Enantioselective synthesis of tetra-substituted tetralines and tetrahydroindolizines by NHC-catalyzed azolium-enolate cascade

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Supporting Information

1. General Information

Unless otherwise specified, all reactions were carried out under an atmosphere of argon in oven-dried reaction vessels with Teflon screw caps. 30 °C Corresponds to the room temperature (rt) of the lab when the experiments were performed. Dry toluene was purchased from commercial sources and stored under argon over sodium wire. The α,β-unsaturated aldehydes **2a, 2b, 2c, 2i, 2u** were purchased from commercial sources and were either distilled (*liquids*) or washed with $NaHCO_3$ (*solids*), prior to use. All other α,β-unsaturated aldehydes were synthesized by following the literature procedure.[1] All the 2-(2-formyl phenyl) aryl ethyl ketone derivatives were prepared following the literature procedure.[2] The triazolium salt **5**[3] was synthesized following the literature procedure. DMAP was purchased from commercial sources and was used without further purification and DBU was distilled prior to use.

Analytical thin layer chromatography was performed on TLC Silica gel 60 F254. Visualization was accomplished with short wave UV light or $KMnO_4$ staining solutions followed by heating. Flash chromatography was performed on silica gel (100-200 mesh) by standard techniques eluting with Pet. Ether-EtOAc solvent system.

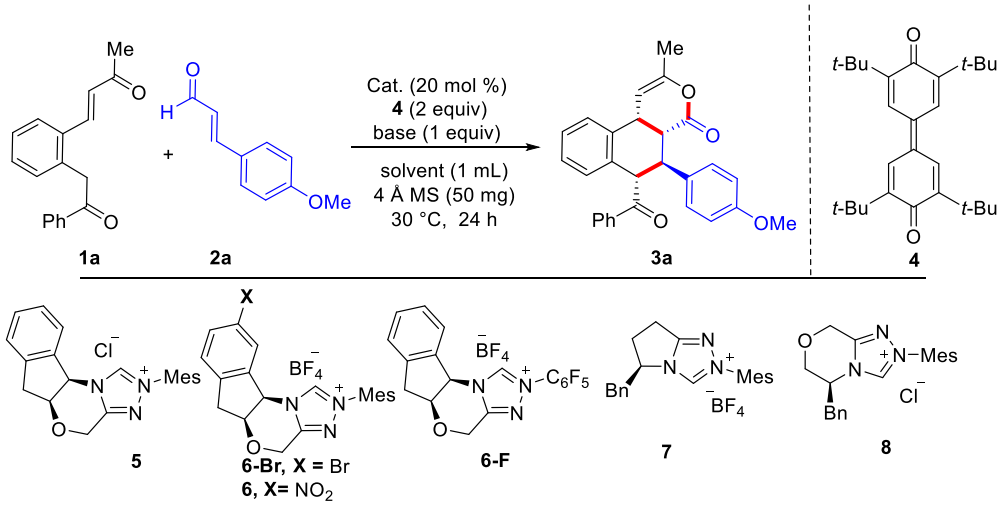
All compounds were fully characterized. ^1H and ^{13}C NMR spectra were recorded on Bruker AV 400 and Bruker Ultra shield spectrometer in solvents as indicated. Chemical shifts (δ) are given in ppm. The residual solvent signals were used as references and the chemical shifts converted to the TMS scale ($CDCl_3$: δH = 7.26 ppm, δC = 77.16 ppm). Infrared (FT-IR) spectra were recorded on a Bruker Alfa FT-IR, ν-max in cm^{-1}. Optical rotations were measured on JASCO P-2000 Polarimeter at room temperature using 50 mm cell of 1.0 mL capacity. HRMS (ESI) data were recorded on a Waters Xevo G2-XS Q-TOF instrument. HPLC analysis was performed on Agilent Technologies 1260 Infinity II with a Variable Wavelength Detector.

[1] A. A. Wubea, A. Hüfner, C. Thomaschitz, M. Blunder, M. Kollroser, Bauer and F. Bucar, *Bioorg. Med. Chem*., 2011, **19**, 567.
[2] S. Zhu, R. Liang, H. Jiang and W. Wu, *Angew. Chem., Int. Ed*., 2012, **51**, 10861.
[3] J. R. Struble and J. W. Bode, *Org. Synth*., 2010, **87**, 362.

2. General Procedure for the Optimization of the Reaction Conditions

2.1 Optimization Studies for Michael-Michael-Lactonization Cascade

To an oven-dried Schlenk reaction vessel equipped with a magnetic stir bar was taken the (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one **1a** (33 mg, 0.125 mmol, 1.0 equiv), triazolium salt (0.025 mmol, 20 mol %), oxidant **4** (102.1 mg, 0.25 mmol, 2.0 equiv), 4 Å MS (50 mg) and (*E*)-3-(4-methoxyphenyl)acrylaldehyde **2a** (40.5 mg, 0.25 mmol, 2.0 equiv). The mixture was kept under argon atmosphere. To this mixture was added the solvent (1.0 mL) under a positive pressure of argon, followed by addition of base (0.125 mmol, 1.0 equiv) and stirring the reaction mixture at 30 °C for 24 h. Filtration of the reaction mixture through a pad of silica gel (and washing using EtOAc), and evaporation of the solvent to obtain the crude products. The yield and diastereomeric ratio were determined by ^{1}H NMR analysis using CH_2Br_2 as the internal standard. The enantiomeric ratio was determined by HPLC analysis on a chiral column.



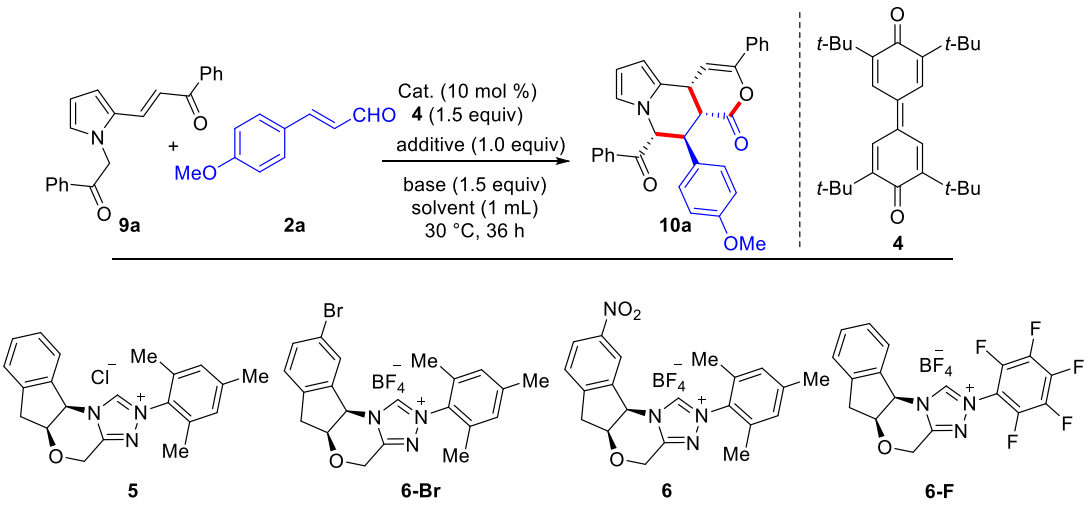
entry[a]	Cat.	base	solvent	additive	yield (%)[b]	dr[b]	er[c]
1	***5***	***DBU***	***$CHCl_3$***	-	***83(80)***	***>20:1***	***97:3***
2	**6-Br**	DBU	$CHCl_3$	-	32	>20:1	85:15
3	**6**	DBU	$CHCl_3$	-	60	>20:1	80:20
4	**6-F**	DBU	$CHCl_3$	-	<5	-	-
5	**7**	DBU	$CHCl_3$	-	65	>20:1	40:60
6	**8**	DBU	$CHCl_3$	-	<5	-	-
7	**5**	DBU	toluene	-	<5	-	-
8	**5**	DBU	THF	-	<5	-	-
9	**5**	DBU	CH_2Cl_2	-	35	>20:1	90:10
10	**5**	DBU	DCE	-	<5	-	-
11	**5**	DMAP	$CHCl_3$	-	43	>20:1	95:5

12	**5**	Et_3N	$CHCl_3$	-	47	>20:1	97:3
13	**5**	DABCO	$CHCl_3$	-	33	>20:1	96:4
14	**5**	K_2CO_3	$CHCl_3$	-	20	>20:1	90:10
15 [d]	**5**	DBU	$CHCl_3$	-	59	>20:1	95:5
16[e]	**5**	DBU	$CHCl_3$	LiOAc	38	>20:1	97:3
17[e]	**5**	DBU	$CHCl_3$	LiCl	14	>20:1	96:4
18[f]	**5**	DBU	$CHCl_3$	-	58	>20:1	97:3

[a] General reaction conditions: **1a** (0.125 mmol), **2a** (2.0 equiv), cat. (20 mol %), base (1.0 equiv), solvent (1.0 mL), 25 °C and 24 h. [b] The yields and dr were determined by ^{1}H NMR analysis of crude product using CH_2Br_2 as the internal standard. [c] The er value was determined by HPLC analysis on a chiral stationary phase. [d] Without 4Å MS. [e] 20 mol % additive was used. [e] 10 mol % of **5** was used.

2.2 Optimization Studies for Tetrahydroindolizines

Under the above optimized reaction conditions (entry 1), the reaction afforded the desired cascade product **10a** in low yield and poor selectivity. At this point, we have started to optimize the reaction conditions by varying all parameters. The detailed studies are given below:



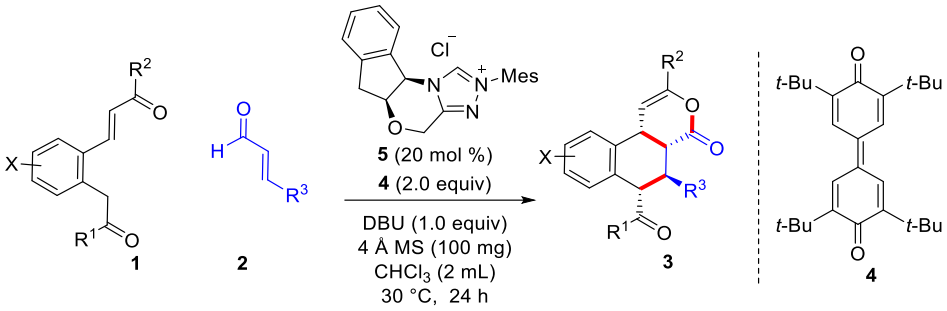
entry	Cat.	base	solvent	additive	yield (%) [b]	dr [b]	er [c]
1	**5**	DABCO	THF	LiCl	29	5:1	99:1
2	**6-Br**	DABCO	THF	LiCl	15	2:1	79:21
3	**6**	DABCO	THF	LiCl	<5	-	-
4	**6-F**	DABCO	THF	LiCl	<5	-	-
5	***5***	*DMAP*	*THF*	*LiCl*	*45 (46)*	*6:1*	*>99:1*
6	**5**	DBU	THF	LiCl	39	1.5:1	>99:1
7	**5**	$^i Pr_2NEt$	THF	LiCl	24	3.5:1	>99:1
8	**5**	Na_2CO_3	THF	LiCl	<5	-	-
9	**5**	DMAP	DME	LiCl	<5	-	-

10	**5**	DMAP	$CHCl_3$	LiCl	<5	-	-
11	**5**	DMAP	toluene	LiCl	<5	-	-
15	**5**	DMAP	THF	-	<5	-	-

[a] General reaction conditions: **9a** (0.1 mmol), **10a** (0.15 mmol), cat. (10 mol %), base (1.5 equiv), LiCl (1.0 equiv), solvent (1.2 mL), 30 °C and 36 h. [b] The yields and dr were determined by ^{1}H NMR analysis of crude product using CH_2Br_2 as the internal standard. [c] The er value was determined by HPLC analysis on a chiral column.

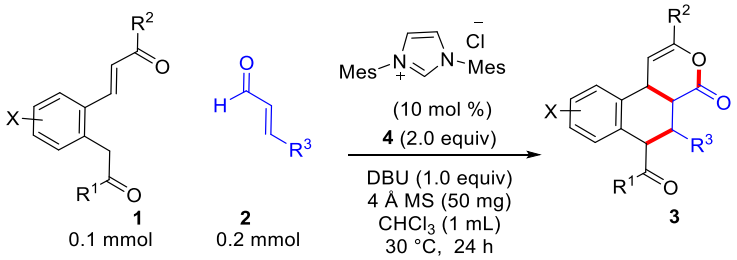
3. General Procedure for the Enantioselective Synthesis of Tricyclic δ-Lactones

Procedure for the Enantioselective Synthesis of Tetrahydro-isochromenone

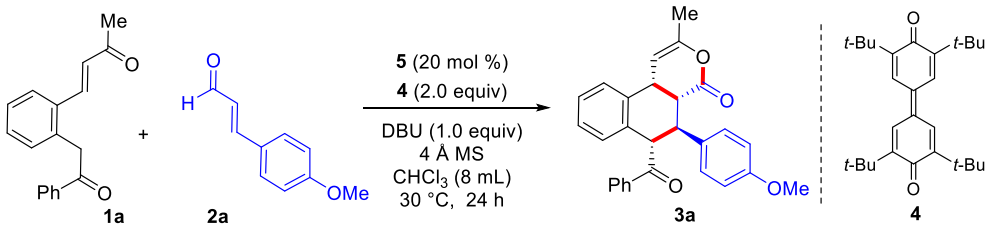


To an oven-dried Schlenk reaction vessel equipped with a magnetic stir bar was taken the (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one **1** (66.1 mg, 0.25 mmol, 1.0 equiv), triazolium salt **5** (18.4 mg, 0.05 mmol, 20 mol %), oxidant **4** (204.3 mg, 0.5 mmol, 2.0 equiv), 4 Å MS (100 mg) and *trans*-cinnamaldehydes **2** (0.5 mmol, 2.0 equiv). The mixture was kept under argon atmosphere. To this mixture was added the $CHCl_3$ (2.0 mL) under a positive pressure of argon, followed by addition of DBU (37.5 µL, 0.25 mmol, 1.0 equiv) and stirring the reaction mixture at 30 °C for 24 h. Then, the reaction mixture was purified using silica gel flash column chromatography to afford the tetrahydro-isochromenone derivatives **3**.

All the racemic tetrahydro-isochromenone derivatives were prepared using *N,N'*-dimesityl imidazolium-derived carbene (IMes).

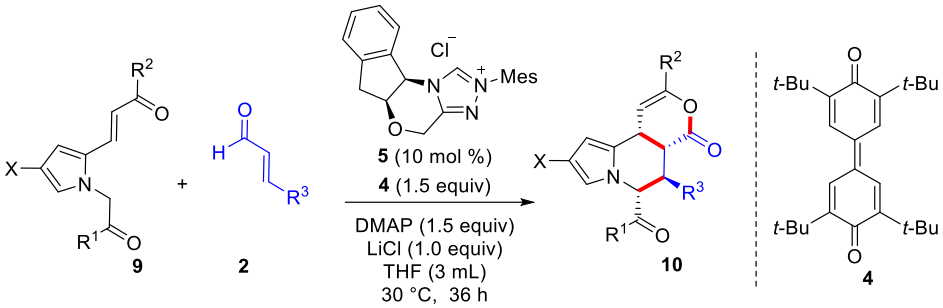


Procedure for the 1 mmol scale experiment



To an oven-dried Schlenk reaction vessel equipped with a magnetic stir bar was taken the (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one **1a** (264.2 mg, 1.0 mmol, 1.0 equiv), triazolium salt **5** (73.6 mg, 0.2 mmol, 20 mol %), oxidant **4** (817.2 mg, 2.0 mmol, 2.0 equiv), 4 Å MS (400 mg) and (*E*)-3-(4-methoxyphenyl)acrylaldehyde **2a** (324.3 mg, 2.0 mmol, 2.0 equiv). The mixture was kept under argon atmosphere. The mixture was kept under argon atmosphere. To this mixture was added the $CHCl_3$ (8.0 mL) under a positive pressure of argon, followed by addition of DBU (149.5 µL, 1.0 mmol, 1.0 equiv) and stirring the reaction mixture at 30 °C for 24 h. the reaction mixture was purified using silica gel flash column chromatography (Pet. ether: EtOAc 95:5 as eluent) to afford the (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3a** as a pale-yellow solid (318 mg, 75% yield, 97:3 er).

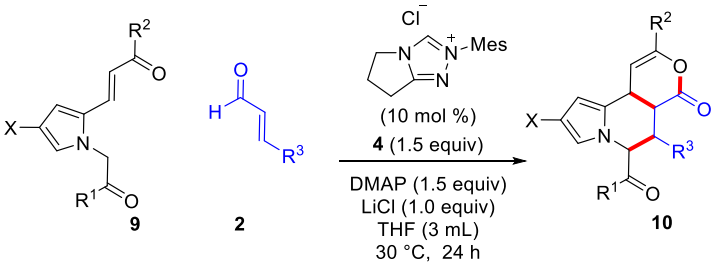
Procedure for the Enantioselective Synthesis of Tetrahydroindolizines



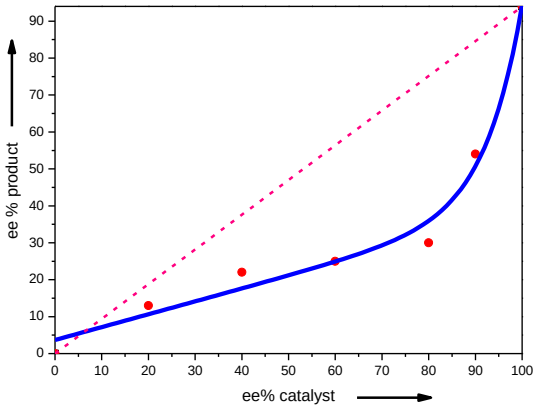
To an oven-dried Schlenk reaction vessel equipped with a magnetic stir bar was taken the (*E*)-3-(1-(2-oxo-2-phenylethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one derivative **1** (0.25 mmol, 1.0 equiv), triazolium salt **5** (9.2 mg, 0.025 mmol, 10 mol %), oxidant **4** (153.2 mg, 0.375 mmol, 1.5 equiv), LiCl (10.6 mg, 0.25 mmol) and *trans*-cinnamaldehydes **2** (0.375 mmol, 1.5 equiv). The mixture was kept under argon atmosphere. To this mixture was added the THF (3.0 mL) under a positive pressure of argon, followed by addition of DMAP (45.8, 0.375 mmol, 1.5 equiv) and stirring the reaction mixture at 30 °C for 36 h. Then, the reaction

mixture was purified using silica gel flash column chromatography to afford the tetrahydro-indolizine derivatives **3**.

All racemic tetrahydro-indolizine derivatives were prepared using *N*-mesityl triazolium-derived carbene.



4. Non-linear effects and Mode of Enantioinduction[4a,b]



ee % catalyst	ee % product **3a**
100	94
90	54
80	30
60	25
40	22
20	13
0	0

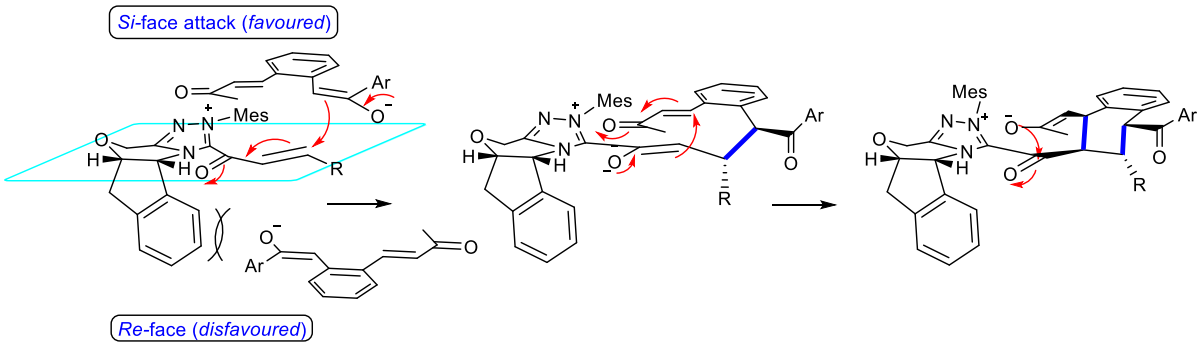
Fig. 1: Nonlinear effects with respect to the product ee and the catalyst ee values

To get insight on the role of NHC catalyst in the cascade process, the reaction of **1a** and **2a** has been conducted under optimized conditions using varying ee values of the triazolium salt **5**. The change in ee values of the product **3a** with the change in ee value of **5** showed a negative non-linear effect. The observation of the negative non-linear effect is an indication that two catalysts are involved in the enantio-determining step of the reaction. Obviously, the NHC is involved in the formation of α,β-unsaturated acylazoliums under oxidative conditions. The negative non-linear effect is an indication of the possible Brønsted base activation of the enones (**1** and **9**) using the NHC derived from **5** under the reaction conditions for the facile Michael addition to catalytically generated α,β-unsaturated acylazoliums.

4. (*a*) D. Guillaneux, S.-H. Zhao, O. Samuel, D. Rainford and H. B. Kagan, *J. Am. Chem. Soc*., 1994, **116**, 9430; (*b*) T. Satyanarayana, S. Abraham and H. B. Kagan, *Angew. Chem., Int. Ed*., 2009, **48**, 456.

Mode of Enantioinduction

The NHC-bound α,β-unsaturated acylazolium has two enantiotopic faces to intercept the in situ generated enolate from arylketone derivative **1**, of them the *re*-face attack is disfavoured due to the presence of bulky chiral indanone fused morpholine core of NHC **5**, while the *si*-face addition is favoured due to less steric interaction. Thus, the stereo-determining *si*-face attack results in the observed stereochemistry in the initial Michael addition step, which directs the second *si*-face Michael addition of the enone appendant by the NHC-bound enolate, followed by a *syn*-selective lactonization to afforded the tricyclic lactone **3**. Moreover, a highly *cis*-selective hetero Diels-Alder reaction can also be invoked for the observed stereochemistry in the lactonization step.



5. X-Ray Data of 3i

X-ray intensity data measurements of compound **3i** was carried out on a Bruker APEX II Ultra diffractometer. The intensity measurements were carried out with Mo rotating anode diffraction source (Mo-K_α= 0.71073 Å) at 100(2) K temperature. The X-ray generator was operated at 45 kV and 80 mA. A preliminary set of cell constants and an orientation matrix were calculated from two matrix sets of 40 frames (each matrix run consists of 20 frames). Data were collected with ω scan width of 0.5° at different settings of φ and 2θ with a frame time of 10 secs keeping the sample-to-detector distance fixed at 5.82 cm. The X-ray data collection was monitored by APEX3 program (Bruker, 2016).[5]All the data were corrected for Lorentzian, polarization and absorption effects using SAINT and SADABS programs (Bruker, 2016). Using the APEX3 (Bruker) program suite, the structure was solved with the ShelXS-97 (Sheldrick, 2008)[6] structure solution program, using direct methods. The model was refined with a version of ShelXL-2018/3 (Sheldrick, 2015)[7] using Least Squares minimization. All

[5] Bruker (2016). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
[6] G. M. Sheldrick, *Acta Crystallogr.* 2008, **A64**, 112.
[7] G. M. Sheldrick, *Acta Crystallogr.* 2015, **C71**, 3.

the hydrogen atoms were placed in a geometrically idealized position and constrained to ride on its parent atoms, An ORTEP III[8] view of the compound was drawn with 50% probability displacement ellipsoids, and H atoms are shown as small spheres of arbitrary radii.

A single crystal of compound **3i** with molecular formula $C_{27}H_{21}ClO_3$, approximate dimensions 0.046 mm x 0.028 mm x 0.25 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured (λ = 0.71073 Å). The integration of the data using a trigonal unit cell yielded a total of 36013 reflections to a maximum θ angle of 30.719° (0.78 Å resolution), of which 12384 were independent (average redundancy 4.468, completeness = 99.2%, R_{int} = 5.82%, R_{sig} = 9.15%) and 8107 were greater than $2\sigma(F^2)$. The final cell constants of a = 8.8163(3) Å, b = 11.2961(4) Å, c =12.0369(4) Å, α = 107.789(2), β = 95.854(2), γ = 110.117(2), volume = 1042.66(6) $Å^3$, are based upon the refinement of the XYZ-centroids of reflections above 20 σ(I). The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.6480 and 0.7480. The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P1, with Z = 2 for the formula unit, $C_{27}H_{21}ClO_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 561 variables converged at R1 = 5.72%, for the observed data and wR2 = 11.41% for all data. The goodness-of-fit was 0.9780. The largest peak in the final difference electron density synthesis was 0.440 $e^-/Å^3$ and the largest hole was -0.480 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.366g/cm^3 and F(000), 448 e^-.

Crystal data of 3i

Crystal Data	**3i**
Formula	$C_{27}H_{21}ClO_3$
Molecular weight	428.1179 g/mol
Crystal Size, mm	0.046 x 0.028 x 0.25
Temp. (K)	100(2)
Wavelength (Å)	0.71073
Crystal Syst.	tetragonal
Space Group	*P1*
a/Å	8.8163(3)
b/Å	11.2961(4)
c/Å	12.0369(4)
α/°	107.789(2)

[8] L. J. Farrugia, *J. Appl. Crystallogr.* 2012, **45**, 849.

β/°	95.854(2)
γ/°	110.117(2)
V/Å^3	1042.66(6)
Z	2
D_{calc}/g cm^{-3}	1.366
μ/mm^{-1}	0.211
F(000)	448
Ab. Correct.	multi-scan
T_{min}/ T_{max}	0.696/0.754
$2\theta_{max}$	61.438
Total reflns.	36013
Unique reflns.	12384
Obs. reflns.	3590
h, k, l (min, max)	(-12, 12), (-16, 16), (-17, 17)
R_{int} / R_{sig}	0.0582/ 0.0915
No. of parameters	561
R1 [$I > 2\sigma(I)$]	0.0572
wR2[$I > 2\sigma(I)$]	0.1141
R1 [all data]	0.1051
wR2 [all data]	0.1353
goodness-of-fit	0.978
$\Delta\rho_{max}$, $\Delta\rho_{min}$(eÅ^{-3})	+0.437, -0.477
CCDC No.	2083530

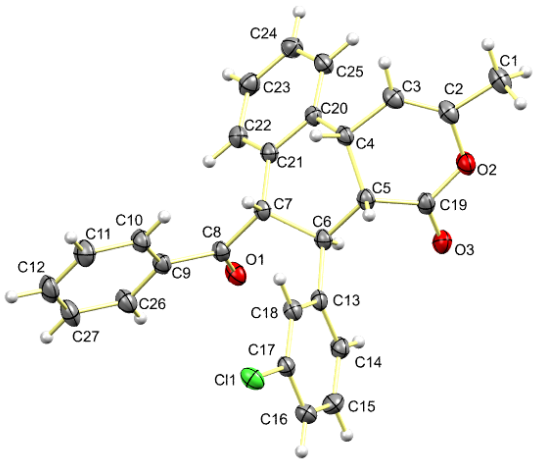
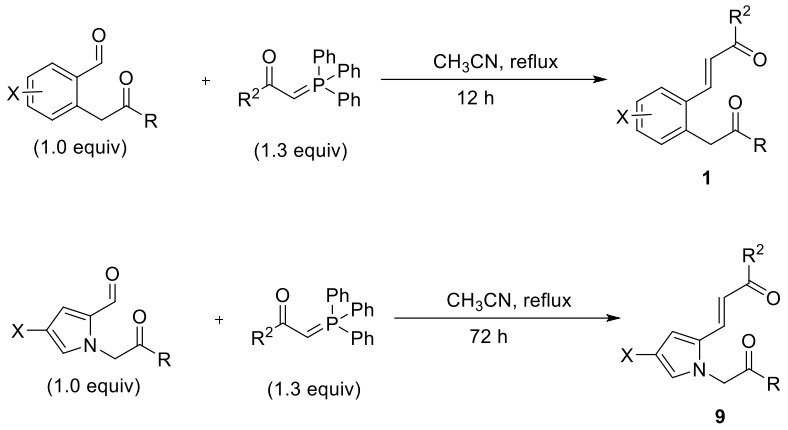


Figure 1. ORTEP Diagram compound 3i showing the atom-numbering scheme, Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres with arbitrary radii.

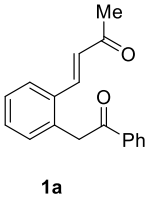
6. Synthesis and Characterization of Michael Acceptors Used

General Procedure for the Synthesis of 1 and 9



The phenacyl enones were synthesized by the following procedure.[2] To a stirred solution of *o*-phenacyl- aldehyde (1.0 equiv) in CH_3CN were added the suitable Wittig salt (1.3 equiv) and reflux for appropriate time. The completion of the reaction was monitored by TLC. Then it was concentrated under reduced pressure and purified by flash column chromatography (eluent: Pet. ether-EtOAc) to afford the desired product **1** and **9**.

(*E*)-4-(2-(2-Oxo-2-phenylethyl)phenyl)but-3-en-2-one (1a)



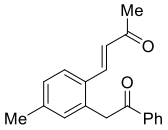
1a

To a stirred solution of 2-(2-oxo-2-phenylethyl)benzaldehyde (1.0 g, 4.5 mmol) and 1-(triphenyl-λ^5-phosphaneylidene)propan-2-one (1.8 g, 5.8 mmol) in CH_3CN (30 mL) were refluxed for 12 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-4-(2-(2-oxo-2-phenylethyl) phenyl)but-3-en-2-one **1a** (540 mg, 45% yield) as a yellow solid.

R_f (Pet. ether /EtOAc = 80/20): 0.42. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.95 (d, *J* = 8.2 Hz, 2H), 7.64-7.51 (m, 3H), 7.44-7.40 (m, 2H), 7.31-7.23 (m, 2H), 7.19-7.16 (m, 1H), 6.56 (d, *J* = 15.9 Hz, 1H), 4.39 (s, 2H), 2.21 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 198.4, 197.0, 140.7, 136.5, 134.7, 134.4, 133.7, 131.6, 130.5, 129.1, 128.9, 128.5, 127.9, 127.1, 43.3, 27.8. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{18}H_{16}O_2Na$ 287.1053; found 287.1048. **FTIR (cm^{-1})** 2923, 2858, 1685, 1599, 1448, 1360, 1329, 1256, 1213.

(*E*)-4-(4-Methyl-2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one (1r)

To a stirred solution of 4-methyl-2-(2-oxo-2-phenylethyl)benzaldehyde (0.59 g, 2.5 mmol) and 1-(triphenyl-λ^5-phosphaneylidene)propan-2-onein (1.1 g, 3.2 mmol) in CH_3CN (15 mL) were

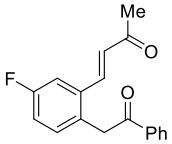


1r

refluxed for 12 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-4-(4-methyl-2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one **1r** (263 mg, 38% yield) as a yellow solid.

R_f (Pet. ether /EtOAc = 80/20): 0.31. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.01 (s, 2H), 7.67-7.47 (m, 5H), 7.12-7.04 (m, 2H), 6.60 (d, *J* = 15.5 Hz, 1H), 4.42 (s, 2H), 2.33-2.24 (m, 6H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 198.3, 197.0, 140.7, 140.5, 136.4, 134.6, 133.5, 132.2, 131.3, 128.8, 128.6, 128.4, 127.9, 126.8, 43.1, 27.5, 21.3. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{19}H_{18}O_2Na$ 301.1199; found 301.1200. **FTIR (cm^{-1})** 2958, 2919, 2858, 1687, 1601, 1449, 1329, 1252, 1211, 1180, 975.

(*E*)-4-(5-Fluoro-2-(2-oxo-2-phenylethyl) phenyl)but-3-en-2-one (1s)

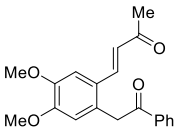


1s

To a stirred solution of 5-fluoro-2-(2-oxo-2-phenylethyl)benzaldehyde (0.61 g, 2.5 mmol) and 1-(triphenyl-λ^5-phosphaneylidene) propan-2-onein (1.1 g, 3.2 mmol) in CH_3CN (15 mL) were refluxed for 12 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-4-(5-fluoro-2-(2-oxo-2-phenylethyl) phenyl)but-3-en-2-one **1s** (282 mg, 40% yield) as a yellow solid.

R_f (Pet. ether /EtOAc = 80/20): 0.34. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.01 (d, *J* = 7.5 Hz, 2H), 7.60-7.47 (m, 4H), 7.31 (d, *J* = 10.1 Hz, 1H), 7.31-7.18 (m, 1H), 7.05 (t, *J* = 7.7 Hz, 1H), 6.60 (d, *J* = 16.1 Hz, 1H), 4.42 (s, 2H), 2.27 (s, 3H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 197.9, 196.7, 162.1 (d, *J* = 247.8 Hz), 139.3 (d, *J* = 2.2 Hz), 136.3, 136.2, 133.7, 133.2 (d, *J* = 8.1 Hz), 130.5 (d, *J* = 2.9 Hz), 129.6, 128.9, 128.4, 117.3 (d, *J* = 21.3 Hz), 113.5 (d, *J* = 22.4 Hz), 42.4, 28.1. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{18}H_{15}FO_2Na$ 305.0948; found 305.0952. **FTIR (cm^{-1})** 2957, 2917, 2855, 1685, 1601, 1445, 1328, 1253, 1210, 1182, 974.

(*E*)-4-(4,5-Dimethoxy-2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one (1t)



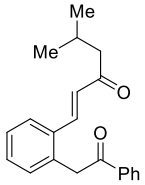
1t

To a stirred solution of 4,5-dimethoxy-2-(2-oxo-2-phenylethyl)benzaldehyde (0.49 g, 1.7 mmol) and 1-(triphenyl-λ^5-phosphaneylidene)propan-2-onein (0.71 g, 2.3 mmol) in CH_3CN (12 mL) were refluxed for 12 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 80:20) to afford the corresponding (*E*)-4-

(4,5-dimethoxy-2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one **1t** (304 mg, 54% yield) as a yellow solid.

R_f (Pet. ether /EtOAc = 80/20): 0.20. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.00 (d, *J* = 7.0 Hz, 2H), 7.62-7.47 (m, 4H), 7.12 (s, 1H), 6.70 (s, 1H), 6.55 (d, *J* = 15.8 Hz, 1H), 4.41 (s, 2H), 3.89-3.85 (m, 6H), 2.24 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 198.2, 197.1, 151.1, 148.4, 140.2, 136.5, 133.6, 128.8, 128.5, 128.4, 126.7, 126.3, 113.9, 109.0, 56.0, 42.6, 27.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{20}H_{20}O_4Na$ 347.1254; found 347.1258. **FTIR (cm^{-1})** 2962, 2932, 2910, 2833, 1688, 1661, 1595, 1514, 1447, 1275, 1253, 1103, 1006.

(*E*)-5-Methyl-1-(2-(2-oxo-2-phenylethyl) phenyl)hex-1-en-3-one (1u)

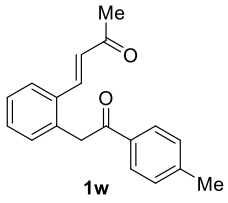


1u

To a stirred solution of 2-(2-oxo-2-phenylethyl) benzaldehyde (0.515 g, 2.3 mmol) and 4-methyl-1-(triphenyl-λ5-phosphaneylidene) pentan-2-one (1.1 g, 7.36 mmol) in CH_3CN (15 mL) were refluxed for 12 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-5-methyl-1-(2-(2-oxo-2-phenylethyl) phenyl)hex-1-en-3-one **1u** (211 mg, 30% yield) as a yellow solid.

R_f (Pet. ether /EtOAc = 80/20): 0.32. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.02 (d, *J* = 7.5 Hz, 2H), 7.71-7.57 (m, 3H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.35-7.30 (m, 2H), 7.24-7.22 (m, 1H), 6.65 (d, *J* = 15.8 Hz, 1H), 4.46 (s, 2H), 2.42 (d, *J* = 7.0 Hz, 2H), 2.20-2.10 (m, 1H), 0.91-0.89 (m, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.3, 196.9, 139.6, 136.5, 134.8, 134.4, 133.6, 131.5, 130.3, 128.9, 128.5, 128.5, 127.8, 126.9, 50.1, 43.1, 25.3, 22.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{21}H_{22}O_2Na$ 329.1512; found 329.1515. **FTIR (cm^{-1})** 2955, 2915, 2856, 1684, 1603, 1445, 1325, 1251, 1211, 1182, 975.

(*E*)-4-(2-(2-Oxo-2-(*p*-tolyl)ethyl)phenyl)but-3-en-2-one (1w)

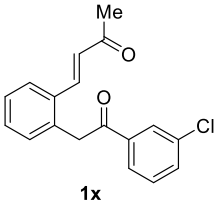


1w

To a stirred solution of 2-(2-oxo-2-(*p*-tolyl)ethyl)benzaldehyde (0.56 g, 2.3 mmol) and 1-(triphenyl-λ^5-phosphaneylidene)propan-2-onein (0.97 g, 3.1 mmol) in CH_3CN (15 mL) were refluxed for 12 h. After completion of the reaction, solvent was evaporated sunder reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-4-(2-(2-oxo-2-(*p*-tolyl)ethyl)phenyl)but-3-en-2-one **1w** (310 mg, 48% yield) as a yellow oil.

***R**f* (Pet. ether /EtOAc = 80/20): 0.31. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.92 (d, *J* = 7.9 Hz, 2H), 7.72-7.63 (m, 2H), 7.36-7.23 (m, 5H), 6.63 (d, *J* = 15.7 Hz, 1H), 4.43 (s, 2H), 2.42 (s, 3H), 2.28 (s, 3H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 198.4, 196.7, 144.5, 140.8, 134.9, 134.4, 134.1, 131.6, 130.5, 129.6, 129.1, 128.7, 127.8, 127.0, 43.2, 27.7, 21.8. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{19}H_{18}O_2Na$ 301.1199; found 301.1205. **FTIR (cm^{-1})** 2919, 2360, 1682, 1603, 1568, 1452, 1360, 1255, 1224, 1179.

(*E*)-4-(2-(2-(3-Chlorophenyl)-2-oxoethyl)phenyl)but-3-en-2-one (1x)

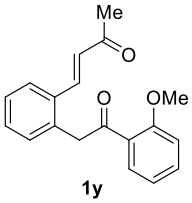


1x

To a stirred solution of 2-(2-(3-chlorophenyl)-2-oxoethyl)benzaldehyde (0.37 g, 1.4 mmol) and 1-(triphenyl-λ^5-phosphaneylidene)propan-2-onein (0.592 g, 1.8 mmol) in CH_3CN (15 mL) were refluxed for 12 h. After completion of the reaction, solvent was evaporated sunder reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-4-(2-(2-(3-chlorophenyl)-2-oxoethyl)phenyl)but-3-en-2-one **1x** (130 mg, 31% yield) as a yellow oil.

***R**f* (Pet. ether /EtOAc = 80/20): 0.45. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.97 (m, 1H), 7.88 (d, *J* = 7.7 Hz, 1H), 7.68-7.49 (m, 3H), 7.44-7.28 (m, 3H), 7.23-7.21 (m, 1H), 6.63 (d, *J* = 15.2 Hz, 1H), 4.42 (s, 2H), 2.29 (s, 3H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 198.1, 195.7, 140.3, 138.0, 135.2, 134.4, 134.1, 133.5, 131.5, 130.5, 130.2, 129.1, 128.6, 128.0, 127.1, 126.6, 43.4, 27.9. **FTIR (cm^{-1})** 2958, 2919, 2858, 1687, 1601, 1449, 1329, 1252, 1211, 1180, 975.

(*E*)-4-(2-(2-(2-Methoxyphenyl)-2-oxoethyl)phenyl)but-3-en-2-one (1y)



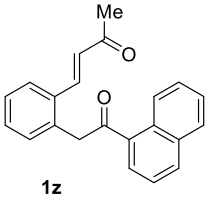
1y

To a stirred solution of 2-(2-(2-methoxyphenyl)-2-oxoethyl)benzaldehyde (0.58 g, 2.3 mmol) and 1-(triphenyl-λ^5-phosphaneylidene)propan-2-onein (0.944 g, 2.9 mmol) in CH_3CN (15 mL) were refluxed for 12 h. After completion of the reaction, solvent was evaporated sunder reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 85:15) to afford the corresponding (*E*)-4-(2-(2-(2-methoxyphenyl)-2-oxoethyl)phenyl)but-3-en-2-one **1y** (322 mg, 47% yield) as a yellow solid.

***R**f* (Pet. ether /EtOAc = 80/20): 0.31. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.70 (d, *J* = 15.9 Hz, 1H), 7.60-7.60 (m, 2H), 7.49-7.45 (m, 1H), 7.43-7.21 (m, 3H), 7.01-6.98 (m, 2H), 6.62 (d, *J* = 15.8 Hz, 1H), 4.49 (s, 2H), 3.96 (s, 3H), 2.28 (s, 3H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 199.6, 198.5, 158.4, 141.1, 135.6, 134.2, 133.9, 131.64 130.6, 130.3, 128.7, 128.1, 127.6, 126.7, 121.0,

111.6, 55.7, 48.2, 27.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{19}H_{18}O_3Na$ 317.1149; found 317.1153. **FTIR (cm^{-1})** 2944, 2916, 2840, 1690, 1670, 1599, 1486, 1360, 1249, 1171.

(*E*)-4-(2-(2-(Naphthalen-1-yl)-2-oxoethyl)phenyl)but-3-en-2-one (1z)

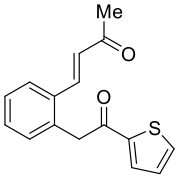


1z

To a stirred solution of 2-(2-(naphthalen-1-yl)-2-oxoethyl)benzaldehyde (0.49 g, 1.8 mmol) and 1-(triphenyl-λ^5-phosphaneylidene)propan-2-onein (0.74 g, 2.3 mmol) in CH_3CN (15 mL) were refluxed for 12 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-4-(2-(2-(naphthalen-1-yl)-2-oxoethyl)phenyl)but-3-en-2-one **1z** (250 mg, 44% yield) as a yellow oil.

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.37. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.45 (d, *J* = 8.8 Hz, 1H), 7.95-7.91 (m, 2H), 7.83-7.81 (m, 1H), 7.72 (d, *J* = 15.9 Hz, 1H), 7.57 (d, *J* = 7.8 Hz, 1H), 7.52-7.44 (m, 3H), 7.33-7.24 (m, 3H), 6.57 (d, *J* = 16.0 Hz, 1H), 4.49 (s, 2H), 2.15 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.8, 198.2, 140.6, 135.3, 134.7, 134.2, 133.9, 132.9, 131.6, 130.4, 130.1, 129.1, 128.5, 128.1, 127.8, 127.6, 126.9, 126.6, 125.5, 124.3, 46.8, 27.3. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{22}H_{18}O_2Na$ 337.1199; found 301.1203. **FTIR (cm^{-1})** 2958, 2923, 2362, 1764, 1673, 1636, 1605, 1511, 1444, 1252, 1151.

(*E*)-4-(2-(2-Oxo-2-(thiophen-2-yl)ethyl) phenyl)but-3-en-2-one (1aa)

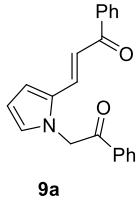


1aa

To a stirred solution of 2-(2-oxo-2-(thiophen-2-yl)ethyl)benzaldehyde (0.57 g, 2.5 mmol) and 1-(triphenyl-λ^5-phosphaneylidene) propan-2-onein (1.1 g, 3.2 mmol) in CH_3CN (15 mL) were refluxed for 12 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-4-(2-(2-oxo-2-(thiophen-2-yl)ethyl) phenyl)but-3-en-2-one **1aa** (216 mg, 32% yield) as a yellow solid.

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.36. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.82-7.78 (m, 2H), 7.68-7.62 (m, 2H), 7.37-7.29 (m, 3H), 7.15 (t, *J* = 4.3 Hz, 1H), 6.62 (d, *J* = 16.0 Hz, 1H), 4.37 (s, 2H), 2.32 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 198.5, 189.7, 143.6, 140.8, 134.5, 134.4, 134.3, 132.7, 131.6, 130.5, 129.2, 128.4, 128.1, 127.1, 44.1, 27.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{16}H_{14}O_2SNa$ 293.0618; found 293.0618. **FTIR (cm^{-1})** 2957, 2855, 1685, 1601, 1445, 1253, 1210, 1182, 974.

(*E*)-3-(1-(2-Oxo-2-phenylethyl)-1H-pyrrol-2-yl)-1-phenylprop-2-en-1-one (9a)

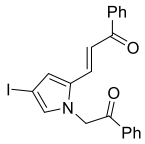


9a

To a stirred solution of 1-(2-oxo-2-phenylethyl)-1*H*-pyrrole-2-carbaldehyde (0.50 g, 2.3 mmol) and 1-phenyl-2-(triphenyl-λ^5-phosphaneylidene)ethan-1-one (1.2 g, 3.0 mmol) in CH_3CN (15 mL) were refluxed for 72 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-3-(1-(2-oxo-2-phenylethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9a** (350 mg, 46% yield) as a yellow solid.

R_f (Pet. ether /EtOAc = 90/10): 0.29. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.00-7.98 (m, 2H), 7.93-7.91 (m, 2H), 7.67-7.50 (m, 5H), 7.44-7.40 (m, 2H), 7.29 (d, *J* = 15.2 Hz, 1H), 6.97-6.96 (m, 1H), 6.87-6.86 (m, 1H), 6.37-6.36 (m, 1H), 5.51 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** 192.4, 189.7, 138.6, 134.4, 134.3, 132.5, 131.7, 130.6, 129.2, 128.9, 128.6, 128.29, 128.2, 128.1, 117.4, 11316, 110.8, 53.2. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{21}H_{17}NO_2Na$ 338.1152; found 338.1156. **FTIR (cm^{-1})** 2923, 2853, 1693, 1592, 1469, 1344, 1290, 1226, 1017.

(*E*)-3-(4-Iodo-1-(2-oxo-2-phenylethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one (9f)

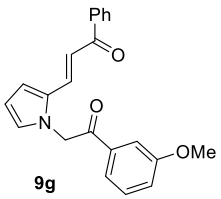


9f

To a stirred solution of 4-iodo-1-(2-oxo-2-phenylethyl)-1*H*-pyrrole-2-carbaldehyde (0.50 g, 1.5 mmol) and 1-phenyl-2-(triphenyl-λ^5-phosphaneylidene)ethan-1-one (0.73 g, 1.9 mmol) in CH_3CN (12 mL) were refluxed for 72 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-3-(4-iodo-1-(2-oxo-2-phenylethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9f** (433 mg, 66% yield) as a yellow solid.

R_f (Pet. ether /EtOAc = 90/10): 0.41. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.97 (d, *J* = 7.1 Hz, 2H), 7.91 (d, *J* = 7.3 Hz, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.54-7.50 (m, 3H), 7.46-7.41 (m, 3H), 7.28 (d, *J* = 14.1 Hz, 1H), 7.00 (s, 1H), 60.87 (d, *J* = 1.2 Hz, 1H), 5.47 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 191.8, 189.4, 139.2, 134.6, 134.1, 132.8, 132.6, 132.0, 130.0, 129.2, 128.7, 128.3, 128.2, 119.2, 118.8, 62.4, 53.0. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{21}H_{16}INO_2Na$ 464.0118; found 464.0125. **FTIR (cm^{-1})** 3126, 3058, 2923, 2853, 1693, 1592, 1469, 1344, 1290, 1226, 1017, 922.

(*E*)-3-(1-(2-(3-Methoxyphenyl)-2-oxoethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one (9g)

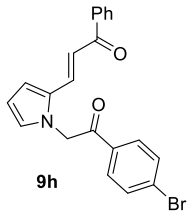


9g

To a stirred solution of 11-(2-(3-methoxyphenyl)-2-oxoethyl)-1*H*-pyrrole-2-carbaldehyde (0.36 g, 1.5 mmol) and 1-phenyl-2-(triphenyl-λ^5-phosphaneylidene) ethan-1-one (0.73 g, 1.9 mmol) in CH_3CN (12 mL) were refluxed for 72 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-3-(1-(2-(3-methoxyphenyl)-2-oxoethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9g** (207 mg, 40% yield) as a yellow solid.

***R*$_f$** (Pet. ether /EtOAc = 90/10): 0.38. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.93-7.91 (m, 2H), 7.60-7.50 (m, 4H), 7.46-7.41 (m, 3H), 7.29 (d, *J* = 15.1 Hz, 1H), 7.21-7.19 (m, 1H), 6.97-6.95 (m, 1H), 6.86-6.85 (m, 1H), 6.37-6.36 (m, 1H), 5.50 (s, 2H), 3.86 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 192.3, 189.8, 160.3, 138.7, 135.7, 132.5, 131.7, 130.7, 130.2, 128.6, 128.3, 128.1, 121.0, 120.6, 117.4, 113.1, 112.5, 110.9, 55.6, 53.4. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{22}H_{19}NO_3Na$ 368.1257; found 368.1261. **FTIR (cm^{-1})** 3125, 3053, 2921, 2850, 1692, 1588, 1465, 1341, 1299, 1224, 10122, 924.

(*E*)-3-(1-(2-(4-Bromophenyl)-2-oxoethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one (9h)



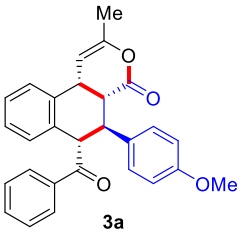
9h

To a stirred solution of 1-(2-(4-bromophenyl)-2-oxoethyl)-1*H*-pyrrole-2-carbaldehyde (0.44 g, 1.5 mmol) and 1-phenyl-2-(triphenyl-λ^5-phosphaneylidene) ethan-1-one (0.73 g, 1.9 mmol) in CH_3CN (12 mL) were refluxed for 72 h. After completion of the reaction, solvent was evaporated under reduced pressure and purified by flash column chromatography (Pet. ether-EtOAc: 90:10) to afford the corresponding (*E*)-3-(1-(2-(4-bromophenyl)-2-oxoethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9h** (354 mg, 60% yield) as a yellow solid.

***R*$_f$** (Pet. ether /EtOAc = 90/10): 0.44. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.92 (d, *J* = 7.1 Hz, 2H), 7.83 (d, *J* = 7.6 Hz, 2H), 7.67-7.65 (m, 2H), 7.57-7.53 (m, 2H), 7.45-7.43 (m, 2H), 7.31-7.26 (m, 1H), 6.95 (s, 1H), 6.85 (s, 1H), 6.36 (s, 1H), 5.47 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 191.7, 189.7, 138.6, 133.0, 132.6, 132.5, 131.5, 130.6, 129.7, 129.6, 128.6, 128.3, 128.0, 117.4, 113.0, 110.9, 53.1. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{21}H_{16}BrNO_2Na$ 416.0257; found 416.0261. **FTIR (cm^{-1})** 3124, 3055, 2925, 2854, 1693, 1590, 1467, 1343, 1289, 1223, 1015, 924.

7. Synthesis and Characterization of Functionalized Tricyclic δ- Lactones

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3a)

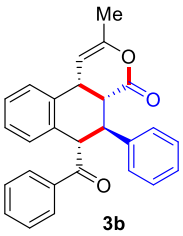


3a

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2a** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo [*f*]isochromen-4-one **3a** as a pale-yellow solid (86 mg, 80% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.37; er = 97:3, $[\alpha]_D^{22}$ = -88.5 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 65:35 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 15.1 min, *Major*: 40.2 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 (d, *J* = 7.3 Hz, 2H), 7.50 (t, *J* = 7.0 Hz, 1H), 7.36 (t, *J* = 7.4 Hz, 2H), 7.27-7.26 (m, 2H), 7.12-7.10 (m, 3H), 6.88 (d, *J* = 7.5 Hz, 1H), 6.73 (d, *J* = 8.3 Hz, 2H), 5.14 (s, 1H), 5.08 (d, *J* = 10.8 Hz, 1H), 4.08- 3.98 (m, 2H), 3.69 (s, 3H), 3.32 (dd, J_1 = 10.1 Hz, J_2 = 6.3 Hz, 1H), 1.95 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.8, 168.6, 158.9, 148.0, 138.1, 135.5, 135.2, 133.3, 131.5, 129.2, 128.7, 128.6, 128.5, 127.9, 127.8, 127.6, 114.2, 104.2, 55.2, 53.2, 45.5, 41.4, 36.2, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{28}H_{24}O_4Na$ 447.1567; found 447.1571. **FTIR (cm^{-1})** 2957, 2925, 2364, 2333, 1755, 1674, 1587, 1513, 1447, 1358, 1252, 1148.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-phenyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3b)



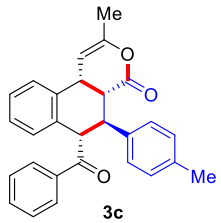
3b

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and *trans*-cinnamaldehyde **2b** (63 µL, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-

benzoyl-2-methyl-5-phenyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one **3b** as a pale-yellow solid (54 mg, 55% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.55; er = >99:1, $[\alpha]_D^{22}$ = -79.5 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 80:20 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 25.2 min, *Major*: 32.9 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 8.1 Hz, 2H), 7.44-7.40 (m, 1H), 7.28 (t, *J* = 7.7 Hz, 2H), 7.21 (d, *J* = 4.4 Hz, 2H), 7.14-7.13 (m, 4H), 7.08-7.04 (m, 2H), 6.81 (d, *J* = 7.7 Hz, 1H), 5.10 (m, 1H), 5.05 (d, *J* = 10.8 Hz, 1H), 4.02- 3.97 (m, 2H), 3.29 (dd, J_1 = 10.2 Hz, J_2 = 6.3 Hz, 1H), 1.88 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.6, 168.6, 148.2, 139.8, 138.1, 135.5, 135.2, 133.3, 128.8, 128.7, 128.6, 128.4, 128.2, 127.9, 127.8, 127.7, 104.0, 52.9, 45.4, 42.2, 36.0, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{27}H_{22}O_3Na$ 417.1461; found 417.1465. **FTIR (cm^{-1})** 2959, 2924, 2364, 2333, 1754, 1675, 1598, 1489, 1449, 1358, 1151.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(*p*-tolyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3c)

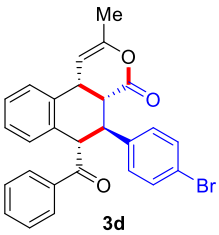


3c

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl) phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(*p*-tolyl) acrylaldehyde **2c** (73.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-2-methyl-5-(*p*-tolyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3c** as a pale-yellow solid (90.2 mg, 85% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.32; er > 99:1, $[\alpha]_D^{22}$ = -152.764 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 80:20 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 24.3 min, *Major*: 42.6 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 7.5 Hz, 2H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.28-7.26 (m, 2H), 7.15-7.09 (m, 3H), 7.02-7.01 (m, 2H), 6.90 (d, *J* = 7.8 Hz, 1H), 5.17 (s, 1H), 5.10 (d, *J* = 10.7 Hz, 1H), 4.08-4.03 (m, 2H), 3.35-3.31 (m, 1H), 2.22 (s, 3H), 1.96 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.6, 168.6, 148.1, 138.1, 137.2, 136.6, 135.5, 135.2, 133.2, 129.5, 128.7, 128.7, 128.4, 127.9, 127.9, 127.8, 127.6, 104.1, 53.1, 45.5, 41.7, 36.1, 21.1, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{28}H_{24}O_3Na$ 431.1618; found 431.1624. **FTIR (cm^{-1})** 3025, 2956, 2921, 2849, 1756, 1675, 1591, 1514, 1448, 1358, 1233, 1149, 976.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-bromophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3d)

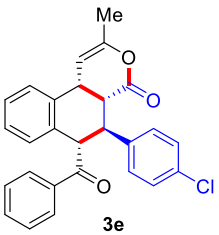


3d

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl) phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(4-bromophenyl) acrylaldehyde **2d** (105.5 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(4-bromophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3d** as a pale-yellow solid (97 mg, 82% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.32; er = 97:3, $[\alpha]_D^{22}$ = -121.132 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 70:30 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 13.8 min, *Major*: 35.7 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 7.9 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.33 (d, *J* = 8.3 Hz, 2H), 7.29-7.26 (m, 2H), 7.15-7.09 (m, 3H), 6.88 (d, *J* = 7.7 Hz, 1H), 5.18 (s, 1H), 5.06 (d, *J* = 10.8 Hz, 1H), 4.13-4.03 (m, 2H), 3.34-3.29 (m, 1H), 1.96 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.1, 168.4, 148.3, 138.9, 137.9, 135.4, 134.9, 133.5, 131.9, 129.9, 128.9, 128.6, 128.3, 127.9, 127.8, 127.7, 121.6, 103.8, 52.7, 45.3, 41.7, 36.0, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{27}H_{21}BrNaO_3$ 495.0566; found 495.0570. **FTIR (cm^{-1})** 3027, 2958, 2923, 2849, 2355, 1753, 1673, 1593, 1485, 1446, 1357, 1232, 1152.

(4a*R*,5S,6*S*,10b*R*)-6-Benzoyl-5-(4-chlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3e)

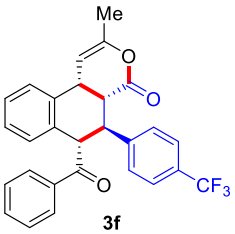


3e

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(4-chlorophenyl)acrylaldehyde **2e** (83.3 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5S,6*S*,10b*R*)-6-benzoyl-5-(4-chlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3e** as a pale-yellow solid (67 mg, 62% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.57; er = 96:4, $[\alpha]_D^{22}$ = -83.0 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralcel ODH, 70:30 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 20.4 min, *Major*: 9.5 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 7.4 Hz, 2H), 7.53 (t, *J* = 7.1 Hz, 1H), 7.39 (t, *J* = 7.4 Hz, 2H), 7.31-7.28 (m, 2H), 7.19-7.12 (m, 5H), 6.88 (d, *J* = 7.4 Hz, 1H), 5.17 (s, 1H), 5.07 (d, *J* = 10.7 Hz, 1H), 4.08- 4.03 (m, 2H), 3.33 (dd, J_1 = 10.2 Hz, J_2 = 6.3 Hz, 1H), 1.96 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.2, 168.4, 148.3, 138.4, 137.9, 135.3, 135.0, 133.5, 133.4, 129.6, 129.0, 128.9, 128.6, 128.4, 127.9, 127.8, 127.7, 103.9, 52.8, 45.4, 41.7, 36.1, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{27}H_{21}ClO_3Na$ 451.1071; found 451.1076. **FTIR (cm^{-1})** 2923, 2365, 2333, 1761, 1678, 1597, 1490, 1446, 1279, 1222, 1152.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(4-(trifluoromethyl)phenyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3f)

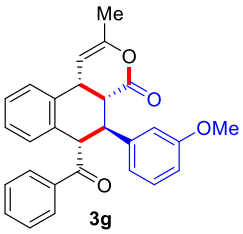


3f

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(4-(trifluoromethyl)phenyl)acrylaldehyde **2f** (100.0 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-2-methyl-5-(4-(trifluoromethyl)phenyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3f** as a white solid (59 mg, 51% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.54; er = 94:6, $[\alpha]_D^{22}$ = -86.7 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralcek ODH, 90:10 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 41.8 min, *Major*: 15.1 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 7.7 Hz, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.47 (d, *J* = 8.2 Hz, 2H), 7.40-7.35 (m, 4H), 7.30-7.29 (m, 2H), 7.17-7.13 (m, 1H), 6.89 (d, *J* = 7.9 Hz, 1H), 5.22 (d, *J* = 2.1 Hz, 1H), 5.12 (d, *J* = 10.6 Hz, 1H), 4.16 (t, *J* = 10.7 Hz, 1H), 4.10- 4.09 (m, 1H), 3.37 (dd, J_1 = 9.9 Hz, J_2 = 6.6 Hz, 1H), 1.97 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.9, 168.3, 148.5, 144.3, 137.9, 135.4, 134.9, 133.6, 129.8 (q, *J* = 32.6 Hz), 128.9, 128.7, 128.6, 128.2, 128.0, 127.8, 127.7, 125.8 (q, *J* = 3.6 Hz), 124.0 (q, *J* = 271.8 Hz), 103.6, 52.5, 45.3, 42.2, 35.9, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{28}H_{21}F_3O_3Na$ 485.1333; found 485.1343. **FTIR (cm^{-1})** 2925, 2364, 2333, 1751, 1674, 1449, 1327, 1159, 1123.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3g)

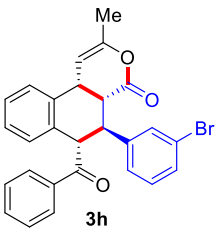


3g

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(3-methoxyphenyl)acrylaldehyde **2g** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(3-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3g** as a pale-yellow solid (81 mg, 76% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.35; er = 96:4, $[\alpha]_D^{22}$ = -126.7 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 60:40 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Major*: 14.9 min, *Minor*: 19.1 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.77 (d, *J* = 7.6 Hz, 2H), 7.51 (t, *J* = 7.1 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 2H), 7.28-7.26 (m, 2H), 7.16-7.12 (m, 2H), 6.89 (d, *J* = 7.8 Hz, 1H), 6.84 (d, *J* = 7.4 Hz, 1H), 6.74 (s, 1H), 6.67 (d, *J* = 8.0 Hz, 1H) 5.19 (s, 1H), 5.12 (d, *J* = 10.5 Hz, 1H), 4.10- 4.05 (m, 2H), 3.69 (s, 3H), 3.33 (dd, J_1 = 9.4 Hz, J_2 = 6.5 Hz, 1H), 1.96 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.4, 168.6, 159.6, 148.3, 141.6, 138.1, 135.6, 135.2, 133.3, 129.8, 128.7, 128.6, 128.2, 127.9, 127.8, 127.6, 120.2, 114.3, 112.7, 103.8, 55.2, 52.5, 45.5, 42.1, 35.8, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{28}H_{24}O_4Na$ 447.1567; found 447.1575. **FTIR (cm^{-1})** 2960, 2924, 2836, 1755, 1674, 1597, 1262, 1232, 1148, 1045, 976.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3-bromophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3h)



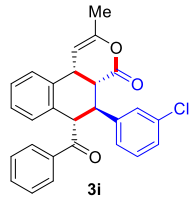
3h

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(3-bromophenyl)acrylaldehyde **2h** (105.5 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(3-bromophenyl)-2-methyl-

4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3h** as a pale-yellow solid (83.1 mg, 71% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.55; er = 93:7, $[\alpha]_D^{22}$ = -112.3 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralcel OD-H, 70:30 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Major*: 10.0 min, *Minor*: 15.7 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 7.8 Hz, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.41-7.36 (m, 3H), 7.29-7.25 (m, 3H), 7.20-7.12 (m, 2H), 7.08 (d, *J* = 7.8 Hz, 1H), 6.88 (d, *J* = 7.7 Hz, 1H), 5.21 (s, 1H), 5.07 (d, *J* = 10.7 Hz, 1H), 4.08- 4.02 (m, 2H), 3.32 (dd, J_1 = 9.8 Hz, J_2 = 6.4 Hz, 1H), 1.97 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.1, 168.3, 148.5, 142.5, 137.9, 135.4, 135.0, 133.5, 131.3, 130.8, 130.4, 128.8, 128.6, 128.1, 127.9, 127.7, 126.9, 122.8, 103.6, 52.5, 45.3, 42.0, 35.8, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{27}H_{21}BrO_3Na$ 495.0566; found 495.0572. **FTIR (cm^{-1})** 3025, 2958, 2922, 2848, 2364, 2333, 1757, 1675, 1447, 1229, 1149, 1074, 975.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3-chlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3i)

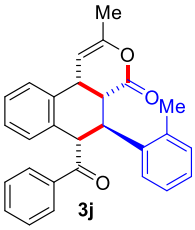


3i

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(3-chlorophenyl)acrylaldehyde **2i** (83.3 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 μL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(3-chlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3i** as a white solid (76 mg, 70% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.57; er = 97:3, $[\alpha]_D^{22}$ = -151.2 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 80:20 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 17.8 min, *Major*: 23.9 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 7.5 Hz, 2H), 7.52 (t, *J* = 7.1 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.29-7.26 (m, 2H), 7.21 (s, 1H), 7.13-7.12 (m, 4H), 6.89 (d, *J* = 7.7 Hz, 1H), 5.20 (s, 1H), 5.07 (d, *J* = 10.8 Hz, 1H), 4.09- 4.04 (m, 2H), 3.33 (dd, J_1 = 9.7 Hz, J_2 = 6.5 Hz, 1H), 1.97 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.1, 168.3, 148.5, 142.3, 138.0, 135.4, 135.1, 134.5, 133.5, 130.1, 128.8, 128.6, 128.4, 128.2, 127.9, 127.9, 127.8, 127.7, 126.4, 103.7, 52.6, 45.4, 42.1, 35.9, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{27}H_{21}ClO_3Na$ 451.1071; found 451.1074. **FTIR (cm^{-1})** 3064, 3024, 2922, 2874, 1759, 1677, 1597, 1447, 1224, 1151, 1049, 977.

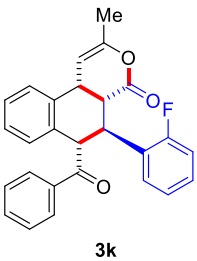
(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(o-tolyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3j)



Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(o-tolyl)acrylaldehyde **2j** (73.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*S*,5*S*,6*S*,10b*R*)-6-benzoyl-2-methyl-5-(o-tolyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one **3j** as a pale-yellow solid (82.1 mg, 80% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.47; er = >99:1, $[\alpha]_D^{22}$ = -209.1 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralcel OD-H, 90:10 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Major*: 19.4 min, *Minor*: 25.9 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 7.4 Hz, 2H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.41-7.29 (m, 5H), 7.17-7.13 (m, 2H), 7.03-6.98 (m, 2H), 6.90 (d, *J* = 7.7 Hz, 1H), 5.20-5.16 (m, 2H), 4.46 (t, *J* = 10.2 Hz, 1H), 4.12-4.10 (m, 1H), 3.33 (dd, J_1 = 10.2 Hz, J_2 = 6.1 Hz, 1H), 2.18 (s, 3H), 1.97 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.8, 168.5, 147.9, 138.1, 137.8, 136.6, 135.5, 135.4, 133.2, 130.9, 128.7, 128.6, 128.5, 127.9, 127.8, 127.6, 127.3, 126.5, 104.6, 53.3, 45.5, 36.4, 19.4, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{28}H_{24}O_3Na$ 431.1618; found 431.1624. **FTIR (cm^{-1})** 3020, 2922, 2852, 1766, 1678, 1491, 1448, 1382, 1448, 1382, 1282, 1221, 1156, 979.

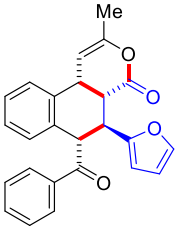
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(2-fluorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3k)



Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(2-fluorophenyl)acrylaldehyde **2k** (75.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(2-fluorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3k** as a white solid (55 mg, 53% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.61; er = 99:1, $[\alpha]_D^{22}$ = -129.3 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 80:20 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 17.0 min, *Major*: 21.5 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.79 (d, *J* = 7.6 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.27-7.25 (m, 2H), 7.19 (t, *J* = 7.2 Hz, 1H), 7.14-7.08 (m, 2H), 6.98-6.86 (m, 3H), 5.35 (d, *J* = 11.2 Hz, 1H), 5.21 (d, *J* = 2.5 Hz, 1H), 4.20 (t, *J* = 9.7 Hz, 1H), 4.11- 4.10 (m, 1H), 3.53 (dd, J_1 = 9.8 Hz, J_2 = 6.3 Hz, 1H), 1.96 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.5, 168.8, 161.4 (d, *J* = 243 Hz), 148.5, 138.07, 135.7 (d, *J* = 8.9 Hz), 133.5, 131.6 (d, *J* = 5 Hz), 129.5 (d, *J* = 9.5), 128.8, 128.5, 128.0, 127.8, 127.6, 127.5, 126.6 (d, *J* = 14.5), 124.5 (d, *J* = 3.6), 116.1, 115.9, 103.6, 50.3, 43.9, 39.1, 35.8, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{27}H_{21}FO_3Na$ 435.1367; found 435.1371. **FTIR (cm^{-1})** 2959, 2924, 2364, 1762, 1680, 1490, 1450, 1279, 1227, 1135.

(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(furan-2-yl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3l)

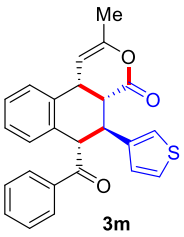


3l

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(furan-2-yl)acrylaldehyde **2l** (61.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*S*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(furan-2-yl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one **3l** as a pale-yellow solid (61 mg, 63% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.52; er = 96:4, $[\alpha]_D^{22}$ = -93.4 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 93:7 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 54.1 min, *Major*: 46.8 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 (d, *J* = 7.4 Hz, 2H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.28-7.25 (m, 3H), 7.14-7.10 (m, 1H), 6.87 (d, *J* = 7.8 Hz, 1H), 6.14-6.13 (m, 1H), 6.05 (d, *J* = 2.9 Hz, 1H), 5.26 (d, *J* = 10.4 Hz, 1H), 5.18 (d, *J* = 2.8 Hz, 1H), 4.20 (t, *J* = 10.0 Hz, 1H), 4.02-4.01 (m, 1H), 3.44 (dd, J_1 = 9.5 Hz, J_2 = 6.4 Hz, 1H), 1.96 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.5, 168.5, 152.7, 148.3, 142.4, 137.8, 135.5, 134.7, 133.5, 128.9, 128.6, 128.1, 127.9, 127.8, 127.6, 110.4, 108.1, 103.8, 49.7, 43.6, 36.0, 35.5, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{25}H_{20}O_4Na$ 407.1254; found 407.1256. **FTIR (cm^{-1})** 2924, 1763, 1681, 1597, 1499, 1447, 1297, 1224, 1150.

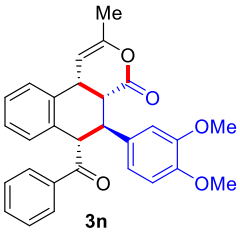
(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(thiophen-3-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3m)



Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl) phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(thiophen-2-yl) acrylaldehyde **2m** (69.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*S*,5*S*,6*S*,10b*R*)-6-benzoyl-2-methyl-5-(thiophen-3-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one **3m** as a pale-yellow solid (84.1 mg, 84% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.33; er = >99:1, $[\alpha]_D^{22}$ = -200.56 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralcell OD-H, 85:15 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Major*: 18.3 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 7.6 Hz, 2H), 7.52 (t, *J* = 7.3 Hz, 1H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.27-7.25 (m, 2H), 7.20.7.18 (m, 1H), 7.16-7.10 (m, 1H), 7.02 (d, *J* = 5.0 Hz, 1H), 6.96 (s, 1H), 6.87 (d, *J* = 7.7 Hz, 1H), 5.16 (s, 1H), 5.05 (d, *J* = 10.5 Hz, 1H), 4.23 (t, *J* = 10.2 Hz, 1H), 4.04-4.03 (m, 1H), 3.33-3.29 (m, 1H), 1.94 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.8, 168.6, 148.1, 140.5, 138.1, 135.5, 134.9, 133.3, 128.8, 128.5, 128.4, 128.0, 127.9, 127.6, 126.6, 126.4, 122.7, 104.0, 52.7, 45.1, 37.6, 35.7, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{25}H_{20}O_3NaS$ 423.1025; found 423.1032. **FTIR (cm^{-1})** 2955, 2925, 2363, 2332, 1754, 1674, 1587, 1513, 1444, 1358, 1252, 1148.

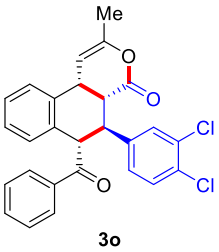
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3,4-dimethoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3n)



Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(3,4-dimethoxyphenyl)acrylaldehyde **2n** (96.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (50 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 70:30) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3,4-dimethoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3n** as a pale-yellow solid (87.2 mg, 76% yield).

***R**f* (Pet. ether /EtOAc = 70/30): 0.35; er = 99:1, $[\alpha]_D^{22}$ = -132.3 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 50:50 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 16.5 min, *Major*: 19.2 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 7.6 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.27-7.26 (m, 2H), 7.15-7.11 (m, 1H), 6.88 (d, *J* = 7.7 Hz, 1H), 6.76-6.73 (m, 1H), 6.69 (d, *J* = 8.3 Hz, 2H), 5.16 (s, 1H), 5.10 (d, *J* = 10.6 Hz, 1H), 4.06- 3.98 (m, 2H), 3.76 (s, 3H), 3.73 (s, 3H), 3.32 (dd, J_1 = 10.2 Hz, J_2 = 6.2 Hz, 1H), 1.95 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.8, 168.6, 148.9, 148.3, 148.1, 138.2, 135.5, 135.3, 133.3, 132.2, 128.8, 128.6, 128.4, 127.9, 127.8, 127.6, 120.2, 111.4, 111.3, 104.1, 55.9, 55.8, 52.9, 45.5, 41.8, 36.1, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{29}H_{26}O_5Na$ 477.1672; found 477.1679. **FTIR (cm^{-1})** 3025, 2961, 2925, 2841, 1757, 1673, 1518, 1447, 1265, 1229, 1146, 1106, 1025, 976.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3,4-dichlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3o)

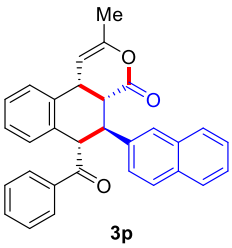


3o

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(3,4-dichlorophenyl)acrylaldehyde **2o** (100.5 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(3,4-dichlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3o** as a pale-yellow solid (51 mg, 45% yield).

***R**f* (Pet. ether /EtOAc = 80/20): 0.5; er = 96:4, $[\alpha]_D^{22}$ = -82.2 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 80:20 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 15.8 min, *Major*: 36.7 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.55 (t, *J* = 7.0 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.30-7.26 (m, 4H), 7.16-7.09 (m, 2H), 6.88 (d, *J* = 8.0 Hz, 1H), 5.20 (s, 1H), 5.03 (d, *J* = 10.8 Hz, 1H), 4.07- 4.02 (m, 2H), 3.33 (dd, J_1 = 9.8 Hz, J_2 = 6.5 Hz, 1H), 1.97 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.7, 168.2, 148.6, 140.6, 137.8, 135.3, 134.8, 133.7, 132.8, 131.7, 130.8, 130.3, 128.9, 128.6, 128.2, 128.1, 127.8, 127.7, 127.6, 103.5, 52.5, 45.3, 41.6, 35.9, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{27}H_{20}Cl_2O_3Na$ 485.0682; found 485.0685. **FTIR (cm^{-1})** 2923, 2365, 1751, 1672, 1590, 1473, 1444, 1352, 1232, 1153.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(naphthalen-2-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3p)

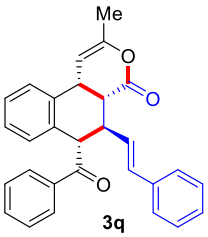


3p

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl)phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (*E*)-3-(naphthalen-2-yl)acrylaldehyde **2p** (91.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-2-methyl-5-(naphthalen-2-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3p** as a white solid (61 mg, 55% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.48; er = 98:2, $[\alpha]_D^{22}$ = -78.3 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 80:20 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 43.5 min, *Major*: 54.6 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.77-7.68 (m, 5H), 7.59 (s, 1H), 7.48-7.37 (m, 4H), 7.33-7.29 (m, 4H), 7.18-7.14 (m, 1H), 6.93 (d, *J* = 7.9 Hz, 1H), 5.27-5.22 (m, 2H), 4.29 (t, *J* = 9.9 Hz, 1H), 4.12-4.11 (m, 1H), 3.45 (dd, J_1 = 10.0 Hz, J_2 = 6.4 Hz, 1H), 1.99 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.3, 168.6, 148.3, 138.0, 137.3, 135.6, 135.2, 133.4, 133.3, 132.8, 128.8, 128.7, 128.6, 128.3, 128.0, 127.9, 127.8, 127.7, 126.2, 125.9, 125.3, 103.9, 52.6, 45.4, 42.2, 35.9, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{31}H_{24}O_3Na$ 467.1618; found 467.1621. **FTIR (cm^{-1})** 2923, 2364, 1754, 1674, 1598, 1446, 1357, 1231, 1148, 1104.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-((*E*)-styryl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3q)



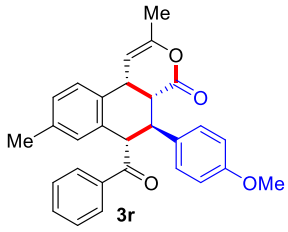
3q

Following the general procedure, (*E*)-4-(2-(2-oxo-2-phenylethyl) phenyl) but-3-en-2-one **1a** (66.1 mg, 0.25 mmol) and (2*E*,4*E*)-5-phenylpenta-2,4-dienal **2q** (79.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-2-methyl-5-((*E*)-styryl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3q** as a pale-yellow solid (68.3 mg, 65% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.31; er >99:1, $[\alpha]_D^{22}$ = -168.64 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 93:7 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 28.8 min, *Major*: 55.1 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 (d, *J* = 7.7 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.44 (t,

J = 7.5 Hz, 2H), 7.27-7.26 (m, 2H), 7.20-7.16 (m, 6H), 6.93 (d, *J* = 7.7 Hz, 1H), 6.31 (d, *J* = 15.7 Hz, 1H), 6.11-6.05 (m, 1H), 5.11 (s, 1H), 4.80 (d, *J* = 10.2 Hz, 1H), 4.08 (s, 1H), 3.61 (q, *J* = 9.9 Hz, 1H), 3.15-3.11 (m, 1H), 1.95 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.7, 168.6, 147.7, 138.2, 136.6, 135.3, 134.2, 133.9, 133.4, 128.9, 128.8, 128.4, 128.3, 128.0, 127.9, 127.7, 127.7, 126.6, 104.6, 52.5, 44.5, 39.9, 35.7, 18.6. **HRMS (ESI)** m/z: $[M+K]^+$ calcd for $C_{29}H_{24}KO_3$ 459.1357; found 459.1360. **FTIR (cm^{-1})** 3609, 2923, 2364, 2332, 1762, 1680, 1598, 1492, 1447, 1350, 1220, 1156.

