Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Modular Three-Component Synthesis of Benzo Chromenone based Blue Luminogens under Catalyst- and Solvent-free Condition

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1. General Information

All chemicals were purchased from Sigma-Aldrich, Merck, Finar and Avra Synthesis, Pvt. Ltd. India and used as received. ACME silica gel (100-200 mesh) was used for column chromatography and thin-layer chromatography (TLC) was performed on Merck-precoated silica gel 60-F254 plates. The absorption and emission spectra of chromenone derivatives were analyzed using JASCO V-730 spectrophotometer and JASCO FP-8350 spectrofluorometer. Two and four-sided transparent quartz cuvettes with a path length of 1 cm were used and spectroscopic grade solvents used for spectral measurements were purchased from Spectrochem. ^{1}H NMR chemical shifts are expressed in parts per million (δ) downfield from tetramethylsilane (with the $CHCl_3$ peak around 7.26 ppm used as standard respectively). ^{13}C NMR chemical shifts are expressed in parts per million (δ) downfield from tetramethylsilane (with the central peak of $CHCl_3$ around 77.2 ppm used as standard respectively). All ^{13}C spectra were measured with complete proton decoupling. NMR coupling constants (*J*) are reported in Hertz (Hz), and splitting patterns are indicated as follows: br, broad; s, singlet; d, doublet; dd, doublet of doublet; ddd, doublet of doublet of doublet; dt, doublet of triplet; t, triplet; q, quartet; m, multiplet.

2. Experimental section:

2.1. Synthesis of 2-hydroxy chalcones

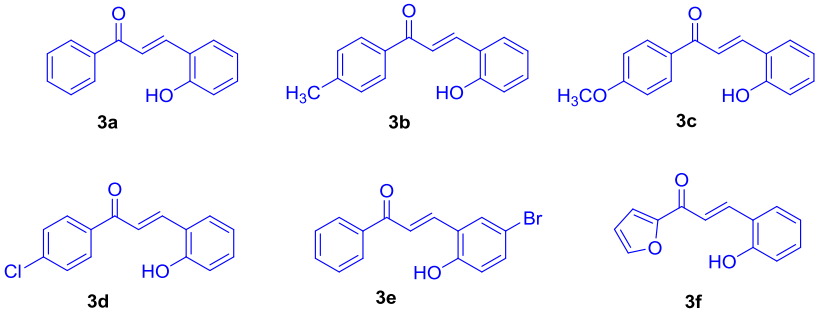
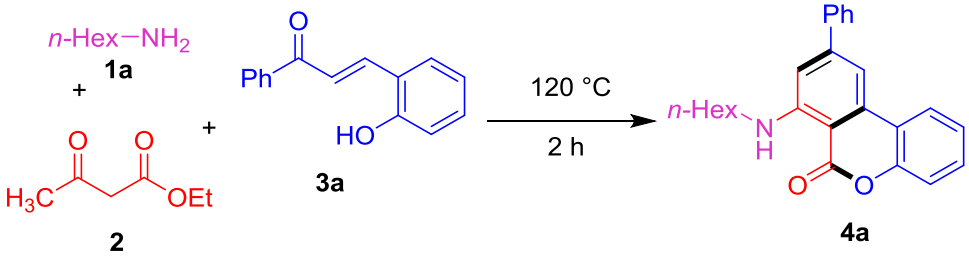


Fig 1. Various 2-hydroxy chalcones employed for the synthesis of 7-amino-6*H*-benzo[*c*]chromen-6-ones.[1]

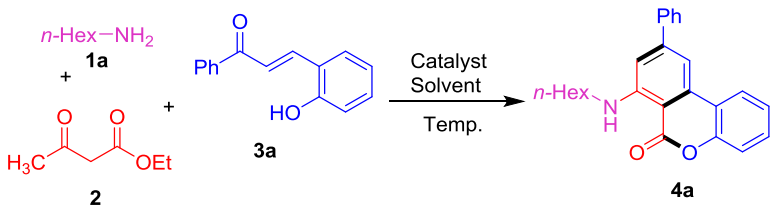
To the solution of substituted acetophenones (1.0 mmol) in 10:1 EtOH/H_2O mixture was added NaOH (1.5 mmol) at 0 °C. After 15 minutes of stirring addition of substituted benzaldehydes to reaction mixture and continued to 2 h at room temperature. Progress of the reaction was monitored by Thin Layer Chromatography (TLC). After completion of the reaction, filter the mixture with suction on a Buckner funnel and washed with cold water. The crude product was recrystallized from ethanol.

2.2. General Procedure for the Synthesis of Substituted 6*H*-benzo[*c*]chromen-6-ones:



To a mixture of hexylamine (**1a**) as 1.0 mmol, ethyl acetoacetate (**2**) as 1.5 mmol, and (E)-3-(2-hydroxyphenyl)-1-phenylprop-2-en-1-one (**3a**) as 1.0 mmol, was added and stirred at 120 °C for 2 h. The reaction progress was monitored by thin layer chromatography (TLC) and after completion of the reaction, the mixture was extracted with water. The organic layer was washed with brine solution and dried over anhydrous Na_2SO_4. Removal of the solvent under vacuum afforded the crude product, which was purified by column chromatography using a hexane/ethyl acetate mixture (19:1).

3. Optimization table for the construction of 6H-benzo[c]chromen-6-ones



Entry	Catalyst	Solvent	Temp. (°C)	Yield (%)[b]
1	S_8 (1.0 equiv.)	-	120	81
2	S_8 (50 mol%)	-	120	83
3	S_8 (20 mol%)	-	120	87
4	S_8 (10 mol%)	-	120	84
5	S_8 (5 mol%)	-	120	88
6	S_8 (5 mol%)	DMSO	120	NR
7	S_8 (5 mol%)	DCB	120	NR
8	S_8 (5 mol%)	PEG-400	120	Trace
9	S_8 (5 mol%)	Glycerol	120	Trace
10	I_2 (5 mol%)	-	120	39
11	$BF_3.Et_2O$ (5 mol%)	-	120	Trace
12	$SnCl_2$ (5 mol%)	-	120	NR
13	$InCl_3$ (5 mol%)	-	120	71
14	**-**	**-**	**120**	**93**
15	-	-	100	64
16	-	-	80	32
17	-	-	RT	NR

Reaction condition: [a] **1a** (1.0 mmol), **2** (1.5 mmol), **3a** (1.0 mmol), 120 °C in 2 h sealed tube. [b] isolated yield.

4. Green Chemistry Metrics:[4]

E-factor

E-factor = [total mass of raw materials minus the total mass of product]/ mass of product

= [0.195+0.099+0.224-0.291] g/ [0.291g]

E-factor = 0.77

Process mass intensity (PMI)

PMI = Σ [mass of stoichiometric (reactants+ reagent+ catalyst)]/ [mass of product]

= [0.195+0.099+0.224] g/ [0.291g]

PMI = 1.1

Reaction mass efficiency (RME)

RME = mass of product /Σ[mass of stoichiometric (reactants+ reagent+ catalyst)] × 100

= [0.291 g/ [0.195+0.099+0.224] g x 100

S. No	Reaction condition	*E*- factor	PMI	RME (%)
1.	$Sc(OTf)_2$ (10 mol%), Glycerol, 100 °C, 24 hr[2]	0.94	1.41	70
2.	$Ca(OTf)_2$ (10 mol%), Chloranil (1.0 equiv.), 100 °C.[3]	1.26	1.99	50
3.	**Our method:** Solvent and Catalyst free, 120 °C.	**0.77**	**1.10**	**89**

5. Single Crystal X-ray Diffraction

5.1. XRD of 4r

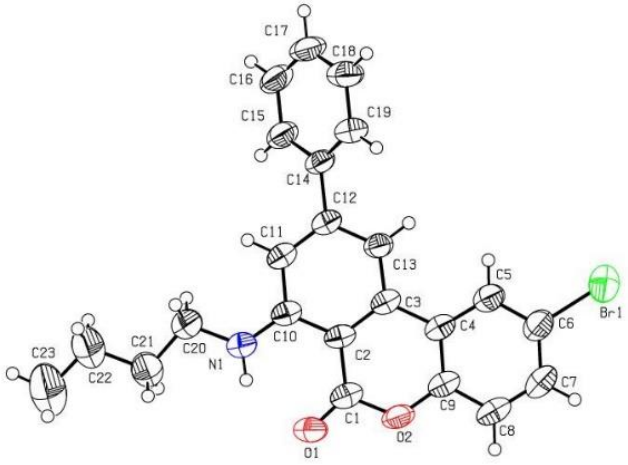


Fig 2. The ORTEP diagram of the compound **4r**

CCDC	**2296386**	
Empirical formula	$C_{23}H_{19}BrNO_2$	
Formula weight	421.30	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.2857(6) Å	□= 74.363(2)°.
	b = 9.3554(6) Å	□= 76.108(2)°.
	c = 12.1715(8) Å	□ = 89.435(2)°.
Final R factor	0.0430	
Structure solution	SHELXT (Sheldrick GM. (2015). Acta. Cryst. A71, 3-8.	
Structure refinement	SHELXL (Sheldrick GM. (2015). Acta. Cryst. C71, 3-8.	

5.2. Crystal packing of 4r

The unit cell packing of crystal structure (**4r**) viewed down a-axis is shown in Fig. 8. According to the packing diagram, no significant classical H-bonds are observed between the molecules. One intramolecular hydrogen bond is formed between the atoms N1 (donor) and O1 (acceptor) with the distance of 2.673(3) A. In addition, the structural stability in the unit cell packing is controlled dominantly by van der Waals forces between the molecules.

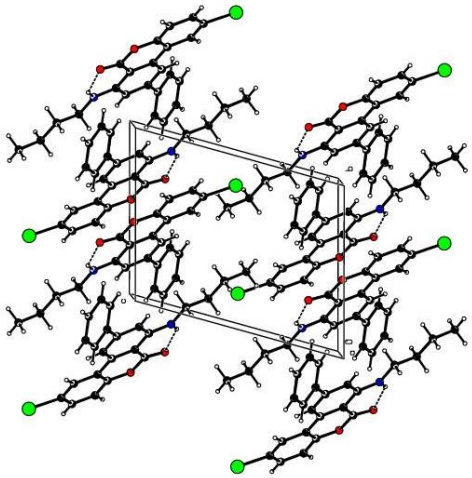


Fig 3. Crystal packing of **4r**

6. Photophysical studies

6. 1. Absorption and Emission spectra

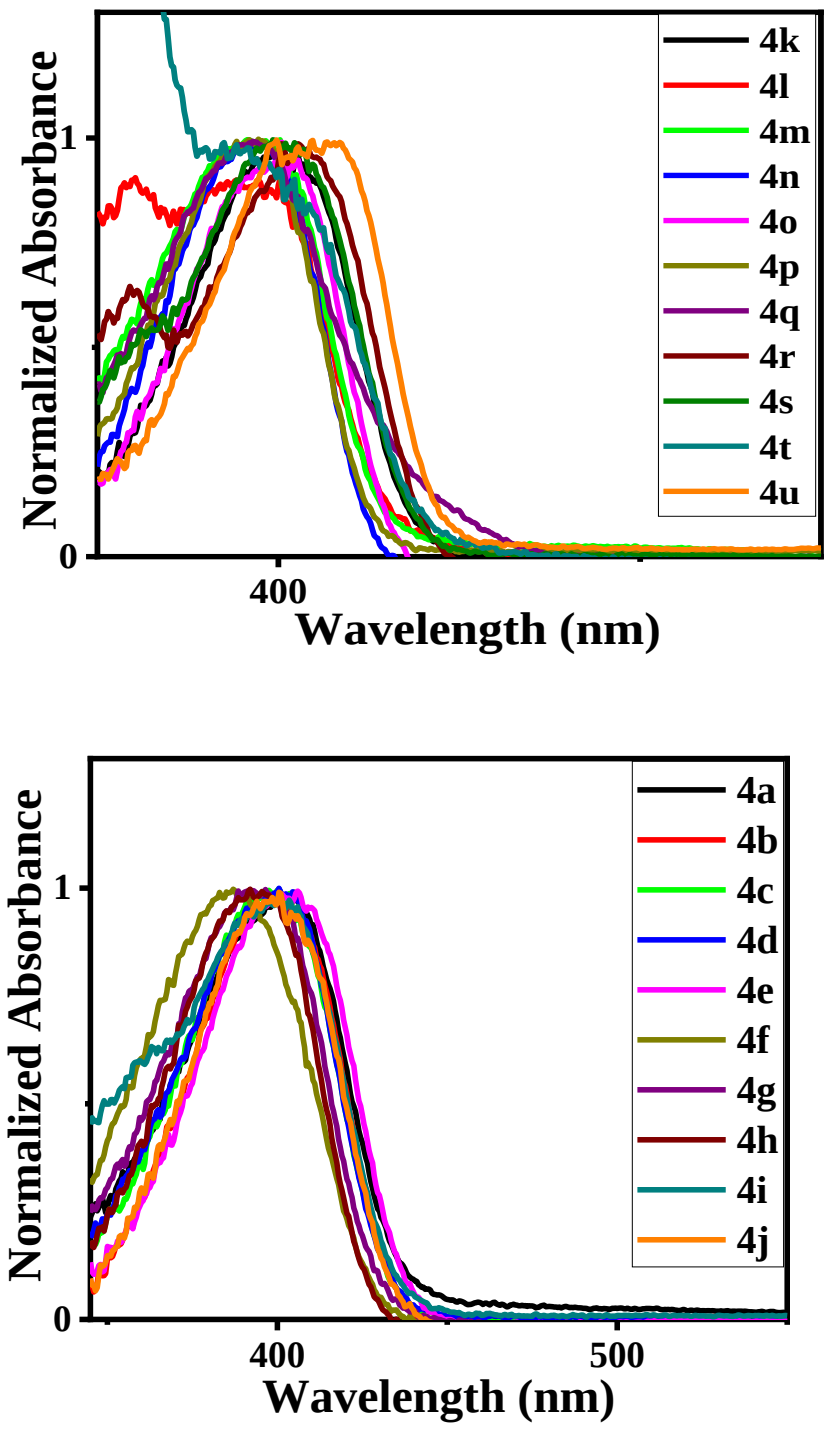
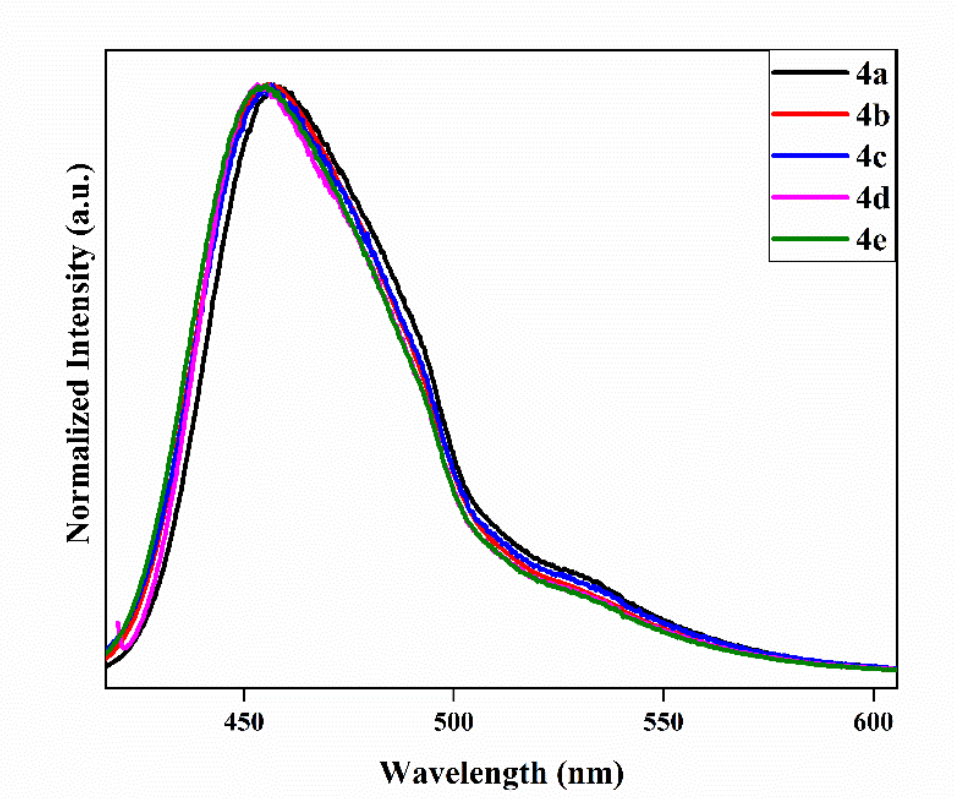
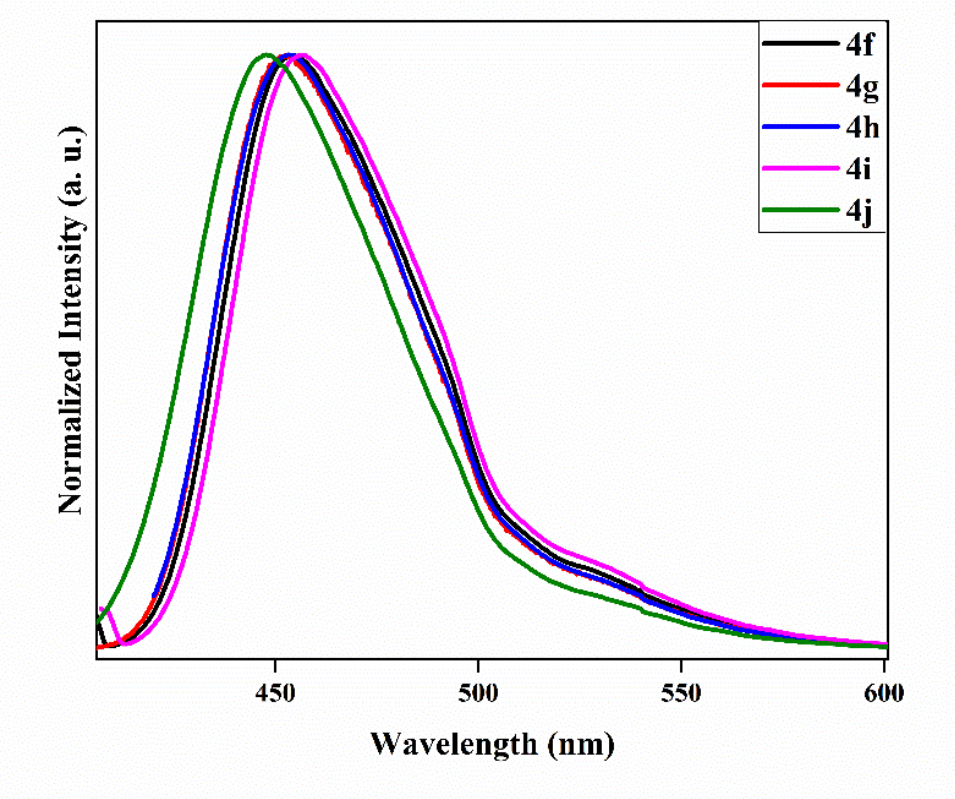
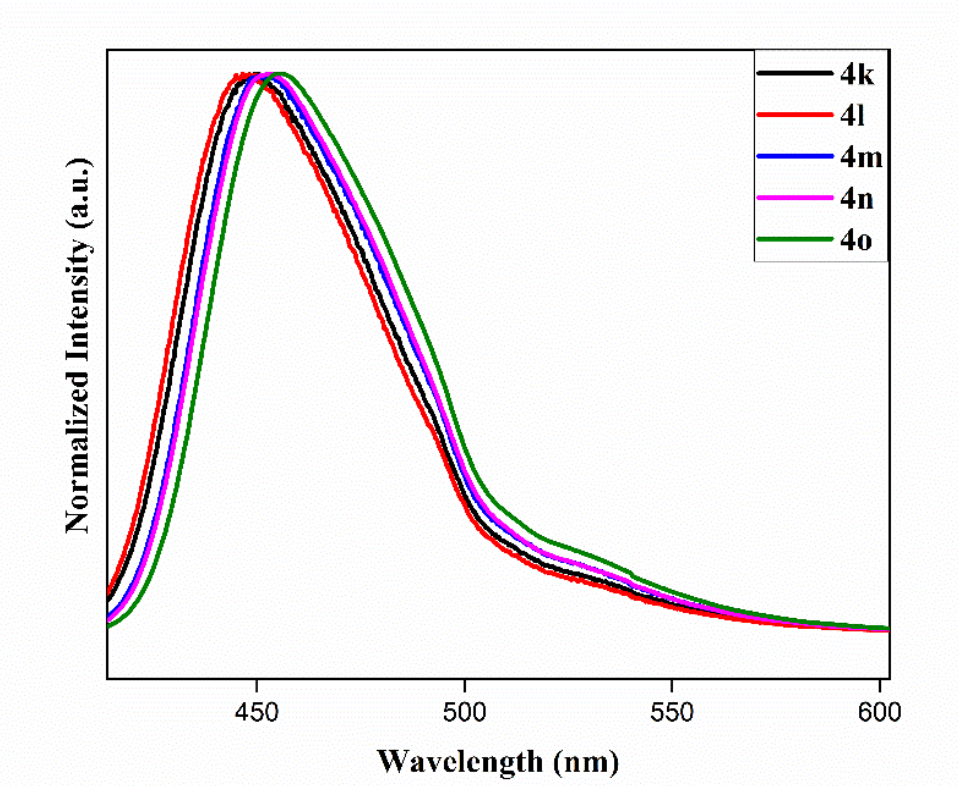


Fig 4. Absorption spectra of compound **4a-4u** in CH_3CN


4a
4b
4c
4d
4e
Normalized Intensity (a.u.)
450
500
550
600
Wavelength (nm)


4f
4g
4h
4i
4j
Normalized Intensity (a. u.)
450
500
550
600
Wavelength (nm)



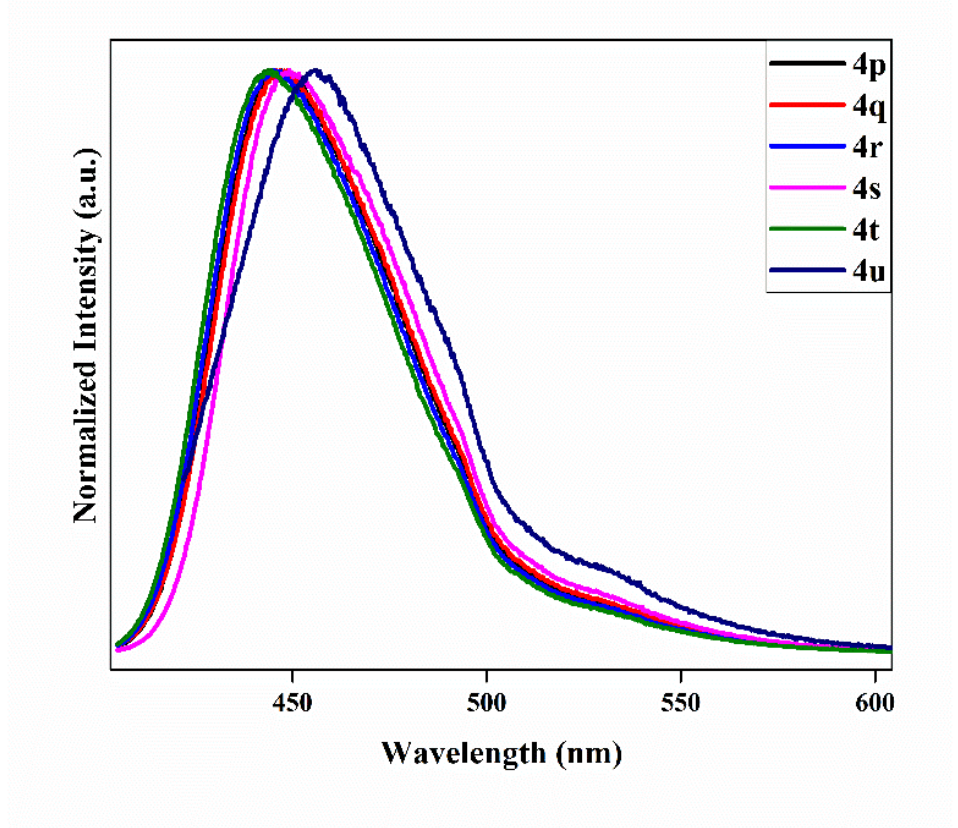


Fig 5. Emission spectra of compound **4a-4e, 4f-4j, 4k-4o, 4p-4u** in CH_3CN

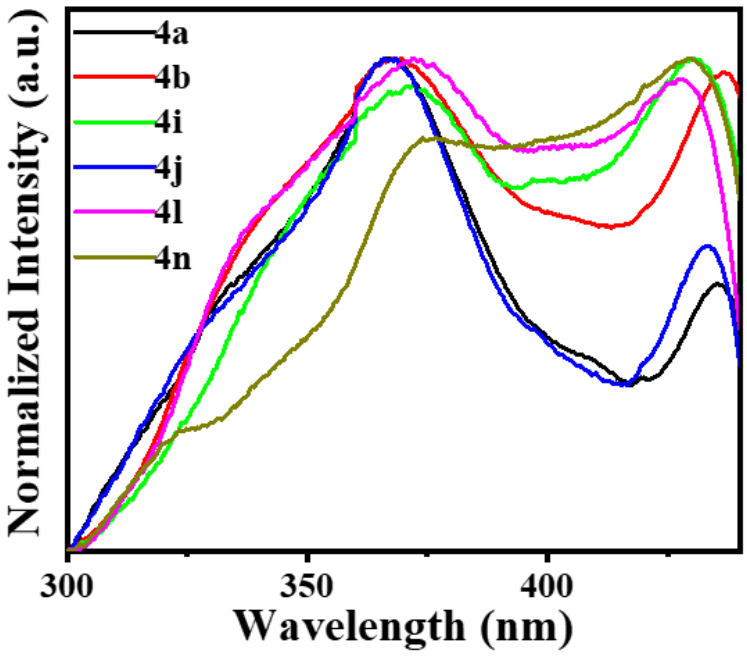


Fig 6. Solid-state excitation spectrum of **4a**, **4b**, **4i**, **4j**, **4l** and **4n**.

6.2 Quantum yield and CIE values

The absorption and emission maxima of the chromenone derivatives lie in the range of 385-410 nm and 445-460 nm, respectively. Relative quantum yield of the chromenone derivatives in acetonitrile was calculated using quinine sulphate in 0.5 M sulphuric acid as a reference (Φ_f = 0.546). The CIE values of the derivatives are close to the CIE coordinates (0.15,0.06) recommended by European Broadcast Union (EBU) for deep blue emission.

