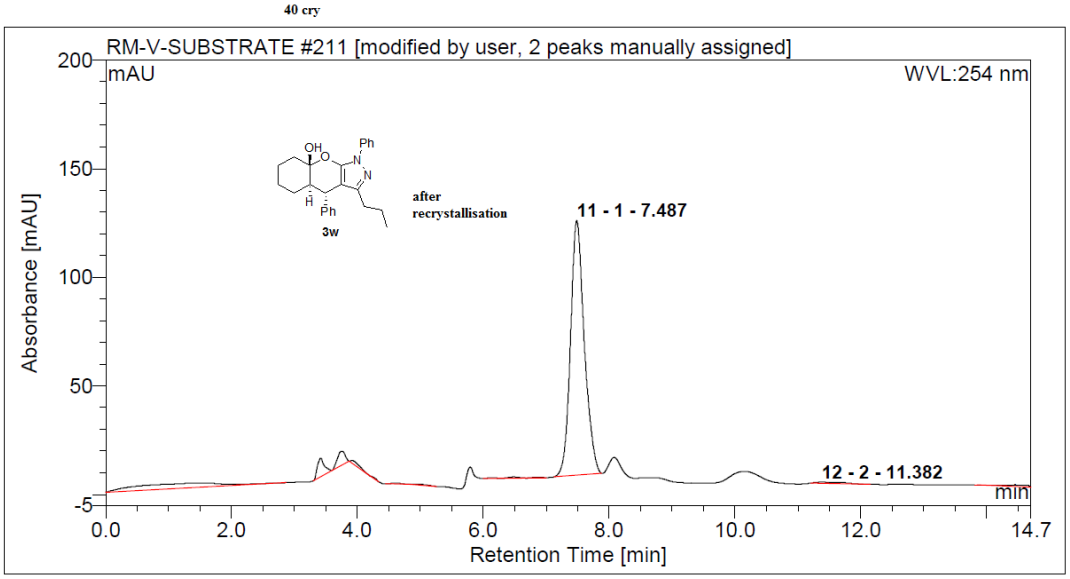
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
23	1	19.29	60.90625	99.86006831	65.71327	n.a.
24	2	21.58	0.085	0.1399316888	0.098	n.a.



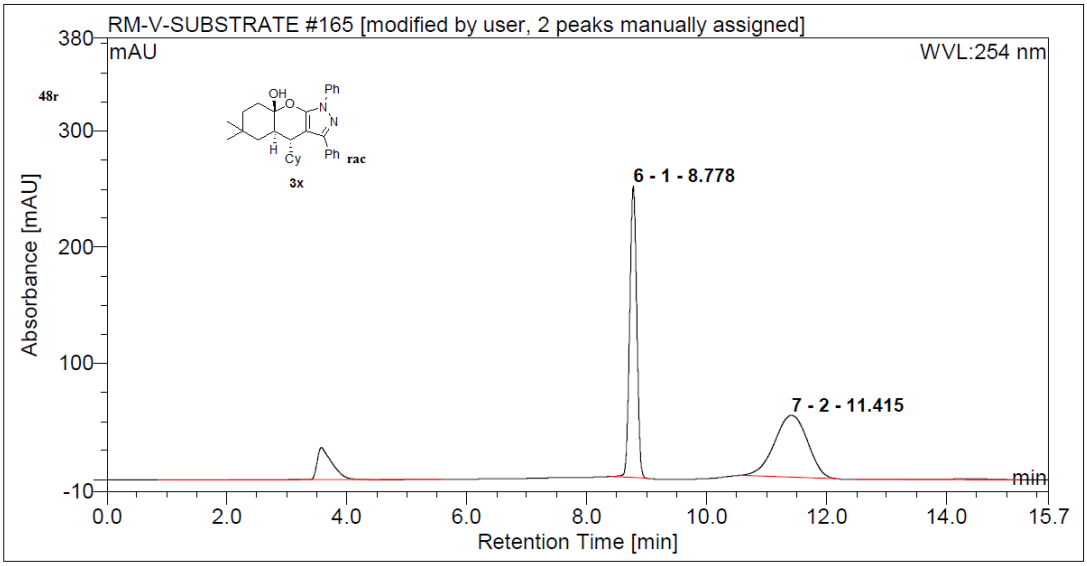
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
15	1	7.74	21.49733	51.37301178	70.45408	n.a.
17	2	11.14	20.348	48.62698822	27.186	n.a.