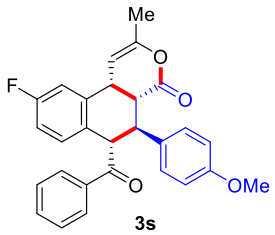
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2,8-dimethyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3r)



Following the general procedure, (*E*)-4-(4-methyl-2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one **1r** (69.6 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2a** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(4-methoxyphenyl)-2,8-dimethyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3r** as a pale-yellow solid (93.3 mg, 85% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.51; er = 99:1, $[\alpha]_D^{22}$ = -209.8 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 60:40 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 12.7 min, *Major*: 37.3 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 (d, *J* = 7.4 Hz, 2H), 7.50 (t, *J* = 7.1 Hz, 1H), 7.37 (t, *J* = 7.2 Hz, 2H), 7.18-7.09 (m, 4H), 6.74-6.71 (m, 3H), 5.14 (s, 1H), 5.03 (d, *J* = 10.4 Hz, 1H), 4.04- 3.96 (m, 2H), 3.69 (s, 3H), 3.30 (dd, J_1 = 9.6 Hz, J_2 = 6.3 Hz, 1H), 2.19 (s, 3H), 1.94 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 202.0, 168.7, 158.8, 147.8, 138.1, 137.3, 134.9, 133.1, 132.4, 131.5, 129.1, 128.7, 128.6, 128.5, 128.3, 114.1, 104.4, 55.1, 53.4, 45.5, 41.5, 35.8, 21.1, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{29}H_{26}O_4Na$ 461.1723; found 461.1729. **FTIR (cm^{-1})** 3017, 2957, 2922, 2837, 1761, 1679, 1511, 1447, 1252, 1154, 1034, 978.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-9-fluoro-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3s)

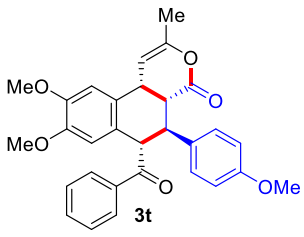


3s

Following the general procedure, (*E*)-4-(5-fluoro-2-(2-oxo-2-phenylethyl) phenyl) but-3-en-2-one **1s** (70.6 mg, 0.25 mmol) and (*E*)-3-(*p*-tolyl) acrylaldehyde **2a** (73.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-9-fluoro-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3s** as a pale-yellow solid (89.6 mg, 81% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.34; er = 98:2, $[\alpha]_D^{22}$ = -162.02 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 70:30 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 16.2 min, *Major*: 22.4 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 (d, *J* = 7.7 Hz, 2H), 7.51 (t, *J* = 7.4 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.12 (d, *J* = 8.3 Hz, 2H), 6.98 (d, *J* = 8.3 Hz, 1H), 6.85 (d, *J* = 6.6 Hz, 2H), 6.74 (d, *J* = 8.5 Hz, 2H), 5.16 (s, 1H), 5.02 (d, *J* = 10.1 Hz, 1H), 4.05-4.00 (m, 2H), 3.69 (s, 3H), 3.31-3.27 (m, 1H), 1.96 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.4, 168.3, 162.0 (d, *J* = 246.9 Hz), 158.9, 148.7, 138.0, 137.9, 137.8, 133.4, 131.6, 130.9 (d, *J* = 2.9 Hz), 129.7 (d, *J* = 8.0 Hz), 129.1, 128.8, 128.6, 114.8 (d, *J* = 21.5 Hz), 114.3, 103.2, 55.2, 52.3, 45.3, 41.4, 35.9, 18.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{28}H_{23}FO_4Na$ 465.1473; found 465.1477. **FTIR (cm^{-1})** 2959, 2927, 2837, 1761, 1678, 1587, 1510, 1447, 1385, 1249, 1148.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-8,9-dimethoxy-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3t)



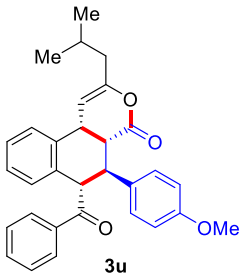
3t

Following the general procedure, (*E*)-4-(4,5-dimethoxy-2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one **1t** (81.1 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2a** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-8,9-

dimethoxy-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3t** as a pale-yellow solid (98.0 mg, 81% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.17; er = 99:1, $[\alpha]_D^{22}$ = -208.1 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 50:50 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 9.1 min, *Major*: 16.1 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.68 (d, *J* = 7.5 Hz, 2H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.36 (t, *J* = 7.7 Hz, 2H), 7.06 (d, *J* = 8.7 Hz, 2H), 6.75-6.72 (m, 3H), 6.34 (s, 1H), 5.13 (s, 1H), 4.93 (d, *J* = 10.2 Hz, 1H), 3.95-3.93 (m, 2H), 3.89 (s, 3H), 3.70 (s, 3H), 3.58 (s, 3H), 3.26 (dd, J_1 = 10.7 Hz, J_2 = 5.8 Hz, 1H), 1.93 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.9, 168.7, 158.9, 148.9, 148.5, 147.8, 138.1, 133.2, 131.2, 129.2, 128.7, 128.6, 127.4, 126.4, 114.2, 111.0, 110.6, 104.4, 56.1, 55.9, 55.2, 53.9, 45.3, 41.2, 35.9, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{30}H_{28}O_6Na$ 507.1778; found 507.1785. **FTIR (cm^{-1})** 3006, 2959, 2925, 2846, 1764, 1680, 1609, 1514, 1445, 1250, 1157, 1034, 974.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-isobutyl-5-(4-methoxyphenyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3u)



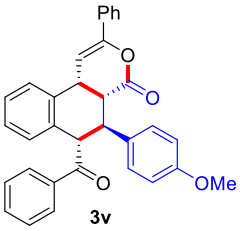
3u

Following the general procedure, ((*E*)-5-methyl-1-(2-(2-oxo-2-phenylethyl) phenyl) hex-1-en-3-one **1u** (76.6 mg, 0.25 mmol) and (*E*)-3-(4-methoxyphenyl) acrylaldehyde **2a** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 μL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-2-isobutyl-5-(4-methoxyphenyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3u** as a pale-yellow solid (87.4 mg, 75% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.35; er = 98:2, $[\alpha]_D^{22}$ = -166.528 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 80:20 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 13.5 min, *Major*: 101.4 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.68 (d, *J* = 7.6 Hz, 2H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.29-7.26 (m, 2H), 7.15-7.08 (m, 3H), 6.90 (d, *J* = 7.9 Hz, 1H), 6.74 (d, *J* = 8.5 Hz, 2H), 5.14-5.13 (m, 1H), 5.02 (d, *J* = 10.9 Hz, 1H), 4.14-4.13 (m, 1H), 4.04 (t, *J* = 10.9 Hz, 1H), 3.7 (s, 3H), 3.36-3.32 (m, 1H), 2.15-2.10 (m, 1H), 2.05-1.90 (m, 2H), 1.0-0.95 (m, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.6, 168.7, 158.9, 150.6, 137.9, 135.4, 135.1, 133.1, 131.2, 129.2, 128.8, 128.7, 127.95, 127.9, 127.7, 114.3, 105.0, 55.2, 54.2, 45.5, 42.0, 41.3,

36.5, 25.6, 22.7, 22.4. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{31}H_{30}O_4Na$ 489.2042; found 489.2043. **FTIR (cm^{-1})** 2957, 2869, 2329, 1753, 1674, 1513, 1454, 1359, 1224, 1147.

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3v)

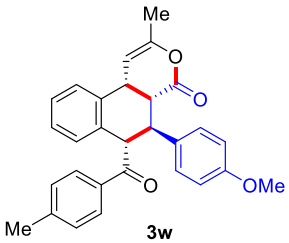


3v

Following the general procedure, (*E*)-3-(2-(2-oxo-2-phenylethyl)phenyl)-1-phenylprop-2-en-1-one **1v** (65.3 mg, 0.20 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2a** (64.8 mg, 0.40 mmol) were treated with the triazolium salt **A** (14.7 mg, 0.04 mmol), oxidant **4** (163.4 mg, 0.40 mmol) and 4 Å MS (80 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (30.4 μL, 0.20 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-benzoyl-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3v** as a pale-yellow solid (50.2 mg, 51% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.37; er = 98:2, $[\alpha]_D^{22}$ = -16.6 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralcel OD-H, 70:30 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Major*: 14.1 min, *Minor*: 24.7 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 7.6 Hz, 2H), 7.66-7.64 (m, 2H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.42-7.30 (m, 7H), 7.17 (t, *J* = 7.2 Hz, 1H), 7.11 (d, *J* = 8.2 Hz, 2H), 6.92 (d, *J* = 7.6 Hz, 1H), 6.74 (d, *J* = 8.5 Hz, 2H), 5.92 (d, *J* = 2.7 Hz, 1H), 5.12 (d, *J* = 10.7 Hz, 1H), 4.32 (dd, J_1 = 5.6 Hz, J_2 = 2.9 Hz, 1H), 4.06 (t, *J* = 10.8 Hz, 1H), 3.70 (s, 3H), 3.45 (dd, J_1 = 10.8 Hz, J_2 = 5.8 Hz, 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.6, 168.2, 158.9, 148.6, 138.0, 135.3, 135.1, 133.3, 132.0, 131.2, 129.4, 129.2, 128.7, 128.6, 128.1, 128.0, 127.9, 124.9, 114.3, 104.4, 55.2, 53.5, 45.6, 41.5, 36.9. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{33}H_{26}O_4Na$ 509.1723; found 509.1728. **FTIR (cm^{-1})** 2958, 2919, 2846, 1767, 1678, 1514, 1459, 1250, 1182, 1079, 1039, 826.

(4a*R*,5*S*,6*S*,10b*R*)-5-(4-Methoxyphenyl)-2-methyl-6-(4-methylbenzoyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3w)



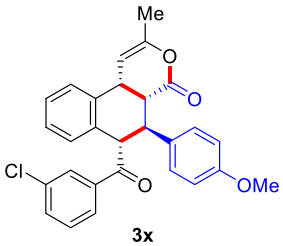
3w

Following the general procedure, (*E*)-4-(2-(2-oxo-2-(p-tolyl)ethyl)phenyl)but-3-en-2-one **1w** (69.6 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2a** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30

°C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-5-(4-methoxyphenyl)-2-methyl-6-(4-methylbenzoyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one e **3w** as a pale-yellow solid (90 mg, 82% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.44; er = 99:1, $[\alpha]_D^{22}$ = -145.1 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralcel ODH, 70:30 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 18.9 min, *Major*: 10.9 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.65 (d, *J* = 7.9 Hz, 2H), 7.25 (d, *J* = 3.6 Hz, 2H), 7.16-7.07 (m, 5H), 6.86 (d, *J* = 7.7 Hz, 1H), 6.72 (d, *J* = 8.2 Hz, 2H), 5.13 (s, 1H), 5.06 (d, *J* = 10.8 Hz, 1H), 4.08- 3.99 (m, 2H), 3.67 (s, 3H), 3.31 (dd, J_1 = 10.3 Hz, J_2 = 6.0 Hz, 1H), 2.35 (s, 3H), 1.94 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.1, 168.6, 158.7, 147.9, 144.2, 135.6, 135.4, 131.6, 129.4, 129.1, 128.8, 128.4, 127.8, 127.7, 127.5, 114.1, 104.2, 55.1, 52.9, 45.5, 41.2, 36.2, 21.7, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{29}H_{26}O_4Na$ 461.1723; found 461.1728. **FTIR (cm^{-1})** 2957, 2925, 1759, 1673, 1606, 1512, 1251, 1180, 1150.

(4a*R*,5*S*,6*S*,10b*R*)-6-(3-Chlorobenzoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3x)



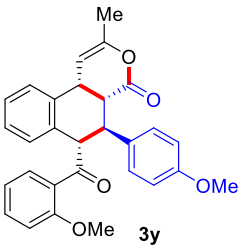
3x

Following the general procedure, (*E*)-4-(2-(2-(3-chlorophenyl)-2-oxoethyl)phenyl)but-3-en-2-one **1x** (74.7 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2a** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-(3-chlorobenzoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3x** as a pale-yellow solid (60 mg, 52% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.4; er = >99:1, $[\alpha]_D^{22}$ = -130.2 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 80:20 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 27.4 min, *Major*: 65.6 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.66 (s, 1H), 7.58 (d, *J* = 7.7 Hz, 1H), 7.47 (t, *J* = 7.3 Hz, 1H), 7.32-7.26 (m, 3H), 7.15-7.09 (m, 3H), 6.86 (d, *J* = 7.7 Hz, 1H), 6.75 (d, *J* = 8.3 Hz, 2H), 5.16 (s, 1H), 4.98 (d, *J* = 10.6 Hz, 1H), 4.08 (s, 1H), 3.97 (t, *J*= 10.5 Hz, 1H), 3.71 (s, 3H), 3.32 (dd, J_1 = 9.9 Hz, J_2 = 6.4 Hz, 1H), 1.95 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.5, 168.4, 159.0, 148.2, 139.4, 135.5, 134.9, 134.7, 133.1, 131.3, 130.1, 129.1, 128.7, 128.6, 128.0,

127.9, 127.7, 126.7, 114.3, 104.1, 45.4, 41.5, 36.2, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{28}H_{23}ClO_4Na$ 481.1177; found 481.1182. **FTIR (cm^{-1})** 2925, 2356, 1763, 1684, 1610, 1567, 1513, 1251, 1180, 1152.

(4a*R*,5*S*,6*S*,10b*R*)-6-(2-Methoxybenzoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3y)



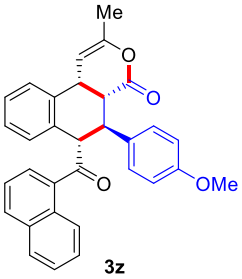
3y

Following the general procedure, (*E*)-4-(2-(2-(2-methoxyphenyl)-2-oxoethyl)phenyl)but-3-en-2-one **1y** (73.6 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2a** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6S,10b*R*)-6-(2-methoxybenzoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one **3y** as a white solid (65 mg, 57% yield).

***R*$_f$** (Pet. ether /EtOAc = 80/20): 0.37; er = 99:1, $[\alpha]_D^{22}$ = -115.9 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 70:30 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 15.3 min, *Major*: 25.3 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.76-7.32 (m, 1H), 7.28-7.22 (m, 2H), 7.19-7.12 (m, 2H), 7.10 (d, *J* = 7.7 Hz, 1H), 7.04 (d, *J* = 8.4 Hz, 2H), 7.84-7.78 (m, 2H), 6.67 (d, *J* = 8.4 Hz, 1H), 5.32 (d, *J* = 10.0 Hz, 1H), 5.12 (s, 1H), 3.98- 3.97 (m, 2H), 3.87- 3.82 (m, 1H), 3.69 (s, 3H), 3.68 (s, 3H), 3.22 (dd, J_1 = 10.4 Hz, J_2 = 6.6 Hz, 1H), 1.92 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 204.5, 168.8, 158.7, 157.8, 147.6, 135.6, 133.4, 132.0, 130.7, 129.6, 129.2, 128.8, 128.2, 127.4, 127.2, 120.7, 113.8, 111.3, 104.5, 56.5, 55.4, 55.2, 45.7, 41.8 35.8, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{29}H_{26}O_5Na$ 477.1672; found 477.1674. **FTIR (cm^{-1})** 2923, 2364, 1764, 1673, 1599, 1512, 1485, 1284, 1249, 1178, 1155.

(4a*R*,5*S*,6*S*,10b*R*)-6-(1-Naphthoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3z)

Following the general procedure, (*E*)-4-(2-(2-(naphthalen-1-yl)-2-oxoethyl)phenyl)but-3-en-2-one **1z** (78.6 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2a** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the

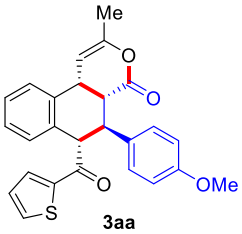


3z

reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*R*,5*S*,6*S*,10b*R*)-6-(1-naphthoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro -4*H*-benzo[*f*]isochromen-4-one **3z** as a white solid (87 mg, 73% yield).

***R**f* (Pet. ether /EtOAc = 80/20): 0.4; er = 99:1, $[\alpha]_D^{22}$ = -181.2 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 75:25 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 20.7 min, *Major*: 39.4 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.14 (d, *J* = 8.1 Hz, 1H), 7.92 (t, *J* = 8.1 Hz, 1H), 7.82 (t, *J* = 7.3 Hz, 1H), 7.50-7.43 (m, 3H), 7.36-7.29 (m, 3H), 7.16-7.12 (m, 3H), 7.02 (d, *J* = 7.8 Hz, 1H), 6.69 (d, *J* = 8.9 Hz, 2H), 5.15 (s, 1H), 5.10 (d, *J* = 11.1 Hz, 1H), 4.10- 4.04 (m, 2H), 3.66 (s, 3H), 3.35 (dd, J_1 = 10.6 Hz, J_2 = 5.8 Hz, 1H), 1.96 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 203.3, 167.6, 157.9, 146.9, 136.3, 134.5, 134.2, 132.9, 131.9, 130.4, 129.1, 128.5, 127.8, 127.3, 126.9, 126.8, 126.7, 126.6, 125.6, 124.9, 123.2, 113.3, 103.3, 55.9, 54.2, 44.2, 40.6, 35.5, 17.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{32}H_{26}O_4Na$ 497.1723; found 497.1728. **FTIR (cm^{-1})** 2361, 2328, 1687, 1671, 1641, 1593, 1575, 1424, 1360, 1220, 1173.

(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(thiophen-2-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3aa)



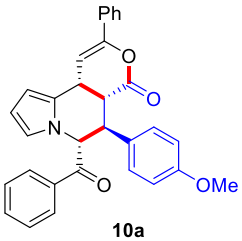
3aa

Following the general procedure, (*E*)-4-(2-(2-oxo-2-(thiophen-2-yl) ethyl)phenyl)but-3-en-2-one **1aa** (67.5 mg, 0.25 mmol) and (*E*)-3-(4-methoxyphenyl)acrylaldehyde **2a** (81.1 mg, 0.50 mmol) were treated with the triazolium salt **5** (18.4 mg, 0.05 mmol), oxidant **4** (204.3 mg, 0.50 mmol) and 4 Å MS (100 mg) in $CHCl_3$ (2 mL) at 30 °C. To this stirring solution at 30 °C was added DBU (37.3 µL, 0.25 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 80:20) to afford (4a*S*,5*S*,6*S*,10b*R*)-6-benzoyl-2-methyl-5-(thiophen-2-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one **3aa** as a pale-yellow solid (91.4 mg, 85% yield).

***R**f* (Pet. ether /EtOAc = 80/20): 0.32; er = > 99:1, $[\alpha]_D^{22}$ = -15.68 (*c* 0.5, $CHCl_3$). **HPLC** (Chiralpak AD, 70:30 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm)*Major*: 42.9 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.58 (d, *J* = 4.5 Hz, 2H), 7.51 (t, *J* = 3.5 Hz, 1H), 7.27-7.26 (m, 2H), 7.15 (d, *J* = 8.4 Hz, 3H), 7.02 (t, *J* = 4.1 Hz, 1H), 6.96 (d, *J* = 7.9 Hz, 1H), 6.75 (d, *J* = 8.4 Hz, 1H), 5.11 (s, 1H), 4.88 (d, *J* = 10.9 Hz, 1H), 4.09 (s, 1H), 3.97 (t, *J* = 11.1 Hz, 1H), 3.70 (s, 3H), 3.35-3.31 (m, 1H), 1.94 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 193.8, 168.5, 158.9, 147.9,

145.3, 135.5, 134.8, 132.9, 131.1, 129.3, 128.6, 128.4, 128.1, 127.9, 127.7, 114.2, 104.4, 55.4, 55.2, 45.4, 41.5, 36.5, 18.6. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{26}H_{22}NaO_4$ 453.1131; found 453.1135. **FTIR (cm^{-1})** 3069, 2957, 2922, 2836, 2332, 1753, 1650, 1512, 1450, 1410, 1355, 1249, 1104, 971.

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10a)

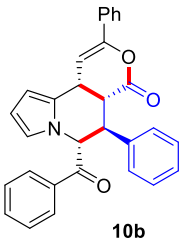


10a

Following the general procedure, (*E*)-3-(1-(2-oxo-2-phenylethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9a** (79.1 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2a** (60.8 mg, 0.375 mmol) were treated with the triazolium salt **5** (9.2 mg, 0.025 mmol), oxidant **4** (153.2 mg, 0.50 mmol) and LiCl (10.6 mg, 0.25 mmol) in THF (3 mL) at 30 °C. To this stirring solution at 30 °C was added DMAP (45.8, 0.375 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 90:10) to afford (4a*R*,5*S*,6*R*,10b*R*)-6-benzoyl-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one **10a** as a white solid (53 mg, 45% yield, 5:1 dr).

R_f (Pet. ether /EtOAc = 85/15): 0.25; Major diastereomer, er = >99:1, Minor diastereomer, er = >99:1. $[\alpha]_D^{22}$ = +128.4 (c 1, $CHCl_3$). HPLC (Chiralpak AD, 70:30 Hexane / IPA, 0.7 mL/min, 254 nm) Major diastereomer: Minor: 31.2 min, Major: 17.6 min, Minor diastereomer: Major: 23.9 min, Minor: 70.2 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81 (d, *J* = 7.5 Hz, 2H), 7.64-7.60 (m, 3H), 7.48-7.44 (m, 2H), 7.38-7.35 (m, 3H), 7.19 (d, *J* = 8.8 Hz, 2H), 6.87 (d, *J* = 8.8 Hz, 2H), 6.62 (m, 1H), 6.33 (d, *J* = 2.1 Hz, 2H), 6.28 (d, *J* = 2.2 Hz, 1H), 5.74 (d, *J* = 3.6 Hz, 1H), 4.07-4.00 (m, 2H), 3.80 (s, 3H), 2.92 (dd, J_1 = 14.4 Hz, J_2 = 9.7 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.8, 168.8, 159.0, 150.4, 135.9, 134.3, 130.9, 129.4, 129.1, 129.0, 128.9, 128.7, 128.6, 124.8, 119.9, 114.6, 109.4, 103.9, 102.8, 65.9, 55.4, 48.4, 42.8, 31.9. **Representative peaks for minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.96 (d, *J* = 7.5 Hz, 2H), 7.55-7.50 (m, 2H), 7.42-7.39 (m, 2H), 7.28 (d, *J* = 8.8 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.67-6.75 (m, 2H), 6.54 (m, 1H), 6.42 (d, *J* = 3.2 Hz, 1H), 6.23 (d, *J* = 1.9 Hz, 1H), 6.18 (d, *J* = 5.3 Hz, 1H), 6.14-6.10 (m, 2H), 4.46 (t, *J* = 5.2 Hz, 1H), 3.70 (s, 3H), 3.33 (dd, J_1 = 6.8 Hz, J_2 = 5.4 Hz 1H), 3.17 (dd, J_1 = 14.7 Hz, J_2 = 2.7 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.9, 167.8, 159.6, 135.9, 134.1, 130.8, 130.4, 128.8, 128.6, 124.7, 120.6, 114.2, 110.2, 104.4, 103.0, 64.9, 55.3, 41.9, 41.5. **HRMS (ESI)** calculated $[M+H]^+$ for $C_{31}H_{26}O_4N$: 476.1856, found: 476.1861. **FTIR (cm^{-1})** 3123, 2921, 1710, 1536, 1469, 1365, 1254, 1194, 1054, 905.

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-2,5-diphenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g] indolizin-4-one (10b)



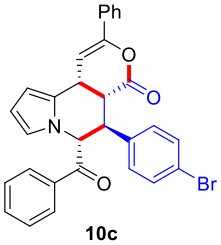
10b

Following the general procedure, (*E*)-3-(1-(2-oxo-2-phenylethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9a** (79.1 mg, 0.25 mmol) and *trans*-cinnamaldehyde **2b** (47.2 μL, 0.375 mmol) were treated with the triazolium salt **5** (9.2 mg, 0.025 mmol), oxidant **4** (153.2 mg, 0.50 mmol) and LiCl (10.6 mg, 0.25 mmol) in THF (3 mL) at 30 °C. To this stirring solution at 30 °C was added DMAP (45.8, 0.375 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 90:10) to afford (4a*R*,5*S*,6*R*,10b*R*)-6-benzoyl-2,5-diphenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g] indolizin-4-one **10b** as a white solid (49 mg, 44% yield, 4:1 dr).

R_f (Pet. ether /EtOAc = 85/15): 0.36; Major diastereomer, er = >99:1, Minor diastereomer, er = >99:1. $[\alpha]_D^{22}$ = +59.9 (c 1, $CHCl_3$). HPLC (Chiralpak AD, 80:20 Hexane / IPA, 0.7 mL/min, 254 nm) Major diastereomer: Minor: 20.4 min, Major: 17.9 min, Minor diastereomer: Major: 32.2 min, Minor: 29.4 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.78-7.74 (m, 2H), 7.63-7.60 (m, 2H), 7.46-7.25 (m, 8H), 7.38-7.35 (m, 3H), 6.62-6.61 (m, 1H), 6.33 (d, *J* = 2.3 Hz, 2H), 6.28 (d, *J* = 2.2 Hz, 1H), 5.76 (d, *J* = 3.5 Hz, 1H), 4.08-4.05 (m, 2H), 2.95 (dd, J_1 = 14.3 Hz, J_2 = 9.5 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.7, 168.8, 150.4, 143.8, 134.3, 134.1, 131.9, 130.9, 129.4, 129.3, 129.1, 129.0, 128.7, 127.8, 124.9, 124.8, 120.0, 109.5, 103.9, 102.8, 65.7, 48.3, 43.5, 31.9. **Representative peaks for minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.76-7.74 (m, 2H), 7.60-7.59 (m, 2H), 7.46-7.25 (m, 9H), 7.04-7.01 (m, 2H), 6.70-6.69 (m, 1H), 6.30 (d, *J* = 3.2 Hz, 1H), 6.20 (d, *J* = 5.6 Hz, 1H), 6.14-6.10 (m, 2H), 4.50 (t, *J* = 5.3 Hz, 1H), 4.08-4.06 (m, 1H), 3.36 (dd, J_1 = 6.9 Hz, J_2 = 4.9 Hz, 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.8, 168.9, 149.6, 136.1, 135.9, 134.3, 134.1, 133.6, 132.1, 130.9, 128.9, 128.2, 128.1, 124.9, 121.2, 109.4, 106.2, 102.9, 59.4, 42.3, 41.9, 31.7. **HRMS (ESI)** calculated $[M+H]^+$ for $C_{30}H_{24}O_3N$: 446.1751, found: 446.1756. **FTIR (cm^{-1})** 3122, 2923, 1714, 1535, 1477, 1360, 1255, 1191, 1054, 913.

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-5-(4-bromophenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10c)

Following the general procedure, (*E*)-3-(1-(2-oxo-2-phenylethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9a** (79.1 mg, 0.25 mmol) and (E)-3-(4-bromophenyl)acrylaldehyde **2d** (79.1 mg, 0.375 mmol) were treated with the triazolium salt **5** (9.2 mg, 0.025 mmol), oxidant **4** (153.2 mg, 0.50 mmol) and LiCl (10.6 mg, 0.25 mmol) in THF (3 mL) at 30 °C. To this

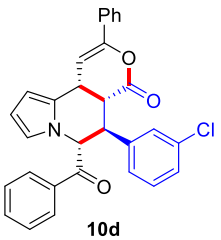


10c

stirring solution at 30 °C was added DMAP (45.8, 0.375 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 90:10) to afford (4a*R*,5*S*,6*R*,10b*R*)-6-benzoyl-5-(4-bromophenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one **10c** as a white solid (67 mg, 51% yield, 4:1 dr).

R*f* (Pet. ether /EtOAc = 70/30): 0.37; Major diastereomer, er = >99:1, Minor diastereomer, er = 97:3. $[\alpha]_D^{22}$ = +70.7 (c 1, $CHCl_3$). HPLC (Chiralcel OD-H, 80:20 Hexane / IPA, 0.7 mL/min, 254 nm) Major diastereomer: Minor: 27.4 min, Major: 21.6 min, Minor diastereomer: Major: 16.8 min, Minor: 13.5 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.76-7.74 (m, 2H), 7.65-7.61 (m, 3H), 7.48-7.45 (m, 4H), 7.38-7.37 (m, 3H), 7.15-7.13 (m, 2H), 6.61 (m, 1H), 6.33-6.27 (m, 3H), 5.69 (d, *J* = 3.9 Hz, 1H), 4.0-4.01 (m, 2H), 2.90 (dd, J_1 = 14.2 Hz, J_2 = 9.6 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.4, 168.6, 150.5, 142.7, 134.4, 134.1, 132.4, 131.9, 130.7, 129.6, 129.5, 129.2, 129.0, 128.7, 124.9, 121.8, 120.0, 109.7, 104.1, 102.7, 65.4, 48.1, 43.1, 31.9. **Representative peaks for minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.82-7.80 (m, 2H), 7.48-7.45 (m, 2H), 7.38-7.37 (m, 5H), 7.28 (m, 2H), 7.01-6.99 (m, 1H), 6.93-6.91 (m, 2H), 6.69 (m, 1H), 6.28-6.27 (m, 1H), 6.20 (d, *J* = 4.9 Hz, 1H), 6.13-6.12 (m, 2H), 4.48 (t, *J* = 4.9 Hz, 1H), 4.01-3.97 (m, 1H), 3.32 (dd, J_1 = 7.1 Hz, J_2 = 4.9 Hz, 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 196.0, 168.6, 131.9, 130.9, 129.6, 129.1, 128.6, 128.3, 124.9, 121.3, 109.6, 104.6, 102.6, 67.6, 53.3, 41.9, 31.7. **HRMS (ESI)** calculated $[M+H]^+$ for $C_{30}H_{23}BrO_3N$: 524.0856, found: 524.0858. **FTIR (cm^{-1})** 2925, 2356, 1763, 1684, 1610, 1567, 1513, 1251, 1180, 1152.

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-5-(3-chlorophenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10d)



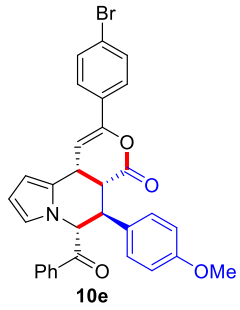
10d

Following the general procedure, (*E*)-3-(1-(2-oxo-2-phenylethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9a** (79.1 mg, 0.25 mmol) and (*E*)-3-(3-chlorophenyl)acrylaldehyde **2i** (62.5 mg, 0.375 mmol) were treated with the triazolium salt **5** (9.2 mg, 0.025 mmol), oxidant **4** (153.2 mg, 0.50 mmol) and LiCl (10.6 mg, 0.25 mmol) in THF (3 mL) at 30 °C. To this stirring solution at 30 °C was added DMAP (45.8, 0.375 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 90:10) to afford (4a*R*,5*S*,6*R*,10b*R*)-6-benzoyl-5-(3-chlorophenyl)-2-phenyl-

4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one **10d** as a white solid (42 mg, 35% yield, 3:1 dr).

R_f (Pet. ether /EtOAc = 85/15): 0.32; Major diastereomer, er = >99:1, Minor diastereomer, er = 97:3. $[\alpha]_D^{22}$ = -79.02 (c 1, $CHCl_3$). HPLC (Chiralpak AD, 85:15 Hexane / IPA, 0.7 mL/min, 254 nm) Major diastereomer: Minor: 20.4 min, Major: 18.8 min, Minor diastereomer: Major: 23.8 min, Minor: 13.6 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.77-7.74 (m, 2H), 7.64-7.60 (m, 3H), 7.48-7.36 (m, 7H), 7.26-7.25 (m, 2H), 6.62 (m, 1H), 6.34-6.32 (m, 2H), 6.28-6.27 (m, 1H), 5.701 (d, *J* = 3.8 Hz, 1H), 4.09-4.02 (m, 2H), 2.93 (dd, J_1 = 14.1 Hz, J_2 = 9.6 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 13C NMR (101 MHz, CDCl3) δ 195.5, 168.6, 150.5, 145.5, 135.1, 134.4, 130.6, 130.5, 129.5, 129.2, 129.1, 129.0, 128.7, 128.1, 127.9, 126.2, 124.93, 124.9, 120.1, 109.7, 104.2, 102.8, 65.3, 47.9, 43.3, 31.9. **Representative peaks for minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81-7.79 (m, 2H), 7.60-7.55 (m, 2H), 7.42-7.40 (m, 1H), 7.26-7.25 (m, 3H), 7.15-7.12 (m, 3H), 7.10-7.06 (m, 1H), 6.01-6.97 (m, 2H), 6.68 (m, 1H), 6.30-6.28 (m, 1H), 6.19-6.10 (m, 3H), 4.47 (t, *J* = 5.2 Hz, 1H), 4.01-3.99 (m, 1H), 3.36 (dd, J_1 = 6.8 Hz, J_2 = 5.1 Hz, 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.0, 166.3, 149.6, 138.1, 133.9, 131.9, 130.6, 130.0, 129.6, 129.4, 128.4, 128.2, 127.4, 124.9, 121.2, 109.6, 106.5, 59.1, 42.1, 41.7, 31.7. **HRMS (ESI)** calculated $[M+H]^+$ for $C_{30}H_{23}ClO_3N$: 480.1361, found: 480.1369. **FTIR (cm^{-1})** 3121, 2923, 1714, 1539, 1470, 1358, 1255, 1192, 1058, 945.

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-2-(4-bromophenyl)-5-(4-methoxyphenyl)-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10e)



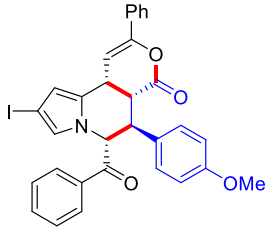
10e

Following the general procedure, (*E*)-1-(4-bromophenyl)-3-(1-(2-oxo-2-phenylethyl)-1H-pyrrol-2-yl)prop-2-en-1-one **9e** (98.6 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl) acrylaldehyde **2a** (60.8 mg, 0.375 mmol) were treated with the triazolium salt **5** (9.2 mg, 0.025 mmol), oxidant **4** (153.2 mg, 0.50 mmol) and LiCl (10.6 mg, 0.25 mmol) in THF (3 mL) at 30 °C. To this stirring solution at 30 °C was added DMAP (45.8, 0.375 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 90:10) to afford (4a*R*,5*S*,6*R*,10b*R*)-6-benzoyl-2-(4-bromophenyl)-5-(4-methoxyphenyl)-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one **10e** as a white solid (57 mg, 48% yield, 1.4:1 dr).

R_f (Pet. ether /EtOAc = 85/15): 0.47; Major diastereomer, er = >99:1, Minor diastereomer, er = >99:1. $[\alpha]_D^{22}$ = +107.2 (c 1, $CHCl_3$). HPLC (Chiralpak ODH, 85:15 Hexane / IPA, 0.7 mL/min, 254 nm) Major diastereomer: Minor: 46.8 min, Major: 40.4 min, Minor diastereomer:

Major: 36.7 min, Minor: 59.7 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.79 (d, *J* = 8.1 Hz, 2H), 7.78-7.45 (m, 4H), 7.38-7.35 (m, 3H), 7.20 (d, *J* = 8.0 Hz, 2H), 6.88 (d, *J* = 8.0 Hz, 2H), 6.62 (m, 1H), 6.35 (m, 1H), 6.17 (m, 1H), 5.68 (m, 1H), 4.02-3.96 (m, 2H), 3.80 (s, 3H), 2.89 (*dd*, J_1 = 14.4 Hz, J_2 = 9.7 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.1, 168.5, 159.2, 150.7, 135.6, 134.6, 132.1, 131.8, 130.7, 129.6, 129.5, 129.2, 129.1, 128.8, 128.7, 124.9, 119.5, 114.7, 106.9, 101.9, 97.3, 65.9, 55.5, 48.1, 42.6, 31.6. **Representative peaks for minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.94 (d, *J* = 7.6 Hz, 2H), 7.78-7.45 (m, 7H), 7.38-7.35 (m, 2H), 6.88 (d, *J* = 8.0 Hz, 2H), 6.54 (m, 1H), 6.36 (m, 1H), 6.13 (m, 1H), 5.98 (m, 1H), 4.21 (m, 1H), 3.86 (m, 1H), 3.70 (s, 3H), 3.15 (d, *J* = 14.3 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 194.8, 167.4, 159.7, 149.9, 134.8, 133.7, 133.2, 131.9, 130.5, 129.4, 129.2, 129.1, 128.8, 128.7, 124.8, 120.2, 114.3, 107.7, 103.6, 98.2, 65.0, 55.4, 39.9, 39.0, 27.9. **HRMS (ESI)** calculated $[M+H]^+$ for $C_{31}H_{25}BrO_4N$: 554.0961, found: 554.0964. **FTIR (cm^{-1})** 2958, 2921, 1699, 1655, 1557, 1454, 1406, 1363, 1252, 1232, 1180.

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-9-iodo-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10f)



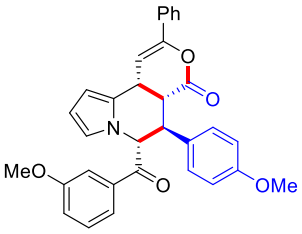
10f

Following the general procedure, (*E*)-3-(4-iodo-1-(2-oxo-2-phenylethyl)-1H-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9f** (110.3 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2b** (60.8 mg, 0.375 mmol) were treated with the triazolium salt **5** (9.2 mg, 0.025 mmol), oxidant **4** (153.2 mg, 0.50 mmol) and LiCl (10.6 mg, 0.25 mmol) in THF (3 mL) at 30 °C. To this stirring solution at 30 °C was added DMAP (45.8, 0.375 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 90:10) to afford (4a*R*,5*S*,6*R*,10b*R*)-6-benzoyl-9-iodo-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one **10f** as a brown solid (95 mg, 62% yield, 5:1 dr).

R_f (Pet. ether /EtOAc = 85/15): 0.25; Major diastereomer, er = >99:1, Minor diastereomer, er = >99:1. $[\alpha]_D^{22}$ = +78.8 (c 1, $CHCl_3$). HPLC (Chiralcel OD-H, 90:10 Hexane / IPA, 0.7 mL/min, 254 nm) Major diastereomer: Minor: 42.2 min, Major: 36.3 min, Minor diastereomer: Major: 28.0 min, Minor: 18.6 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.79-7.77 (m, 2H), 7.62-7.60 (m, 3H), 7.49-7.45 (m, 2H), 7.38-7.36 (m, 3H), 7.21-7.18 (m, 2H), 6.89-6.87 (m, 2H), 6.68 (m, 1H), 6.42 (m, 1H), 6.18 (d, *J* = 2.0 Hz, 1H), 5.72 (d, *J* = 3.2 Hz, 1H), 4.04-3.95 (m, 2H), 3.80 (s, 3H), 2.89 (dd, J_1 = 14.2 Hz, J_2 = 9.6 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 13C NMR (101 MHz, CDCl3) δ 193.9, 168.7, 159.3, 150.0, 135.6, 134.54, 133.7, 133.2, 131.8,

129.6, 129.2, 129.1, 128.8, 128.7, 124.8, 124.6, 114.7, 111.4, 101.9, 65.8, 61.0, 55.4, 48.1, 42.7, 31.6. **Representative peaks for minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.79-7.77 (m, 2H), 7.65-7.63 (m, 3H), 7.47-7.39 (m, 5H),6.96-6.90 (m, 3H), 6.68 (m, 1H), 6.66-6.65 (m, 1H), 6.22 (m, 1H), 6.17-6.15 (m, 1H), 6.08 (d, J = 6.6 Hz, 1H), 4.44 (t, *J* = 5.3 Hz, 1H), 3.98-3.95 (m, 1H), 3.71 (s, 3H), 3.23 (dd, J_1 = 6.8 Hz, J_2 = 5.2 Hz, 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 193.9, 168.7, 159.2, 150.0, 133.8, 131.8, 130.4, 130.3, 129.6, 128.2, 127.5, 126.2, 124.9, 114.1, 113.7, 102.1, 61.5, 59.5, 55.3, 41.9, 41.4, 31.7. **HRMS (ESI)** calculated $[M+H]^+$ for $C_{31}H_{25}IO_4N$: 602.0823, found: 602.0826. **FTIR (cm^{-1})** 3348, 2948, 1740, 1686, 1601, 1513, 1350, 1124, 1022,.

(4a*R*,5*S*,6*R*,10b*R*)-6-(3-Methoxybenzoyl)-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10g)

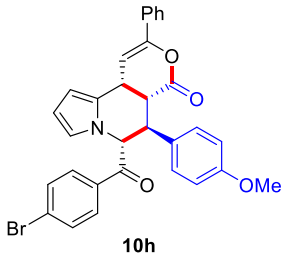


10g

Following the general procedure, (*E*)-3-(1-(2-(3-methoxyphenyl)-2-oxoethyl)-1H-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9g** (86.3 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2b** (60.8 mg, 0.375 mmol) were treated with the triazolium salt **5** (9.2 mg, 0.025 mmol), oxidant **4** (153.2 mg, 0.50 mmol) and LiCl (10.6 mg, 0.25 mmol) in THF (3 mL) at 30 °C. To this stirring solution at 30 °C was added DMAP (45.8, 0.375 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 90:10) to afford (4a*R*,5*S*,6*R*,10b*R*)-6-(3-methoxybenzoyl)-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one **10g** as a white solid (66 mg, 52% yield, 5:1 dr). **R_f** (Pet. ether /EtOAc = 84/15): 0.21; Major diastereomer, er = >99:1, Minor diastereomer, er = 96:4. $[\alpha]_D^{22}$ = +80.04 (c 1, $CHCl_3$). HPLC (Chiralpak AD, 80:20 Hexane / IPA, 0.7 mL/min, 254 nm) Major diastereomer: Minor: 58.9 min, Major: 24.4 min, Minor diastereomer: Major: 83.6 min, Minor: 19.0 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.63-7.60 (m, 2H), 7.40-7.32 (m, 6H), 7.21-7.14 (m, 3H), 6.87-6.85 (m, 2H), 6.62 (m, 1H), 6.33-6.32 (m, 2H), 6.28 (d, *J* = 2.0 Hz, 1H), 5.71 (d, *J* = 3.7 Hz, 1H), 4.10-4.03 (m, 2H), 3.79 (s, 6H), 2.93 (dd, J_1 = 14.3 Hz, J_2 = 9.2 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 13C NMR (101 MHz, CDCl3) δ 195.7, 168.8, 160.2, 159.1, 150.4, 135.9, 135.5, 132.0, 130.9, 130.0, 129.4, 128.9, 128.6, 124.8, 121.5, 121.1, 119.9, 114.6, 113.0, 109.5, 103.9, 102.8, 66.1, 55.5, 55.4, 48.4, 42.8, 31.9. **Representative peaks for minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.85-7.82 (m, 1H), 7.63-7.60 (m, 2H), 7.45-7.43 (m, 5H), 7.01-6.96 (m, 3H), 6.83-6.77 (m, 2H), 6.69-6.67 (m, 3H), 6.15-6.10 (m, 2H), 4.48 (t, *J* = 5.7 Hz, 1H), 3.86-3.83 (m, 1H), 3.81 (s, 3H), 3.71 (s, 3H), 3.34 (dd,

J_1 = 6.7 Hz, J_2 = 5.1 Hz, 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 194.8, 168.4, 159.2, 141.0, 130.4, 129.9, 125.8, 120.6, 114.0, 106.2, 103.6, 59.6, 55.4, 55.3, 42.0, 41.7. **HRMS (ESI)** calculated $[M+H]^+$ for $C_{32}H_{28}O_5N$: 506.1962, found: 506.1967. **FTIR (cm^{-1})** 2923, 1764, 1673, 1599, 1485, 1284, 1249, 1178, 1155.

(4a*R*,5*S*,6*R*,10b*R*)-6-(4-Bromobenzoyl)-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10h)

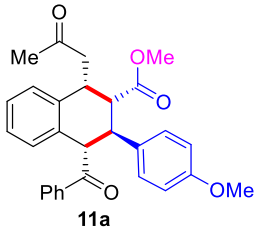


10h

Following the general procedure, (*E*)-3-(1-(2-(4-bromophenyl)-2-oxoethyl)-1H-pyrrol-2-yl)-1-phenylprop-2-en-1-one **9h** (98.6 mg, 0.25 mmol) and *(E)*-3-(4-methoxyphenyl)acrylaldehyde **2b** (60.8 mg, 0.375 mmol) were treated with the triazolium salt **5** (9.2 mg, 0.025 mmol), oxidant **4** (153.2 mg, 0.50 mmol) and LiCl (10.6 mg, 0.25 mmol) in THF (3 mL) at 30 °C. To this stirring solution at 30 °C was added DMAP (45.8, 0.375 mmol) and stirred the reaction mixture at 30 °C for 24 h. Then the reaction mixture was purified using flash column chromatography (Pet. ether-EtOAc: 90:10) to afford (4a*R*,5*S*,6*R*,10b*R*)-6-(4-bromobenzoyl)-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro -4*H*-pyrano[3,4-g] indolizin-4-one **10h** as a white solid (62 mg, 45% yield, 6:1 dr).

R_f (Pet. ether /EtOAc = 85/15): 0.27; Major diastereomer, er = 90:10. $[\alpha]_D^{22}$ = 121.4 (c 1, $CHCl_3$). HPLC (Chiralpak AD, 75:25 Hexane / IPA, 0.7 mL/min, 254 nm) Major diastereomer: Minor: 57.5 min, Major: 25.2 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.64-7.58 (m, 6H), 7.38-7.35 (m, 3H), 7.18-7.16 (m, 2H), 6.87-6.85 (m, 2H), 6.59 (m, 1H), 6.34-6.32 (m, 2H), 6.26 (d, *J* = 2.2 Hz, 1H), 5.66 (d, *J* = 3.9 Hz, 1H), 4.04-3.95 (m, 2H), 3.80 (s, 3H), 2.92 (dd, J_1 = 14.2 Hz, J_2 = 9.6 Hz 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.0, 168.7, 159.1, 150.5, 135.5, 132.9, 132.5, 131.9, 130.8, 130.5, 129.7, 129.5, 128.8, 128.7, 124.8, 119.9, 114.7, 109.6, 104.0, 102.8, 65.9, 55.4, 48.2, 42.8, 31.9. **Representative peaks for minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.52-7.57 (m, 6H), 7.40-7.38 (m, 3H), 6.96-6.92 (m, 3H), 1H), 6.68-6.63 (m, 3H), 6.29-6.28 (m, 1H), 6.13-6.10 (m, 2H), 4.42 (t, *J* = 5.1 Hz, 1H), 4.07-4.04 (m, 1H), 3.71 (s, 3H), 3.31 (dd, J_1 = 6.8 Hz, J_2 = 5.2 Hz, 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.9, 168.9, 149.6, 132.2, 130.4, 128.9, 128.5, 124.9, 121.1, 119.9, 114.1, 109.5, 106.35 102.9, 66.7, 55.3, 41.6, 31.7, 27.2. **HRMS (ESI)** calculated $[M+H]^+$ for $C_{31}H_{25}BrO_4N$: 554.0961, found: 554.0964. **FTIR (cm^{-1})** 2958, 2923, 2355, 1753, 1673, 1593, 1485, 1446, 1357, 1152.

Methyl (2*S*,3*S*,4*S*)-4-benzoyl-3-(4-methoxyphenyl)-1-(2-oxopropyl)-1,2,3,4-tetrahydro naphthalene-2-carboxylate (11a)

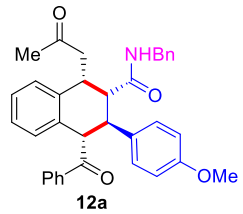


11a

To a dry Schlenk tube containing compound **3a** (53.1 mg, 0.125 mmol) MeOH (3.0 mL) and DMAP (15.3 mg, 0.125 mmol) was added. The resulting mixture was allowed to stir overnight at 60 °C. Then the solution was concentrated under reduced pressure and the residue was purified by flash column chromatography on silica gel using petroleum ether/EtOAc (80:20) to afford the methyl (2*S*,3*S*,4*S*)-4-benzoyl-3-(4-methoxyphenyl)-1-(2-oxopropyl)-1,2,3,4-tetrahydro naphthalene-2-carboxylate **11a** as a oily liquid (46.2 mg, 81% yield).

***R*$_f$** (Pet. ether /EtOAc = 70/30): 0.41; er = 95:5, $[\alpha]_D^{22}$ = -36.4 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 60:40 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 7.9 min, *Major*: 26.8 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.63-7.61 (m, 2H), 7.51-7.48 (m, 1H), 7.37-7.33 (m, 2H), 7.24-7.22 (m, 1H), 7.19-7.16 (m, 1H), 7.10-7.07 (m, 2H), 7.06-7.03 (m, 1H), 6.79 (d, *J* = 7.5 Hz, 1H), 6.74-6.71 (m, 2H), 5.09 (d, *J* = 9.7 Hz, 1H), 4.14-4.06 (m, 1H), 3.87- 3.77 (m, 1H), 3.71 (s, 3H), 3.52-3.46 (m, 1H), 3.41 (m, 3H), 3.35 (dd, J_1 = 12.7 Hz, J_2 = 4.3 Hz, 1H), 2.83 (dd, J_1 = 18.4 Hz, J_2 = 5.2 Hz, 1H), 2.15 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 207.0, 203.3, 173.6, 158.5, 140.2, 138.3, 134.5, 134.1, 133.3, 129.4, 128.82, 128.78, 128.1, 127.4, 127.2, 114.2, 55.2, 54.7, 51.8, 48.2, 47.9, 41.6, 35.8, 30.6. **HRMS (ESI)** m/z: [M+Na]+ calcd for $C_{29}H_{28}O_5Na$ 479.1829; found 479.1832. **FTIR (cm^{-1})** 2958, 2922, 2364, 2330, 1736, 1727, 1676, 1613, 1514, 1364, 1251, 1202, 1161.

(2*S*,3*S*,4*S*)-4-Benzoyl-N-benzyl-3-(4-methoxyphenyl)-1-(2-oxopropyl)-1,2,3,4-tetrahydro naphthalene-2-carboxamide (12a)



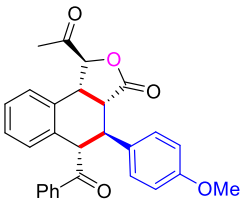
12a

To a dry Schlenk tube containing compound **3a** (48.0 mg, 0.11 mmol) in THF (4.0 mL) was added benzylamine (24.7 µL, 0.22 mmol) at 30 °C. The resulting mixture was allowed to stirr 36 h. Then the solution was concentrated under reduced pressure and the residue was purified by flash column chromatography on silica gel using petroleum ether/EtOAc (70:30) to afford the (2*S*,3*S*,4*S*)-4-benzoyl-N-benzyl-3-(4-methoxyphenyl)-1-(2-oxopropyl)-1,2,3,4-tetrahydro naphthalene-2-carboxamide **12a** as a white solid (42.2 mg, 72% yield).

***R*$_f$** (Pet. ether /EtOAc = 60/40): 0.41; er = 99:1, $[\alpha]_D^{22}$ = -117.3 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 60:40 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 12.0 min, *Major*: 7.3 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.62 (d, *J* = 7.8 Hz, 2H), 7.49 (t, *J* = 7.1 Hz, 1H), 7.34 (t,

J = 7.1 Hz, 2H), 7.25-7.23 (m, 1H), 7.18-7.03 (m, 7H), 6.81 (d, *J* = 7.3 Hz, 1H), 6.73 (d, *J* = 7.7 Hz, 2H), 6.66 (d, *J* = 6.5 Hz, 2H), 5.92 (bs, 1H), 5.03 (d, *J* = 9.9 Hz, 1H), 4.42 (dd, J_1 = 14.9 Hz, J_2 = 7.3 Hz, 1H), 4.06- 4.02 (m, 1H), 3.91-3.84 (m, 2H), 3.74-3.67 (m, 4H), 2.93 (dd, J_1 = 12.1 Hz, J_2 = 3.9 Hz, 1H), 2.81 (dd, J_1 = 18.3 Hz, J_2 = 5.3 Hz, 1H), 2.15 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 207.4, 203.2, 172.1, 158.5, 140.6, 138.1, 138.0, 134.5, 134.3, 133.2, 129.4, 129.2, 128.8, 128.7, 128.4, 128.1, 127.3, 127.1, 127.1, 114.2, 55.2, 54.7, 50.4, 47.8, 43.1, 42.0, 36.4, 30.7. **HRMS (ESI)** m/z: $[M+Na]^+$ calcd for $C_{35}H_{33}O_4NNa$ 554.2302; found 554.2306. **FTIR (cm^{-1})** 3291, 3087, 2957, 2924, 2364, 2333, 1714, 1675, 1555, 1513, 1494, 1357, 1248.

(1*S*,3a*R*,4*S*,5S,9b*R*)-1-Acetyl-5-benzoyl-4-(4-methoxyphenyl)-3a,4,5,9b-tetrahydro naphtho[1,2-c]furan-3(1*H*)-one (13a)



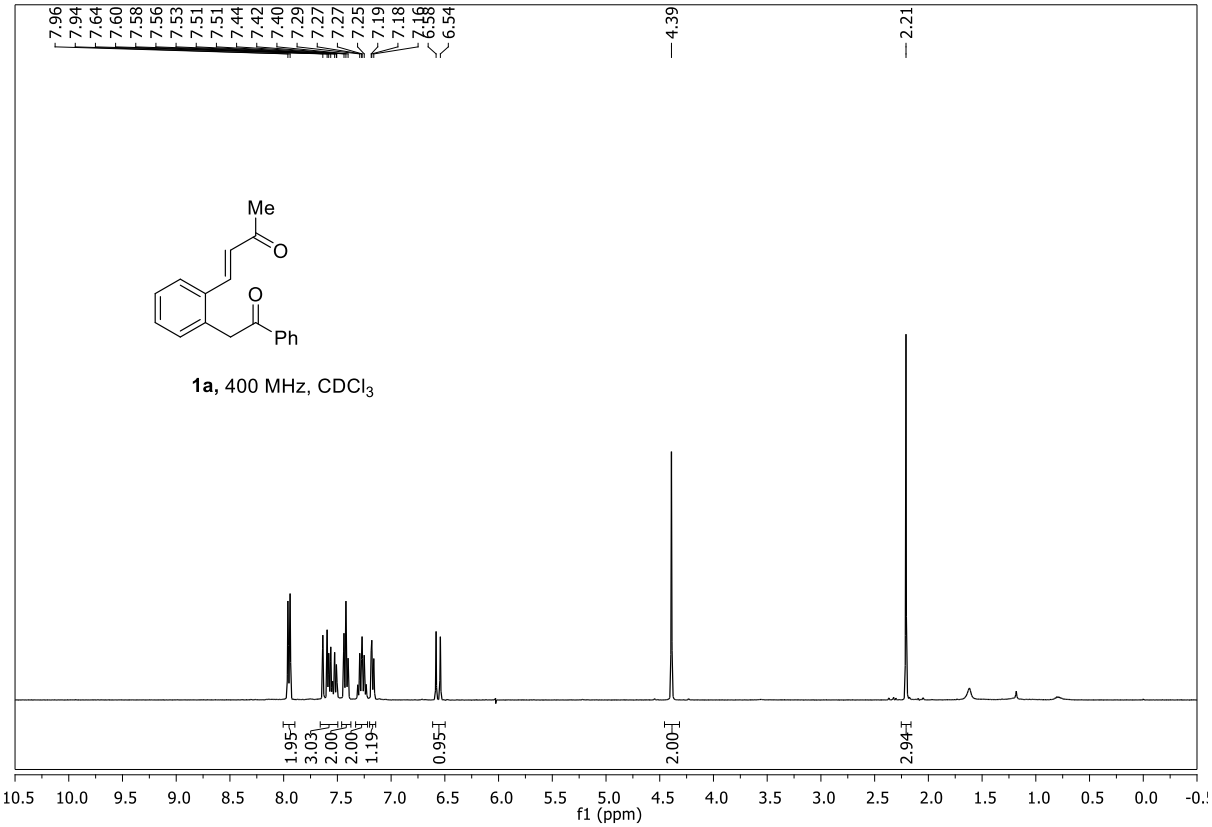
13a

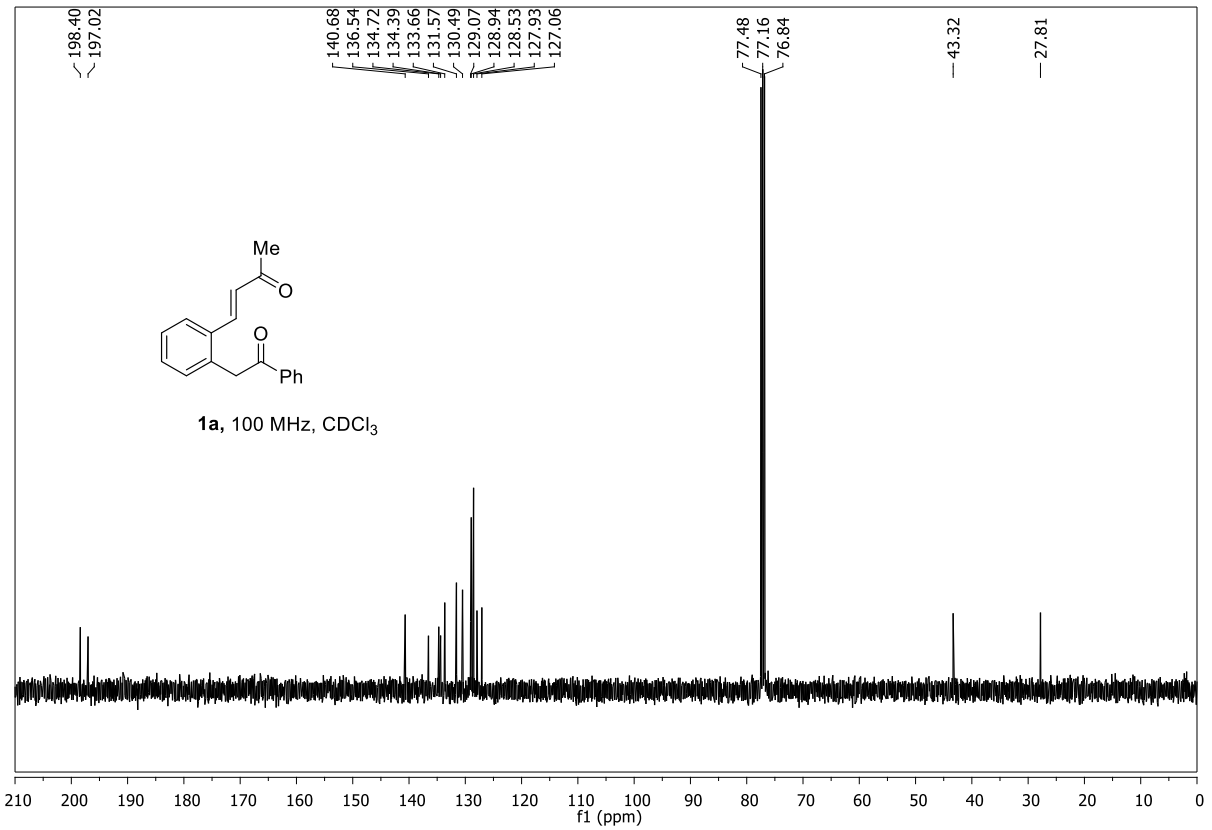
To a dry Schlenk tube containing compound **3a** (53.1 mg, 0.125 mmol) in DCM (2.0 mL) mCPBA (32.3 mg, 0.1875 mmol) was added at 0 °C and the resulting mixture was allowed to stir overnight at room temperature. After the completion of reaction, PTSA (2 mg, 0.0125 mmol) was added, and the reaction was stirred for another 20 min at room temperature, quenched with aqueous $NaHCO_3$ solution and extracted with DCM for three times, the combined organic layer was washed with saturated brine and dried over anhydrous Na_2SO_4. DCM was removed under reduced pressure, and crude reaction mixture was purified by flash column chromatography through silica gel (petroleum ether: ethyl acetate 80:20) to afford (1*S*,3a*R*,4*S*,5S,9b*R*)-1-Acetyl-5-benzoyl-4-(4-methoxyphenyl)-3a,4,5,9b-tetrahydro naphtho [1,2-c]furan-3(1*H*)-one **13a** as oily liquid (36 mg, 66% yield).

***R*$_f$** (Pet. ether /EtOAc = 70/30): 0.36; er = 98:2, $[\alpha]_D^{22}$ = -164.1 (*c* 1.0, $CHCl_3$). **HPLC** (Chiralpak AD, 70:30 Hexane / *i*-PrOH, 0.7 mL/min, 254 nm) *Minor*: 16.0 min, *Major*: 17.4 min. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.01-7.99 (m, 2H), 7.65-7.61 (m, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.31-7.26 (m, 1H), 7.24-7.21 (m, 1H), 7.17 (d, *J* = 7.4 Hz, 1H), 7.01 (d, *J* = 7.0 Hz, 1H), 6.96-6.94 (m, 2H), 6.77-6.57 (m, 2H), 5.05 (d, *J* = 8.4 Hz, 1H), 5.00 (d, *J* = 1.6 Hz, 1H), 4. 30 (t, *J* = 9.2 Hz, 1H), 4.13 (m, 1H), 3.74 (s, 3H), 3.30 (dd, J_1 = 9.9 Hz, J_2 = 2.8 Hz, 1H), 2.06 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** 208.1, 200.6, 176.2, 158.9, 136.1, 134.3, 134.2, 133.7, 131.3, 130.7, 130.6, 129.0, 128.9, 128.4, 128.3, 127.9, 114.6, 85.5, 55.4, 51.81, 44.2, 39.6, 39.0, 27.7. **HRMS (ESI)** m/z: [M+Na]+ calcd for $C_{28}H_{24}O_5Na$ 463.1516; found 463.1516.**FTIR (cm^{-1})** 2959, 2923, 2365, 2332, 1780, 1718, 1678, 1513, 1455, 1364, 1252, 1153.

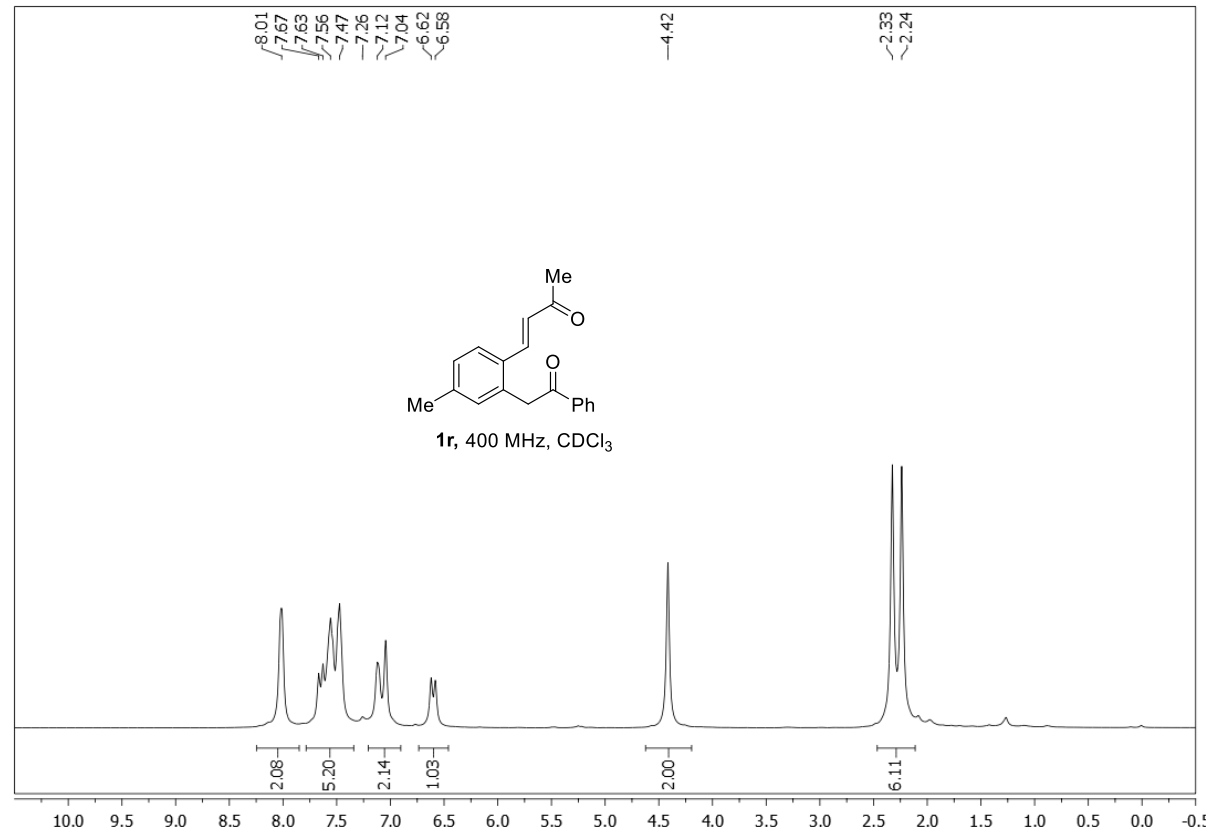
8. ^{1}H and ^{13}C NMR Spectra of Michael Acceptors Used

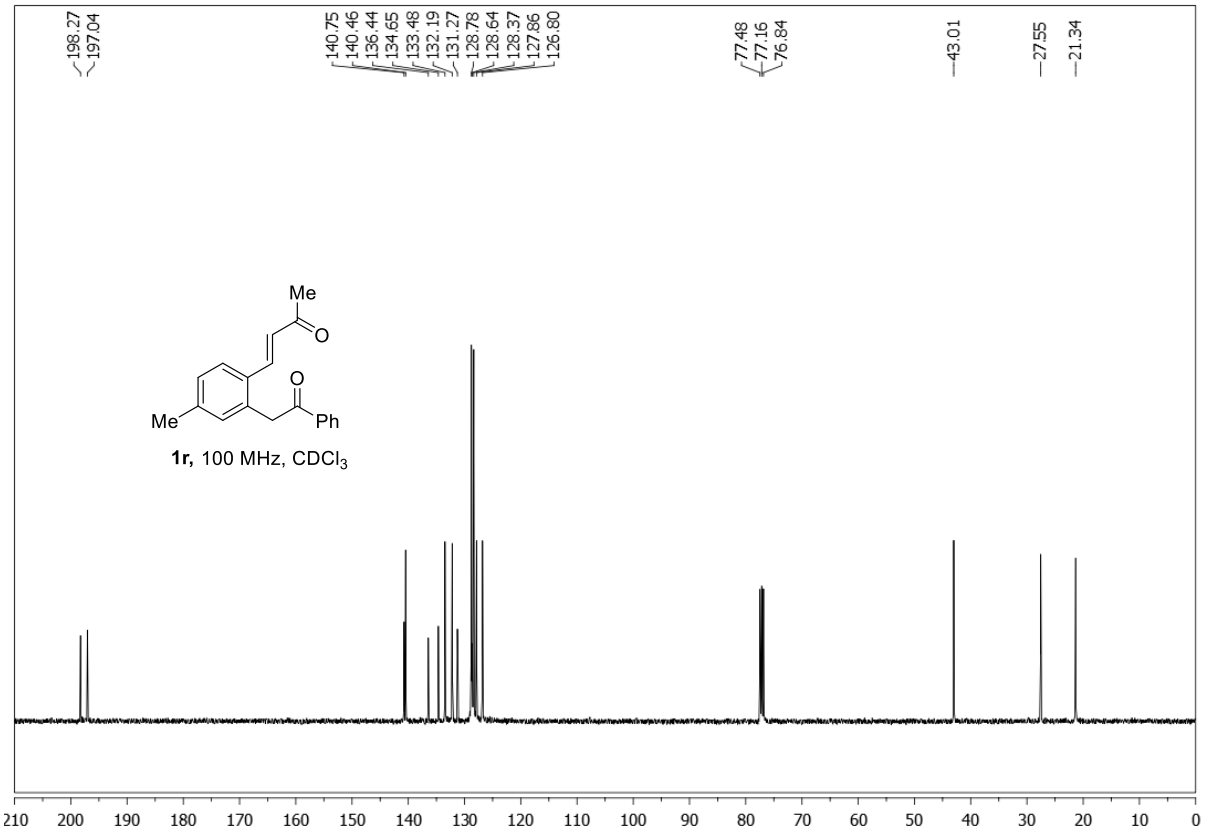
(*E*)-4-(2-(2-Oxo-2-phenylethyl)phenyl)but-3-en-2-one (1a)



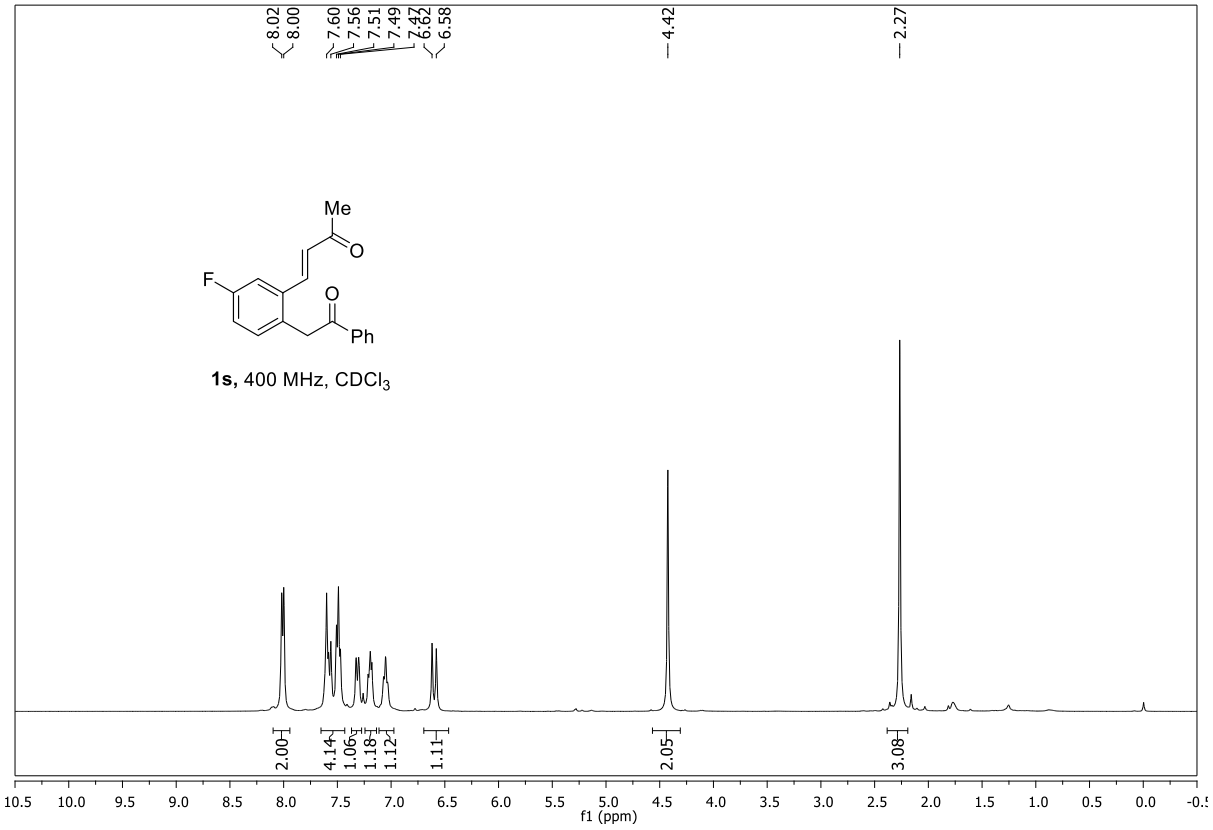


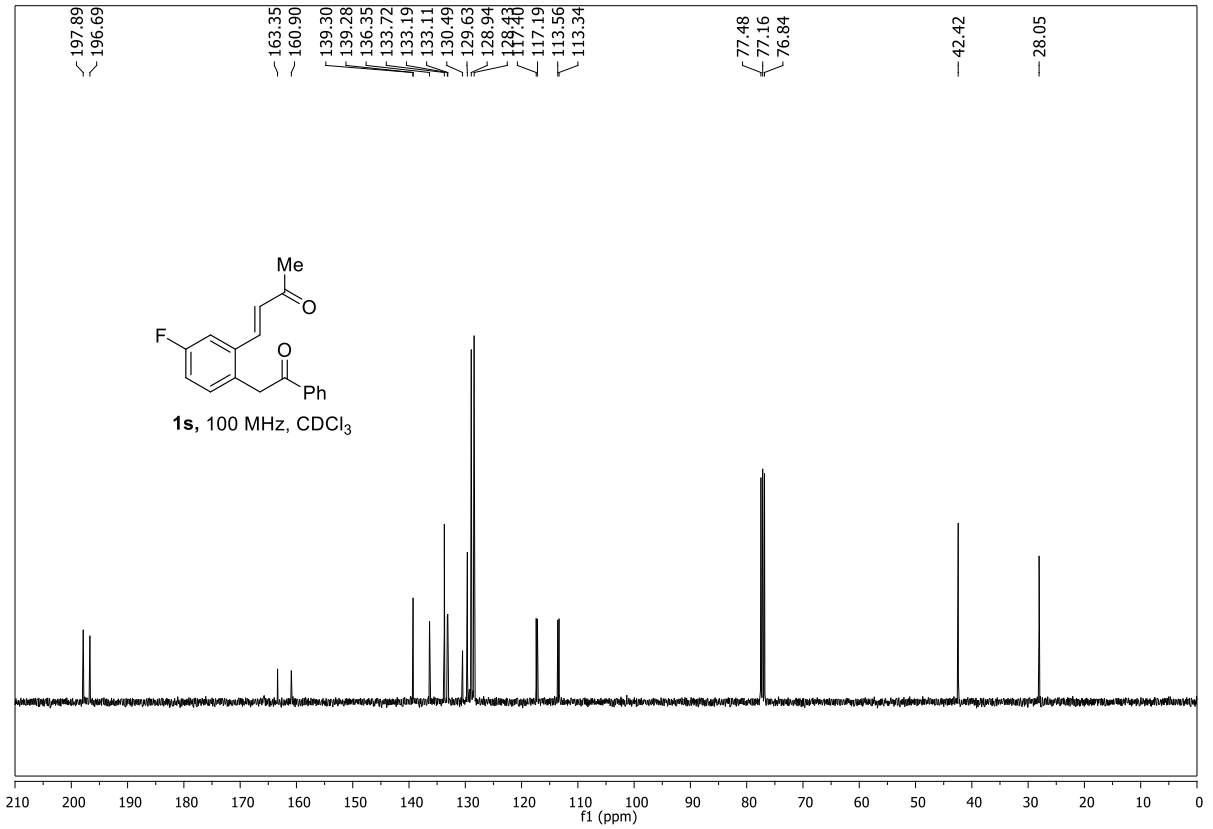
(*E*)-4-(4-Methyl-2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one (1r)



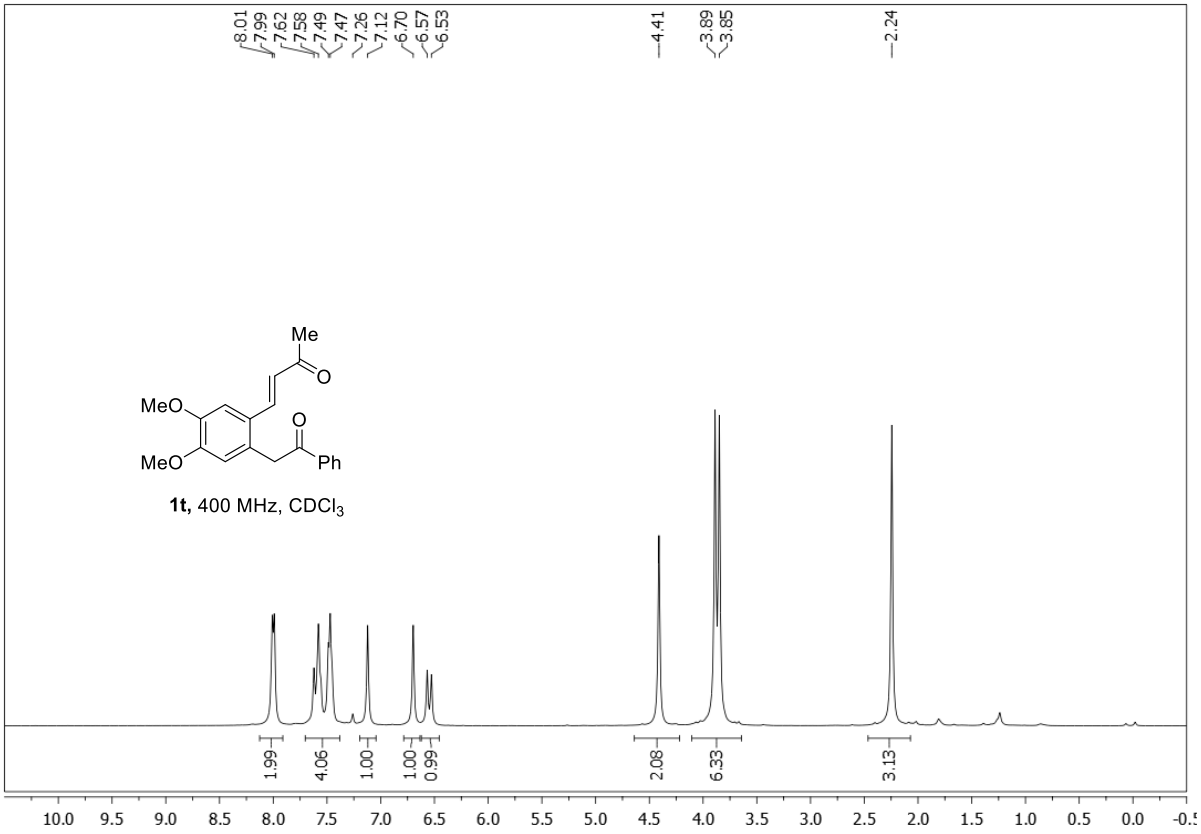


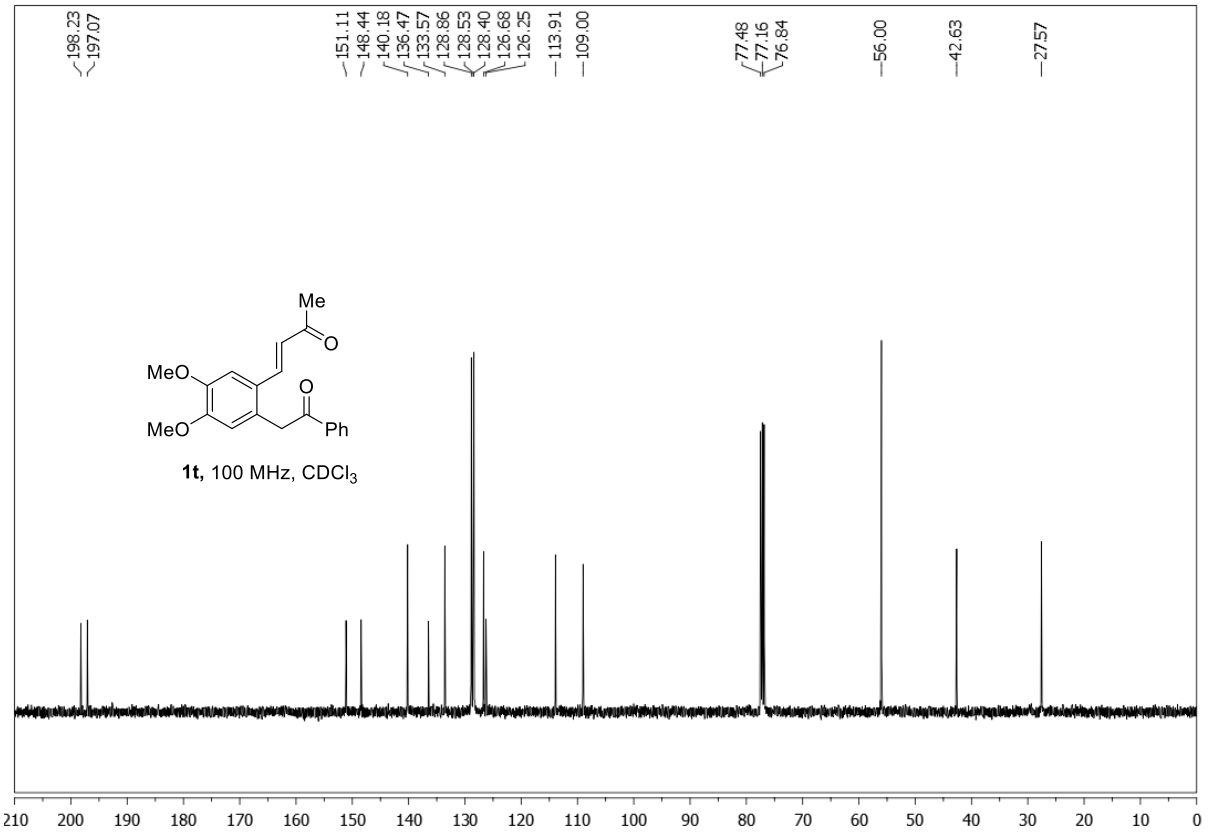
(*E*)-4-(5-Fluoro-2-(2-oxo-2-phenylethyl) phenyl)but-3-en-2-one (1s)



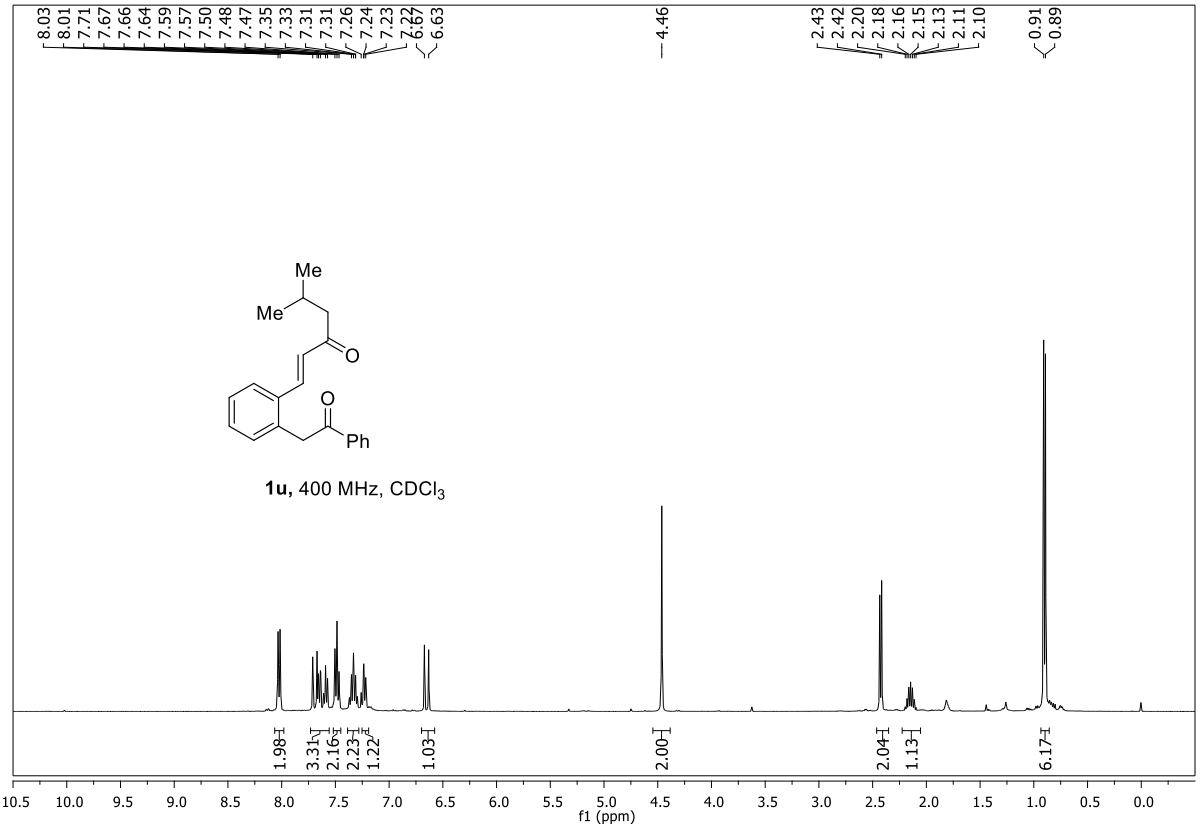


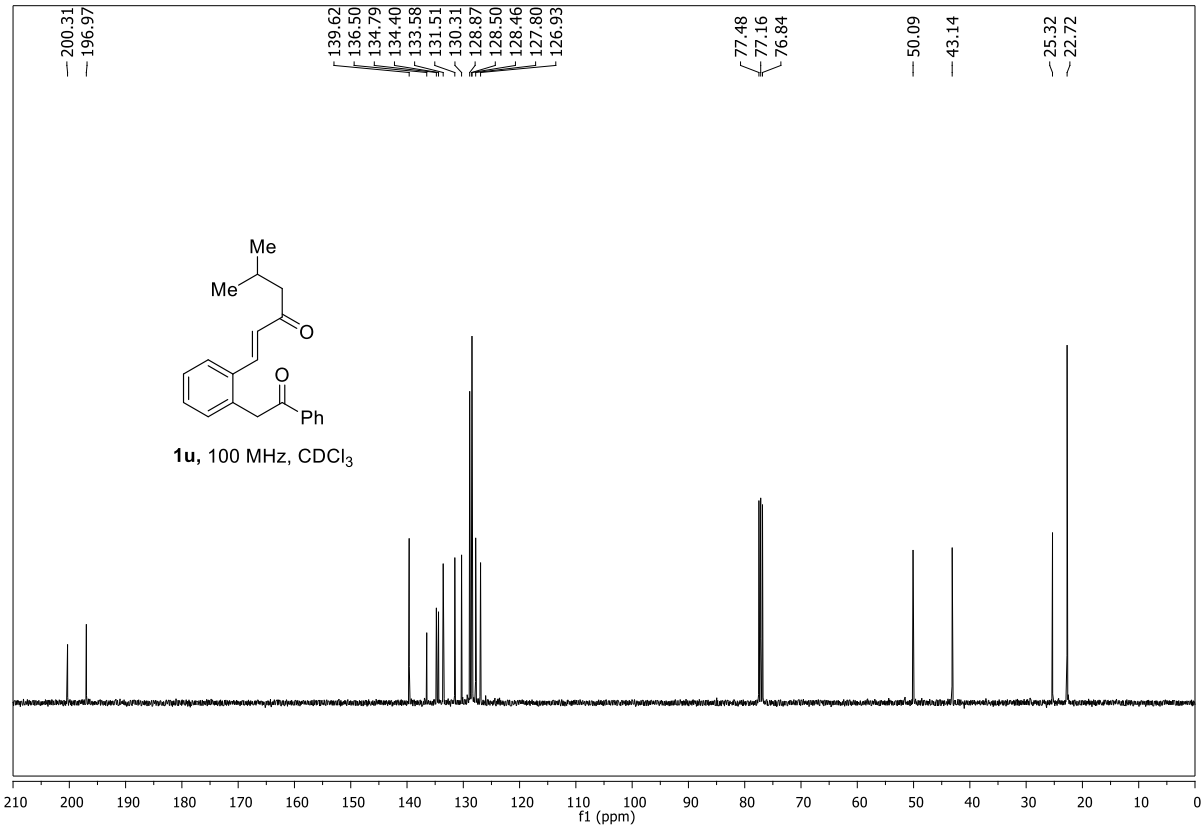
(*E*)-4-(4,5-Dimethoxy-2-(2-oxo-2-phenylethyl)phenyl)but-3-en-2-one (1t)