S. No.	Sample	Absorption maxima (nm)	Emission maxima (nm)	Quantum yield (%)	CIE Value	Color purity (%)
1.	**4a**	405	455	29	(0.15,0.12)	85
2.	**4b**	402	455	29	(0.15,0.12)	85
3.	**4c**	398	453	28	(0.15,0.11)	85
4.	**4d**	402	454	31	(0.15,0.11)	86
5.	**4e**	406	457	33	(0.15,0.13)	84
6.	**4f**	387	448	29	(0.15,0.09)	89
7.	**4g**	397	450	28	(0.15,0.09)	89
8.	**4h**	397	447	27	(0.15,0.09)	89
9.	**4i**	401	453	25	(0.15,0.11)	86
10.	**4j**	401	453	26	(0.15,0.11)	86
11.	**4k**	401	456	33	(0.15,0.12)	84
12.	**4l**	400	447	21	(0.15,0.09)	89
13.	**4m**	397	447	21	(0.15,0.09)	89
14.	**4n**	396	445	24	(0.15,0.09)	90
15.	**4o**	401	449	26	(0.15,0.10)	88
16.	**4p**	396	445	30	(0.15,0.08)	90
17.	**4q**	393	456	21	(0.15,0.12)	84
18.	**4r**	406	457	27	(0.15,0.14)	81
19.	**4s**	402	456	32	(0.15,0.13)	84
20.	**4t**	397	457	14	(0.15,0.13)	82
21.	**4u**	405	455	18	(0.15,0.12)	84

6. 3. Solid-State emission spectra and fluorescent images

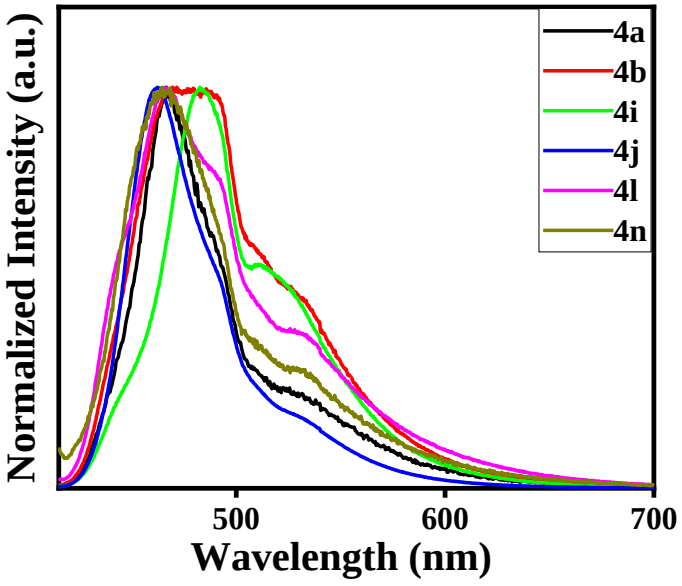


Fig 7. Solid-state emission spectra of compound **4a**, **4b**, **4i**, **4j**, **4j**, **4l** and **4n**.

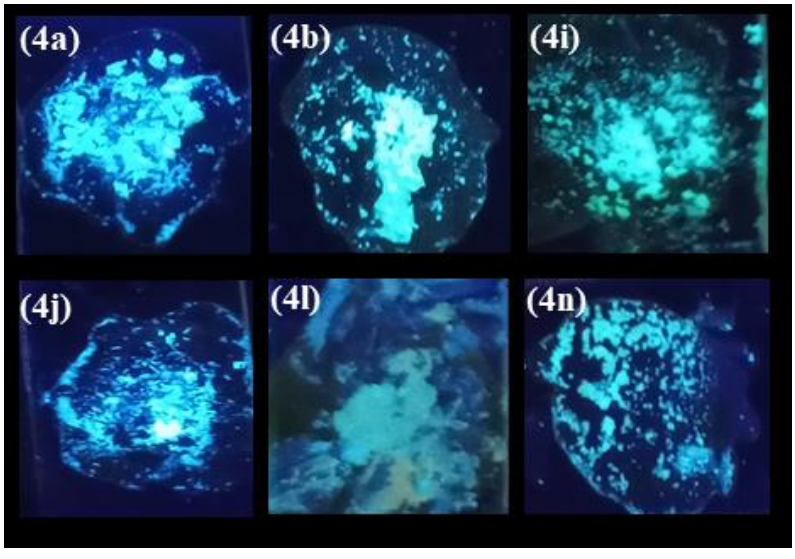
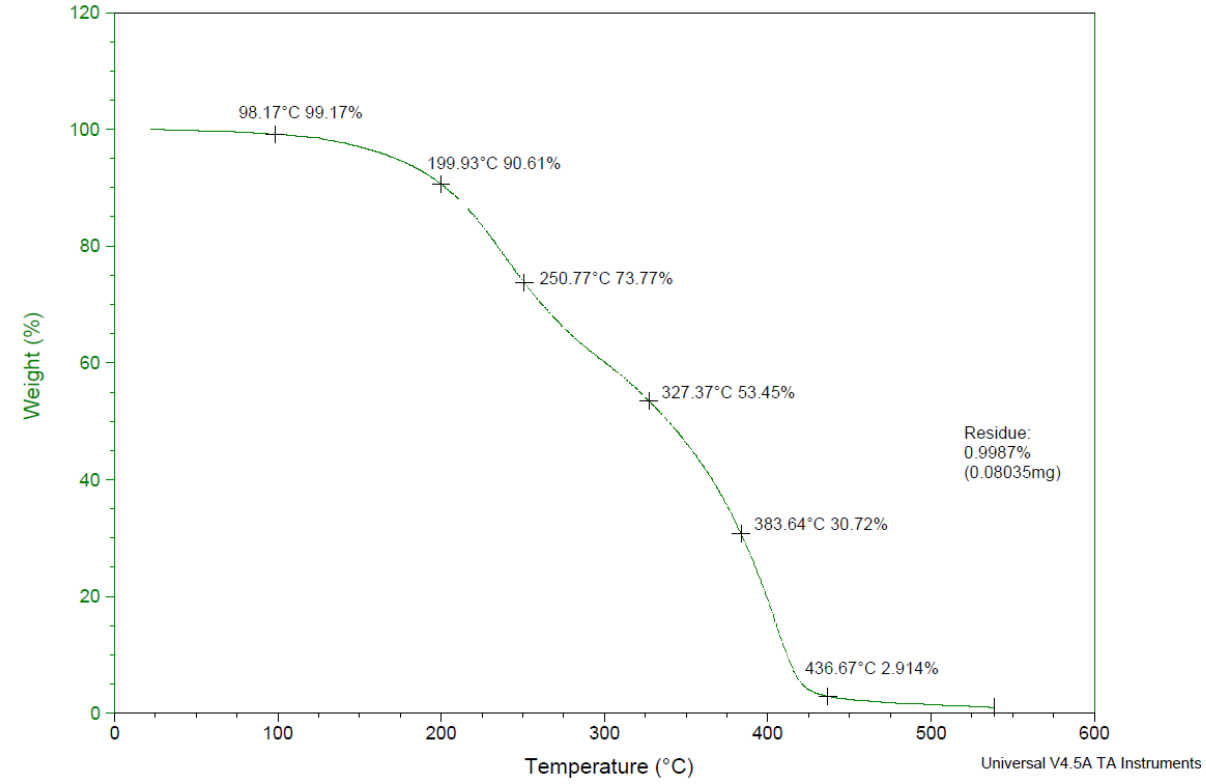
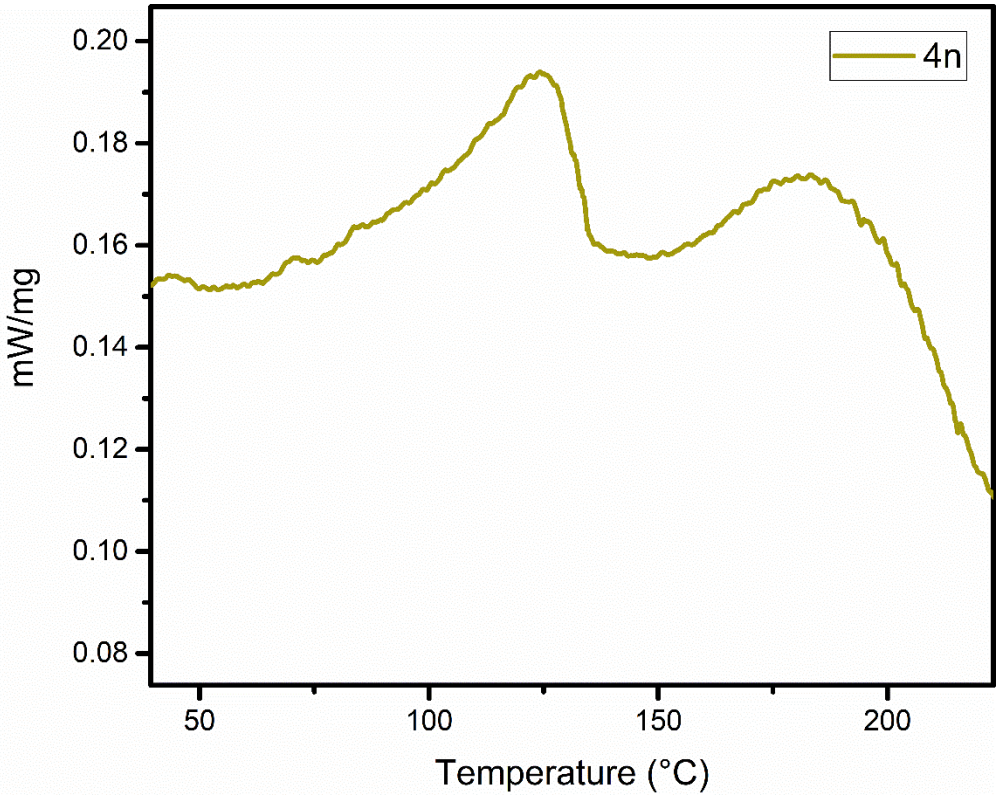


Fig 8. Solid-state fluorescent images of compound **4a**, **4b**, **4i**, **4j**, **4j**, **4l** and **4n**.