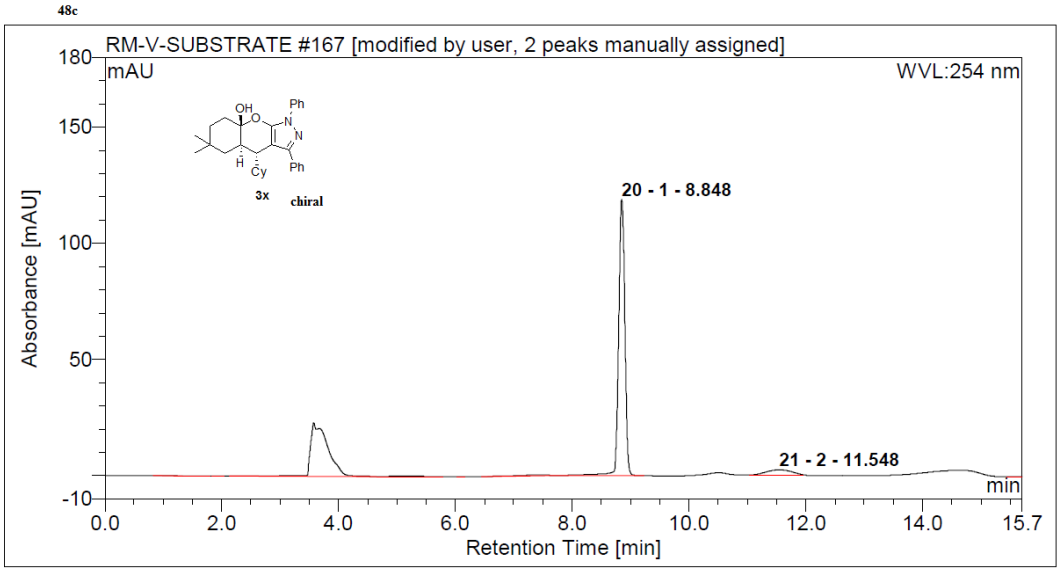
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
23	1	7.53	25.6955	91.65795549	85.07796	n.a.
25	2	11.52	2.339	8.342016122	3.490	n.a.



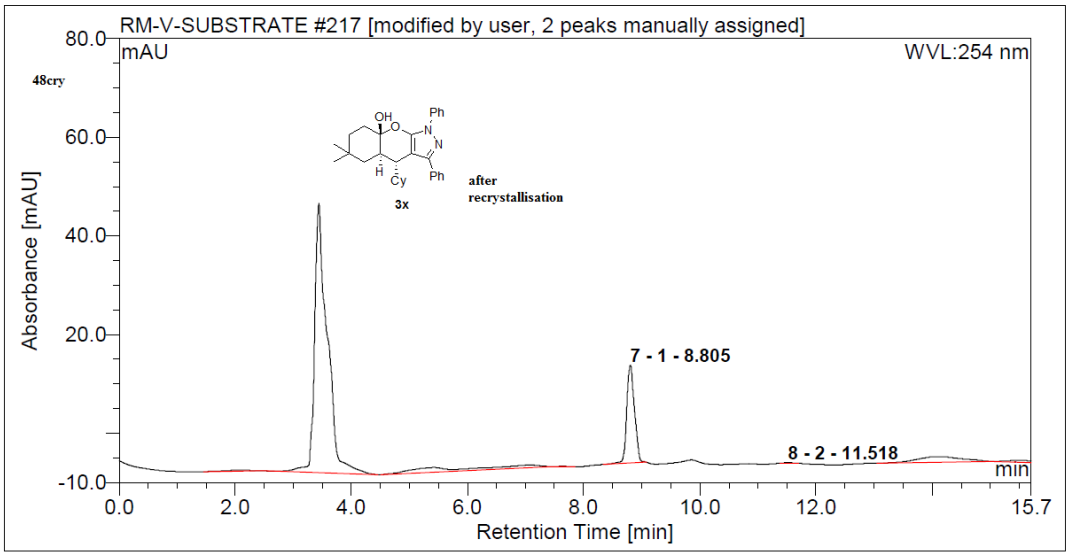
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
11	1	7.49	29.80076	99.0064909	117.1744	n.a.
12	2	11.38	0.299	0.9935091048	0.573	n.a.



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
6	1	8.78	34.11079	49.95030592	251.0615	n.a.
7	2	11.42	34.179	50.04969408	53.241	n.a.