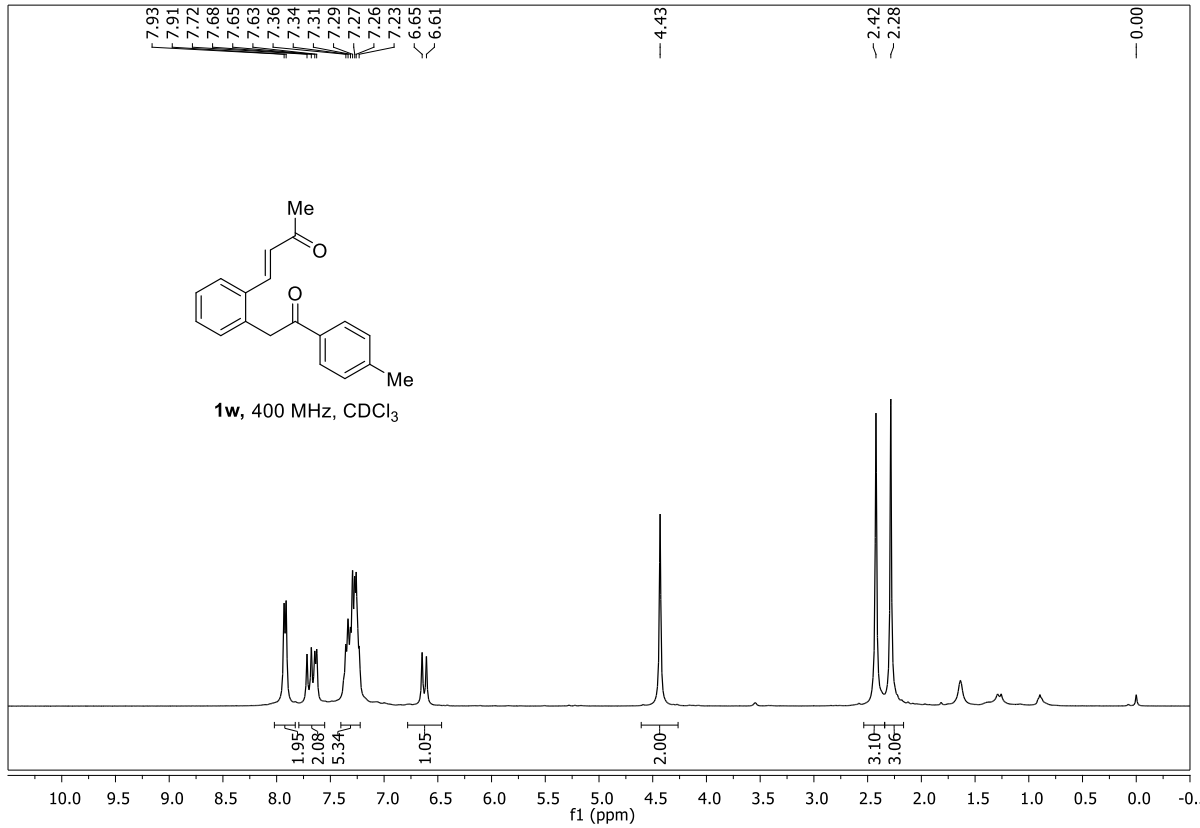


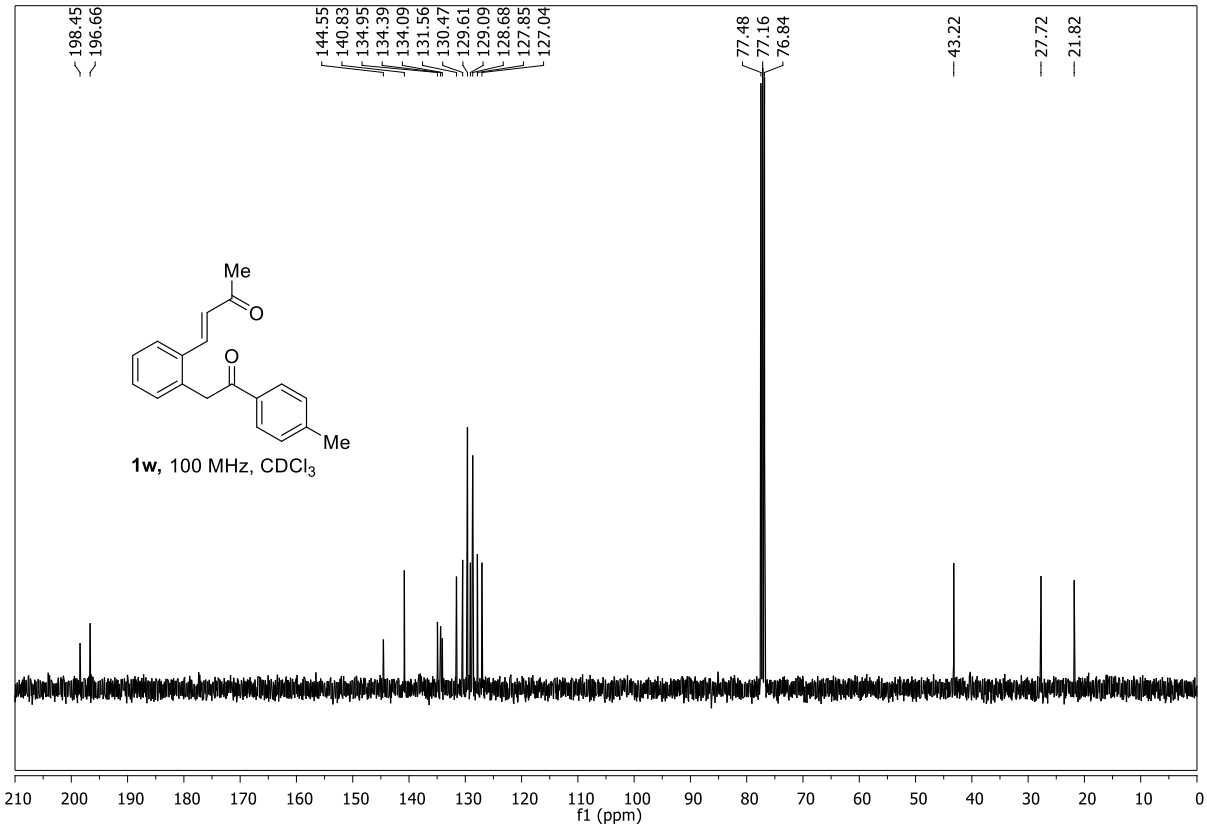
(*E*)-5-Methyl-1-(2-(2-oxo-2-phenylethyl) phenyl)hex-1-en-3-one (1u)



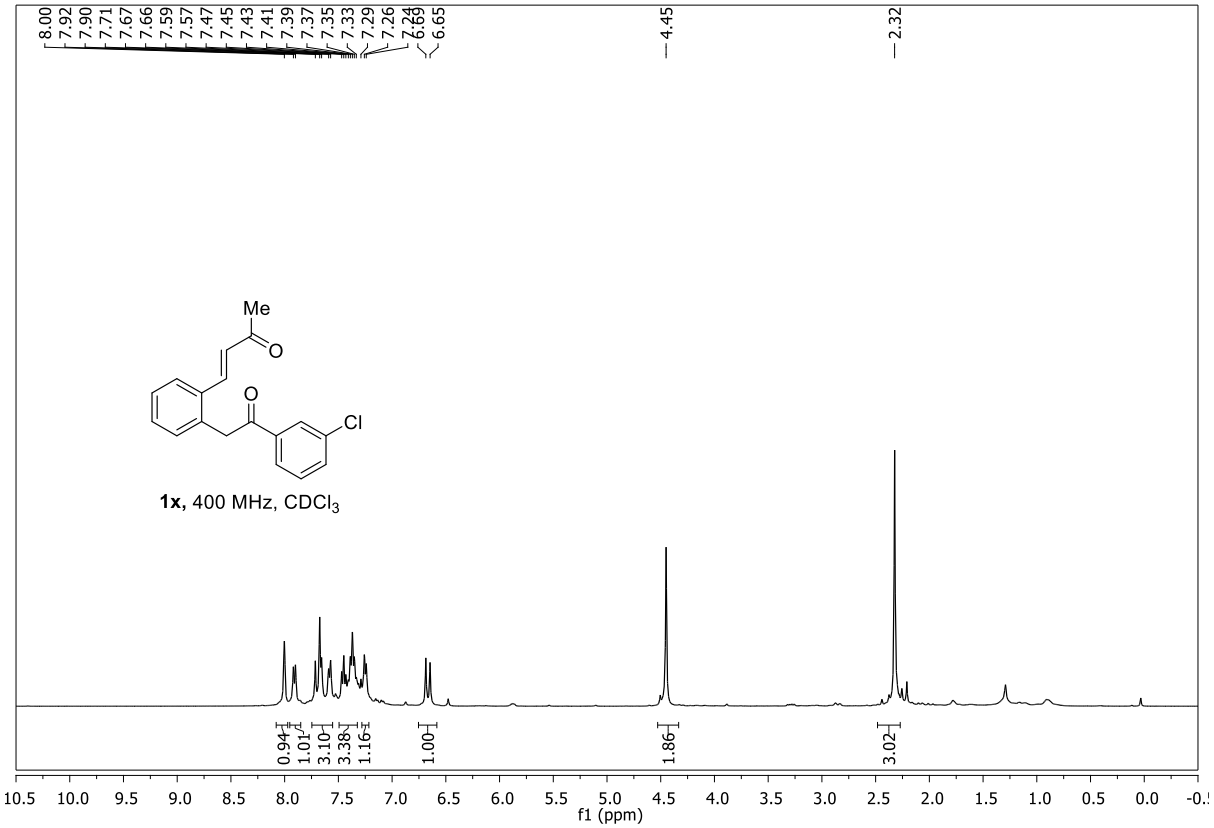


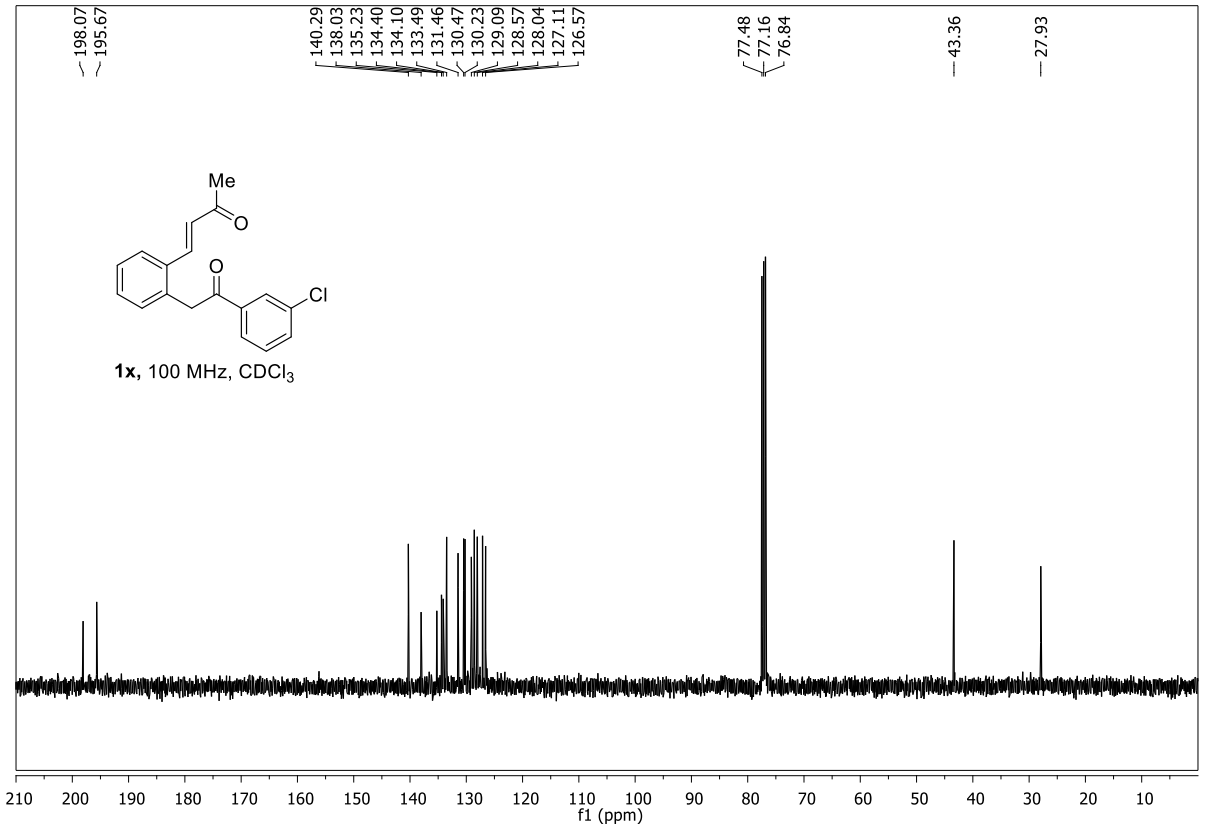
(*E*)-4-(2-(2-Oxo-2-(*p*-tolyl)ethyl)phenyl)but-3-en-2-one (1w)



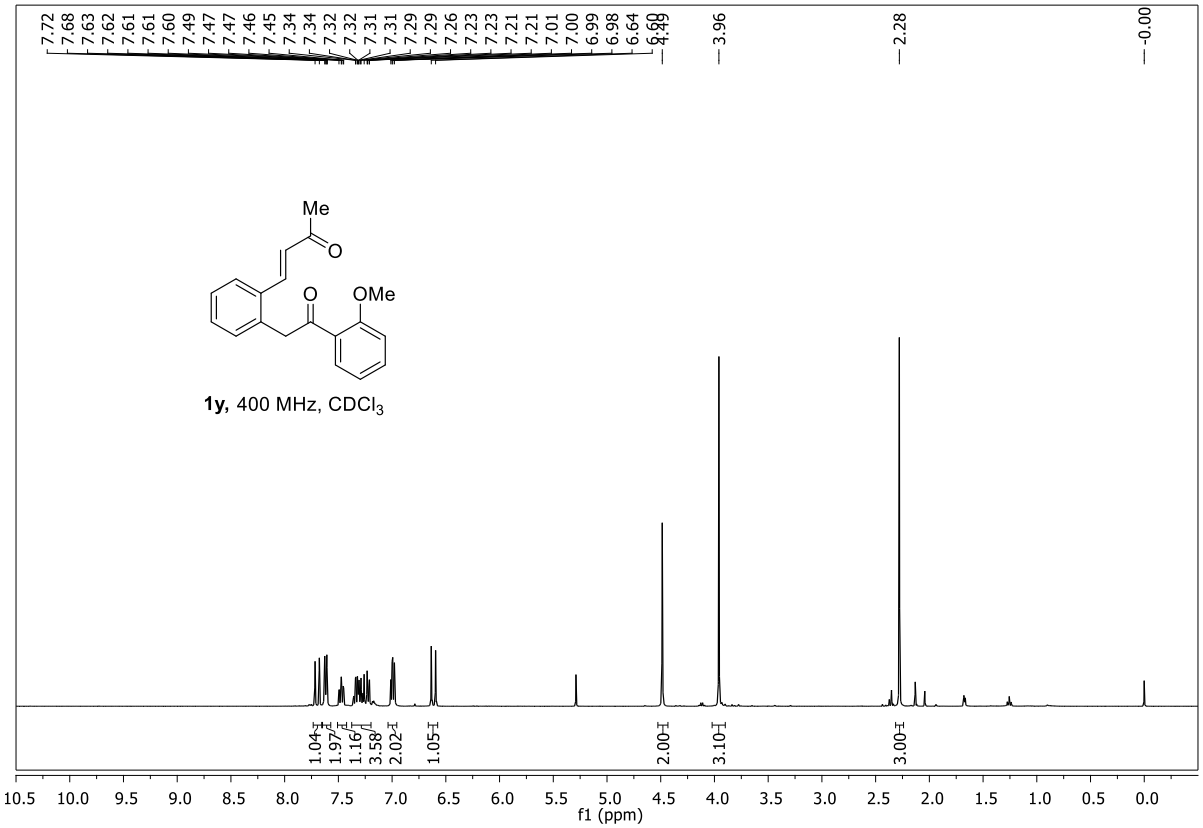


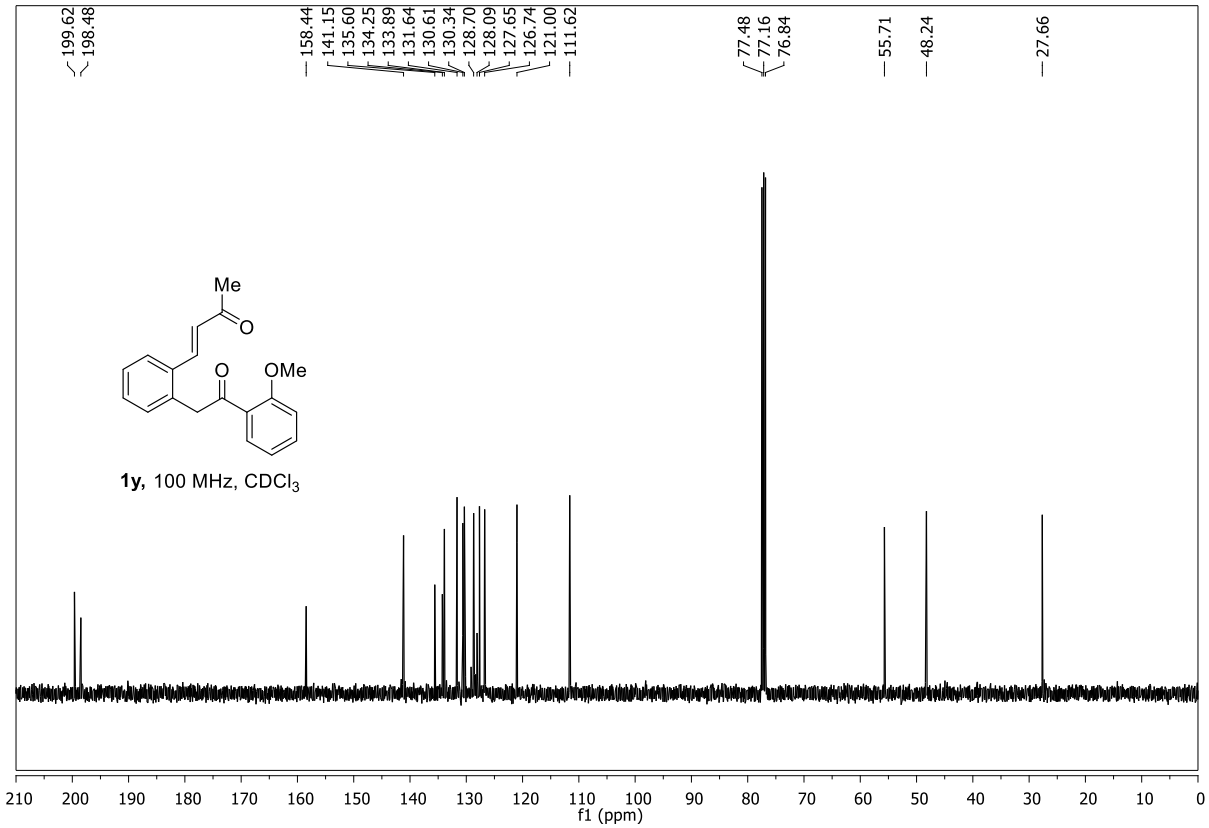
(*E*)-4-(2-(2-(3-Chlorophenyl)-2-oxoethyl)phenyl)but-3-en-2-one (1x)



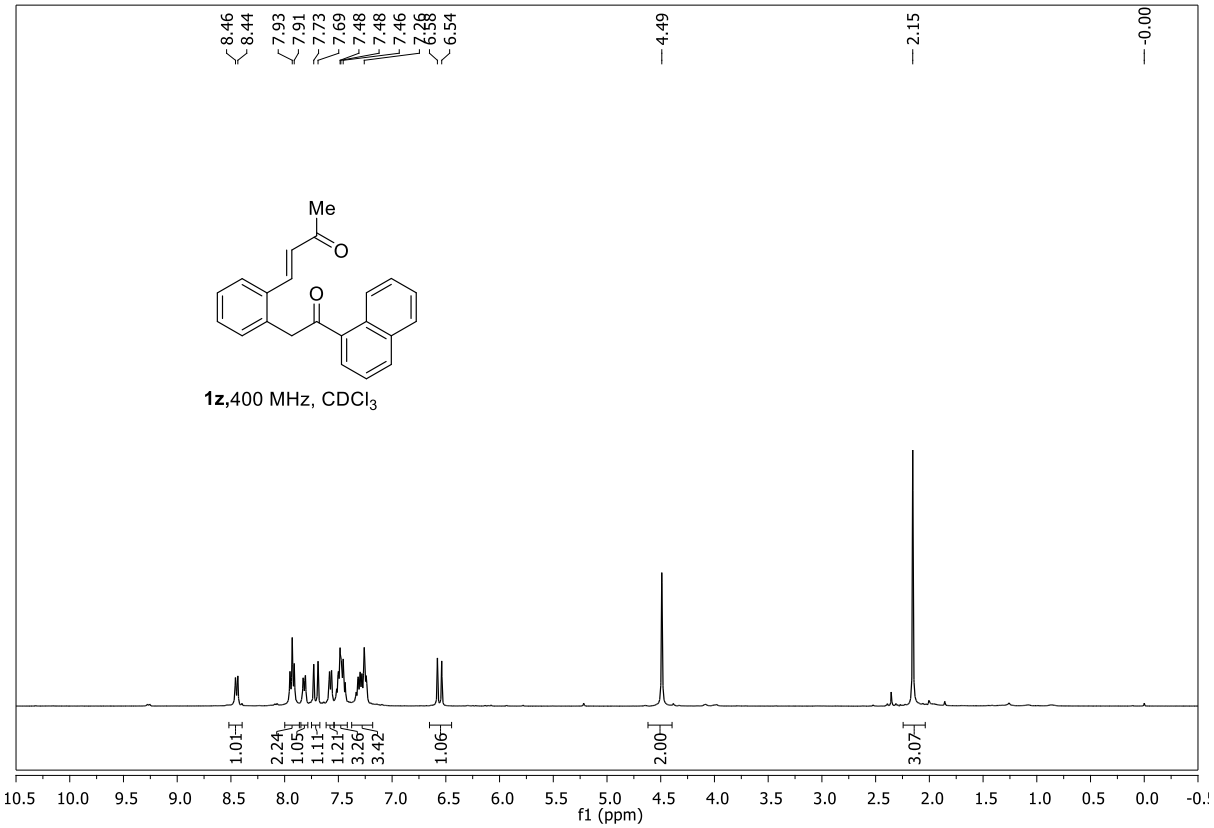


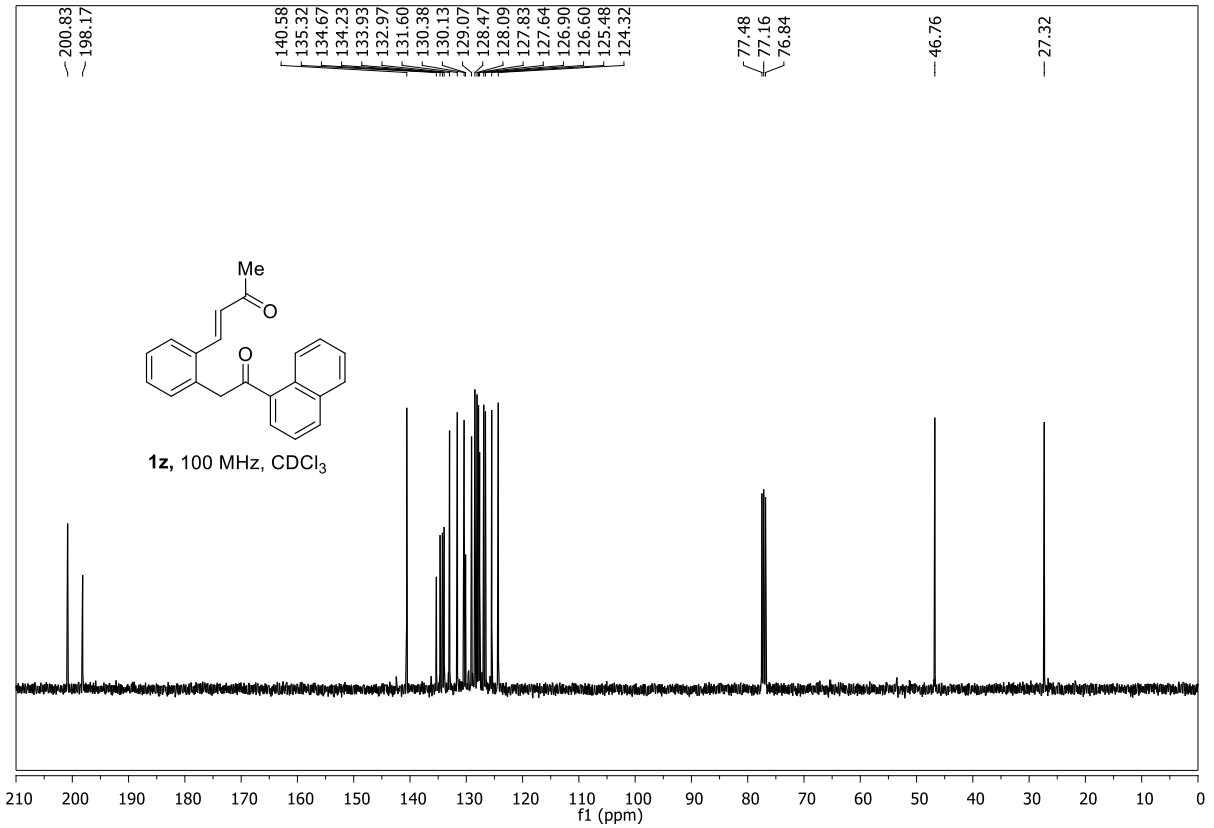
(*E*)-4-(2-(2-(2-Methoxyphenyl)-2-oxoethyl)phenyl)but-3-en-2-one (1y)



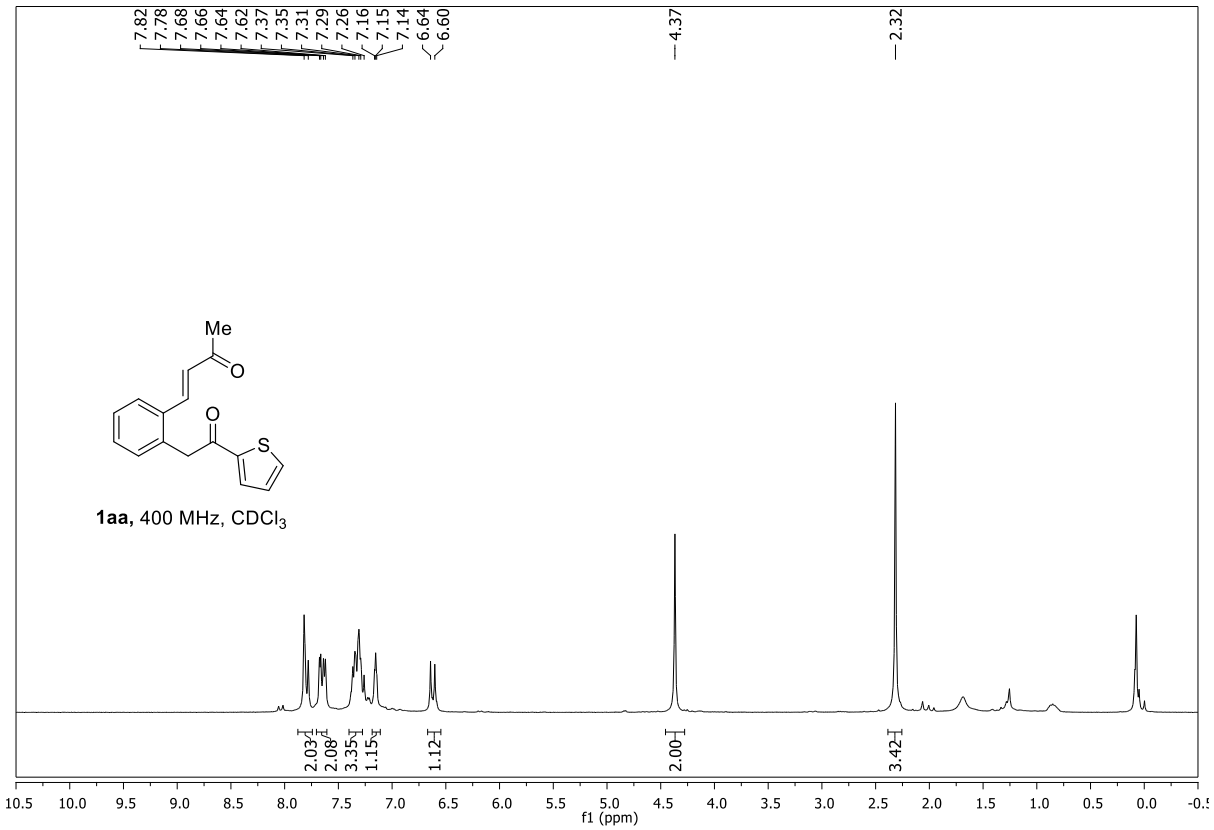


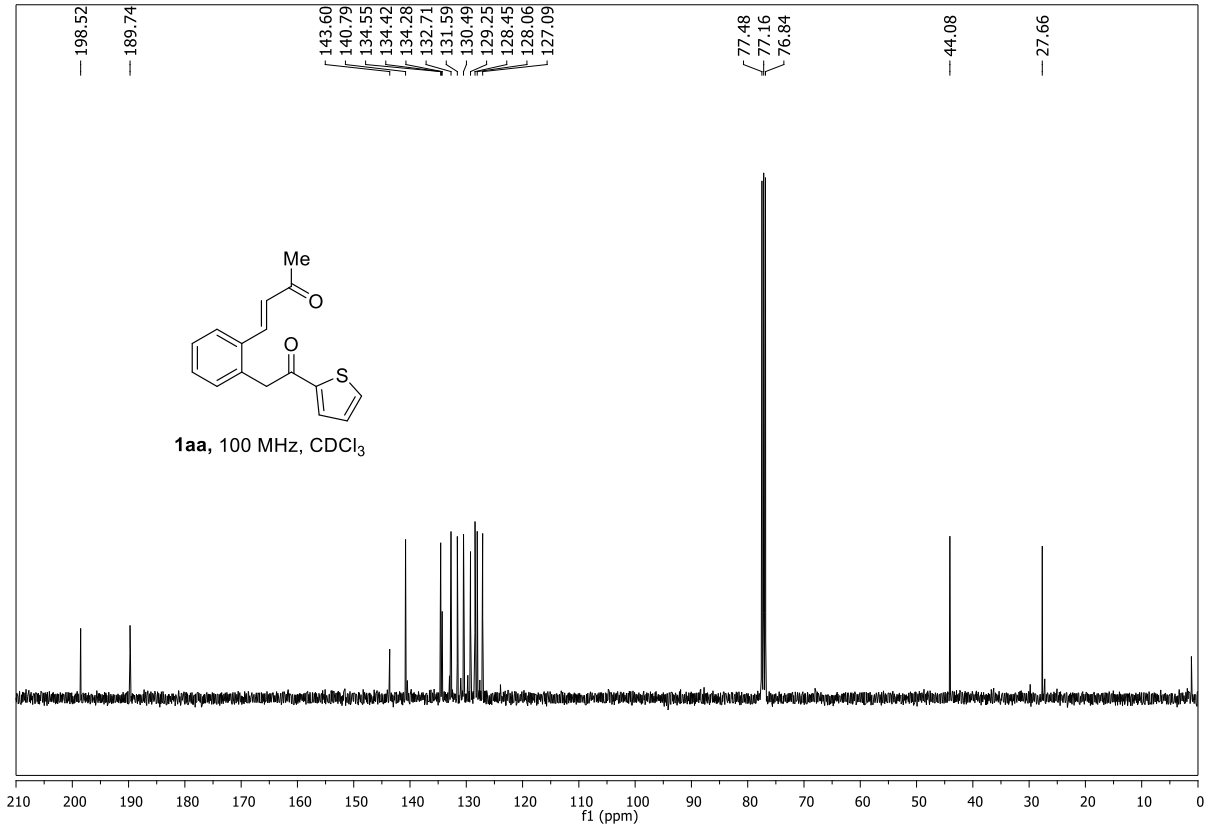
(*E*)-4-(2-(2-(Naphthalen-1-yl)-2-oxoethyl)phenyl)but-3-en-2-one (1z)



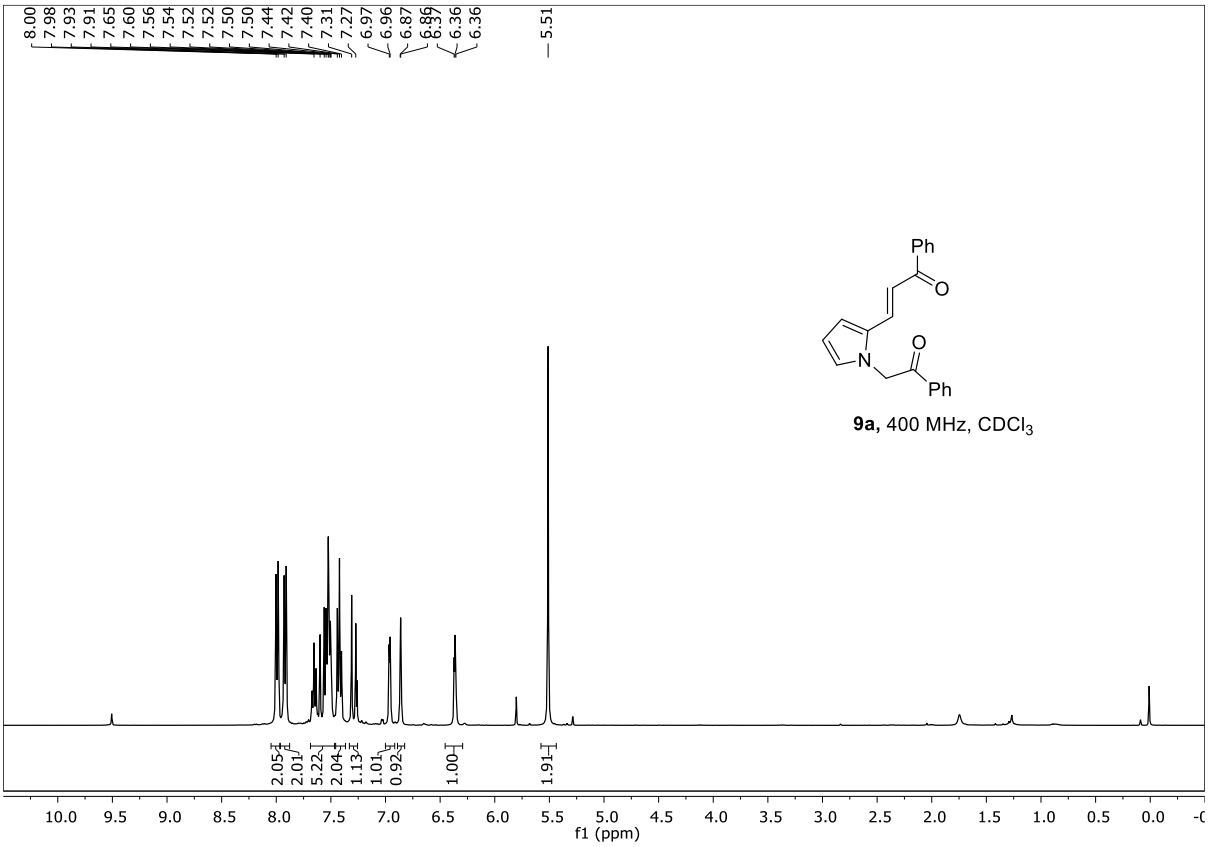


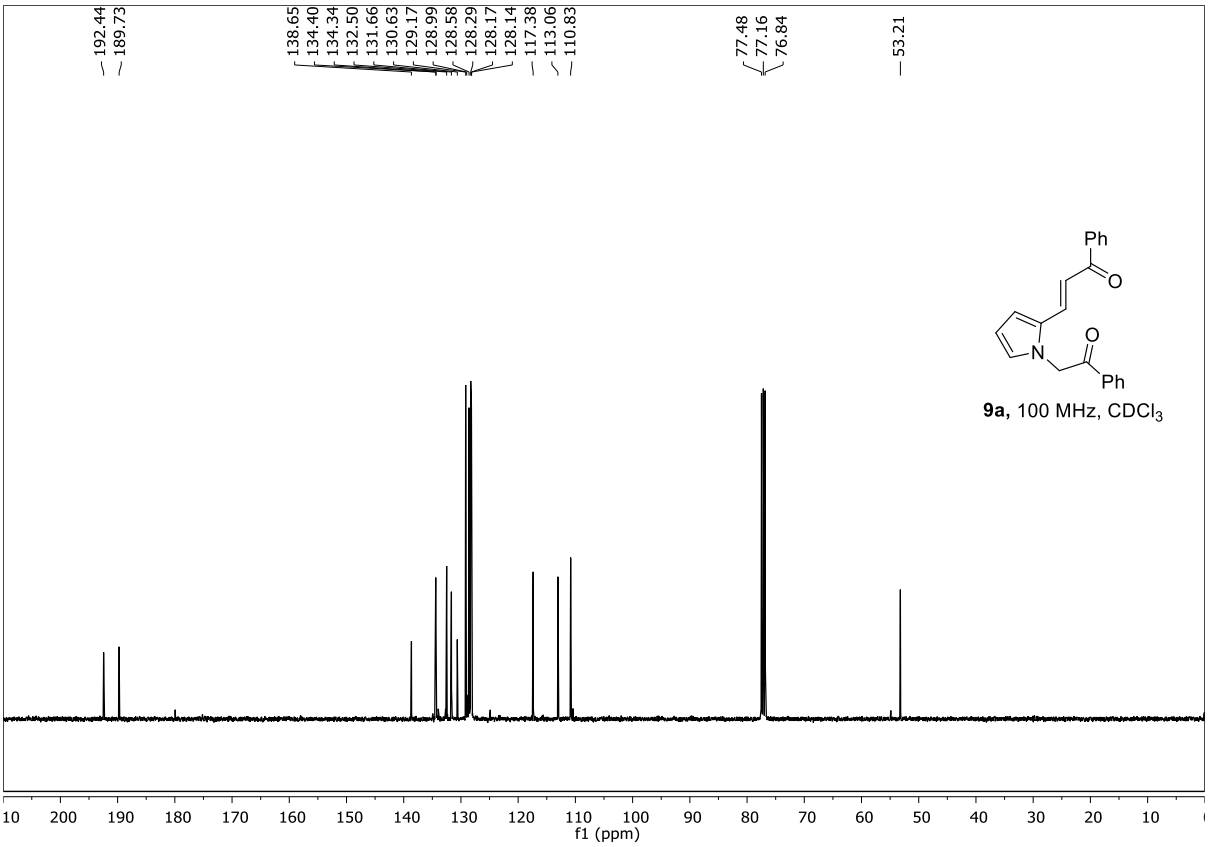
(*E*)-4-(2-(2-Oxo-2-(thiophen-2-yl)ethyl) phenyl)but-3-en-2-one (1aa)



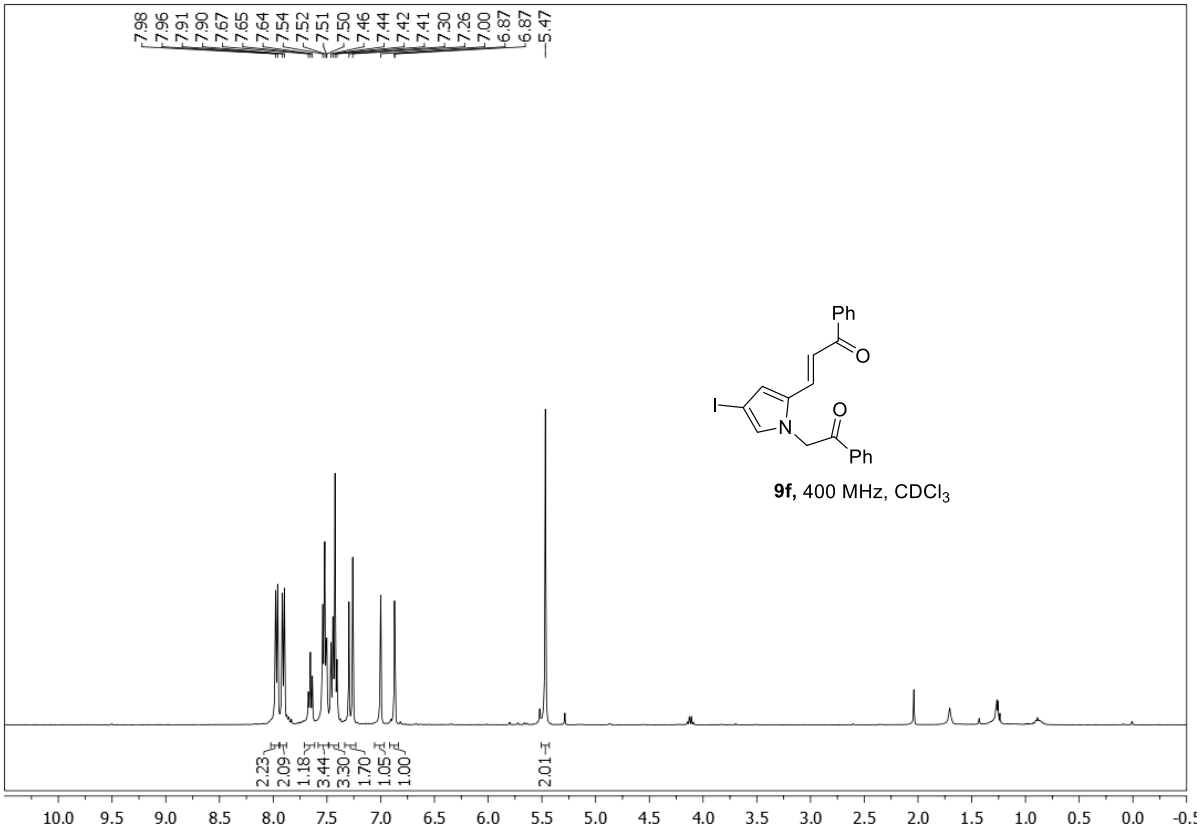


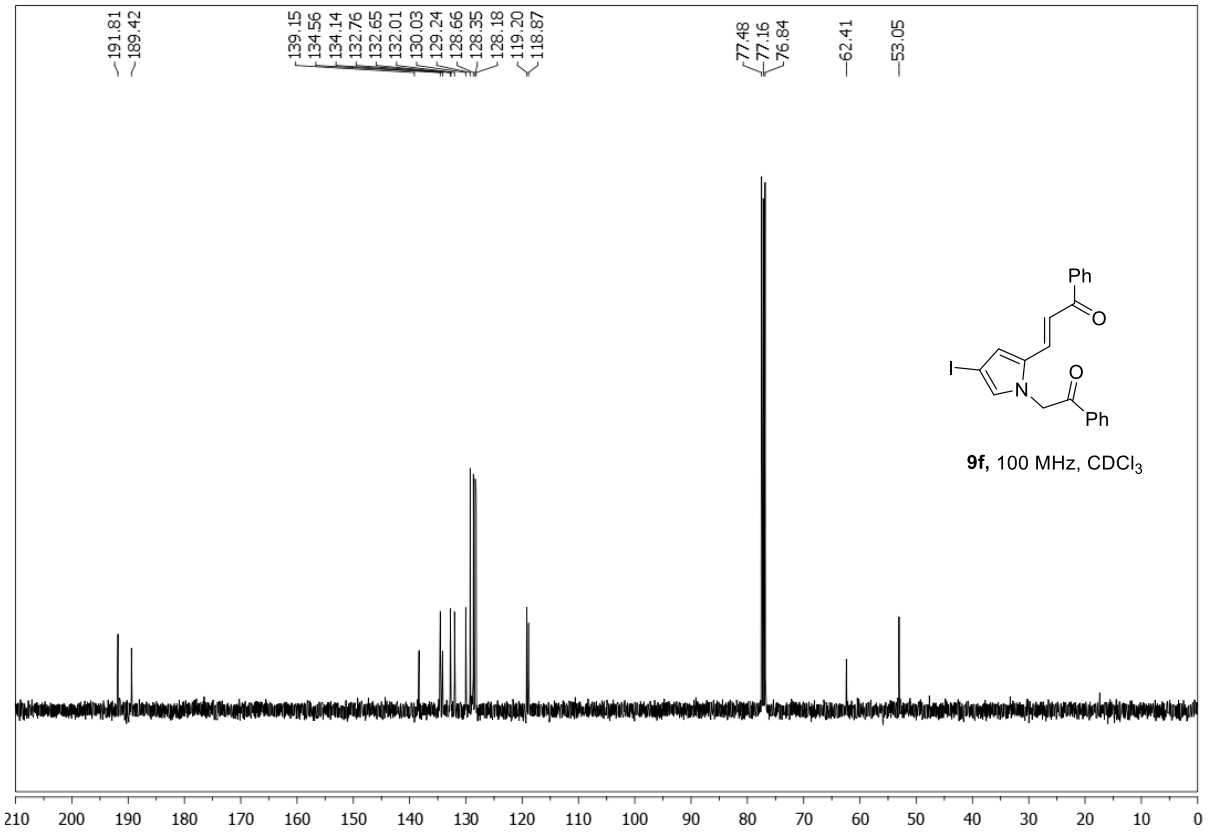
(*E*)-3-(1-(2-Oxo-2-phenylethyl)-1H-pyrrol-2-yl)-1-phenylprop-2-en-1-one (9a)



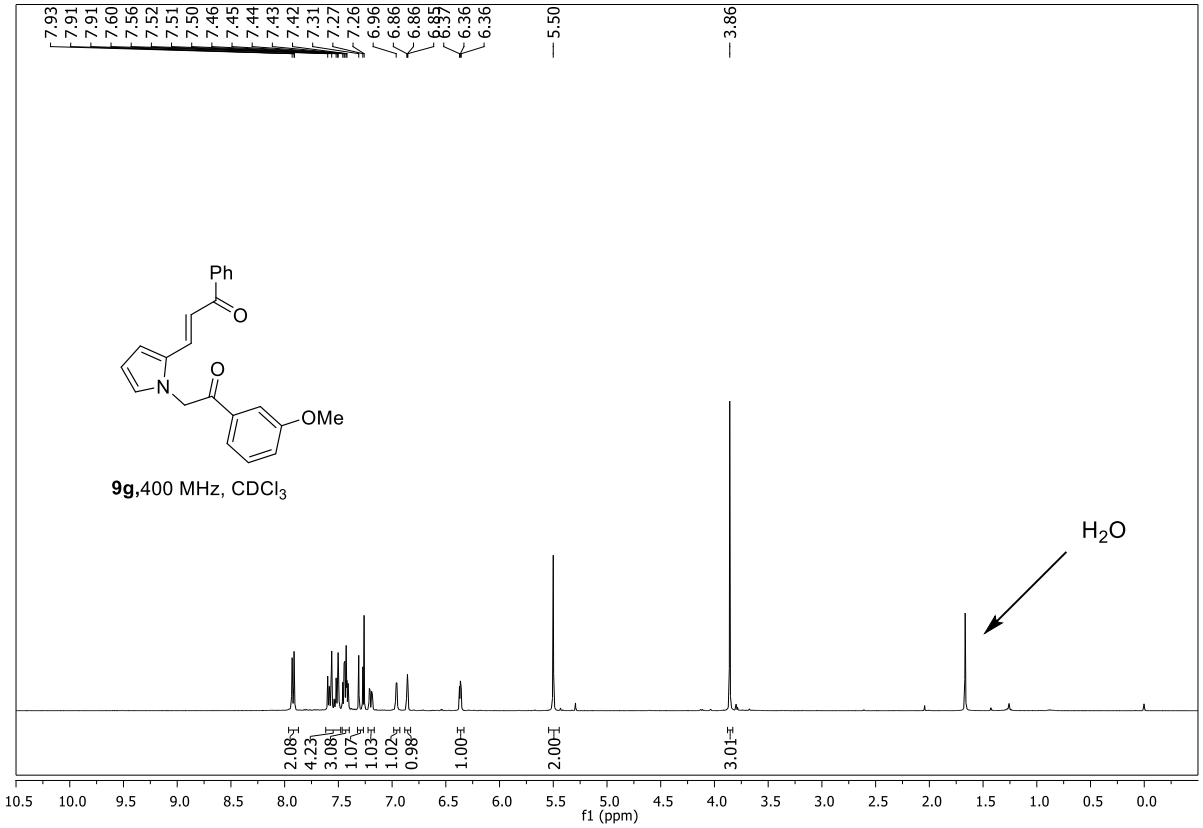


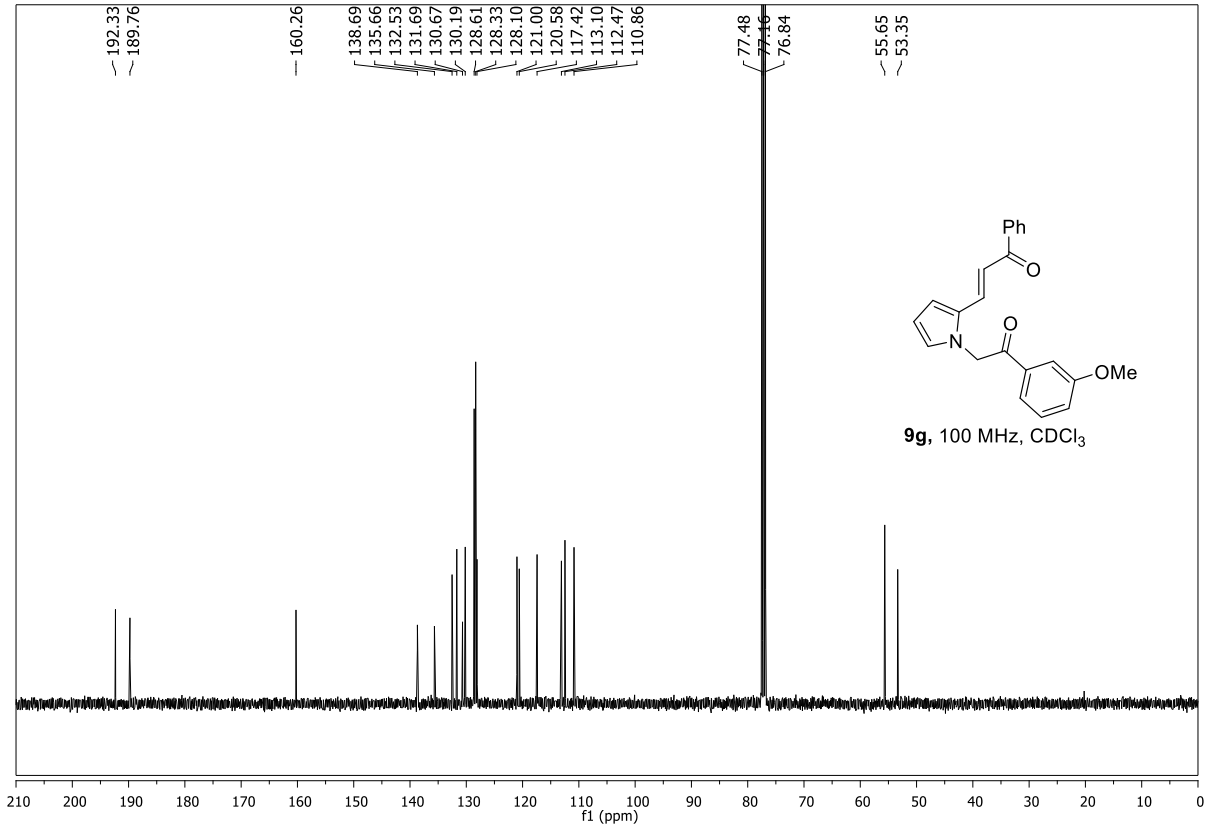
(*E*)-3-(4-Iodo-1-(2-oxo-2-phenylethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one (9f)



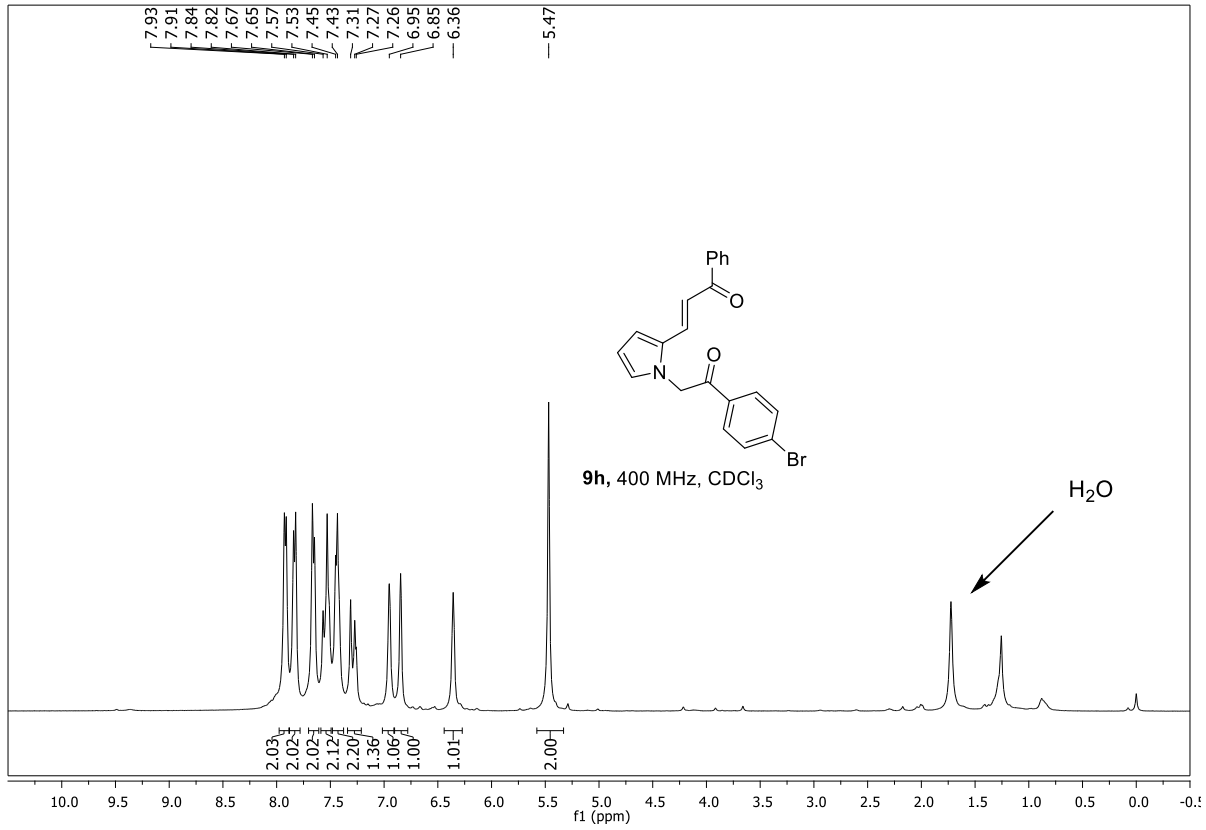


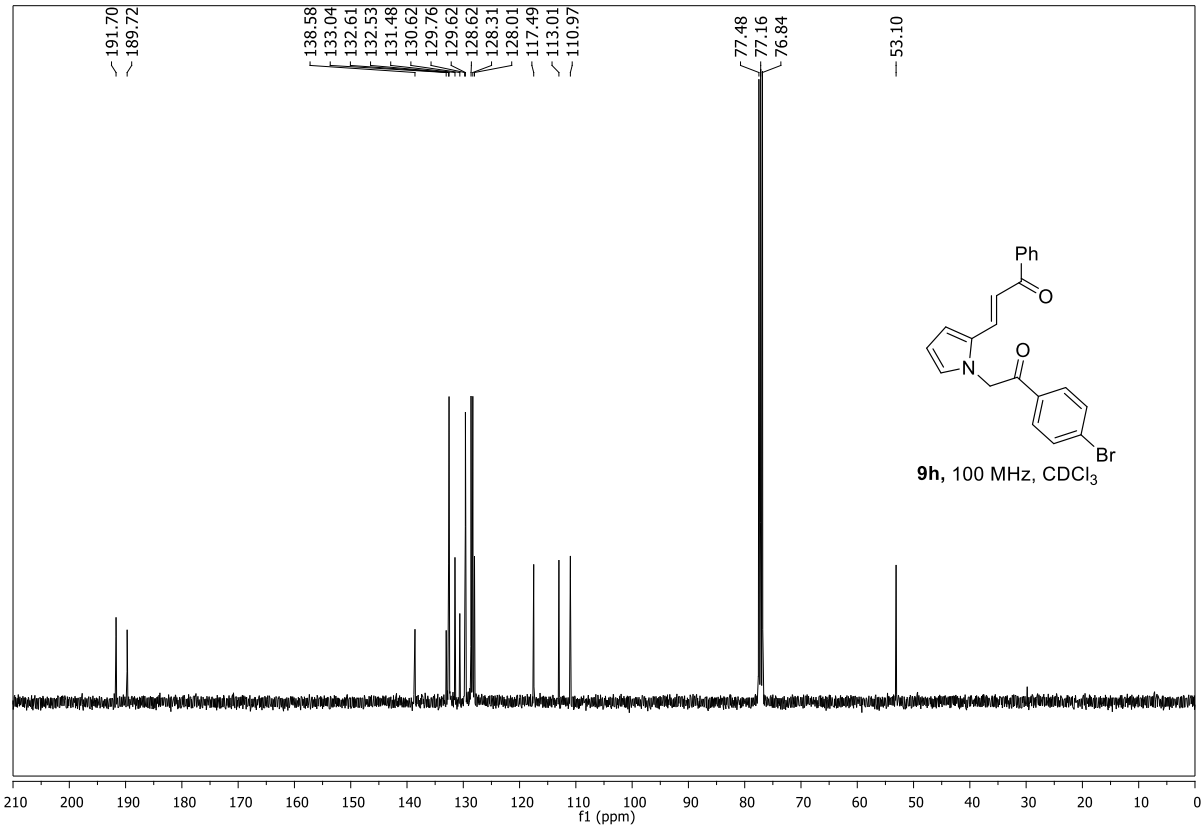
(*E*)-3-(1-(2-(3-Methoxyphenyl)-2-oxoethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one (9g)





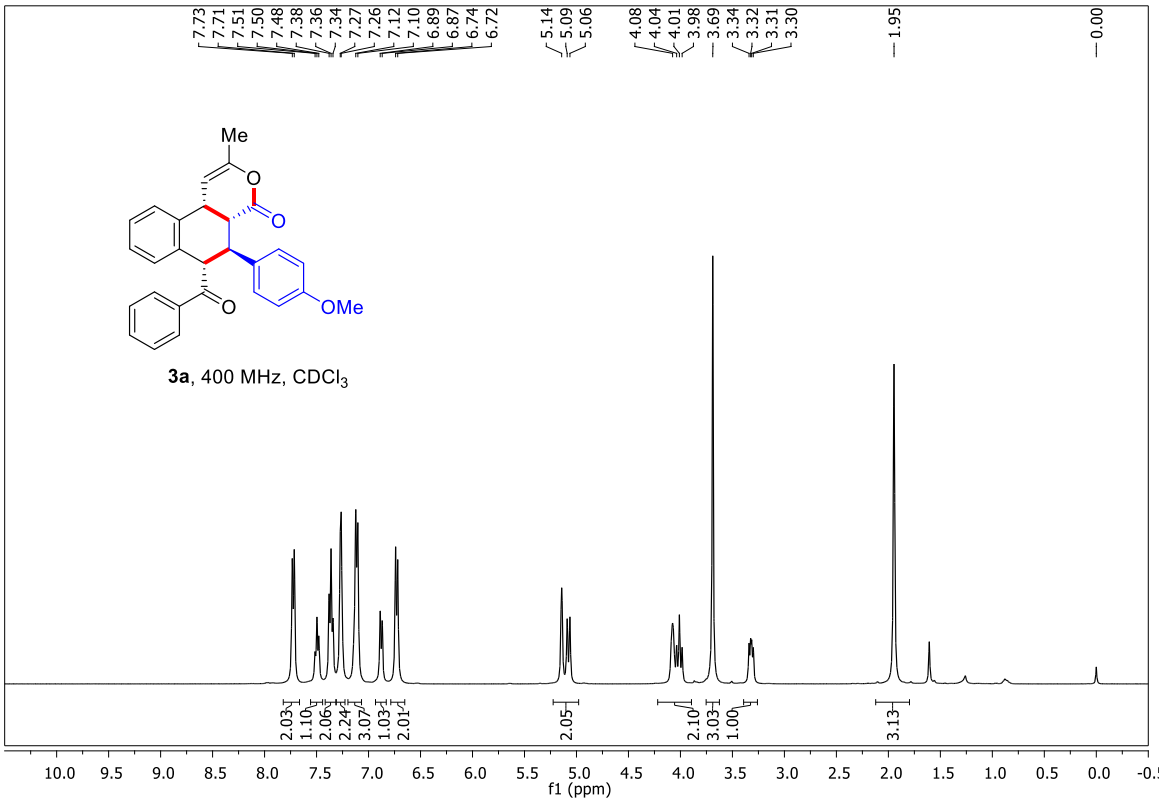
(*E*)-3-(1-(2-(4-Bromophenyl)-2-oxoethyl)-1*H*-pyrrol-2-yl)-1-phenylprop-2-en-1-one (9h)

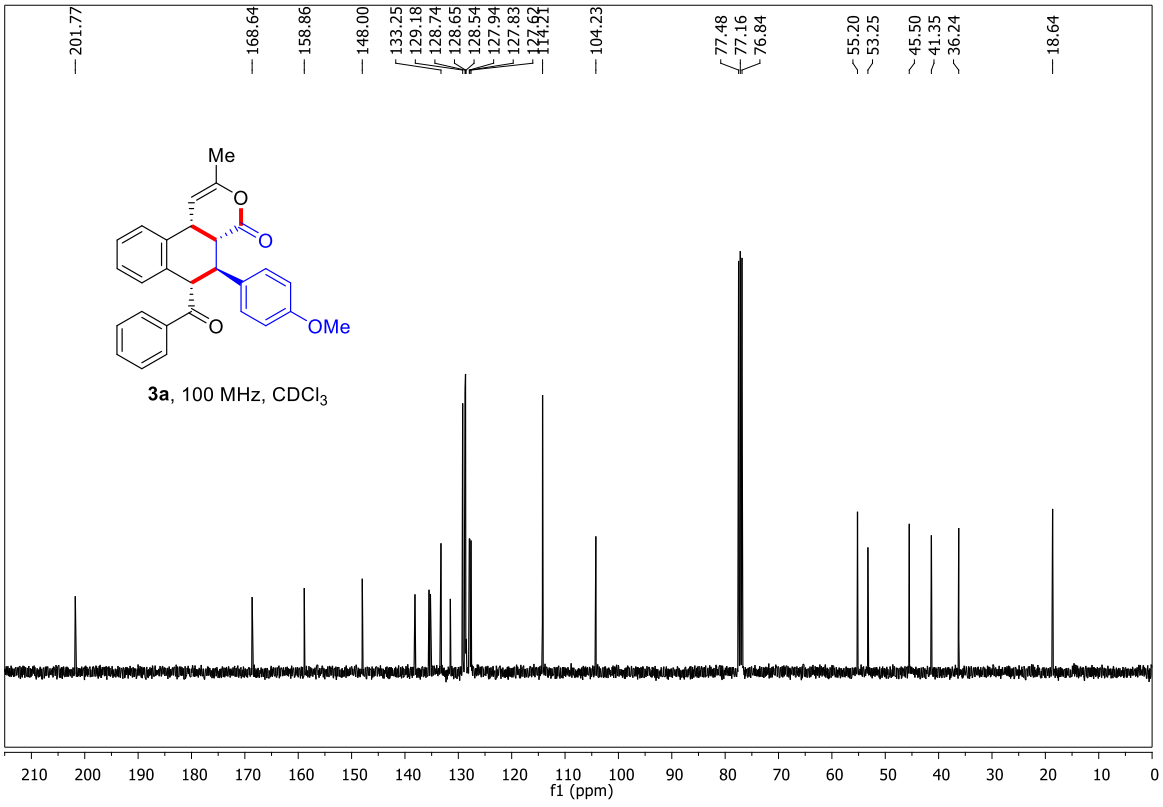




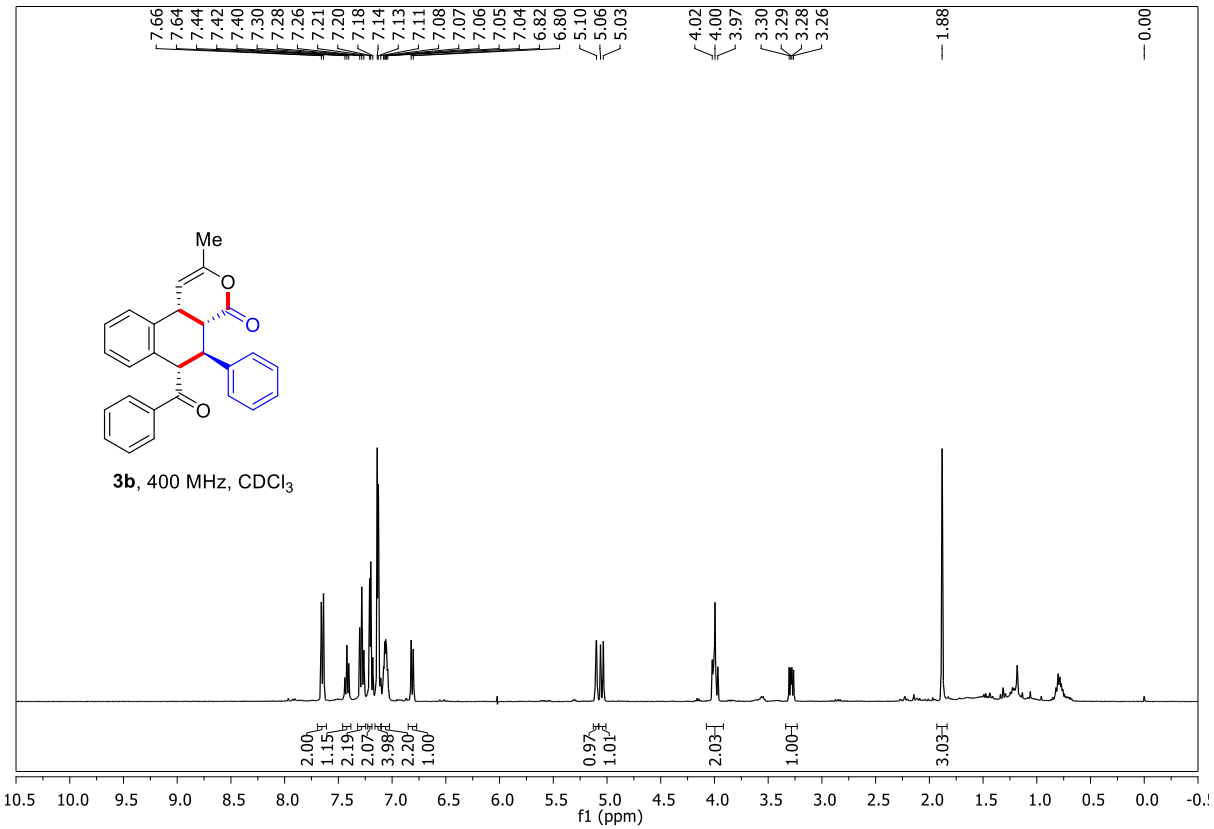
9. ^{1}H and ^{13}C NMR Spectra of Functionalized Tricyclic δ- Lactones

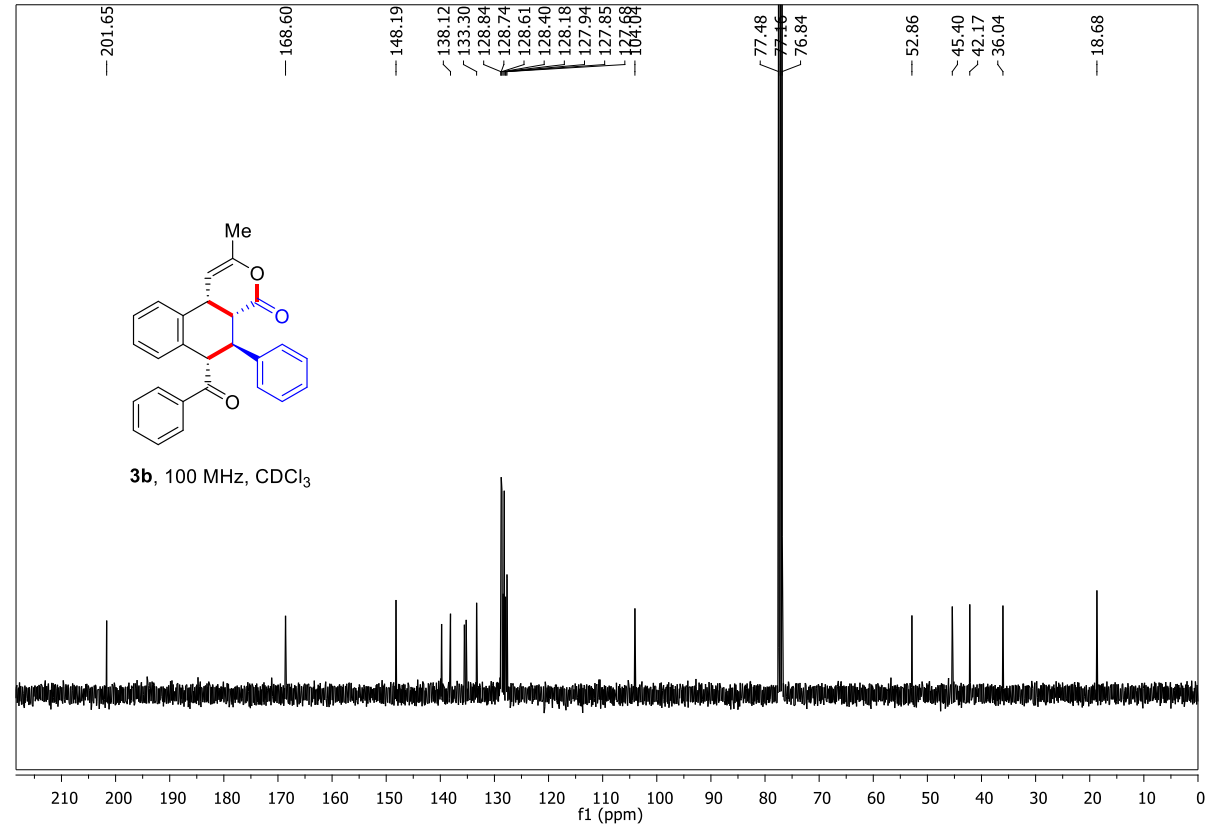
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3a)



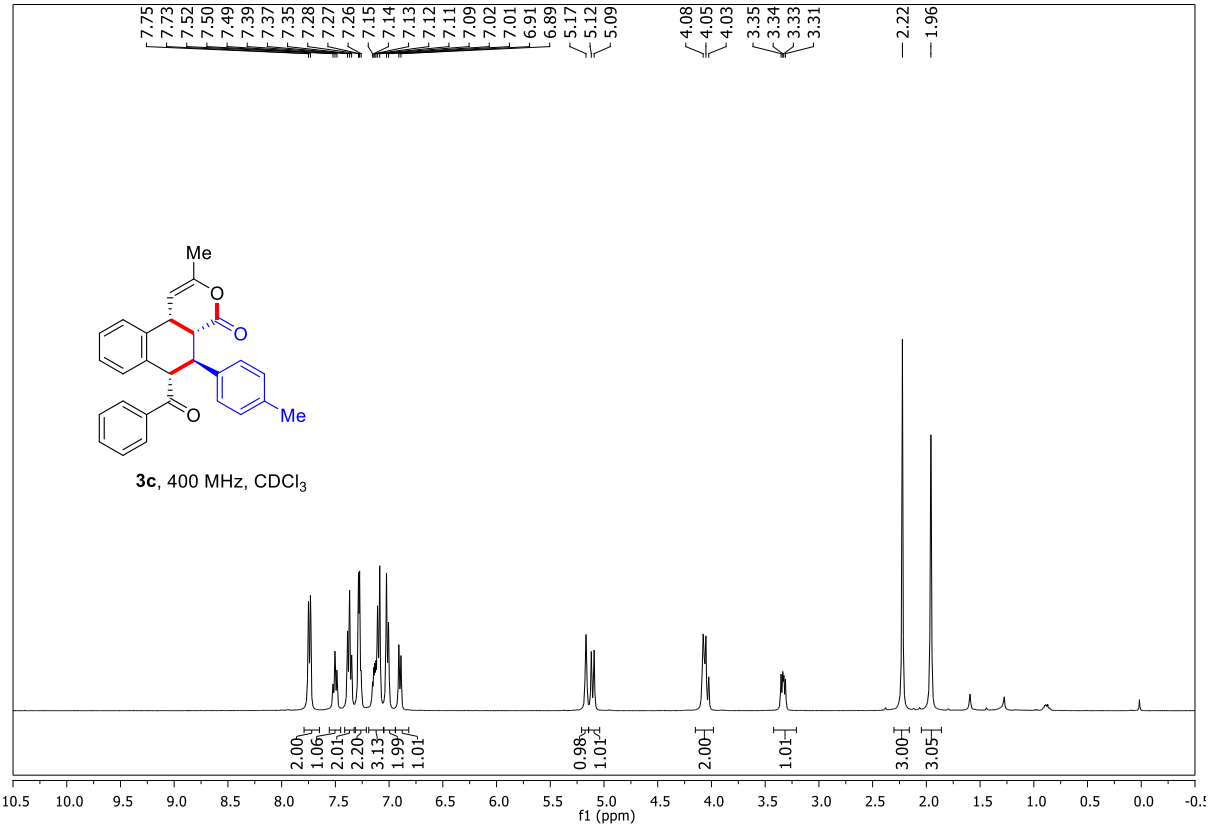


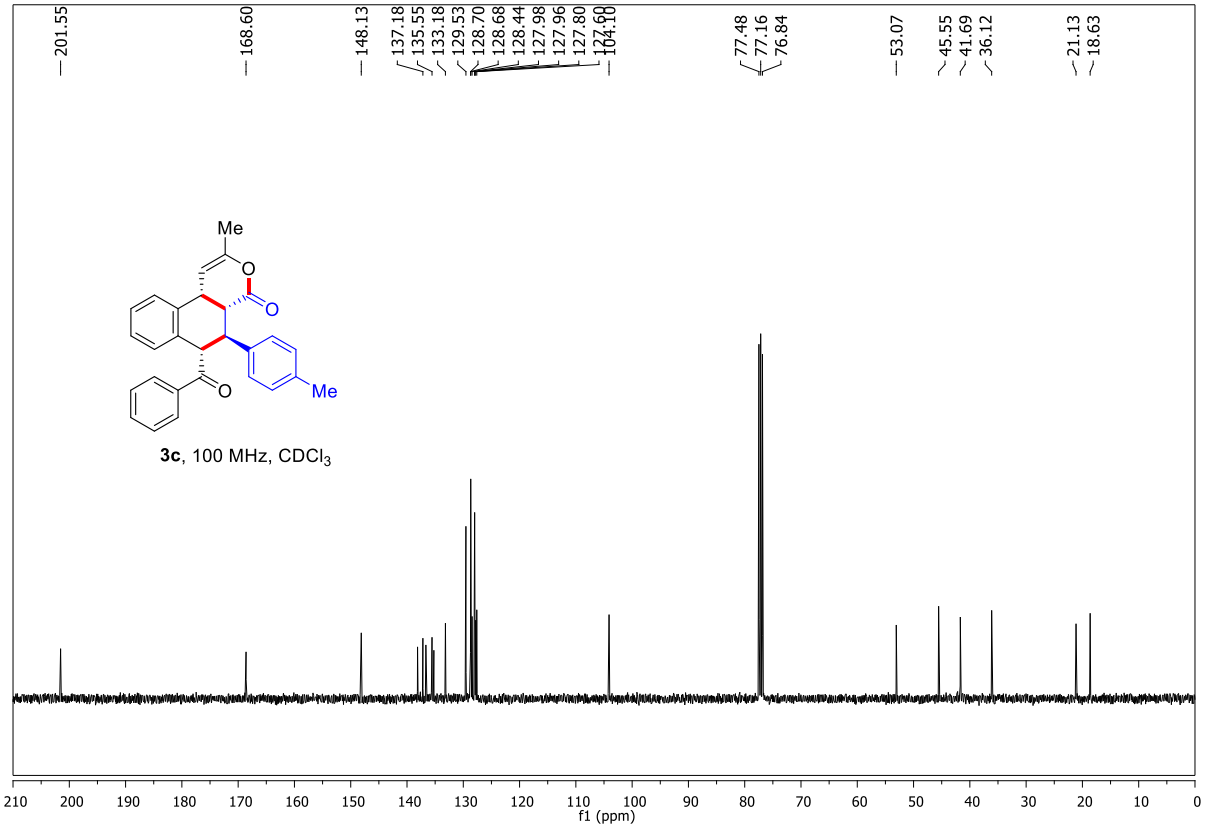
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-phenyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3b)