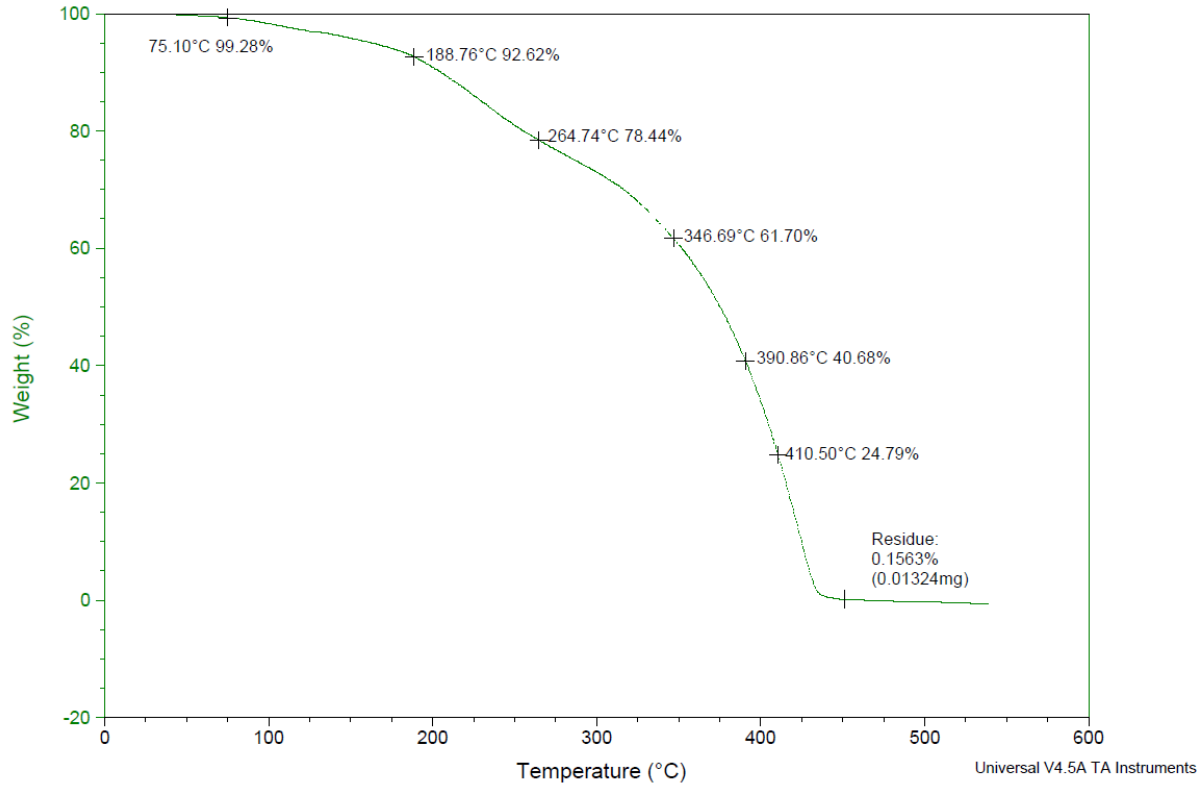
7. TGA and DSC analysis

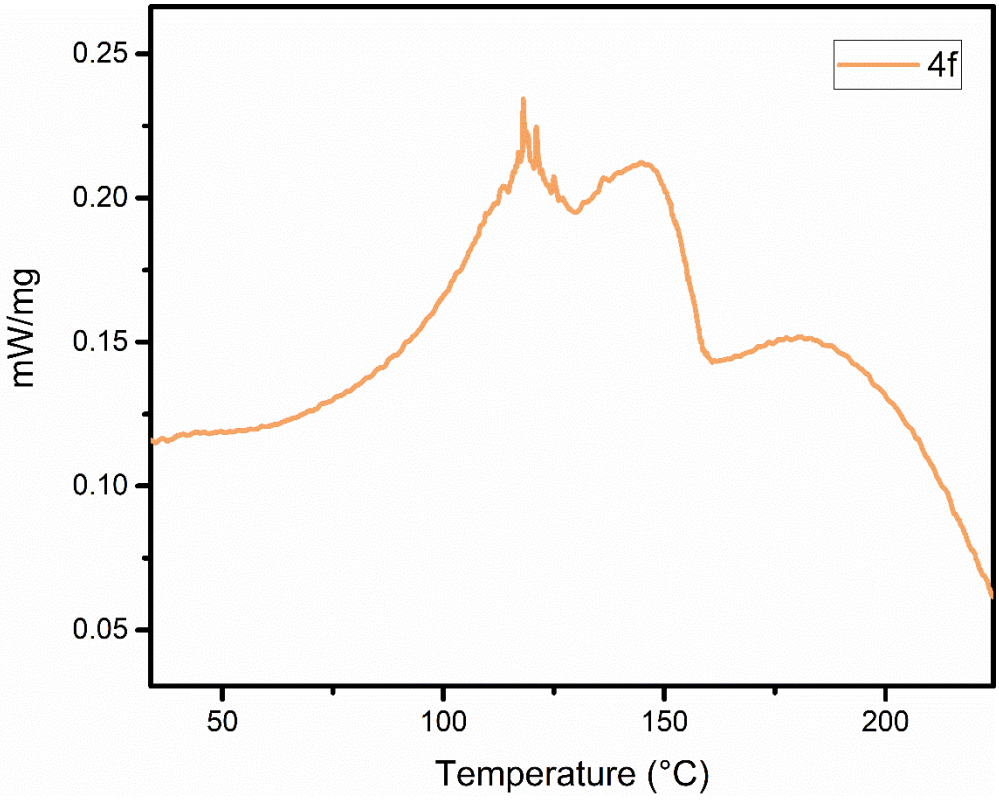
(i) TGA and DSC analysis of **4n**



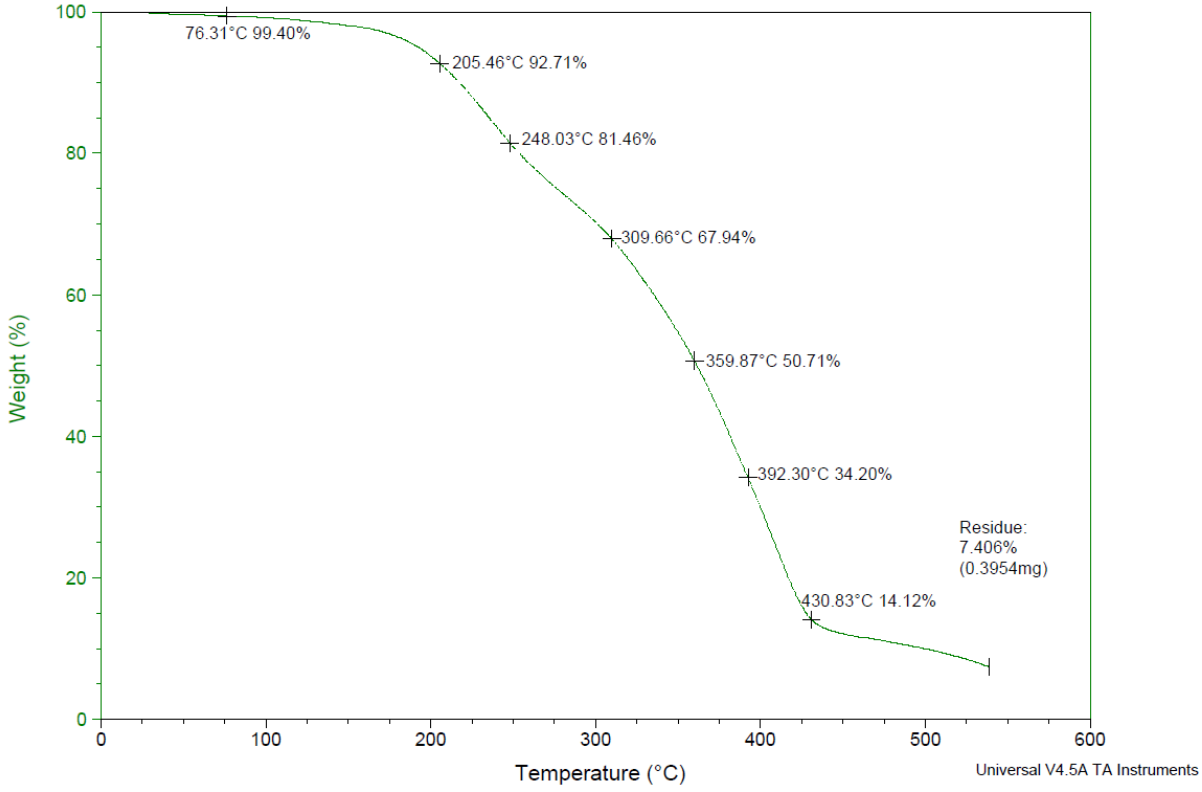


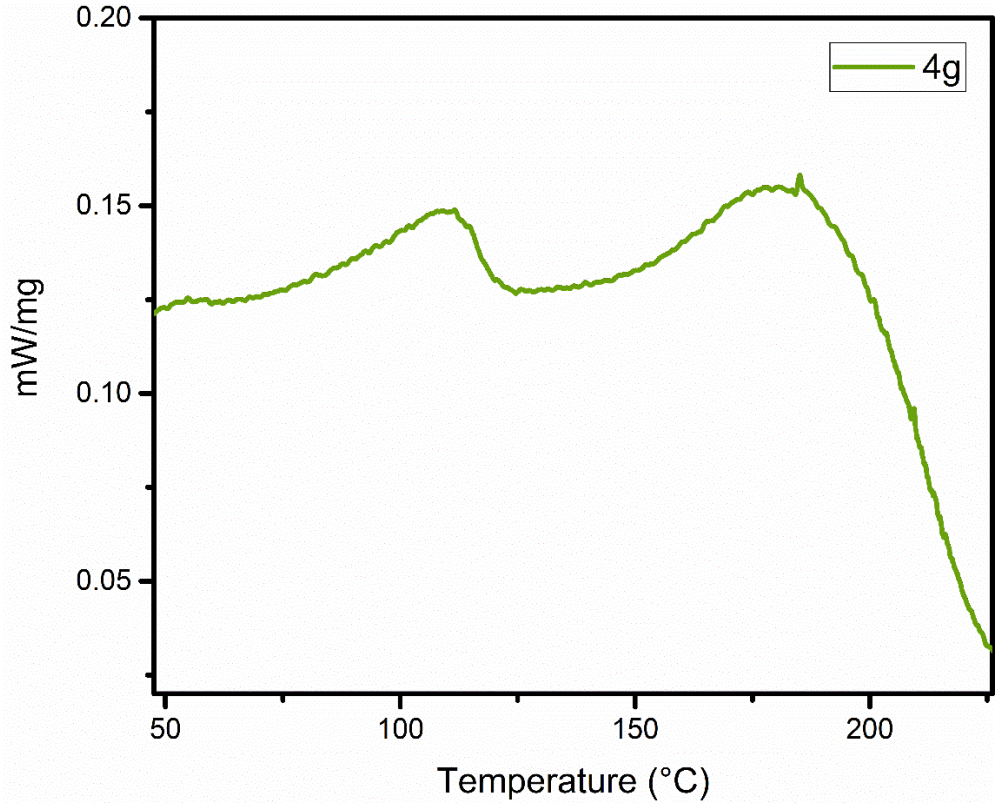
(ii) TGA and DSC analysis of **4f**



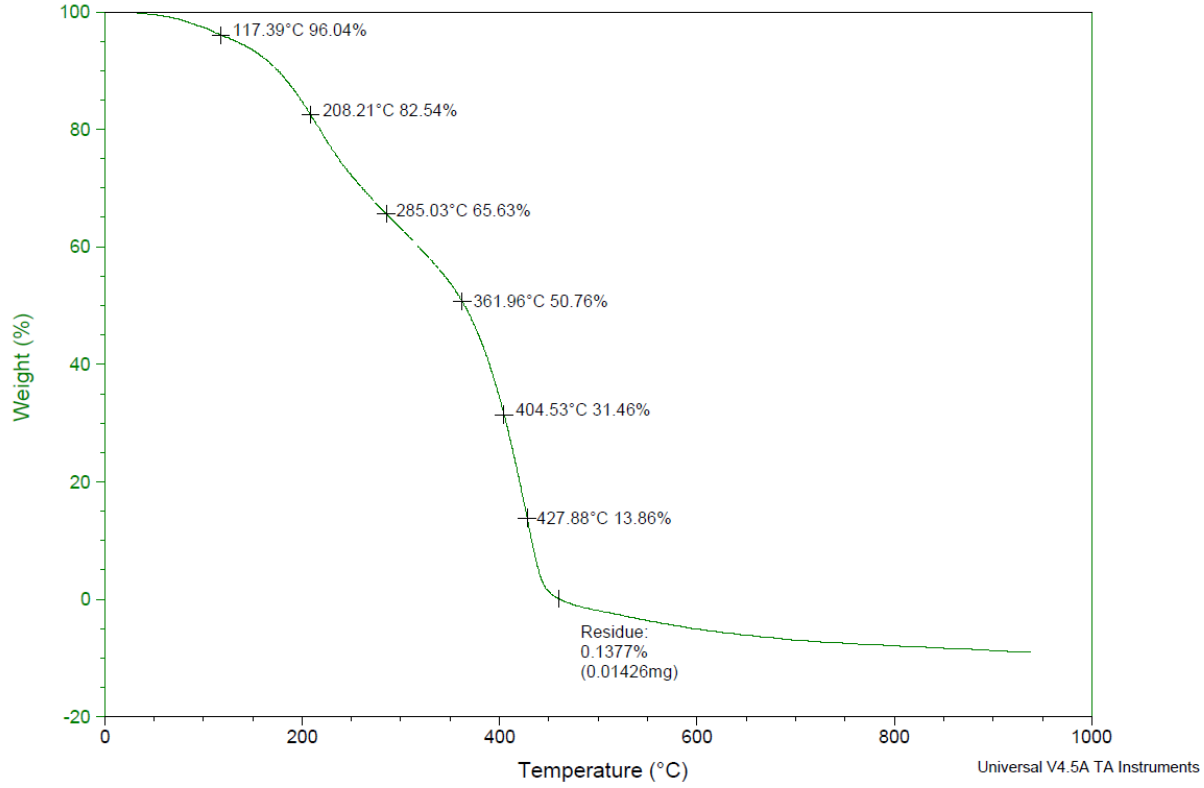


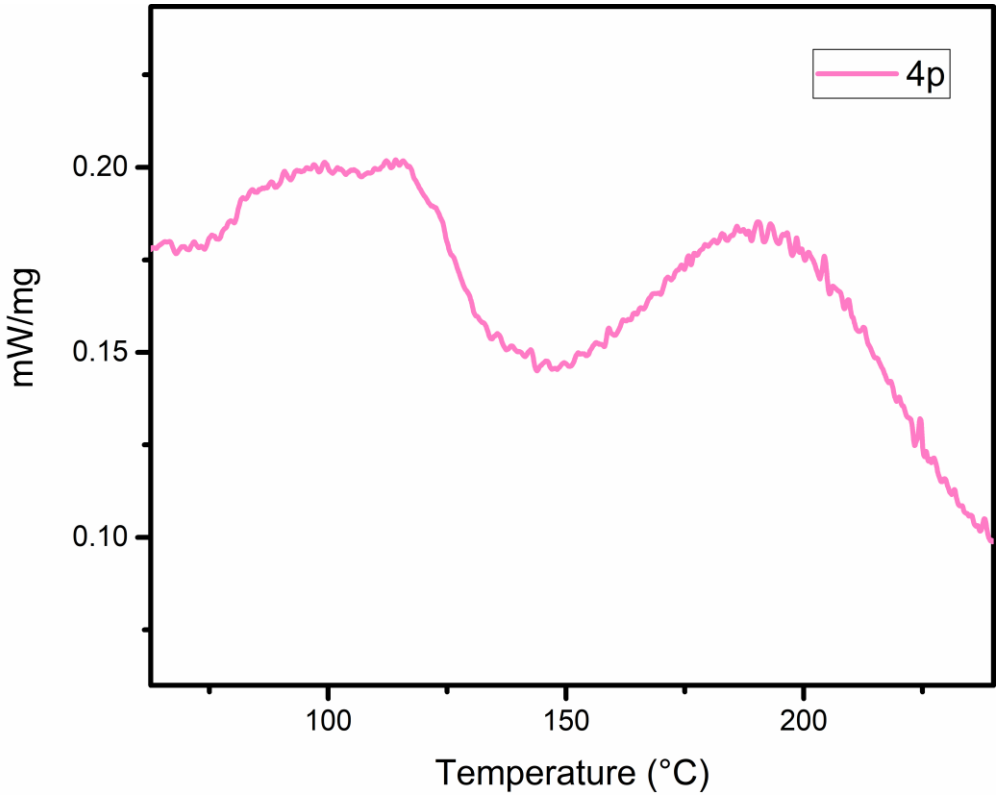
(iii) TGA and DSC analysis of **4g**





(iv) TGA and DSC analysis of **4p**





8. Electrochemical studies

8.1 Cyclic voltammetry for compound 4n, 4f, 4g, 4p

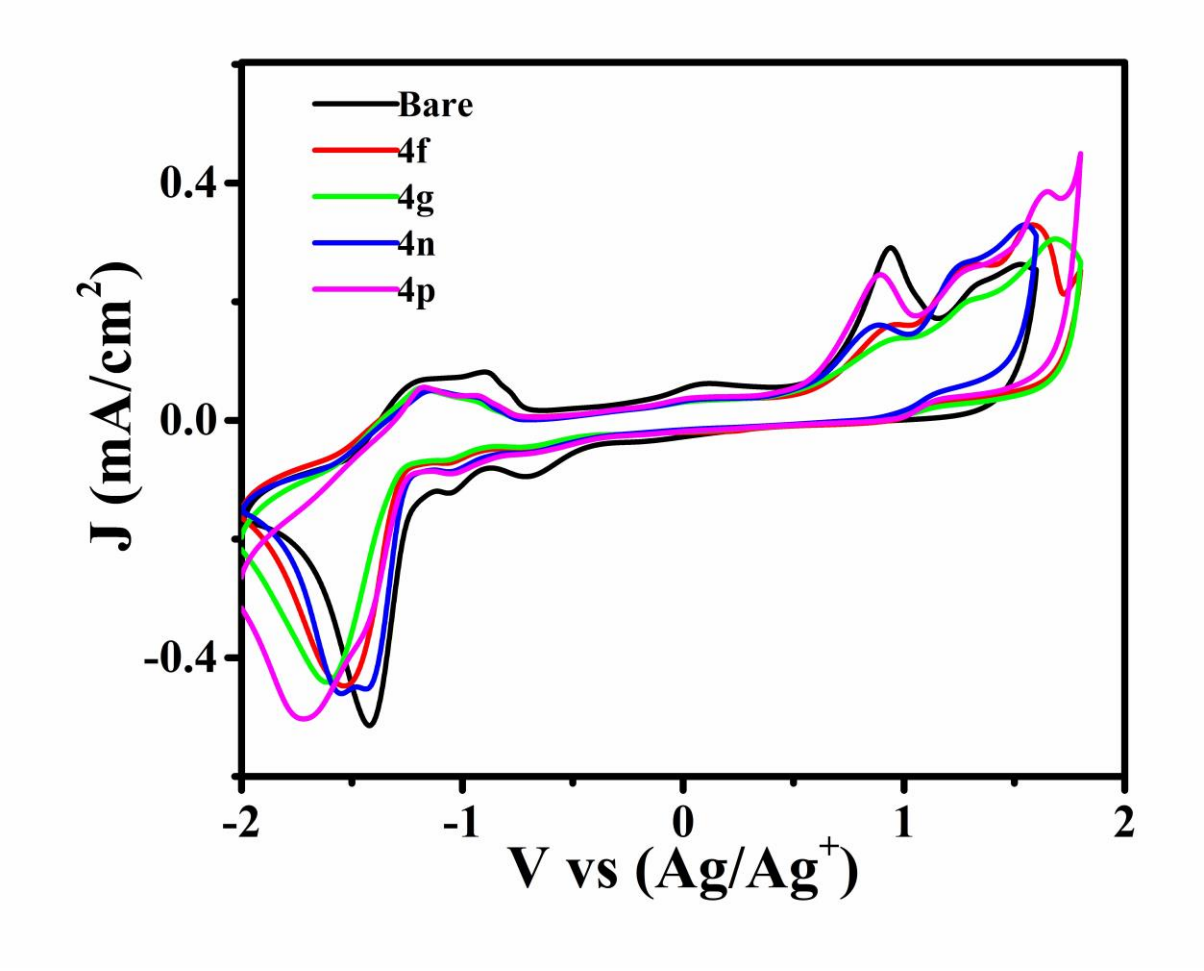


Fig 9. CV plot of compounds **4f, 4g, 4n, 4p.**

Working electrode : Platinum disc electrode

Counter electrode : Pt wire

Reference electrode : Saturated Ag/Ag^+

Electrolyte : 0.1 M

Solvent : Acetonitrile

Solute Concentration : 1mM

9. DFT Calculation

Computational studies of **4f**, **4g**, **4n** and **4p** were performed using schrödinger suite of programs. The ground-state geometry and energy of HOMO and LUMO levels of **4f, 4g, 4n** and, **4p** in acetonitrile was optimized by employing B3LYP-D3/6-31G(d,p) density functional theory and CPCM model is used to include the solvent effects. The results are shown in **Fig 8.**

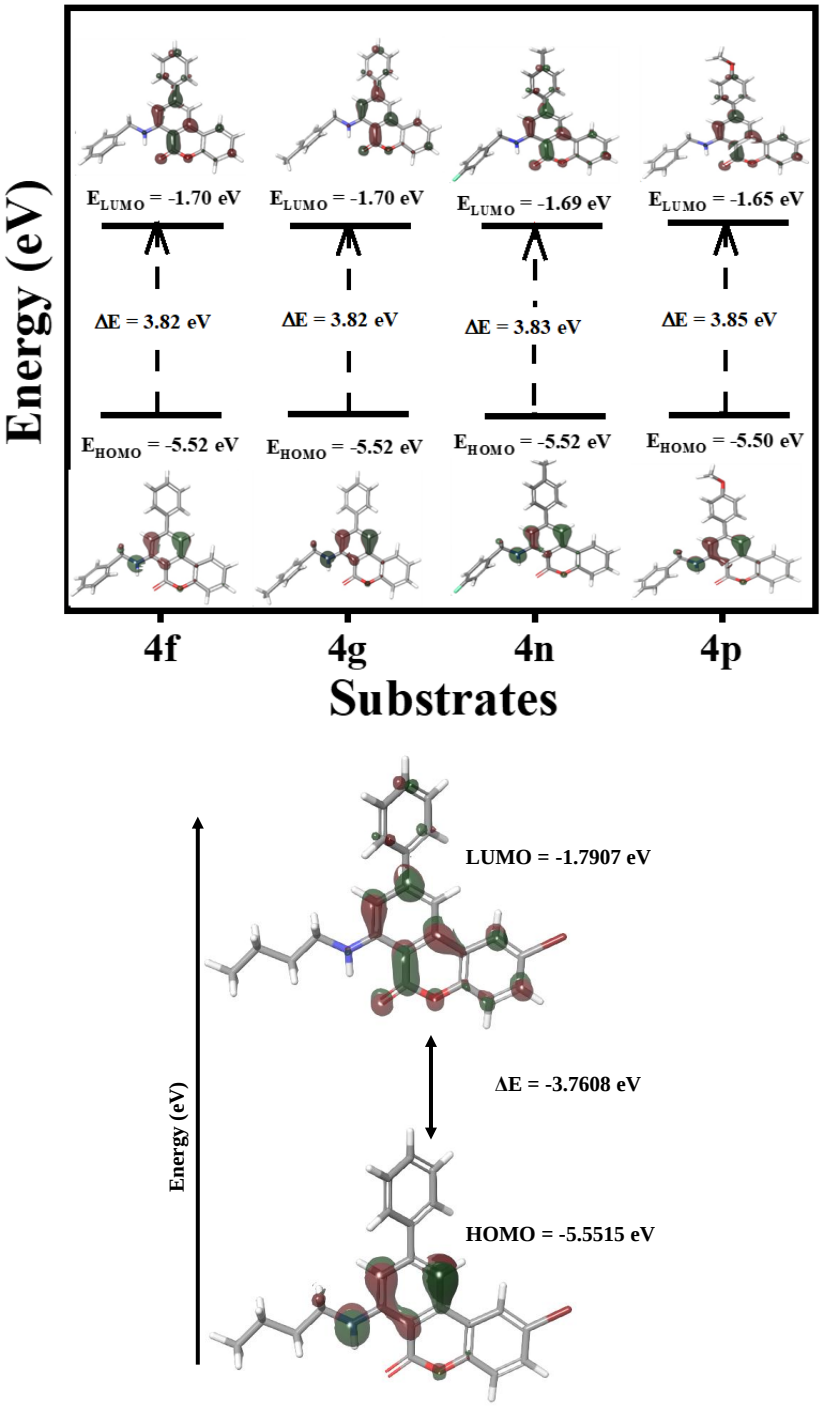
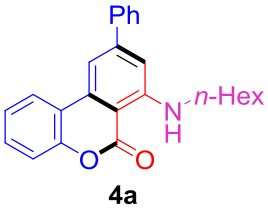


Fig 10. Frontier molecular orbitals of **4r**, **4f**, **4g**, **4n**, and **4p** in acetonitrile

10. Characterization Data (4a-u)

(7-(Hexylamino)-9-phenyl -6*H*-benzo[*c*]chromen-6-one (4a)



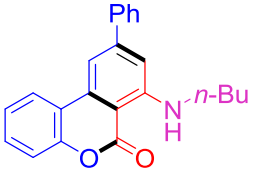
4a

Isolated yield = 93%, 345 mg, yellow solid; mp: 94-96 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.16 (t, *J* = 4.8 Hz, 1H), 8.07 (d, *J* = 7.8 Hz, 1H), 7.70-7.69 (m, 2H), 7.52-7.50 (m, 2H), 7.47-7.43 (m, 3H), 7.33-7.28 (m, 2H), 6.86 (s, 1H), 3.33 (q, *J* = 7.2 Hz, 2H), 1.78 (quin, *J* = 7.2 Hz, 2H), 1.52-1.47 (m, 2H), 1.36-1.33 (m, 2H), 0.91 (quin, *J* = 7.2 Hz, 3H).^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 152.6, 151.3, 148.8, 140.9, 136.6, 133.7, 130.1, 128.9, 128.6, 127.5, 124.3, 123.4, 118.9, 117.4, 108.5, 106.6, 101.9, 43.3, 31.7, 29.8, 28.8, 26.8, 22.6, 14.1.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

(7-(Butylamino)-9-phenyl-6*H*-benzo[*c*]chromen-6-one (4b)



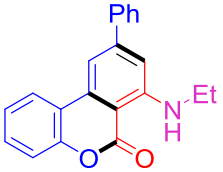
4b

Isolated yield = 85%, 291 mg, yellow solid; mp: 98-100 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.60 (t, *J* = 4.8 Hz, 1H), 8.06 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.69-7.67 (m, 2H), 7.51-7.47 (m, 2H), 7.45-7.42 (m, 3H), 7.32-7.27 (m, 2H), 6.85 (d, *J* = 1.2 Hz, 1H), 3.32 (q, *J* = 7.2 Hz, 2H), 1.76 (quin, *J* = 7.2 Hz, 2H), 1.51 (sextet, *J* = 7.8 Hz, 2H), 0.99 (t, *J* = 7.2 Hz, 3H).^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 152.6, 151.2, 148.6, 141.0, 136.6, 130.1, 128.9, 128.6, 127.4, 124.3, 123.4, 118.9, 117.3, 108.5, 106.6, 101.9, 42.9, 31.1, 20.5, 13.9.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

7-(ethylamino)-9-phenyl-6*H*-benzo[*c*]chromen-6-one (4c)

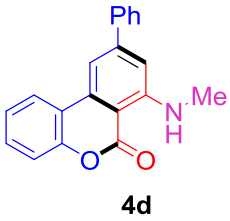


4c

Isolated yield = 84%, 264 mg, yellow solid; mp: 97-99 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.53 (t, *J* = 4.8 Hz, 1H), 8.06 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.68 (dd, *J* = 7.8, 1.2 Hz, 2H), 7.51-7.48 (m, 2H), 7.45-7.42 (m, 3H), 7.32-7.27 (m, 2H), 6.85 (d, *J* = 1.2 Hz, 1H), 3.37 (q, *J* = 7.2 Hz, 2H), 1.39 (t, *J* = 7.2Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.3, 152.5, 151.2, 148.8, 140.9, 136.6, 130.1, 128.9, 128.6, 127.5, 124.3, 123.3, 119.1, 117.4, 108.5, 106.6, 101.8, 37.8, 14.4. HRMS (ESI): calculated for m/z 315.1259 ($[M+H]^+$); found. m/z 316.1301.

7-(methylamino)-9-phenyl-6*H*-benzo[*c*]chromen-6-one (4d)



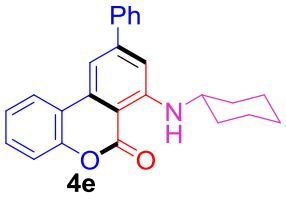
4d

Isolated yield = 86% 258 mg, yellow solid; mp: 100-103 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.55 (d, *J* = 4.8Hz, 1H), 8.07 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.70-7.68 (m, 2H), 7.51-7.49 (m, 2H), 7.46-7.42 (m, 3H), 7.32-7.27 (m, 2H), 6.84 (d, *J* = 1.8 Hz, 1H), 3.04 (d, *J* = 4.8 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 153.3, 151.2, 148.8, 140.9, 136.6, 130.2, 129.0, 128.7, 127.5, 124.3, 123.3, 118.9, 117.4, 108.1, 106.7, 102.0, 29.8.

Physical and spectral properties of this compound were identical to those previously reported in literature.[3]

(7-(Cyclohexylamino)-9-phenyl -6*H*-benzo[*c*]chromen-6-one (4e)

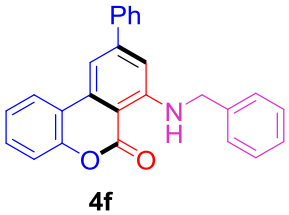


4e

Isolated yield = 79%, 291 mg, yellow solid; mp: 96-100 °C

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.70 (d, *J* = 7.2 Hz, 1H), 8.05 (dd, *J* = 7.8, 1.8 Hz 1H), 7.67-7.65 (m, 2H), 7.52-7.48 (m, 2H), 7.45-7.41 (m, 2H), 7.37-7.38 (m, 1H), 7.31-7.25 (m, 2H), 6.86 (s, 1H), 3.57-3.52 (m, 1H), 2.11-2.07 (m, 2H), 1.84-1.79 (m, 2H), 1.66-1.62 (m, 1H), 1.48-1.40 (m, 4H), 1.36-1.31 (m, 1H). Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

7-(benzylamino)-9-phenyl-6*H*-benzo[*c*]chromen-6-one (4f)



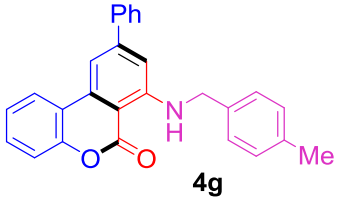
4f

Isolated yield = 75%, 282 mg, yellow solid; mp: 149-151°C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 9.10 (t, *J* = 5.4 Hz, 1H), 8.06 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.55-7.53 (m, 2H), 7.47-7.43 (m, 4H), 7.42-7.40 (m, 3H), 7.37-7.35 (m, 2H), 7.33-7.32 (m, 1H), 7.30-7.27 (m, 2H), 6.83 (d, *J*= 2.4 Hz, 1H), 4.59 (d, *J* = 6.0 Hz, 2H).^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 152.4, 151.2, 148.7, 140.8, 138.2, 136.7, 130.3, 129.0, 128.8, 128.6, 127.4, 127.2, 124.4, 123.4, 118.9, 117.4, 109.3, 107.3, 102.4, 47.3.

Physical and spectral properties of this compound were identical to those previously reported in literature.[3]

7-((4-methylbenzyl)amino)-9-phenyl-6H-benzo[c]chromen-6-one (4g)



4g

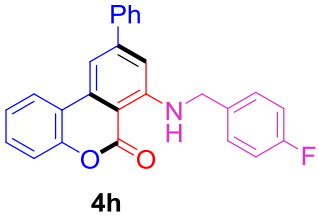
Isolated yield = 80%, 312 mg, yellow solid; mp: 130-133 °C

^{1}H NMR (600 MHz, $CDCl_3$): δ 9.06 (t, *J* = 2.8 Hz, 1H), 8.08 (dd, *J* = 4.1, 0.7 Hz, 1H), 7.58 (d, *J* = 4.0 Hz, 2H), 7.51 – 7.36 (m, 5H), 7.35 – 7.28 (m, 4H), 7.17 (d, *J* = 4.0 Hz, 2H), 6.87 (s, 1H), 4.56 (d, *J* = 2.8

Hz, 2H), 2.35 (s, 1H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 162.31, 151.30, 150.16, 147.63, 139.71, 135.97, 135.57, 134.01, 129.12, 128.44, 127.86, 127.51, 126.36, 126.11, 123.27, 122.26, 117.82, 116.33, 108.11, 106.07, 101.29, 45.94, 20.09.

HRMS (ESI): calculated for m/z 391.1572 ($[M+H]^+$); found. m/z 392.1594.

7-((4-fluorobenzyl)amino)-9-phenyl-6*H*-benzo[*c*]chromen-6-one (4h)

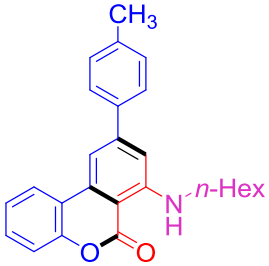


4h

Isolated yield = 72%, 284 mg, yellow solid; mp: 155-158 °C

^{1}H NMR (600 MHz, $CDCl_3$): δ 9.07 (t, *J* = 5.4 Hz, 1H), 8.06 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.55-7.53 (m, 2H), 7.48-7.43 (m, 4H), 7.43-7.40 (m, 1H), 7.38-7.36 (m, 2H), 7.33-7.28 (m, 2H), 7.06-7.03 (m, 2H), 6.79 (d, *J* = 1.2 Hz, 1H), 4.56 (d, *J* = 6.0 Hz, 2H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 160.2, 152.4, 150.7, 148.2, 138.3, 136.6, 133.0, 130.2, 129.2, 128.6, 127.5, 124.2, 123.3, 119.0, 117.4, 114.4, 108.7, 105.4, 102.5, 47.3. HRMS (ESI): calculated for m/z 395.1322 ($[M+H]^+$); found. m/z 396.1375.

(7-(Hexylamino)-9-(*p*-tolyl)-6*H*-benzo[*c*]chromen-6-one (4i)



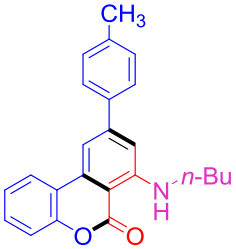
4i

Isolated yield = 89%, 342 mg, yellow solid; mp: 114-116 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.58 (t, *J* = 4.8 Hz, 1H), 8.05 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.58-7.57 (m, 2H), 7.43-7.41 (m, 2H), 7.31-7.26 (m, 4H), 6.84 (d, *J* = 1.2 Hz, 1H), 3.31 (q, *J* = 7.2Hz, 2H), 2.48 (s, 3H), 1.76 (quin, *J* = 7.2 Hz, 2H), 1.47 (quin, *J* = 7.2 Hz, 2H), 1.36-1.33 (m, 2H), 0.90 (quin, *J* = 7.2 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.3, 152.6, 151.2, 148.0, 138.7, 138.0, 136.5, 130.0, 129.8, 129.6, 127.3, 124.2, 119.0, 117.4, 108.2, 106.4, 101.6, 43.2, 31.7, 28.9, 27.0, 21.3, 14.1.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

(7-(Butylamino)-9-(*p*-tolyl)-6*H*-benzo[*c*]chromen-6-one (4j)



4j

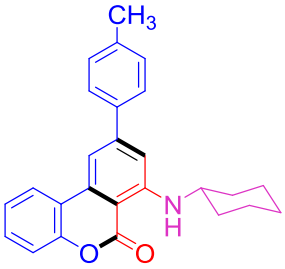
Isolated yield = 83%, 296 mg, yellow solid; mp: 147-151 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.58 (t, *J* = 4.8 Hz, 1H), 8.06 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.58 (d, *J* = 8.4 Hz, 2H), 7.44-7.41 (m, 2H), 7.31-7.27 (m, 4H), 6.84 (s, 1H), 3.32 (q, *J* = 7.2 Hz, 2H), 2.43 (s, 3H), 1.76 (quin, *J* = 7.2 Hz, 2H), 1.51 (sextet, *J* = 7.8 Hz, 2H), 0.98 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (150

MHz, $CDCl_3$): δ 163.4, 152.6, 151.3, 148.7, 138.6, 138.1, 136.6, 130.0, 129.6, 127.3, 124.3, 123.3, 119.0, 117.4, 108.3, 106.4, 101.6, 42.9, 31.1, 21.3, 20.5, 13.9.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

(7-(Cyclohexylamino)-9-(*p*-tolyl)-6*H*-benzo[*c*]chromen-6-one (4k)



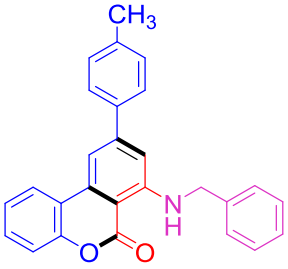
4k

Isolated yield = 78%, 298 mg, yellow solid; mp: 138-141 °C

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.68 (d, *J* = 7.2 Hz, 1H), 8.04 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.57-7.56 (m, 2H), 7.43-7.40 (m, 1H), 7.37 (d, *J* = 1.2 Hz, 1H), 7.31-7.25 (m, 4H), 6.85 (s, 1H), 3.55-3.53 (m, 1H), 2.43 (s, 3H), 2.10-2.07 (m, 2H), 1.82-1.79 (m, 2H), 1.67-1.63 (m, 1H), 1.48-1.39 (m, 4H), 1.37-1.31 (m, 1H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 151.8, 151.2, 148.6, 138.6, 138.2, 136.7, 130.0, 129.7, 127.4, 124.2, 123.3, 119.1, 117.4, 108.8, 106.2, 101.6, 50.9, 32.6, 25.9, 24.7, 21.3.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

(7-(Benzylamino)-9-(*p*-tolyl)-6*H*-benzo[*c*]chromen-6-one (4l)



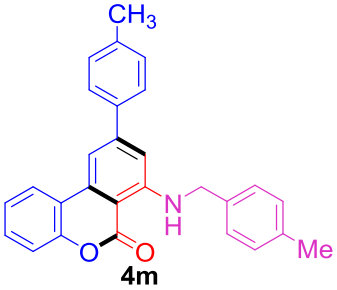
4l

Isolated yield = 76%, 297 mg, yellow solid; mp: 157-161°C

^{1}H NMR (600 MHz, $CDCl_3$): δ 9.08 (t, *J* = 5.4 Hz, 1H), 8.06 (dd, *J* = 7.8 1.2 Hz, 1H), 7.46-7.43 (m, 4H), 7.41-7.39 (m, 2H), 7.37-7.34 (m, 2H), 7.32-7.31 (m, 1H), 7.29-7.25 (m, 4H), 4.59 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.6, 152.4, 151.2, 148.6, 138.7, 138.3, 137.8, 136.6, 130.2, 129.7, 128.9, 127.4, 127.2, 124.4, 123.4, 118.9, 117.4, 109.0, 107.1, 102.3, 47.3, 21.3.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

7-((4-methylbenzyl)amino)-9-(*p*-tolyl)-6*H*-benzo[*c*]chromen-6-one (4m)



4m

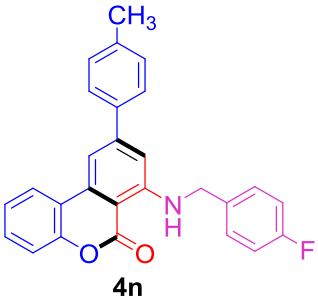
Isolated yield = 73%, 295 mg, yellow solid; mp: 158-161°C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 9.03 (t, *J* = 5.4 Hz, 1H), 8.06 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.47-7.4 (m, 4H), 7.33-7.25 (m, 6H), 7.16-7.15 (m, 2H), 6.84 (s, 1H), 4.54 (d, *J* = 6.0 Hz, 2H), 2.04 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 152.4, 151.3, 148.6, 138.7, 137.8, 137.0, 136.6, 135.2, 130.1, 129.7, 129.5,

127.2, 124.3, 123.3, 118.9, 117.4, 108.9, 106.9, 102.2, 47.0, 21.2.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

7-((4-fluorobenzyl)amino)-9-(*p*-tolyl)-6*H*-benzo[*c*]chromen-6-one (4n)

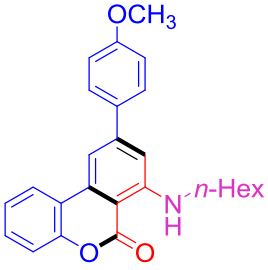


4n

Isolated yield = 71%, 290 mg, yellow solid; mp: 152-156 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 9.05 (t, *J* = 6.0 Hz, 1H), 8.06 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.48-7.43 (m, 4H), 7.38-7.35 (m, 2H), 7.33-7.25 (m, 4H), 7.05-7.02 (m, 2H), 6.78 (d, *J* = 1.8 Hz, 1H), 4.55 (d, *J* = 6.00 Hz, 2H), 2.41 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 163.0, 161.4, 152.2, 151.2, 148.7, 138.8, 137.7, 136.7, 133.9, 130.2, 129.7, 128.7, 127.3, 124.4, 123.4, 118.9, 117.4, 115.8, 115.6, 108.9, 107.3, 102.4, 46.6, 21.3. HRMS (ESI): calculated for m/z 409.1478; ($[M+H]^+$); found. m/z 410.1547.

(7-(Hexylamino)-9-(4-methoxyphenyl)-6*H*-benzo[*c*]chromen-6-one (4o)



4o

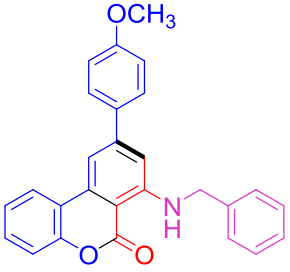
Isolated yield = 88%, 352 mg, yellow solid; mp: 81-85 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.58 (t, *J* = 6.0 Hz, 1H), 8.06 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.64-7.63 (m, 2H), 7.44-7.40 (m, 2H), 7.37-7.26 (m, 2H), 7.03-7.02 (m, 2H), 6.81 (d, *J* = 1.2 Hz, 1H), 3.88 (s, 3H), 3.31 (q, *J* = 7.2, 1.8 Hz, 2H), 1.77 (quin, *J* = 7.2 Hz, 2H), 1.49-1.46 (m, 2H), 1.37-1.32 (m, 4H), 0.90 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 160.2, 152.6, 151.3, 148.3, 136.6, 133.3, 130.1, 128.6, 124.2, 123.3, 119.0, 117.4, 114.4, 107.9, 106.1, 101.5, 55.5, 43.2, 31.7, 29.0, 27.0, 22.7, 14.1.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

7-(benzylamino)-9-(4-methoxyphenyl)-6*H*-benzo[*c*]chromen-6-one (4p)



4p

Isolated yield = 78%, 317 mg, yellow solid; mp: 152-155 °C.