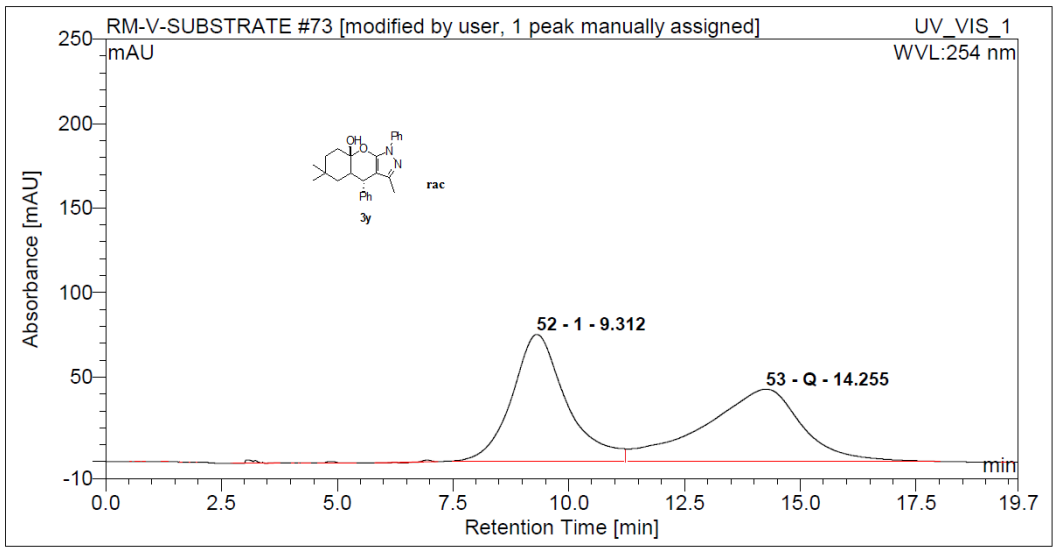


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
20	1	8.85	14.71986	93.23273559	119.0033	n.a.
21	2	11.55	1.068	6.767264406	2.170	n.a.

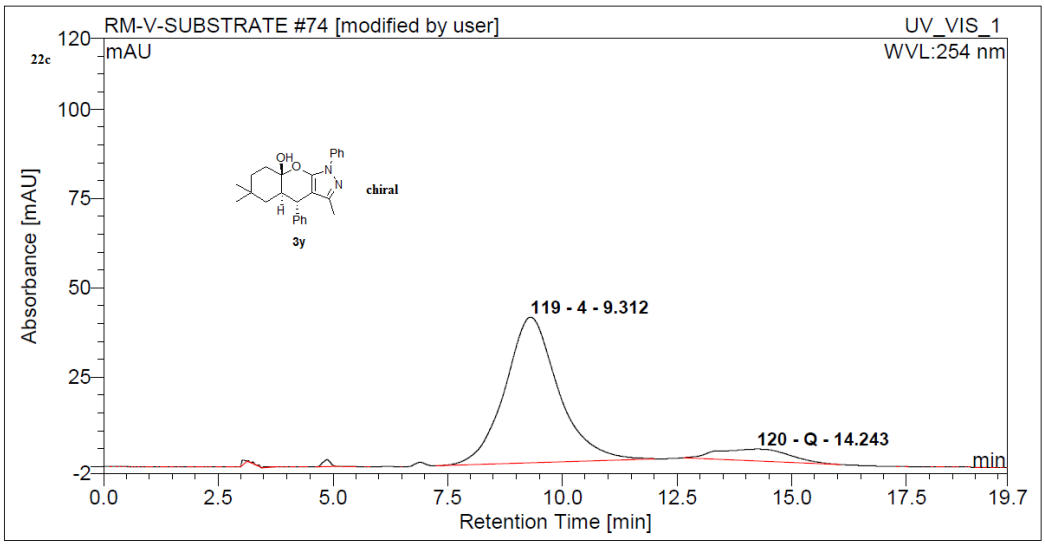


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
7	1	8.81	3.085155	99.47463739	19.82312	n.a.
8	2	11.52	0.016	0.52536261	0.079	n.a.

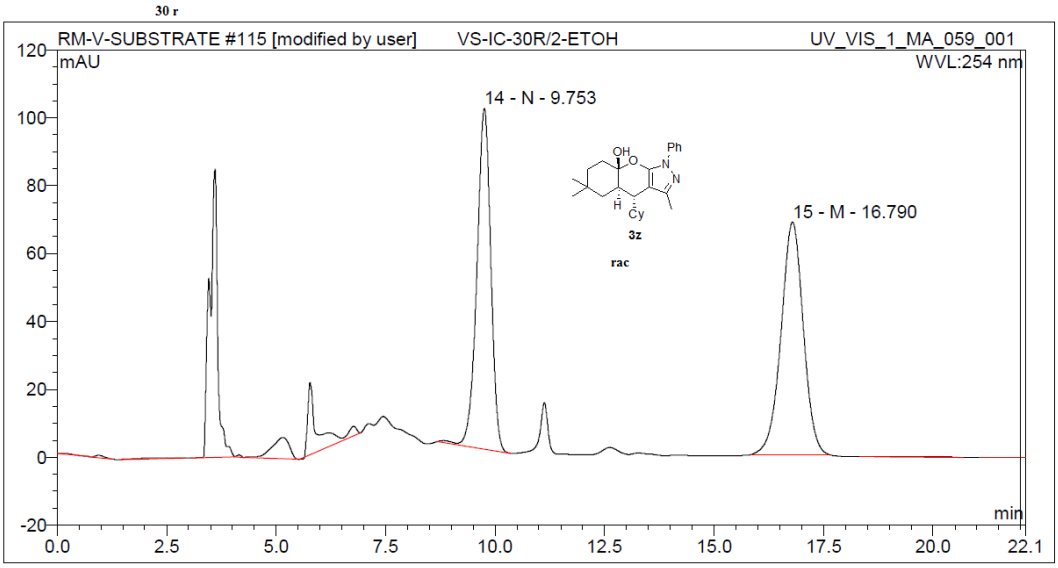
22R



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
52	1	9.31	101.0112	49.23770803	74.89293	n.a.
53	Q	14.26	104.139	50.76229197	42.821	n.a.