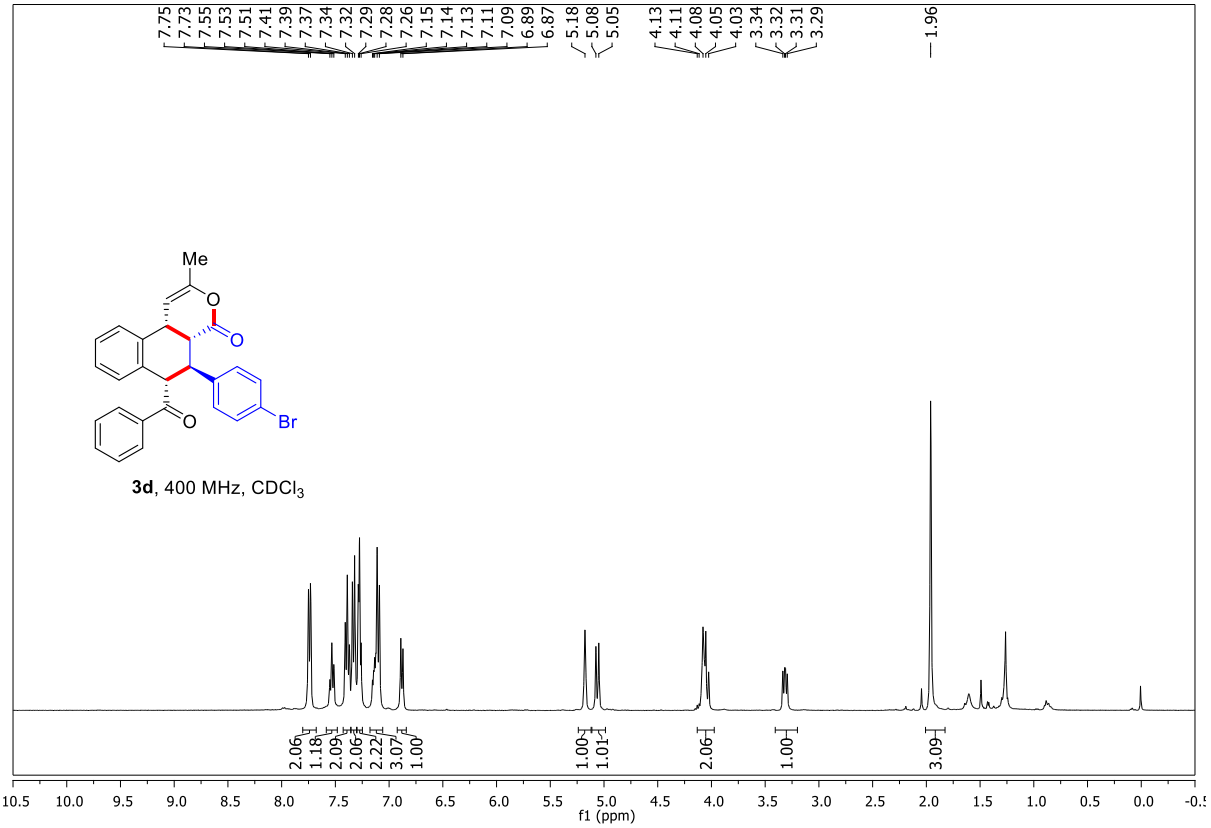


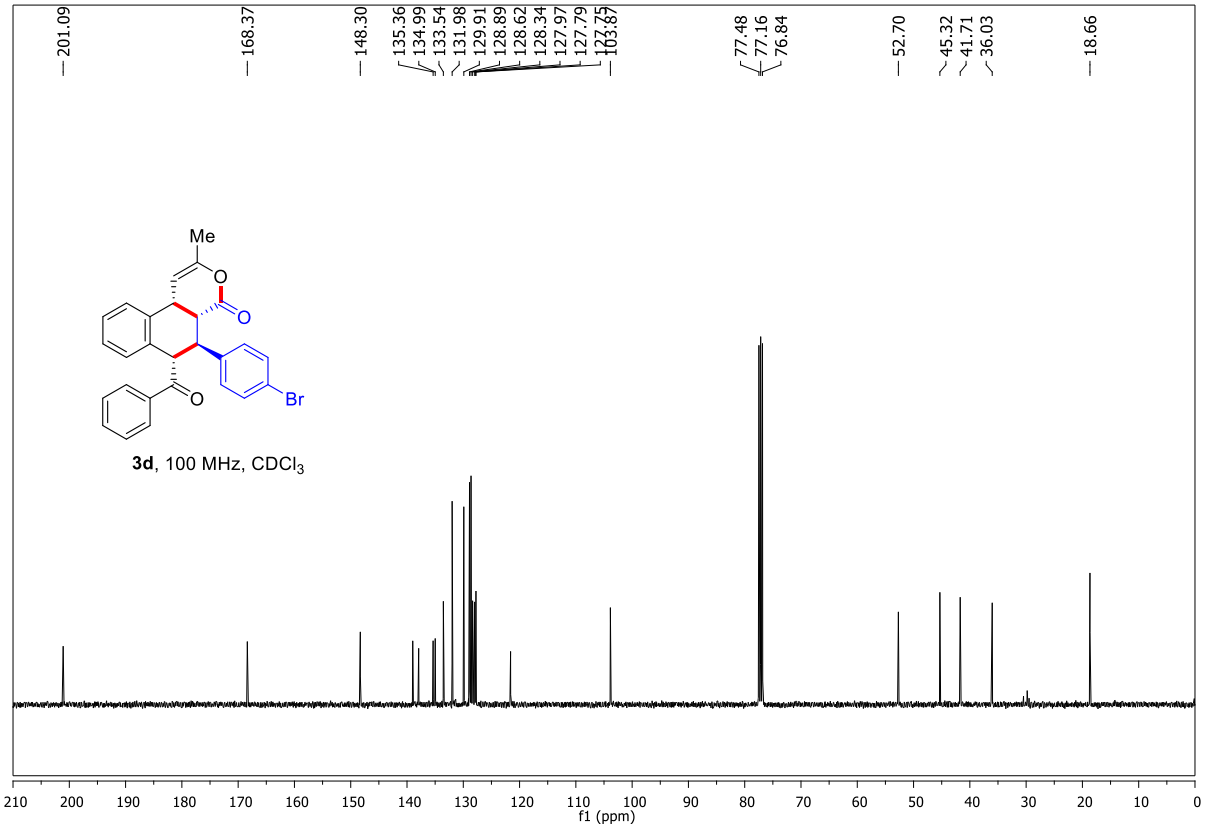
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(*p*-tolyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3c)



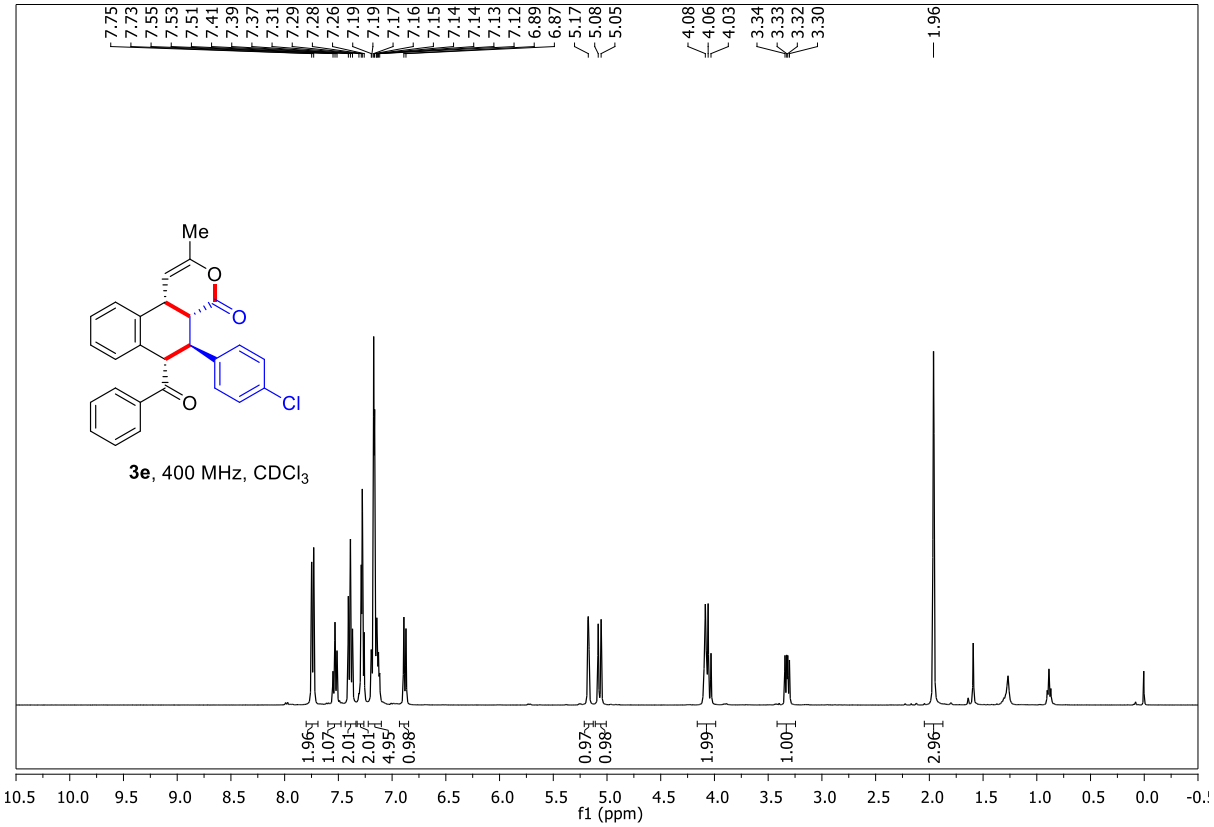


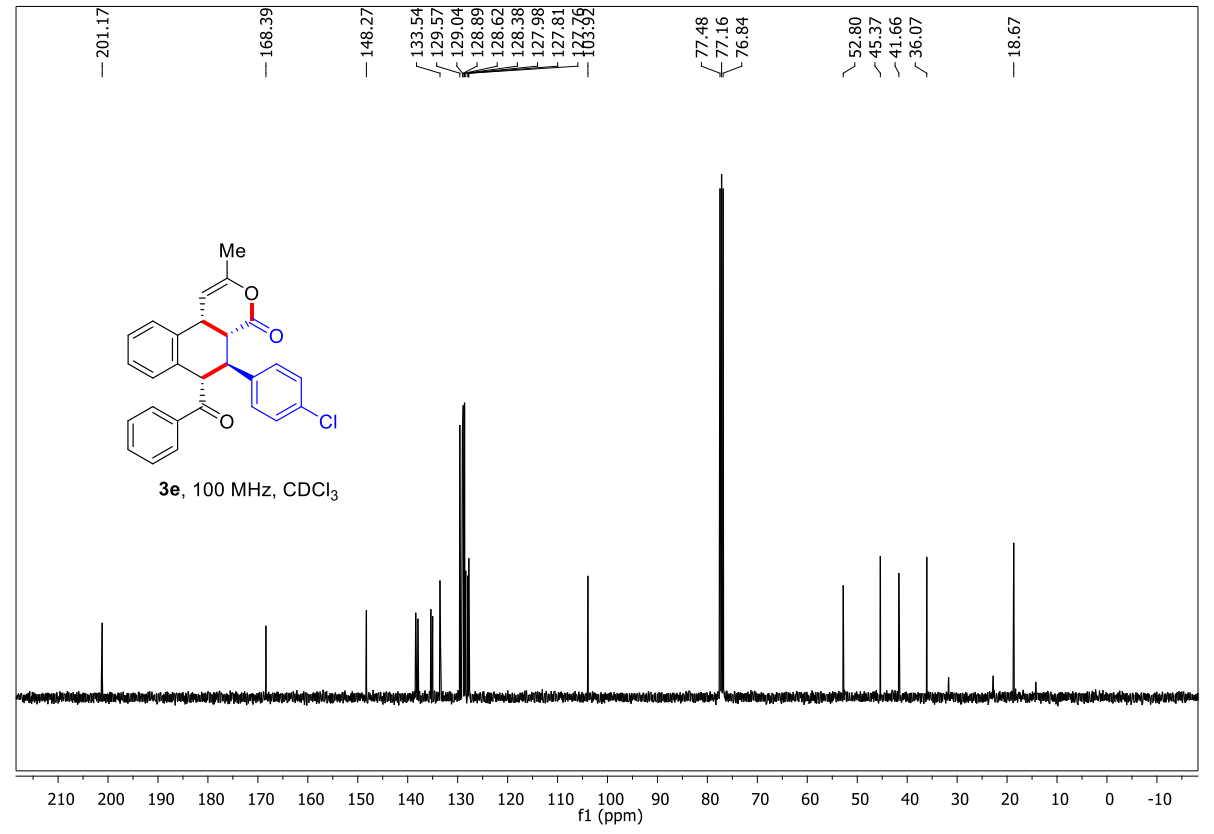
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-bromophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3d)



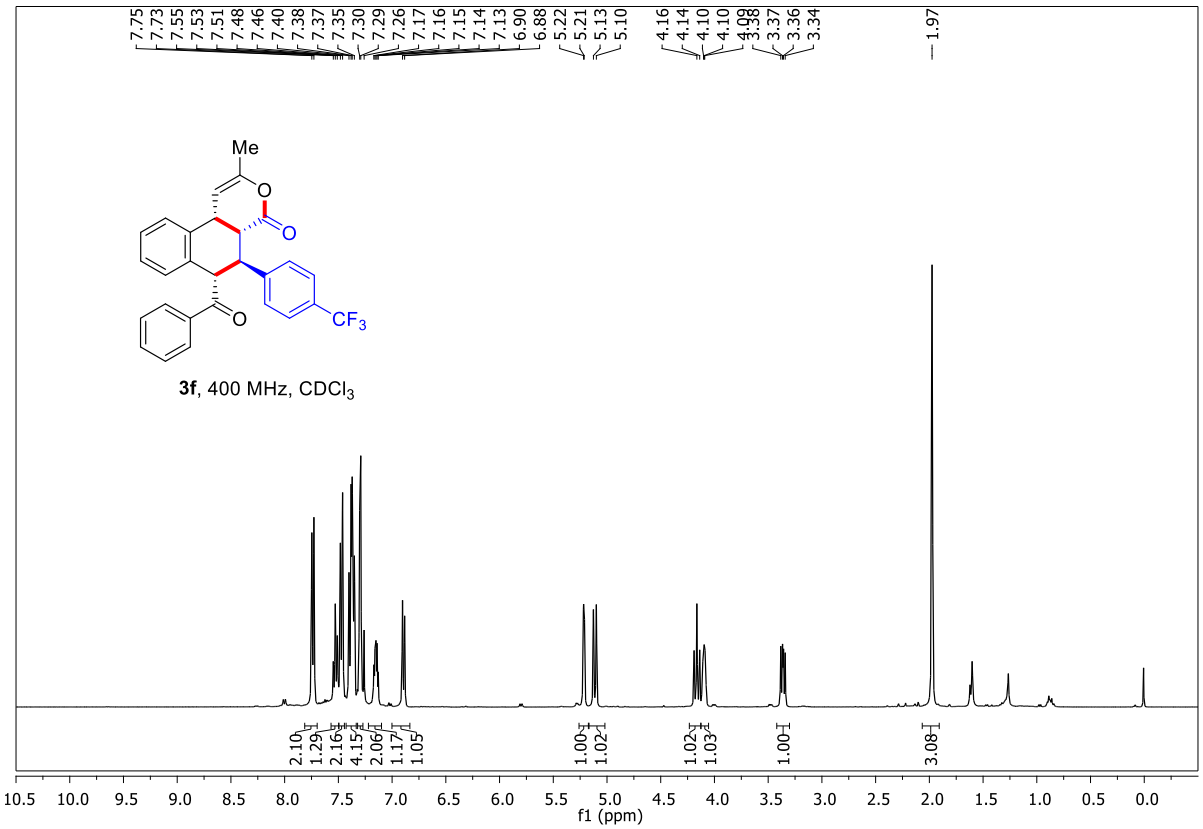


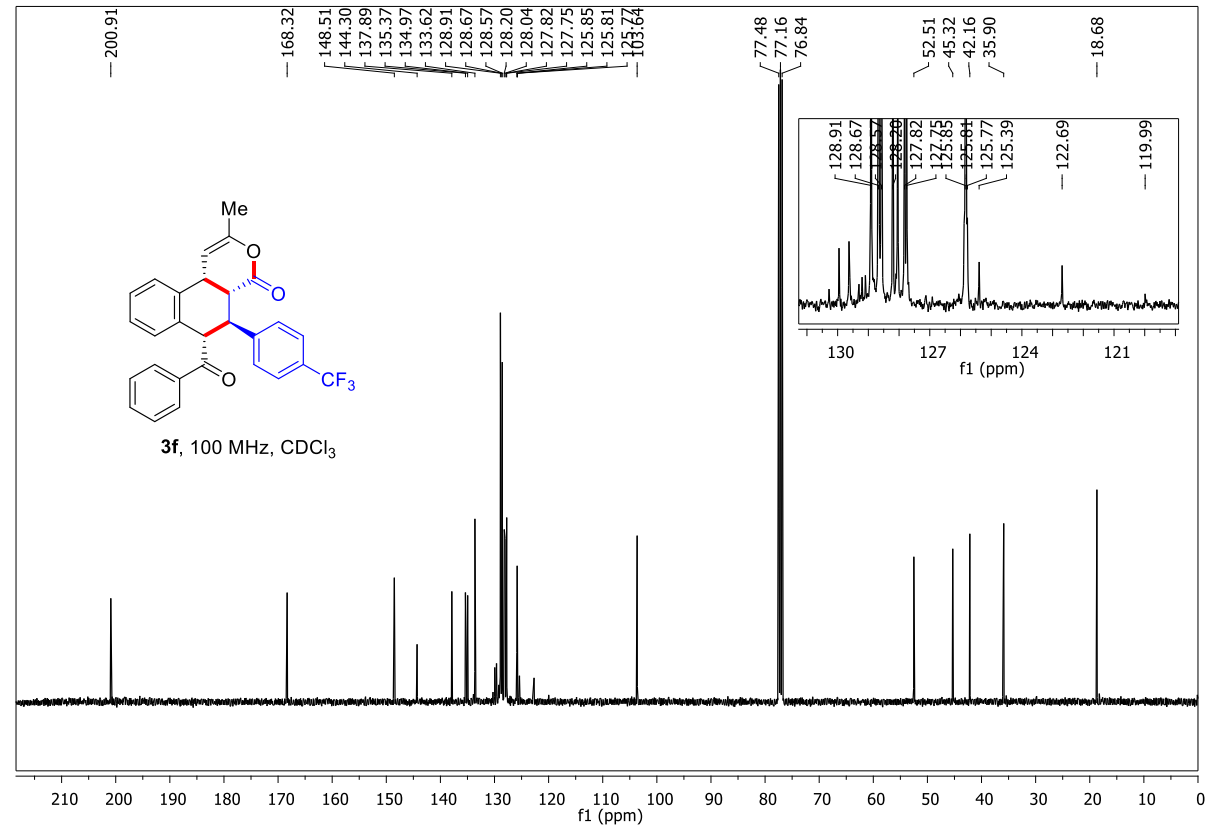
(4a*R*,5S,6*S*,10b*R*)-6-Benzoyl-5-(4-chlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3e)



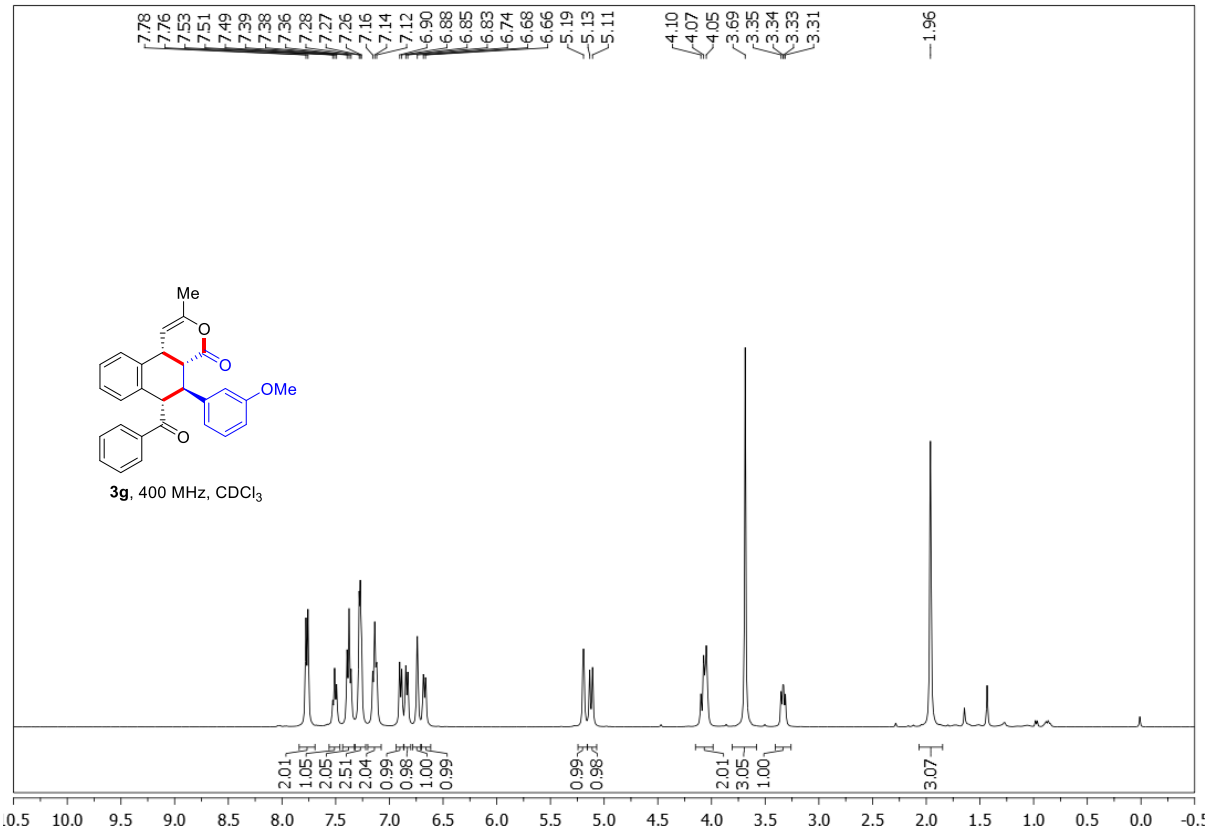


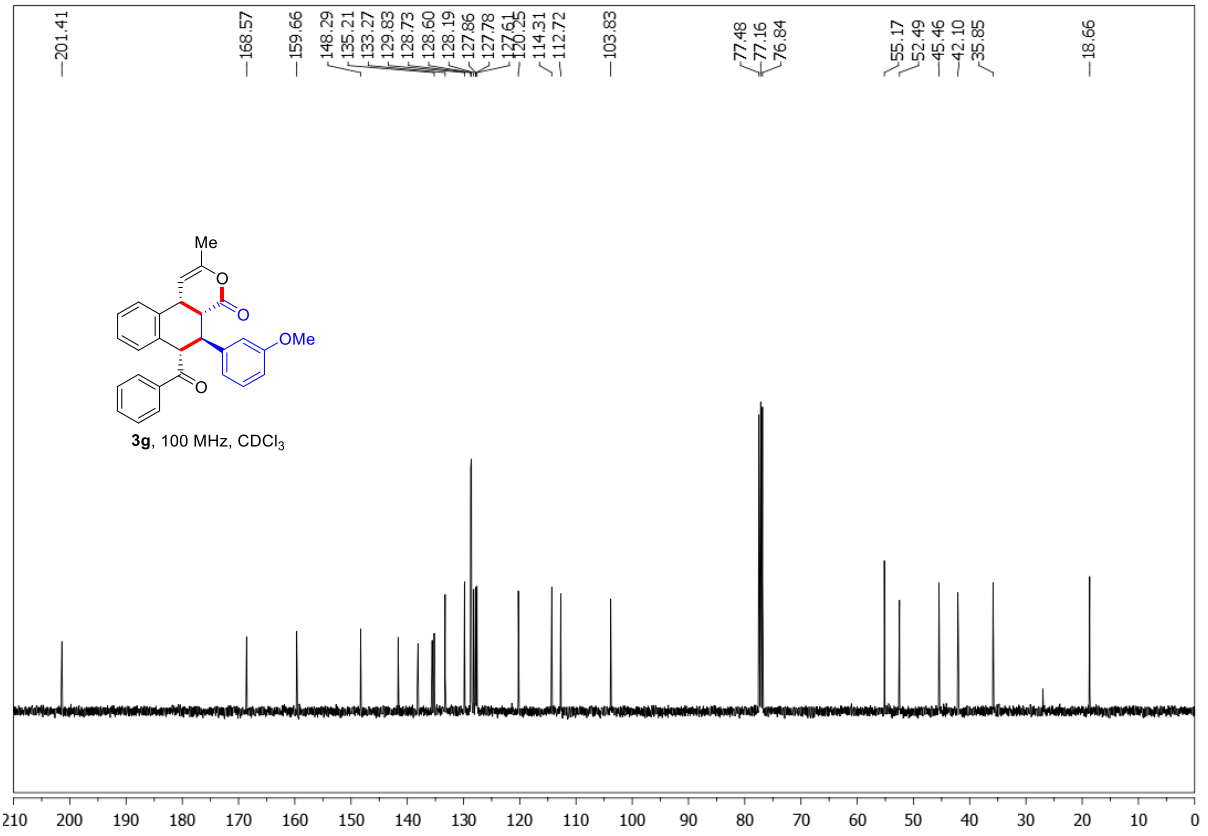
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(4-(trifluoromethyl)phenyl)-4a,5,6,10b-tetrahydro-4H-benzo[*f*]isochromen-4-one (3f)



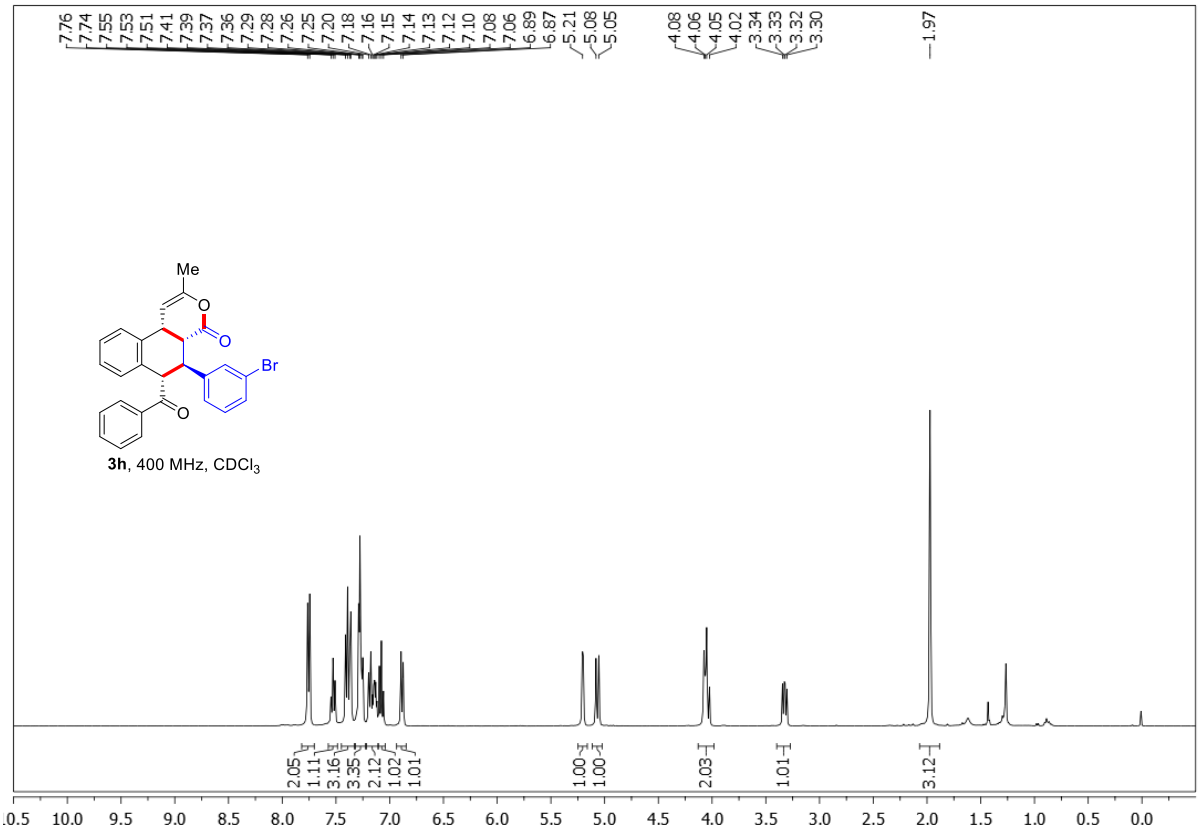


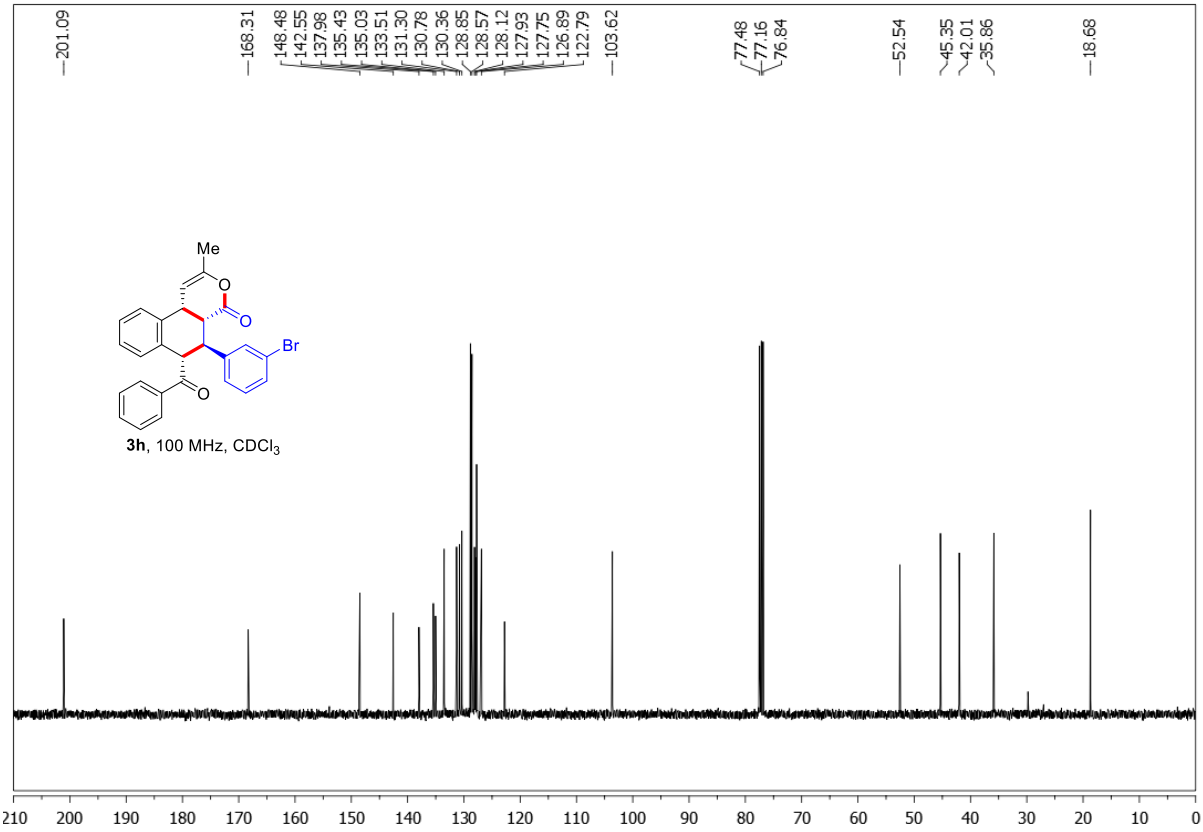
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3g)



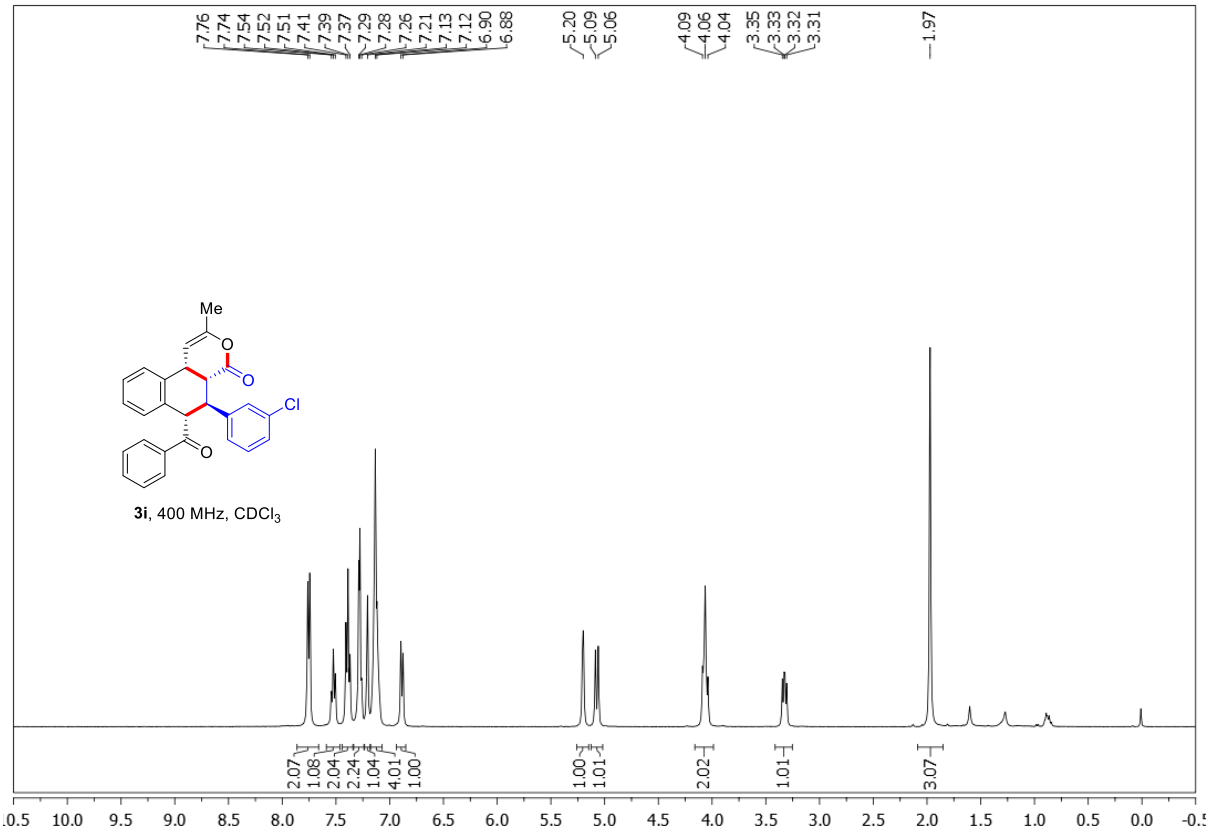


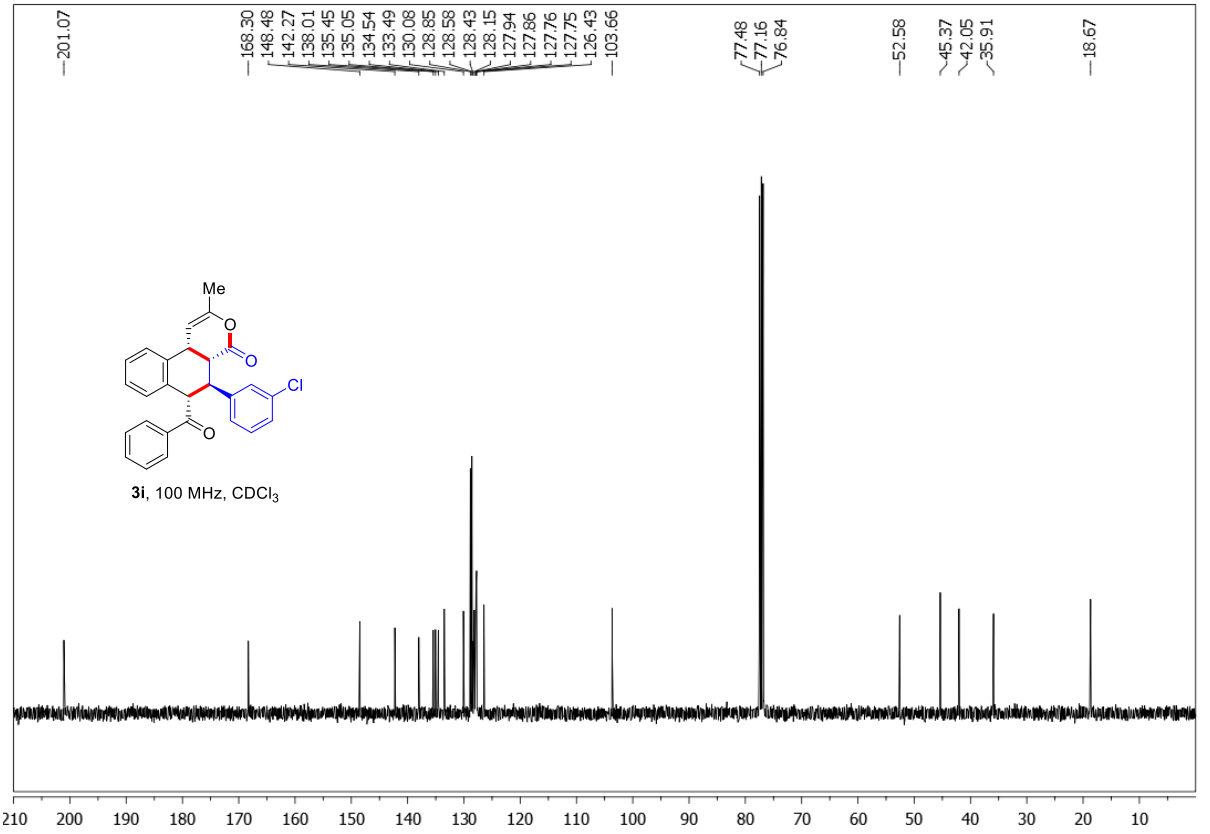
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3-bromophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3h)



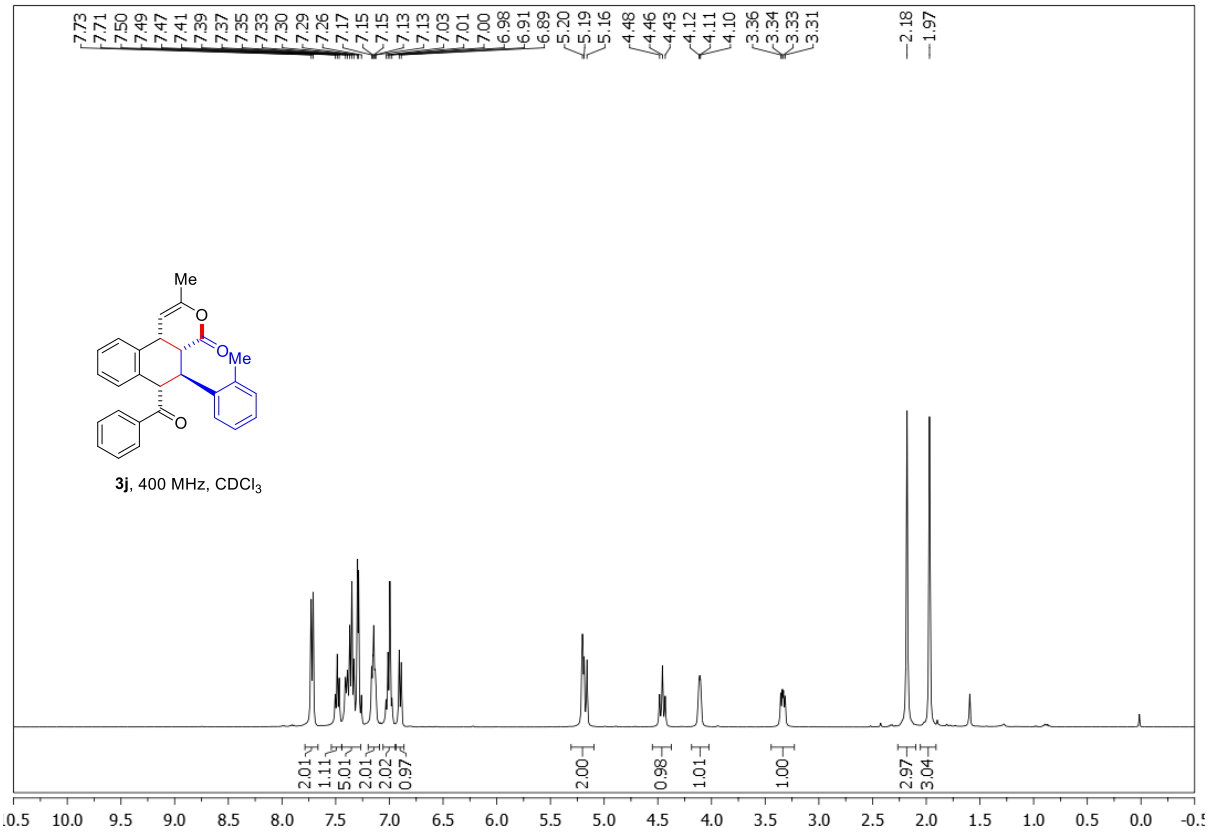


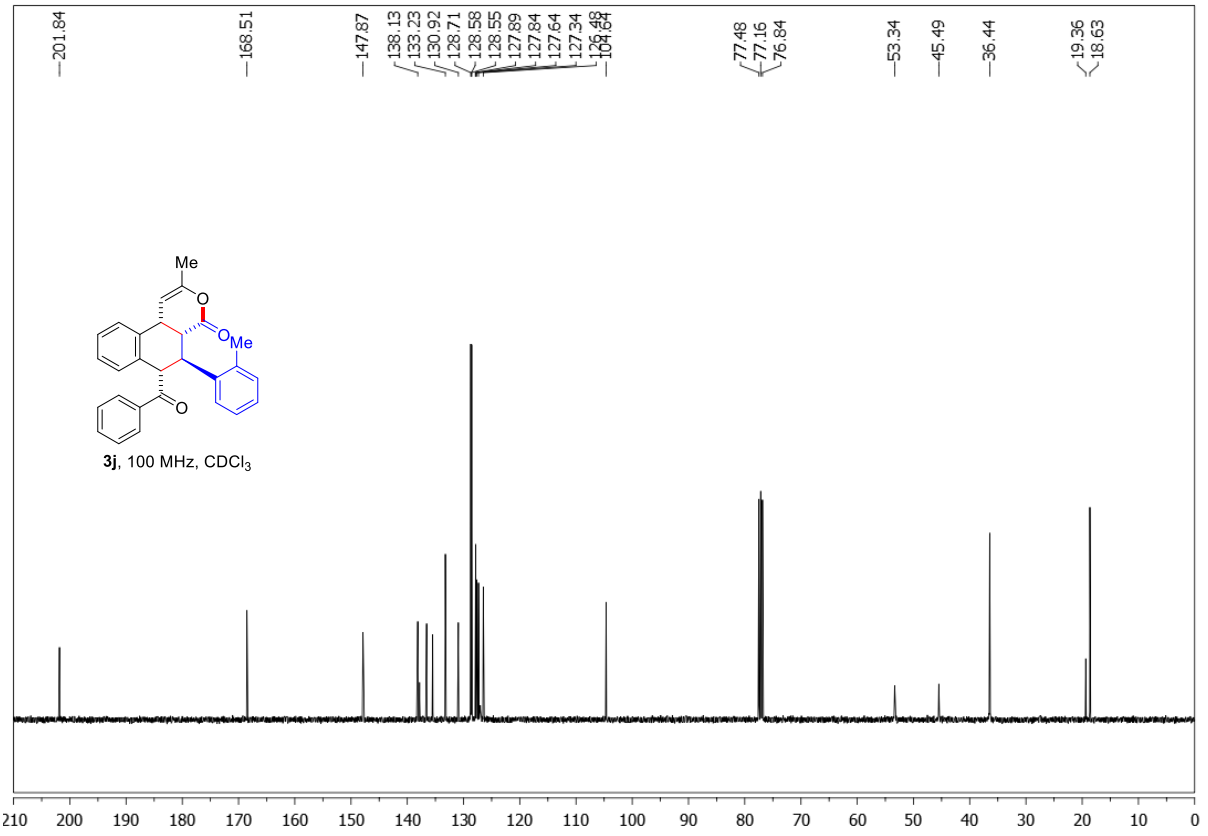
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3-chlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3i)



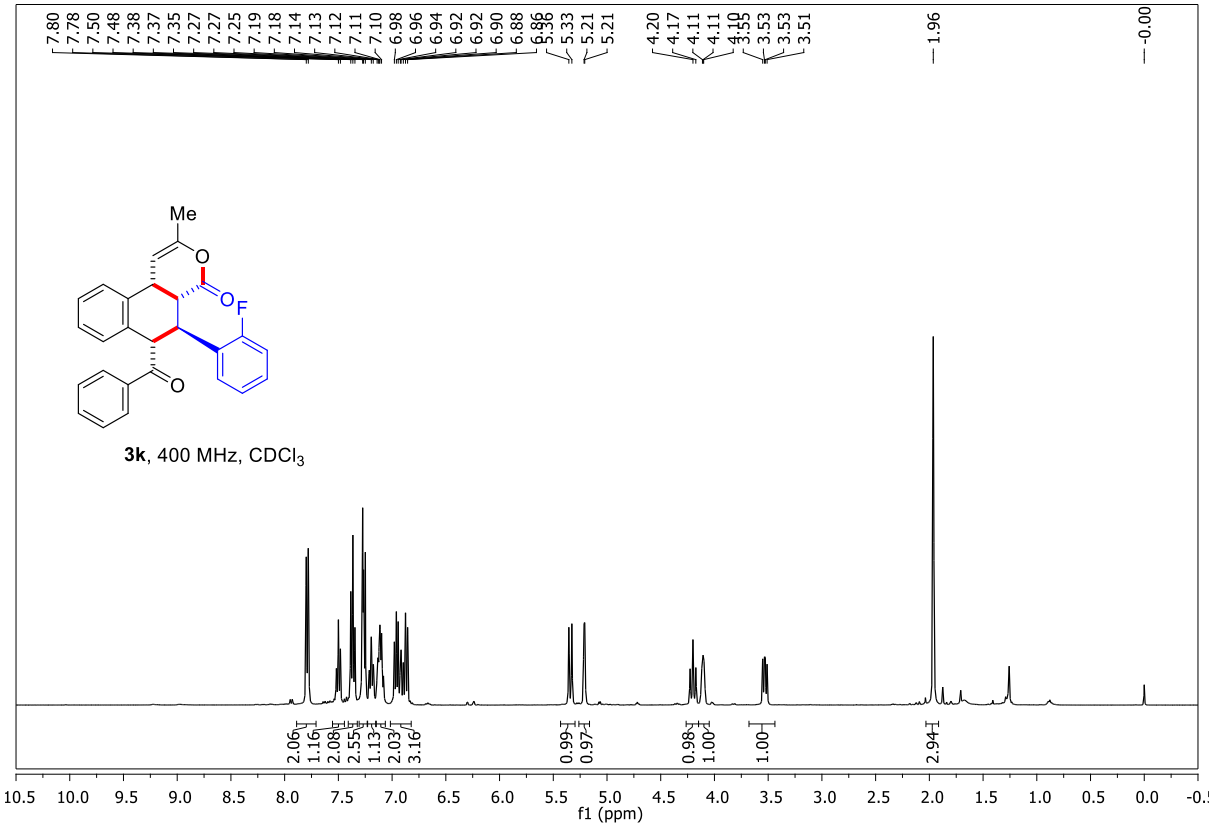


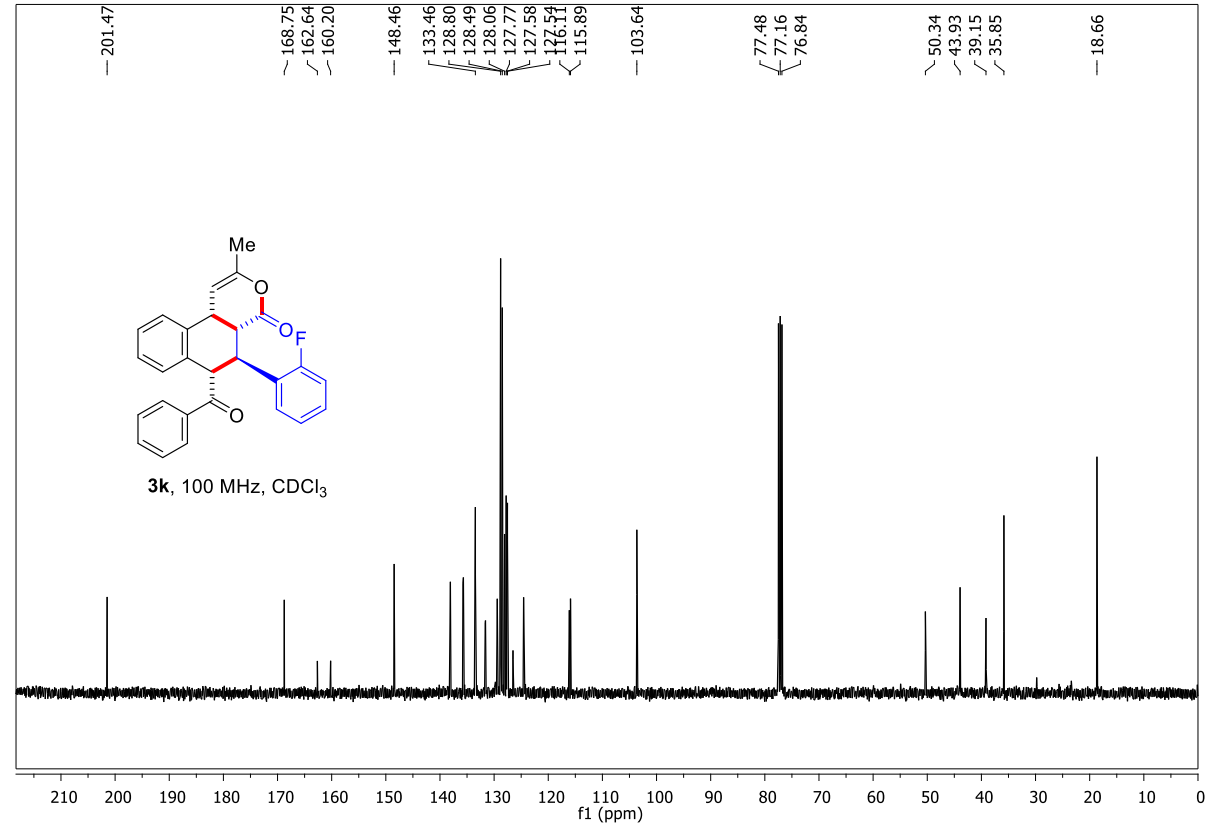
(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(o-tolyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3j)



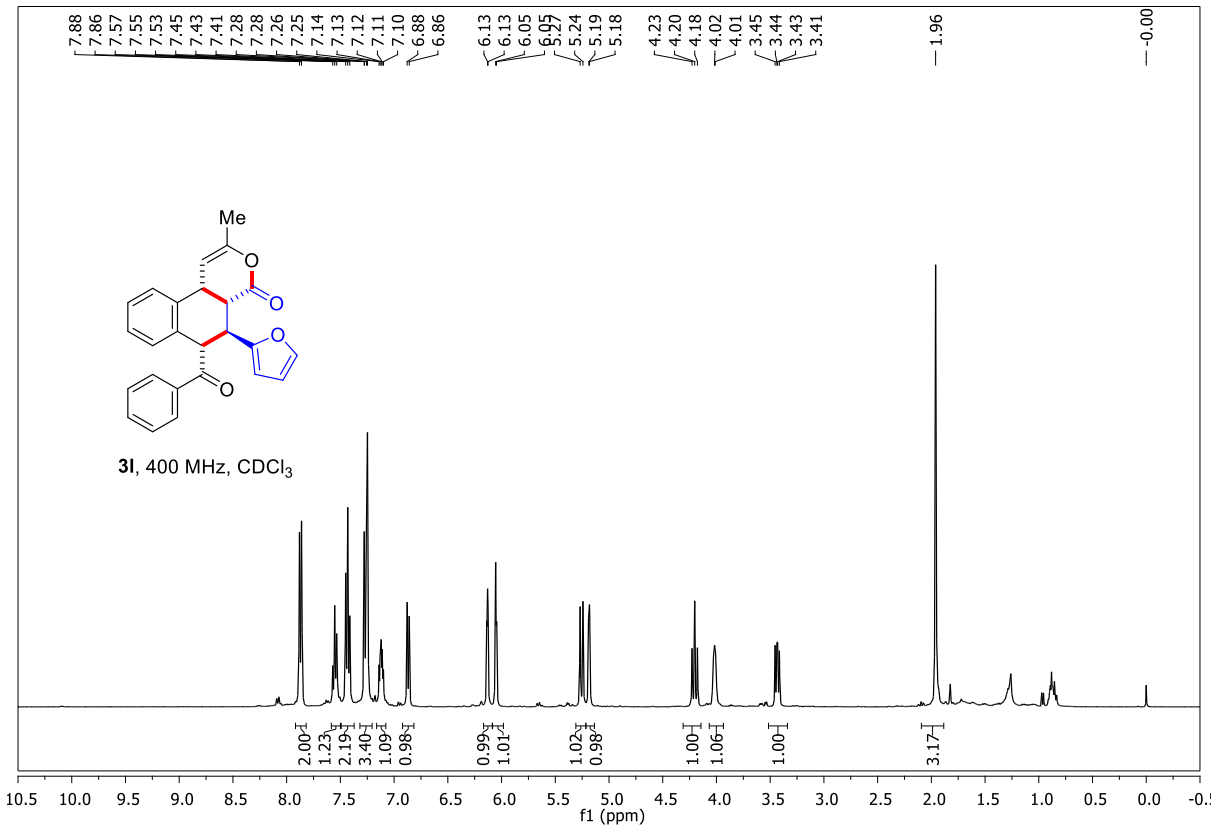


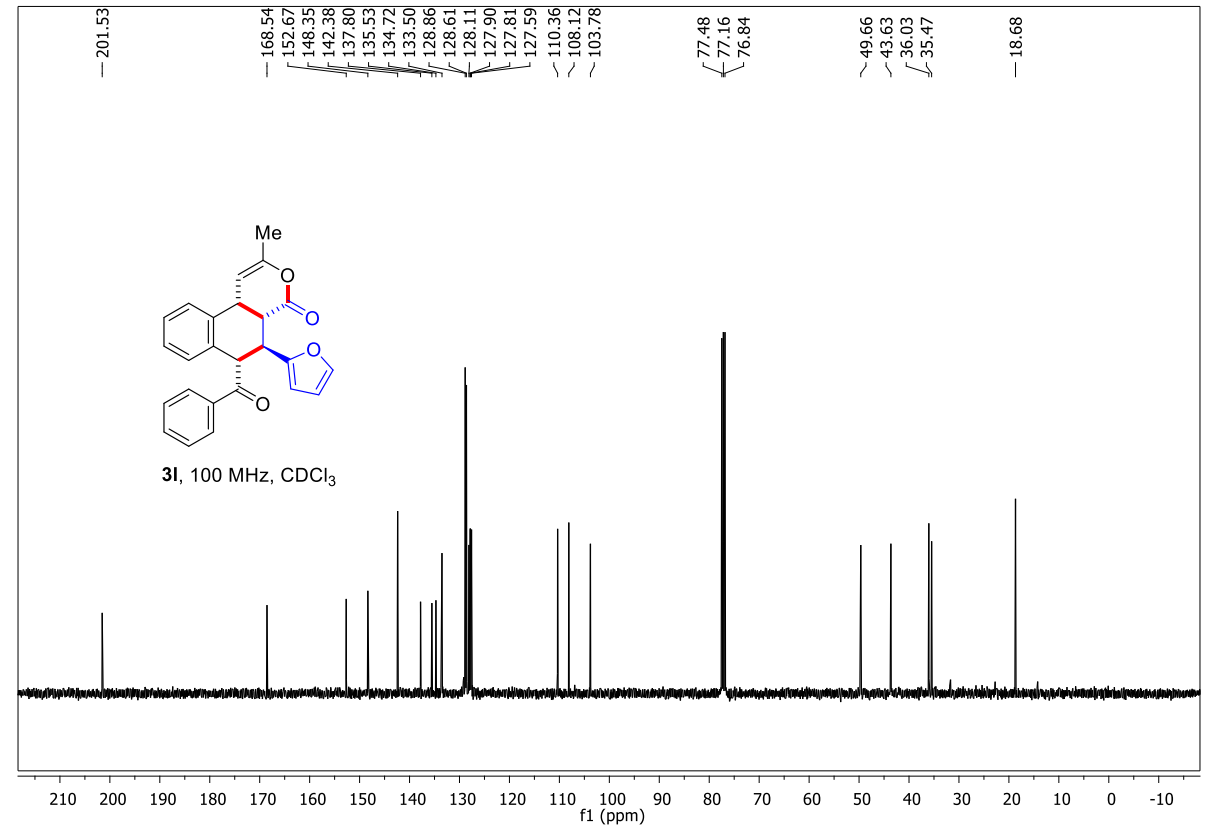
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(2-fluorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3k)



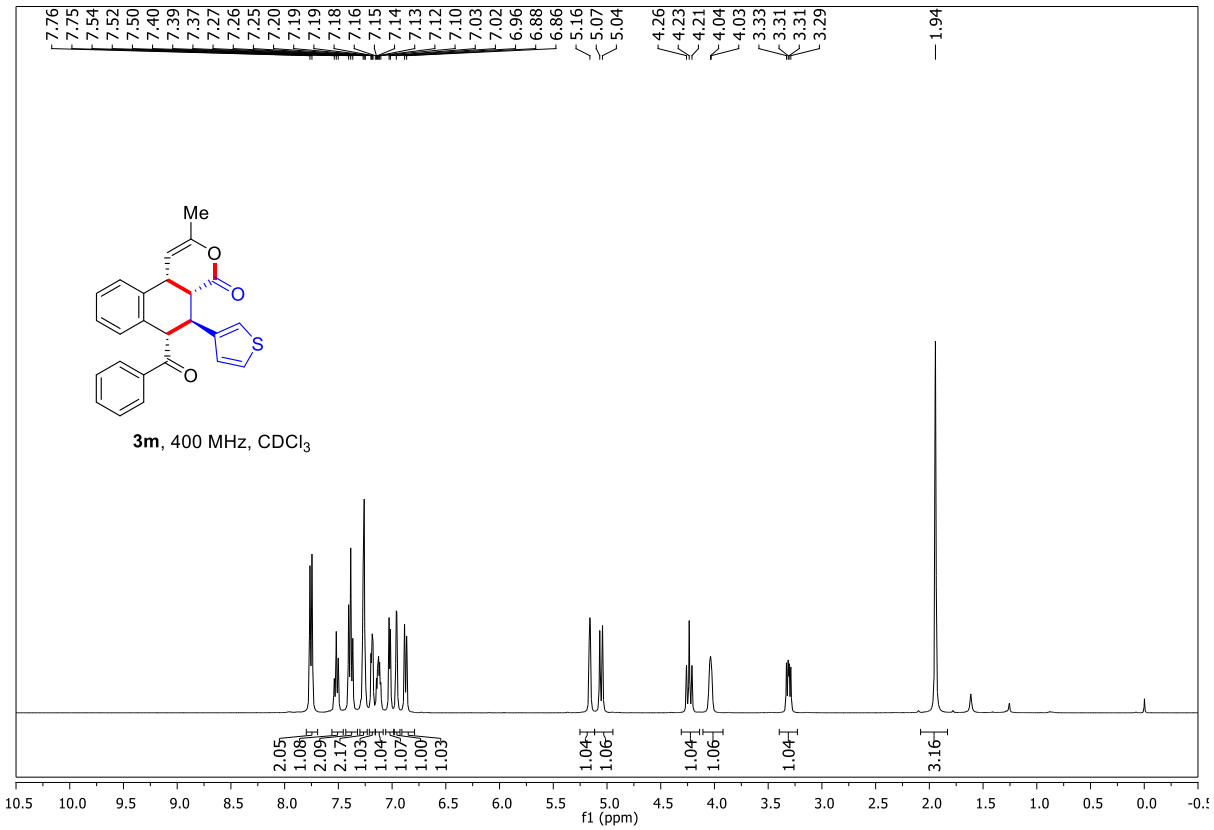


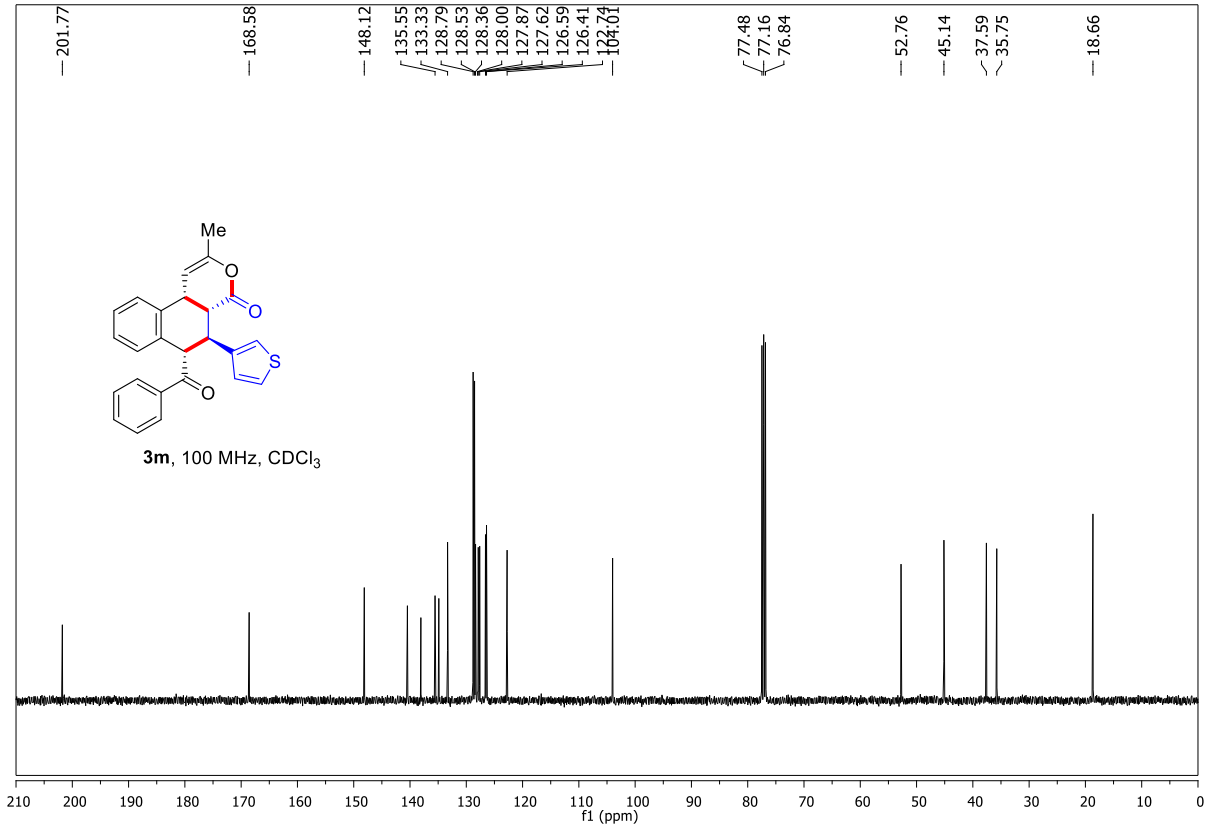
(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(furan-2-yl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3l)



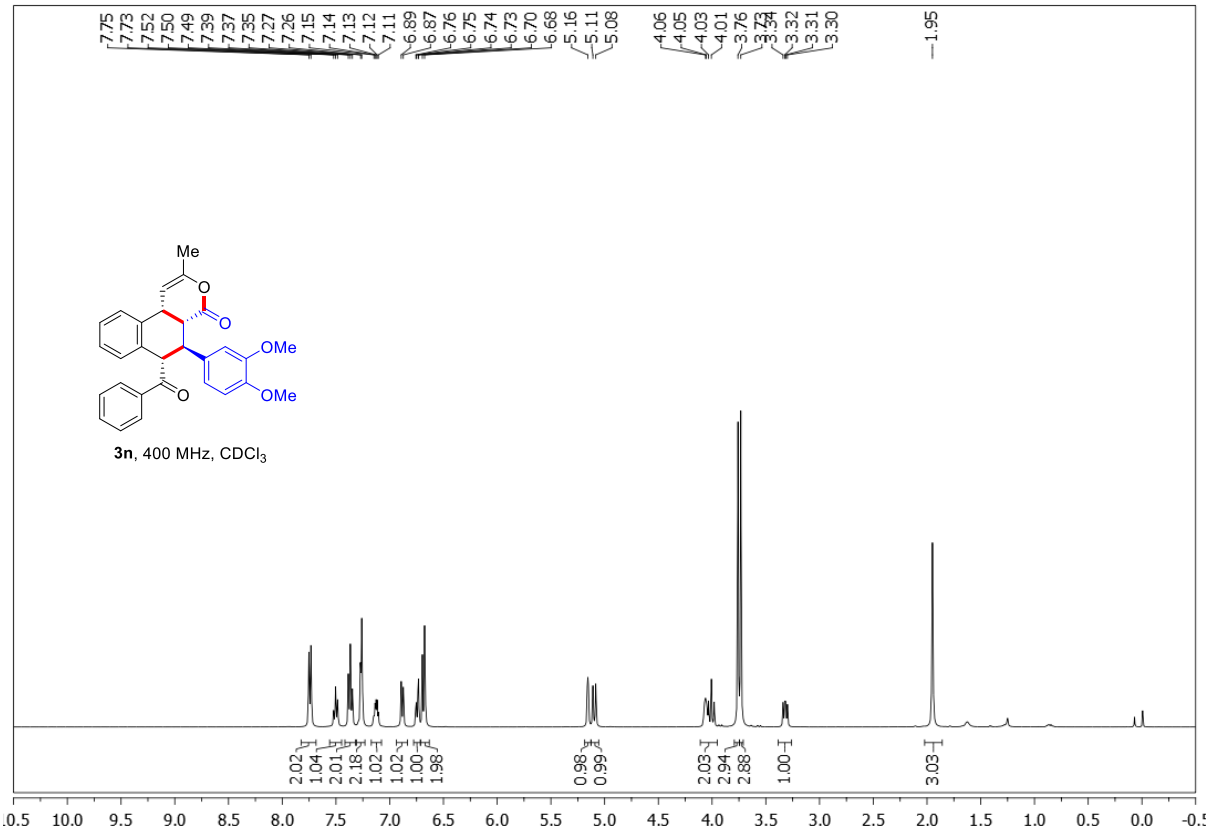


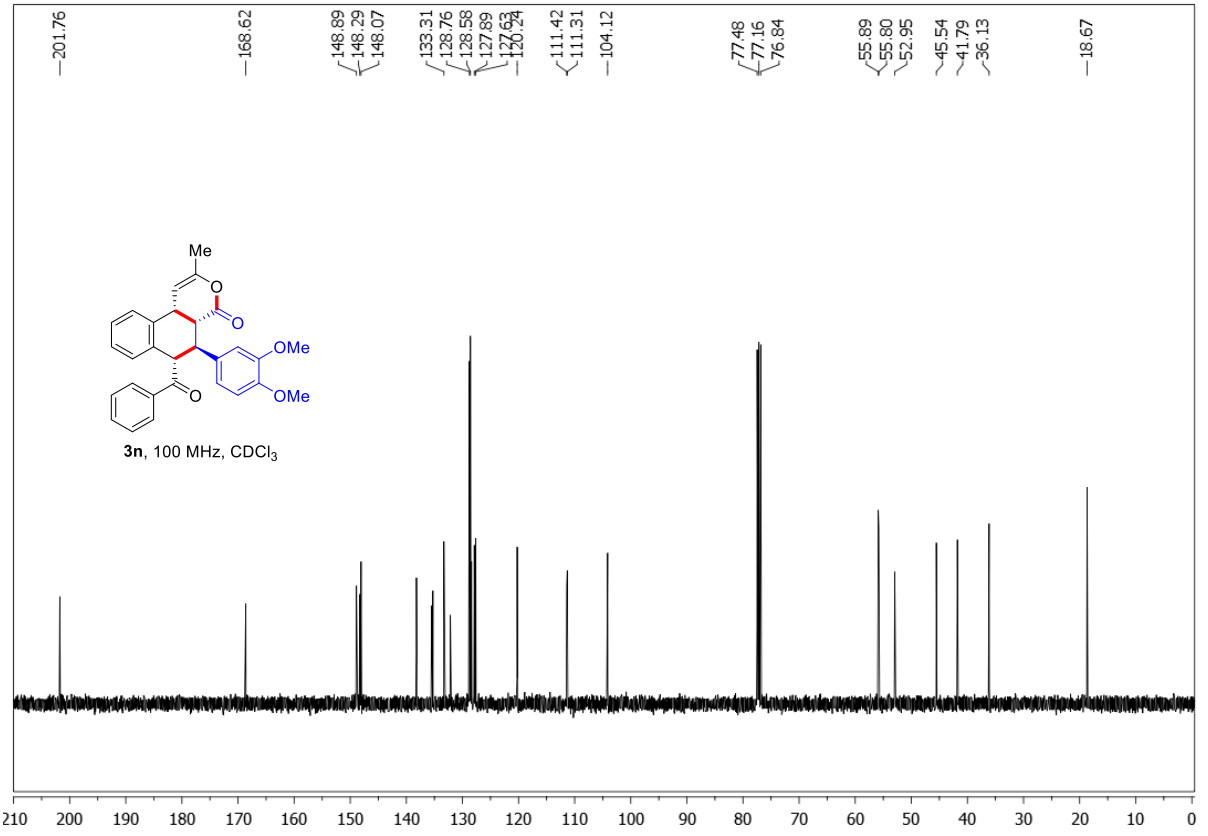
(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(thiophen-3-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3m)



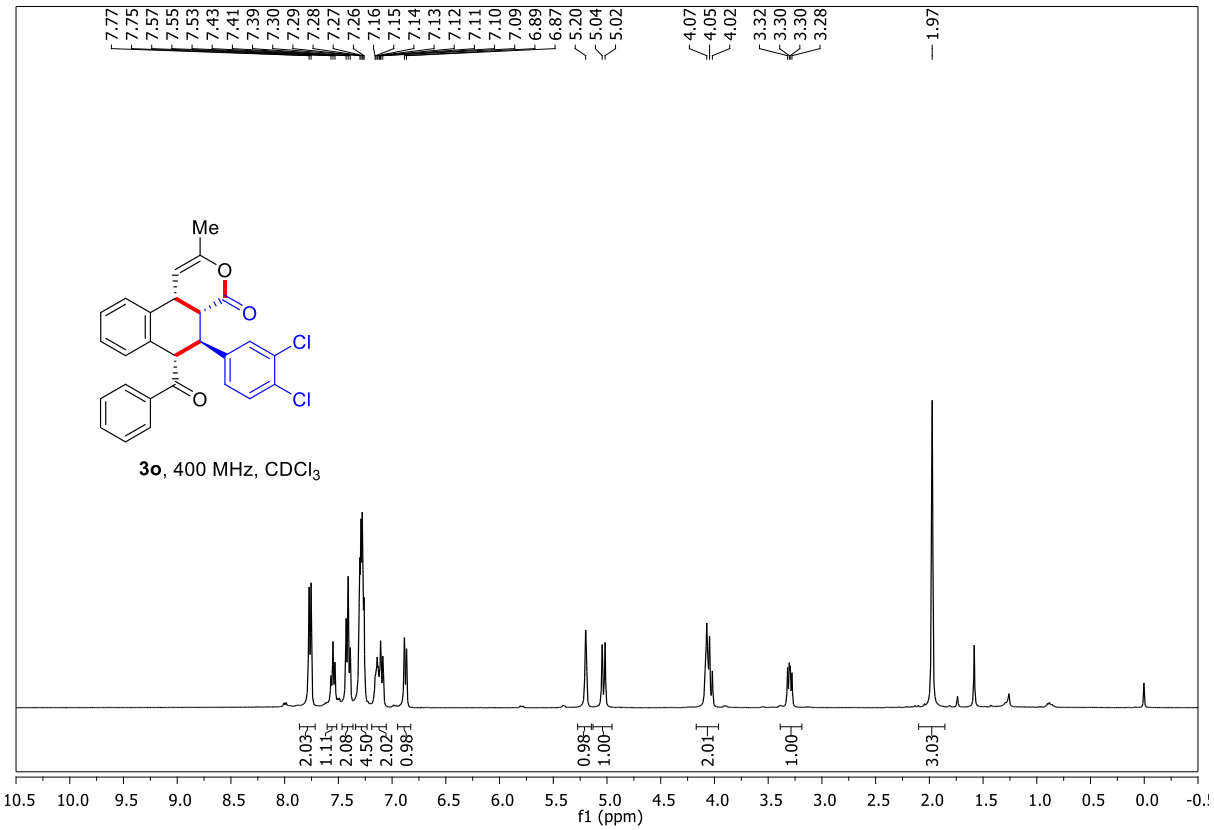


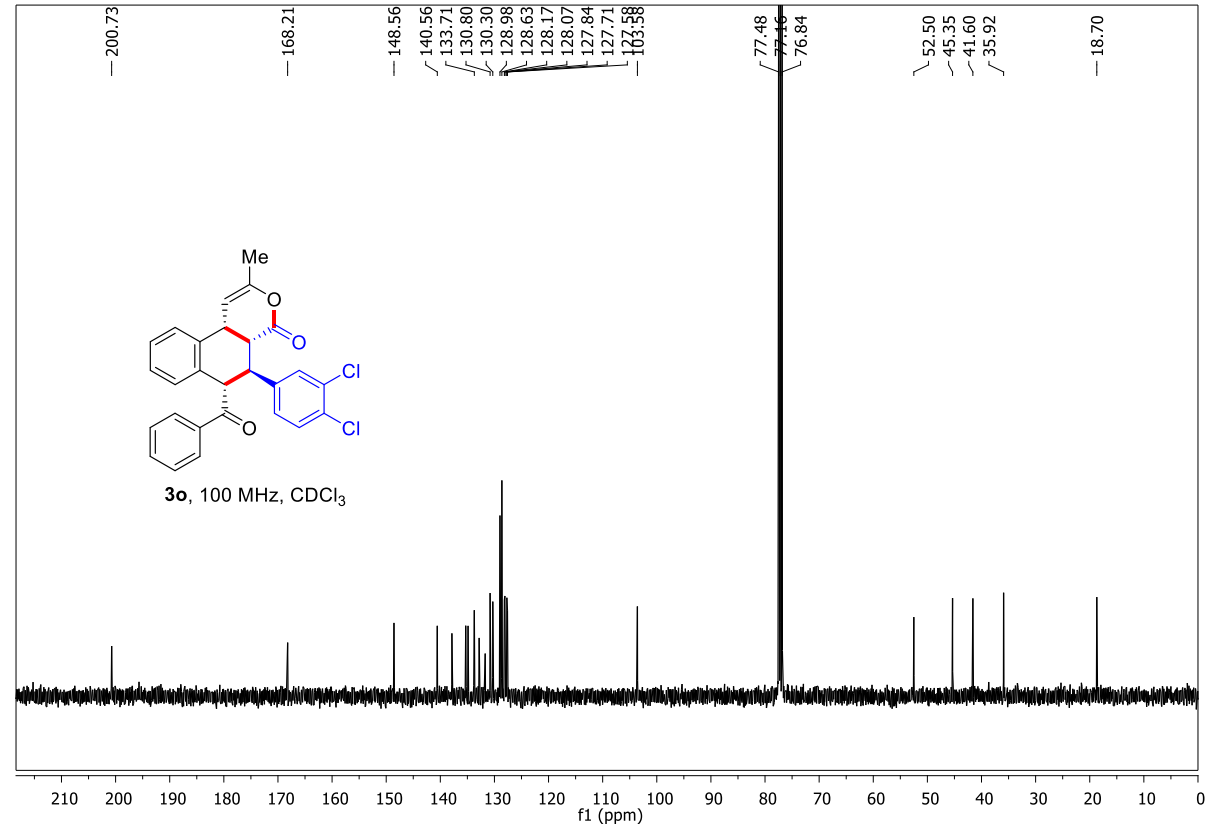
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3,4-dimethoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3n)



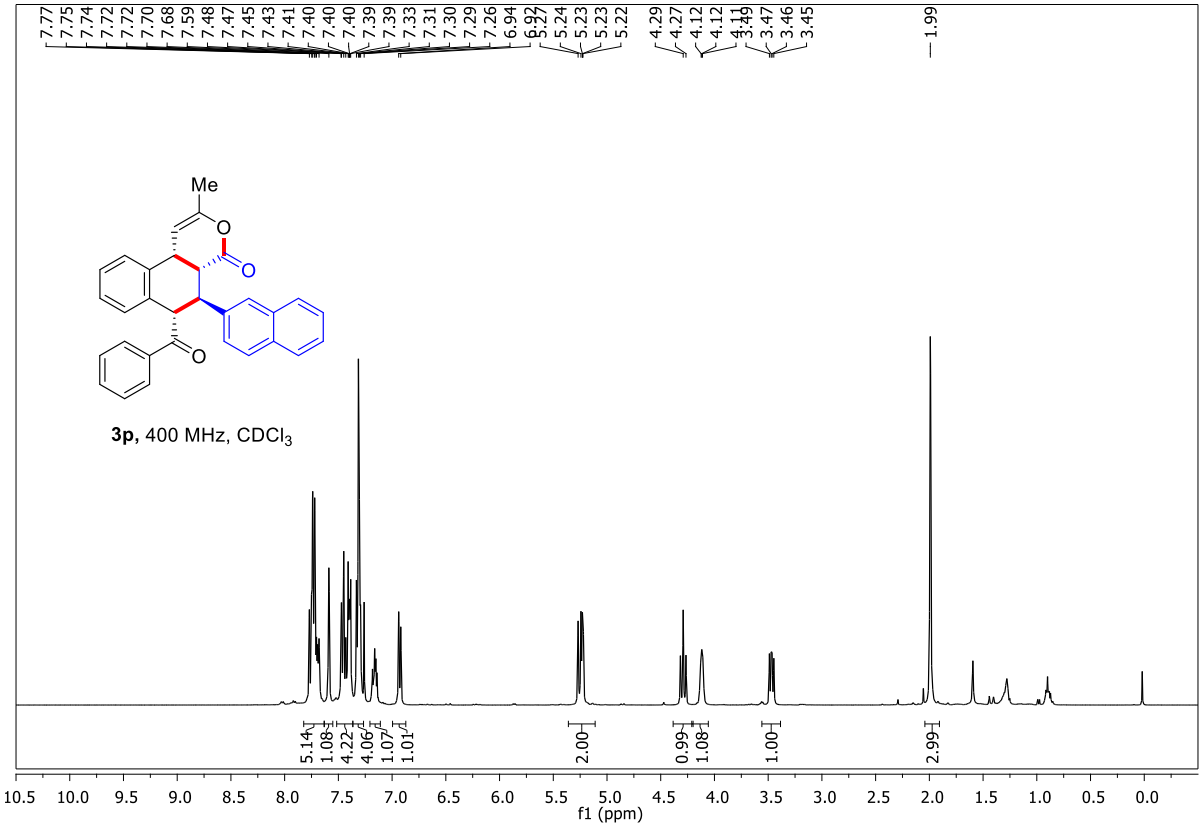


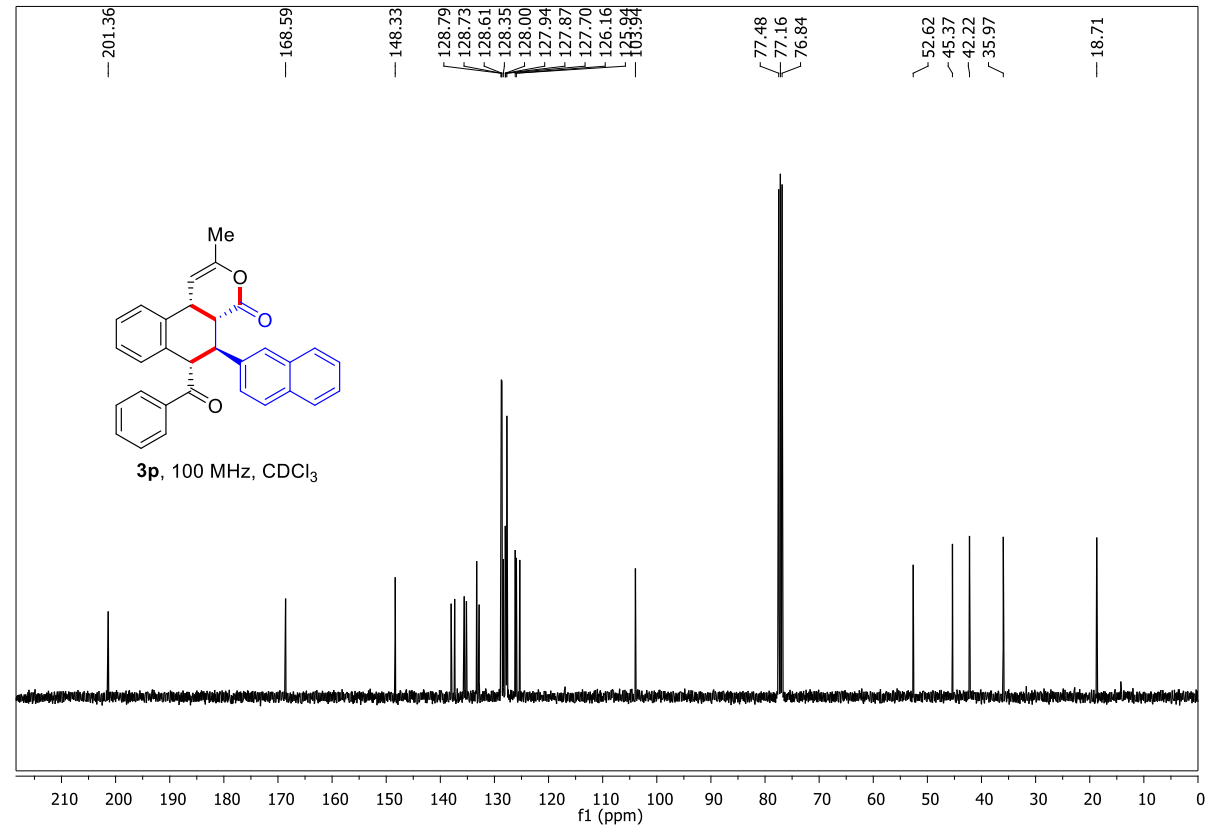
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3,4-dichlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3o)