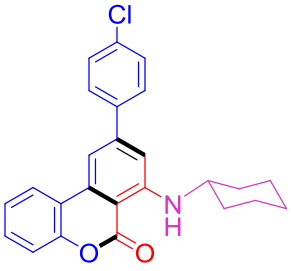
^{1}H NMR (600 MHz, $CDCl_3$): δ 9.07 (t, *J* = 6.0 Hz, 1H), 8.06 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.50- 7.47 (m, 2H), 7.45-7.40 (m, 4H), 7.37-7.34 (m, 2H), 7.32-7.26 (m, 3H), 6.98-6.96 (m, 2H), 6.79 (s, 1H), 4.59 (d, *J* = 6.0 Hz, 2H), 3.85 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 163.4, 160.2, 152.4, 151.3, 148.2, 138.3, 136.6, 133.0, 130.2, 128.9, 128.6, 127.4, 127.2, 124.3, 123.3,

118.9, 117.4, 114.4, 108.7, 106.8, 102.0, 55.5, 47.3.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

9-(4-Chlorophenyl)-7-(cyclohexylamino)-6*H*-benzo[*c*]chromen-6-one (4q)



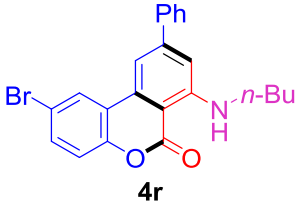
4q

Isolated yield = 80%, 322 mg, yellow solid; mp: 135-138 °C

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.72 (d, *J* = 7.2 Hz, 1H), 8.03 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.59-7.56 (m, 2H), 7.47-7.42 (m, 3H), 7.32-7.26 (m, 3H), 6.80 (s, 1H), 3.54-3.51 (m, 1H), 2.09-2.03 (m, 2H), 1.83-1.79 (m, 2H), 1.65-1.63 (m, 1H), 1.48-1.39 (m, 4H), 1.36-1.31 (m, 1H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.3, 151.7, 151.2, 147.4, 139.5, 137.0, 134.7, 130.2, 129.2, 128.8, 124.3, 123.3, 118.8, 117.4, 108.6, 106.0, 102.0, 50.9, 32.6, 25.9, 24.7.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

9-(4-bromophenyl)-7-(butylamino)-6*H*-benzo[*c*]chromen-6-one (4r)



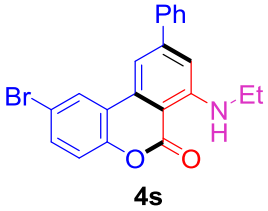
4r

Isolated yield = 89%, 374 mg, yellow solid; mp: 177-181 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.56 (t, *J* = 6.0 Hz, 1H), 8.14 (d, *J* = 1.8 Hz, 1H), 7.67-7.65 (m, 2H), 7.52-7.49 (m, 2H), 7.46-7.44 (m, 1H), 7.32 (d, *J* = 1.8 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 1H), 3.31 (q, *J* = 6.6 Hz, 2H), 1.75 (quin, *J* = 7.2 Hz, 2H), 1.51 (sextet, *J* = 7.2 Hz, 2H), 0.98 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 162.7, 152.6, 149.9, 148.8, 140.6, 135.2, 132.8, 129.0, 128.7, 127.5, 126.1, 120.8, 119.1, 117.2, 109.1, 106.6, 101.4, 42.9, 31.1, 20.4, 13.9. HRMS (ESI): calculated for m/z 421.0677 ($[M+H]^+$); found. m/z 422.0748, 424.0941.

2-bromo-7-(ethylamino)-9-phenyl-6*H*-benzo[*c*]chromen-6-one (4s)



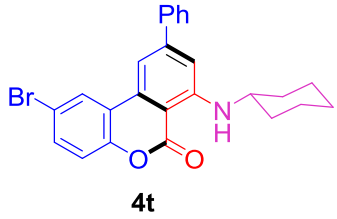
4s

Isolated yield = 81%, 318 mg, yellow solid; mp: 112-115 °C.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.49-8.48 (m, 1H), 8.14 (d, *J* = 1.8 Hz, 1H), 7.67-7.66 (m, 2H), 7.52-7.49 (m, 3H), 7.46-7.45 (m, 1H), 7.32 (d, *J* = 1.8 Hz, 1H), 7.18 (d, *J* = 7.8 Hz, 1H), 6.87 (s, 1H), 3.39-3.34 (m, 2H), 1.39 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 162.7, 152.5, 150.1, 148.9, 140.6, 135.2, 132.8, 129.0, 128.8, 127.5, 126.1, 120.8, 119.1, 117.2, 109.1,

106.6, 101.4, 37.8, 14.4. HRMS (ESI): calculated for m/z 393.0364 ([M+H]$^+$); found. m/z 394.0406; 396.0601

(2-Bromo-7-(cyclohexylamino)-9-phenyl-6*H*-benzo[*c*]chromen-6-one(4t)

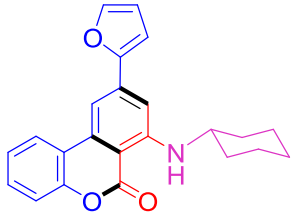


4t

Isolated yield = 77%, 344 mg, yellow solid; mp: 179-182 °C. ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.66 (d, *J* = 7.2 Hz, 1H), 8.16 (d, *J* = 2.4 Hz, 1H), 7.66-7.64 (m, 2H), 7.52-7.49 (m, 2H), 7.46-7.45 (m, 1H), 7.19 (s, 1H), 7.17 (d, *J* = 8.4 Hz, 1H), 6.89 (s, 1H), 3.55-3.54 (m, 1H), 2.10-2.08 (m, 2H), 1.82-1.80 (m, 2H), 1.66-1.64 (m, 2H), 1.45-1.41 (m, 4H), 1.36-1.32 (m, 1H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 162.8, 151.8, 150.1, 148.9, 140.8, 135.4, 132.9, 129.0, 128.7, 127.5, 126.1, 120.9, 119.1, 117.2, 109.7, 106.5, 101.5, 51.0, 32.6, 25.8, 24.7.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

(7-(Cyclohexylamino)-9-(furan-2-yl)-6*H*-benzo[*c*]chromen-6-one (4u)

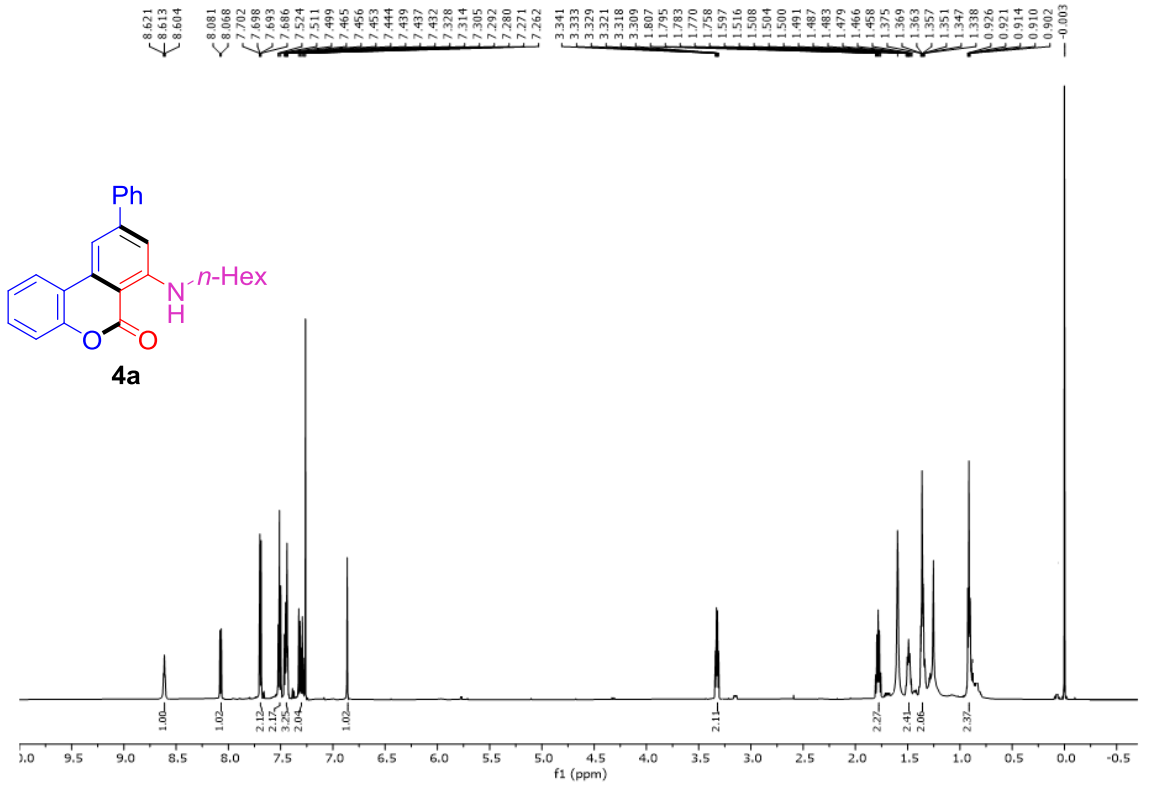


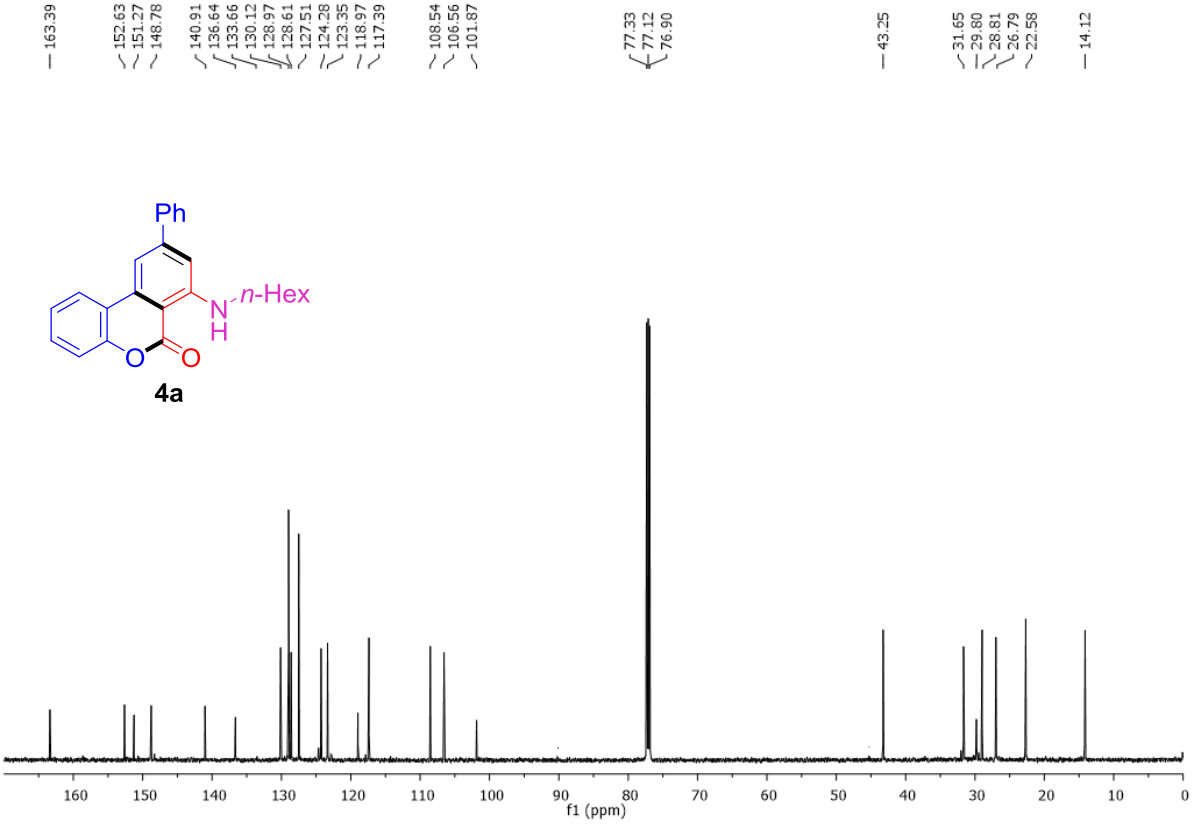
4u

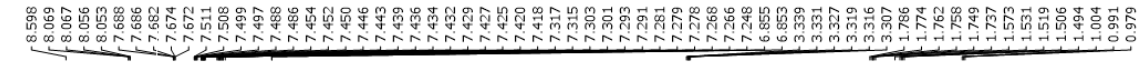
Isolated yield = 84%, 301 mg, yellow solid; mp: 162-164 °C. ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.69 (d, *J* = 7.8 Hz, 1H), 8.04 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.56 (d, *J* = 1.8 Hz, 1H), 7.48 (s, 1H), 7.43-7.40 (m, 1H), 7.29-7.26 (m, 2H), 6.99 (s, 1H), 6.87 (d, *J* = 1.2 Hz, 1H), 6.55-6.54 (m, 1H), 3.58-3.53 (m, 1H), 2.12-2.07 (m, 2H), 1.84-1.79 (m, 2H), 1.67-1.64 (m, 1H), 1.49-1.41(m, 4H), 1.36-1.32 (m, 1H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.0, 153.3, 151.7, 151.2, 143.4, 137.0, 136.9, 130.1, 124.2, 123.3, 118.8, 117.3, 112.1, 108.1, 104.7, 102.5, 101.6, 50.8, 32.6, 25.9, 24.6.

Physical and spectral properties of this compound were identical to those previously reported in literature.[2]

11. Copies of ^{1}H NMR and ^{13}C NMR Spectra



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7.486
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7.452
7.450
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7.439
7.436
7.434
7.432
7.429
7.427
7.425
7.420
7.418
7.317
7.315
7.303
7.301
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7.291
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7.278
7.268
7.266
7.248
6.855
6.853
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3.331
3.327
3.319
3.316
3.307
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1.506
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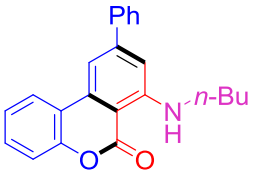
Ph
n-Bu
N
H
O
O

4b

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2.05
3.07
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1.99
2.05
2.23
3.12
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
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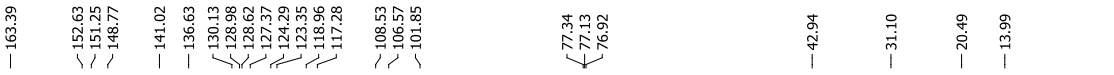
f1 (ppm)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127.37
124.29
123.35
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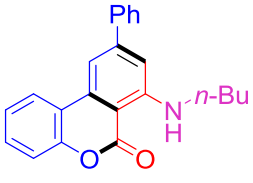
76.92
42.94
31.10
20.49
13.99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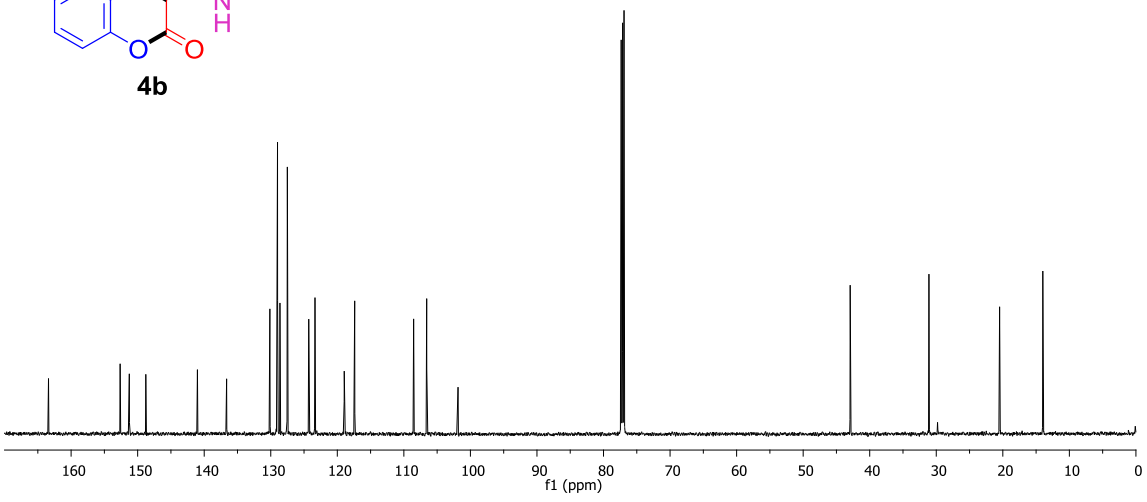
O

4b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50
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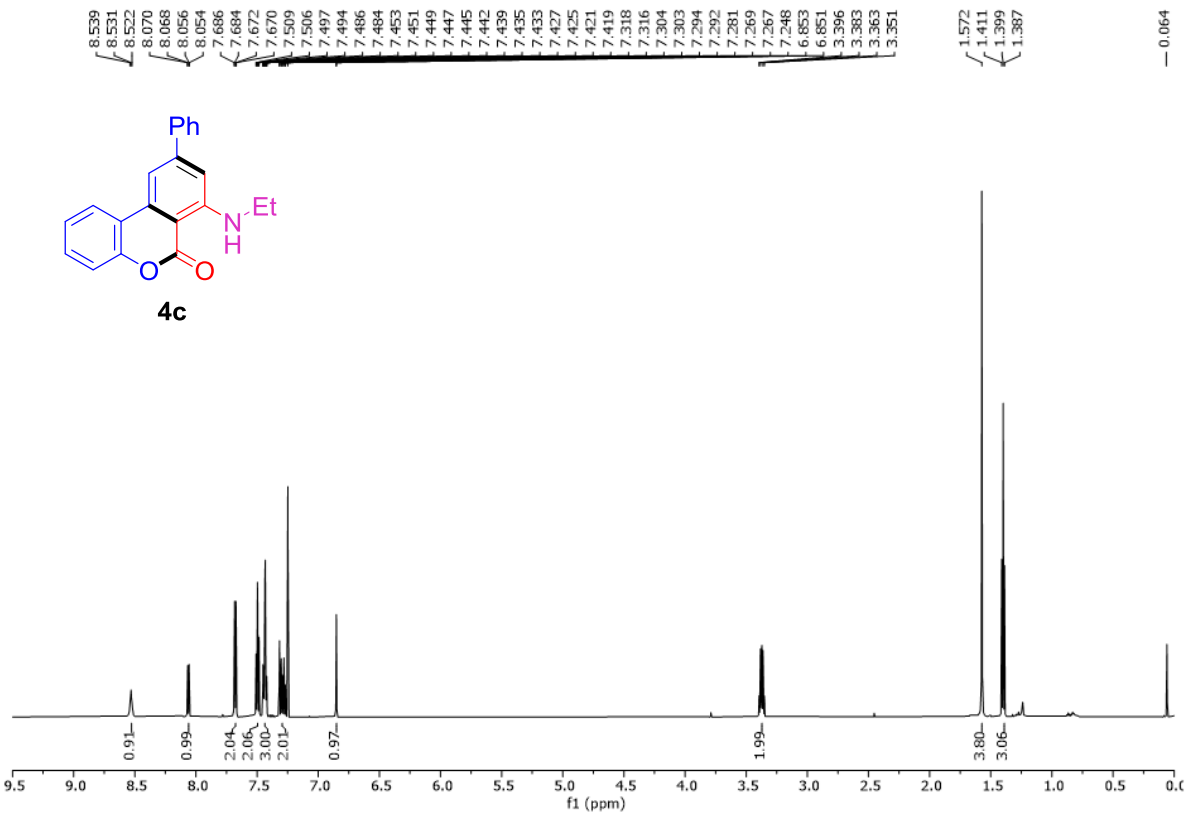
10
0
f1 (ppm)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7.506
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7.494
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7.484
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7.447
7.445
7.442
7.439
7.435
7.433
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7.425
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3.351
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O
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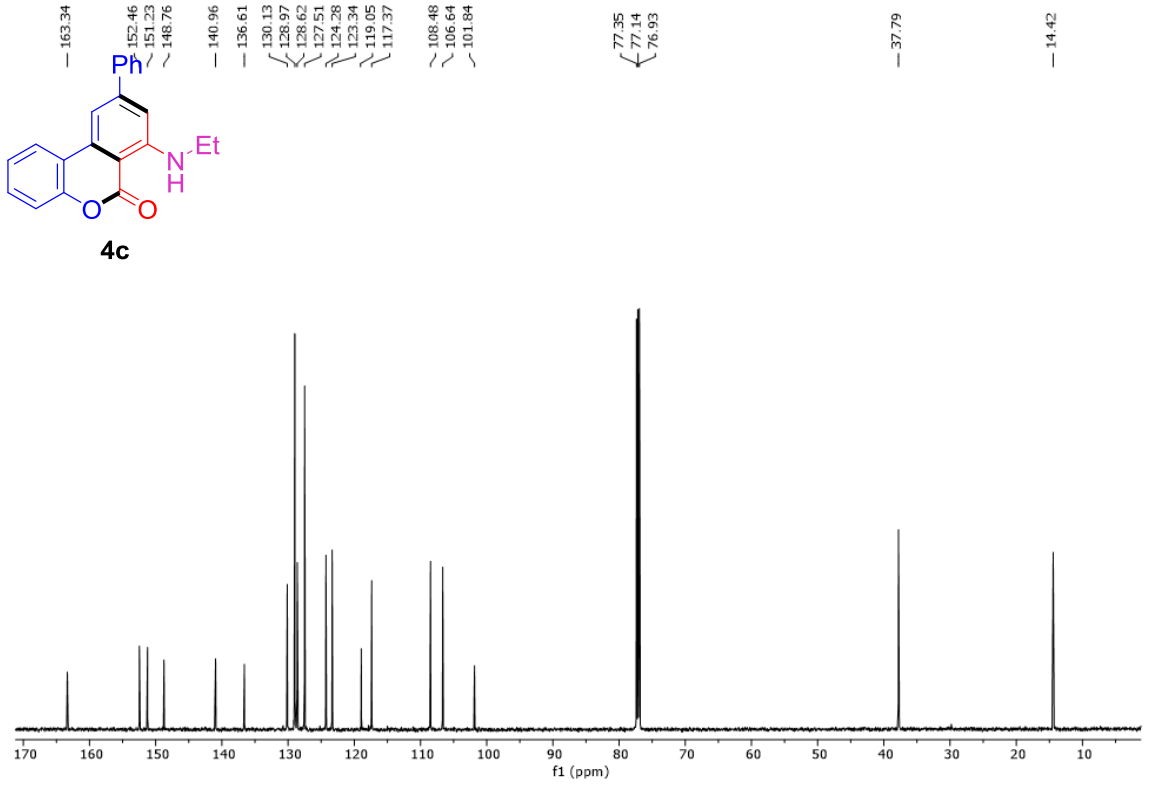
f1 (ppm)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117.37
108.48
106.64
101.84
77.35
77.14
76.93
37.79
14.42
Ph
Et
N
H
O
O
4c
f1 (ppm)


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8.058
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7.698
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7.688
7.685
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7.510
7.504
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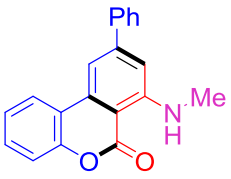
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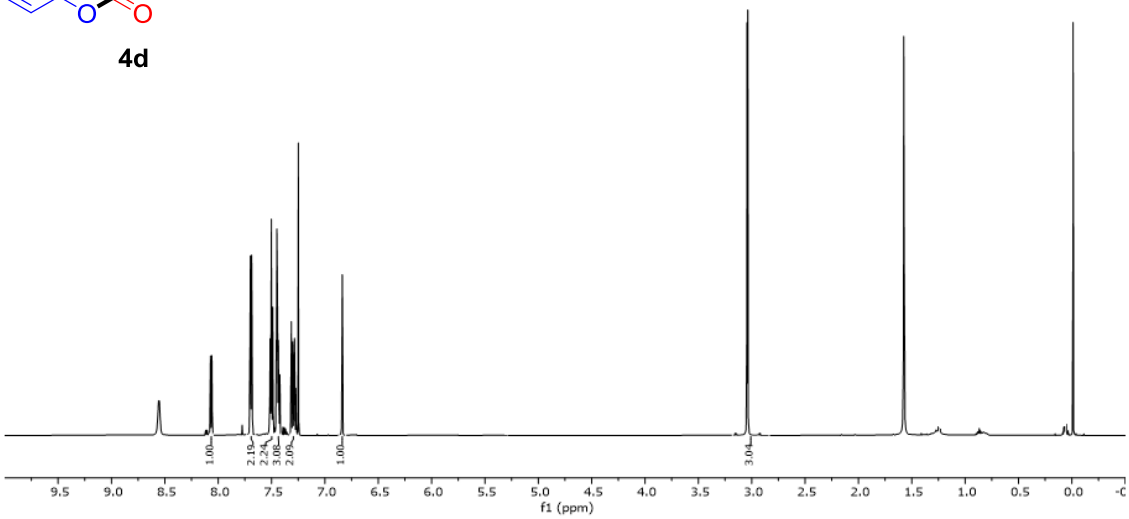
H
Me
O
O

4d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8.0
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6.5
6.0
5.5
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2.5
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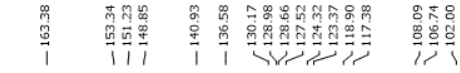
f1 (ppm)