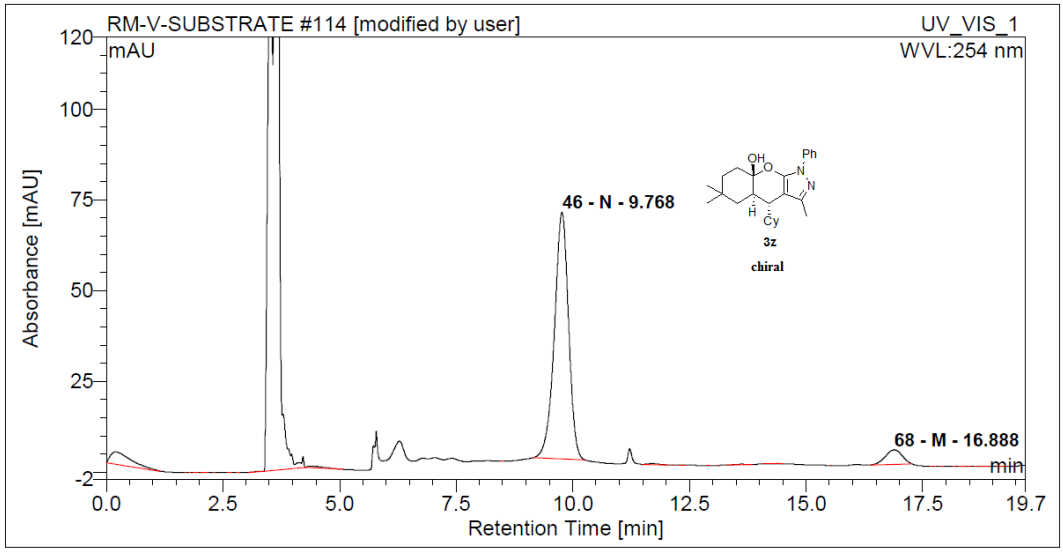


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
119	4	9.31	54.97422	90.18845278	40.83166	n.a.
120	Q	14.24	5.981	9.81154722	3.333	n.a.



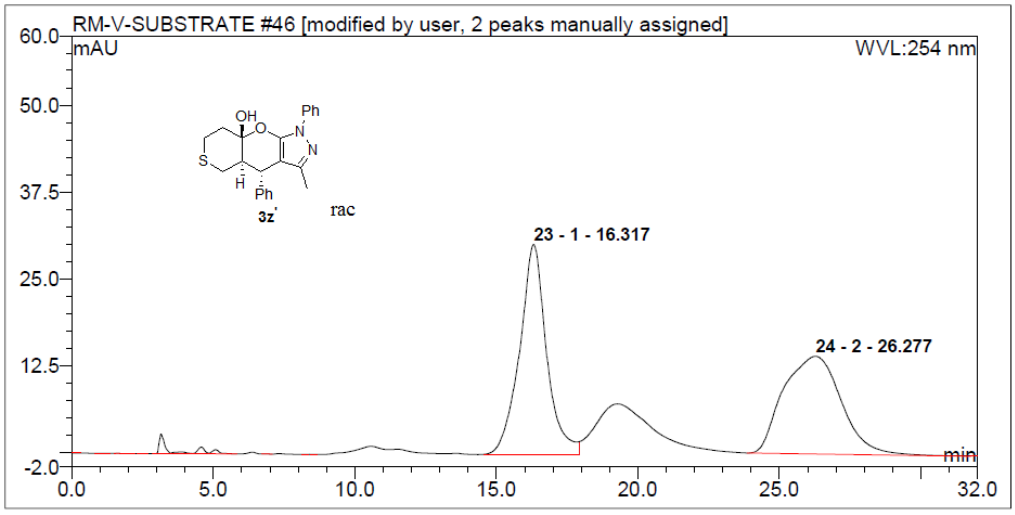
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
14	N	9.75	37.17943	47.95690377	100.4029	n.a.
15	M	16.79	40.347	52.04309623	68.537	n.a.