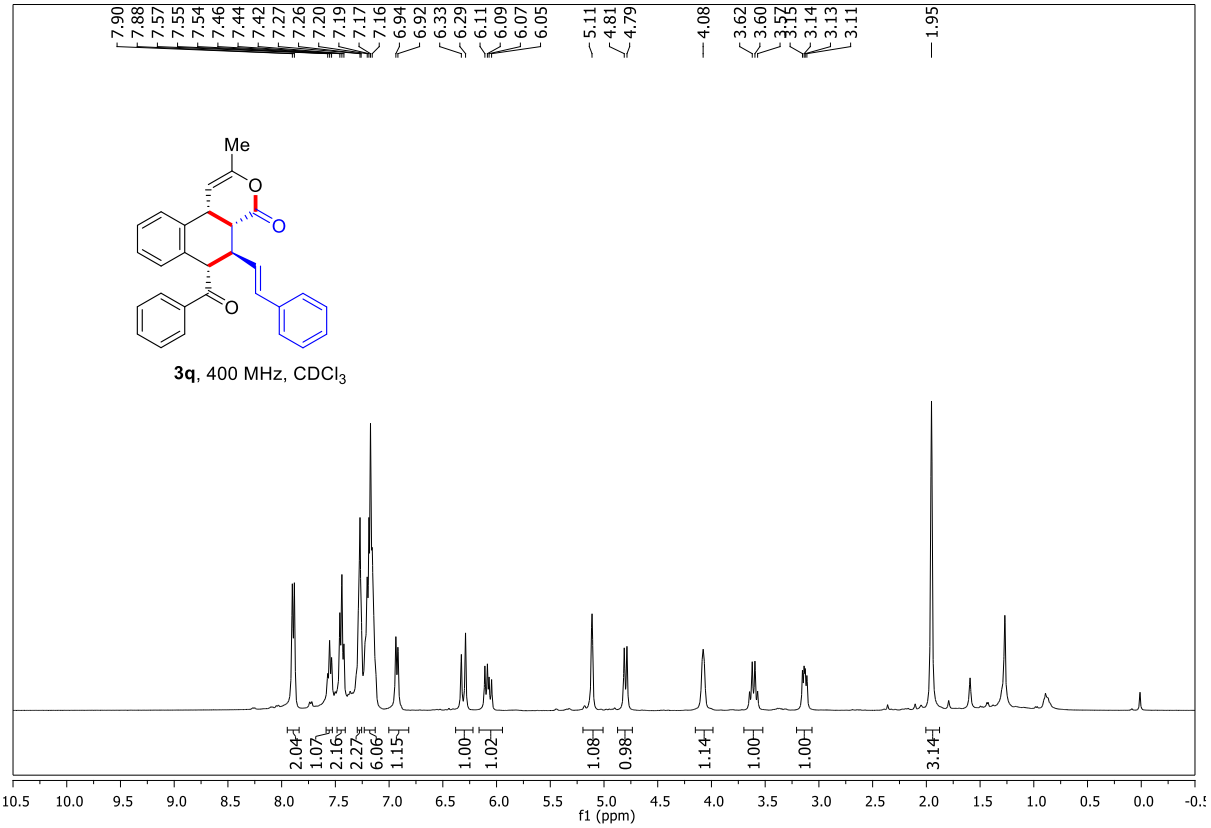


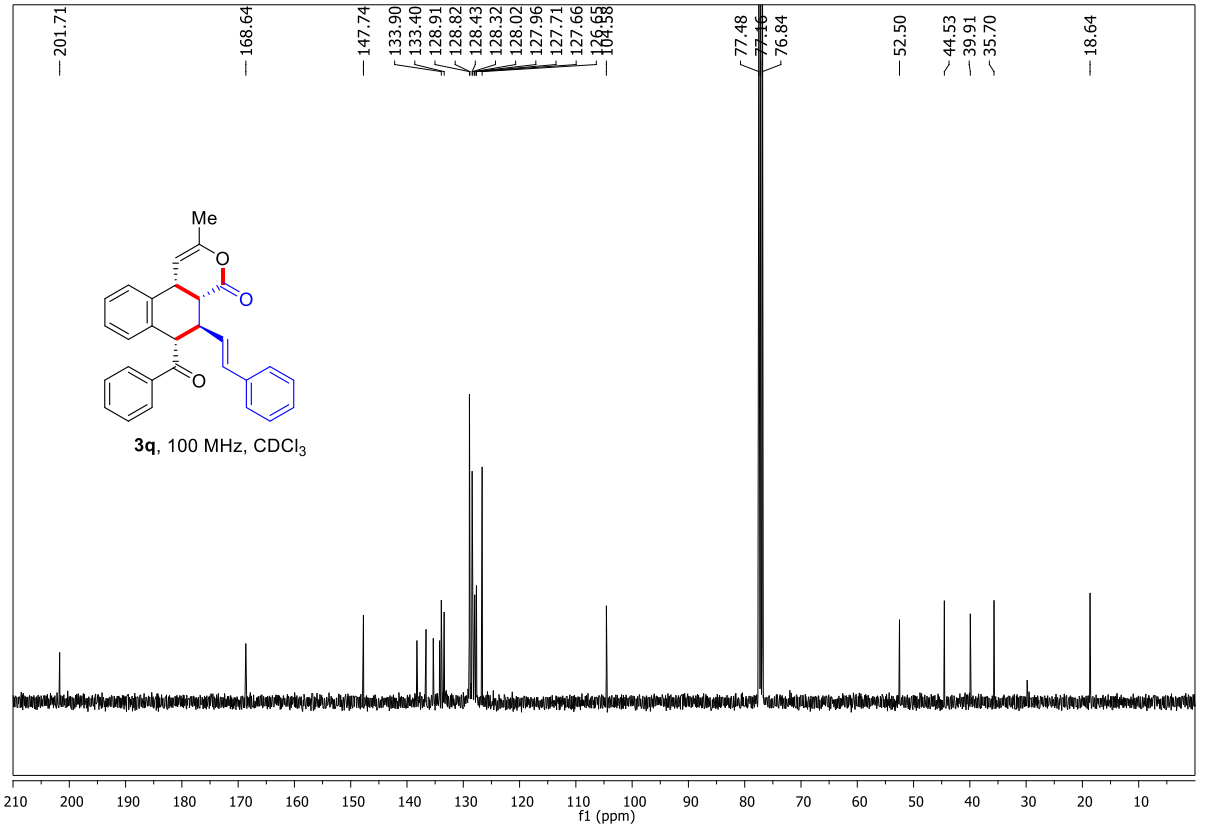
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(naphthalen-2-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3p)



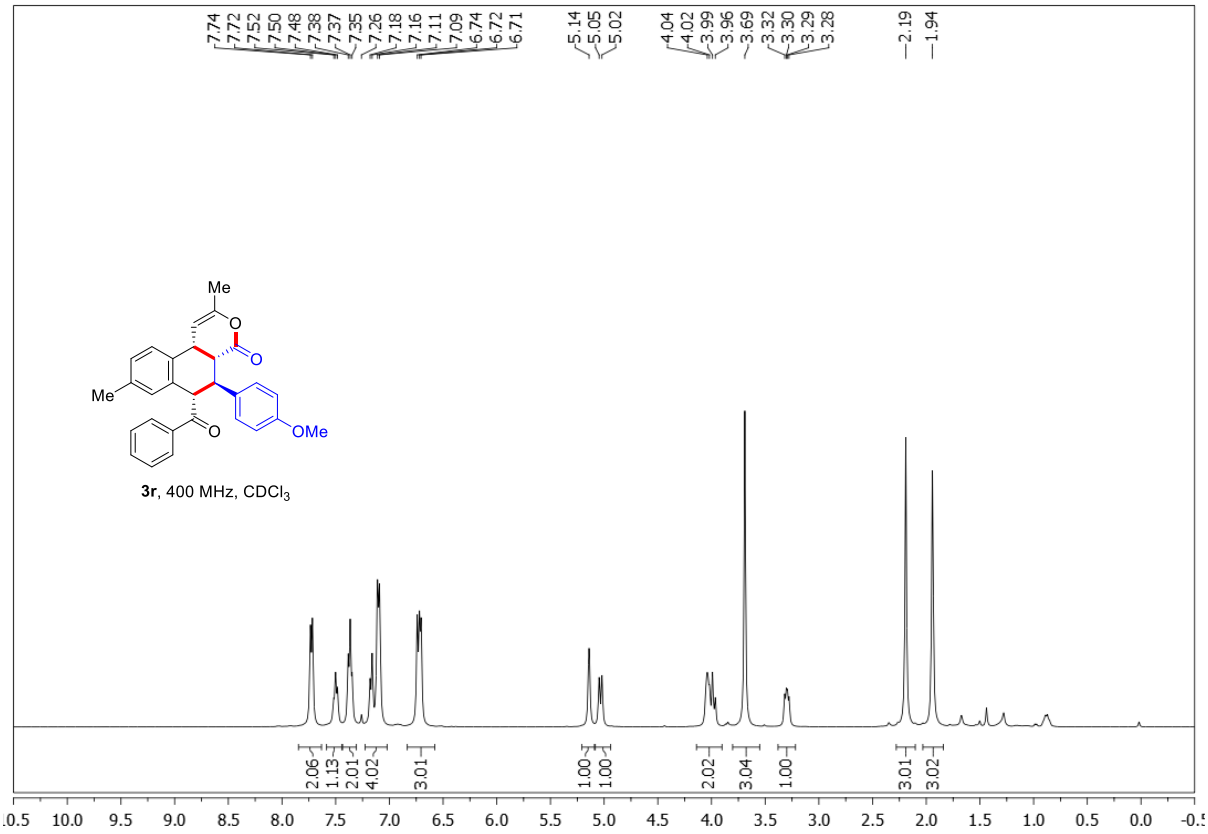


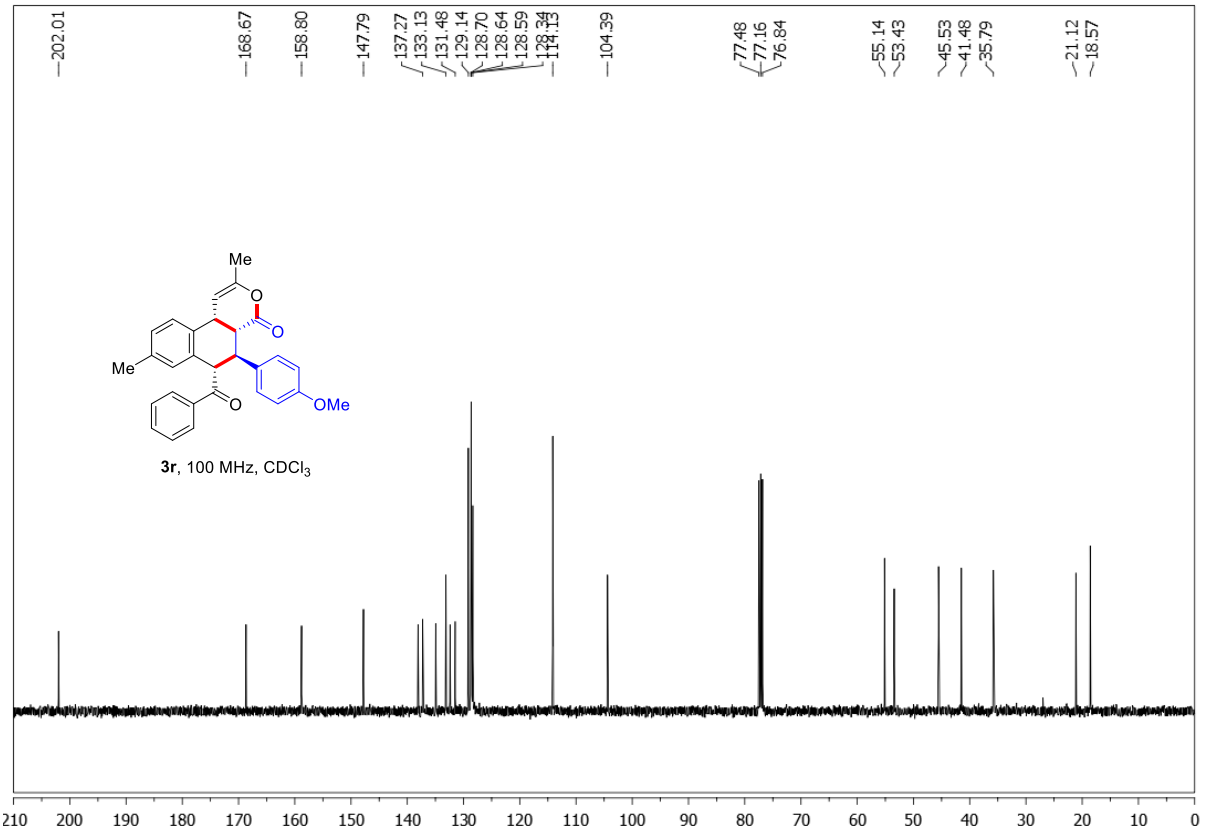
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-((*E*)-styryl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3q)



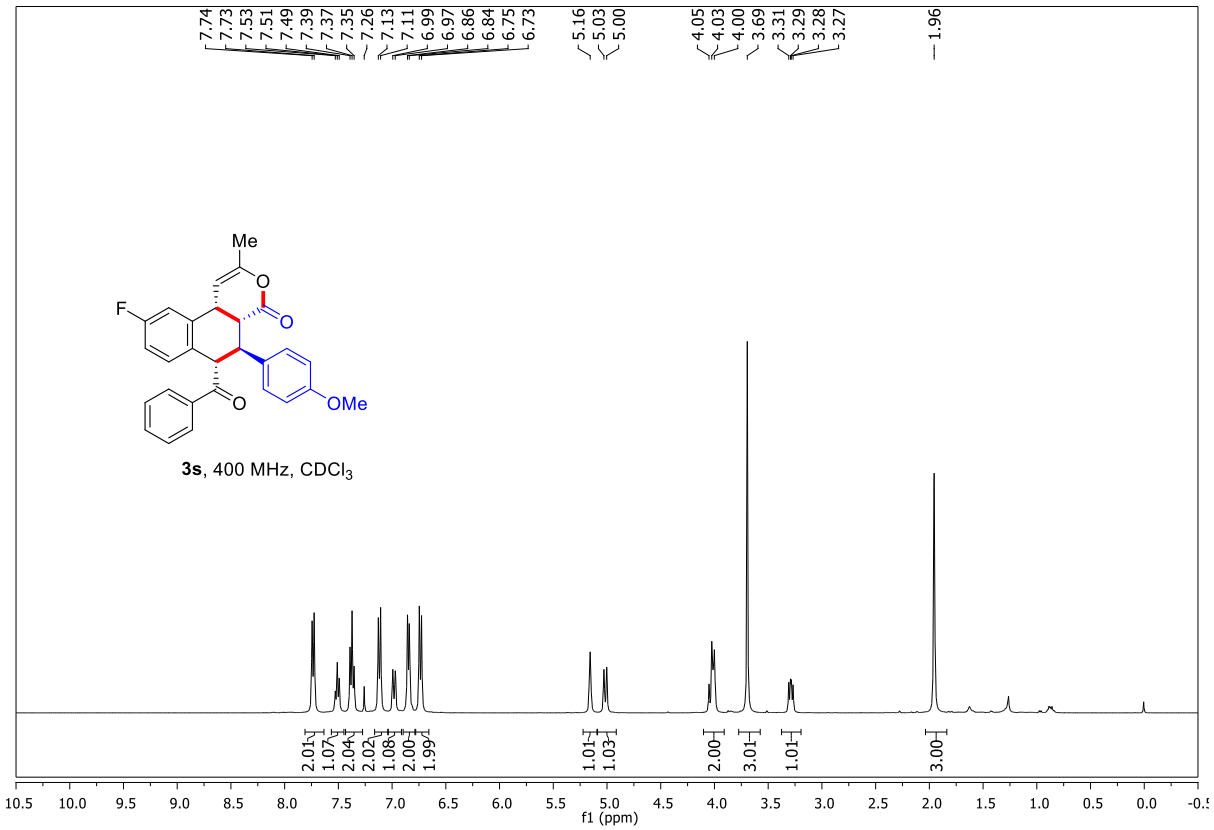


(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2,8-dimethyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3r)

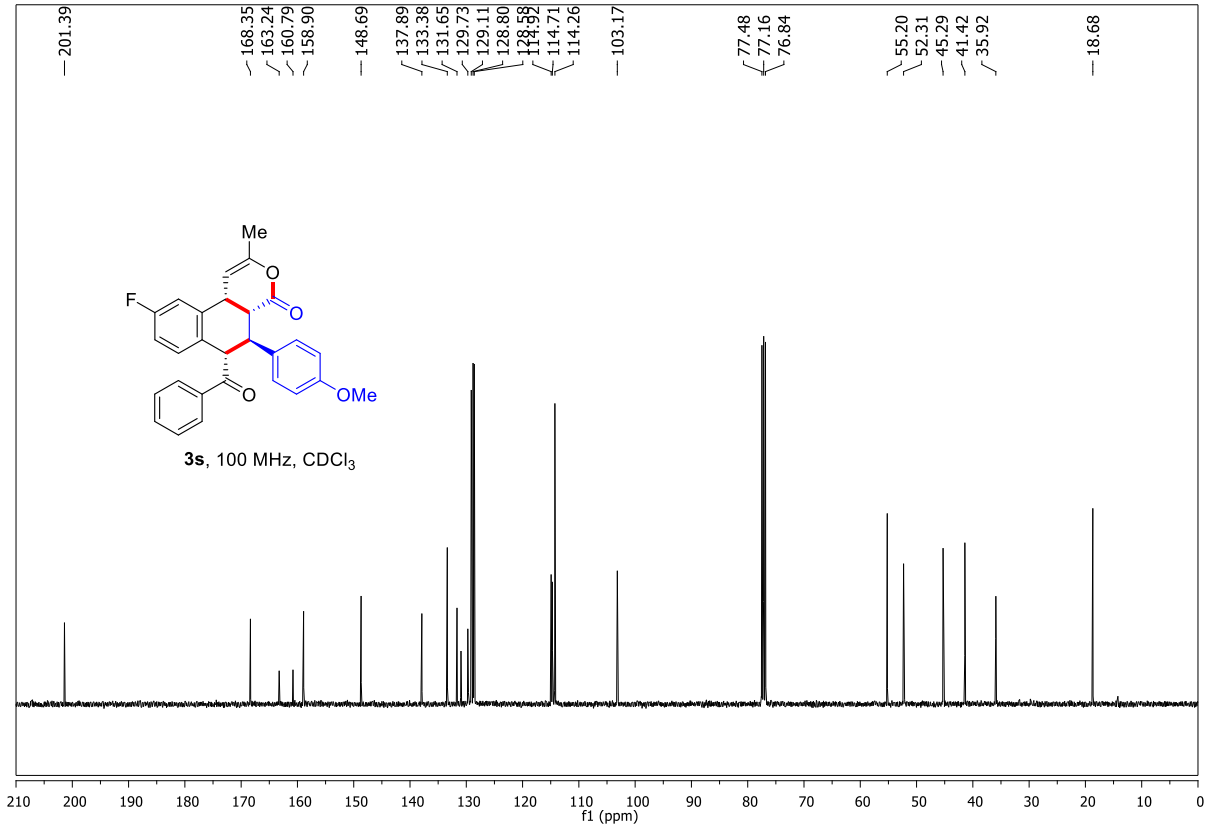




(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-9-fluoro-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3s)

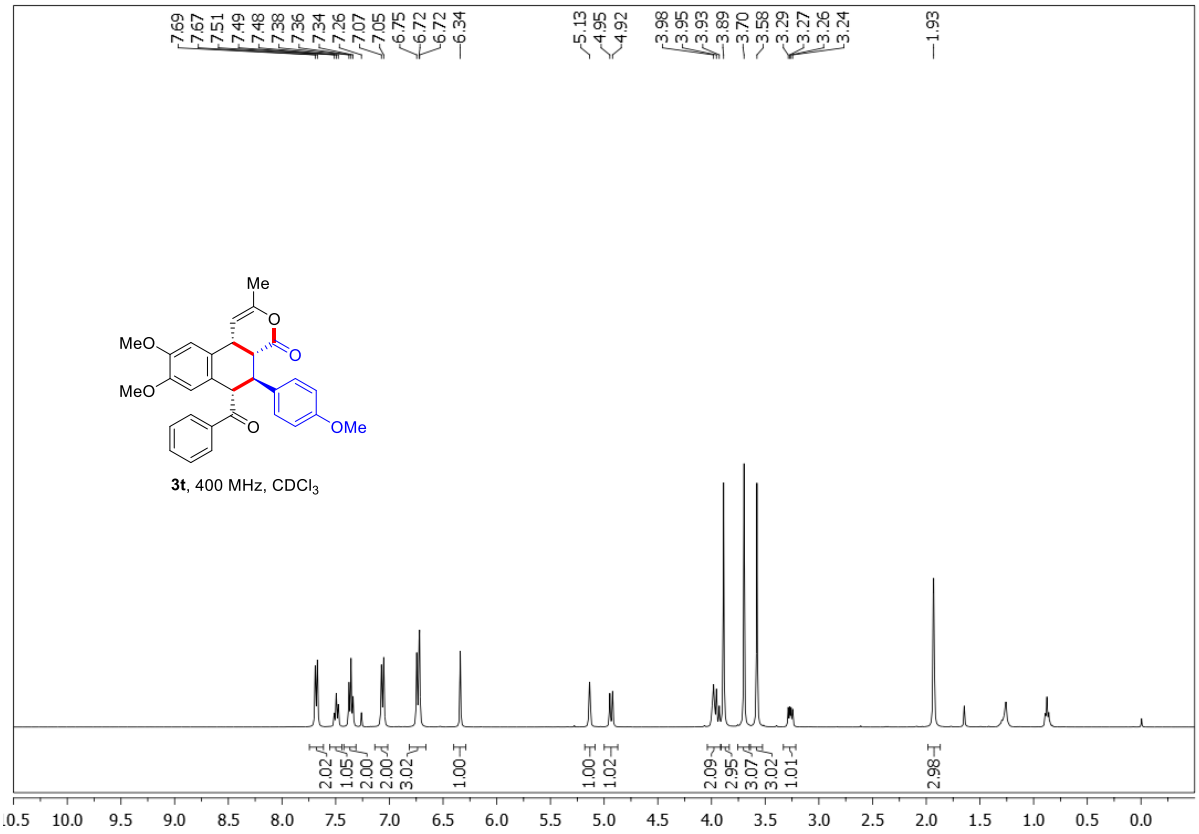


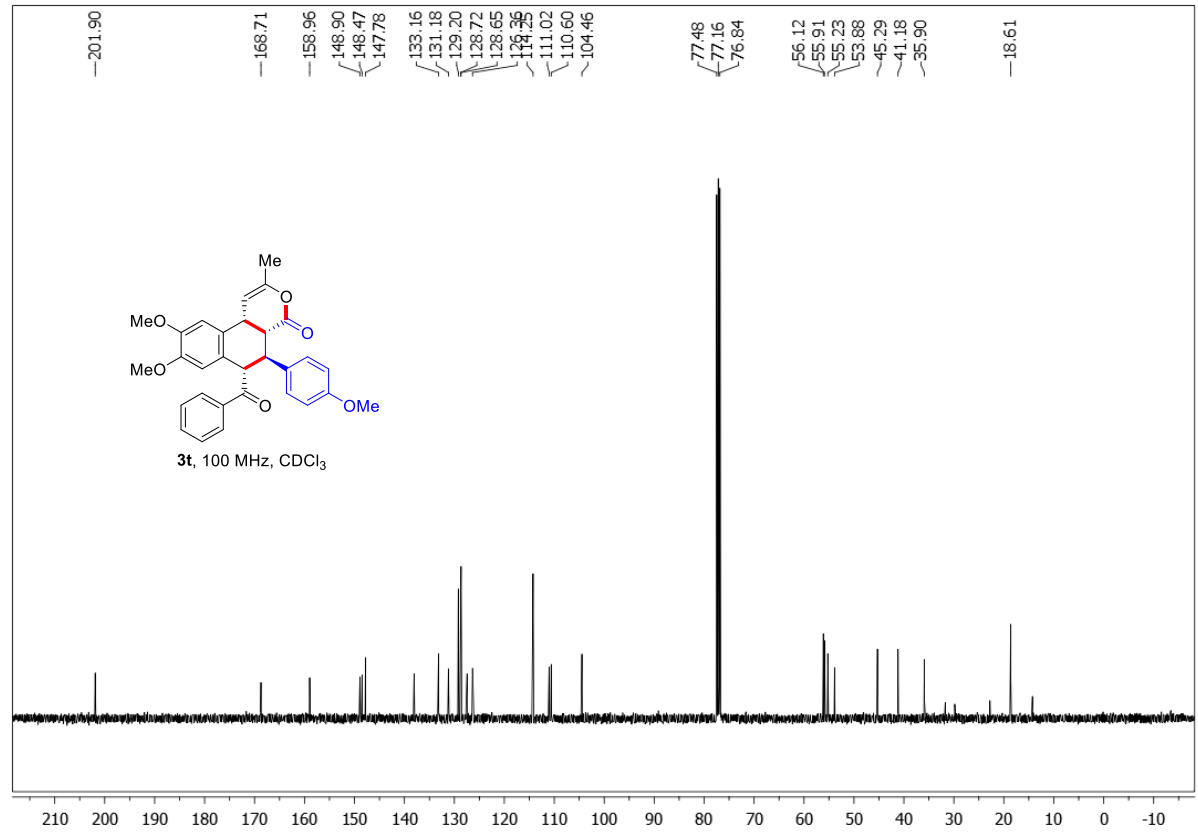
3s, 400 MHz, $CDCl_3$



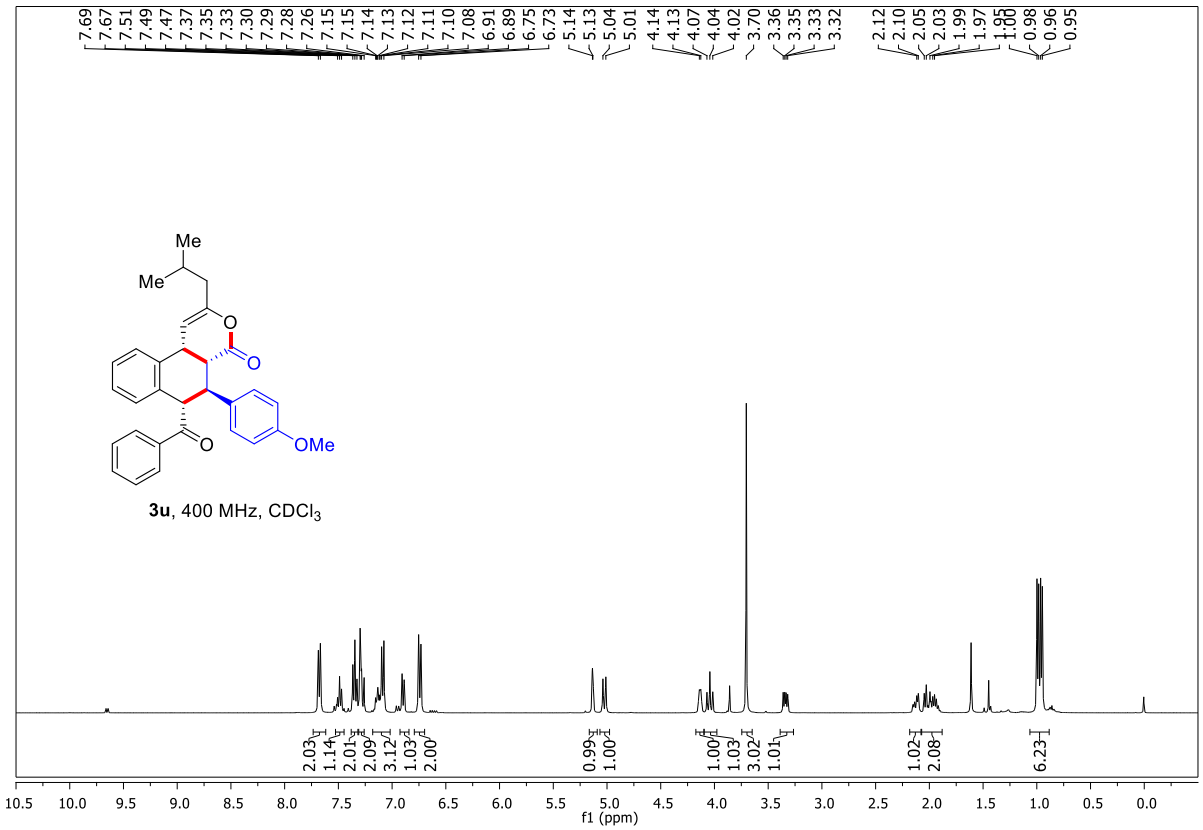
3s, 100 MHz, $CDCl_3$

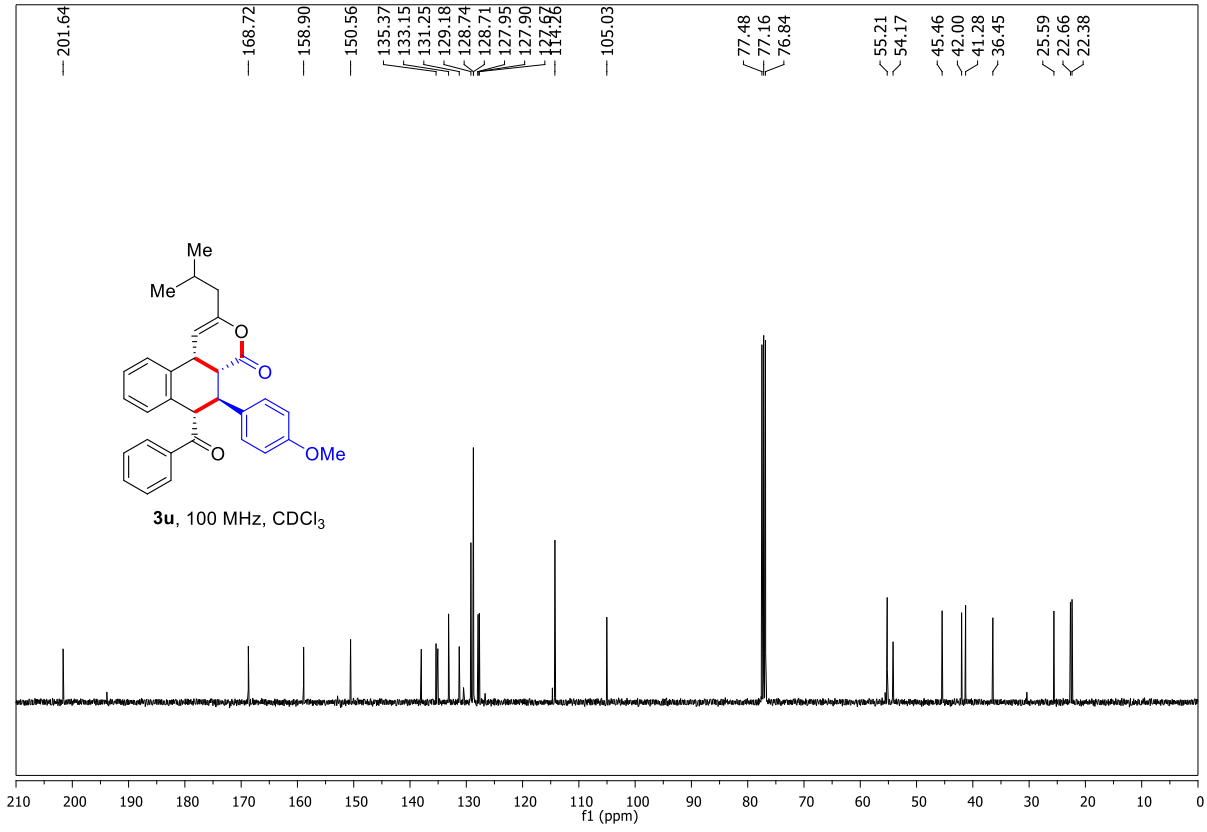
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-8,9-dimethoxy-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3t)



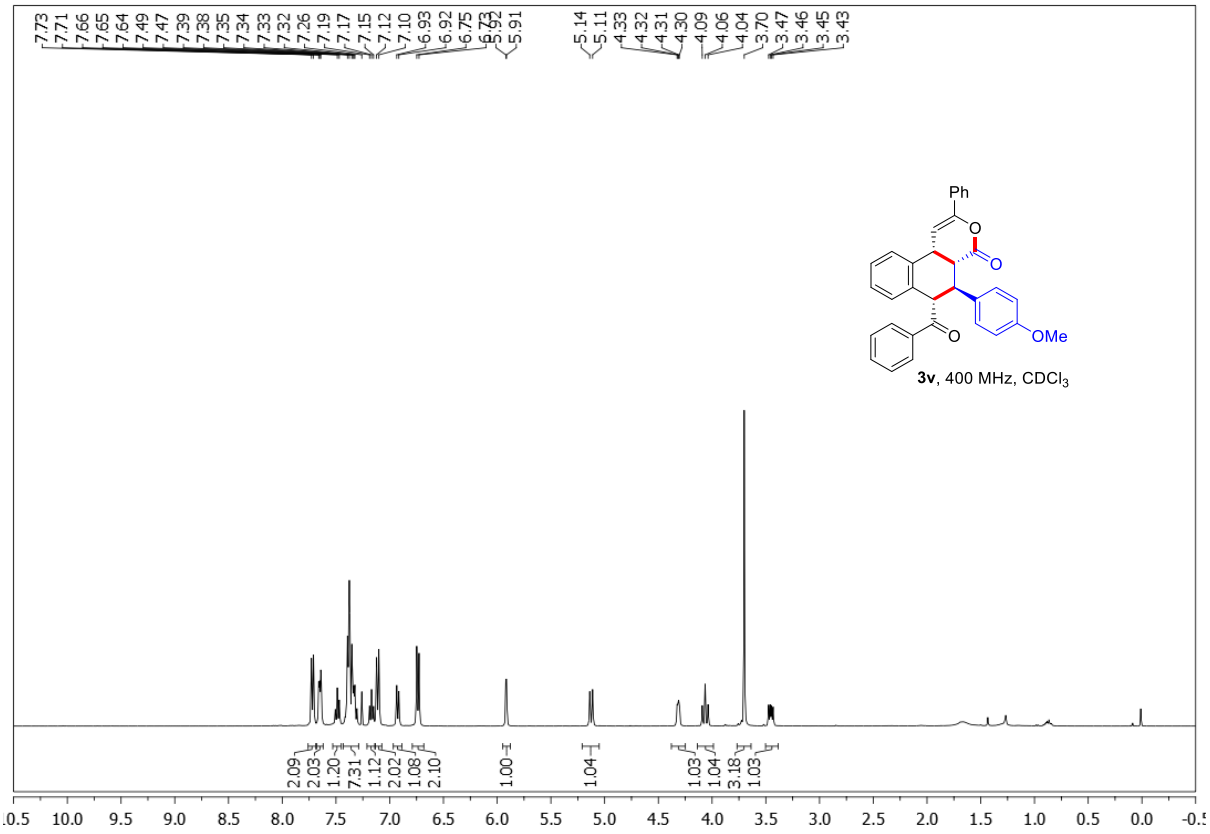


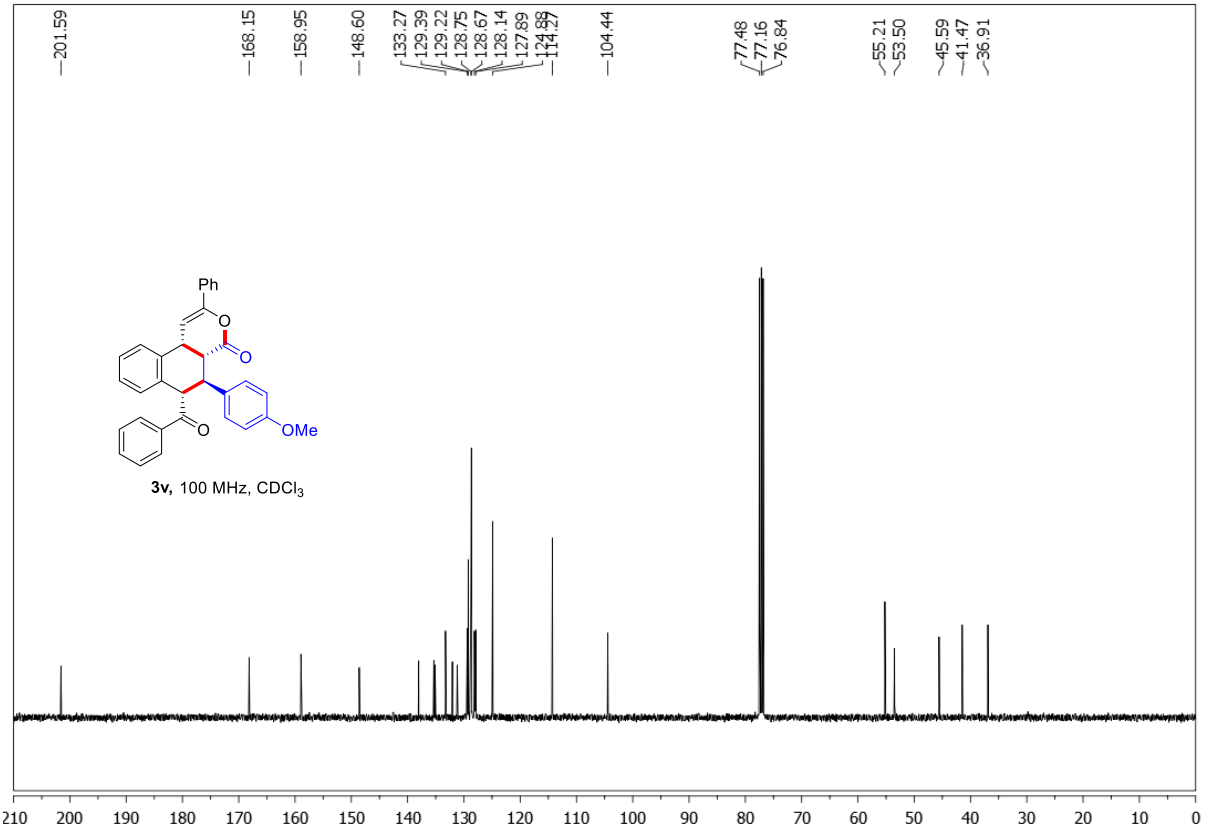
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-isobutyl-5-(4-methoxyphenyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3u)



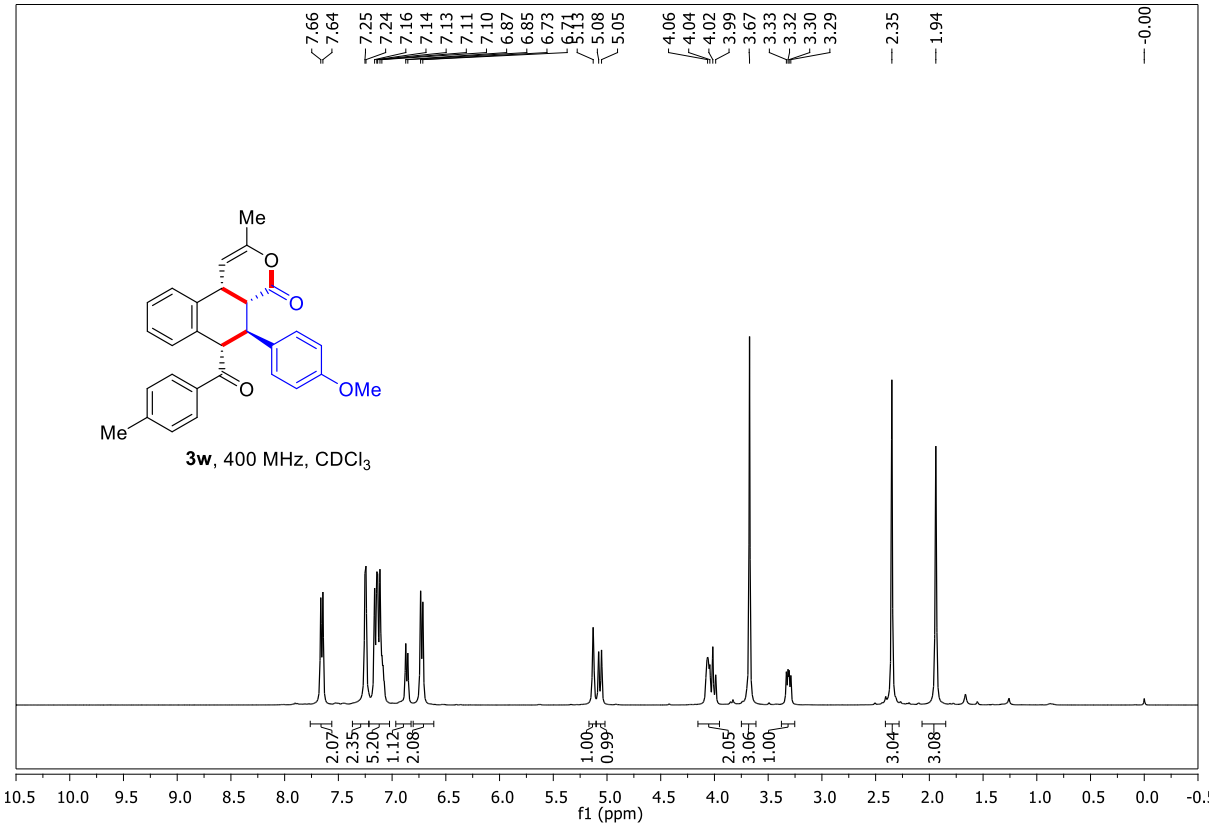


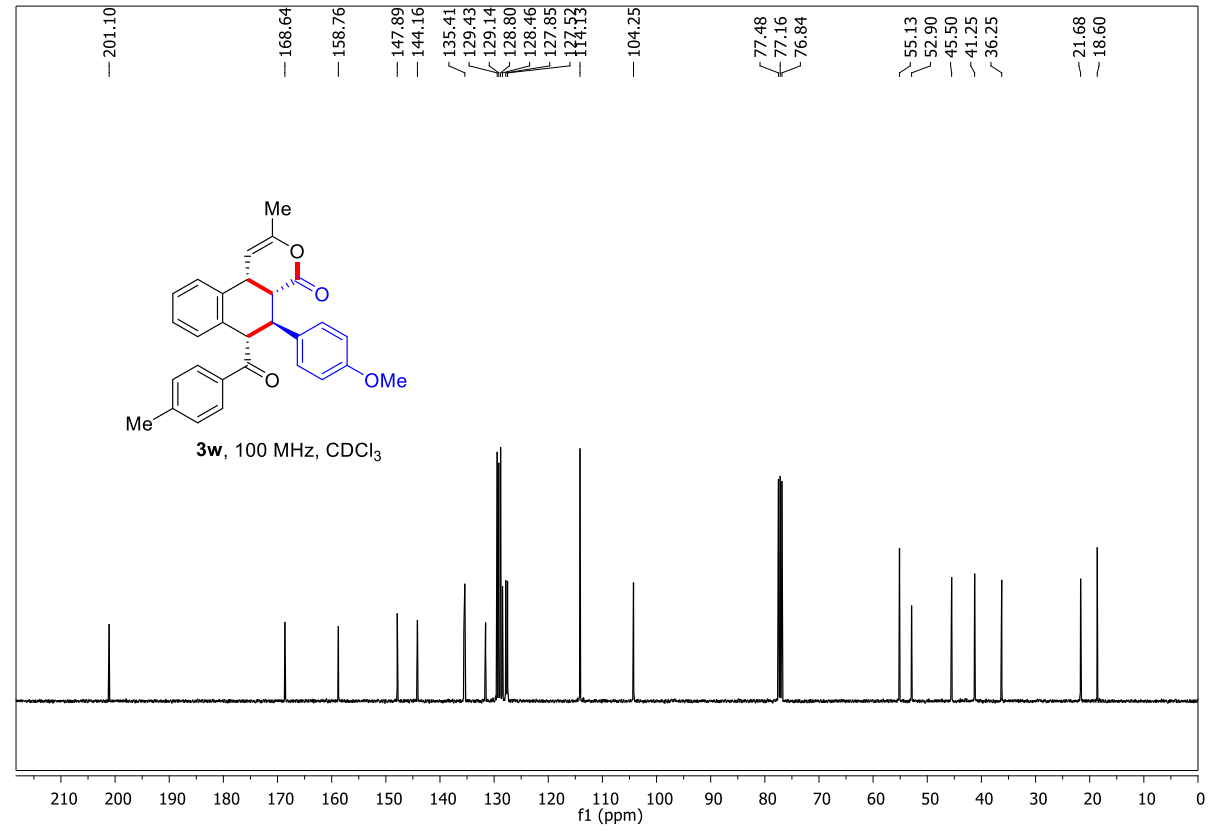
(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3v)



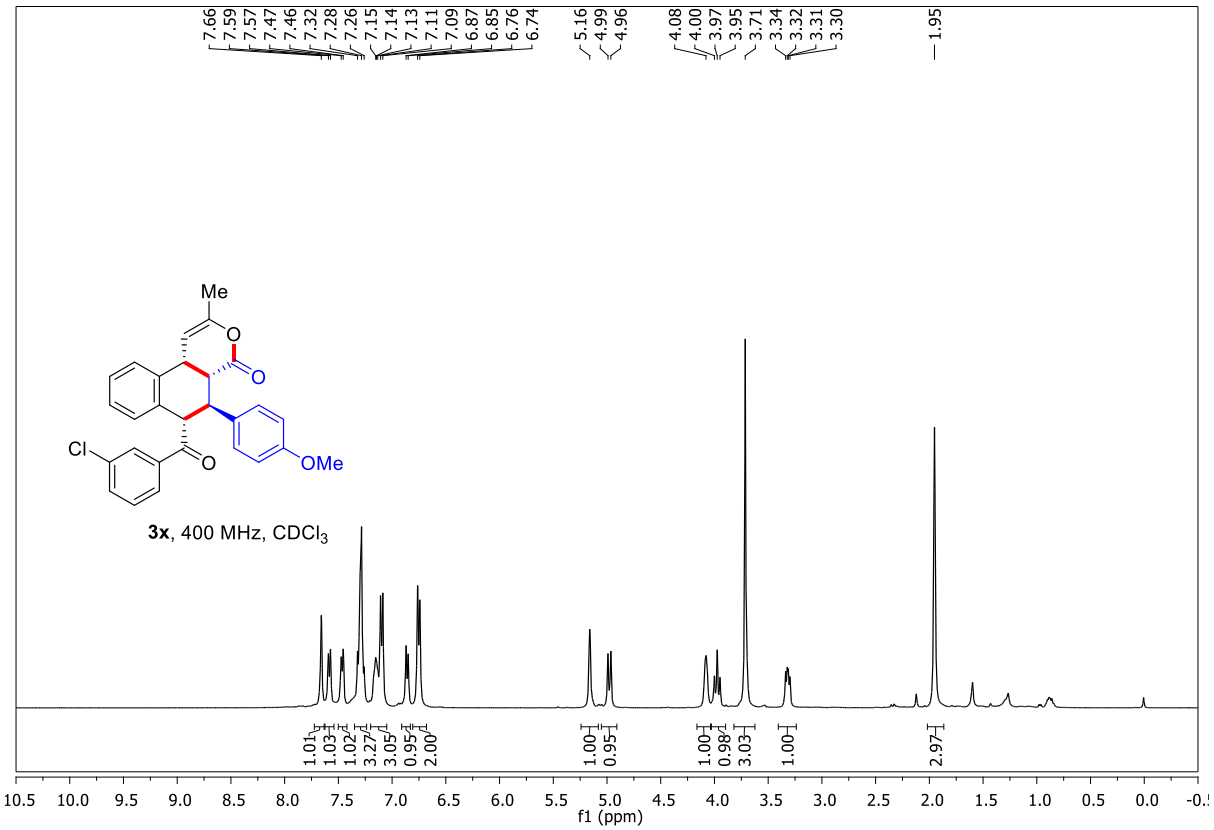


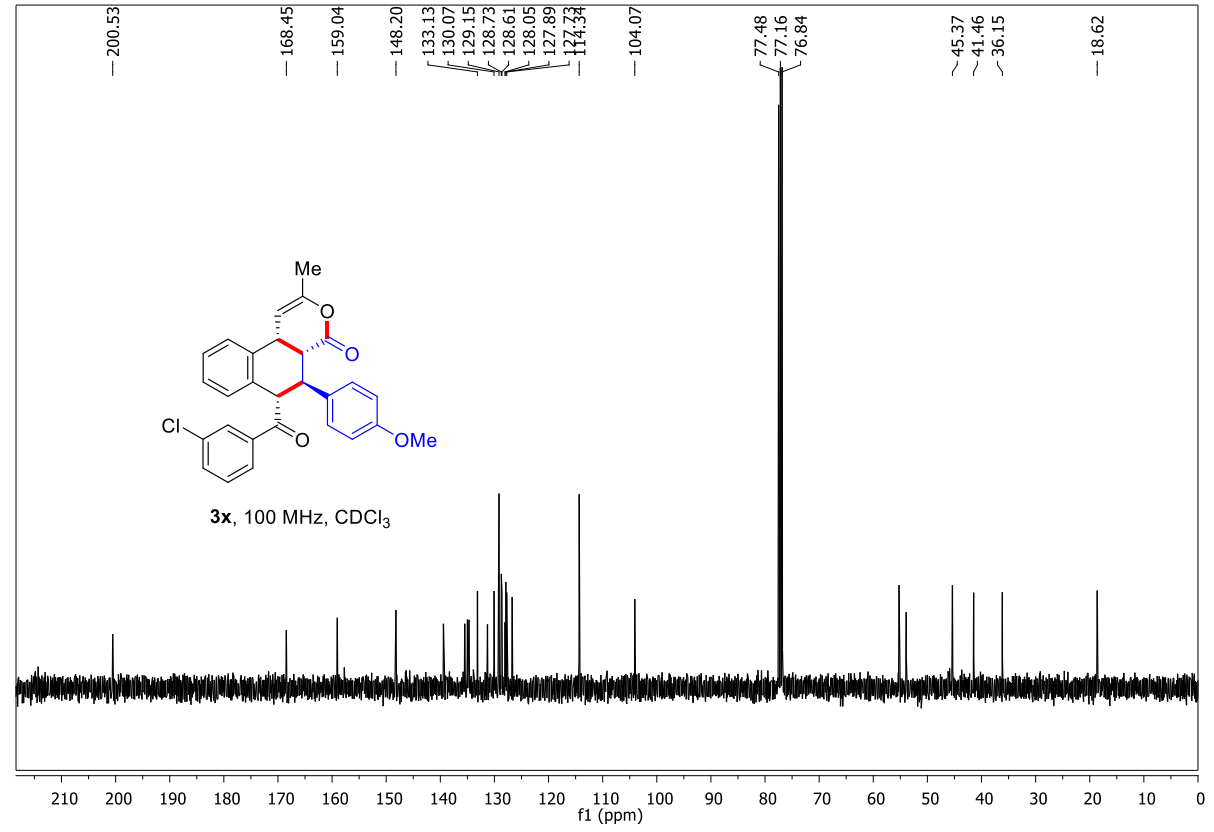
(4a*R*,5*S*,6*S*,10b*R*)-5-(4-Methoxyphenyl)-2-methyl-6-(4-methylbenzoyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3w)



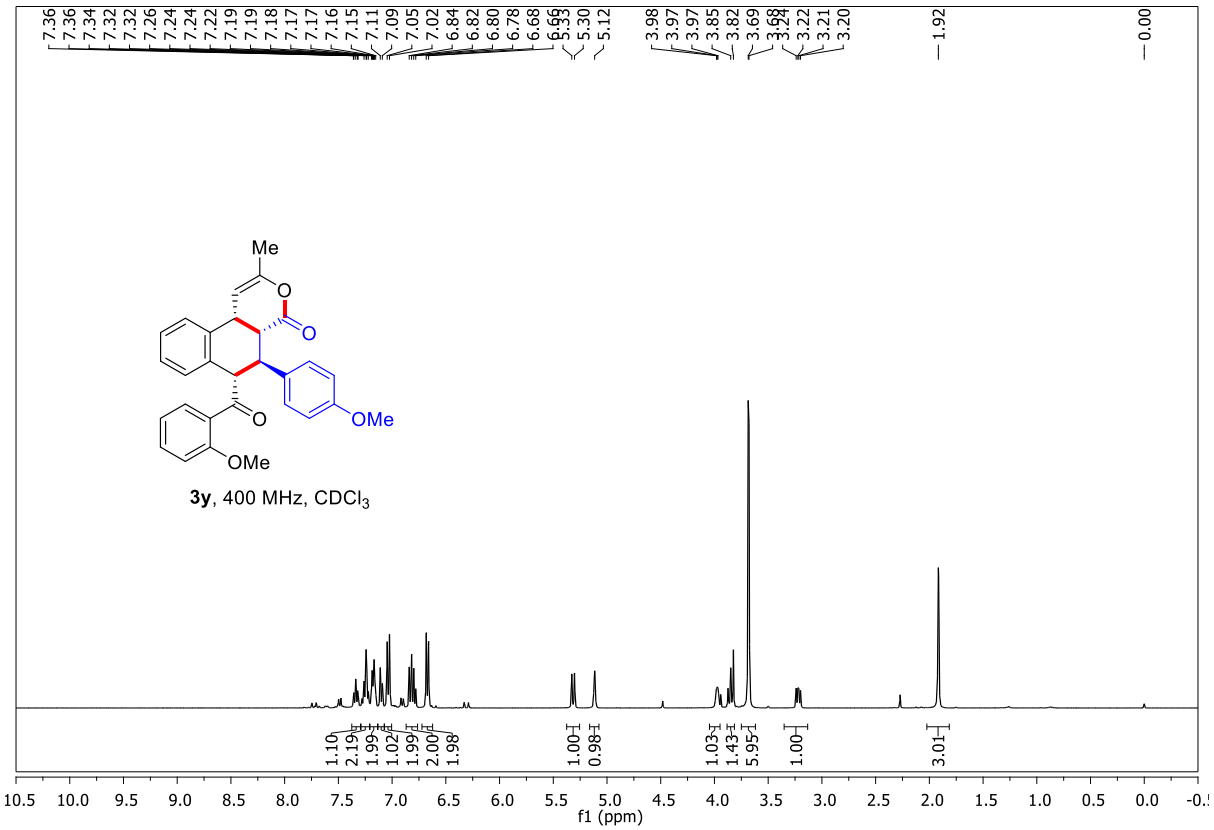


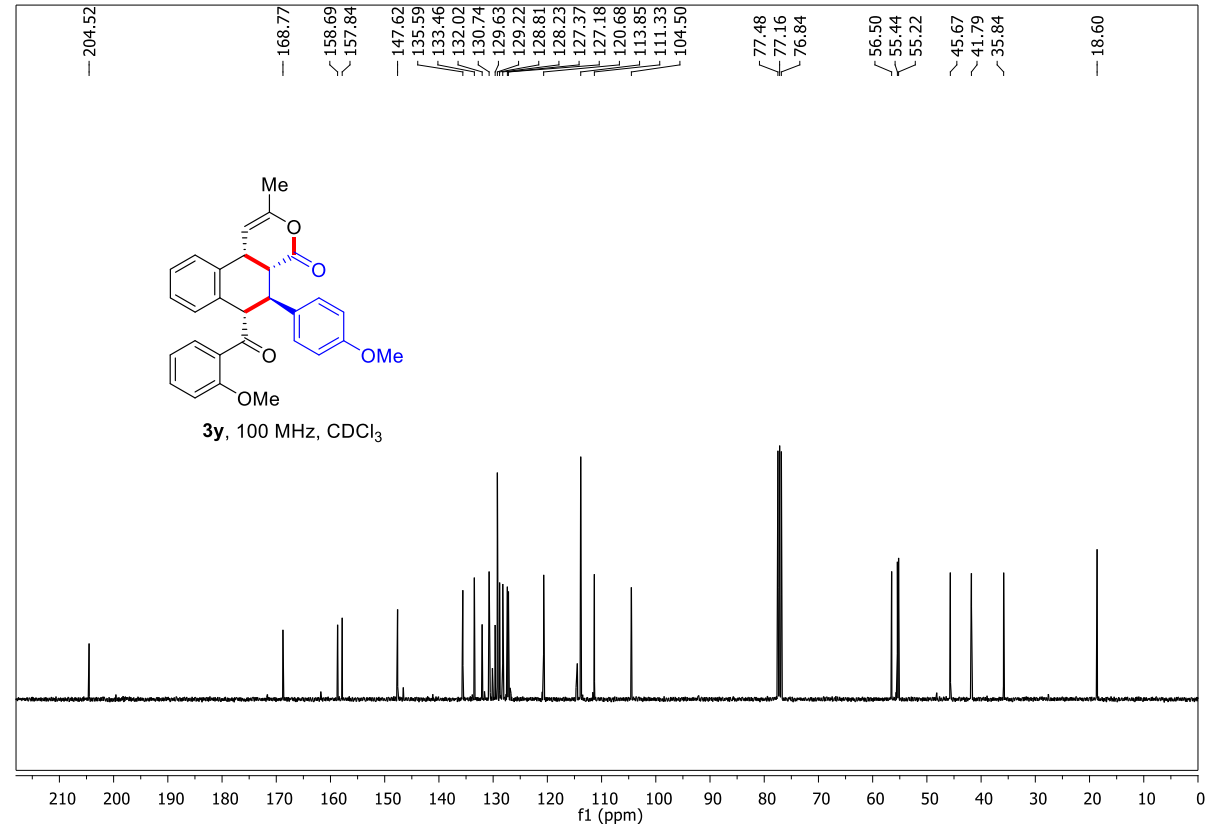
(4a*R*,5*S*,6*S*,10b*R*)-6-(3-Chlorobenzoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3x)



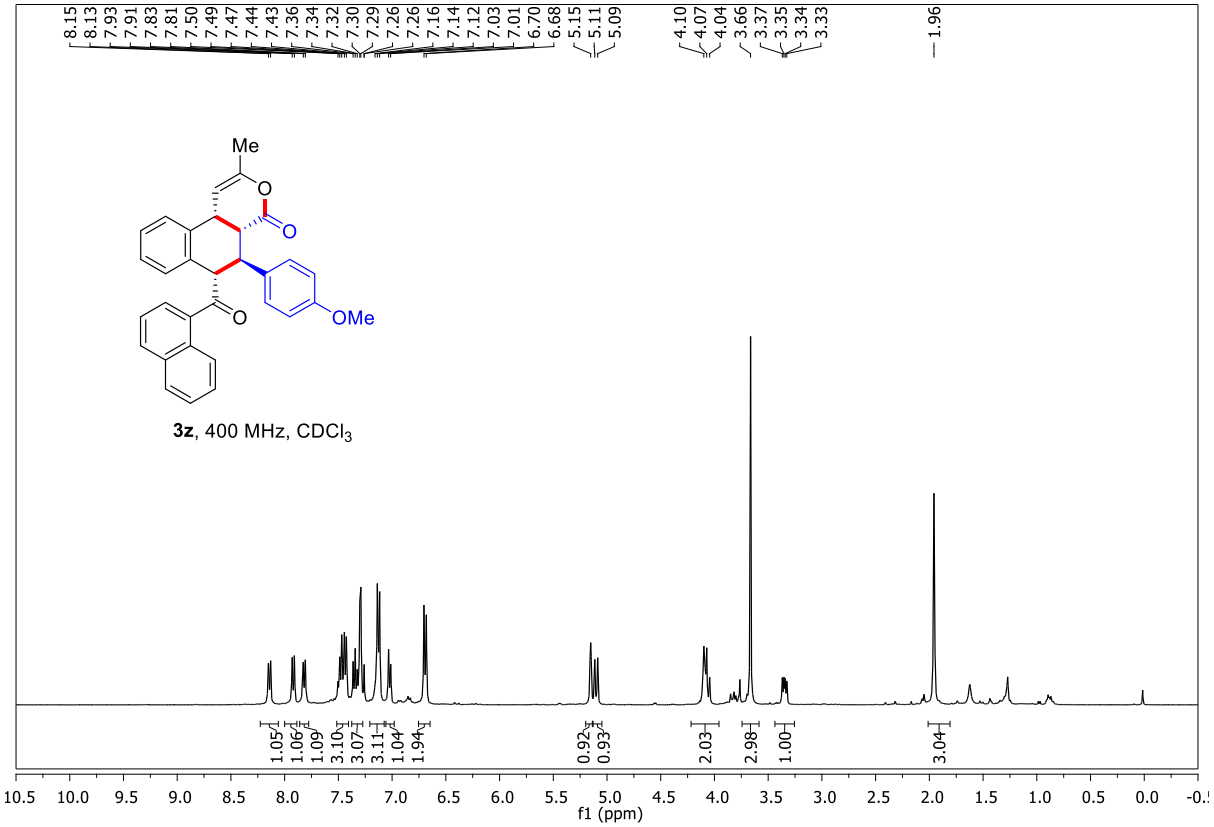


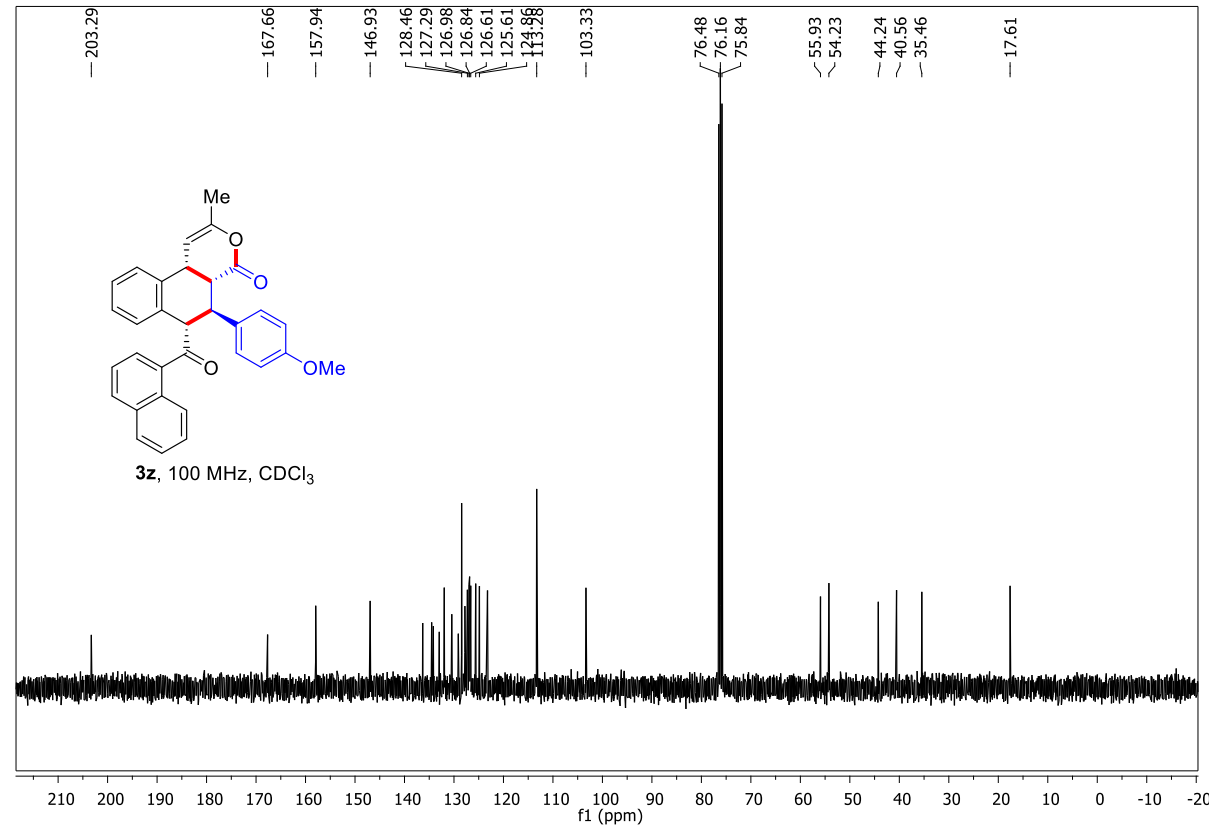
(4a*R*,5*S*,6*S*,10b*R*)-6-(2-Methoxybenzoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3y)



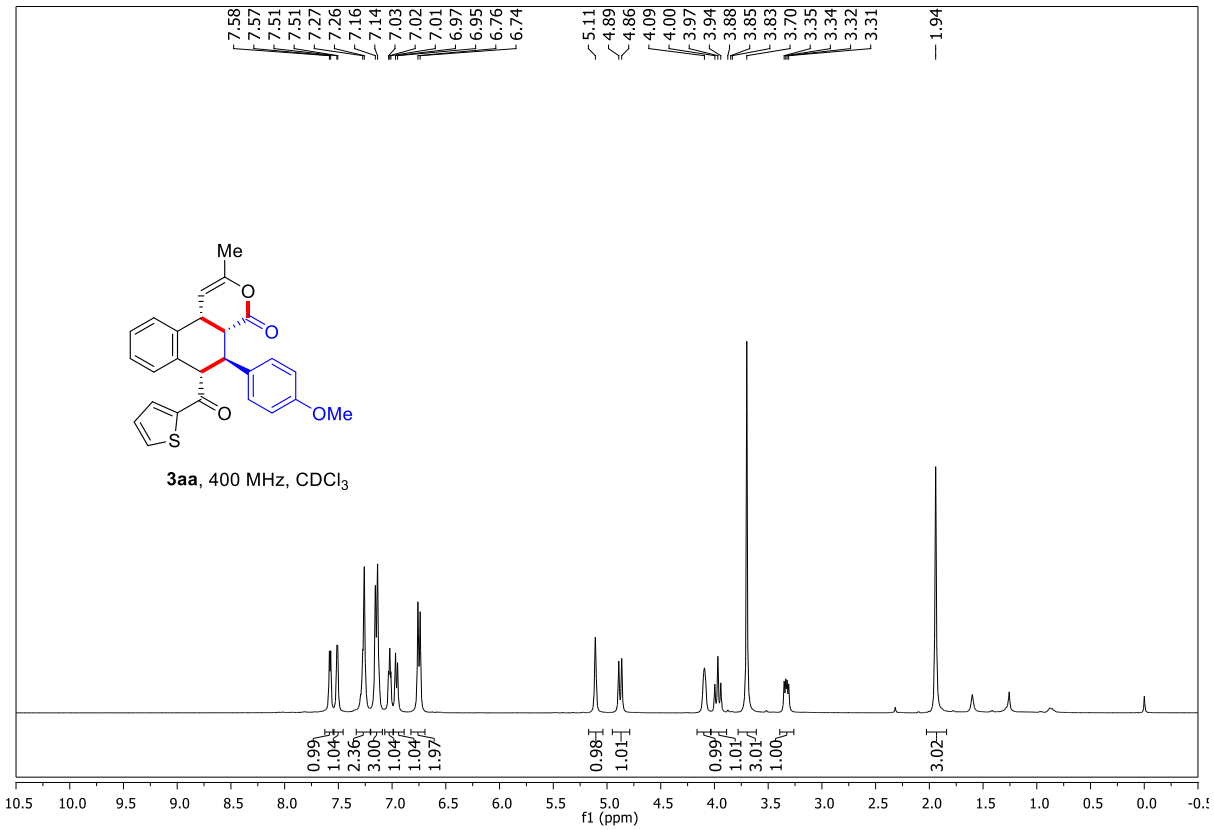


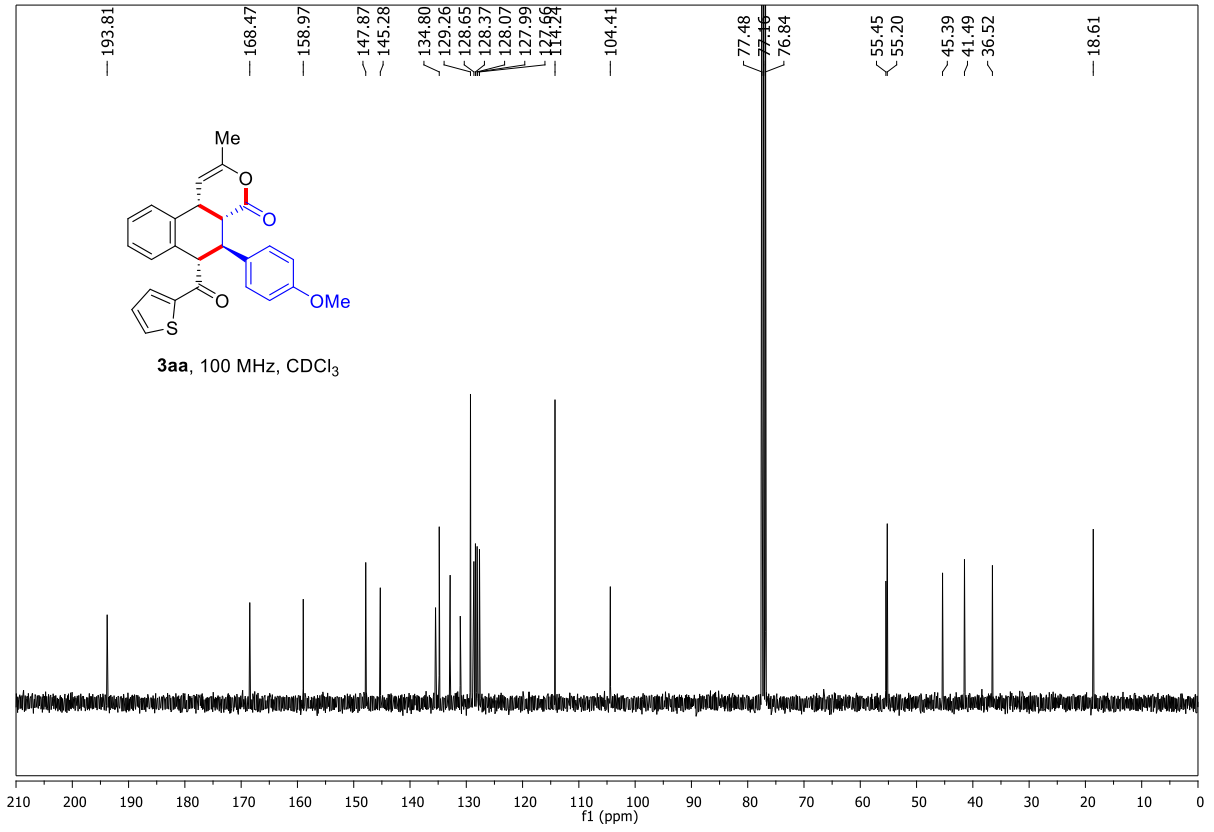
(4a*R*,5*S*,6*S*,10b*R*)-6-(1-Naphthoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3z)



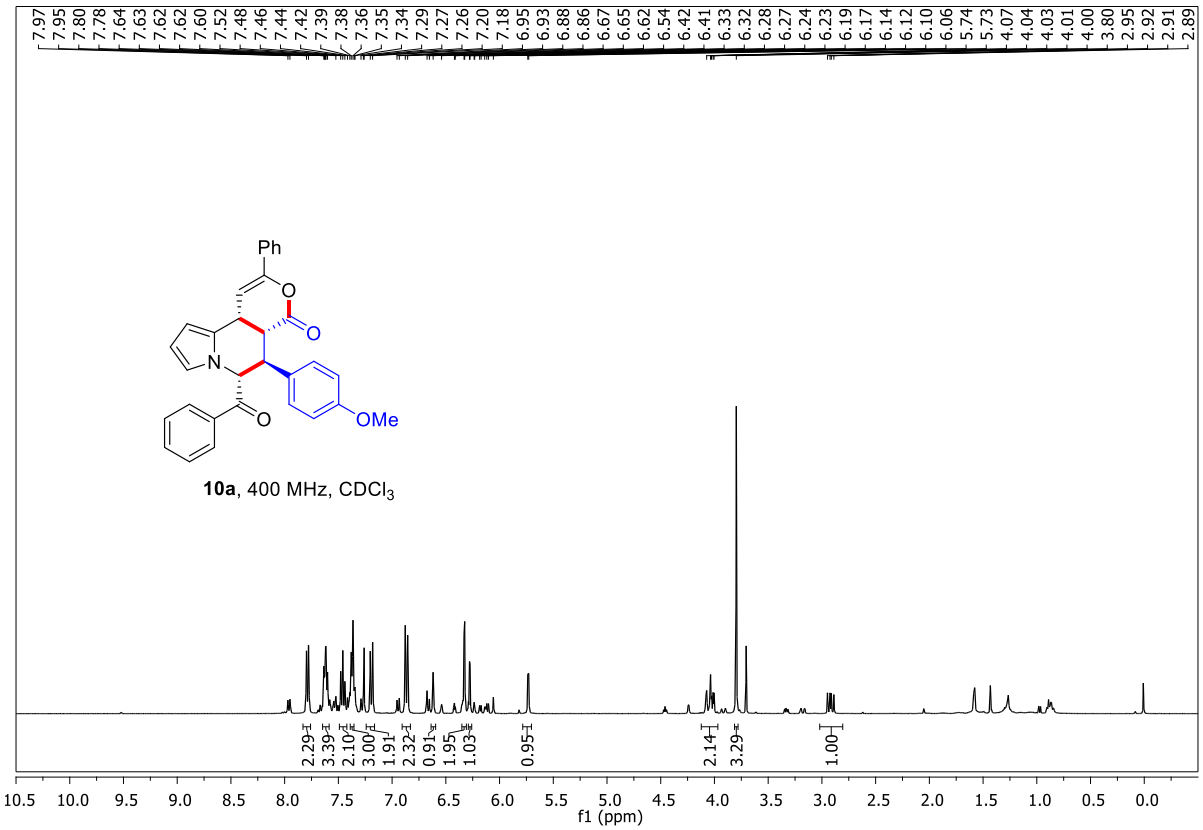


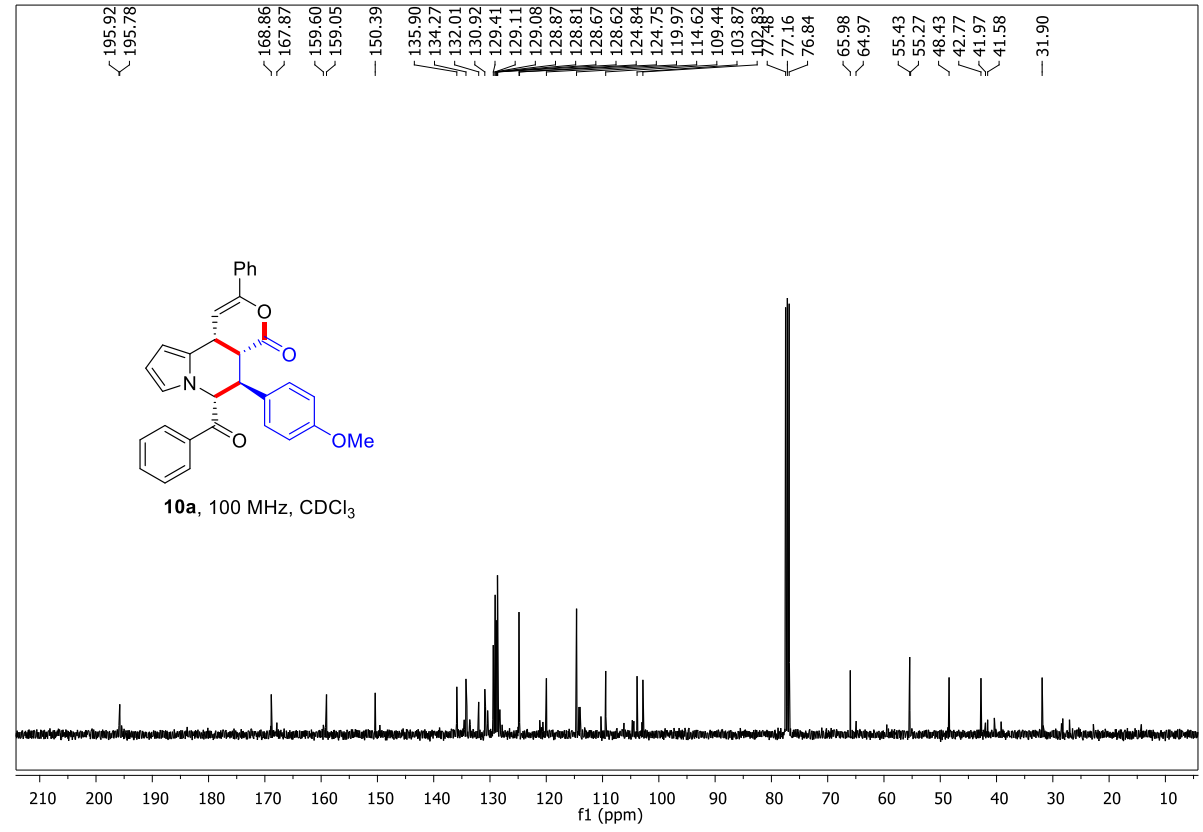
(4a*R*,5*S*,6*S*,10b*R*)-5-(4-Methoxyphenyl)-2-methyl-6-(thiophene-2-carbonyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3aa)



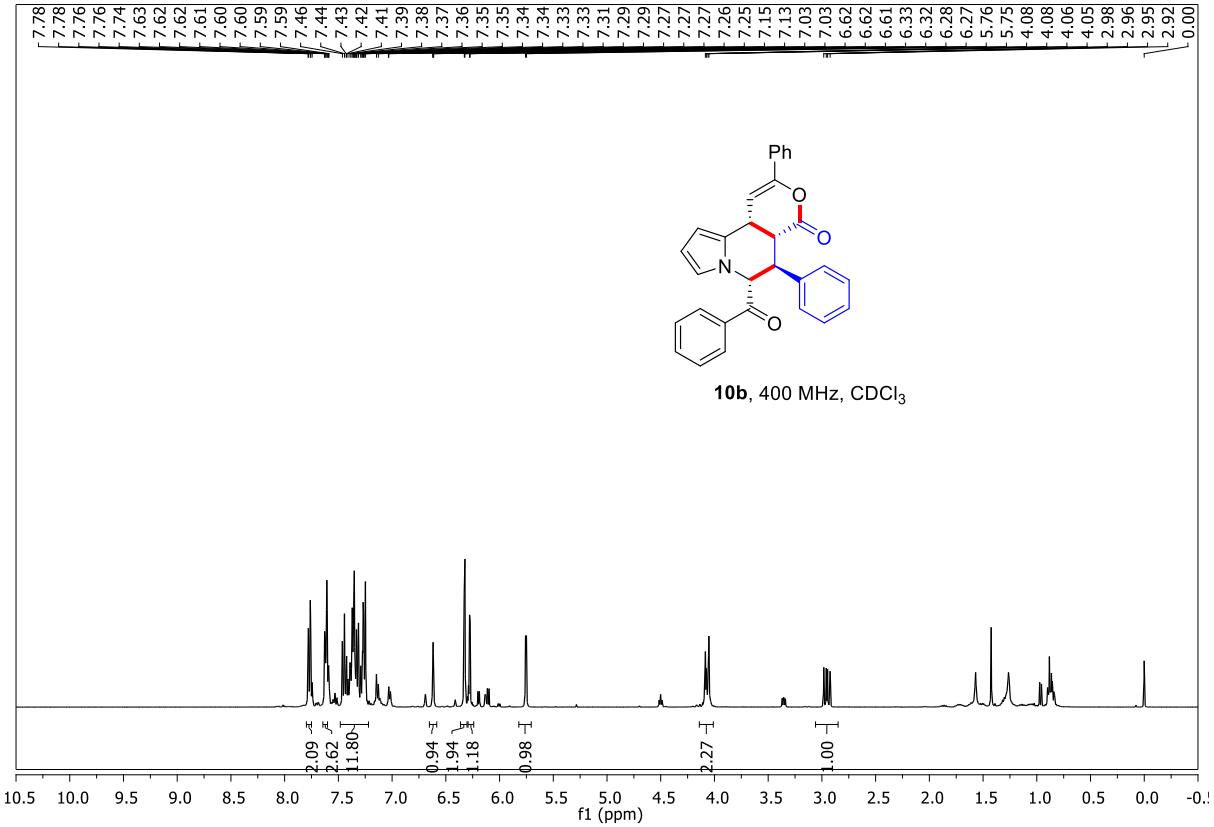


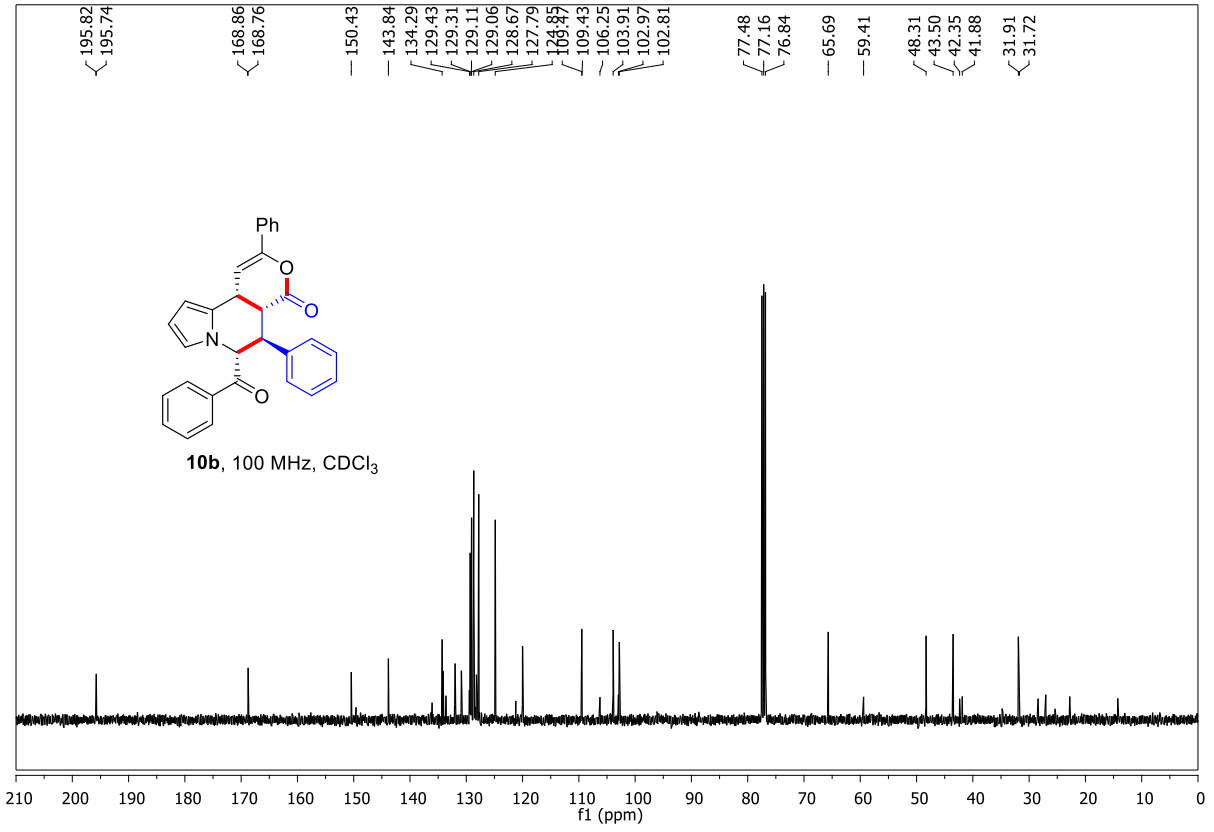
(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10a)