Ph
N
H
Me
O
O

4d

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123.37
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108.09
106.74
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77.35
77.14
76.92
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60
50
40
30
20
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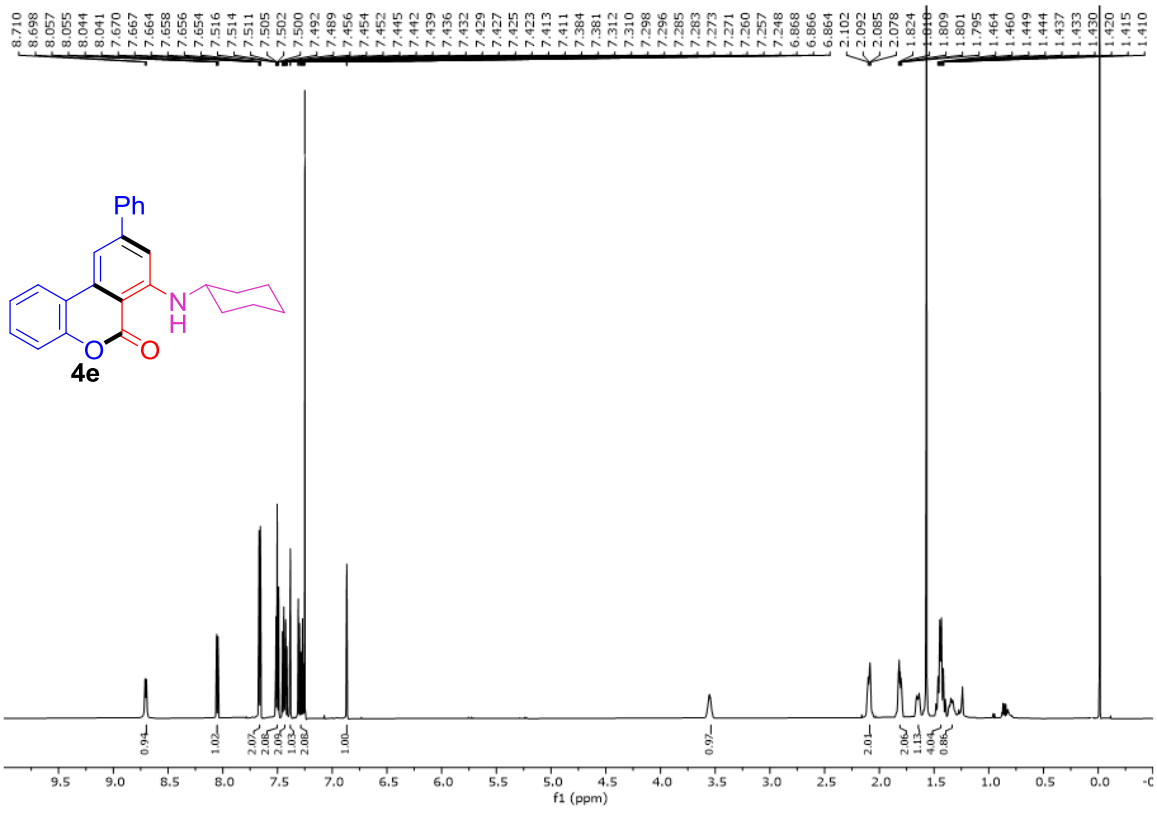
f1 (ppm)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O
O
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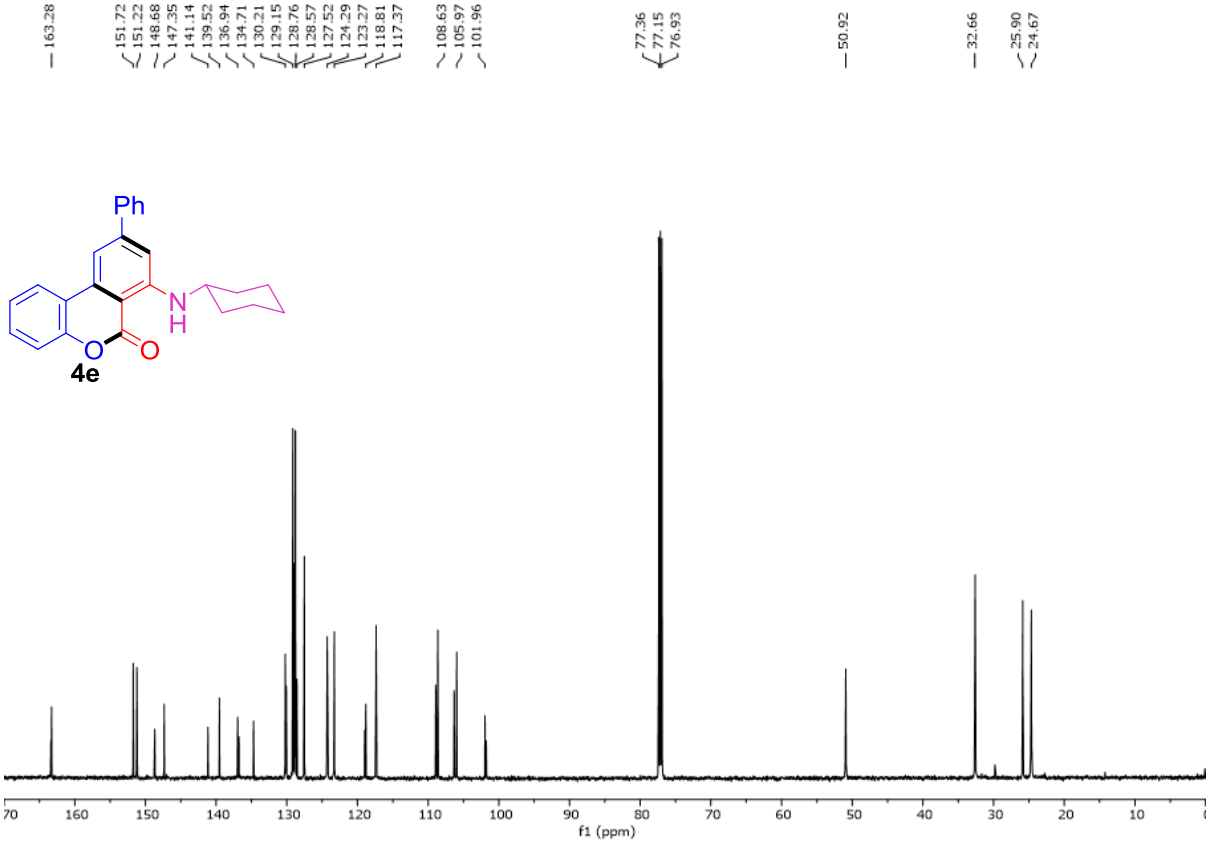
f1 (ppm)