30 c



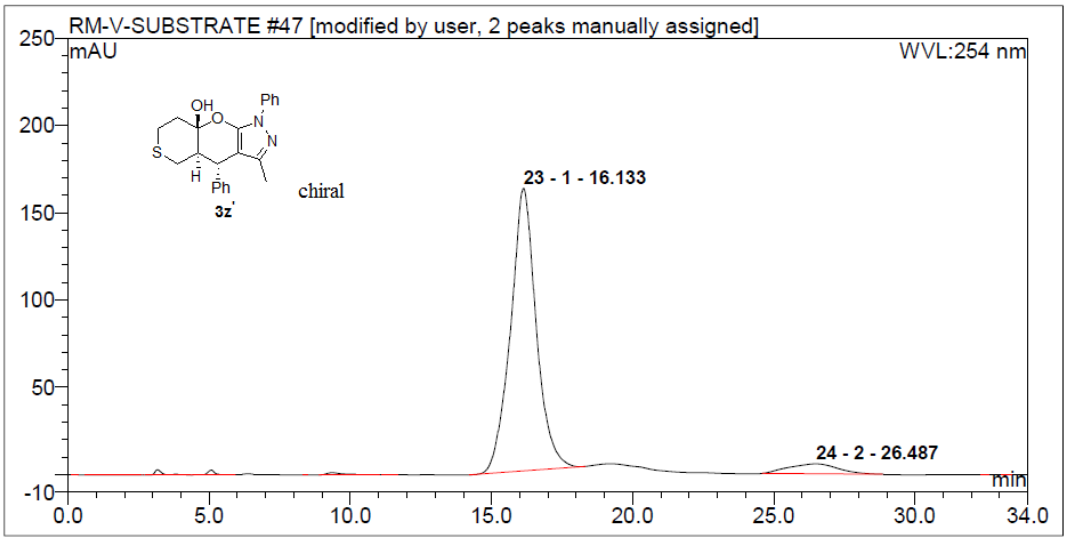
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
46	N	9.77	23.91971	93.65341987	68.0469	n.a.
68	M	16.89	1.621	6.346580128	3.968	n.a.

RM-VS-57R



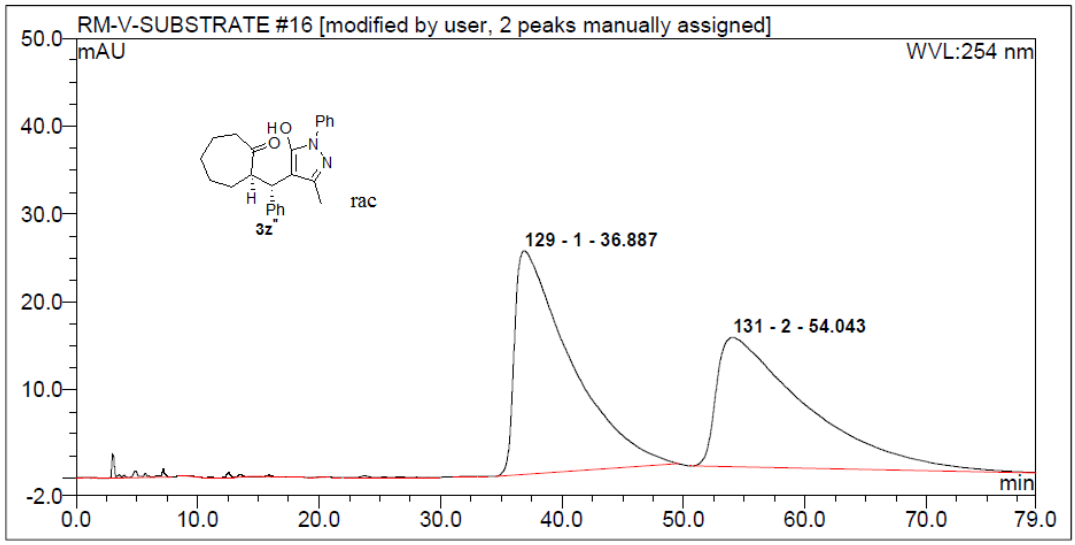
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
23	1	16.32	33.28038	50.06313551	30.1469	n.a.
24	2	26.28	33.196	49.93686449	14.037	n.a.

RM-VS-57C



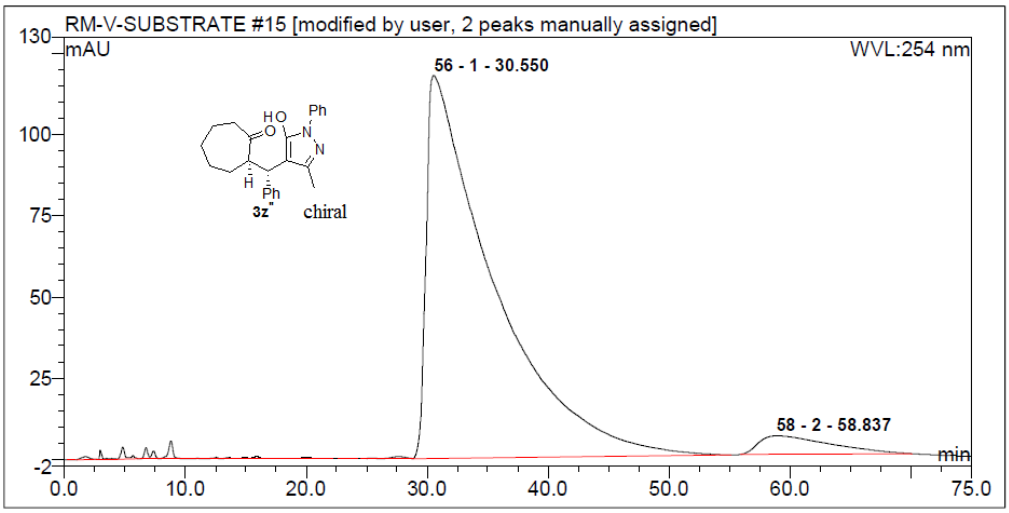
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
23	1	16.13	170.7182	93.63533161	161.7796	n.a.
24	2	26.49	11.604	6.364668394	5.761	n.a.