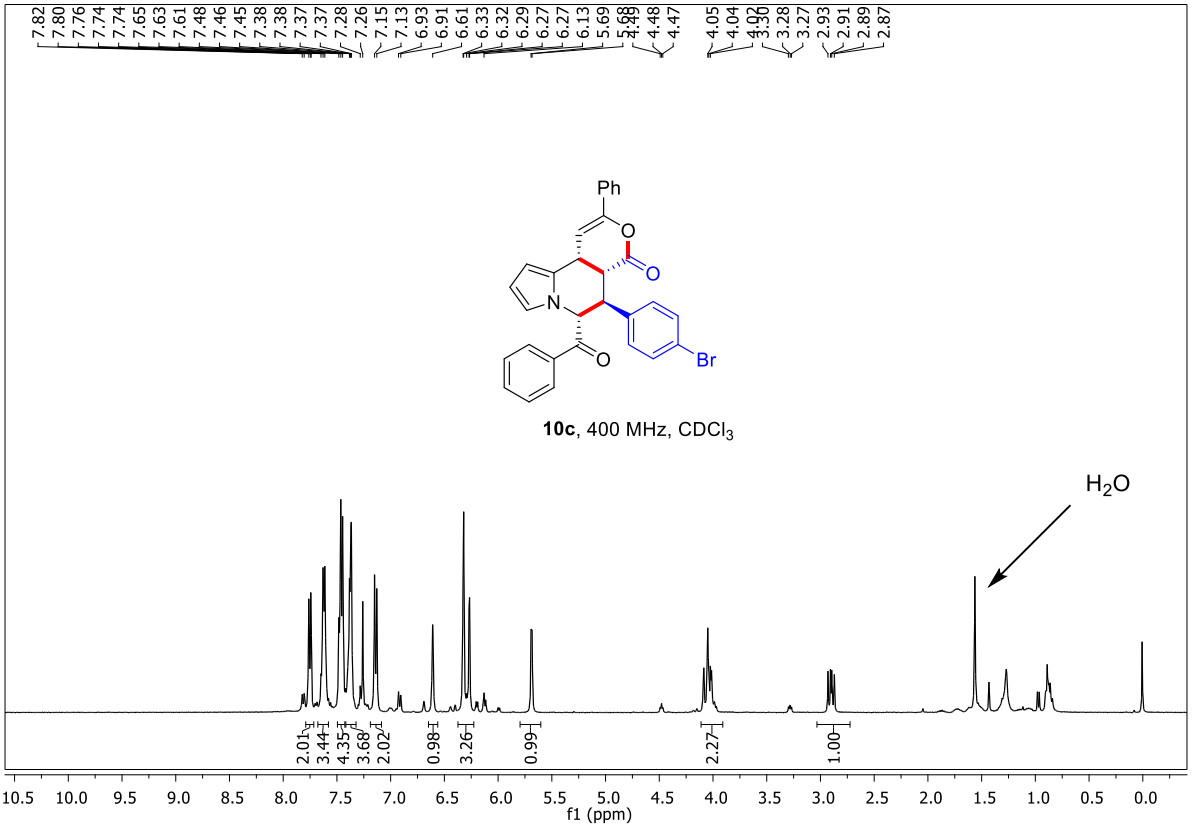


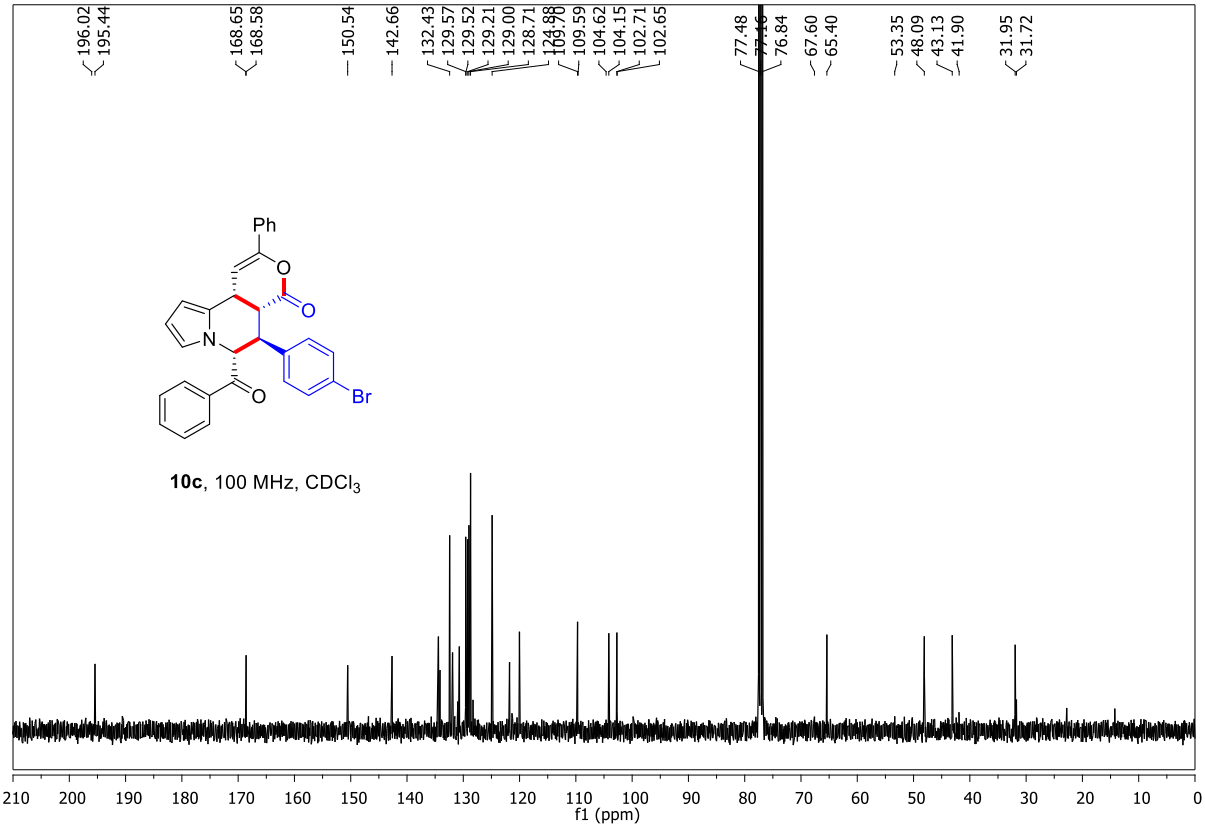
(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-2,5-diphenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g] indolizin-4-one (10b)



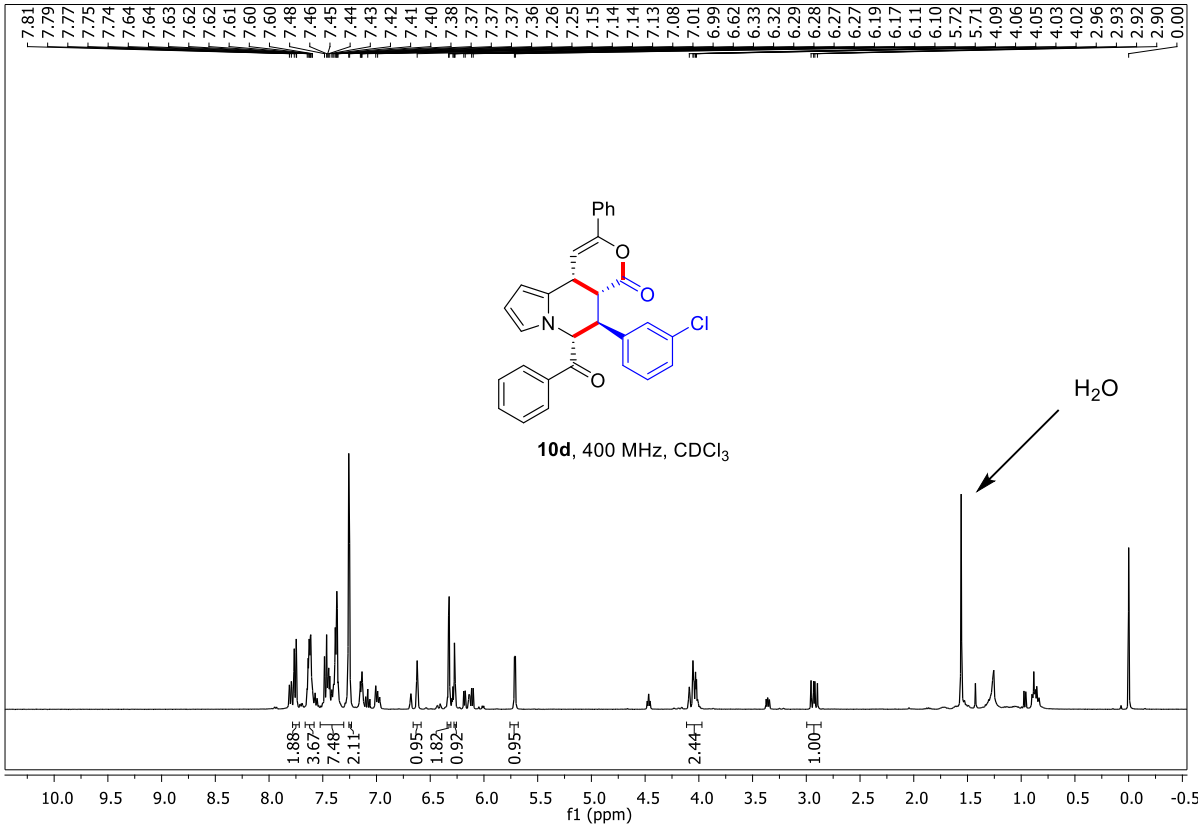


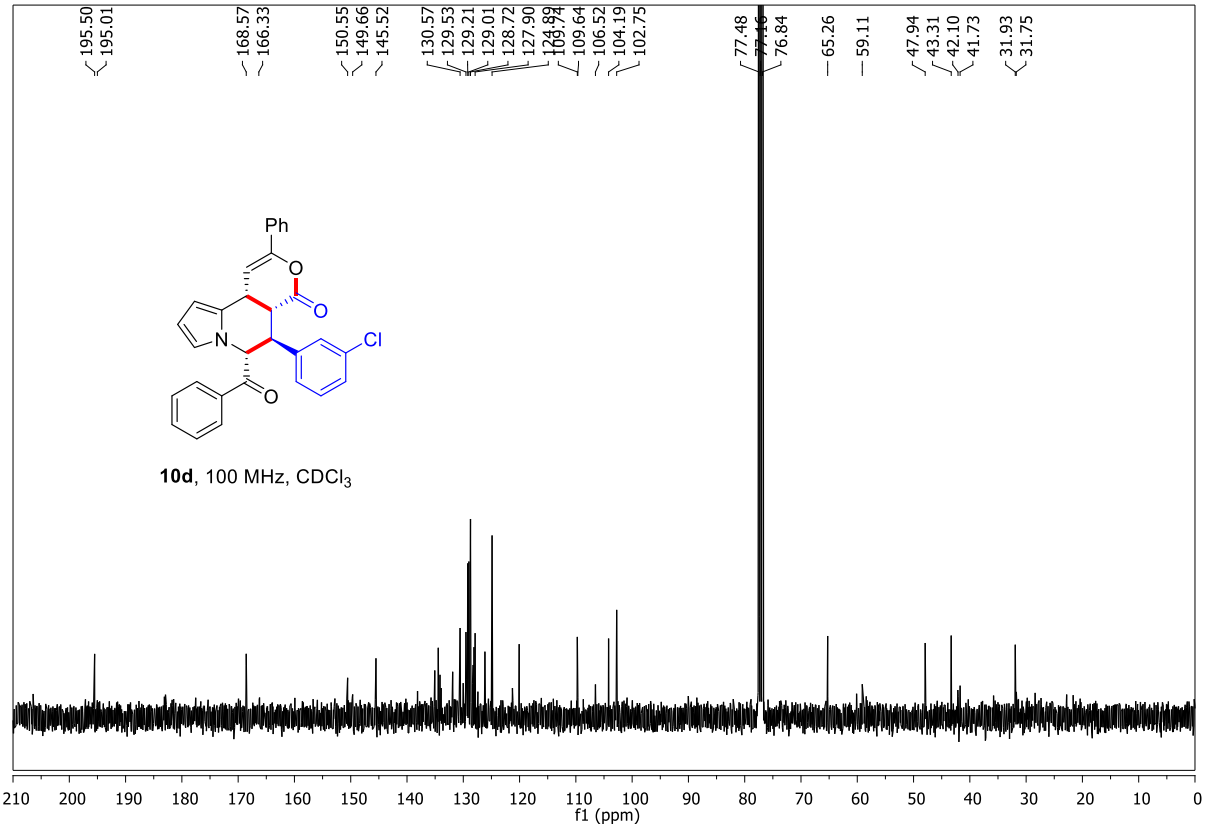
(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-5-(4-bromophenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10c)



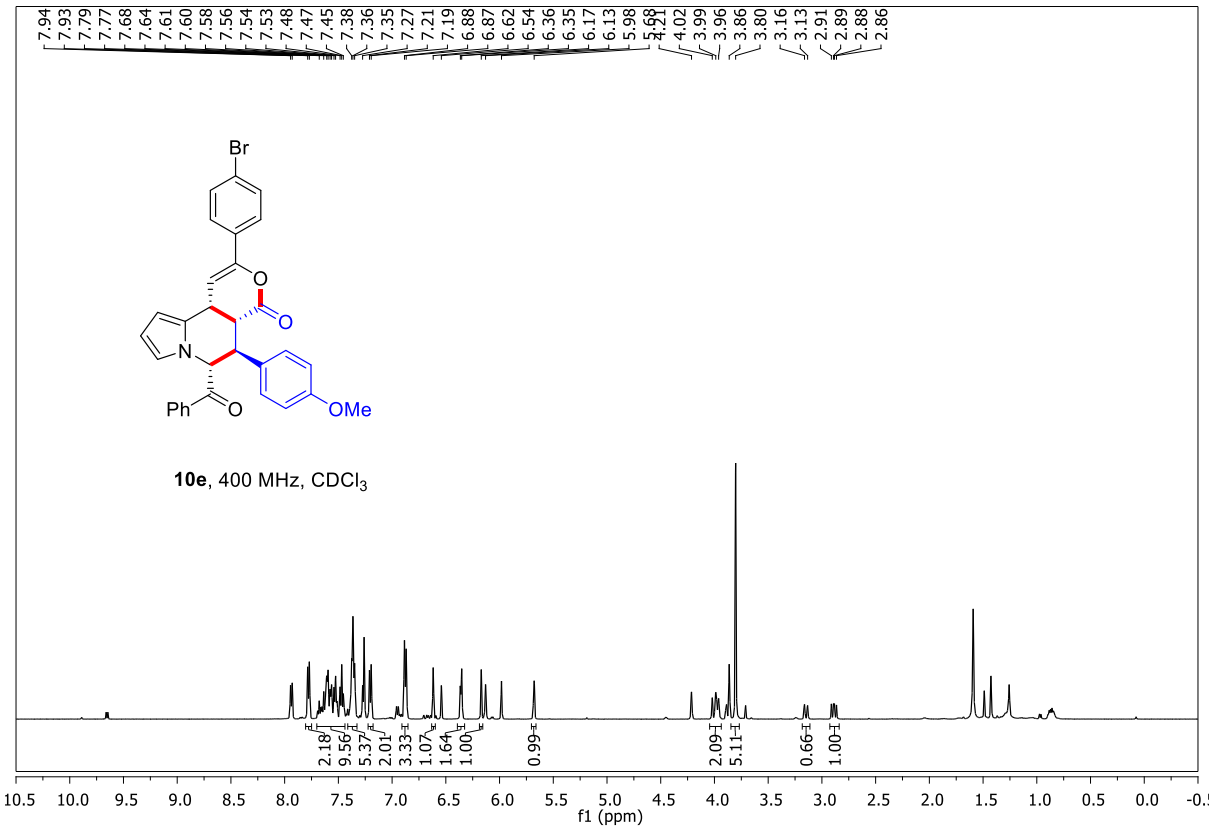


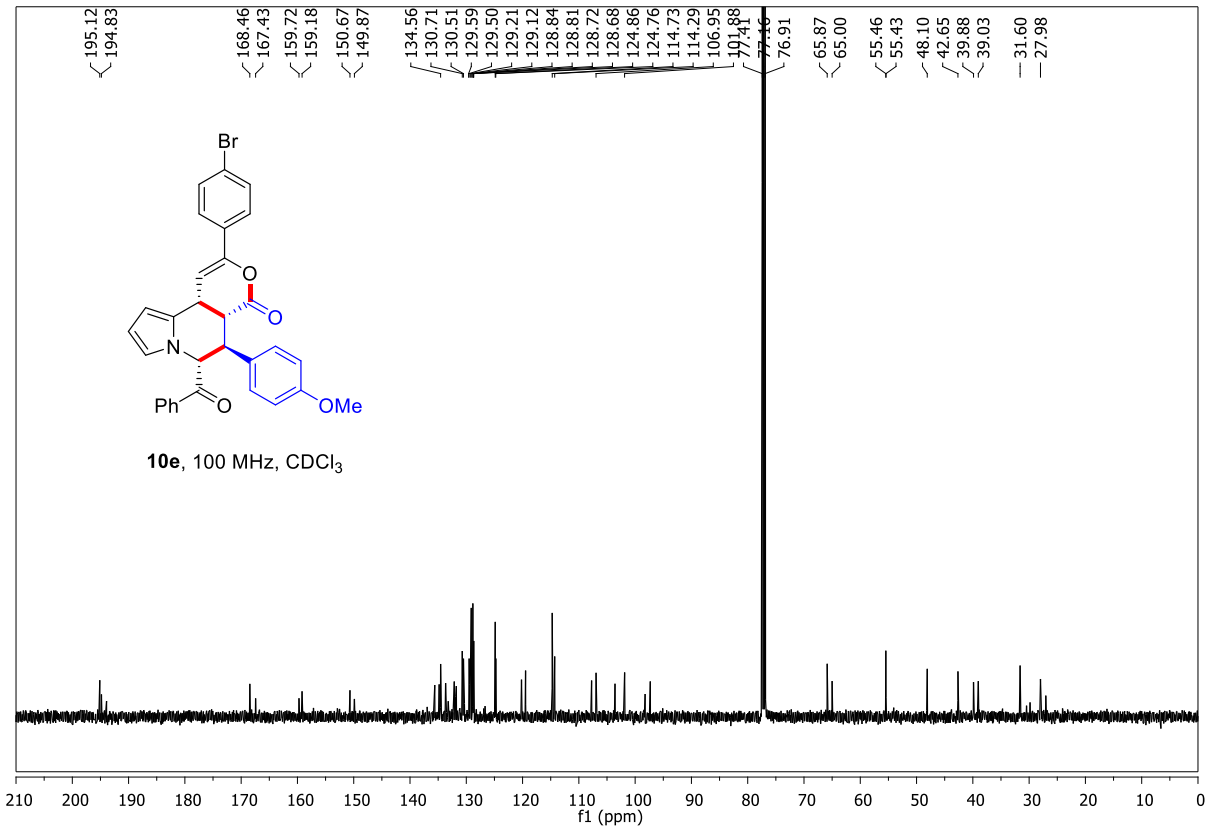
(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-5-(3-chlorophenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10d)



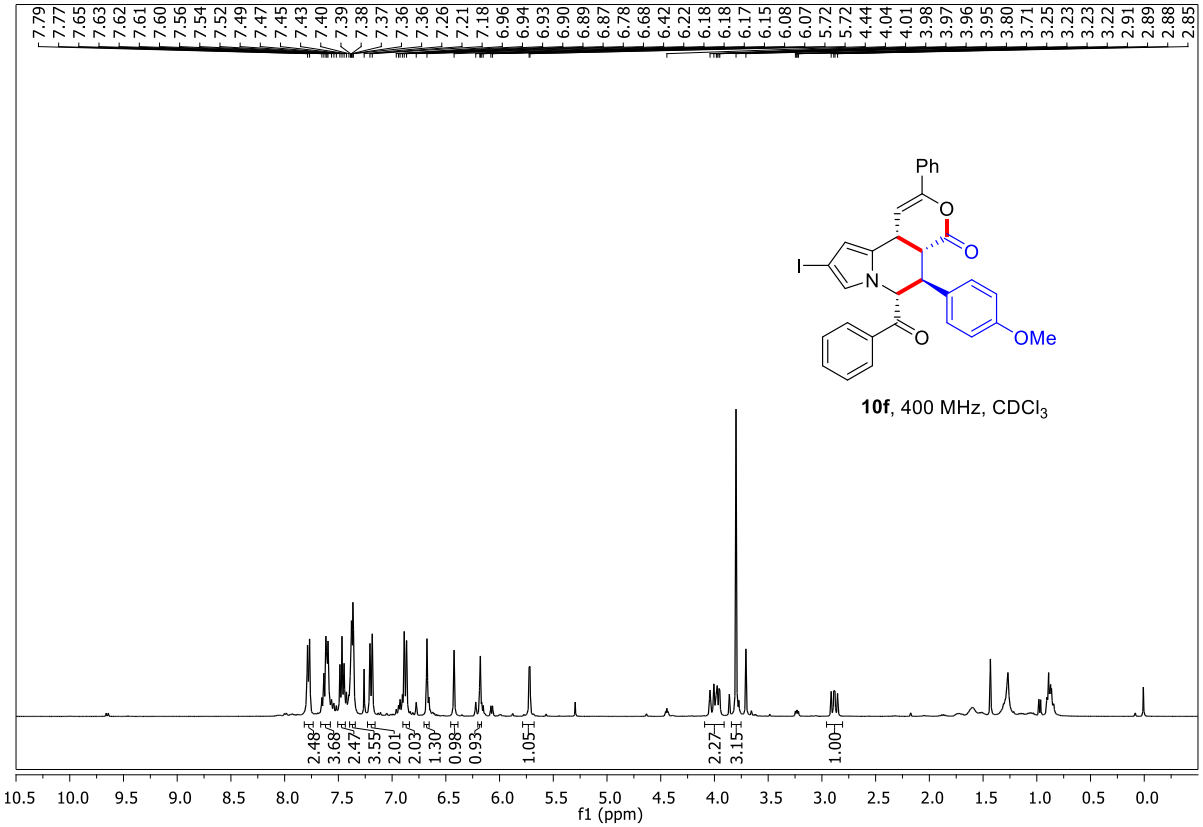


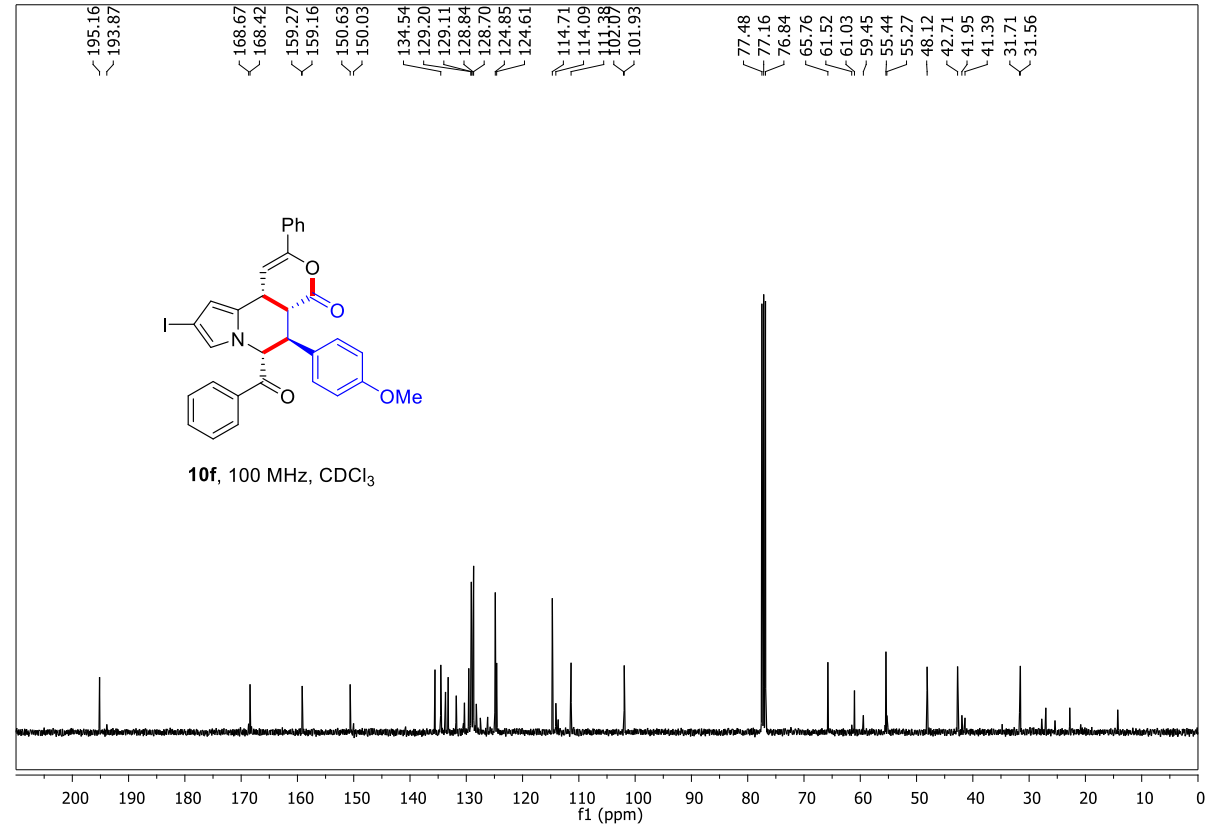
(4a*R*,5*S*,6*R*,10*bR*)-6-Benzoyl-2-(4-bromophenyl)-5-(4-methoxyphenyl)-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10e)



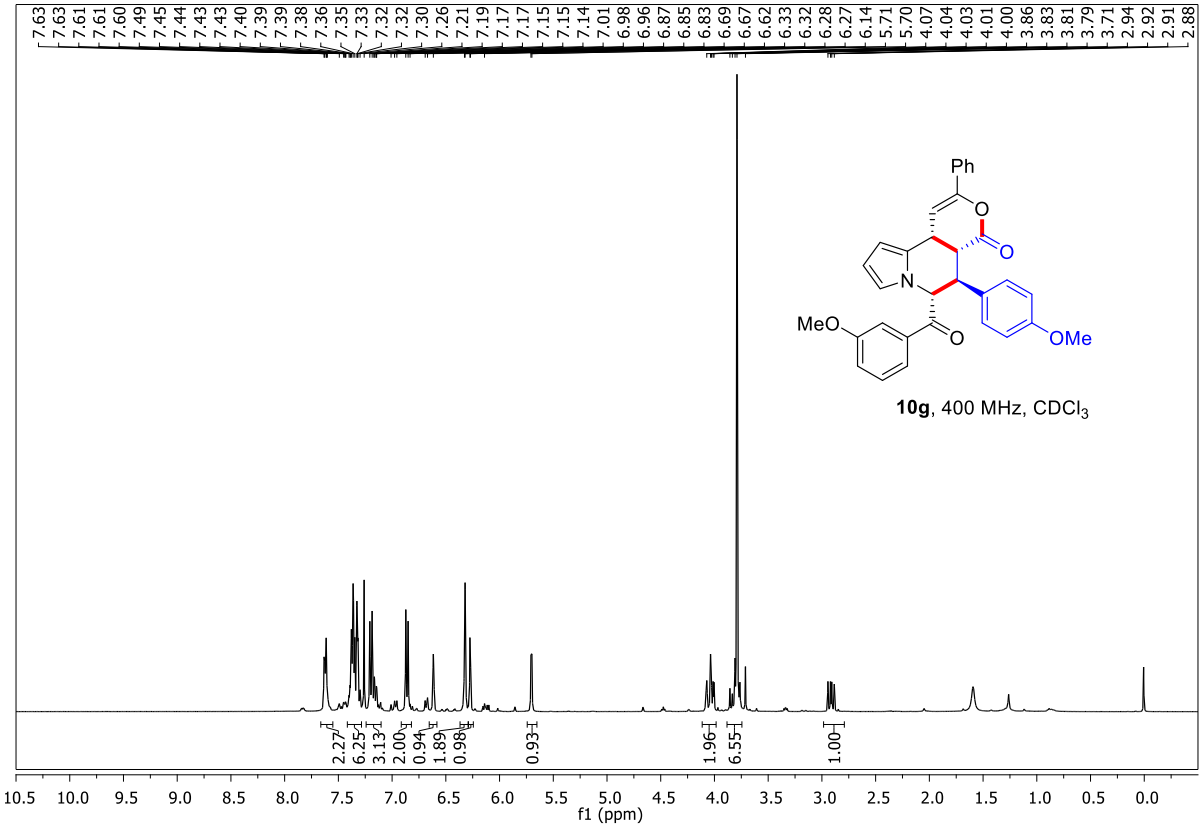


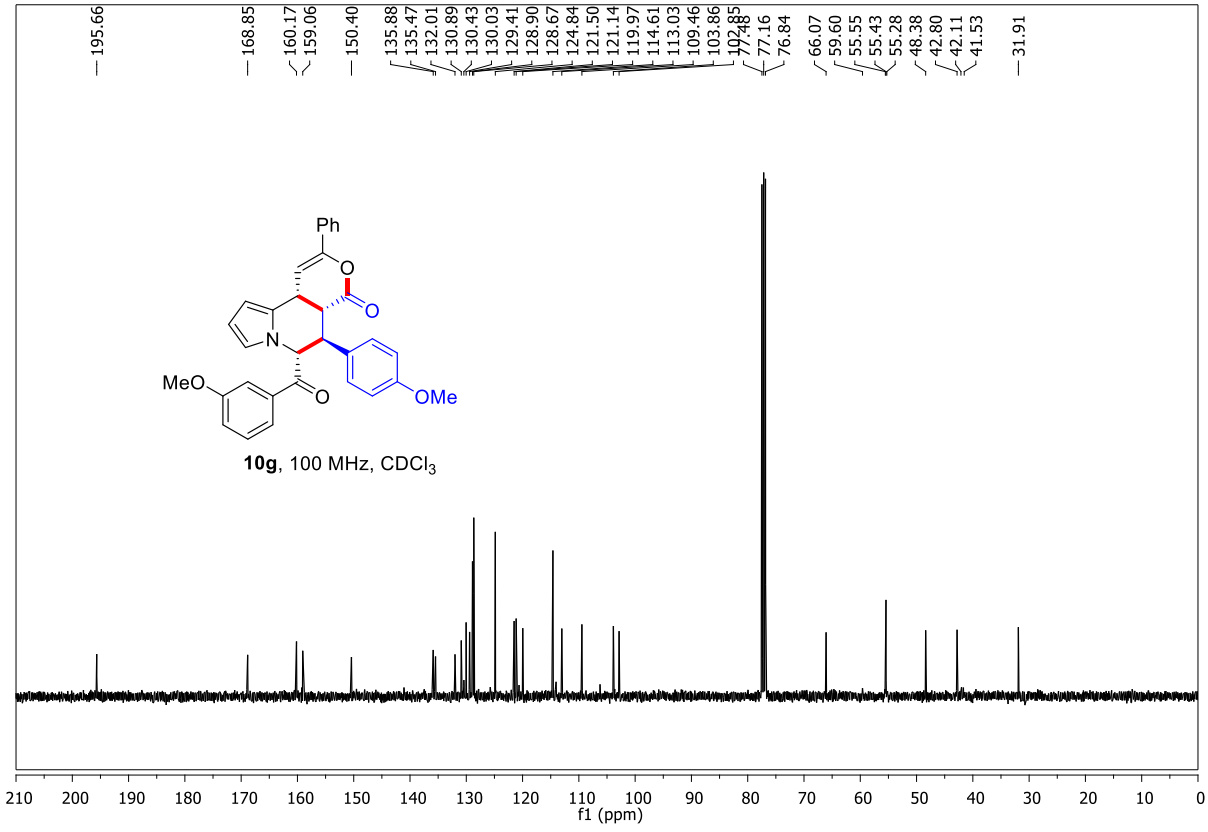
(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-9-iodo-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10f)



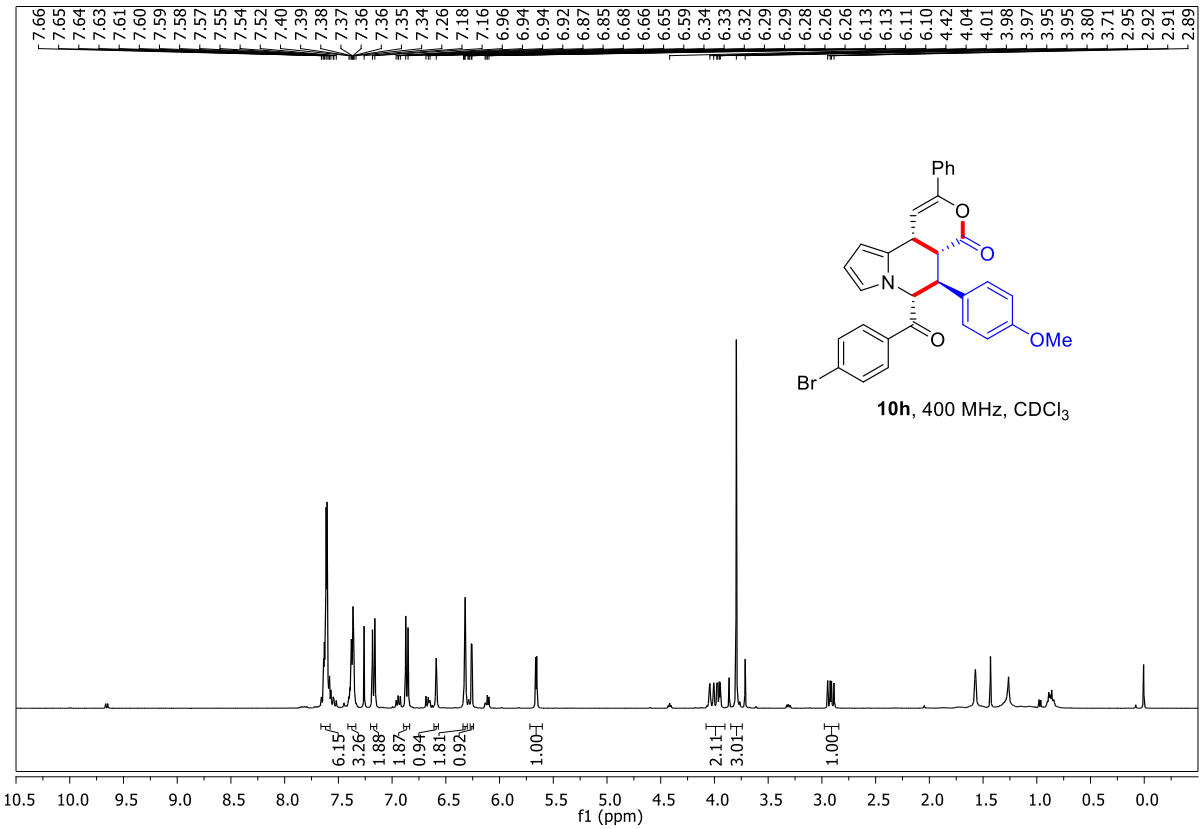


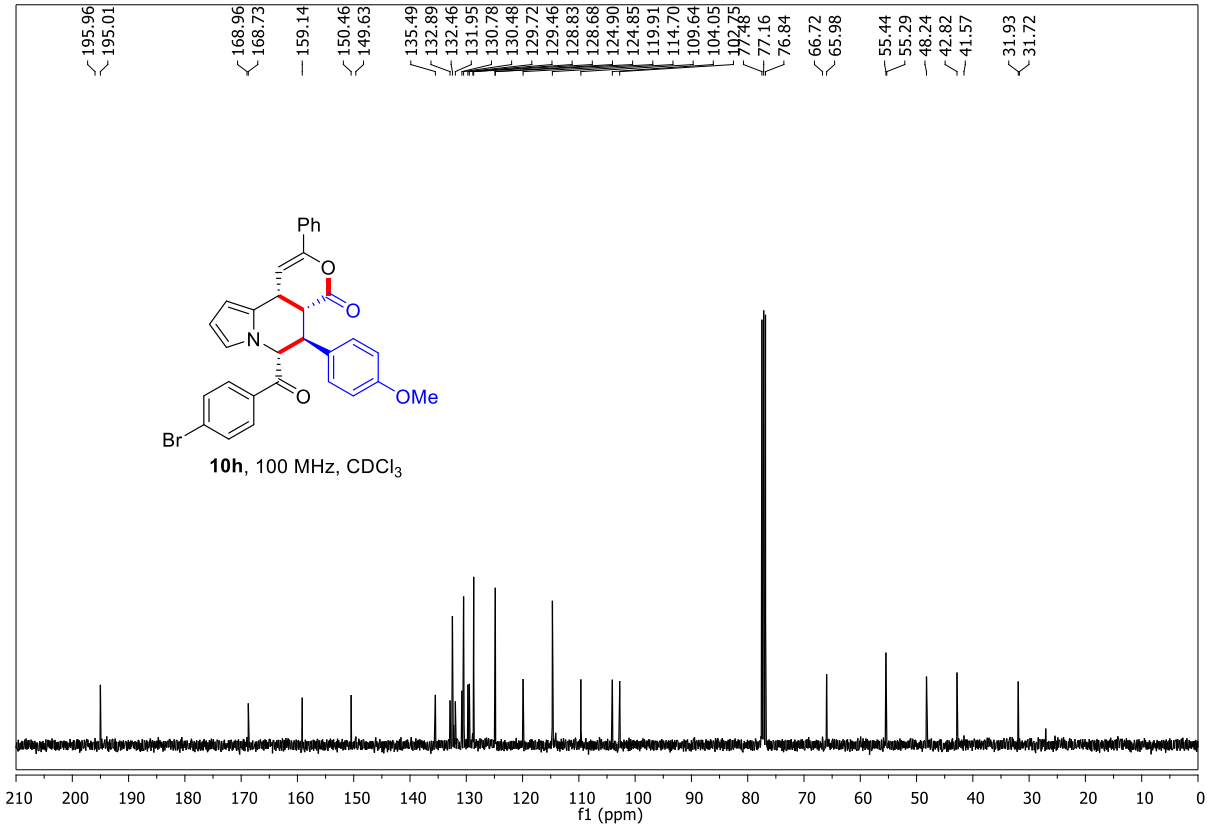
(4a*R*,5*S*,6*R*,10b*R*)-6-(3-Methoxybenzoyl)-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10g)



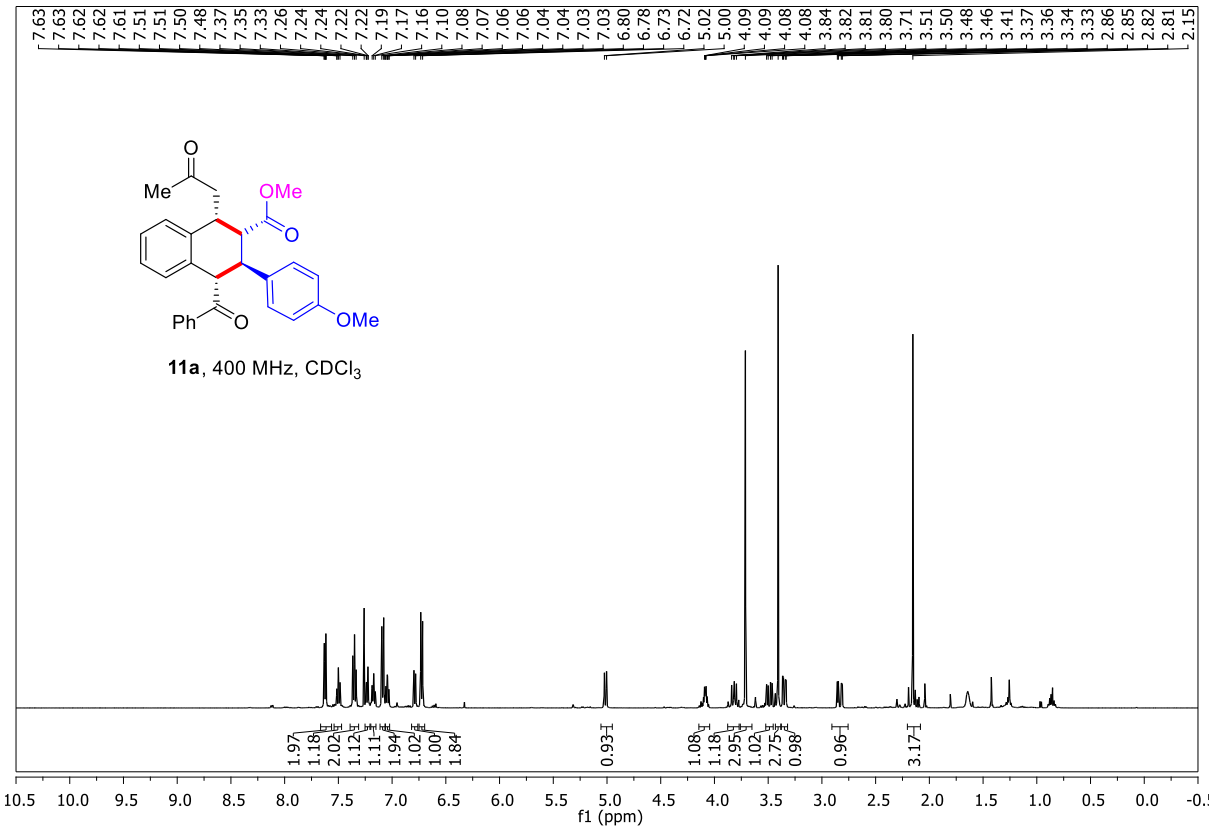


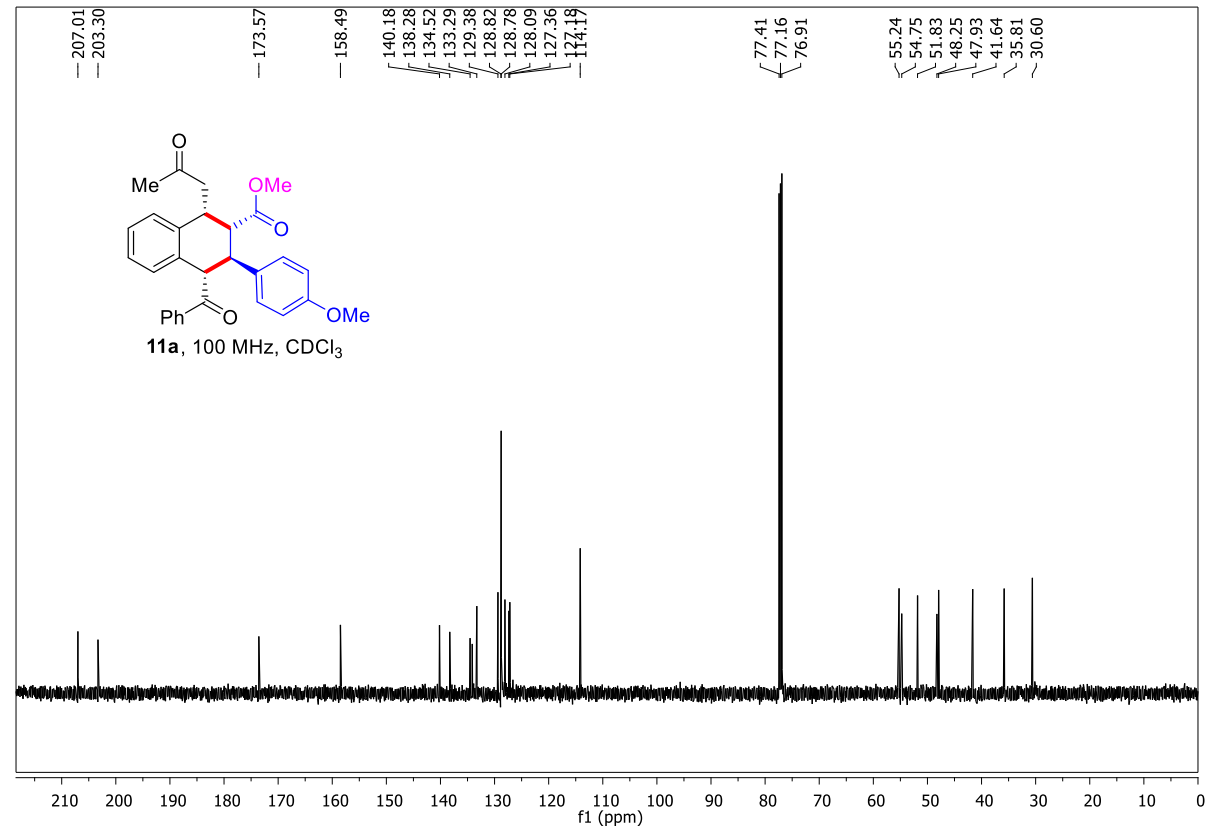
(4a*R*,5*S*,6*R*,10b*R*)-6-(4-Bromobenzoyl)-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10h)



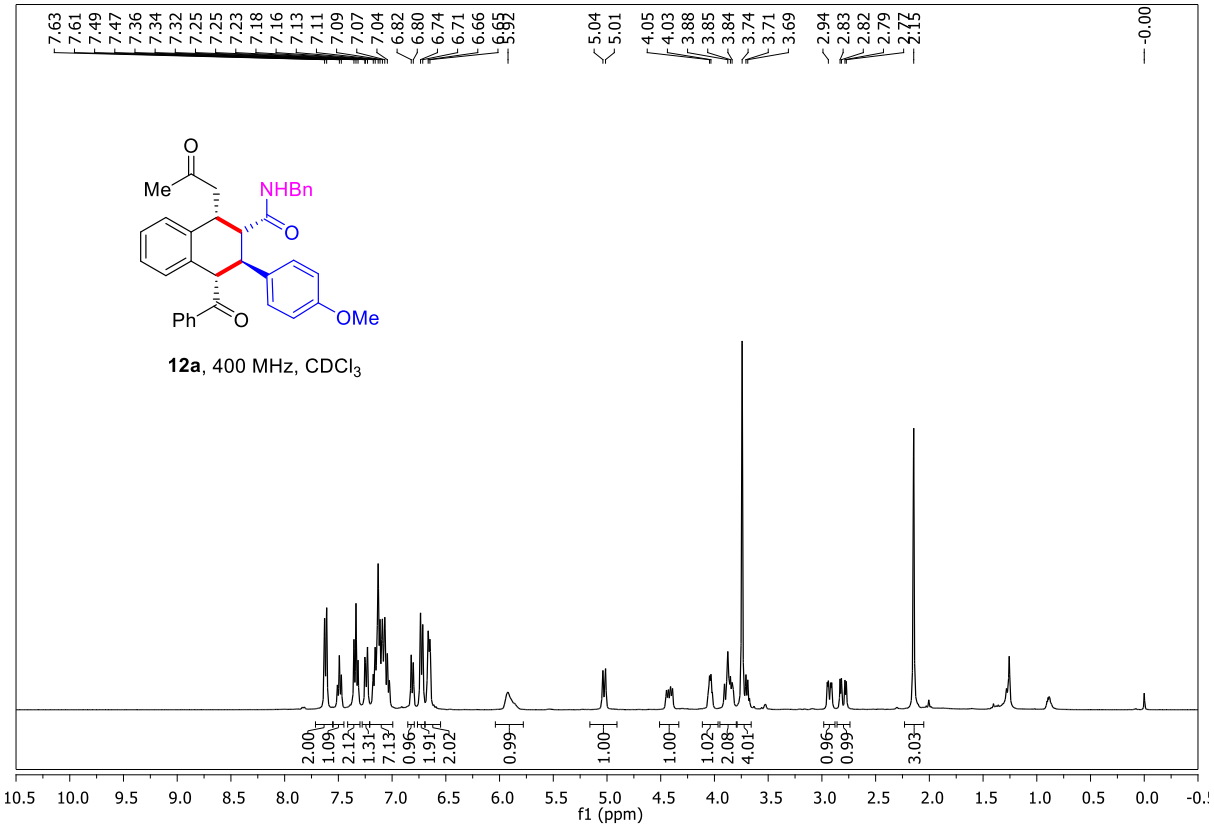


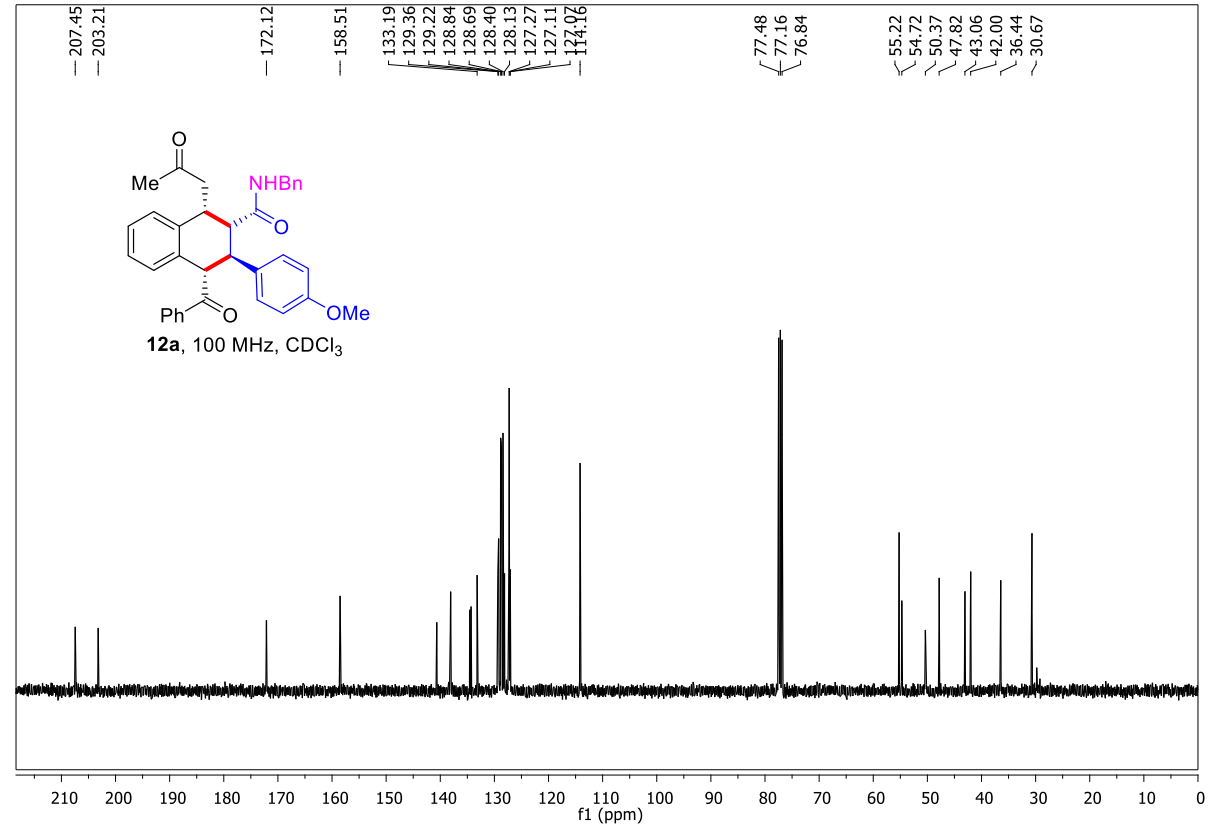
Methyl (2*S*,3*S*,4*S*)-4-benzoyl-3-(4-methoxyphenyl)-1-(2-oxopropyl)-1,2,3,4-tetrahydro naphthalene-2-carboxylate (11a)



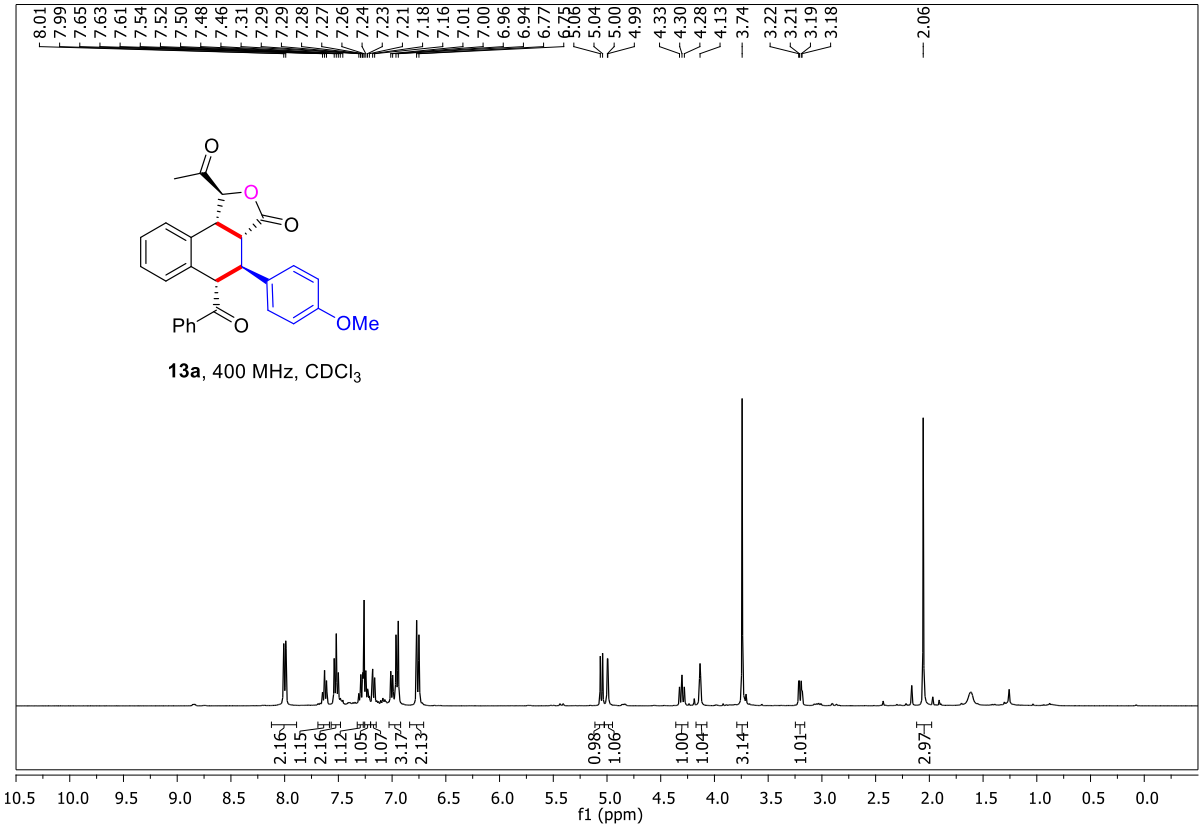


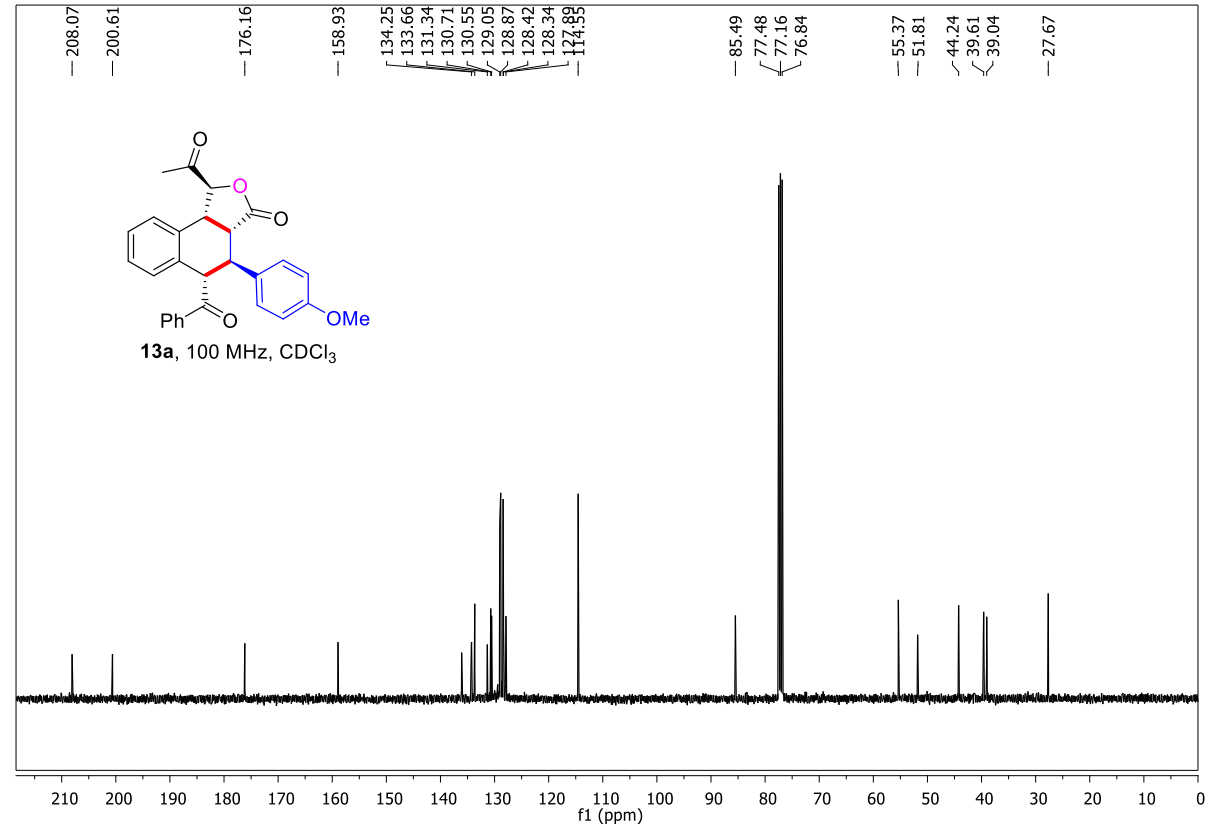
(2*S*,3*S*,4*S*)-4-Benzoyl-N-benzyl-3-(4-methoxyphenyl)-1-(2-oxopropyl)-1,2,3,4-tetrahydro naphthalene-2-carboxamide (12a)





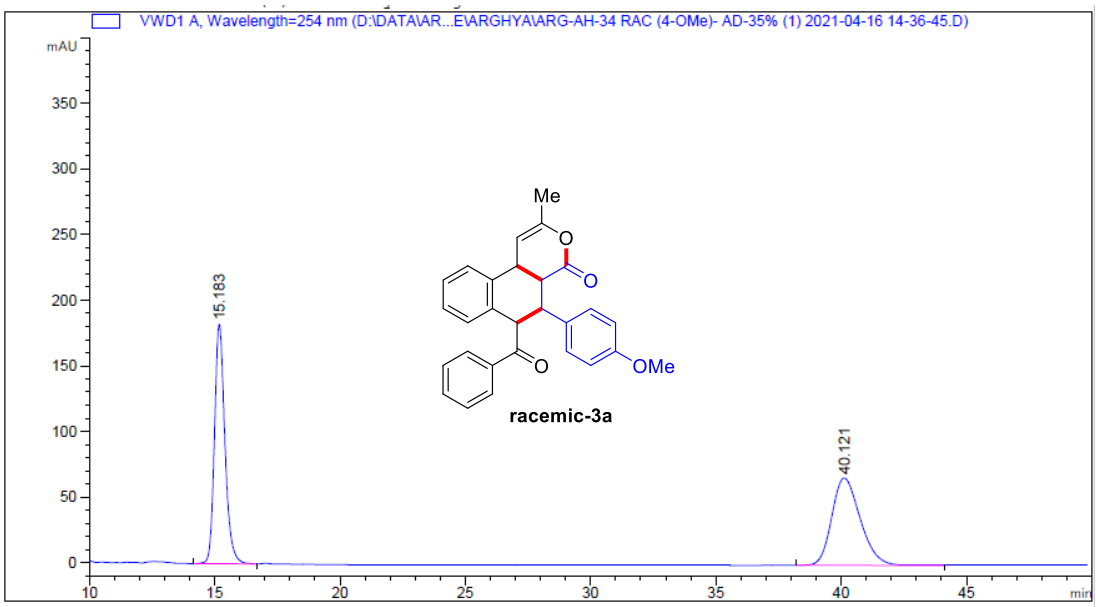
(1*S*,3a*R*,4*S*,5S,9b*R*)-1-Acetyl-5-benzoyl-4-(4-methoxyphenyl)-3a,4,5,9b-tetrahydro naphtho[1,2-c]furan-3(1*H*)-one (13a)



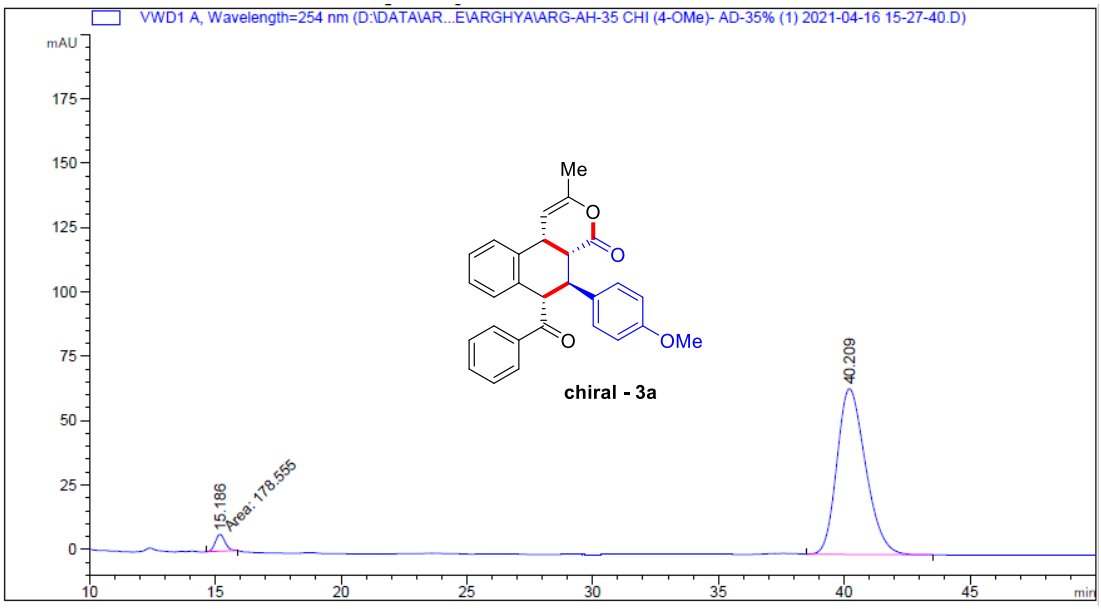


10.HPLC Data of Functionalized Tricyclic δ- Lactones

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3a)



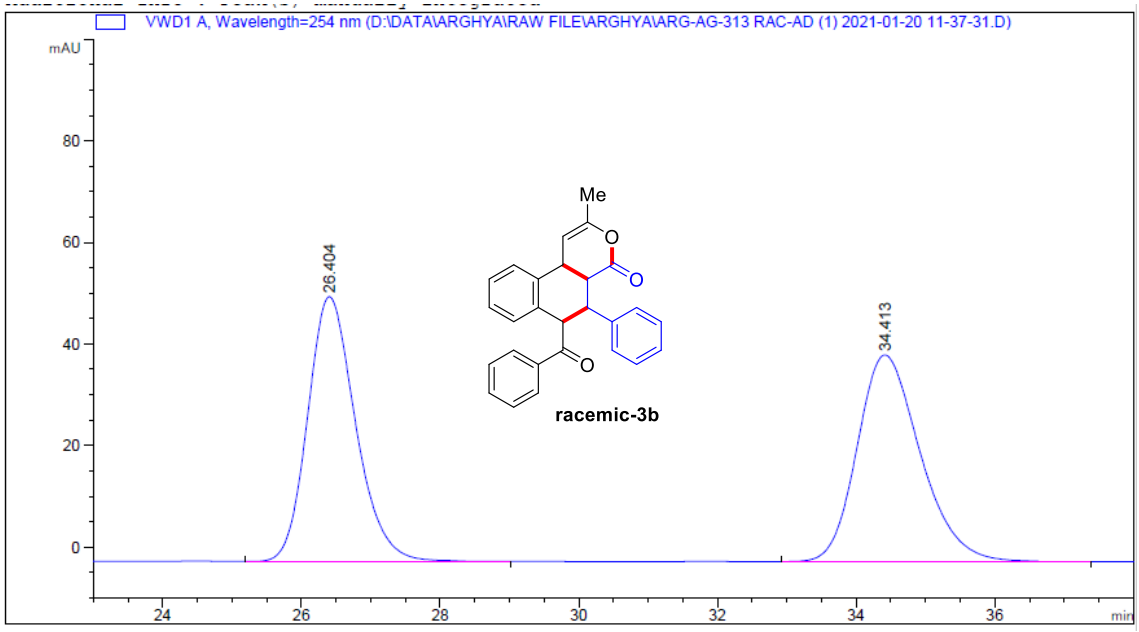
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.183	BB	0.4420	5287.00488	182.47456	50.2245
2	40.121	BB	1.2106	5239.74219	66.54926	49.7755



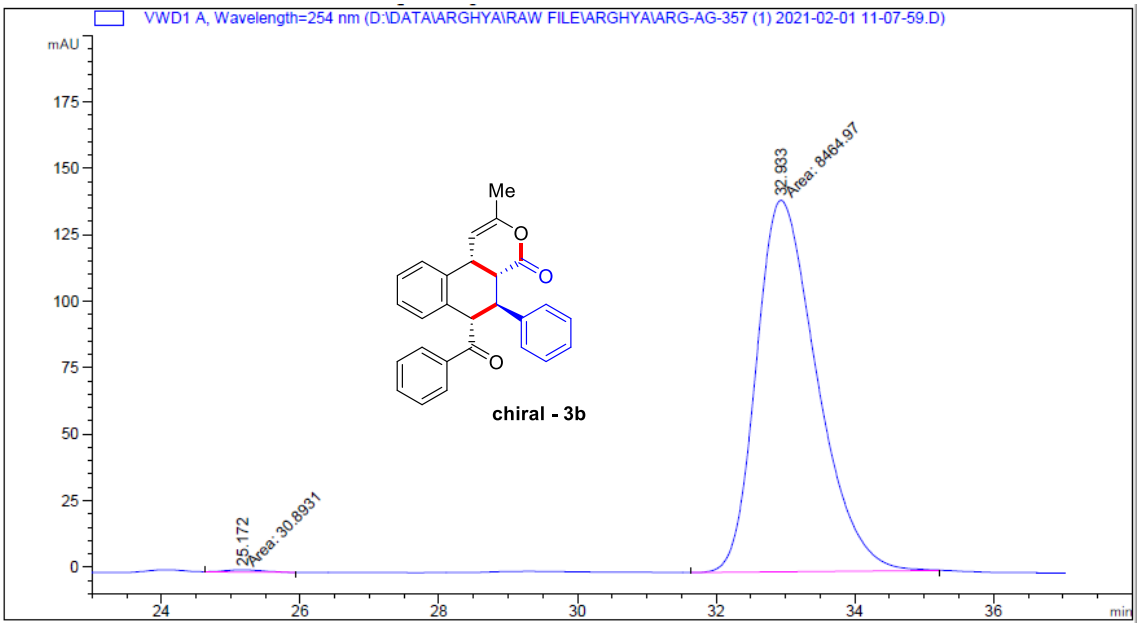
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.186	MM	0.4536	178.55479	6.56136	3.3868
2	40.209	BB	1.2182	5093.52344	64.30157	96.6132

Sample Info : CHIRALPAK AD, 35% IPA-HEXANE, .7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-phenyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*] isochromen-4-one (3b)



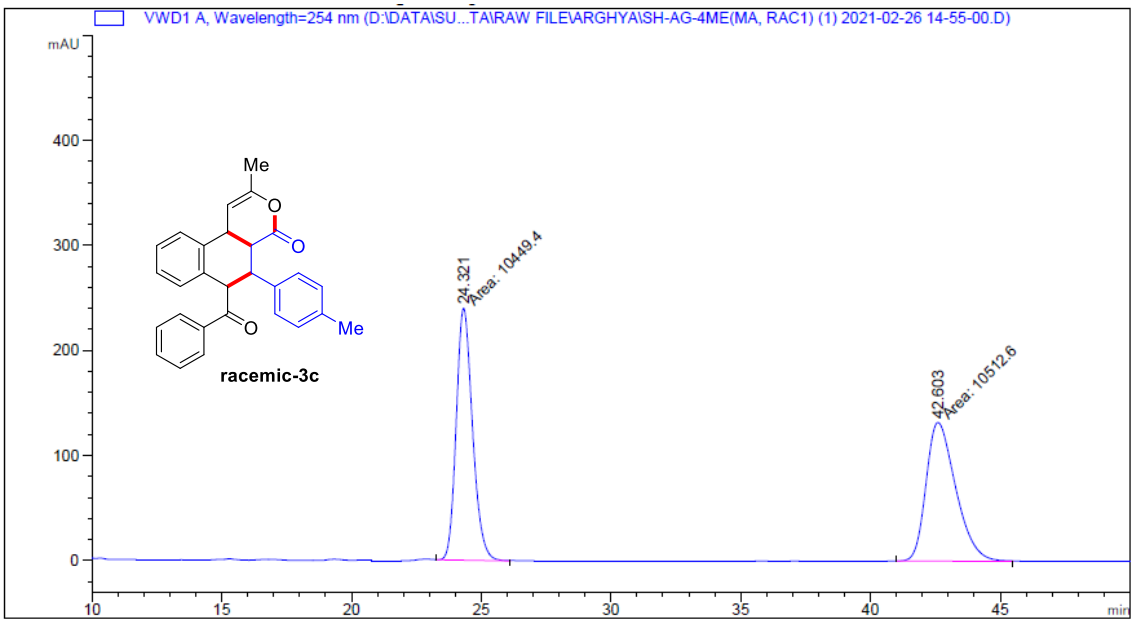
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.404	BB	0.7343	2500.38354	52.15963	49.2873
2	34.413	BB	0.9666	2572.69678	40.69034	50.7127



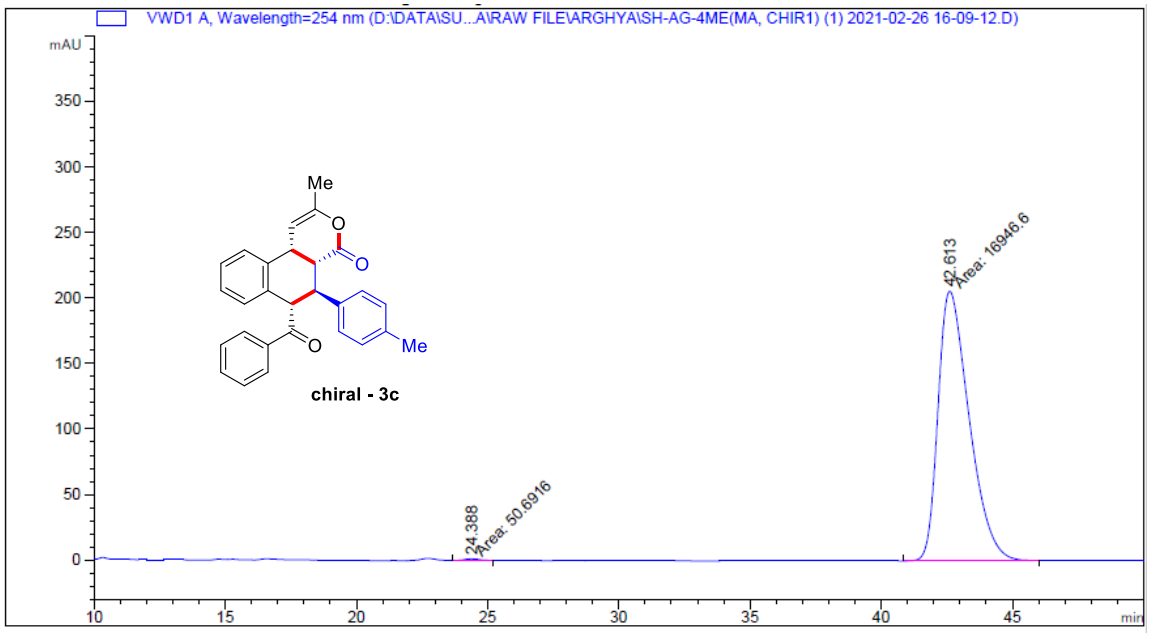
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.172	MM	0.6127	30.89309	8.40368e-1	0.3636
2	32.933	MM	1.0101	8464.97461	139.66795	99.6364

Sample Info : CHIRALPAK AD, 20% IPA-HEXANE, .7 mL/min.,254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(*p*-tolyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3c)



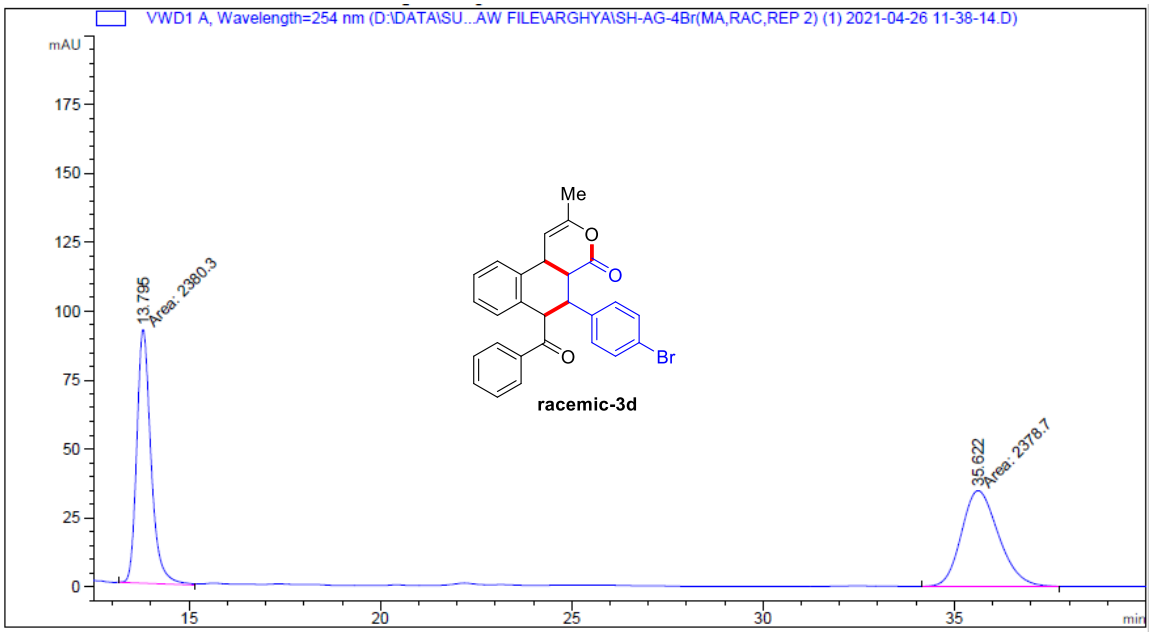
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.321	MM	0.7264	1.04494e4	239.75114	49.8493
2	42.603	MM	1.3275	1.05126e4	131.98260	50.1507



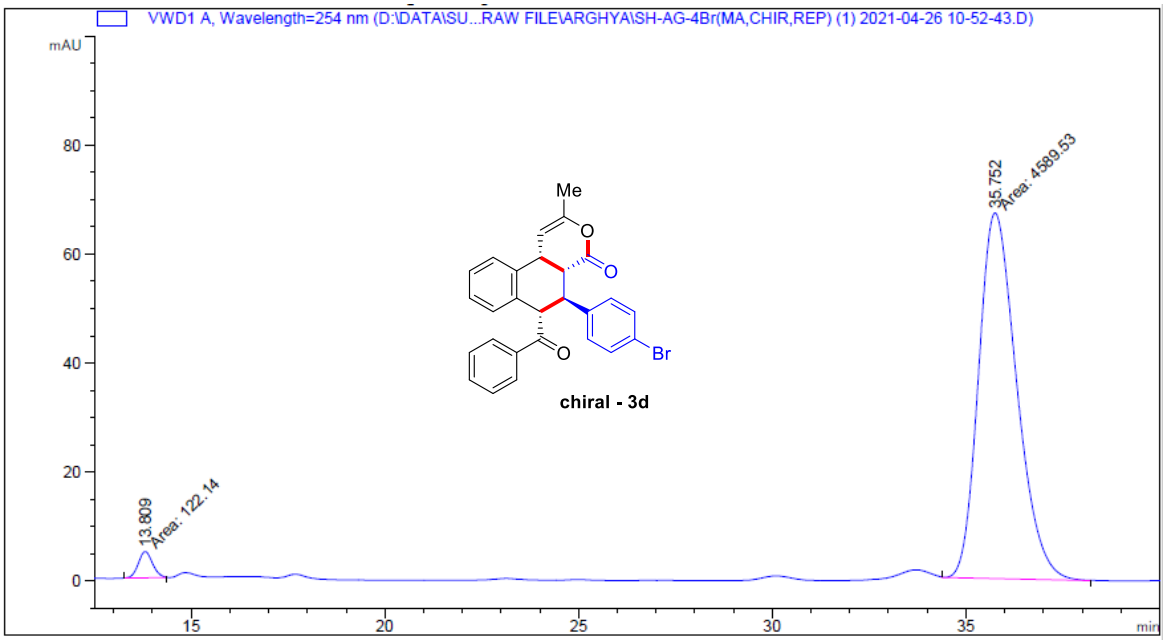
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.388	MM	0.6689	50.69158	1.26310	0.2982
2	42.613	MM	1.3741	1.69466e4	205.55139	99.7018

Sample Info : CHIRALPAK AD, 20 % IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-bromophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3d)



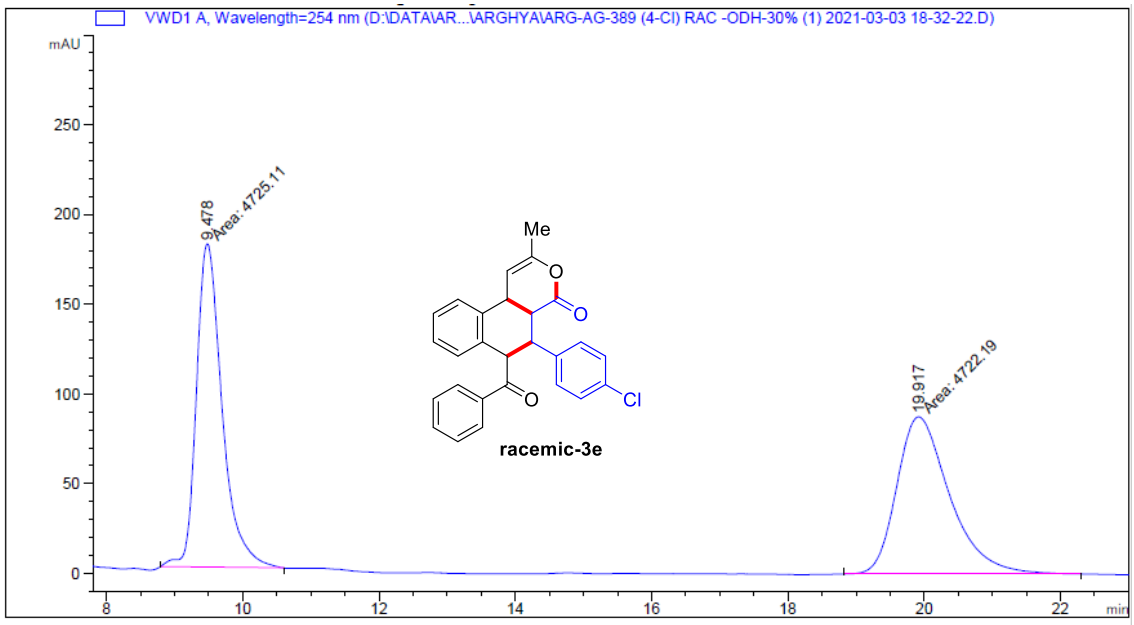
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.795	MM	0.4312	2380.29907	92.00716	50.0168
2	35.622	MM	1.1359	2378.70215	34.90316	49.9832