Ph
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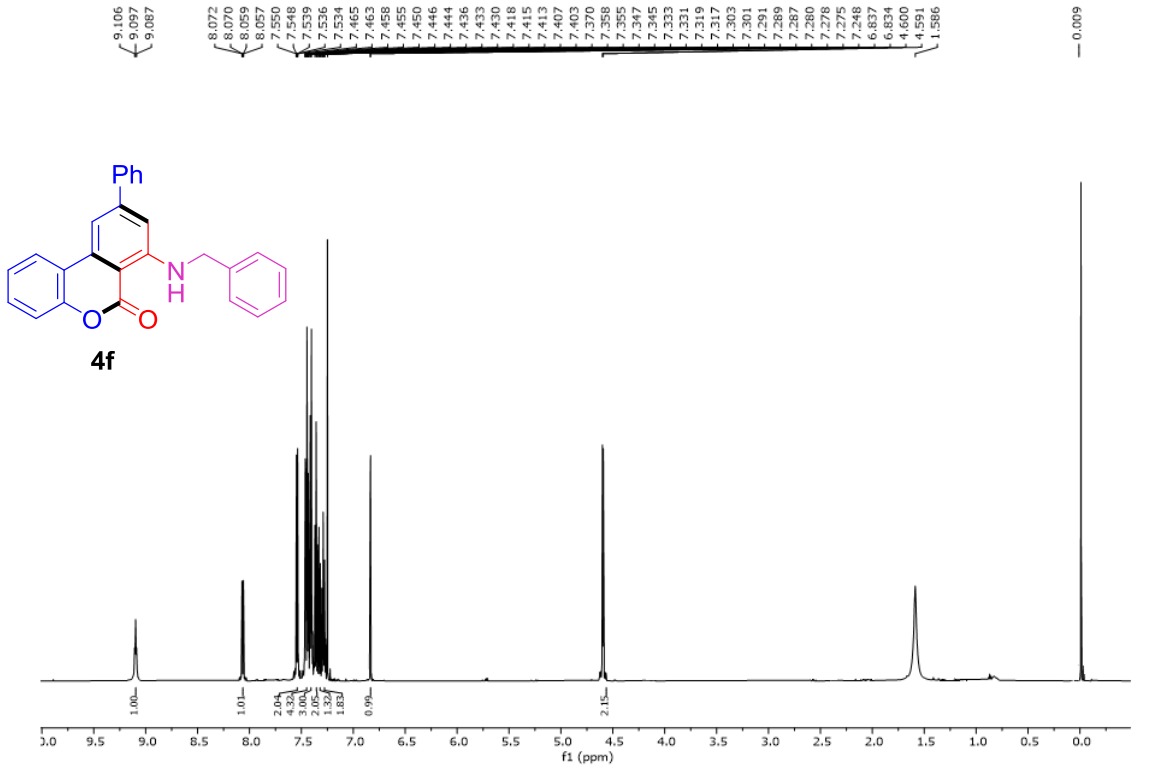
H
O
O
4e
f1 (ppm)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O
O
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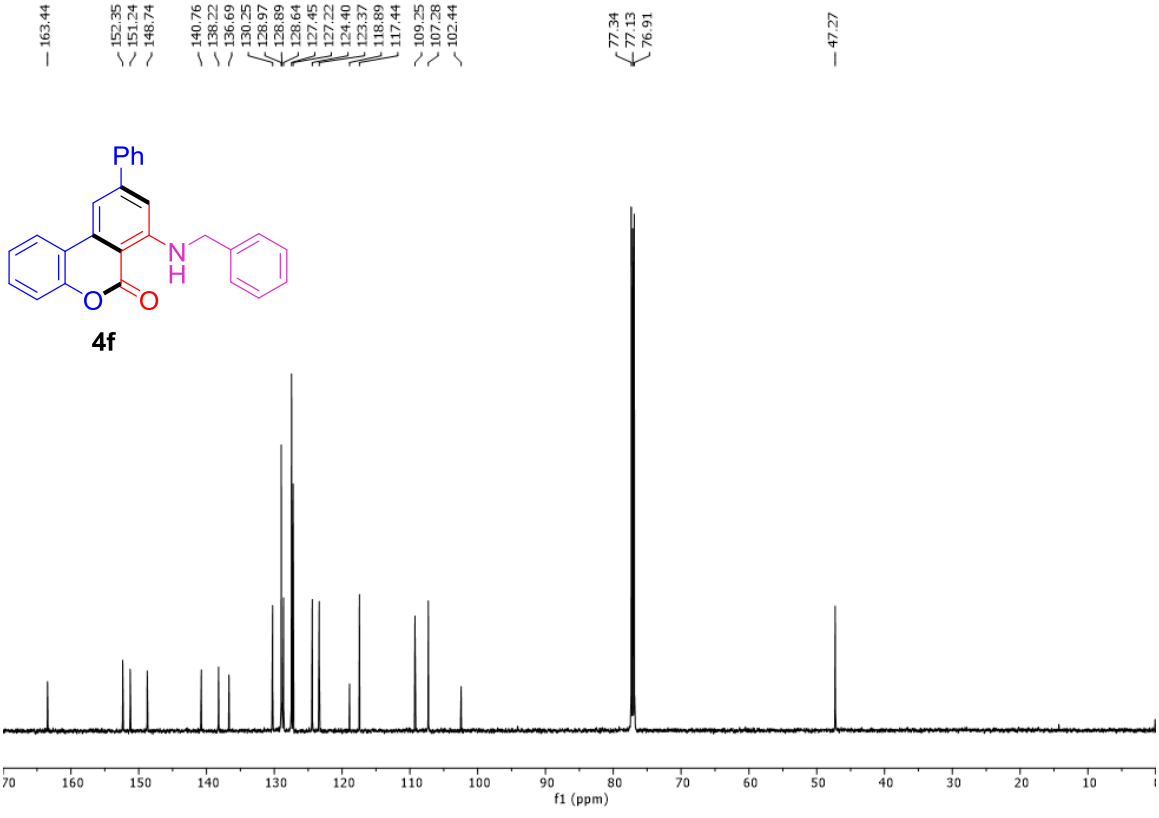
f1 (ppm)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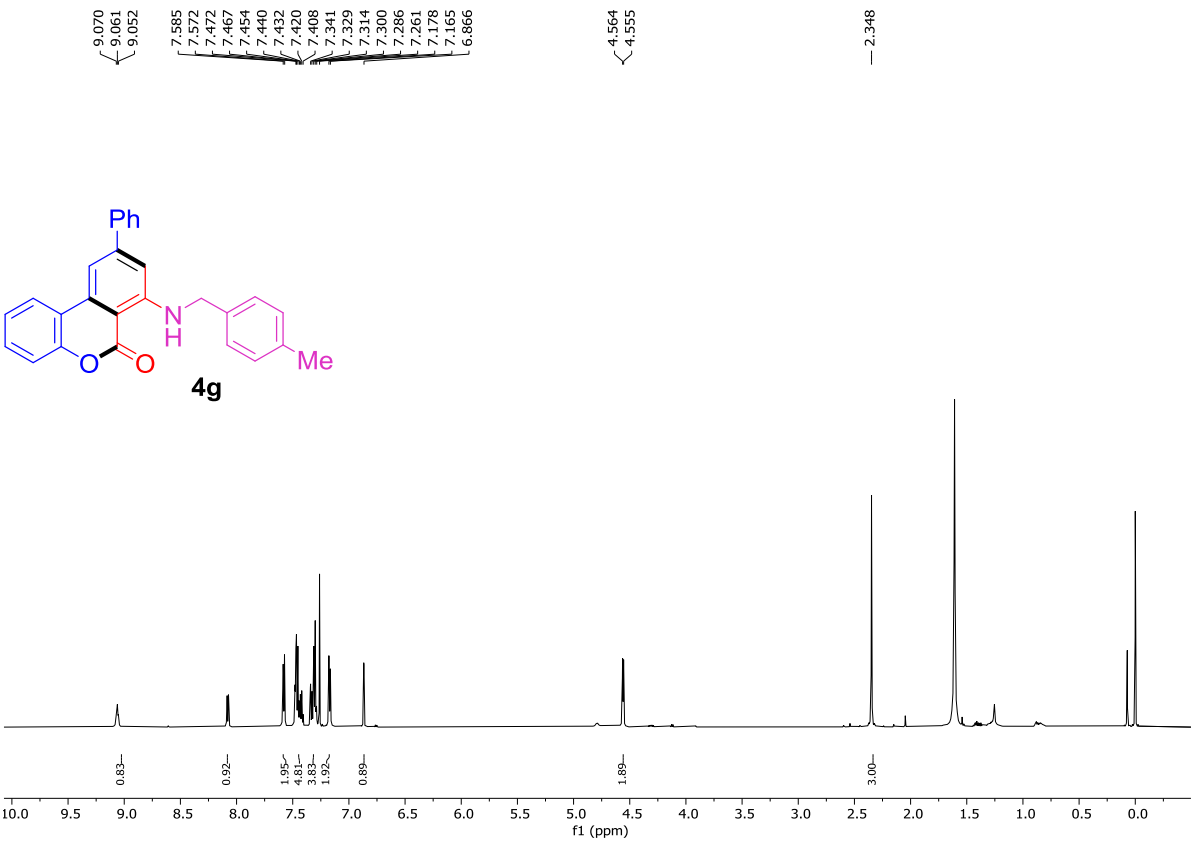
f1 (ppm)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Ph
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H
O
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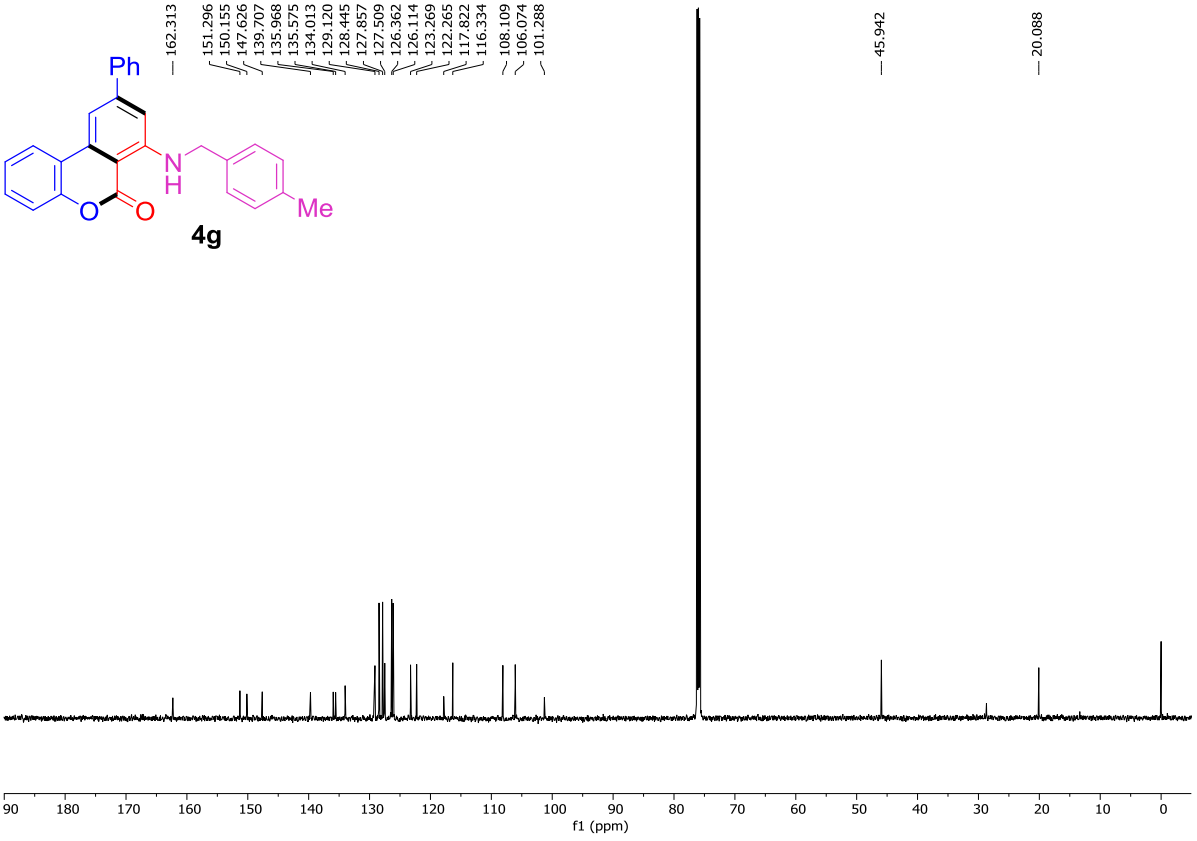
f1 (ppm)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126.362
126.114
123.269
122.265
117.822
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H
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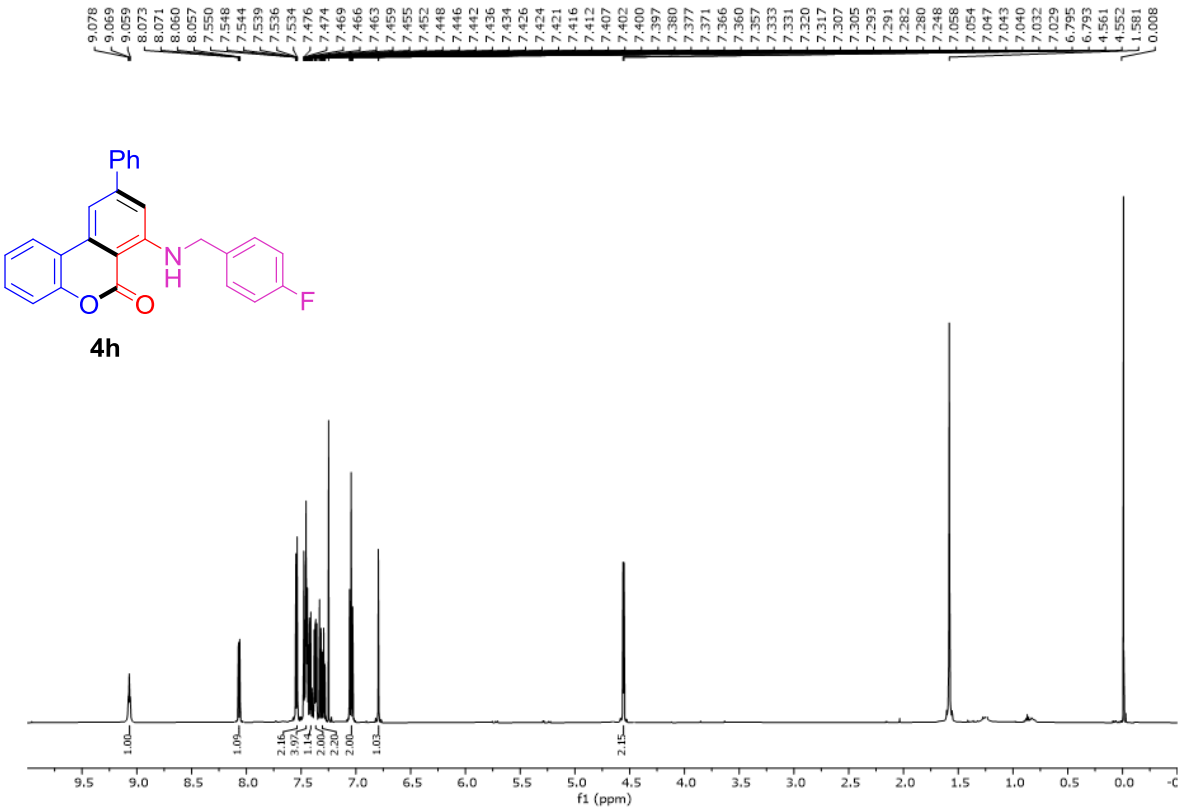
Me
4g
f1 (ppm)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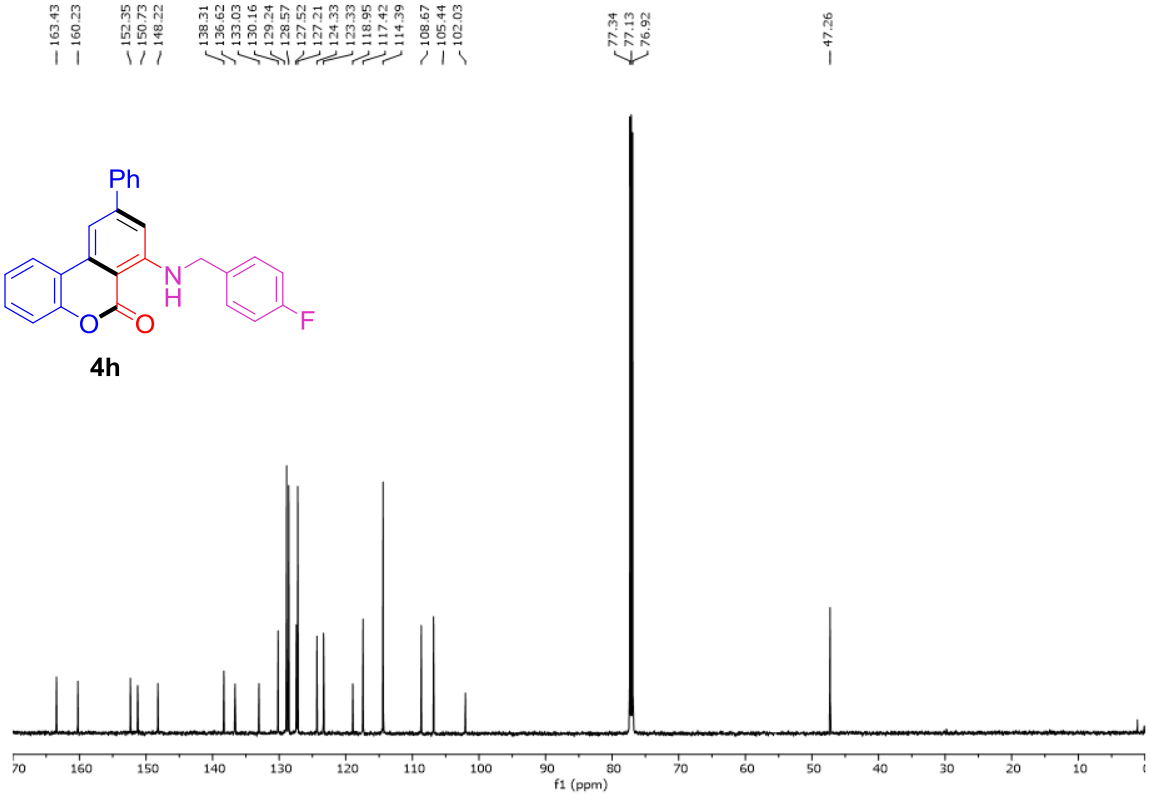
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124.33
123.33
118.95
117.42
114.39
108.67
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77.34
77.13
76.92
47.26
Ph
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H
O
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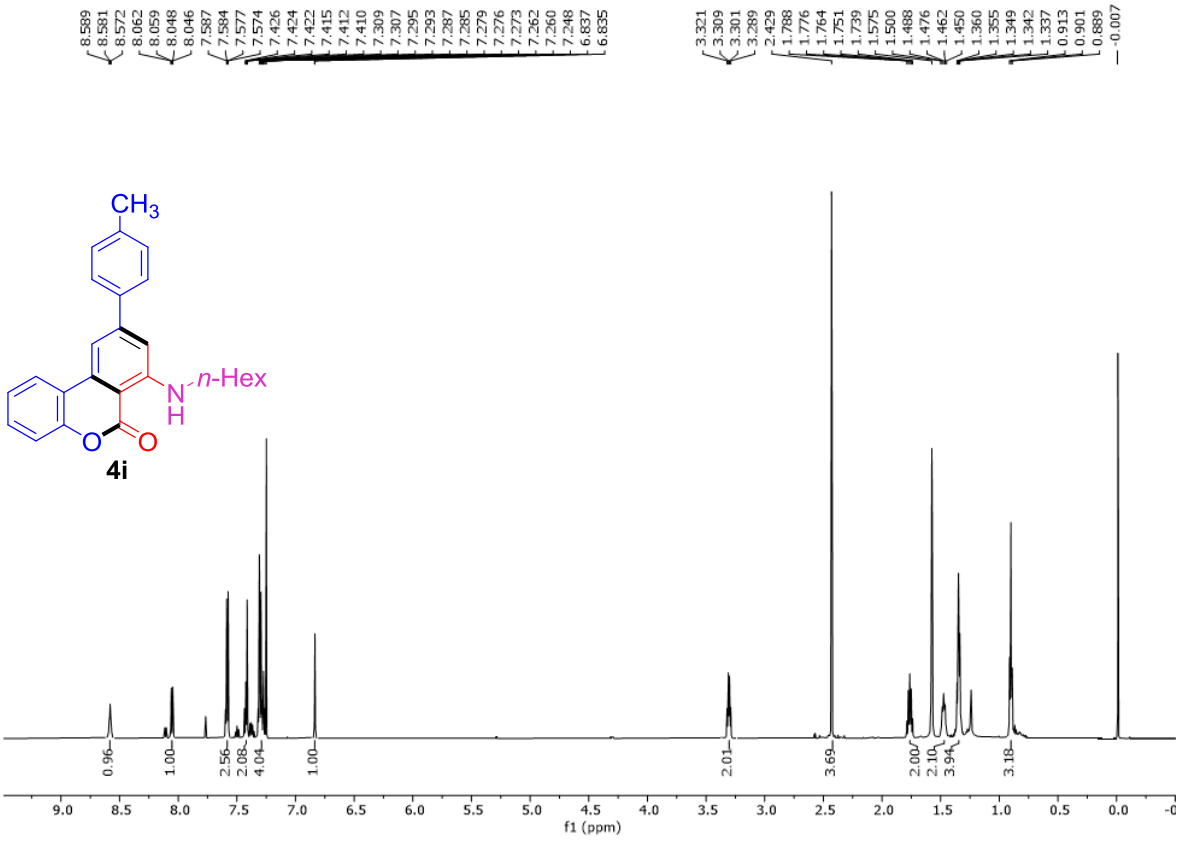
F
4h
f1 (ppm)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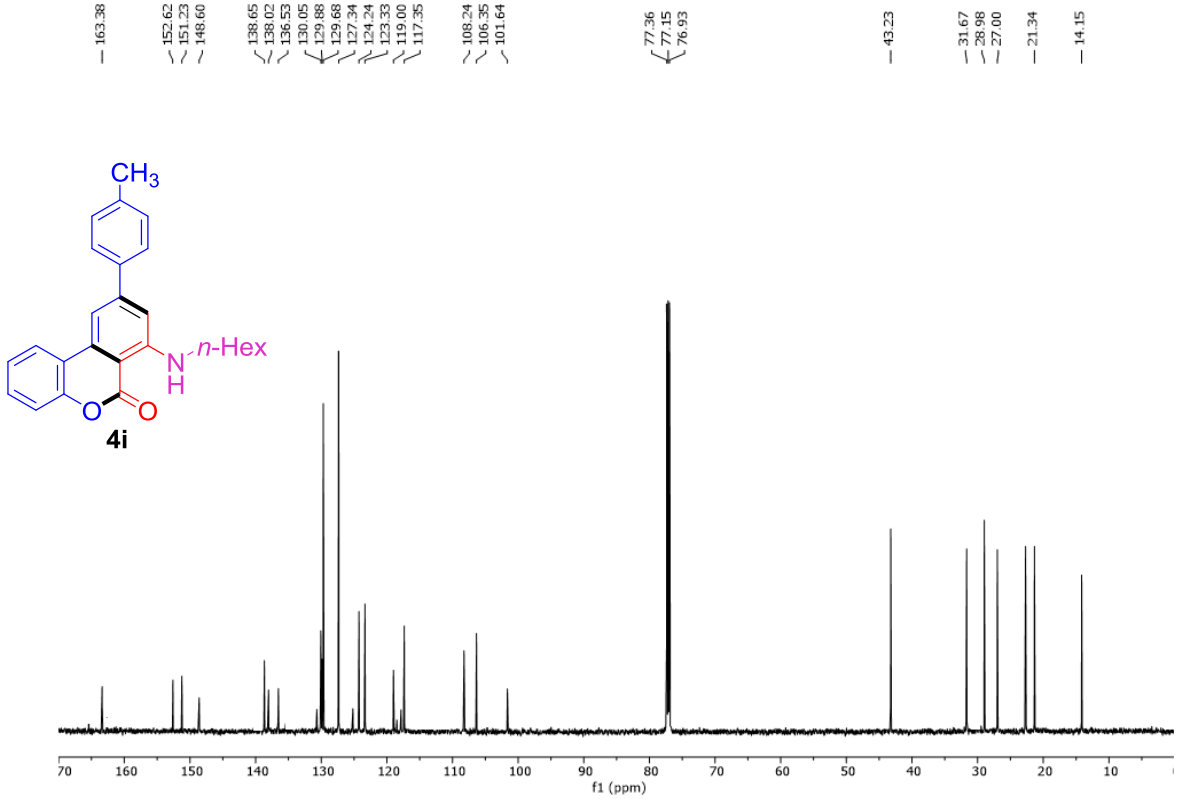
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101.64
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O
O
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20
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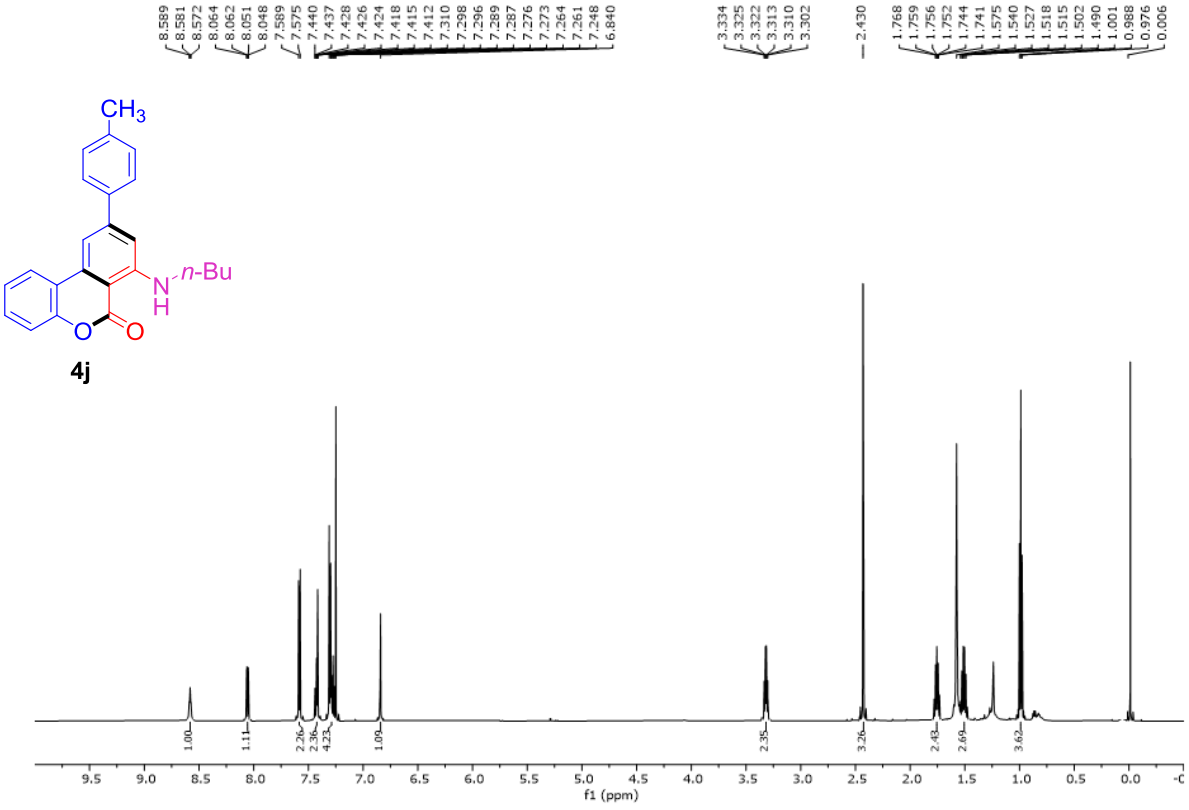
f1 (ppm)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7.273
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7.248
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3.302
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1.518
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n-Bu
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H
O
O
4j
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4.23
1.09
2.35
3.26
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3.62
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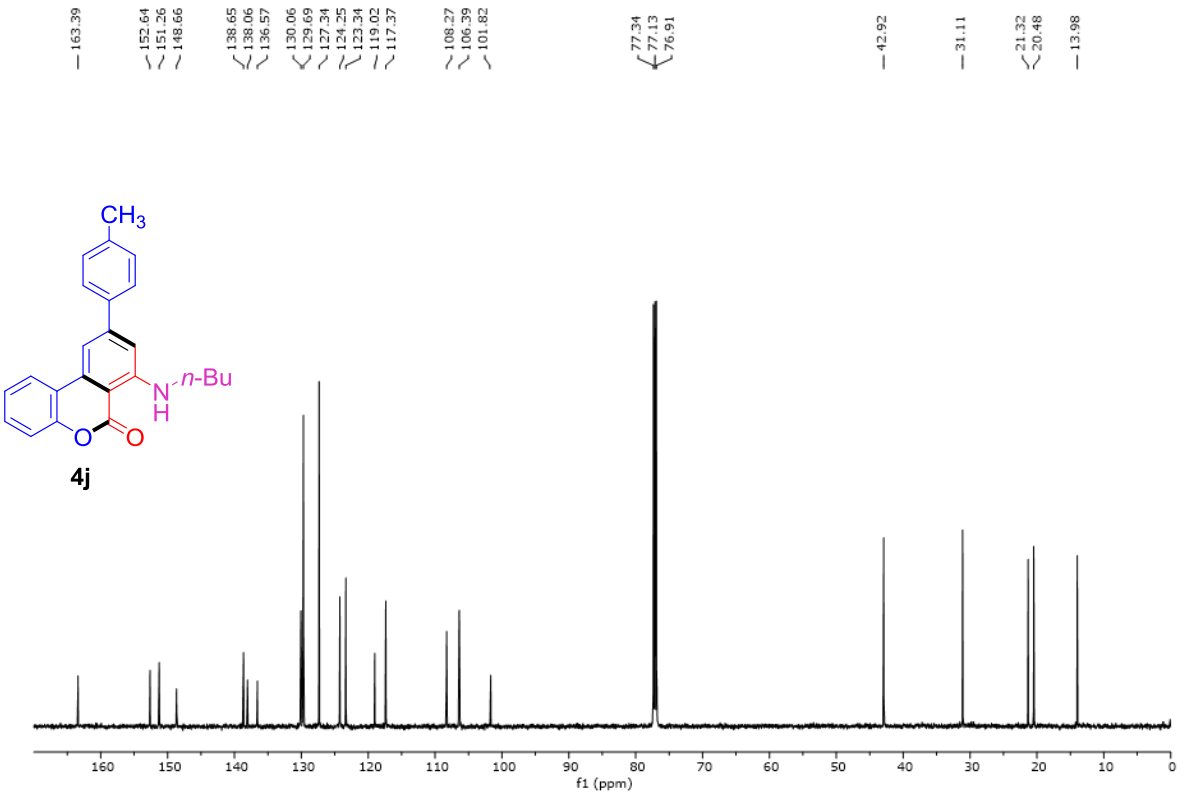
f1 (ppm)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123.34
119.02
117.37
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77.13
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21.32
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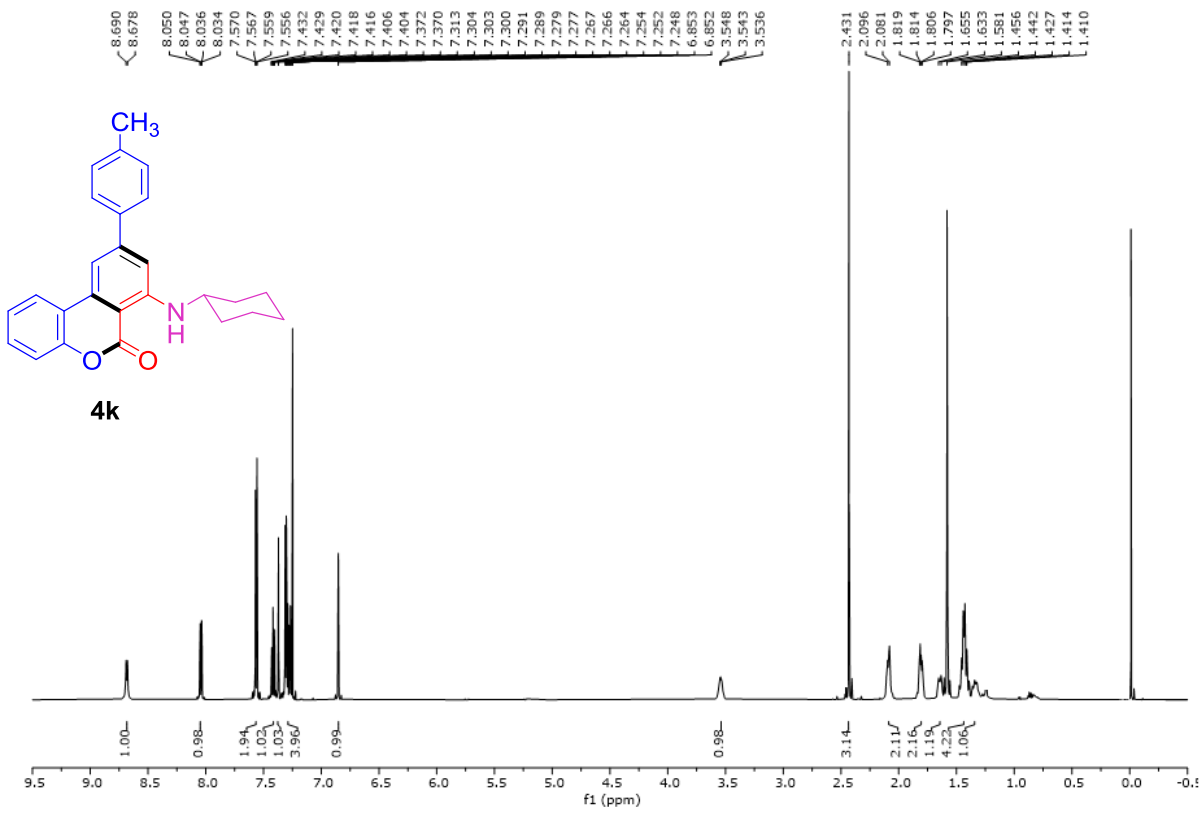
f1 (ppm)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7.252
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CH3
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H
O
O
4k
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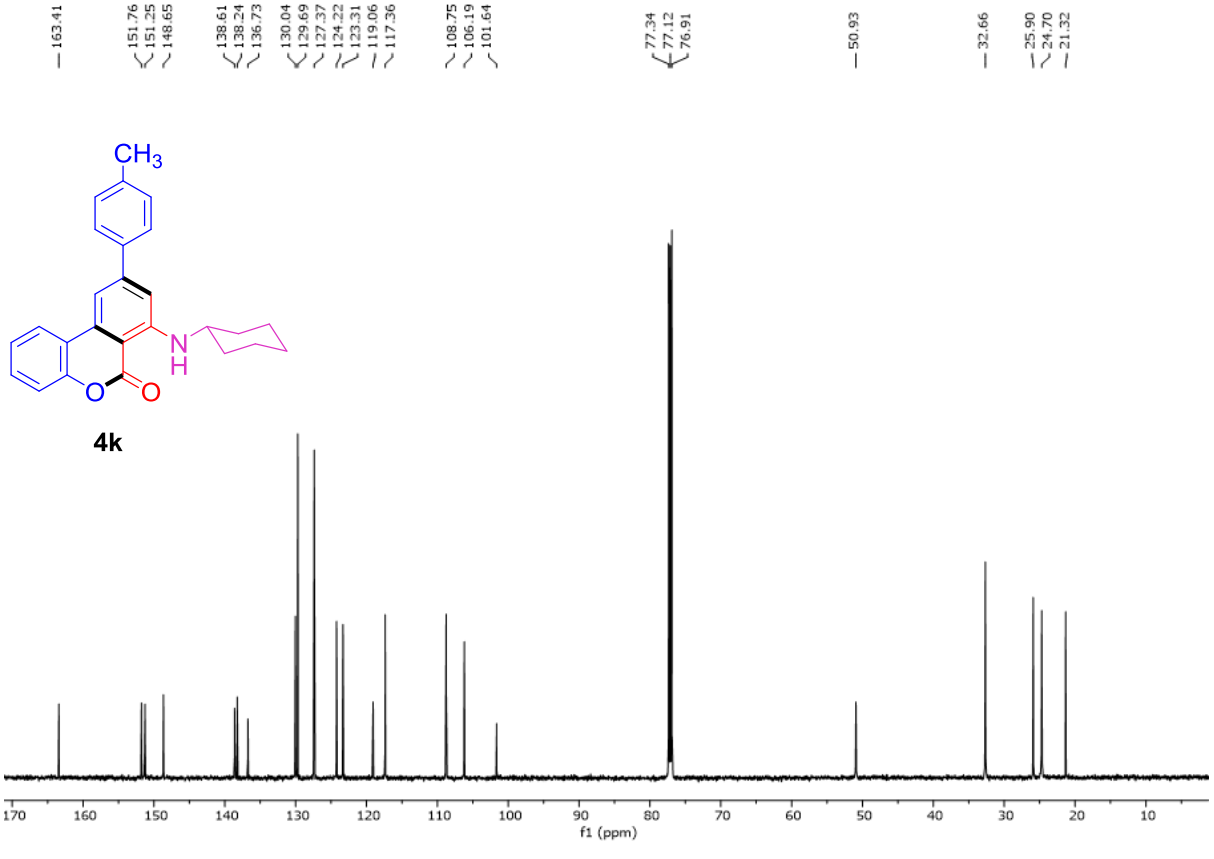
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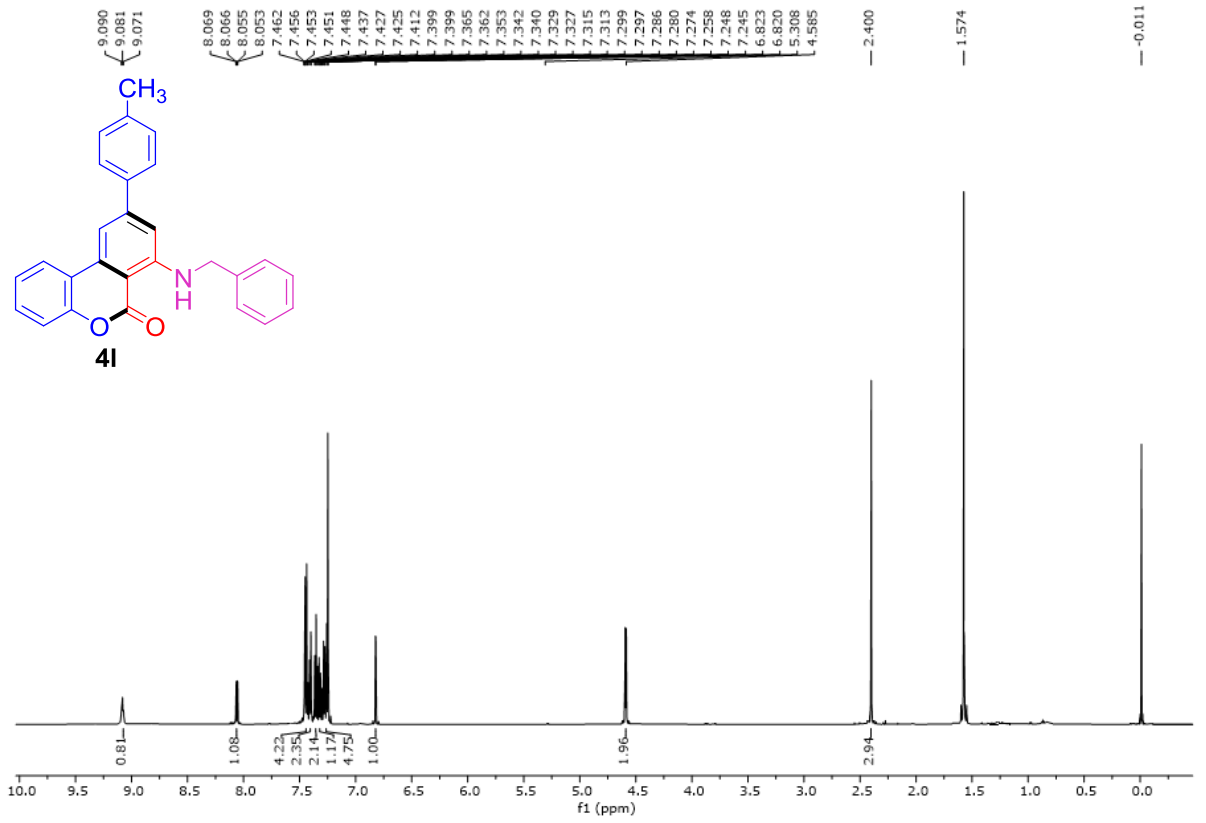
21.32
CH3
N
H
O
O
4k
f1 (ppm)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H
O
O
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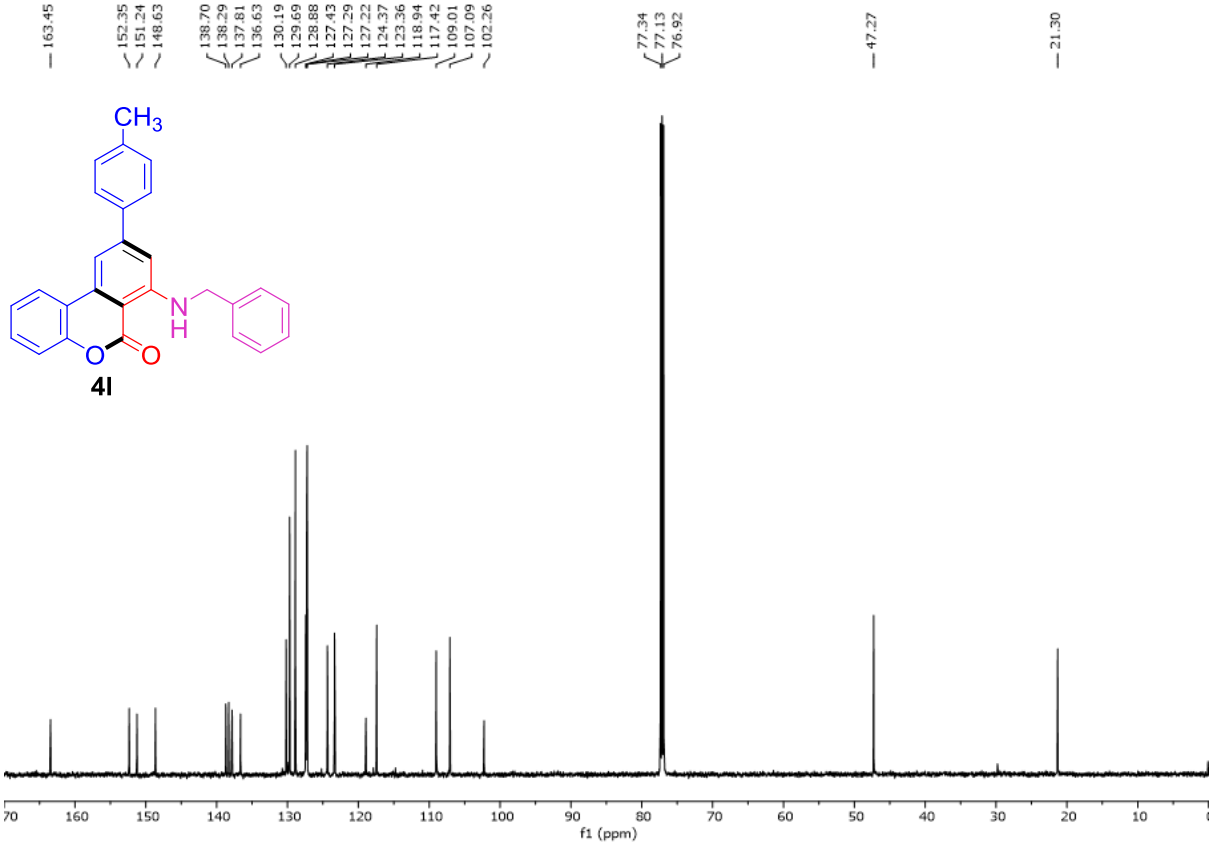
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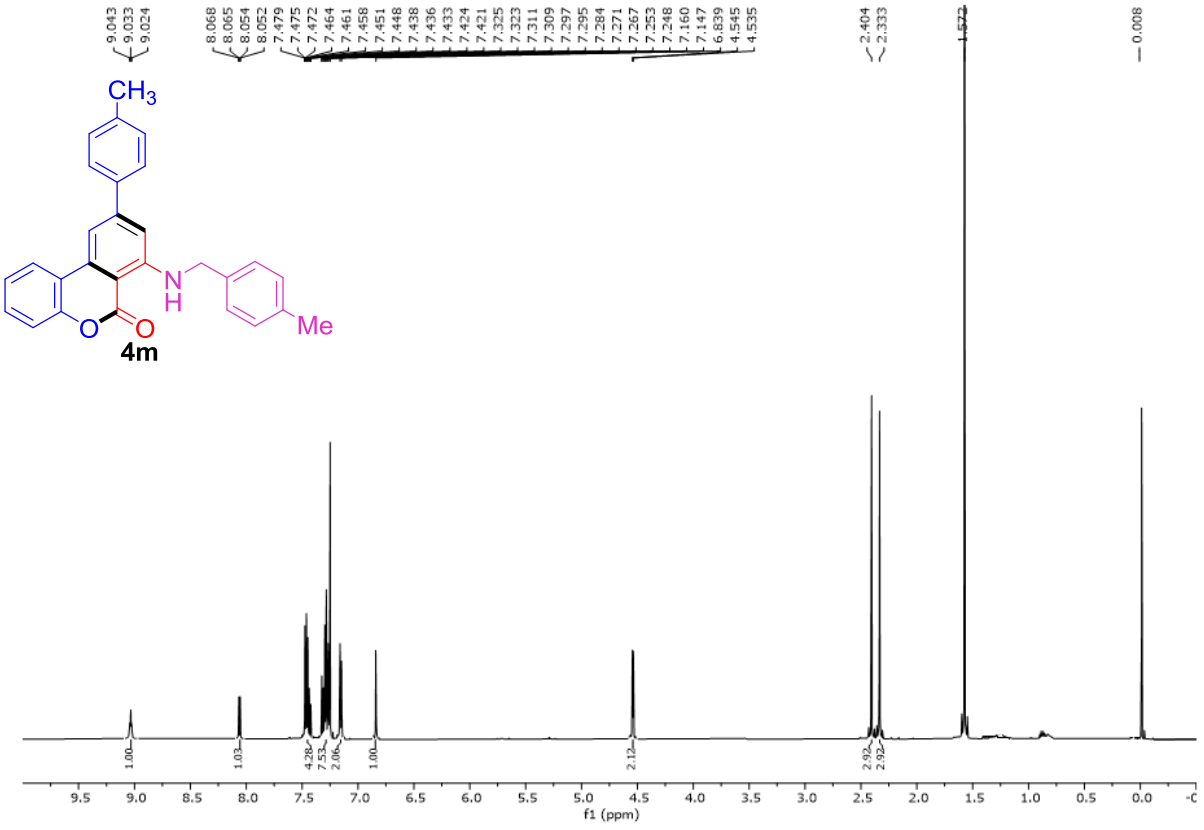
H
O
O
4l
f1 (ppm)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CH3
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O
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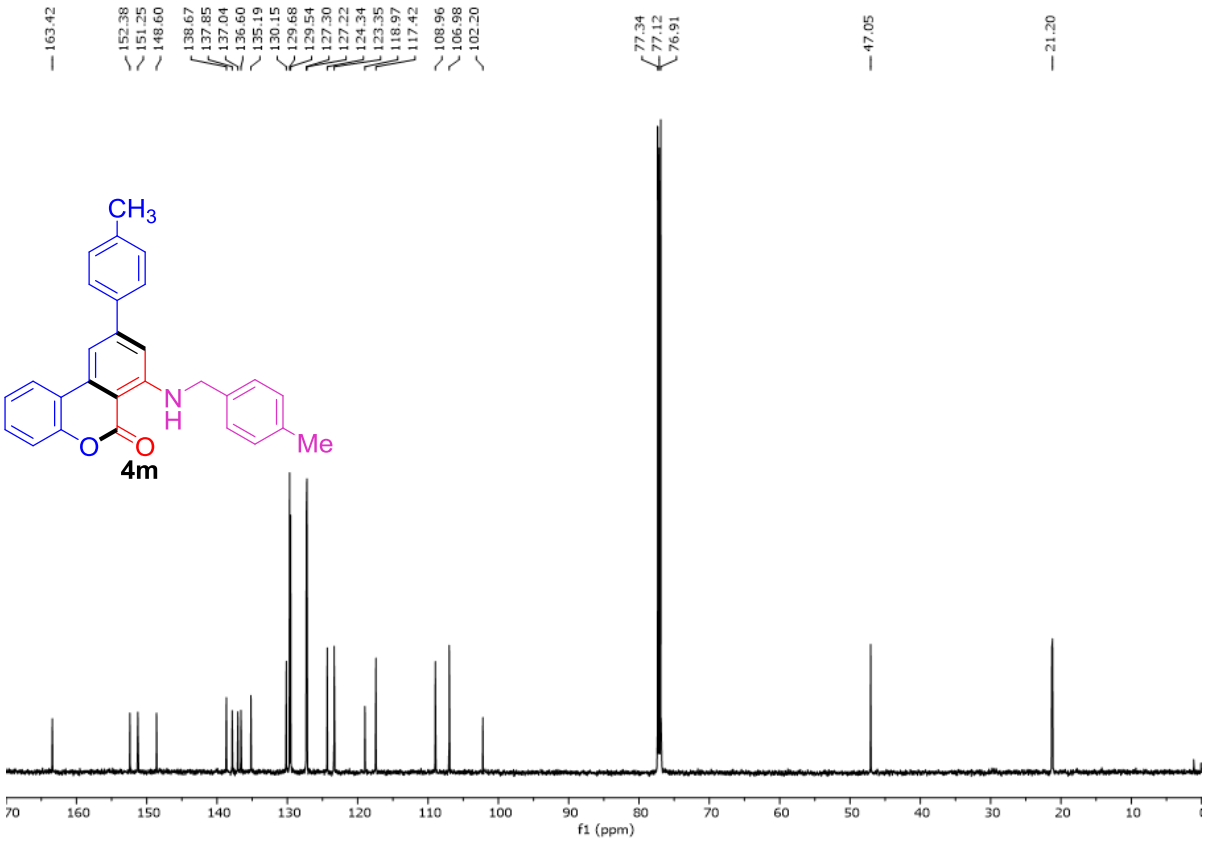