RM-VS-5R

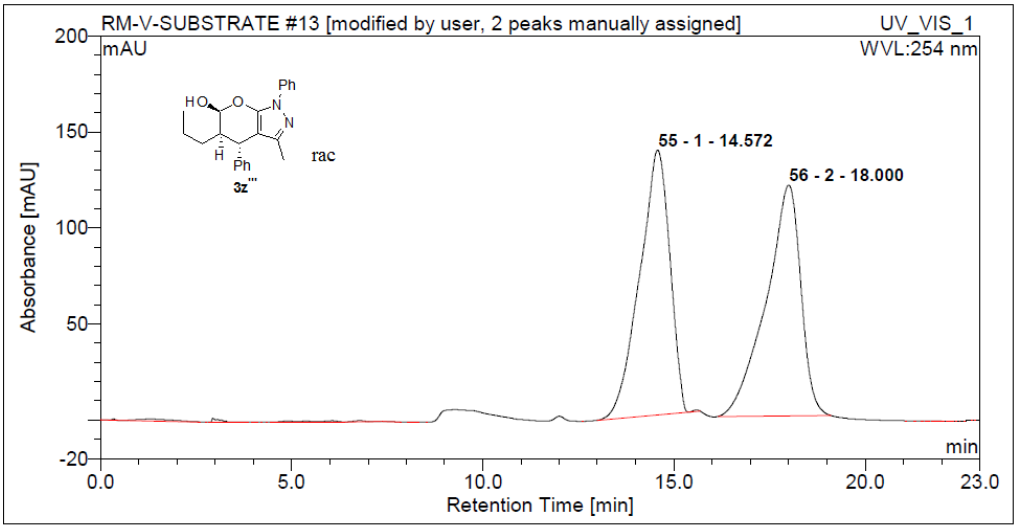


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
129	1	36.89	135.6926	52.00518445	25.42198	n.a.
131	2	54.04	125.229	47.99481555	14.699	n.a.