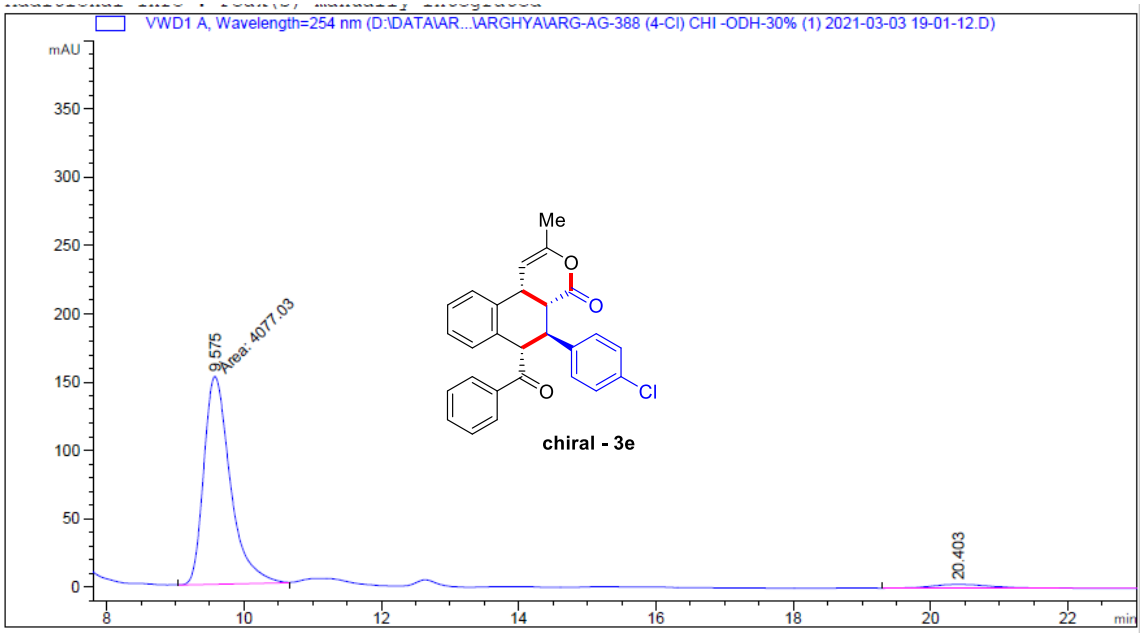
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.809	MM	0.4232	122.14046	4.81041	2.5923
2	35.752	MM	1.1404	4589.53271	67.07635	97.4077

Sample Info : CHIRALPAK AD, 30% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5S,6*S*,10b*R*)-6-Benzoyl-5-(4-chlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3e)



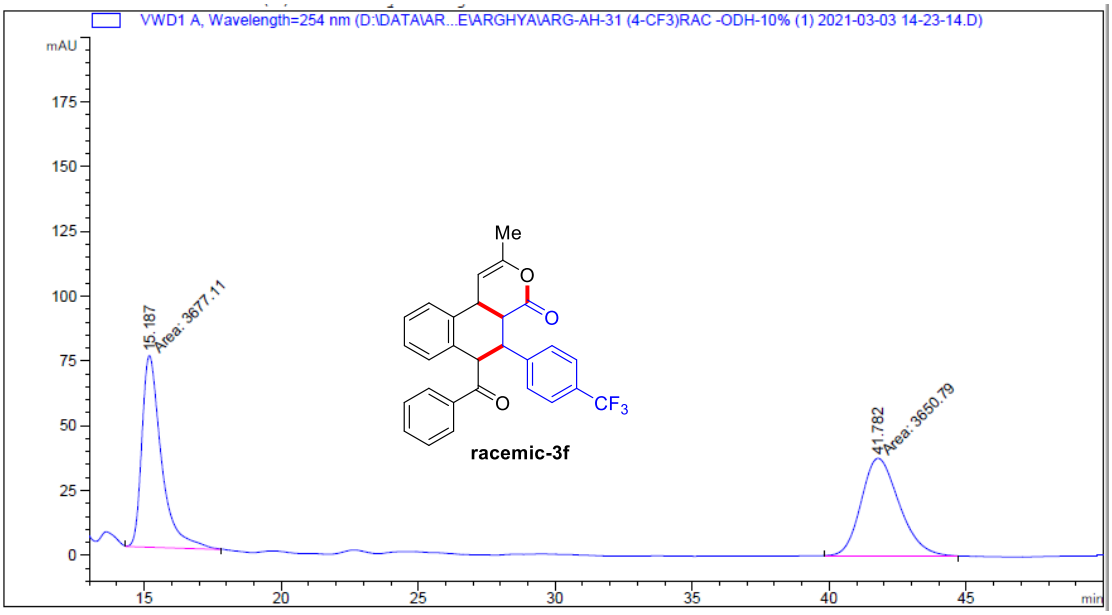
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.478	MM	0.4372	4725.11230	180.14320	50.0154
2	19.917	MM	0.8991	4722.19434	87.53761	49.9846



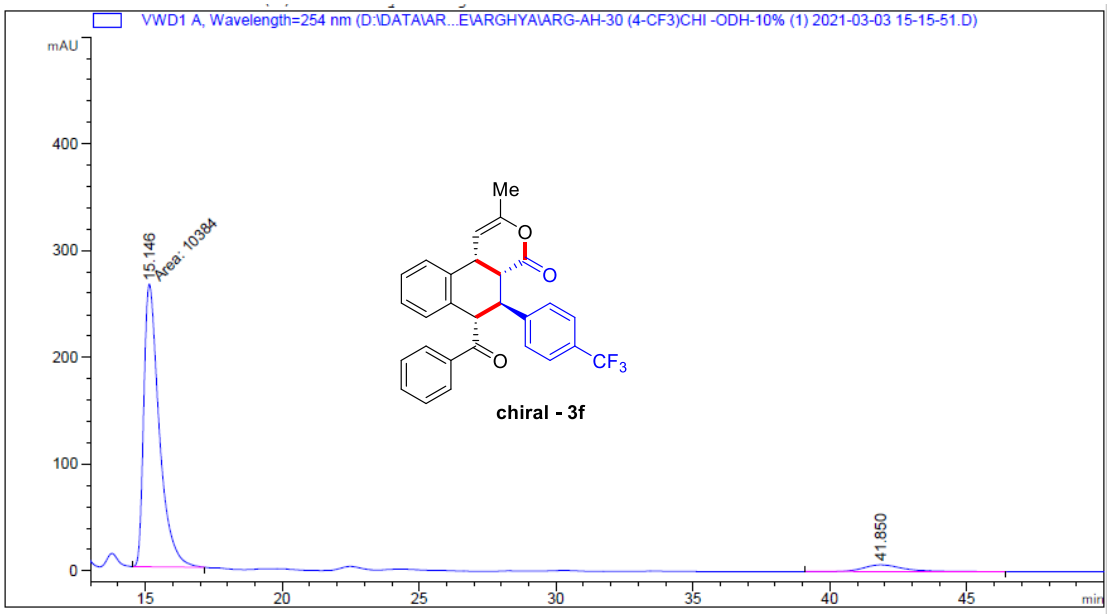
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.575	MM	0.4469	4077.03345	152.03781	95.8818
2	20.403	BB	0.8603	175.11208	2.77459	4.1182

Sample Info : CHIRALCEL-ODH, 30% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(4-(trifluoromethyl)phenyl)-4a,5,6,10b-tetrahydro-4H-benzo[*f*]isochromen-4-one (3f)



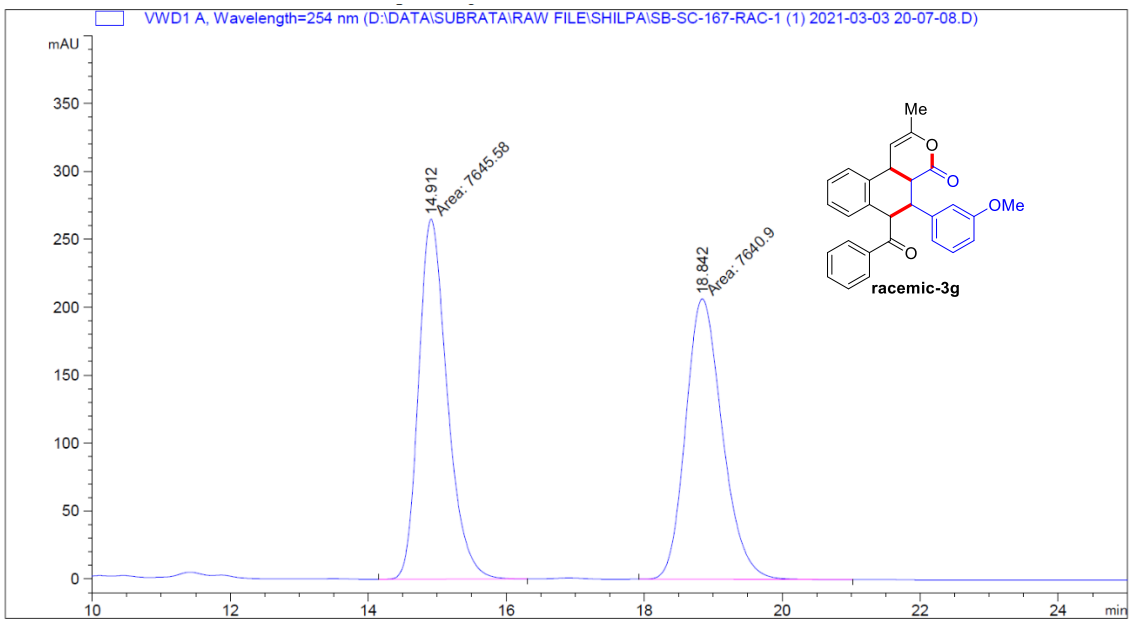
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.187	MM	0.8287	3677.11255	73.94974	50.1796
2	41.782	MM	1.6152	3650.79272	37.67184	49.8204



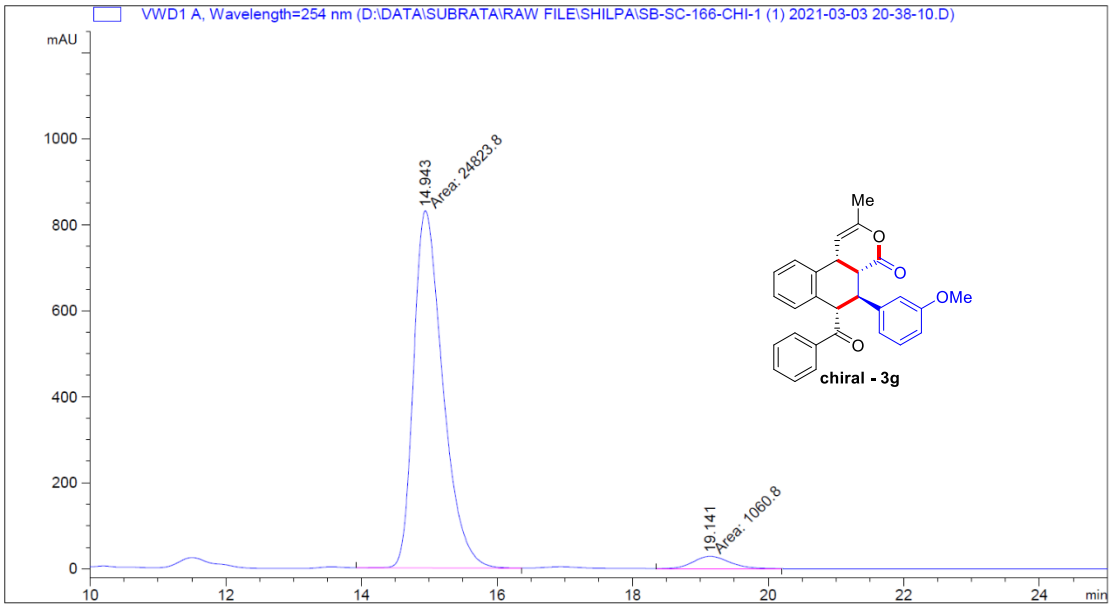
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.146	MM	0.6539	1.03840e4	264.67667	94.0625
2	41.850	BB	1.3802	655.47418	6.31387	5.9375

Sample Info : CHIRALCEL-ODH, 10% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3g)



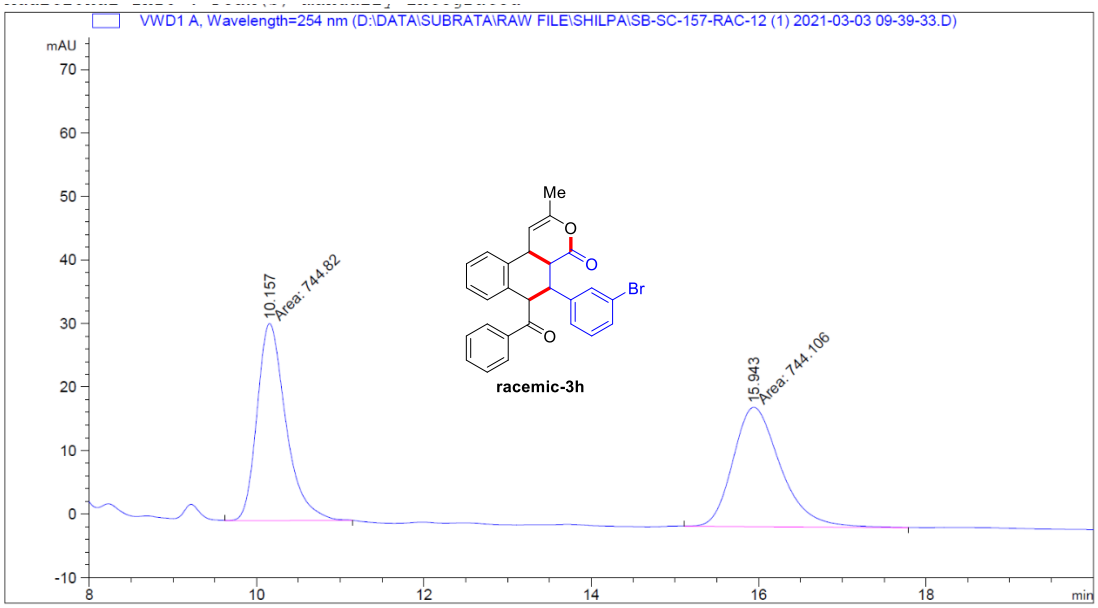
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.912	MM	0.4806	7645.57813	265.14667	50.0153
2	18.842	MM	0.6166	7640.89746	206.54028	49.9847



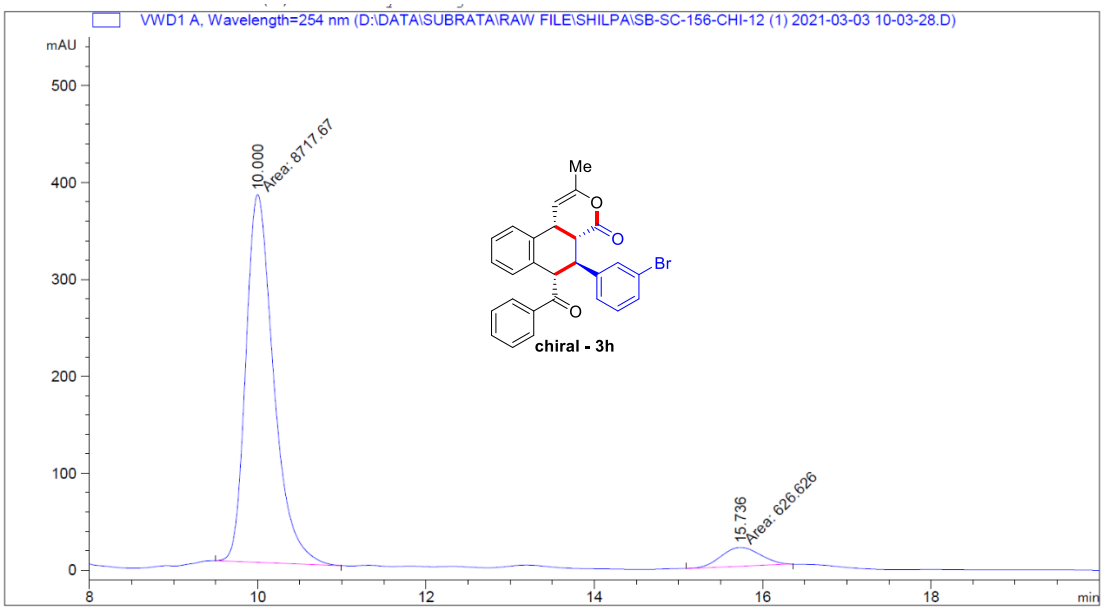
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.943	MM	0.4984	2.48238e4	830.15332	95.9018
2	19.141	MM	0.6179	1060.80078	28.61245	4.0982

Sample Info : CHIRALPAK-AD, 40% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3-bromophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3h)



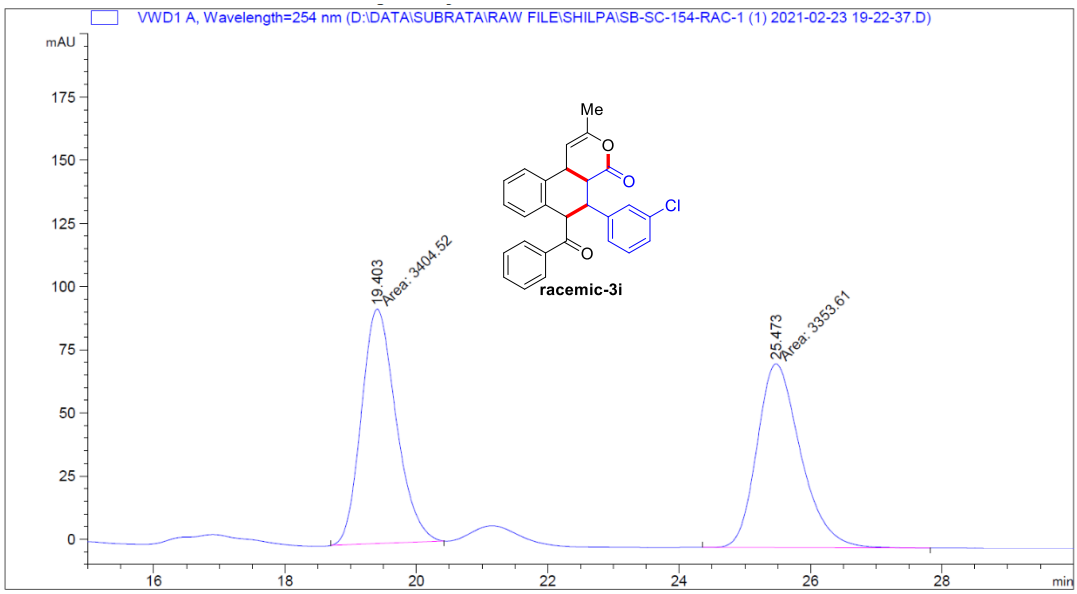
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.157	MM	0.3998	744.81976	31.05234	50.0240
2	15.943	MM	0.6596	744.10590	18.80138	49.9760



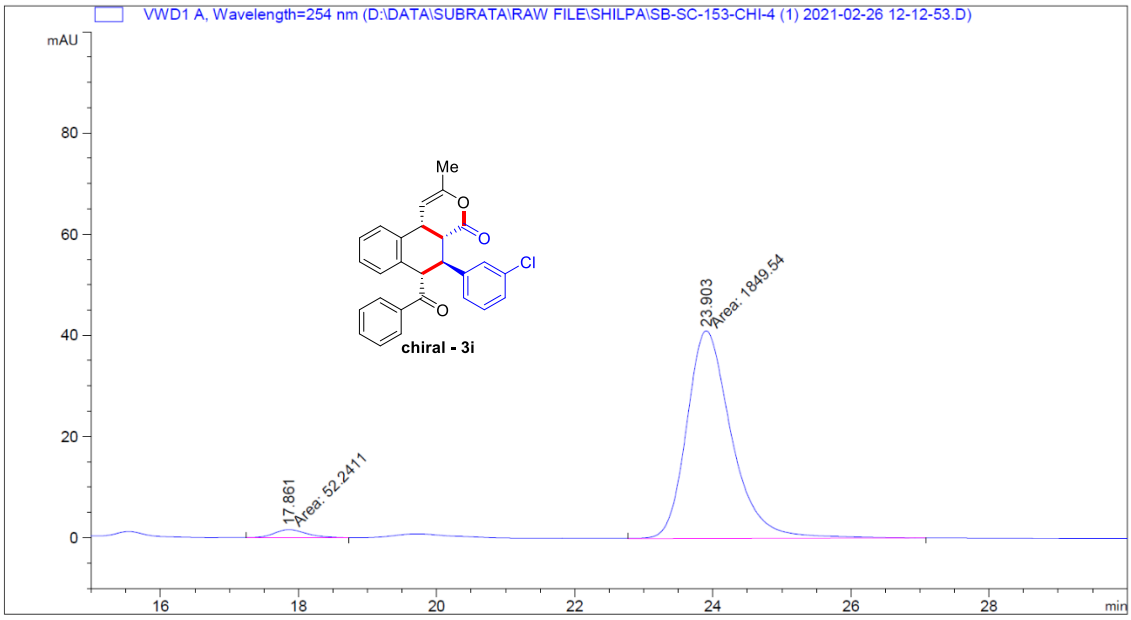
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.000	MM	0.3827	8717.67480	379.68018	93.2940
2	15.736	MM	0.5352	626.62598	19.51251	6.7060

Sample Info : CHIRALCEL-ODH, 30% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3-chlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3i)



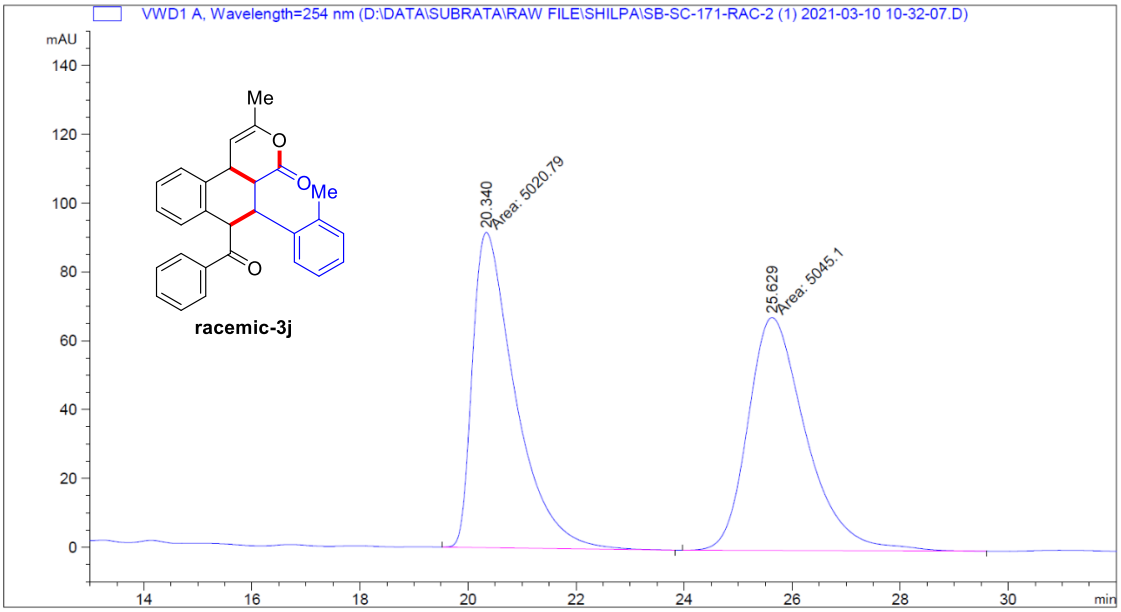
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.403	MM	0.6115	3404.52319	92.78700	50.3767
2	25.473	MM	0.7685	3353.61279	72.72897	49.6233



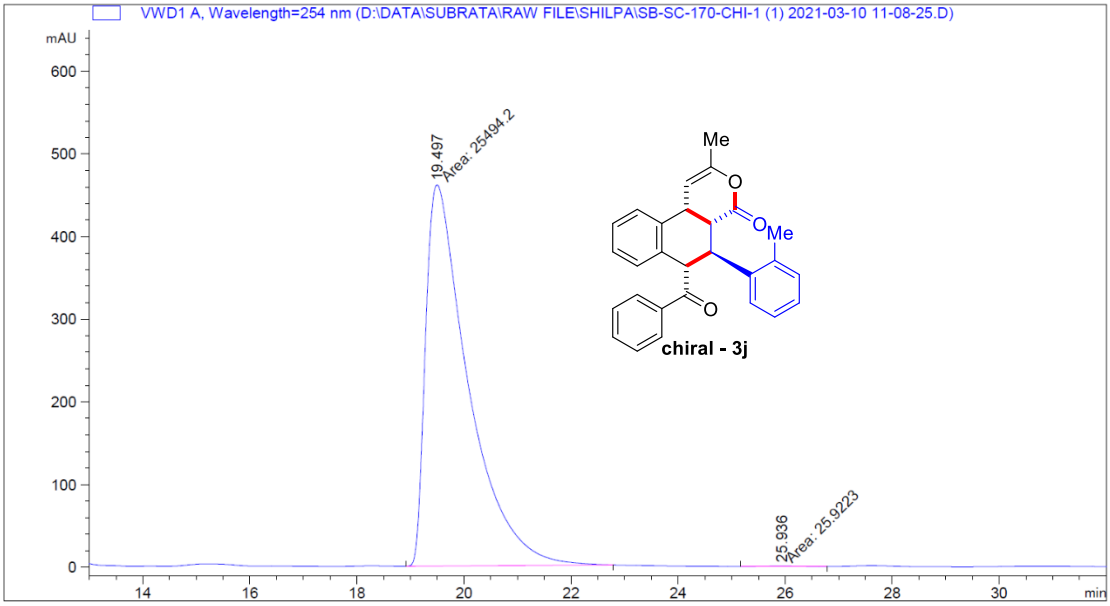
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.861	MM	0.5449	52.24108	1.59798	2.7470
2	23.903	MM	0.7514	1849.53821	41.02214	97.2530

Sample Info : CHIRALPAK AD, 20 % IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(o-tolyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3j)



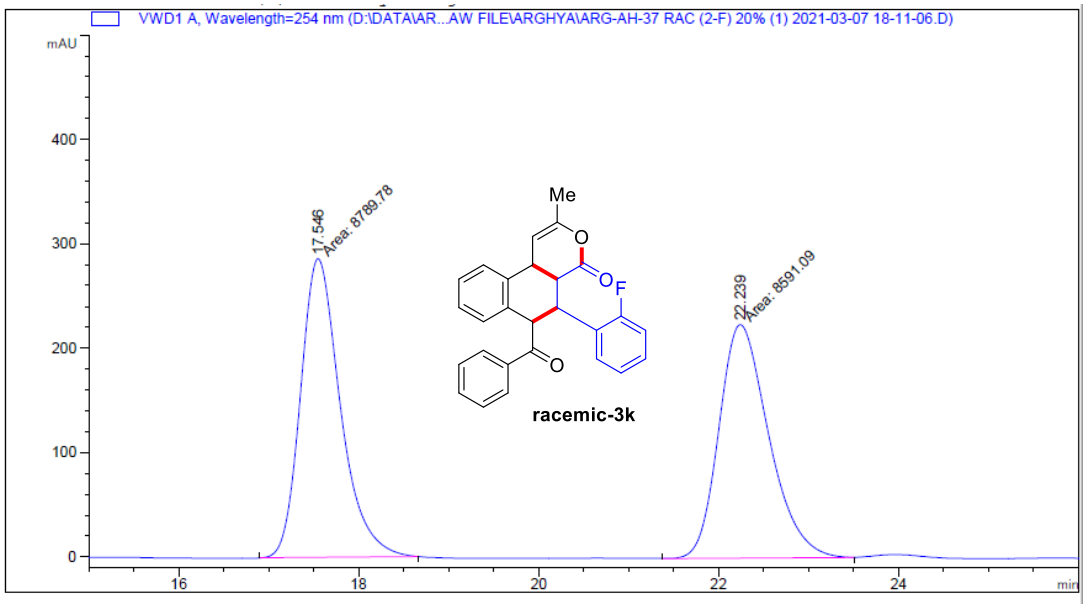
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.340	MM	0.9139	5020.79297	91.56218	49.8793
2	25.629	MM	1.2427	5045.09961	67.66344	50.1207



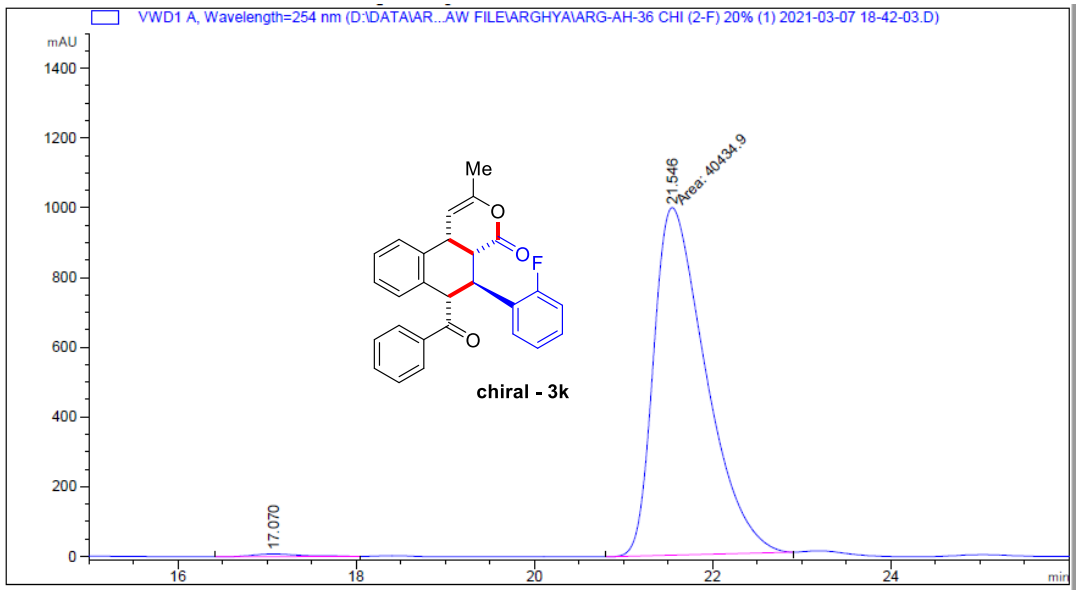
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.497	MM	0.9212	2.54942e4	461.24158	99.8984
2	25.936	MM	0.7956	25.92232	5.43040e-1	0.1016

Sample Info : CHIRALCEL-OD-H, 10% IPA-HEXANE, .7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(2-fluorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3k)



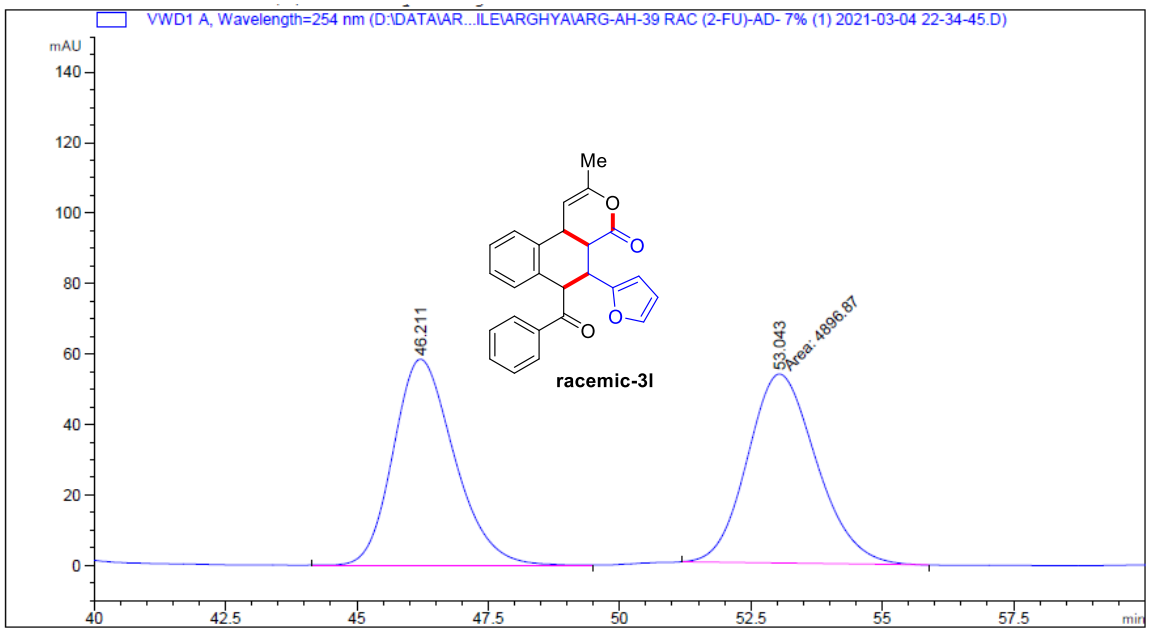
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.546	MM	0.5120	8789.78027	286.10803	50.5716
2	22.239	MM	0.6401	8591.09082	223.69762	49.4284



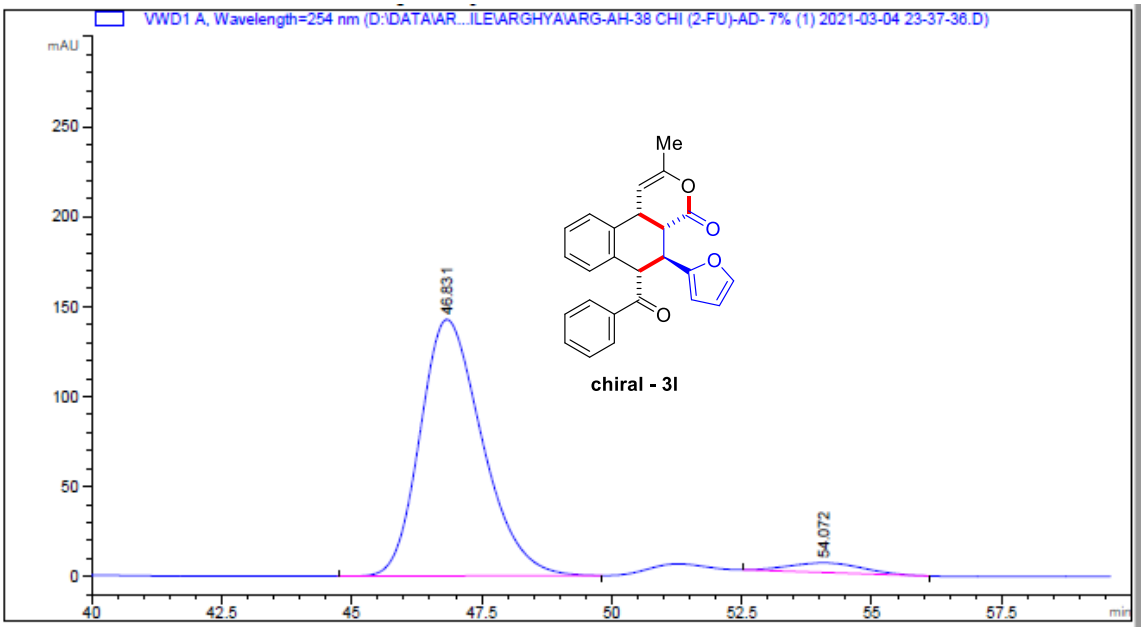
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.070	BB	0.5273	286.39078	7.98820	0.7033
2	21.546	MM	0.6762	4.04349e4	996.66321	99.2967

Sample Info : CHIRALPAK-AD, 20% IPA-HEXANE, .7 mL/min, 254 nm

(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(furan-2-yl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3l)



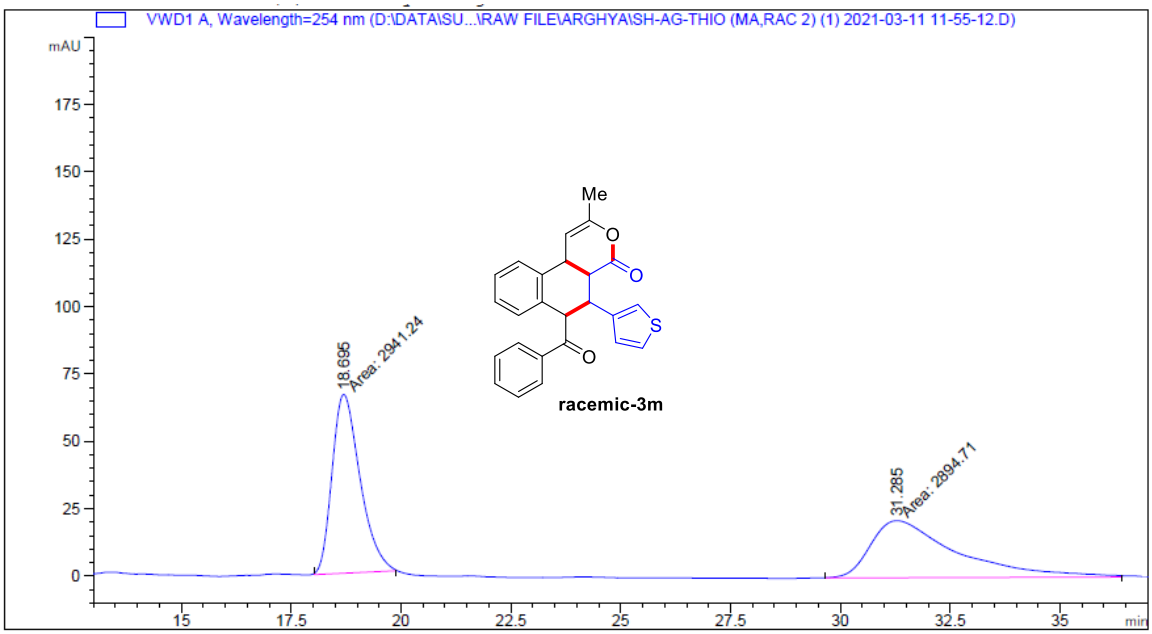
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	46.211	BB	1.2434	4723.75244	58.41530	49.1003
2	53.043	MM	1.5222	4896.86816	53.61612	50.8997



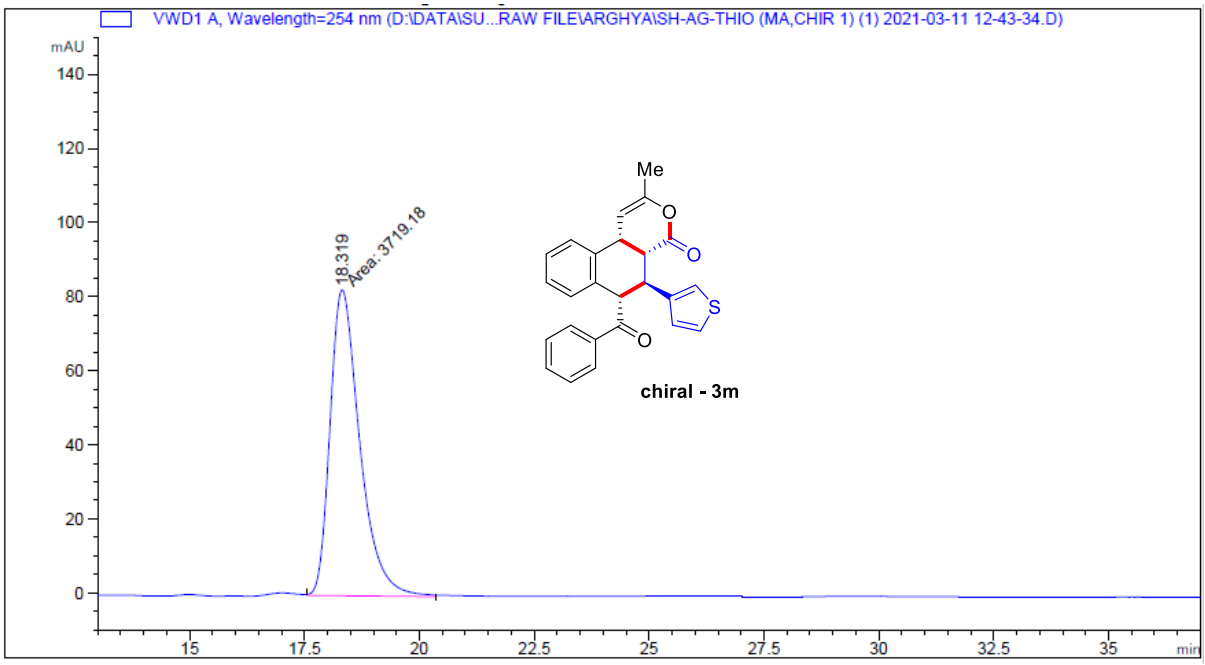
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	46.831	BB	1.2764	1.18338e4	142.41867	95.7405
2	54.072	BB	1.1826	526.48718	5.35247	4.2595

Sample Info : CHIRALPAK-AD, 7% IPA-HEXANE, .7 mL/min, 254 nm

(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(thiophen-3-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3m)



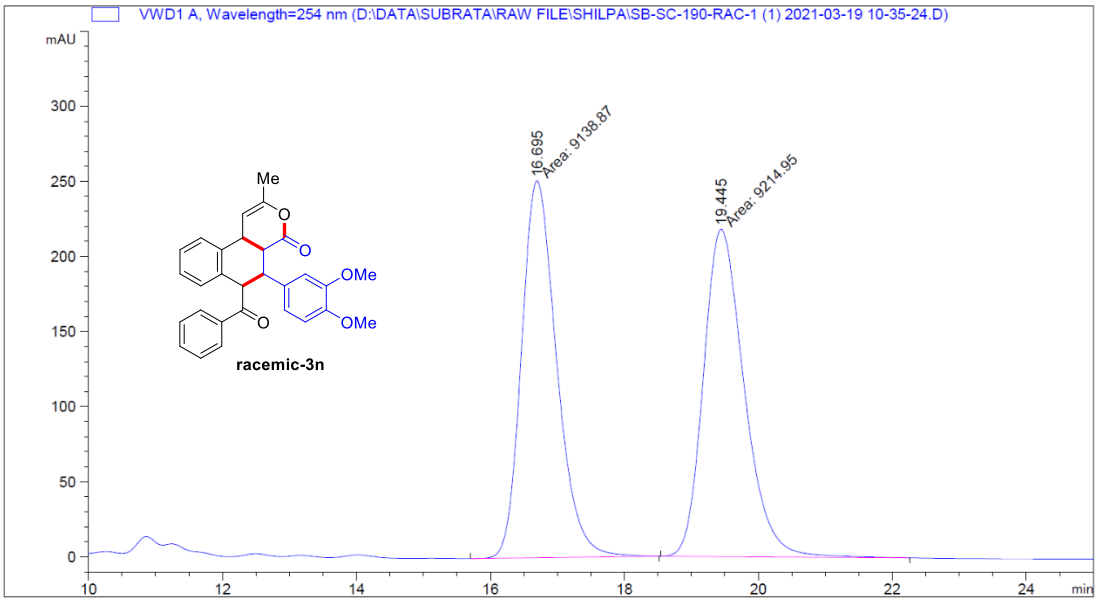
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.695	MM	0.7390	2941.24219	66.33076	50.3987
2	31.285	MM	2.2711	2894.70605	21.24267	49.6013



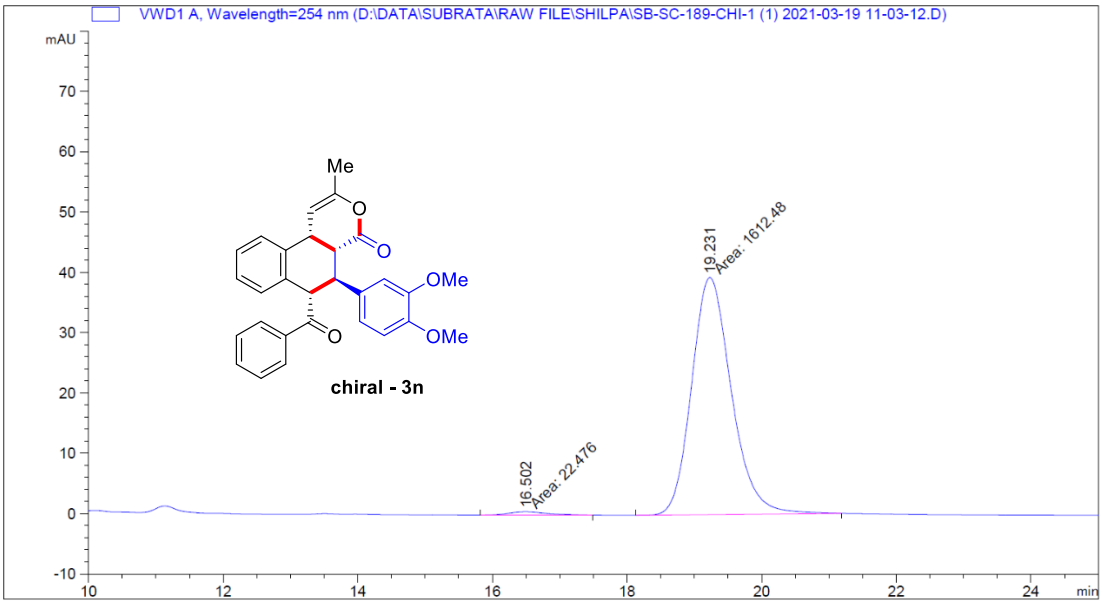
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.319	MM	0.7508	3719.17944	82.55823	100.0000

Sample Info : CHIRALCELL OD-H, 15% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3,4-dimethoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one(3n)



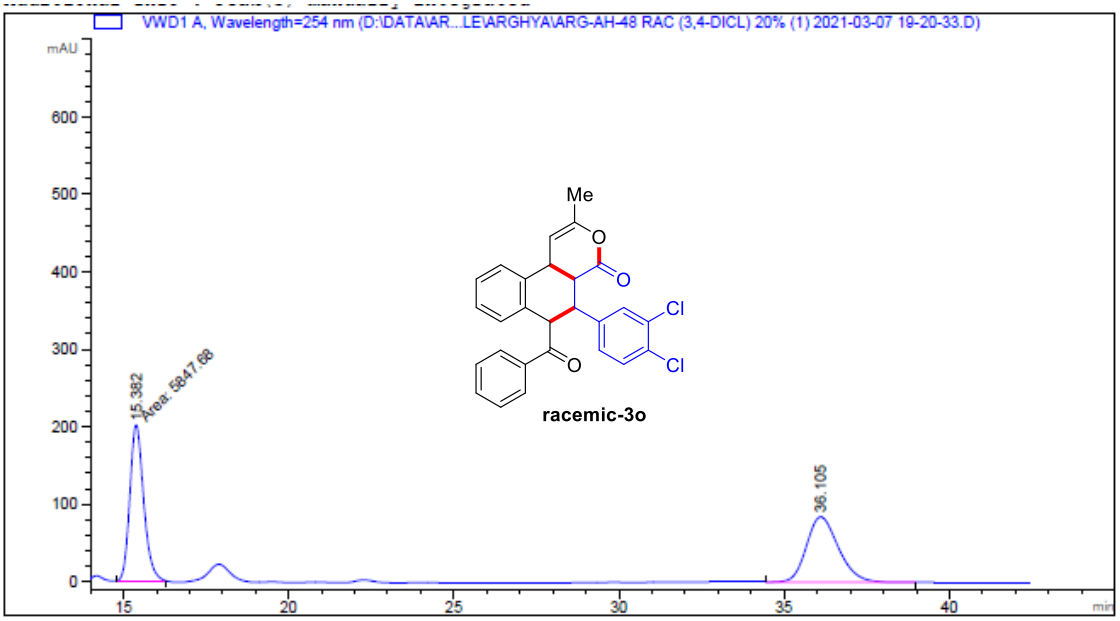
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.695	MM	0.6069	9138.87305	250.95570	49.7927
2	19.445	MM	0.7048	9214.95215	217.90891	50.2073



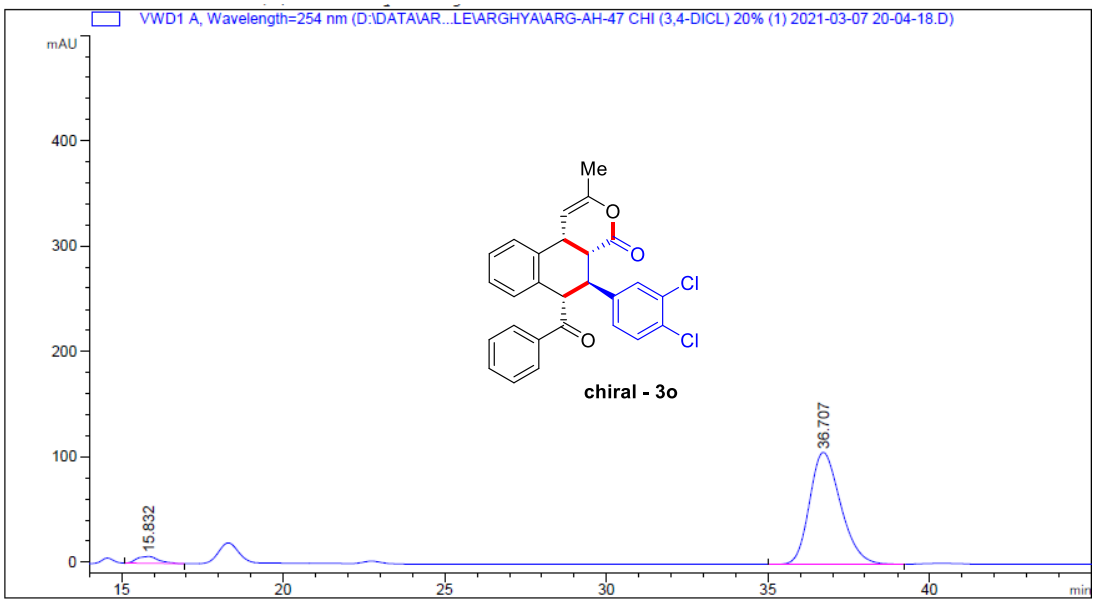
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.502	MM	0.6699	22.47602	5.59212e-1	1.3747
2	19.231	MM	0.6838	1612.47778	39.30384	98.6253

Sample Info : CHIRALPAK AD, 50 % IPA-HEXANE, 0.7mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(3,4-dichlorophenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3o)



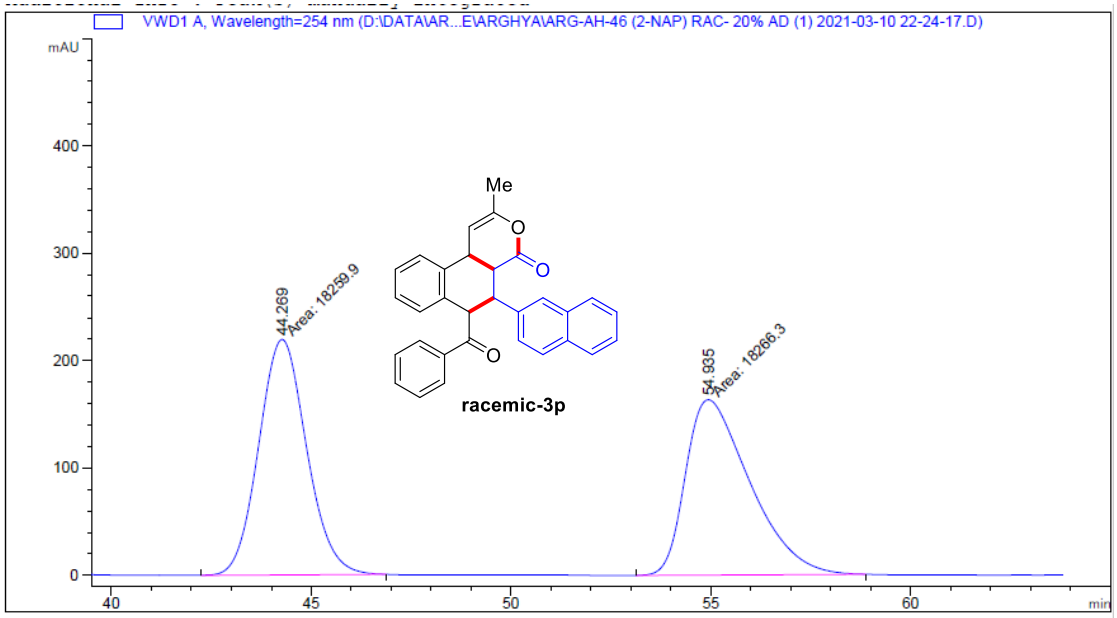
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.382	MM	0.4845	5847.67529	201.15695	50.8863
2	36.105	BB	1.0321	5643.97217	84.19189	49.1137



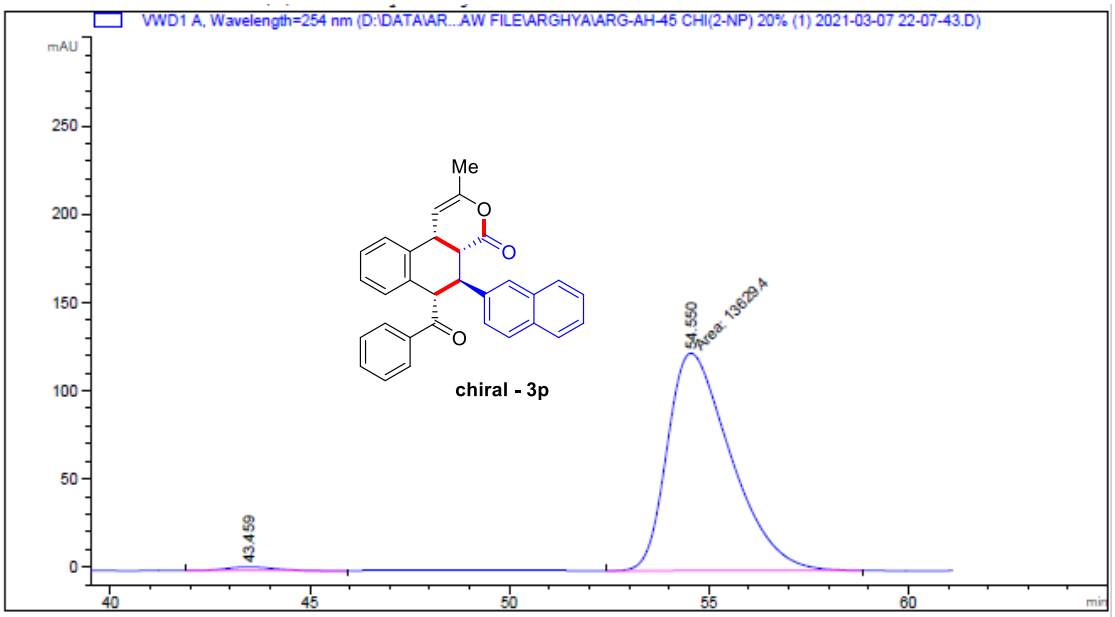
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.832	BB	0.6298	303.36142	6.58129	4.0918
2	36.707	BB	1.0332	7110.46240	106.19379	95.9082

Sample Info : CHIRALPAK-AD, 20% IPA-HEXANE, .7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(naphthalen-2-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3p)



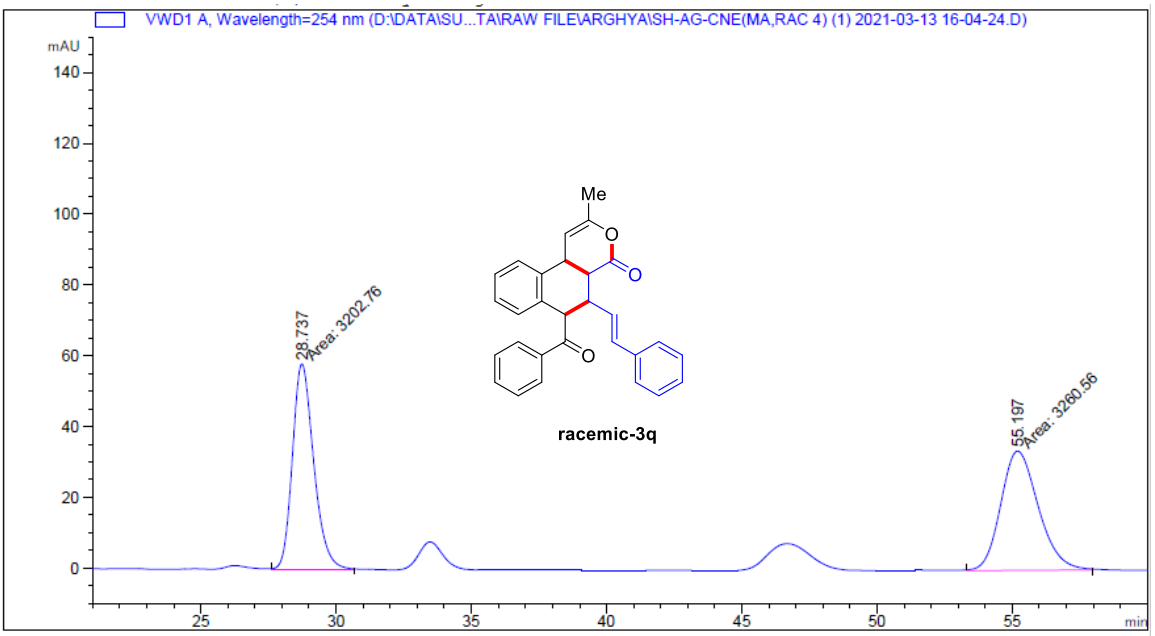
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	44.269	MM	1.3881	1.82599e4	219.24704	49.9913
2	54.935	MM	1.8639	1.82663e4	163.33215	50.0087



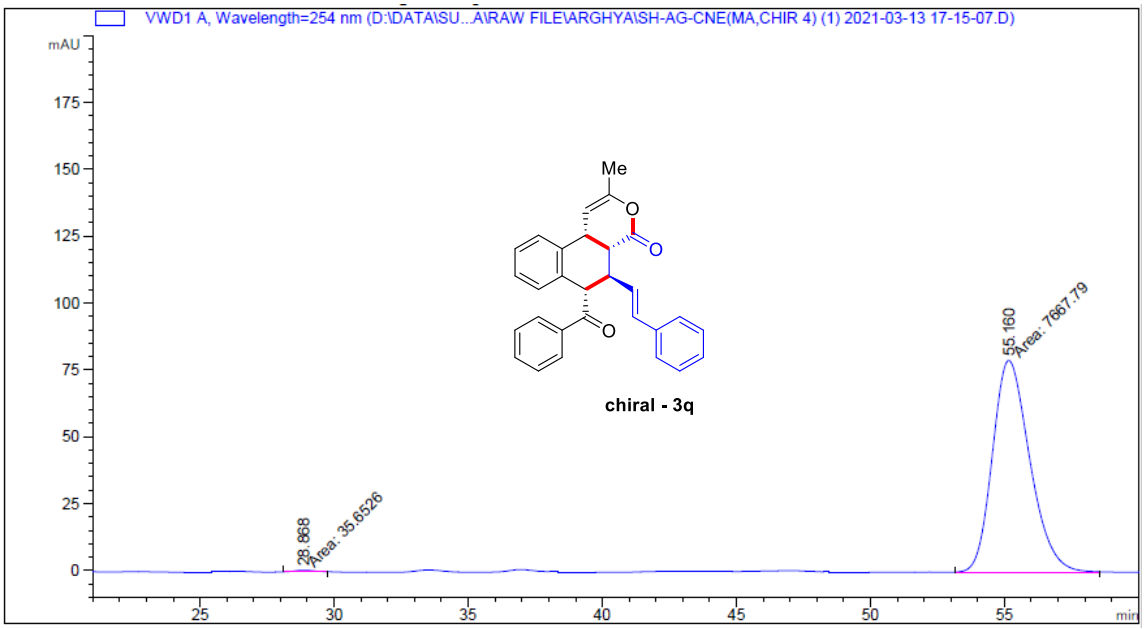
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	43.459	BB	1.0229	180.68040	2.08931	1.3083
2	54.550	MM	1.8452	1.36294e4	123.10655	98.6917

Sample Info : CHIRALPAK-AD, 20% IPA-HEXANE, .7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-((*E*)-styryl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3q)



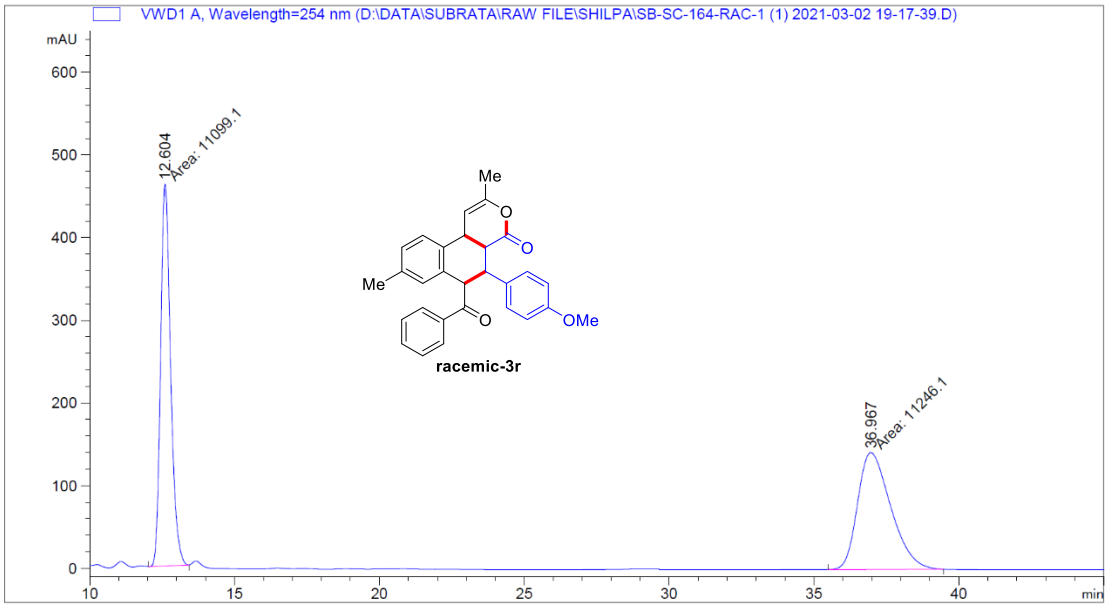
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.737	MM	0.9179	3202.76147	58.15466	49.5529
2	55.197	MM	1.6136	3260.55518	33.67784	50.4471



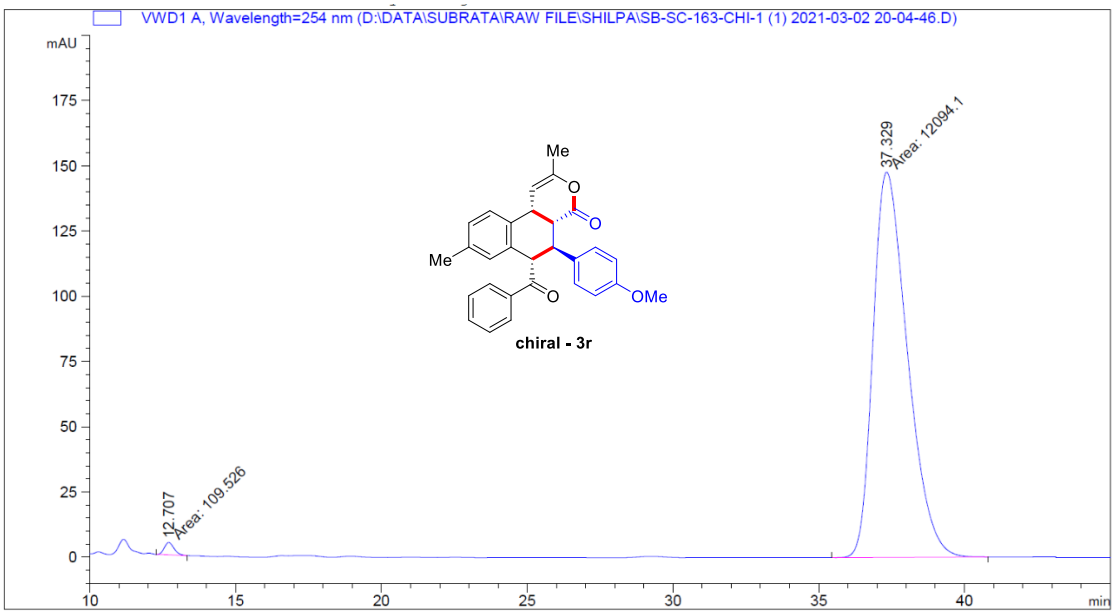
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.868	MM	0.9101	35.65258	6.52930e-1	0.4628
2	55.160	MM	1.6144	7667.79248	79.16170	99.5372

Sample Info : CHIRAl PAK AD,7% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2,8-dimethyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3r)



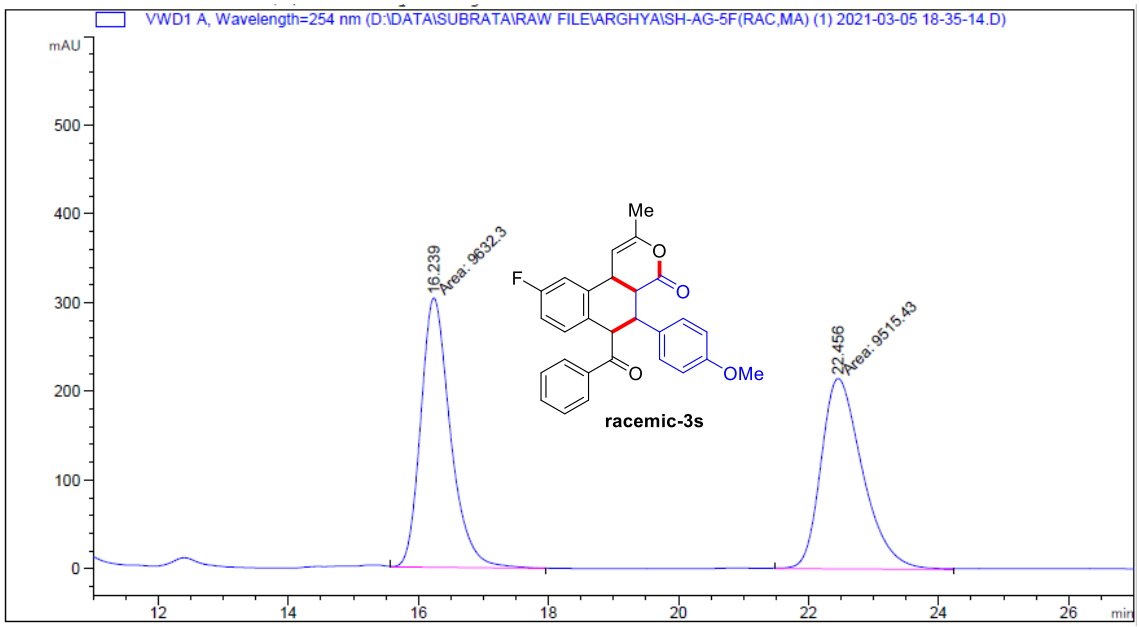
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.604	MM	0.4003	1.10991e4	462.08380	49.6712
2	36.967	MM	1.3266	1.12461e4	141.29347	50.3288



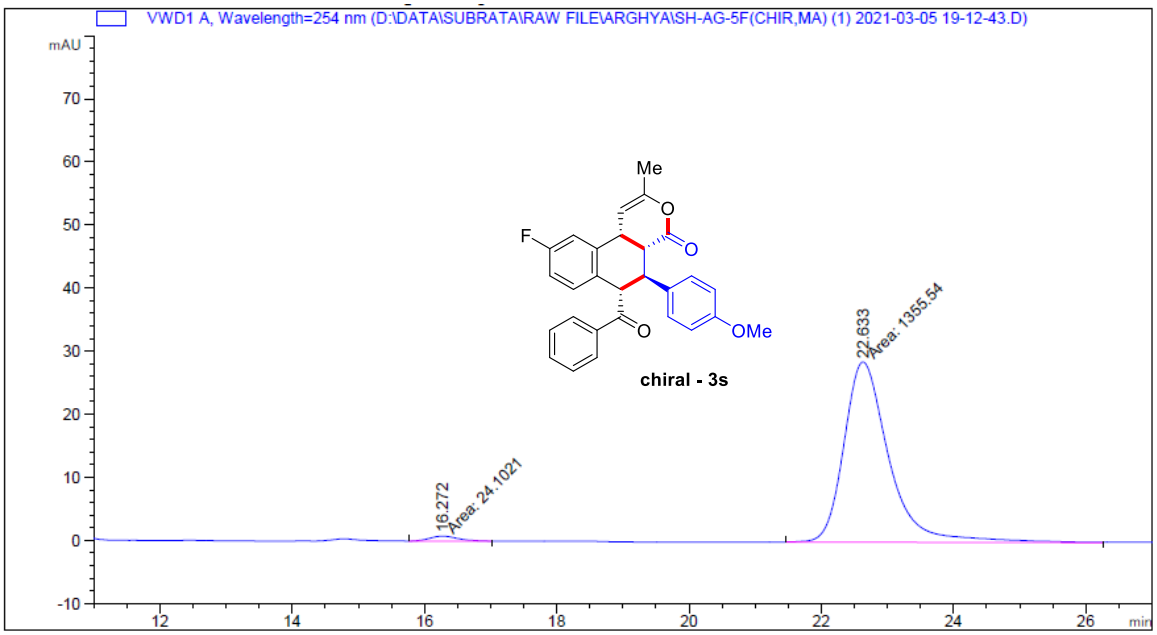
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.707	MM	0.3874	109.52612	4.71262	0.8975
2	37.329	MM	1.3651	1.20941e4	147.66252	99.1025

Sample Info : CHIRALPAK-AD, 40% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-9-fluoro-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3s)



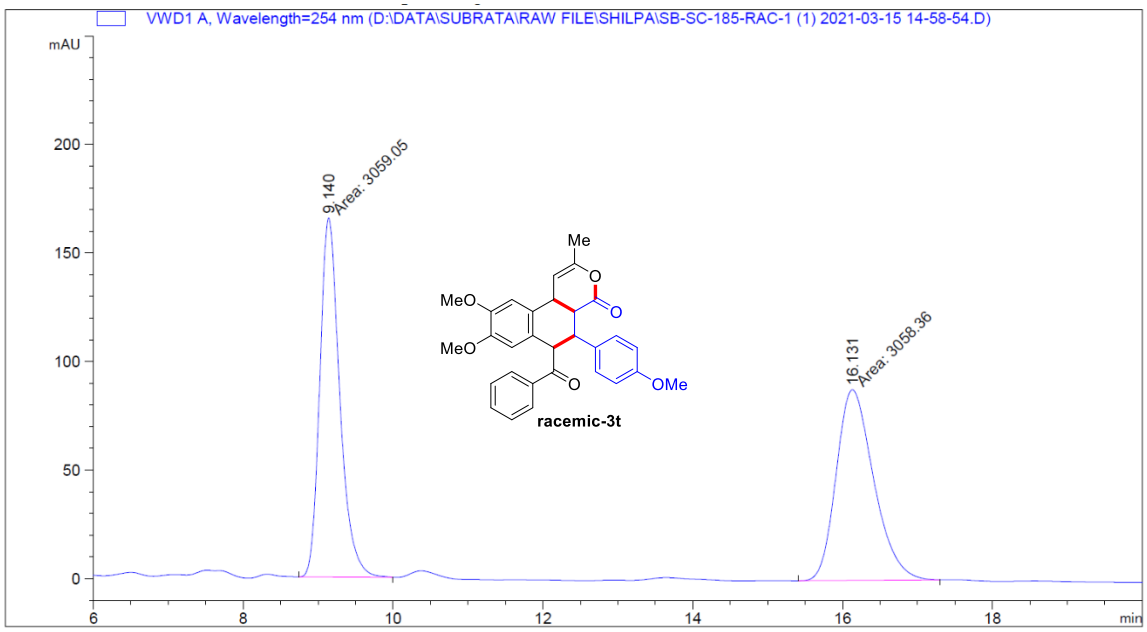
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.239	MM	0.5287	9632.29883	303.67035	50.3052
2	22.456	MM	0.7390	9515.43359	214.60608	49.6948



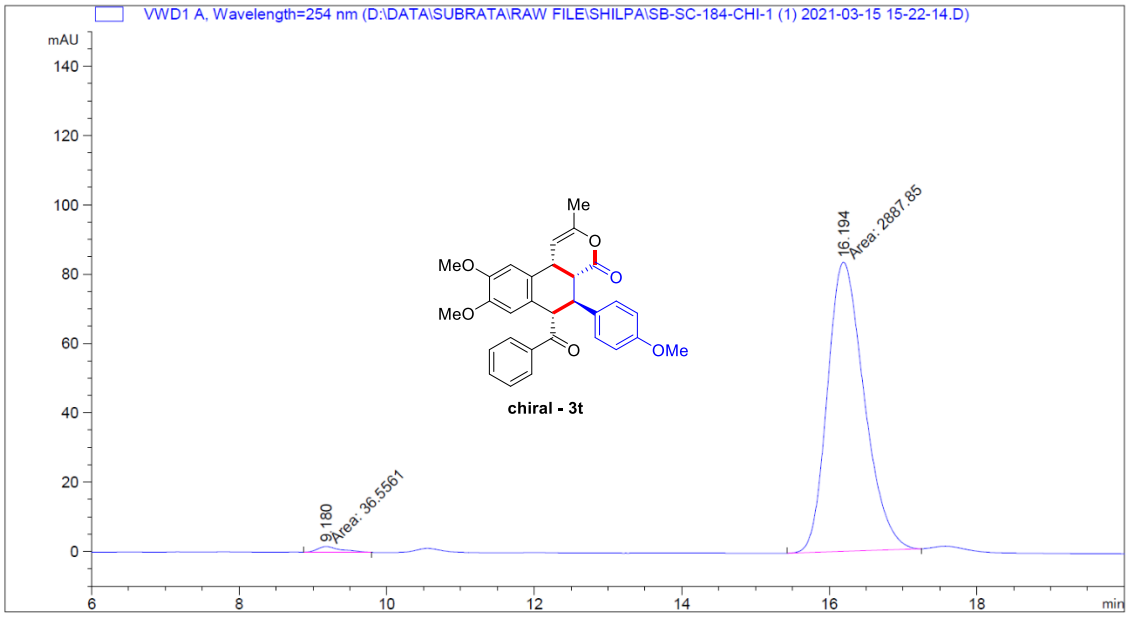
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.272	MM	0.5022	24.10212	7.99881e-1	1.7470
2	22.633	MM	0.7909	1355.54016	28.56595	98.2530

Sample Info : CHIRALPAK-AD, 30% IPA-HEXANE, .7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-8,9-dimethoxy-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one(3t)



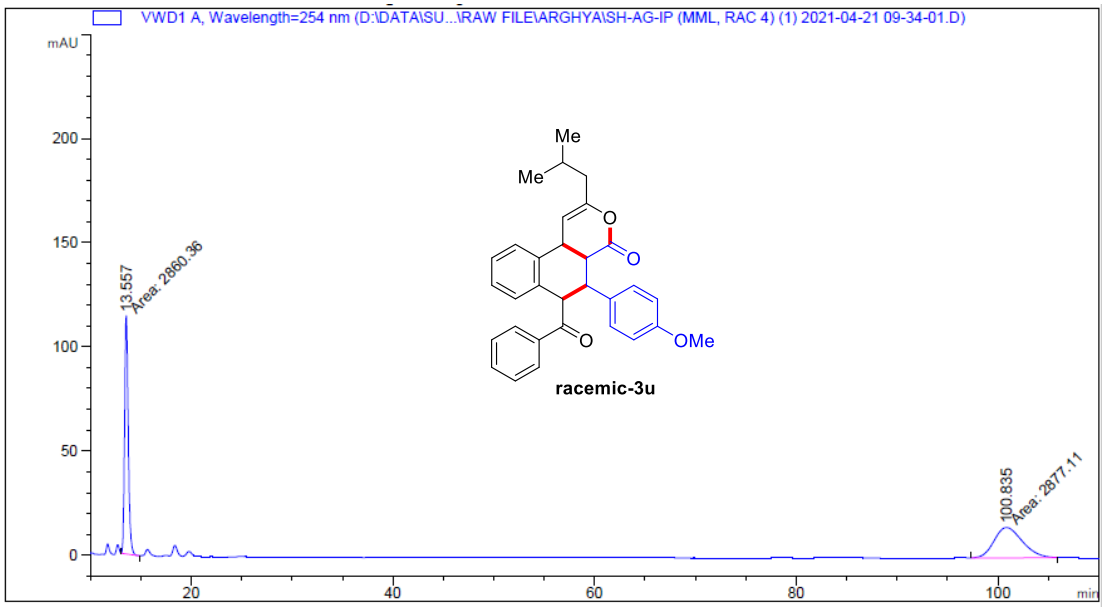
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.140	MM	0.3081	3059.04810	165.49138	50.0056
2	16.131	MM	0.5803	3058.35742	87.84579	49.9944



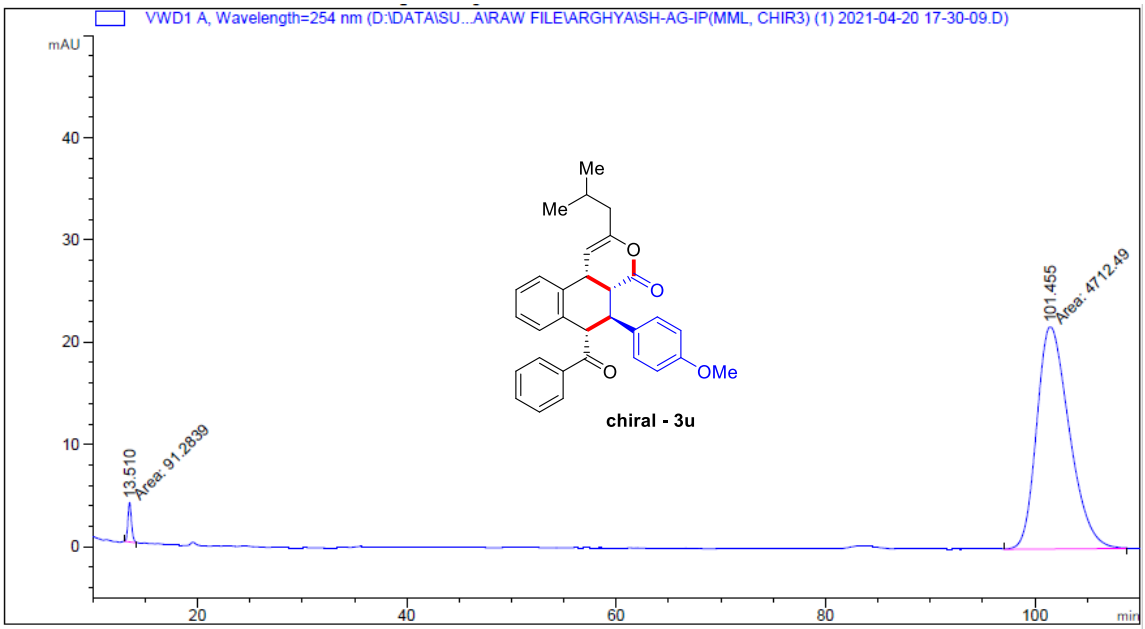
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.180	MM	0.3686	36.55605	1.65312	1.2500
2	16.194	MM	0.5771	2887.84619	83.39981	98.7500

Sample Info : CHIRAl PAK AD,50% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-isobutyl-5-(4-methoxyphenyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3u)



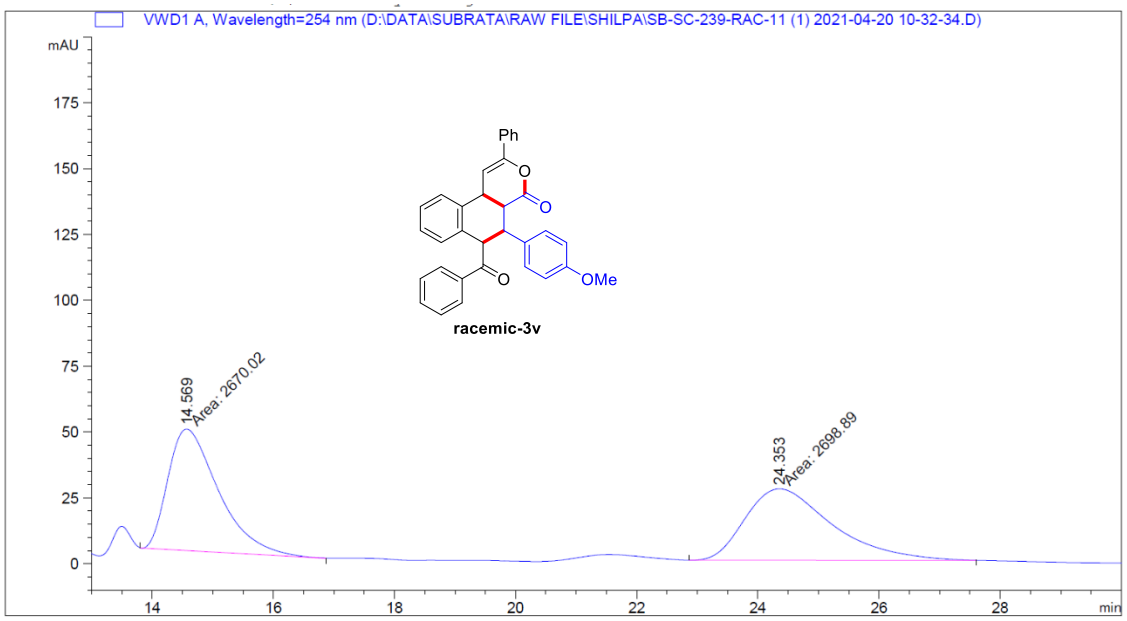
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.557	MM	0.4170	2860.35522	114.32616	49.8540
2	100.835	MM	3.2767	2877.11230	14.63398	50.1460