f1 (ppm)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123.35
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117.42
108.96
106.98
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76.91
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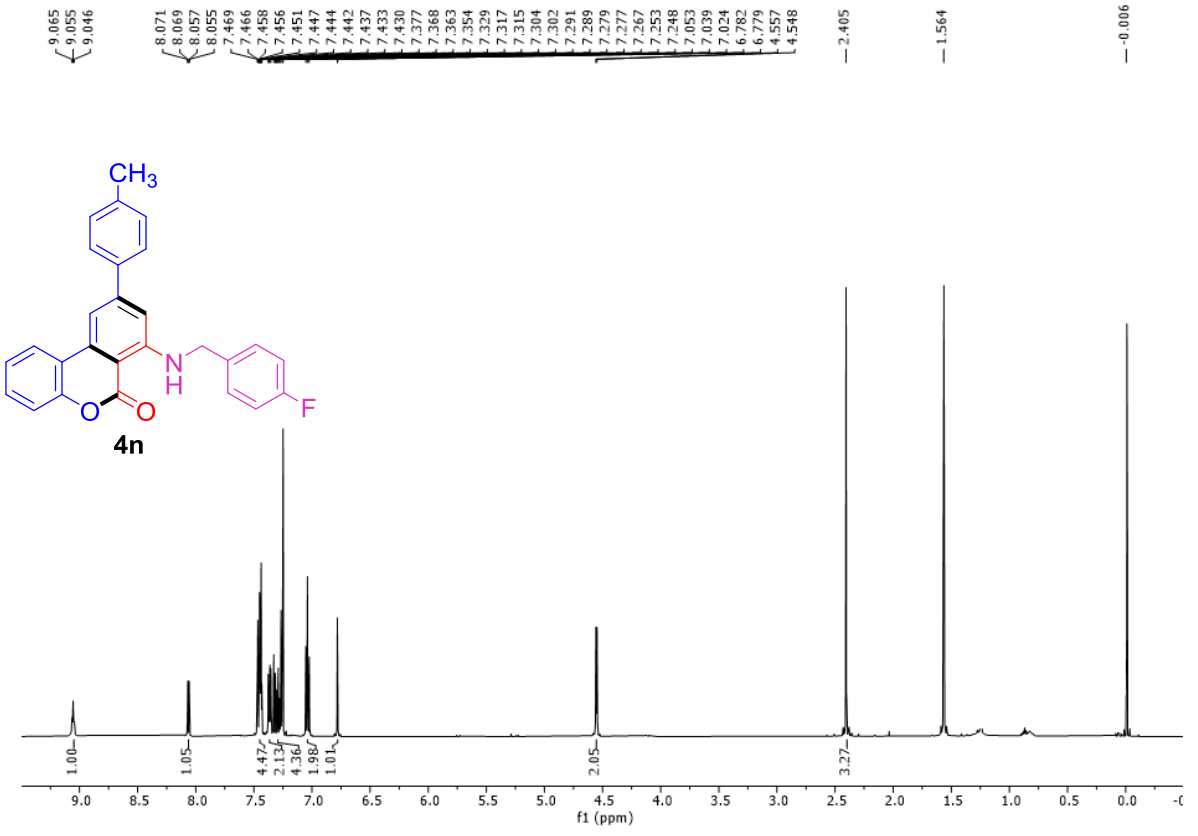
H
O
O
Me
4m
f1 (ppm)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7.442
7.437
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CH3
N
H
O
O
F
4n
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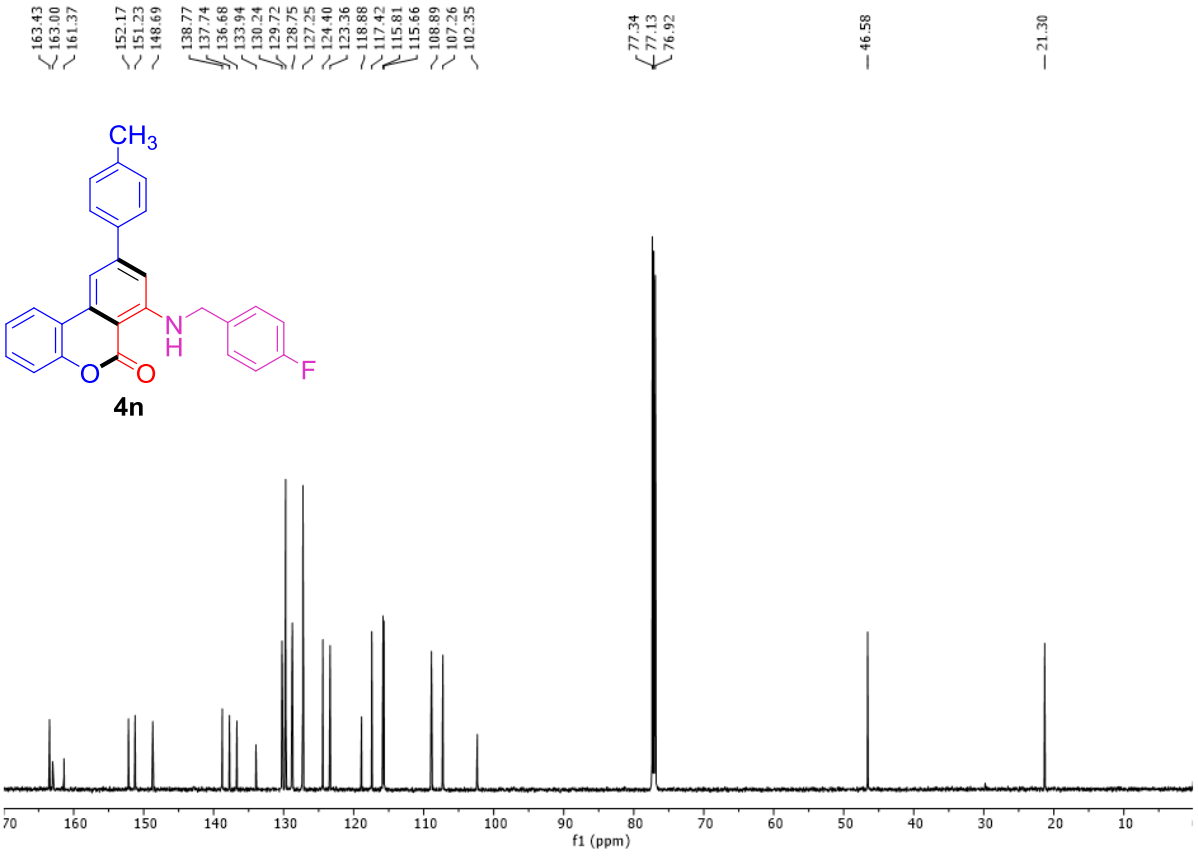
1.01
2.05
3.27
f1 (ppm)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115.66
108.89
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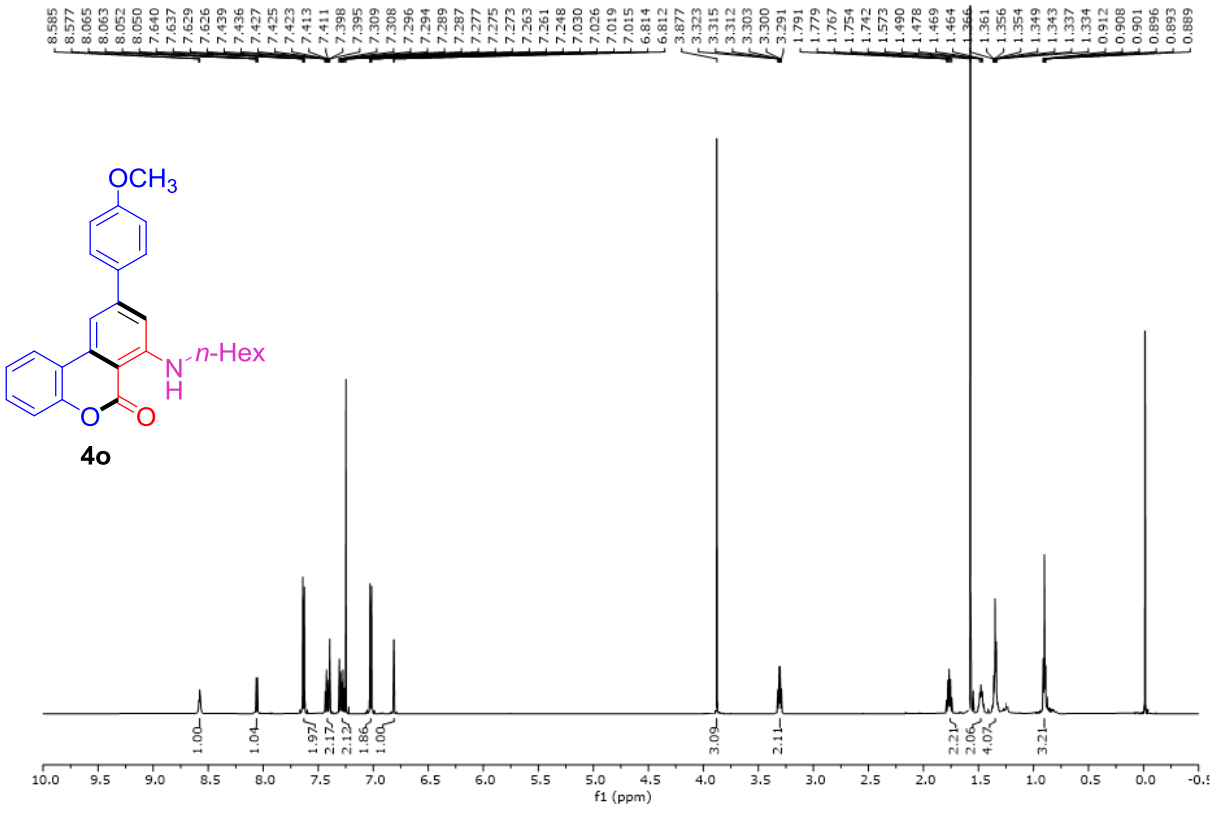
21.30
CH3
N
H
O
O
F
4n
f1 (ppm)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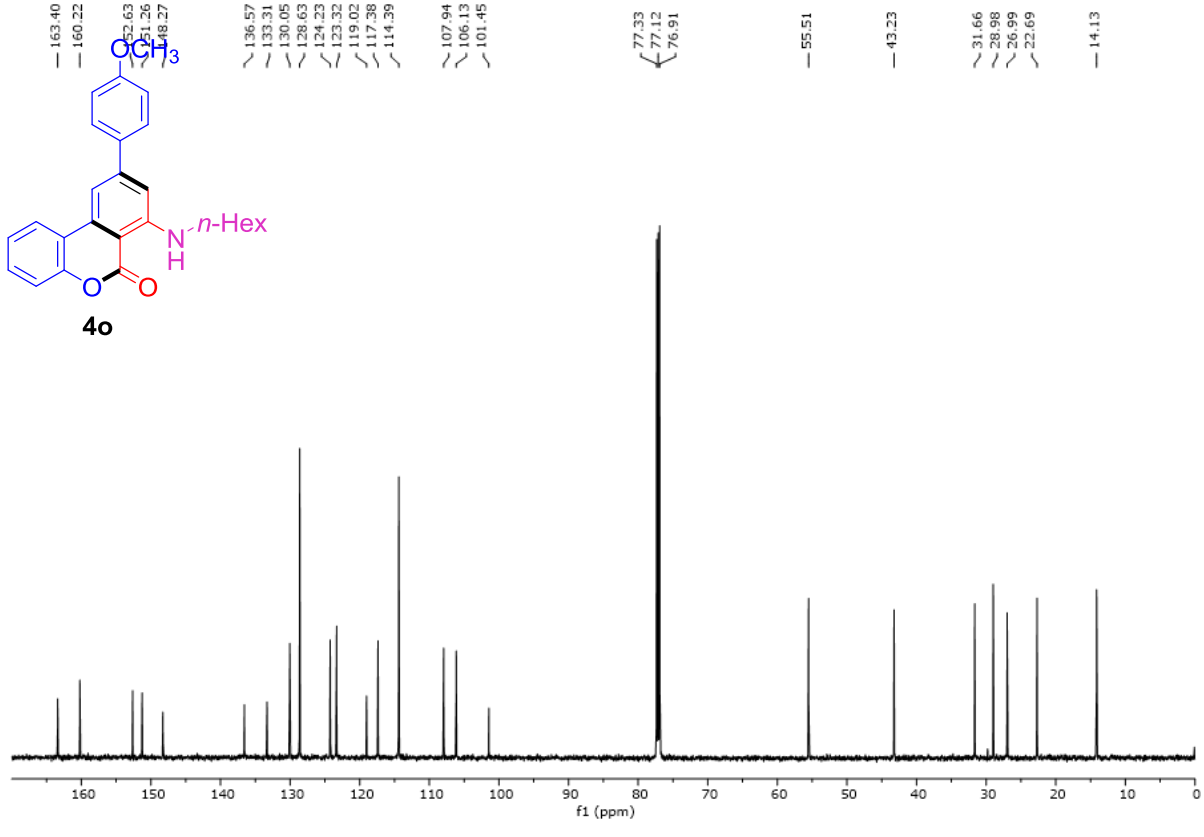
n-Hex
N
H
O
O
4o
f1 (ppm)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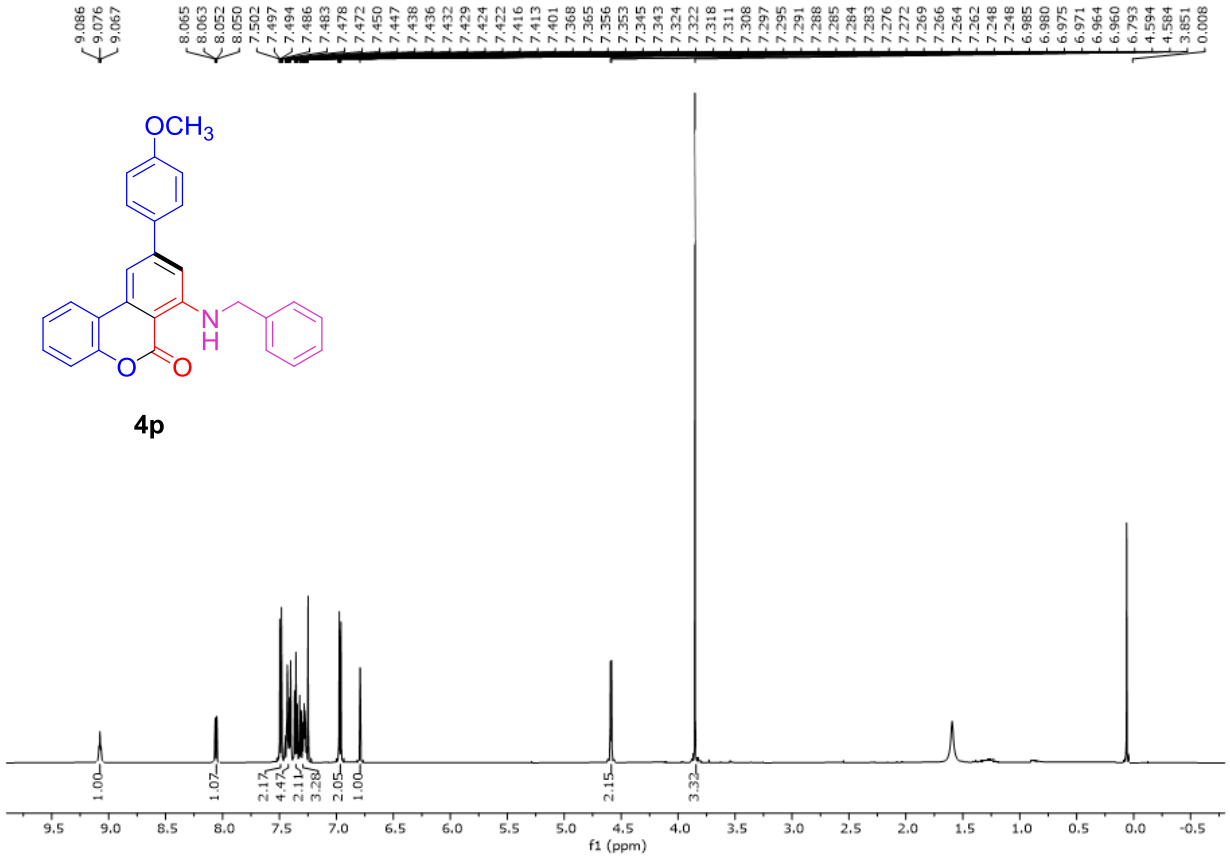
H
O
O
4o
f1 (ppm)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7.438
7.436
7.432
7.429
7.424
7.422
7.416
7.413
7.401
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7.356
7.353
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7.343
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7.248
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6.964
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6.793
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0.008
OCH3
N
H
O
O
4p
1.00
1.07
2.17
4.42
2.11
3.28
2.05
1.00
2.15
3.32
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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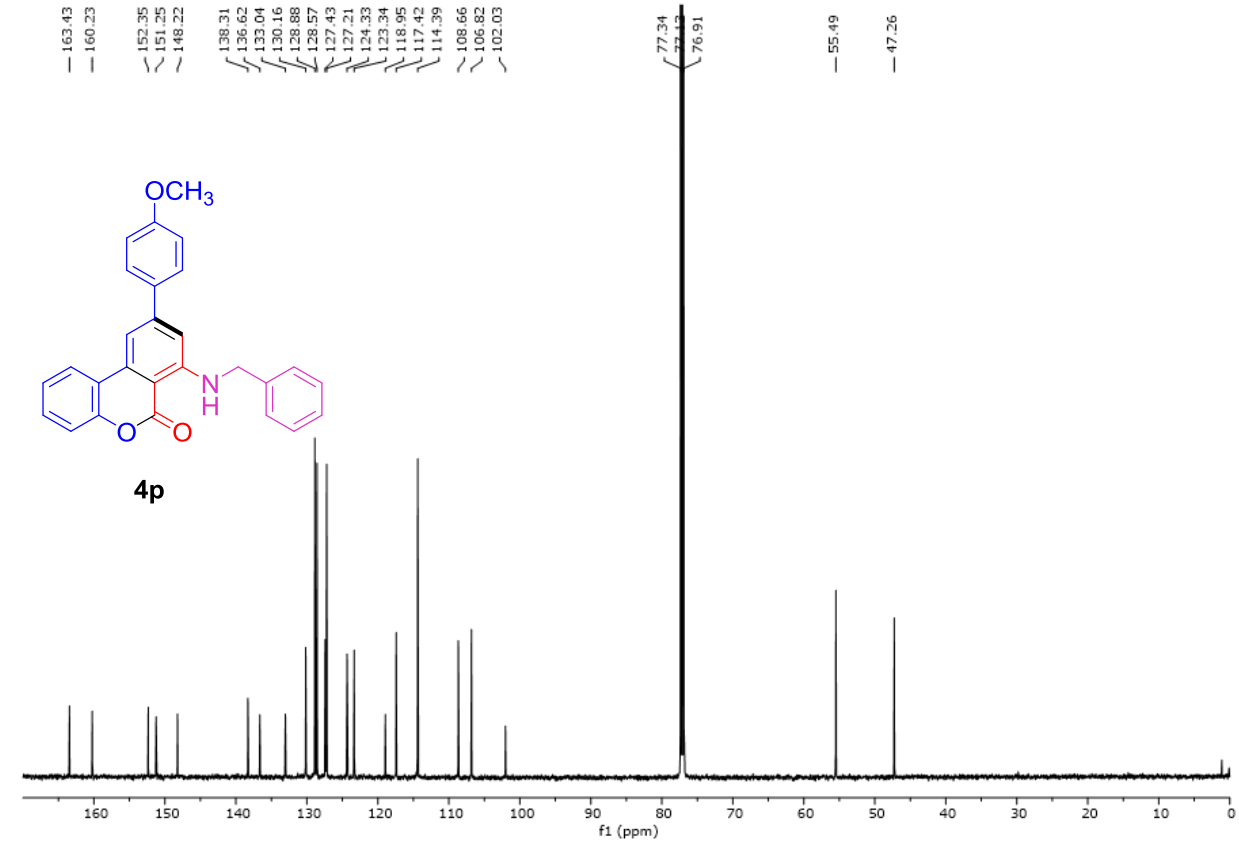
0.5
0.0
-0.5
f1 (ppm)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124.33
123.34
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114.39
108.66
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102.03
77.34
77.13
76.91
55.49
47.26
OCH3
N
H
O
O
4p
160
150
140
130
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110
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50
40
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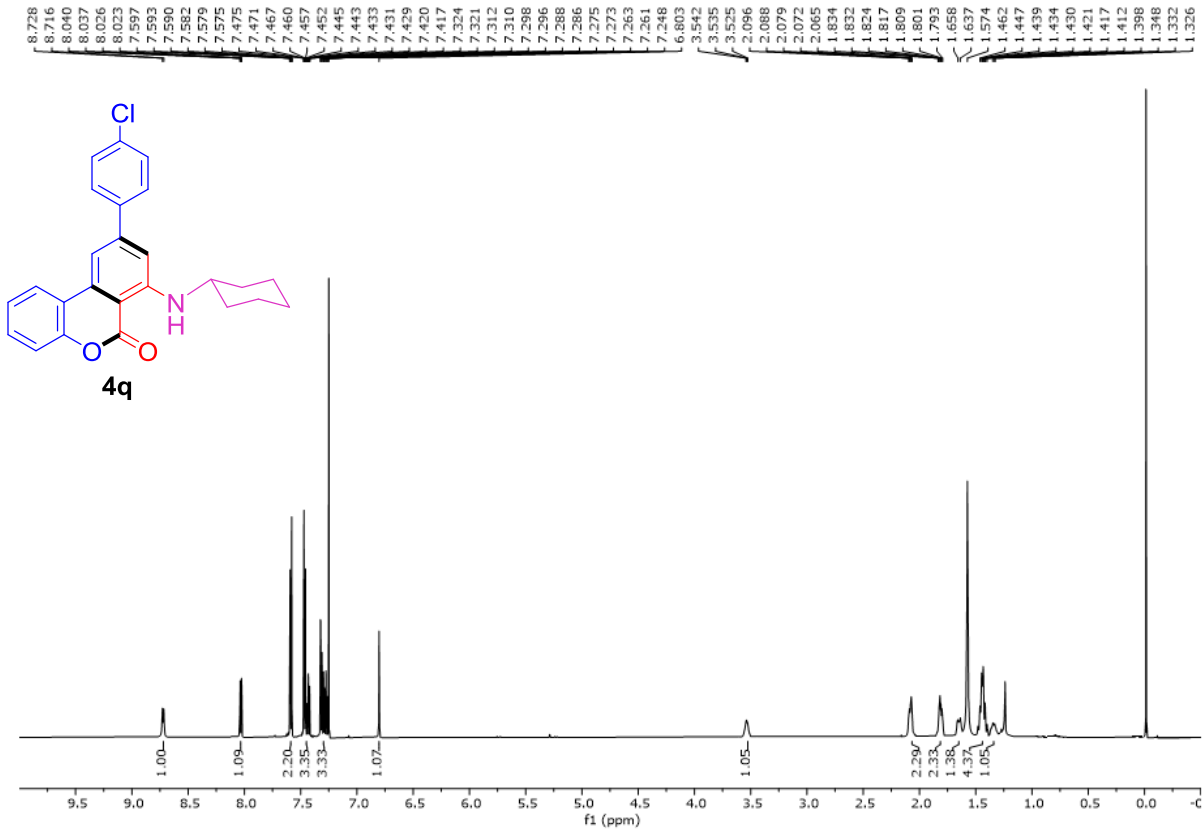
f1 (ppm)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7.457
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7.443
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7.429
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7.417
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7.321
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7.288
7.286
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7.273
7.263
7.261
7.248
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1.817
1.809
1.801
1.793
1.658
1.637
1.574
1.462
1.447
1.439
1.434
1.430
1.421
1.417
1.412
1.398
1.348
1.332
1.326
Cl
N
H
O
O
4q
1.00
1.09
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2.29
2.33
1.38
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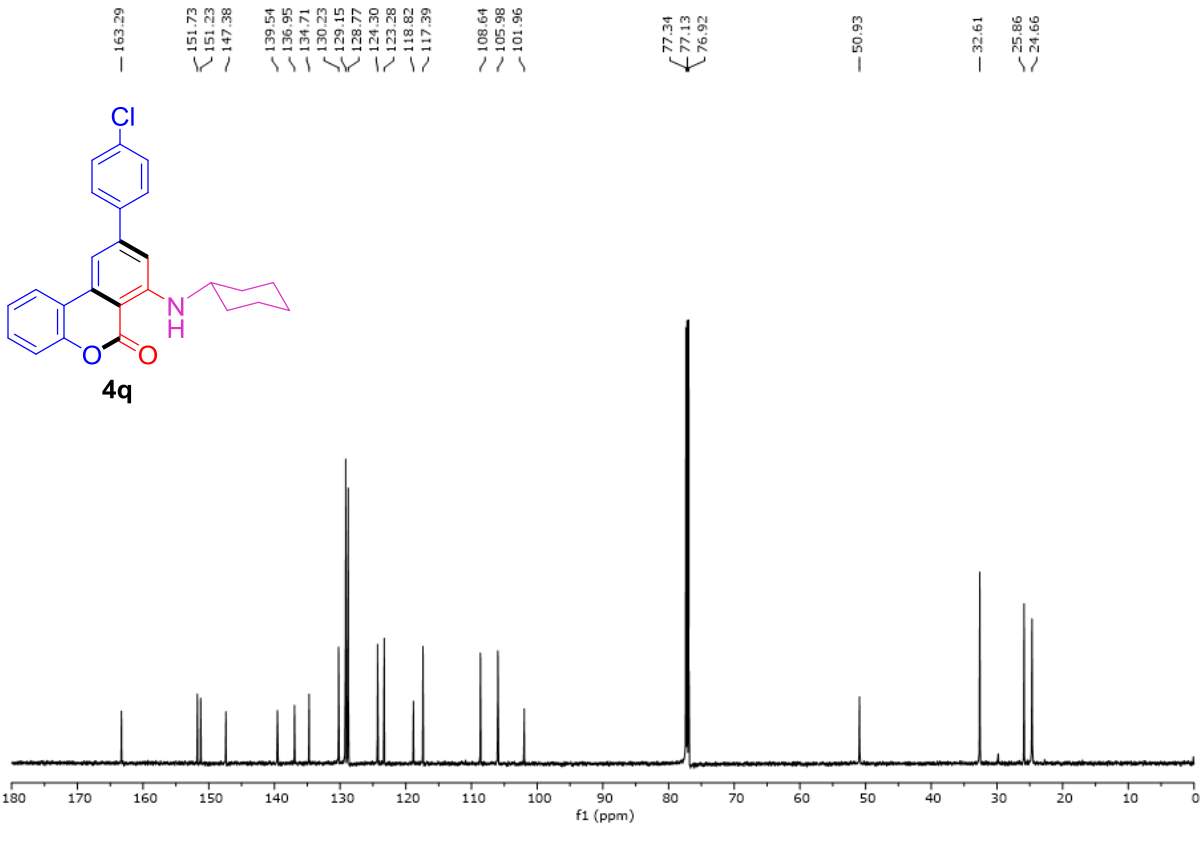
1.05
f1 (ppm)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117.39
108.64
105.98
101.96
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77.13
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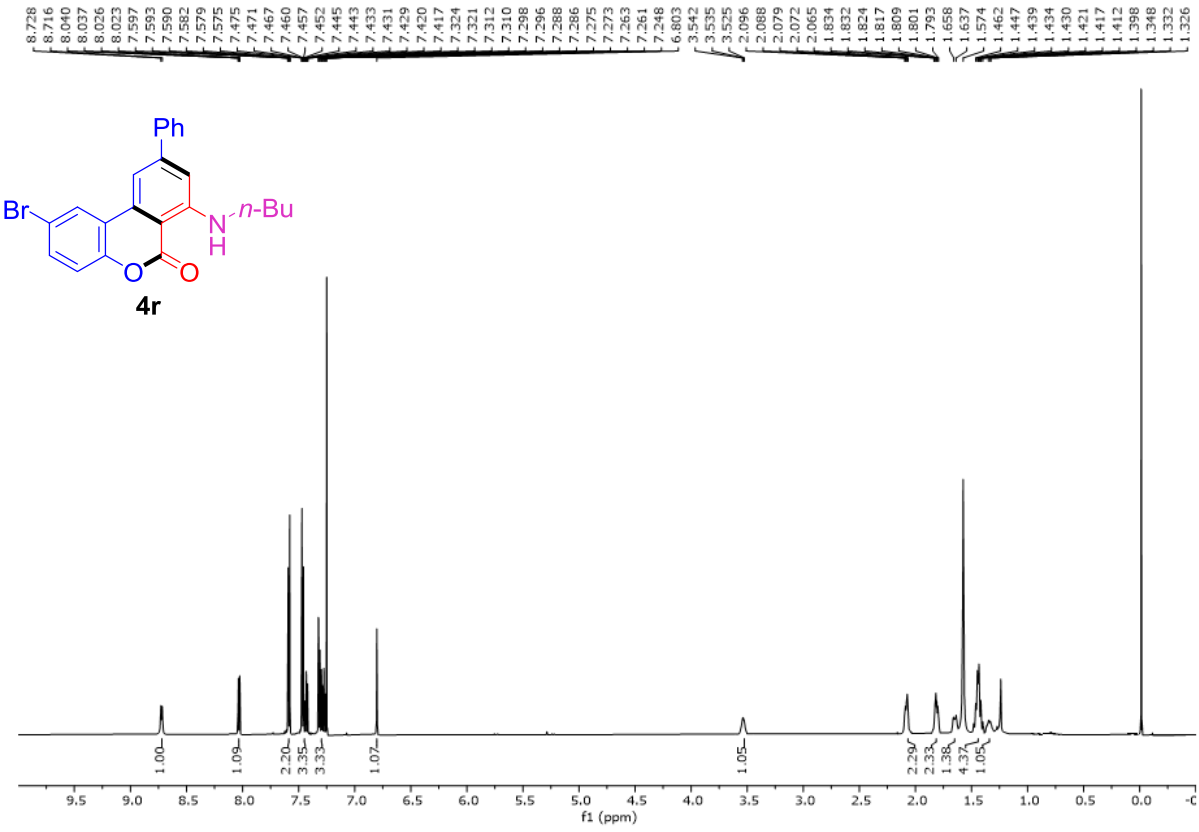
24.66
Cl
N
H
O
O
4q
f1 (ppm)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O
4r
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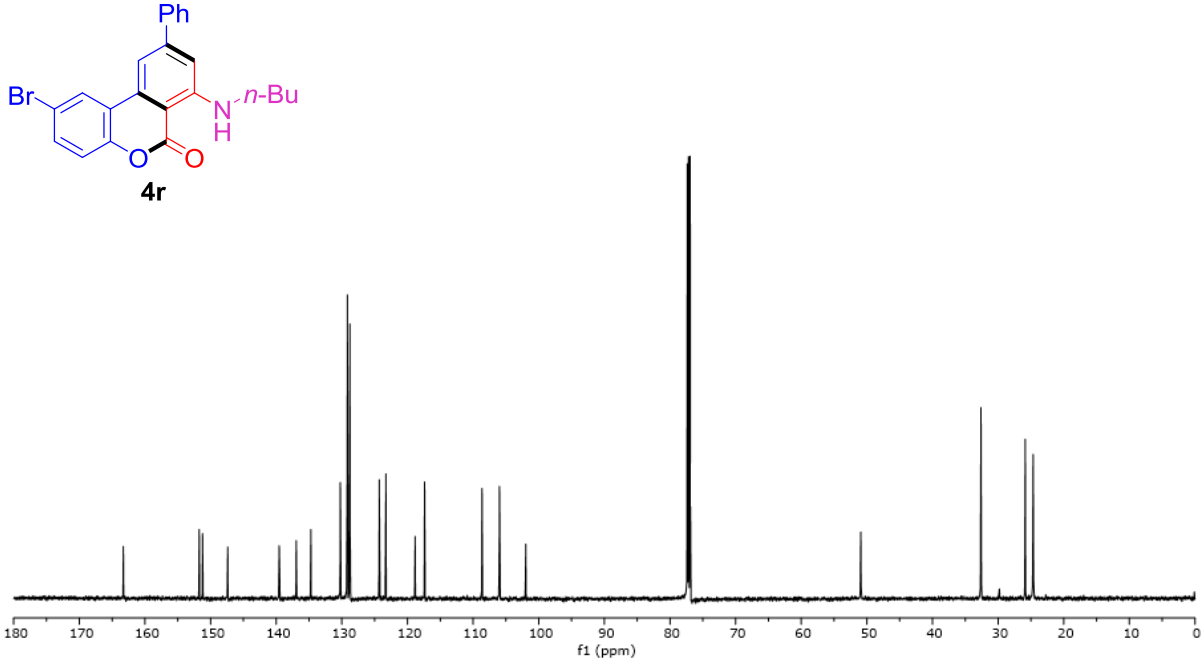
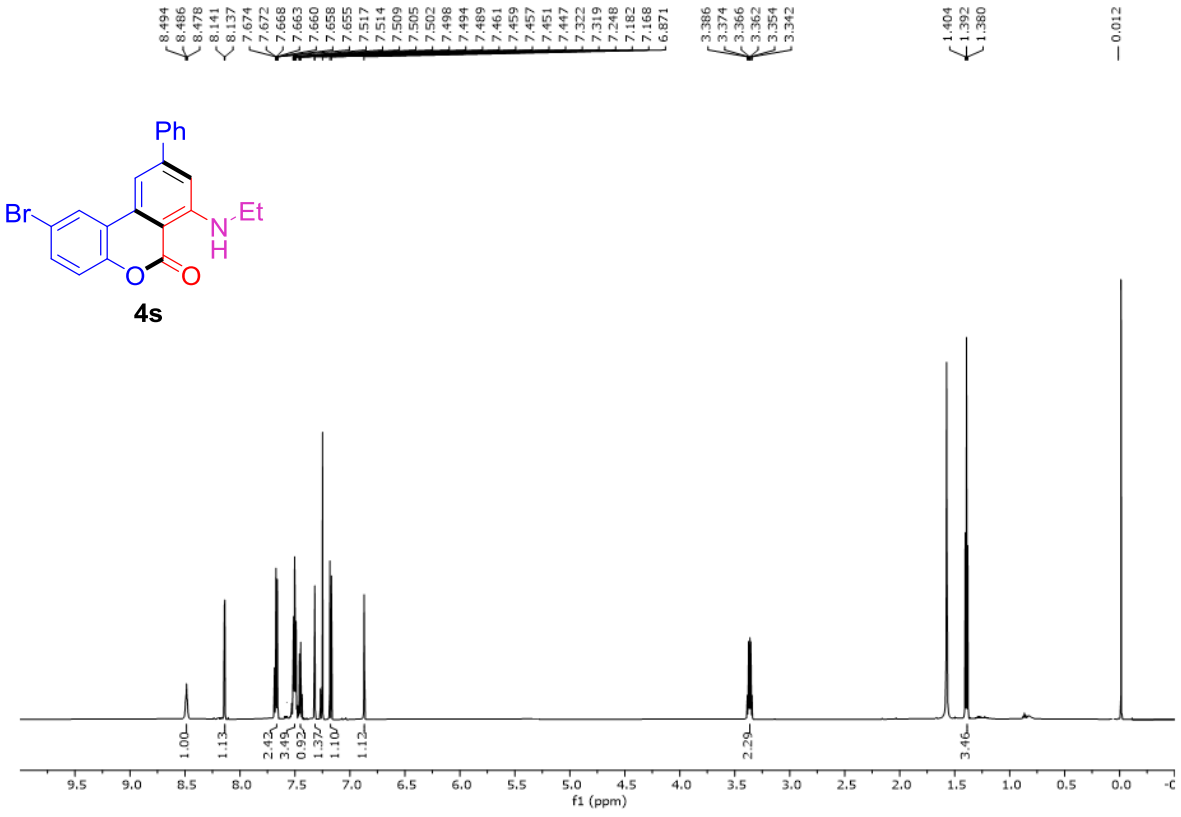