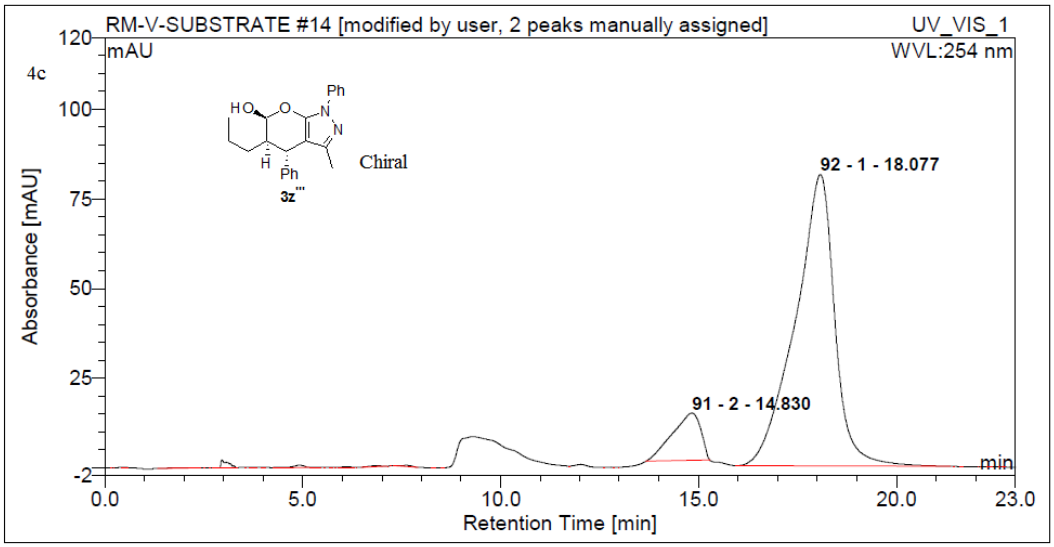
RM-VS-5C



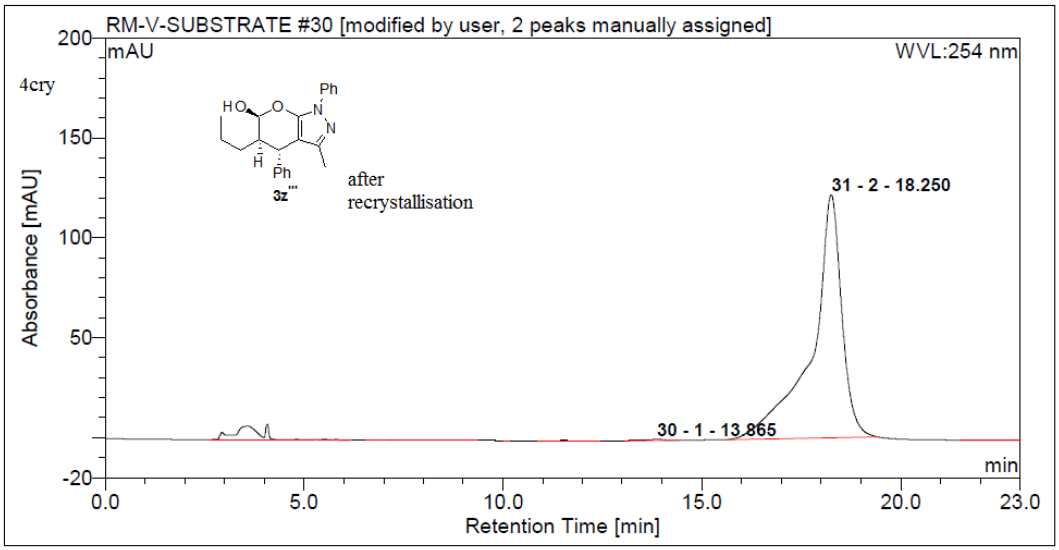
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
56	1	30.55	750.7649	95.11885852	117.705	n.a.
58	2	58.84	38.526	4.881141475	5.706	n.a.



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
55	1	14.57	125.9671	49.95247853	137.7555	n.a.
56	2	18.00	126.207	50.04752147	120.144	n.a.

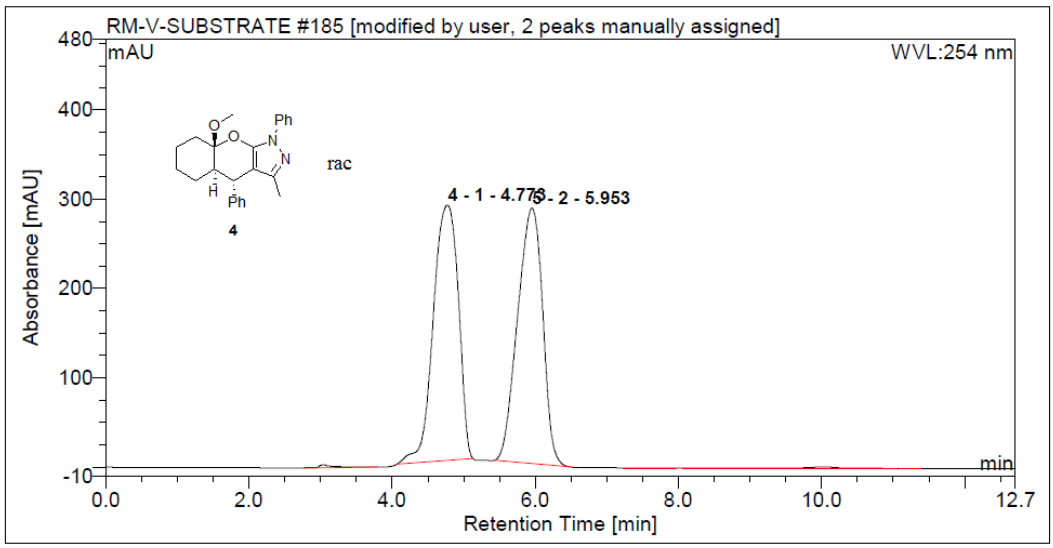


No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
91	2	14.83	10.99359	10.48909078	13.15575	n.a.
92	1	18.08	93.816	89.51090922	81.270	n.a.



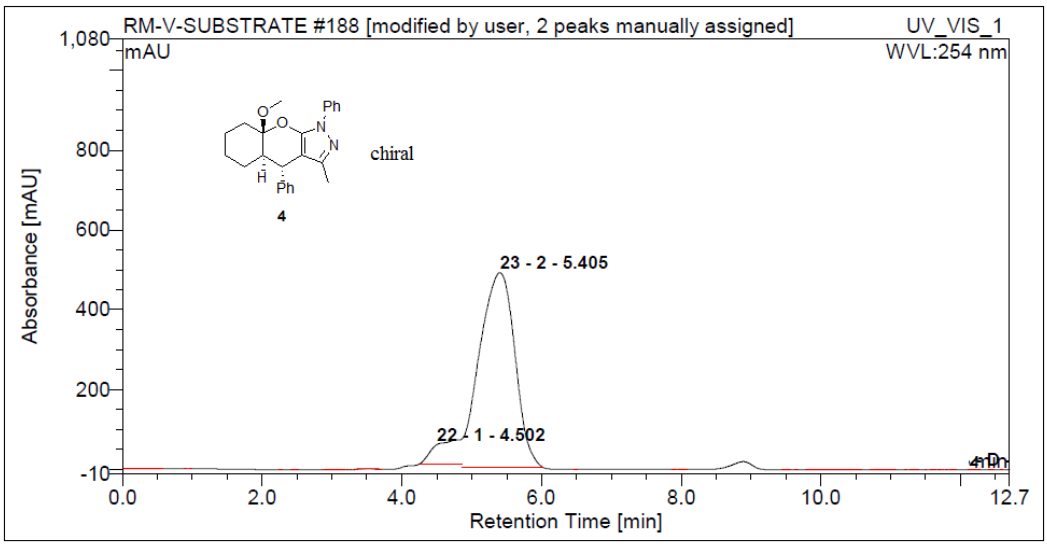
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
30	1	13.87	0.302203	0.2832764455	0.69237	n.a.
31	2	18.25	106.379	99.71672355	121.523	n.a.