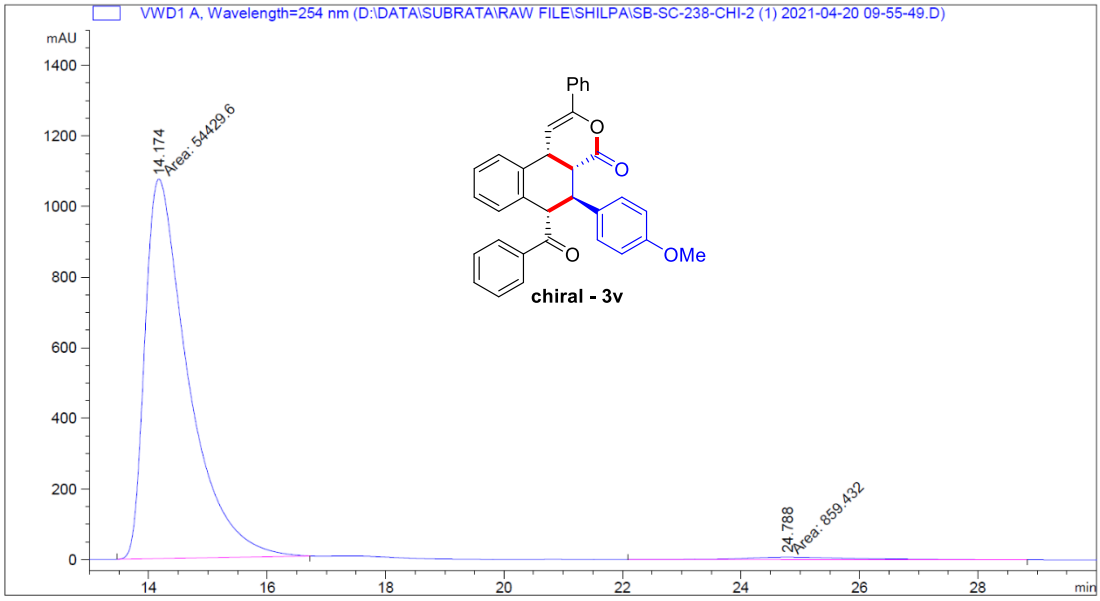
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.510	MM	0.3948	91.28386	3.85373	1.9003
2	101.455	MM	3.6141	4712.48877	21.73170	98.0997

Sample Info : CHIRALPAK AD, 20 % IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one(3v)



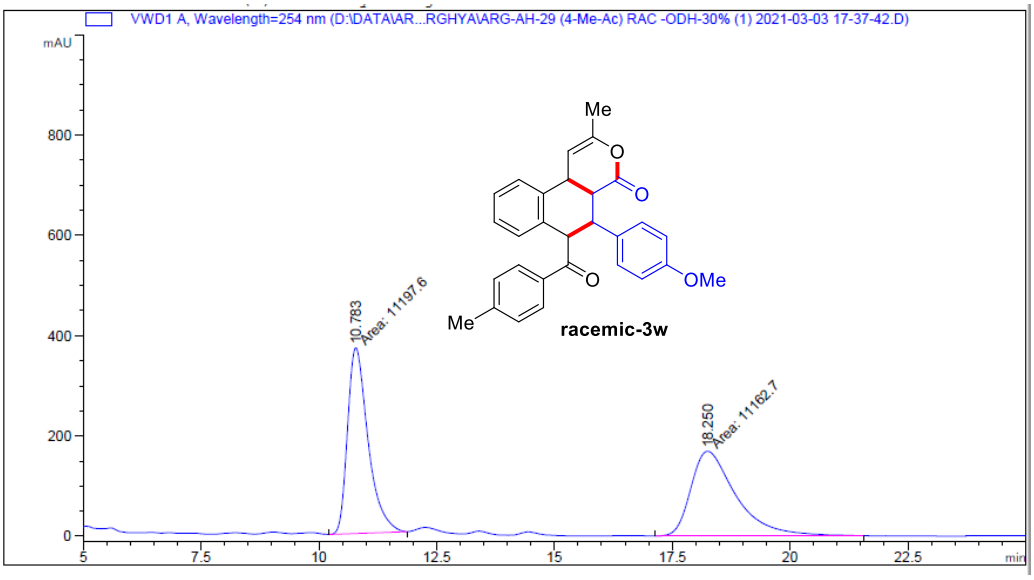
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.569	MM	0.9648	2670.01758	46.12535	49.7311
2	24.353	MM	1.6601	2698.88794	27.09529	50.2689



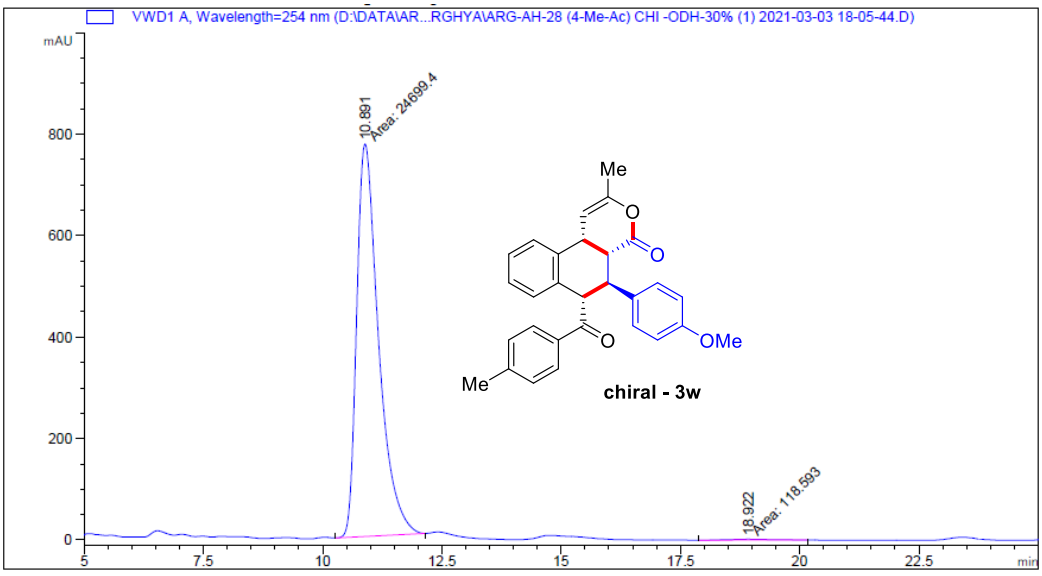
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.174	MM	0.8440	5.44296e4	1074.85742	98.4456
2	24.788	MM	2.2115	859.43213	6.47697	1.5544

Sample Info : CHIRALCEL-OD-H, 30% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-5-(4-Methoxyphenyl)-2-methyl-6-(4-methylbenzoyl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3w)



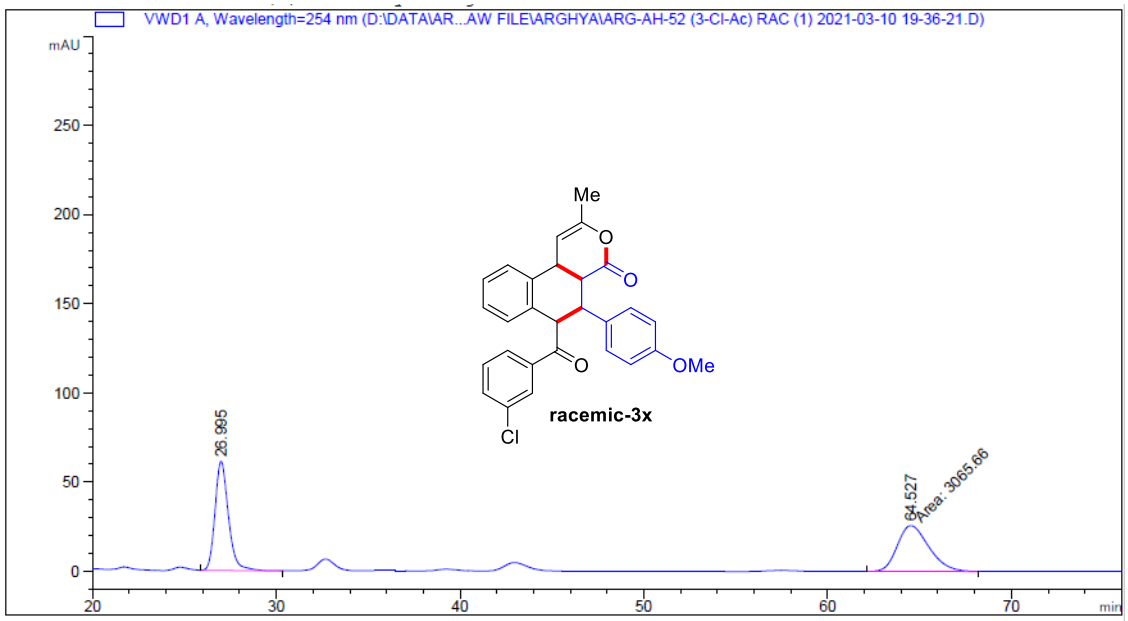
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.783	MM	0.5042	1.11976e4	370.11960	50.0779
2	18.250	MM	1.1039	1.11627e4	168.54013	49.9221



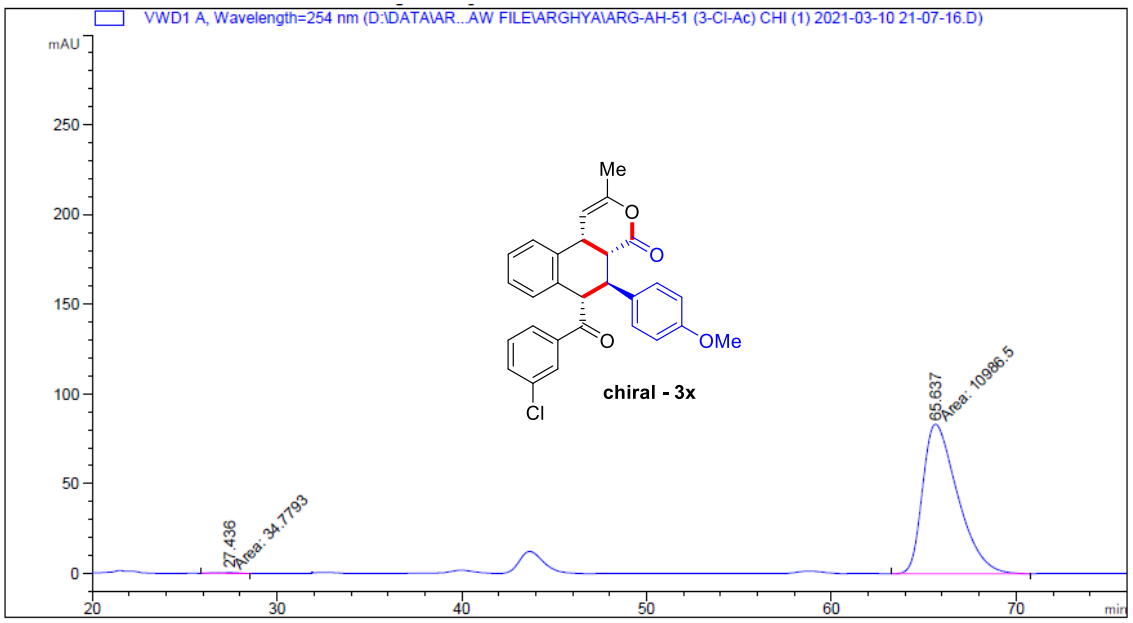
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.891	MM	0.5320	2.46994e4	773.75909	99.5221
2	18.922	MM	1.1131	118.59294	1.77579	0.4779

Sample Info : CHIRALCEL-ODH, 30% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-(3-Chlorobenzoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3x)



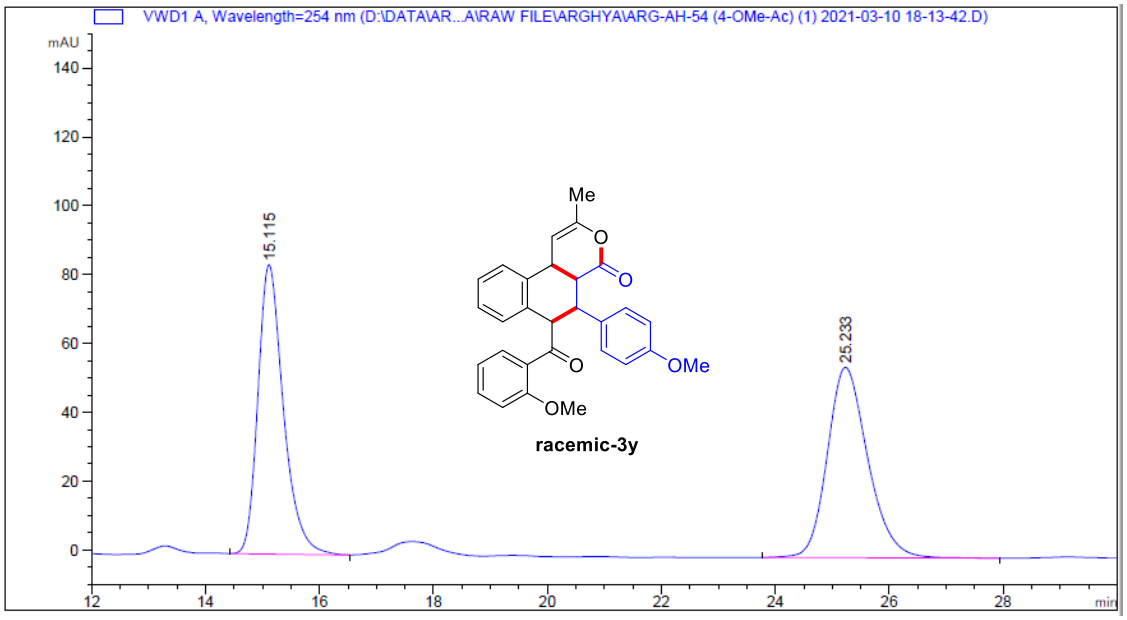
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.995	BB	0.7680	3090.35327	61.21006	50.2005
2	64.527	MM	1.9889	3065.66235	25.69009	49.7995



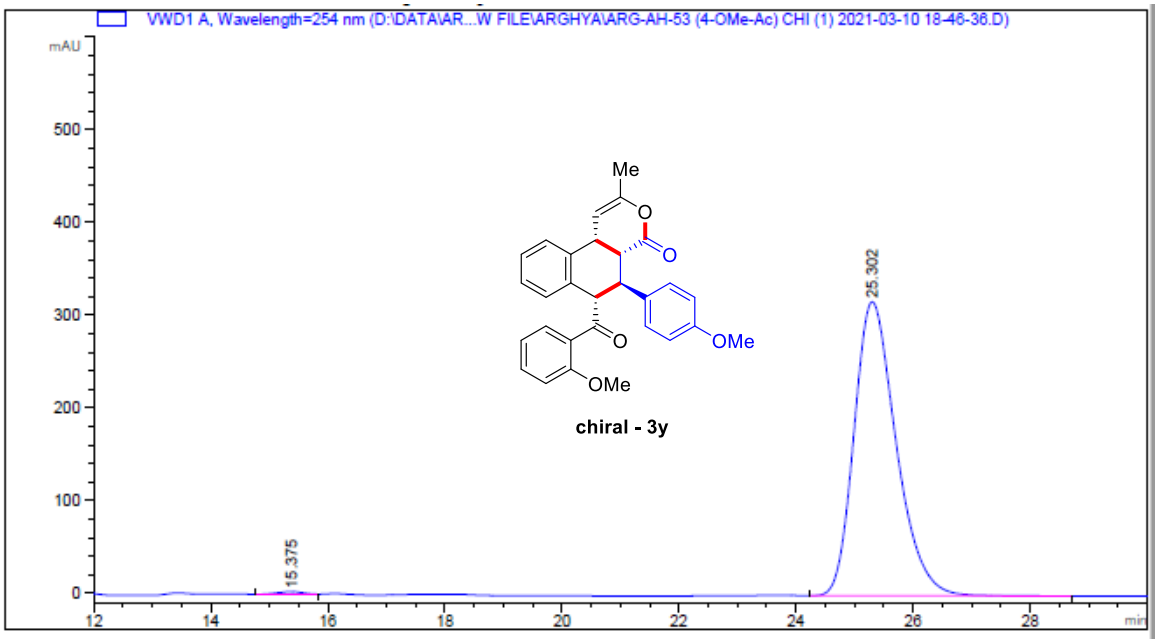
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.436	MM	1.1294	34.77931	5.13250e-1	0.3156
2	65.637	MM	2.1976	1.09865e4	83.32234	99.6844

Sample Info : CHIRALPAK AD, 20% IPA-HEXANE, .7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-(2-Methoxybenzoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3y)



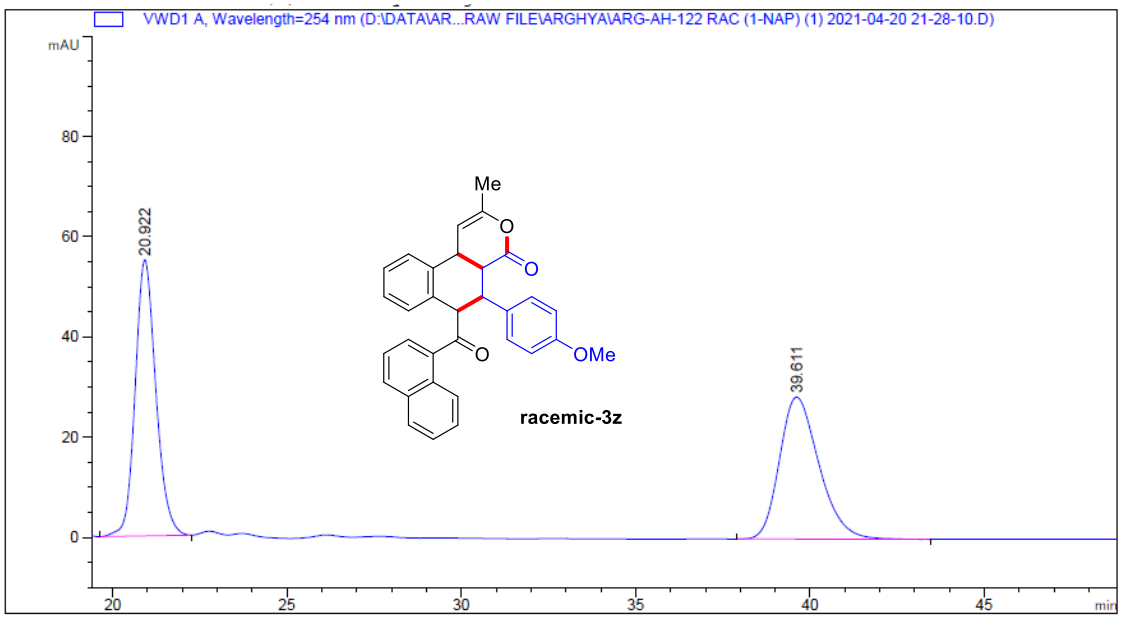
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.115	BB	0.4694	2610.16211	84.03398	48.9814
2	25.233	BB	0.7503	2718.71973	55.23158	51.0186



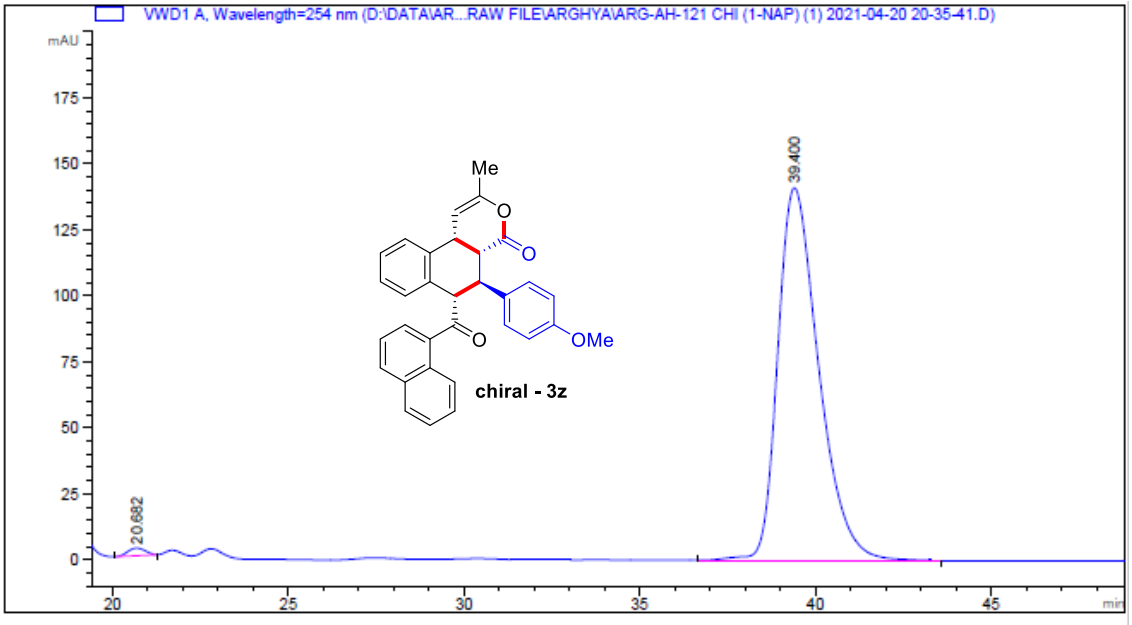
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.375	BB	0.4886	81.41607	2.63374	0.5128
2	25.302	BB	0.7635	1.57960e4	316.94006	99.4872

Sample Info : CHIRALPAK AD, 30% IPA-HEXANE, .7 mL/min, 254 nm

(4a*R*,5*S*,6*S*,10b*R*)-6-(1-Naphthoyl)-5-(4-methoxyphenyl)-2-methyl-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3z)



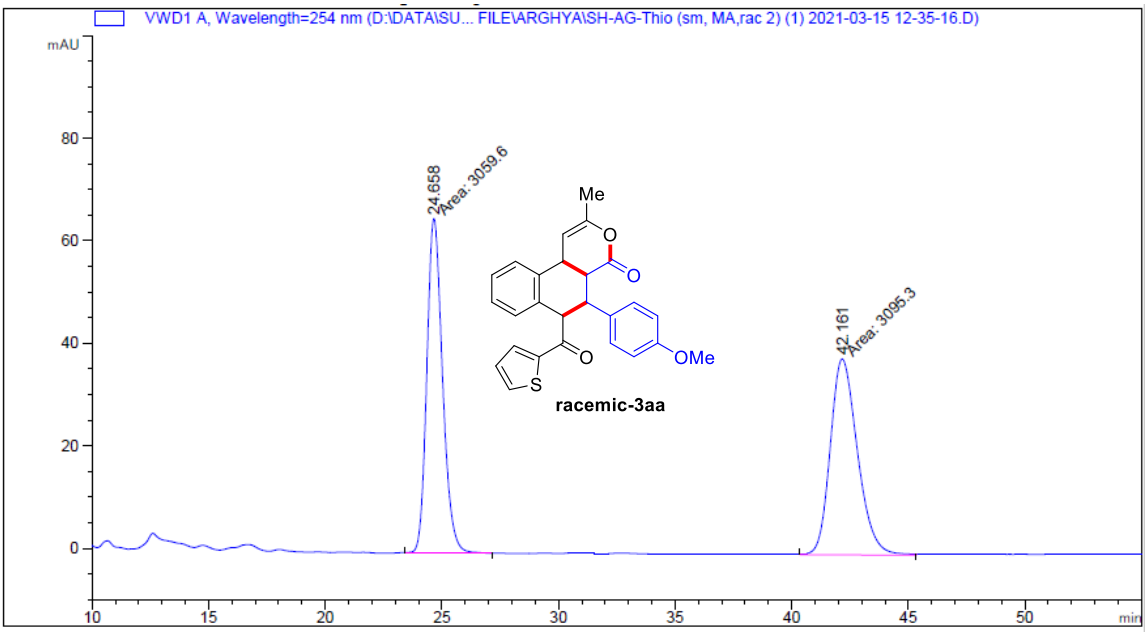
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.922	BB	0.6310	2275.20898	55.07272	50.4655
2	39.611	BB	1.1979	2233.23193	28.32182	49.5345



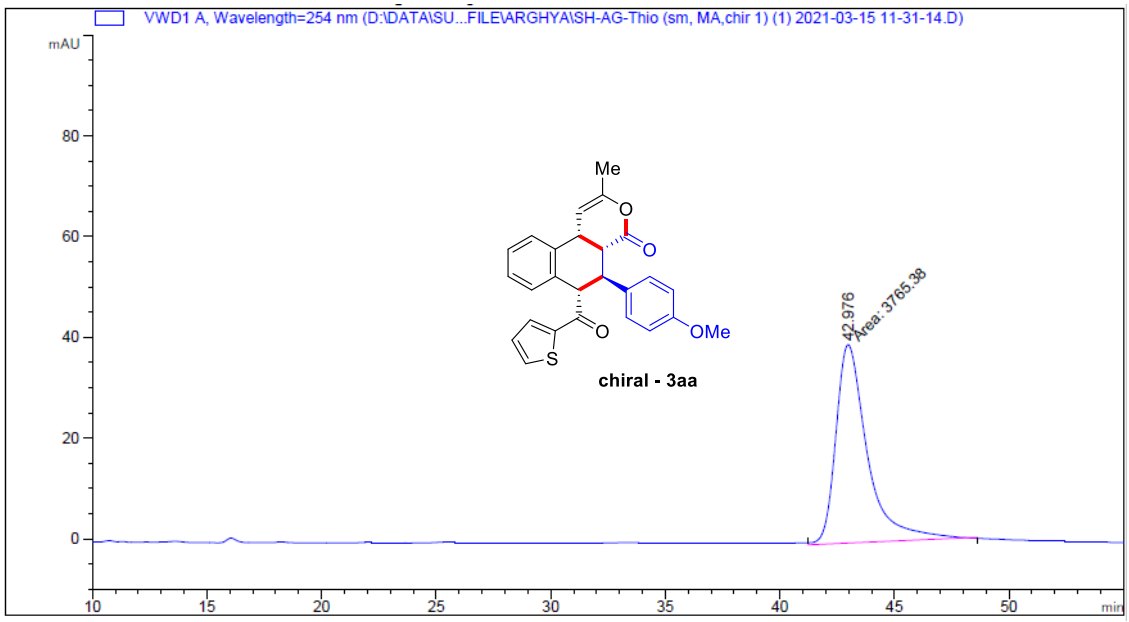
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.682	BB	0.5456	99.97591	2.89019	0.8713
2	39.400	BB	1.2365	1.13748e4	140.94965	99.1287

Sample Info : CHIRALPAK AD, 25% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*S*,5*S*,6*S*,10b*R*)-6-Benzoyl-2-methyl-5-(thiophen-2-yl)-4a,5,6,10b-tetrahydro-4*H*-benzo[*f*]isochromen-4-one (3aa)



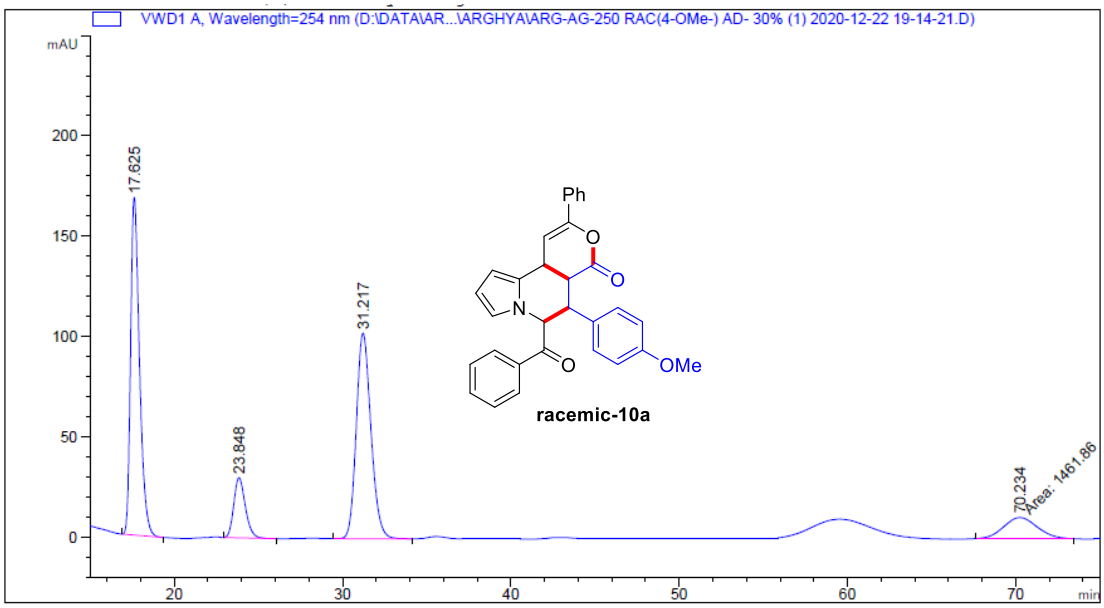
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.658	MM	0.7815	3059.60425	65.25004	49.7100
2	42.161	MM	1.3512	3095.30103	38.17882	50.2900



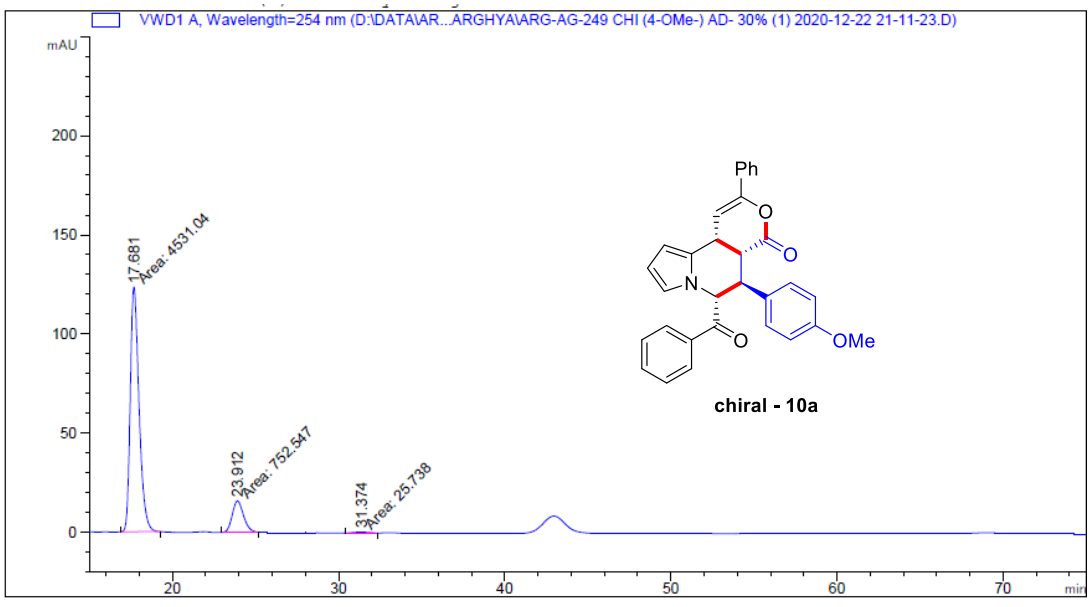
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	42.976	MM	1.5951	3765.38306	39.34288	100.0000

Sample Info : CHIRAl PAK AD,30% IPA-HEXANE, 0.7 mL/min, 254 nm

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10a)



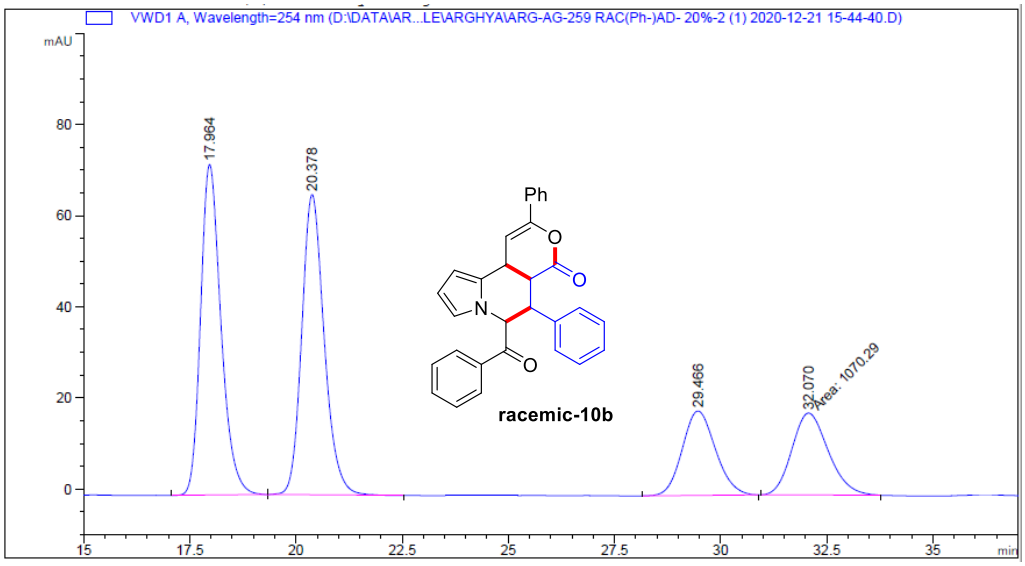
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.625	BB	0.5571	6163.84863	168.35533	40.4143
2	23.848	BB	0.7303	1430.62756	30.00208	9.3802
3	31.217	BB	0.9288	6195.30469	102.41341	40.6206
4	70.234	MM	2.3156	1461.86133	10.52194	9.5849



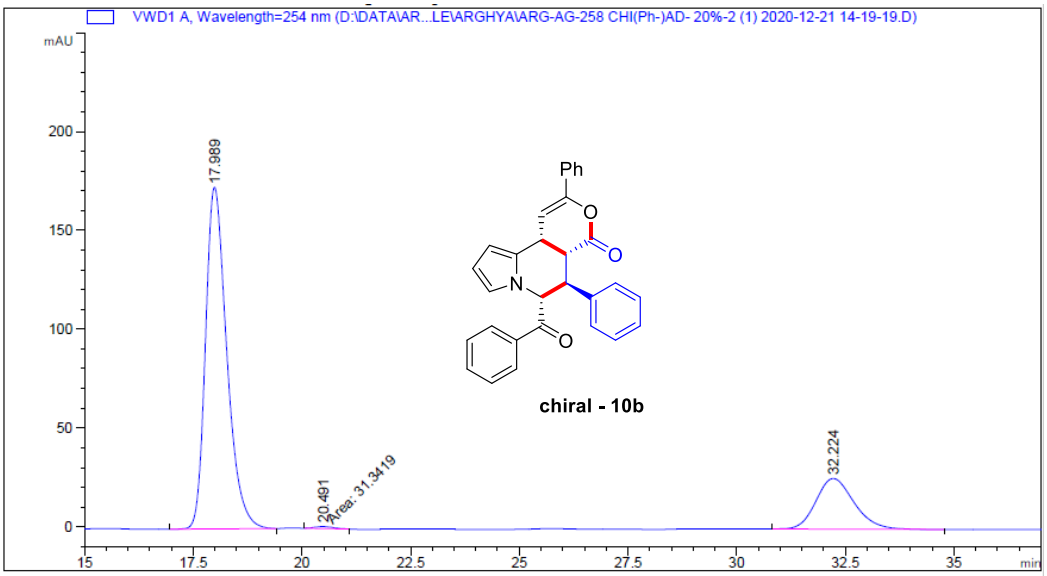
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.681	MM	0.6114	4531.03564	123.51097	85.3412
2	23.912	MM	0.7912	752.54694	15.85250	14.1741
3	31.374	MM	0.8940	25.73804	4.79828e-1	0.4848

Sample Info : CHIRALPAK AD , 30% IPA-HEXANE, .7 mL/min.

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-2,5-diphenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g] indolizin-4-one (10b)



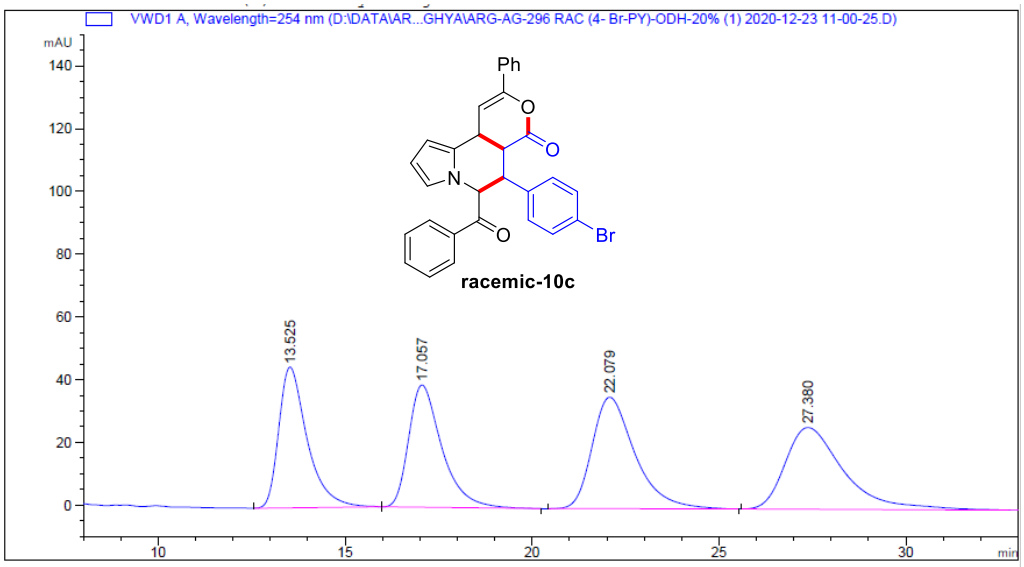
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.964	BB	0.5103	2419.71753	72.52306	34.9907
2	20.378	BB	0.5612	2418.16943	65.88641	34.9683
3	29.466	BB	0.8369	1007.15167	18.50794	14.5641
4	32.070	MM	0.9930	1070.28638	17.96461	15.4770



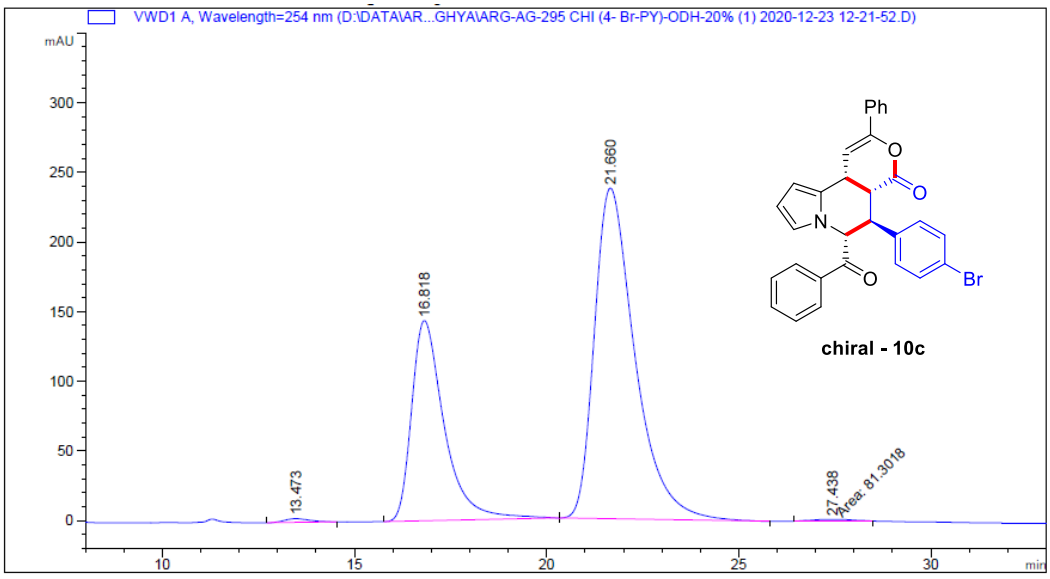
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.989	BB	0.5200	5882.62451	172.88060	78.7049
2	20.491	MM	0.5088	31.34187	1.02664	0.4193
3	32.224	BB	0.9311	1560.31506	25.63548	20.8758

Sample Info : CHIRALPAK AD , 20% IPA-HEXANE, .7 mL/min.

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-5-(4-bromophenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10c)



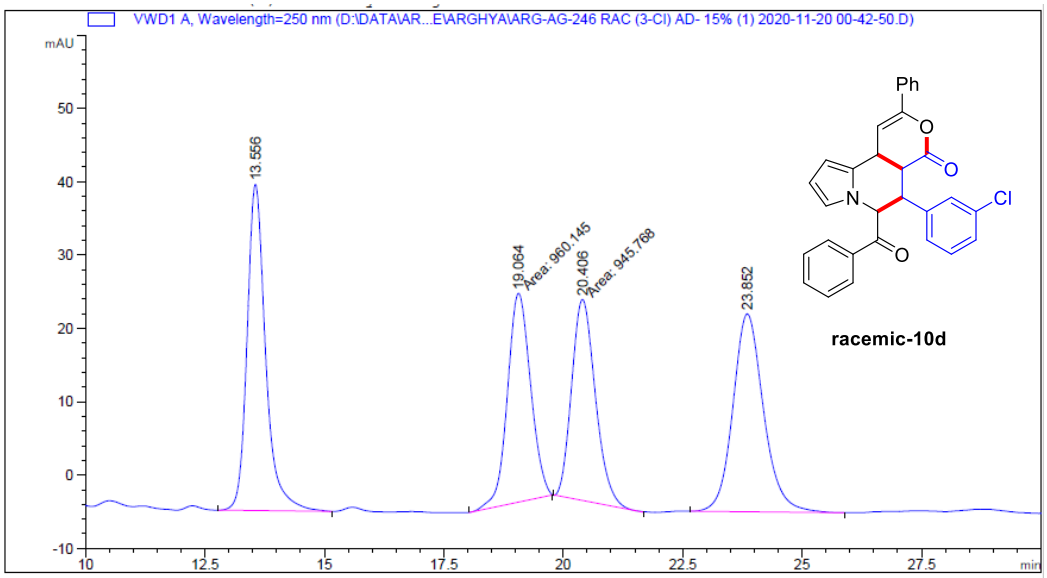
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.525	BB	0.7926	2373.79175	44.83872	22.7230
2	17.057	BB	0.9266	2406.54590	38.96359	23.0365
3	22.079	BB	1.2051	2830.43701	35.50526	27.0942
4	27.380	BB	1.5797	2835.87646	26.05798	27.1463



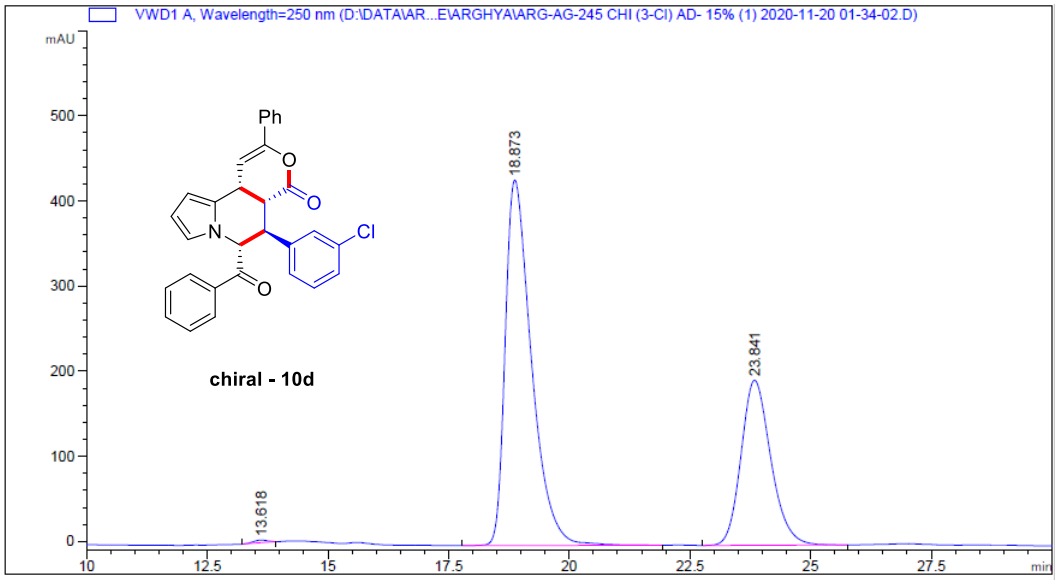
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.473	BB	0.6989	121.08150	2.59228	0.4644
2	16.818	BB	0.8870	8485.18066	143.67177	32.5422
3	21.660	BB	1.1057	1.73869e4	237.32100	66.6817
4	27.438	MM	1.1796	81.30183	1.14867	0.3118

Sample Info : CHIRALCEL ODH , 20% IPA-HEXANE, .7 mL/min.

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-5-(3-chlorophenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10d)



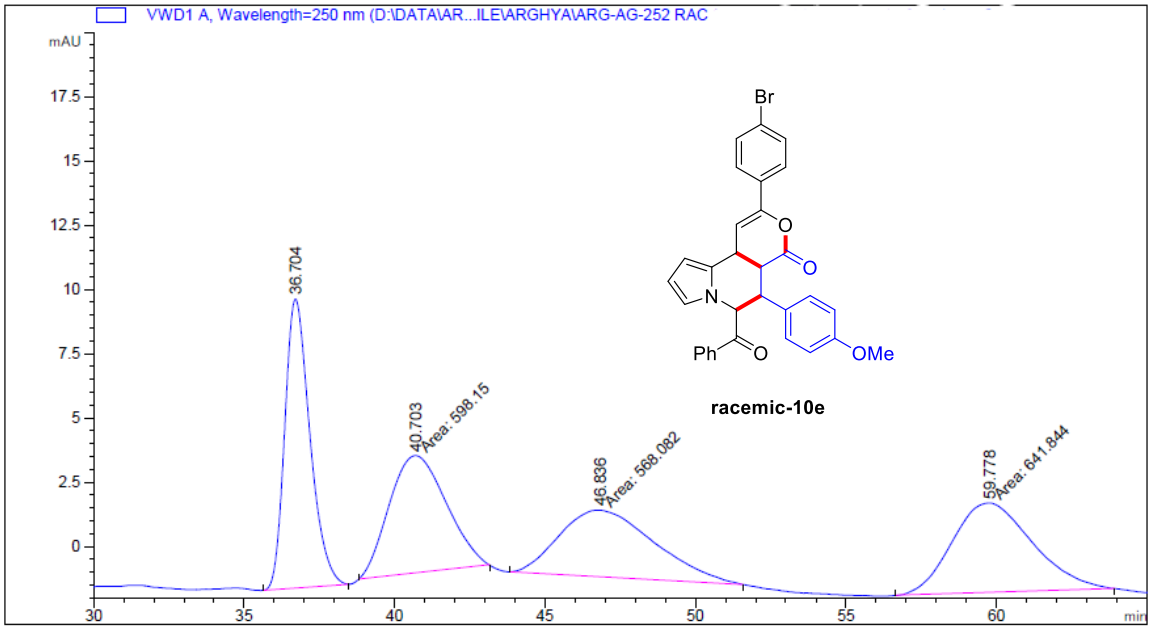
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.556	BB	0.4078	1203.81201	44.46981	27.8995
2	19.064	MM	0.5620	960.14526	28.47199	22.2523
3	20.406	MM	0.5752	945.76758	27.40399	21.9191
4	23.852	BB	0.6791	1205.08618	26.98148	27.9291



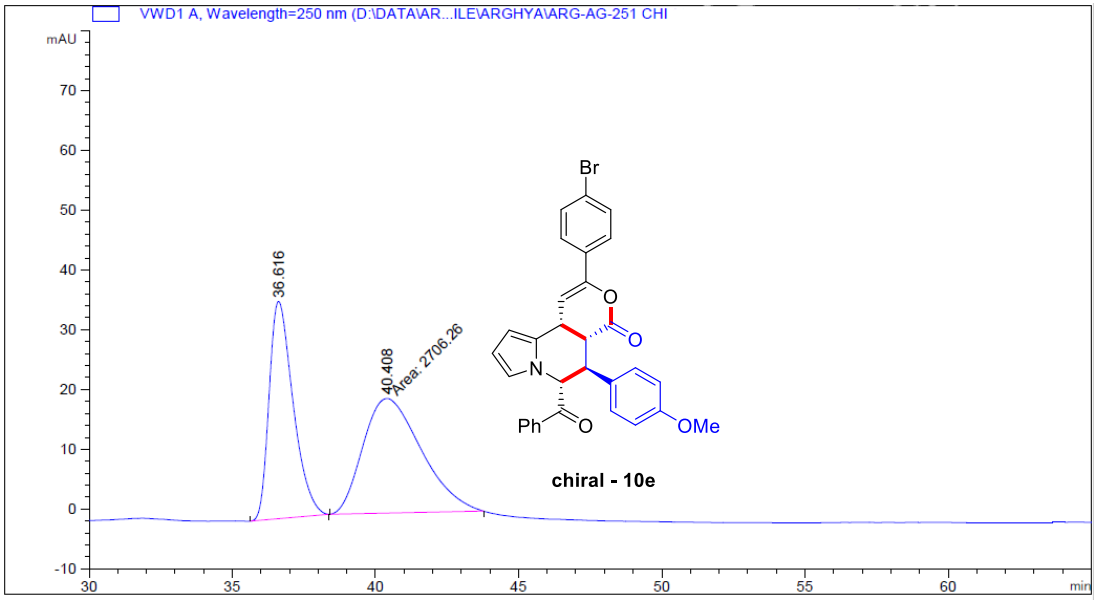
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.618	BB	0.3174	61.38850	3.05608	0.2499
2	18.873	BB	0.5712	1.61976e4	429.33289	65.9266
3	23.841	BB	0.6583	8310.12793	193.80508	33.8235

Sample Info : CHIRALPAK- AD, 15 % IPA/HEXANE, .7 mL -min, 254 nM

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-2-(4-bromophenyl)-5-(4-methoxyphenyl)-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10e)



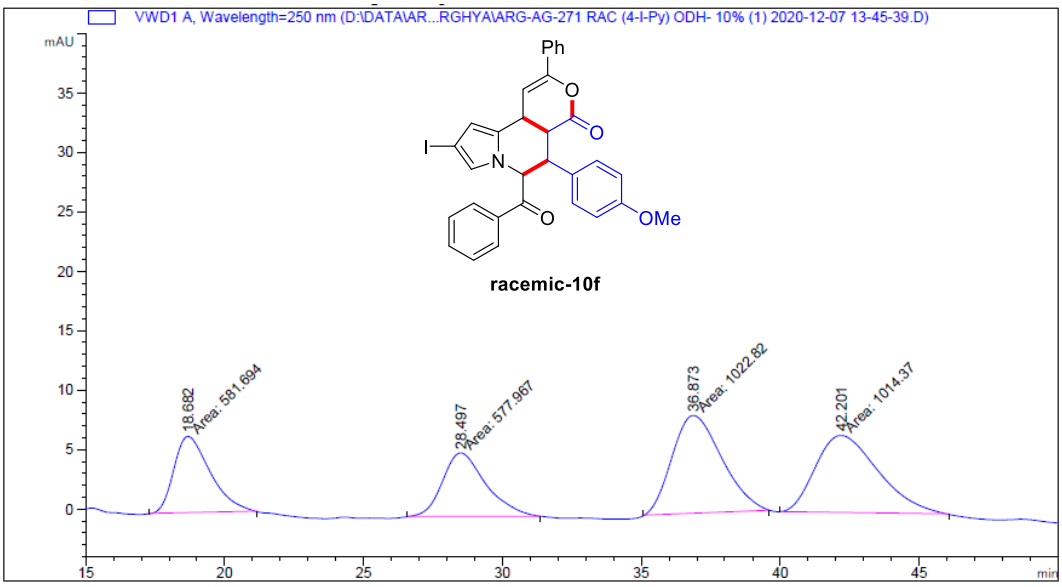
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	36.704	BB	0.8821	652.05682	11.23482	26.5049
2	40.703	MM	1.5726	598.15027	4.55471	24.3137
3	46.836	MM	3.6536	568.08191	2.59142	23.0915
4	59.778	MM	3.0794	641.84424	3.47381	26.0898



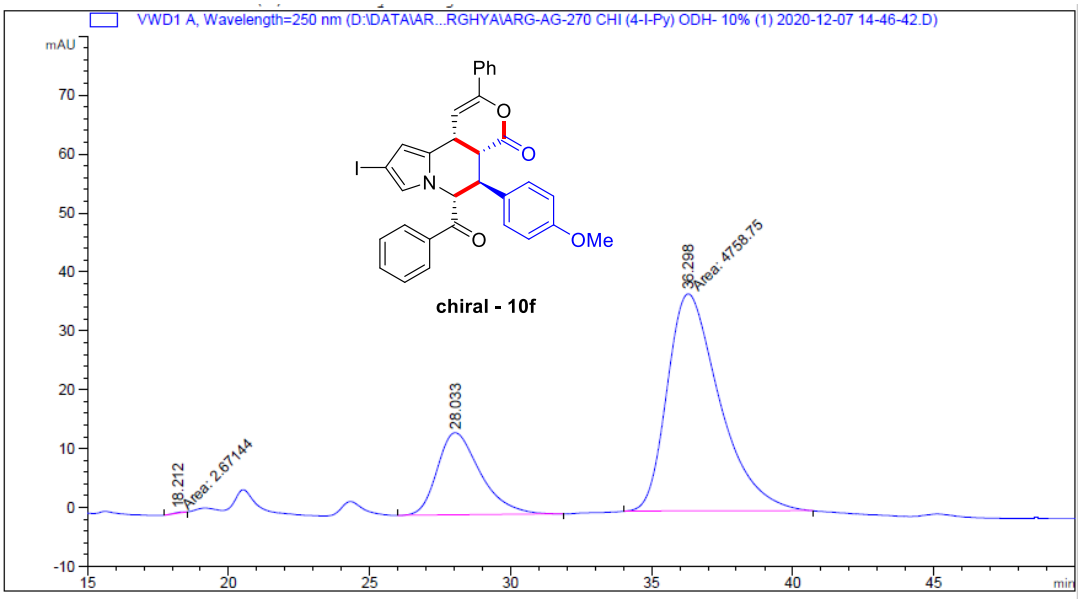
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	36.616	BB	0.8849	2117.62183	36.33844	43.8987
2	40.408	MM	2.3546	2706.26343	19.15561	56.1013

Sample Info : CHIRALPAK- ODH, 15 % IPA/HEXANE, .7 mL -min, 254 nM

(4a*R*,5*S*,6*R*,10b*R*)-6-Benzoyl-9-iodo-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10f)



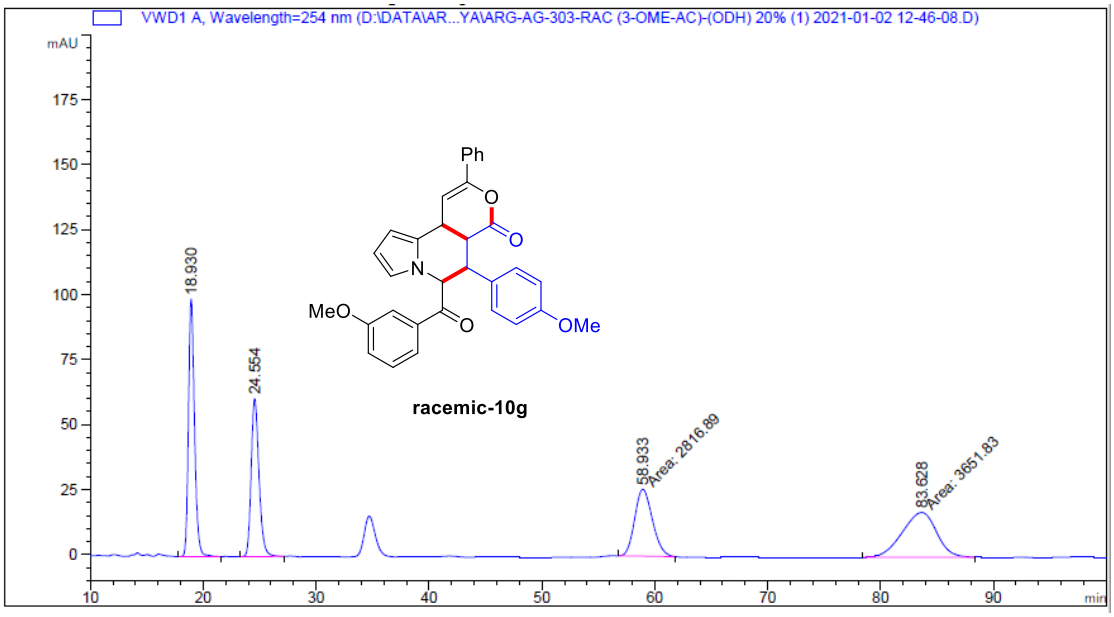
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.682	MM	1.5138	581.69360	6.40423	18.1959
2	28.497	MM	1.8068	577.96674	5.33142	18.0793
3	36.873	MM	2.0732	1022.82001	8.22237	31.9947
4	42.201	MM	2.6096	1014.36646	6.47836	31.7302



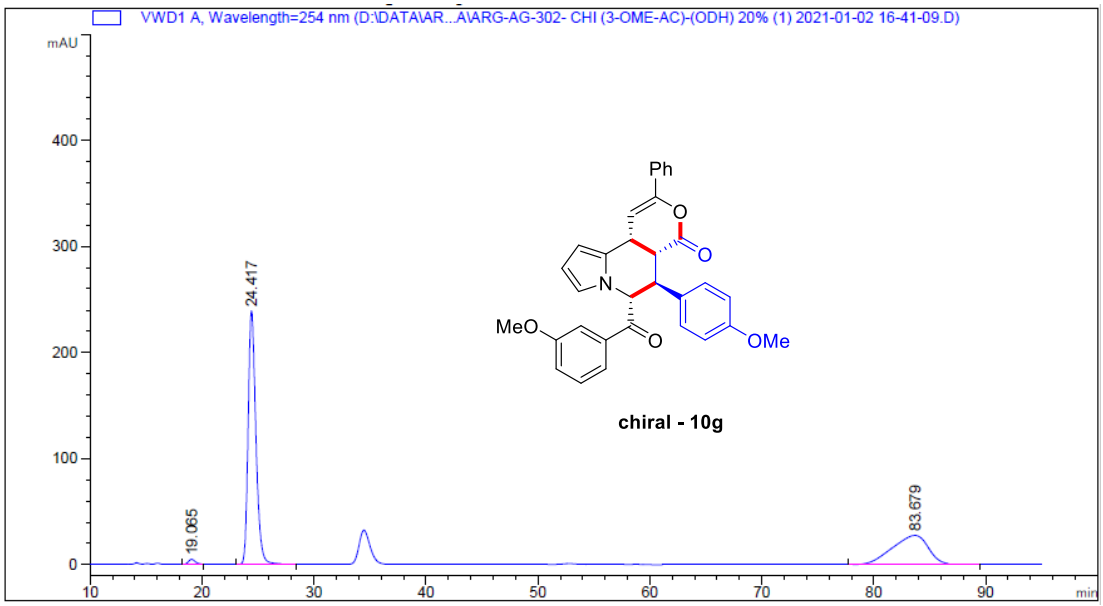
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.212	MM	0.3626	2.67144	1.22803e-1	0.0428
2	28.033	BB	1.5223	1478.41638	13.94061	23.6932
3	36.298	MM	2.1527	4758.74902	36.84281	76.2640

Sample Info : CHIRALCEL ODH, 10 % IPA/HEXANE, .7 mL -min, 254 nM

(4a*R*,5*S*,6*R*,10b*R*)-6-(3-Methoxybenzoyl)-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10g)



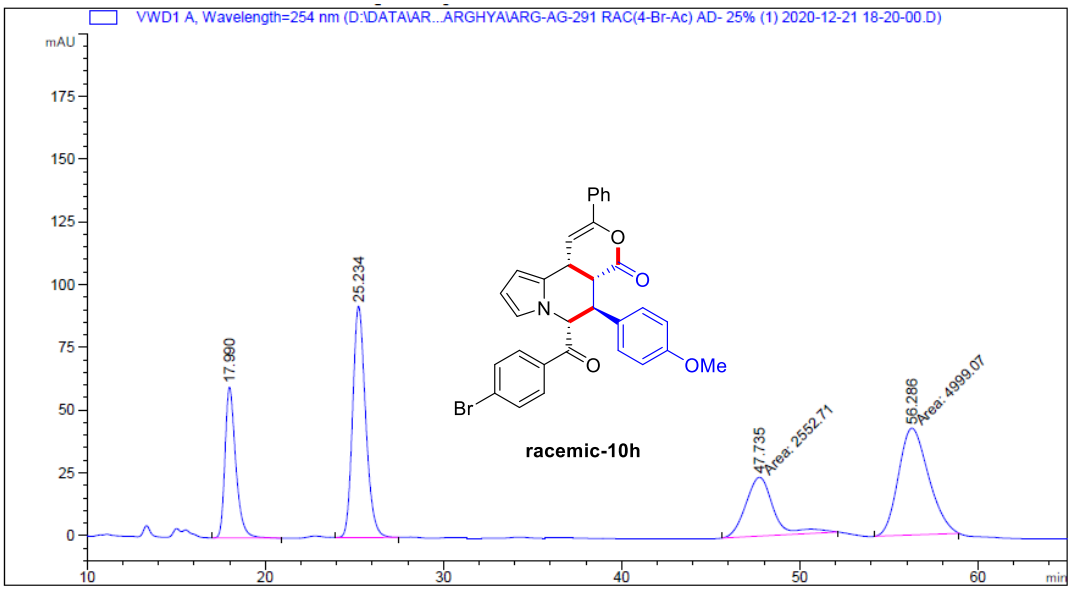
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.930	BB	0.5673	3680.00659	99.09443	28.1534
2	24.554	BB	0.7371	2922.54956	60.77134	22.3586
3	58.933	MM	1.8218	2816.89014	25.77083	21.5502
4	83.628	MM	3.5284	3651.82983	17.24960	27.9378



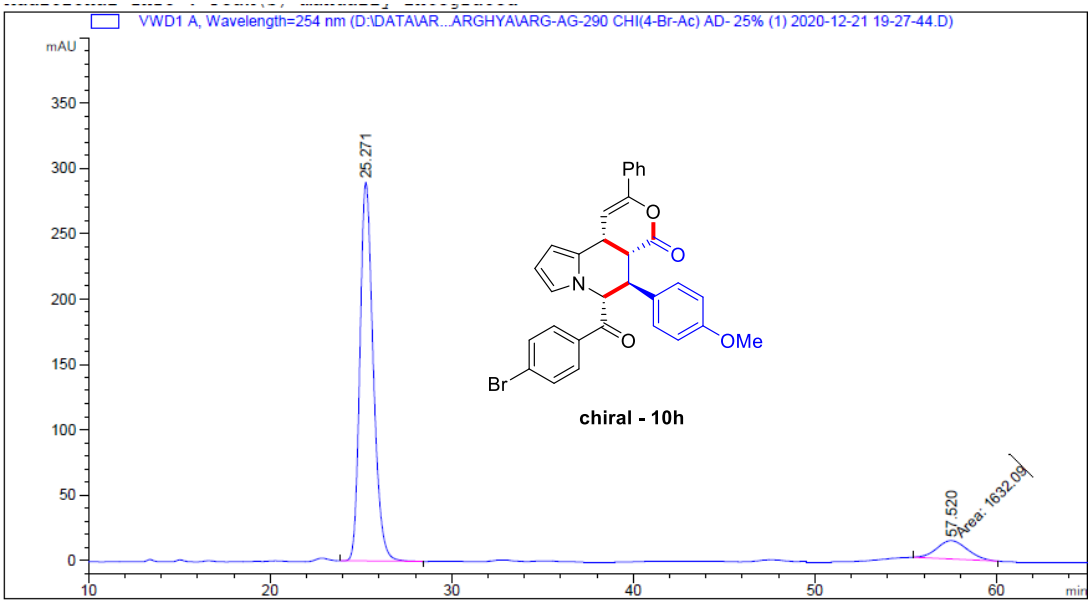
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.065	BB	0.6228	189.87975	4.62691	1.0579
2	24.417	BB	0.7329	1.14795e4	238.79106	63.9558
3	83.679	BB	2.9064	6279.71680	27.71510	34.9863

Sample Info : CHIRAPAK AD , 20% IPA-HEXANE, .7 mL/min.

(4a*R*,5*S*,6*R*,10b*R*)-6-(4-Bromobenzoyl)-5-(4-methoxyphenyl)-2-phenyl-4a,5,6,10b-tetrahydro-4*H*-pyrano[3,4-g]indolizin-4-one (10h)



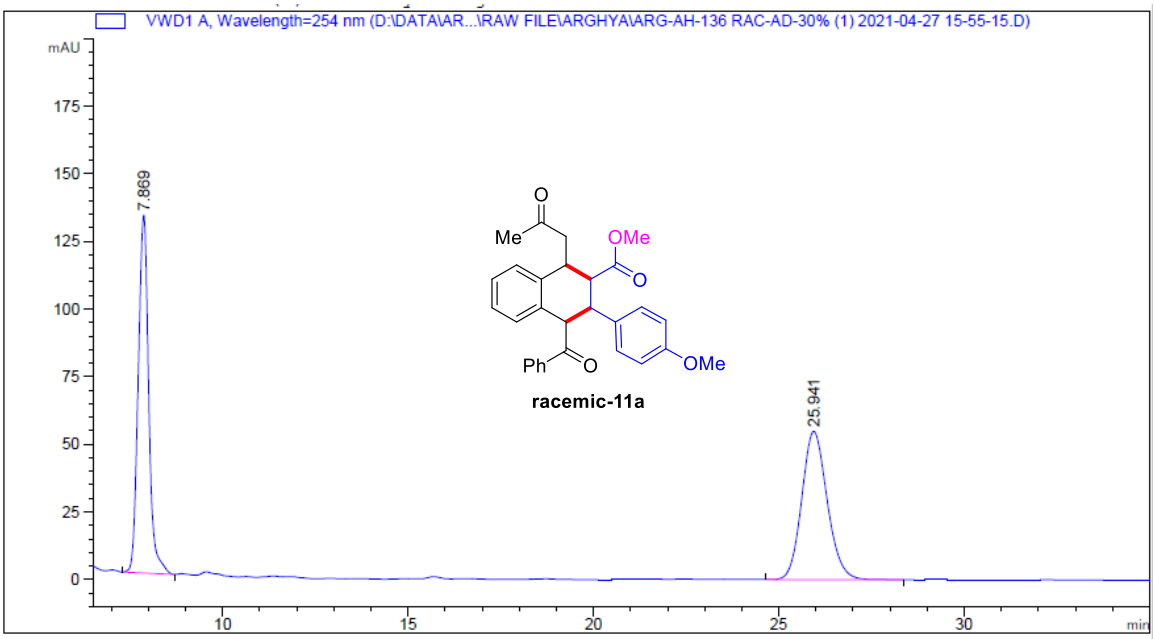
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.990	BB	0.6335	2519.45923	60.04304	17.0867
2	25.234	BB	0.7754	4673.93506	92.21004	31.6981
3	47.735	MM	1.8152	2552.70996	23.43846	17.3122
4	56.286	MM	1.9576	4999.06689	42.56183	33.9031



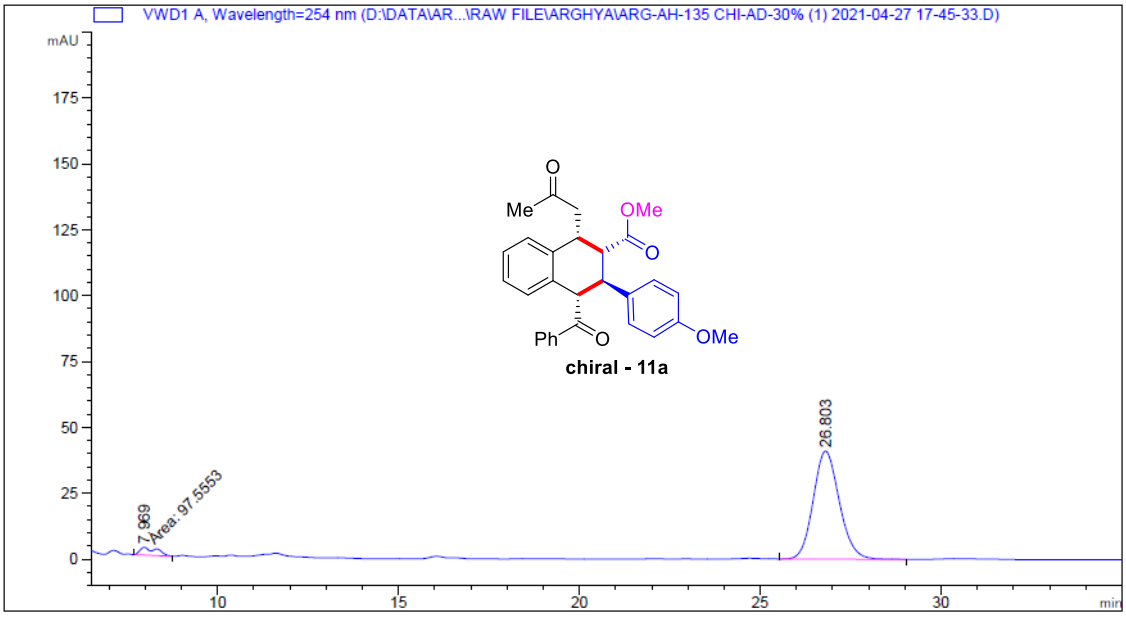
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.271	BB	0.7796	1.47636e4	289.65424	90.0456
2	57.520	MM	1.9389	1632.09155	14.02932	9.9544

Sample Info : CHIRALPAK AD , 25% IPA-HEXANE, .7 mL/min.

Methyl (2S,3S,4S)-4-benzoyl-3-(4-methoxyphenyl)-1-(2-oxopropyl)-1,2,3,4-tetrahydro naphthalene-2-carboxylate (11a)



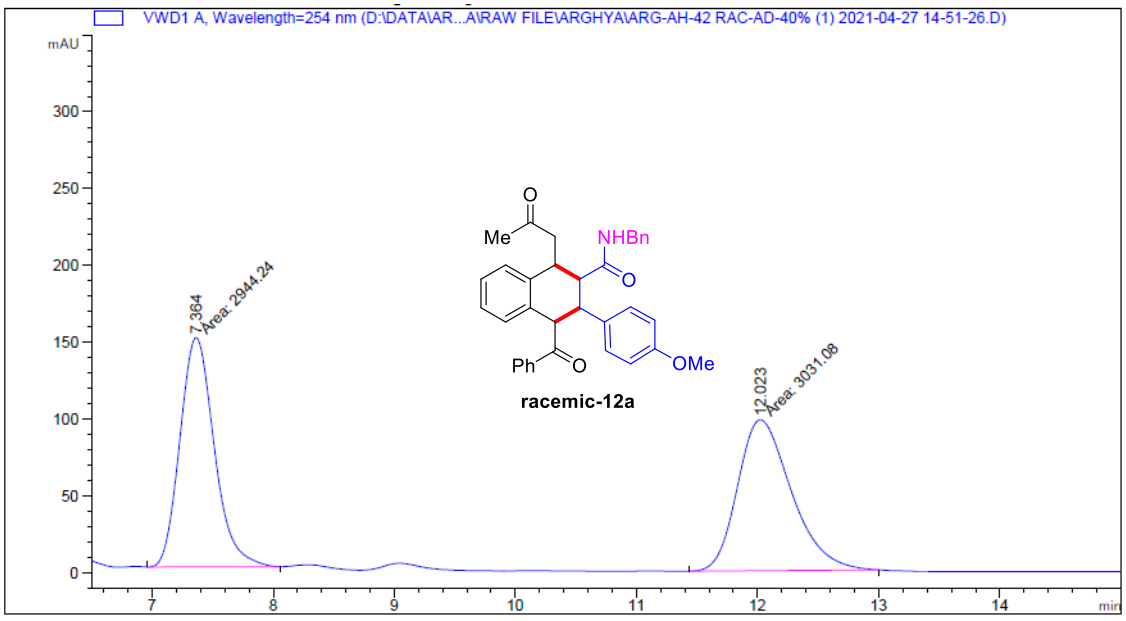
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.869	BB	0.3065	2591.43237	132.33221	49.7804
2	25.941	BB	0.7379	2614.29517	54.77166	50.2196



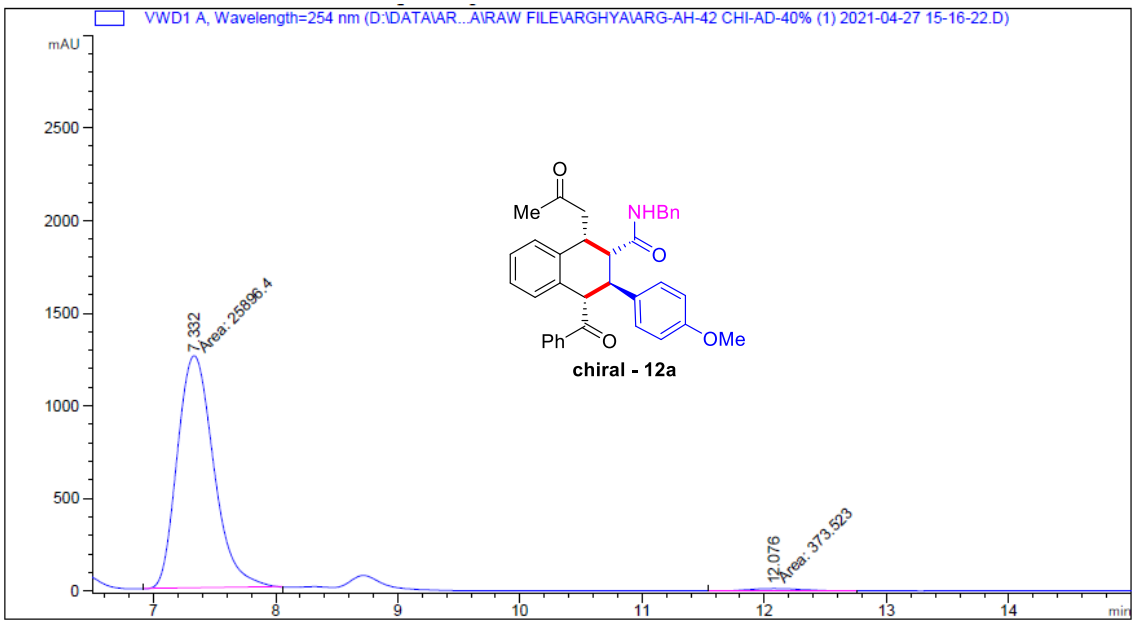
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.969	MM	0.5420	97.55525	2.99960	4.5384
2	26.803	BB	0.7715	2052.01953	40.96047	95.4616

Sample Info : CHIRALPAK AD, 30% IPA-HEXANE, .7 mL/min, 254 nm

(2*S*,3*S*,4*S*)-4-Benzoyl-N-benzyl-3-(4-methoxyphenyl)-1-(2-oxopropyl)-1,2,3,4-tetrahydro naphthalene-2-carboxamide (12a)



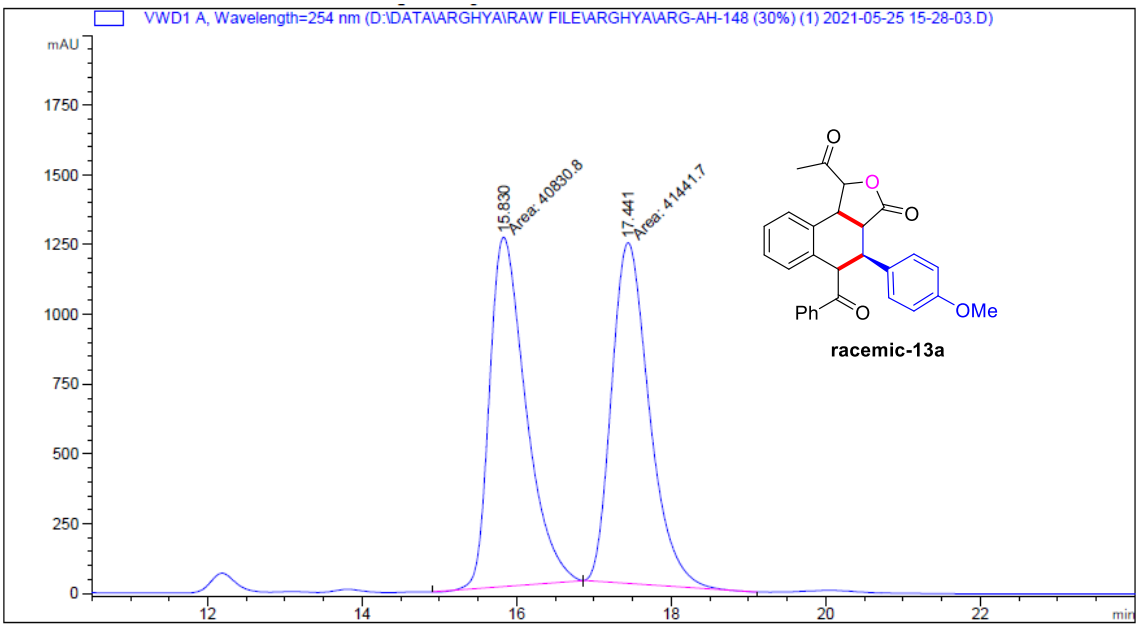
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.364	MM	0.3288	2944.23535	149.24939	49.2733
2	12.023	MM	0.5144	3031.08008	98.21487	50.7267



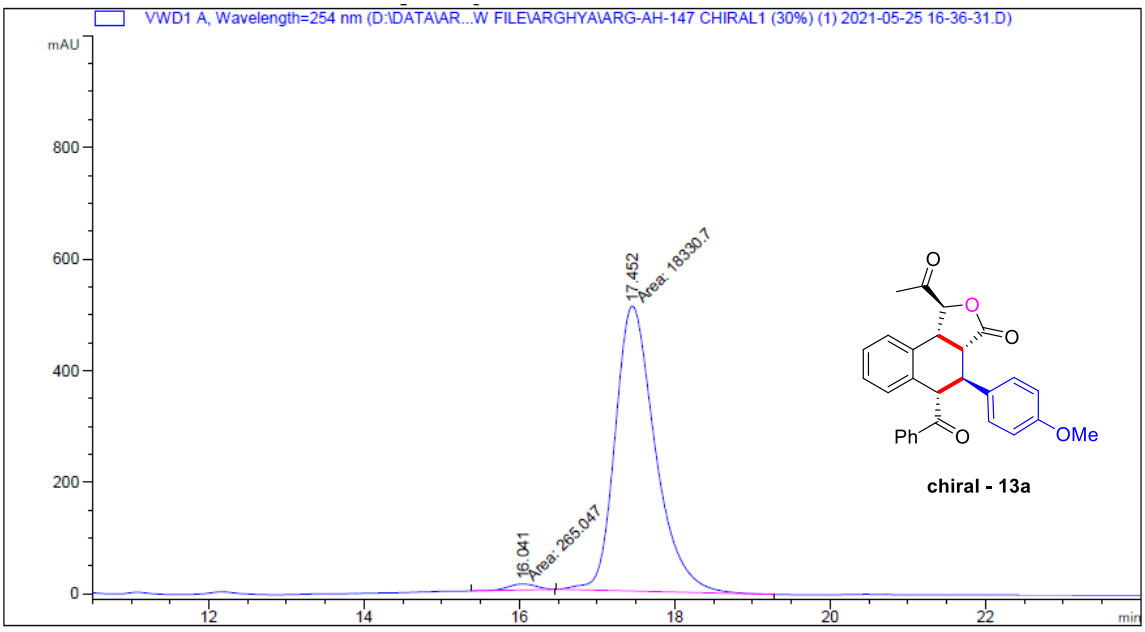
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.332	MM	0.3447	2.58964e4	1252.11340	98.5781
2	12.076	MM	0.4973	373.52286	12.51868	1.4219

Sample Info : CHIRALPAK AD, 40% IPA-HEXANE, .7 mL/min, 254 nm

(1*S*,3a*R*,4*S*,5S,9b*R*)-1-Acetyl-5-benzoyl-4-(4-methoxyphenyl)-3a,4,5,9b-tetrahydro naphtho[1,2-c]furan-3(1*H*)-one (13a)



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.830	MM	0.5437	4.08308e4	1251.72986	49.6287
2	17.441	MM	0.5657	4.14417e4	1220.90332	50.3713



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.041	MM	0.4132	265.04749	10.69192	1.4253
2	17.452	MM	0.5984	1.83307e4	510.56180	98.5747

Sample Info : CHIRALPAK AD, 30% IPA-HEXANE, .7 mL/min, 254 nm