v-1-3r



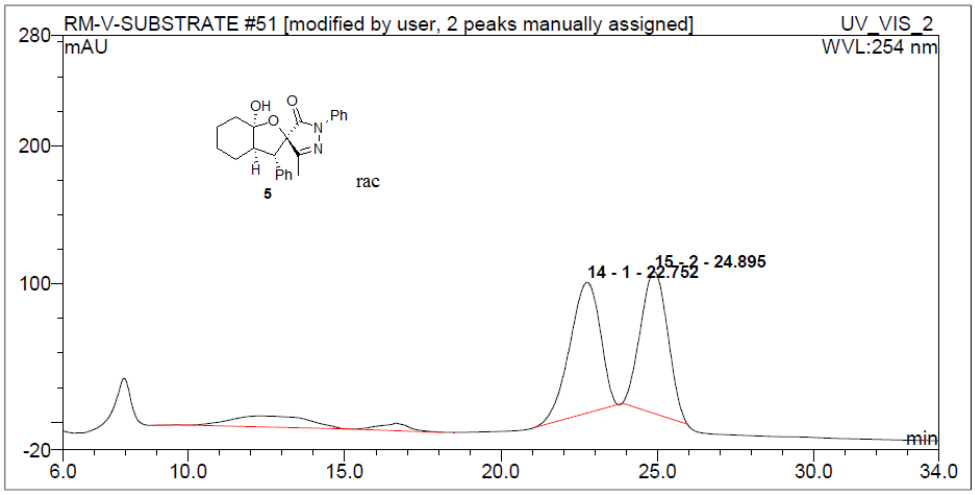
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
4	1	4.77	116.7397	50.06781189	286.4928	n.a.
5	2	5.95	116.423	49.93218811	286.645	n.a.

v-1-3c



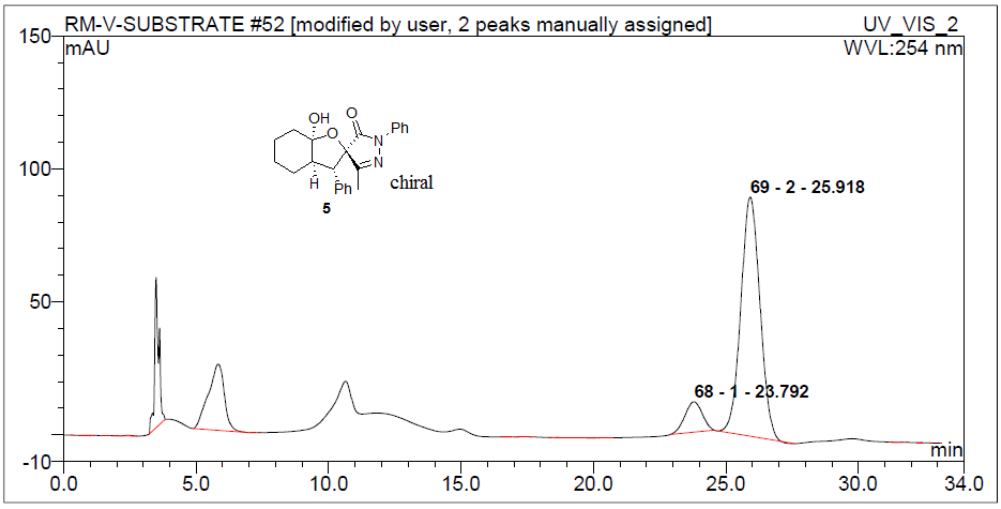
No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
22	1	4.50	26.02767	8.373569699	48.57277	n.a.
23	2	5.41	284.803	91.62635599	487.470	n.a.

RM-V-1-4C



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
14	1	22.75	106.7035	50.7354729	94.36267	n.a.
15	2	24.90	103.610	49.2645271	102.453	n.a.

RM-V-1-4C



No.	Peak Name	Ret.Time (detected) min	Area mAU*min	Rel.Area(ident.) %	Height mAU	Amount
68	1	23.79	8.674532	10.33547803	11.40714	n.a.
69	2	25.92	75.255	89.66452197	89.898	n.a.