Ph
Br
n-Bu
N
H
O
O
4r
f1 (ppm)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7.168
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3.366
3.362
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3.342
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1.392
1.380
0.012
Ph
Br
N
Et
H
O
O
4s
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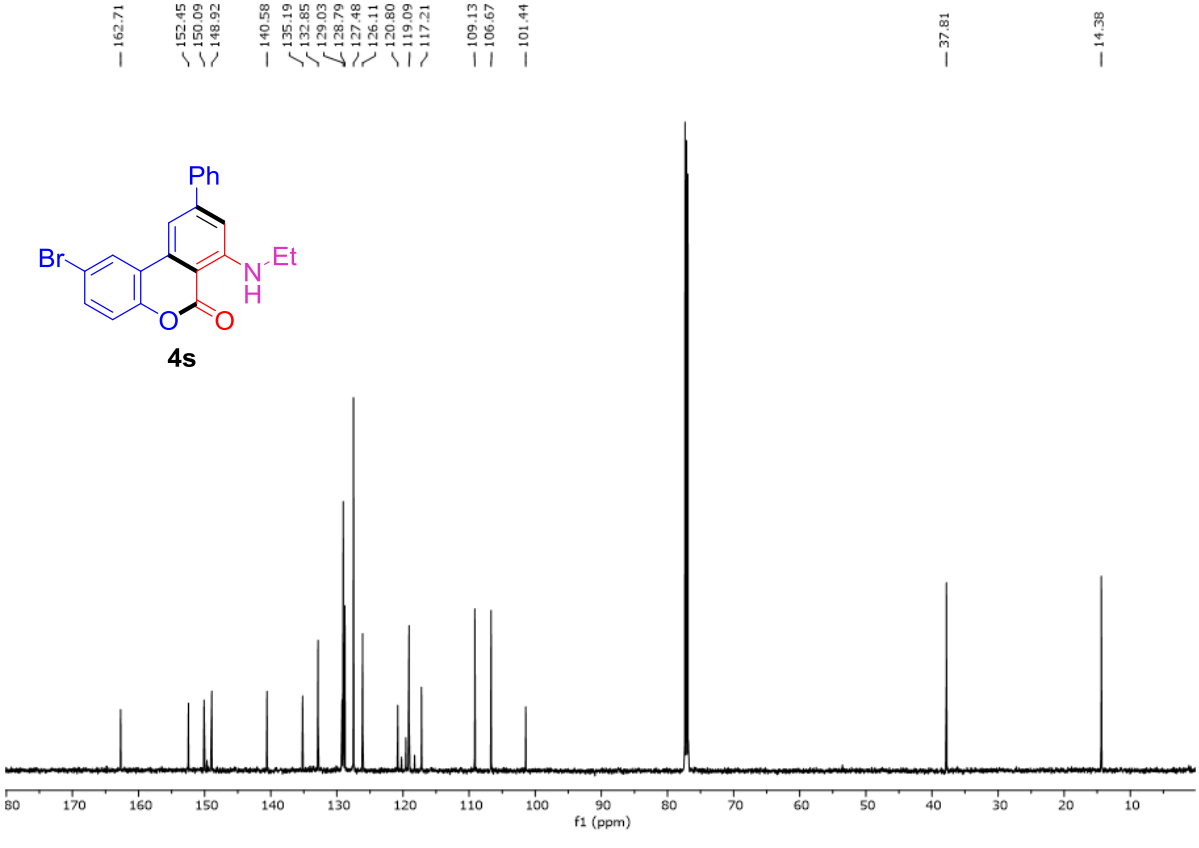
f1 (ppm)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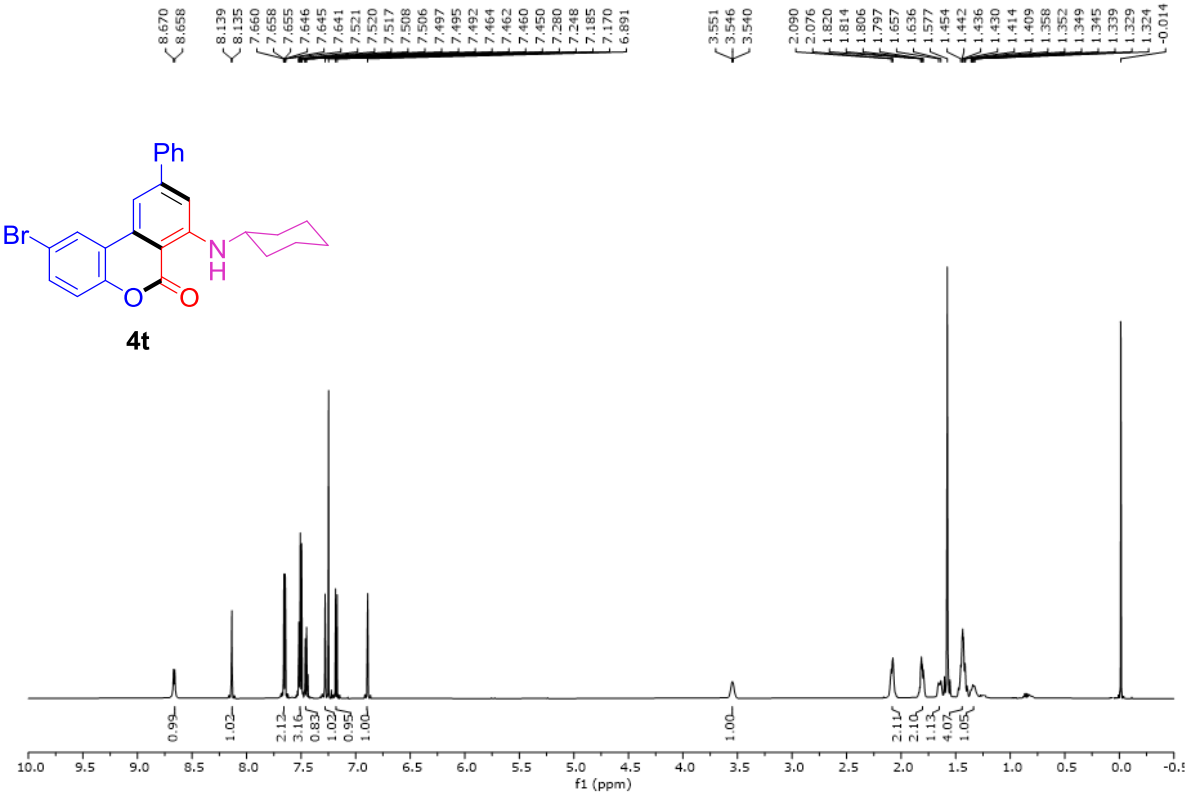
14.38
Ph
Br
N
Et
H
O
O
4s
f1 (ppm)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1.324
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H
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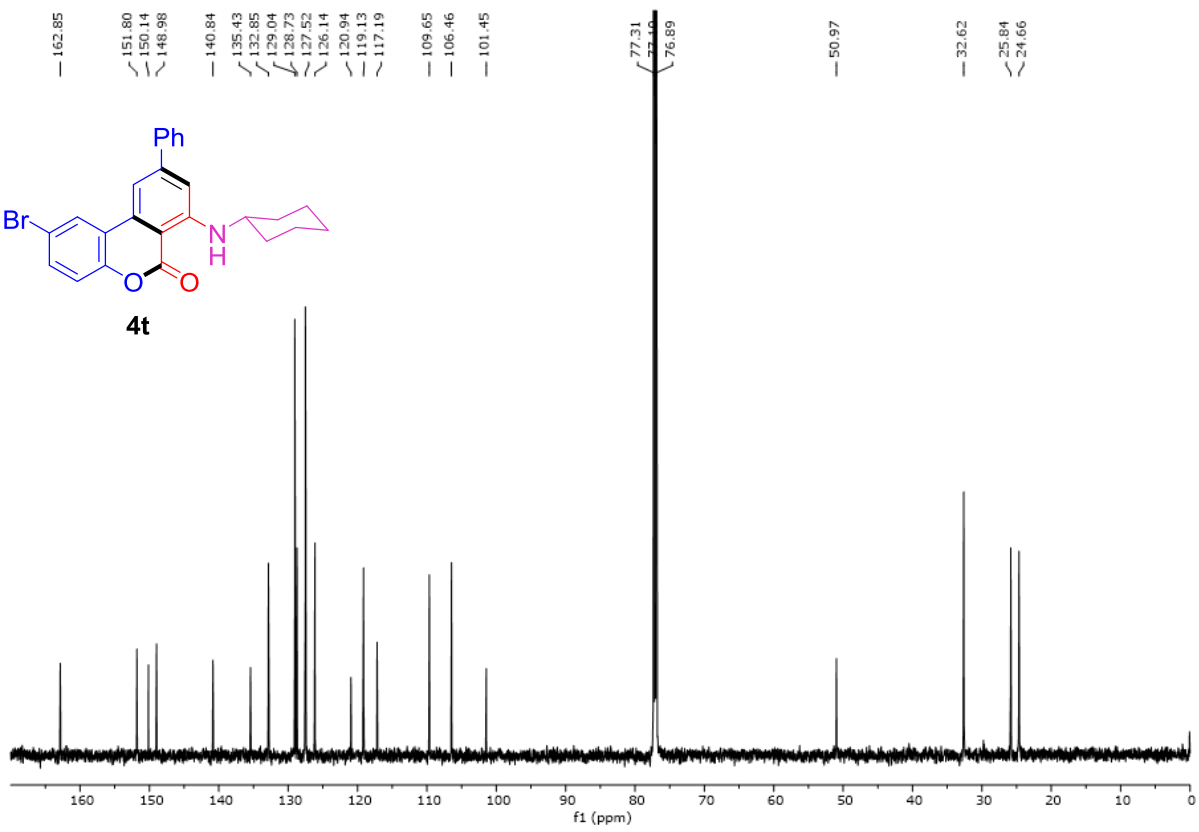
1.05
f1 (ppm)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77.31
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32.62
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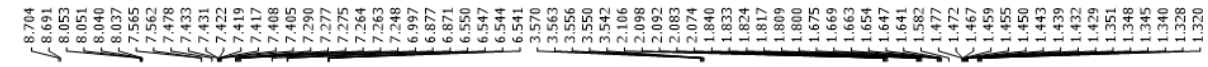
24.66
Ph
Br
N
H
O
O
4t
f1 (ppm)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3.556
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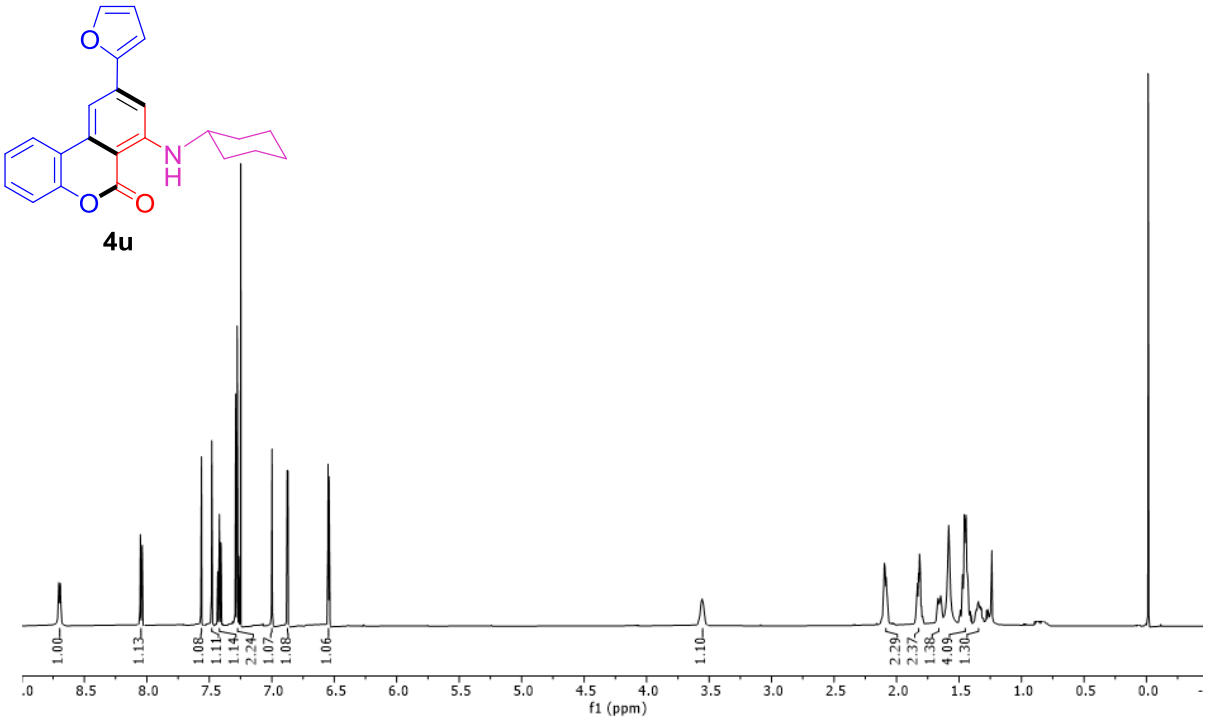
1.320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H
O
O
4u
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1.06
1.10
2.29
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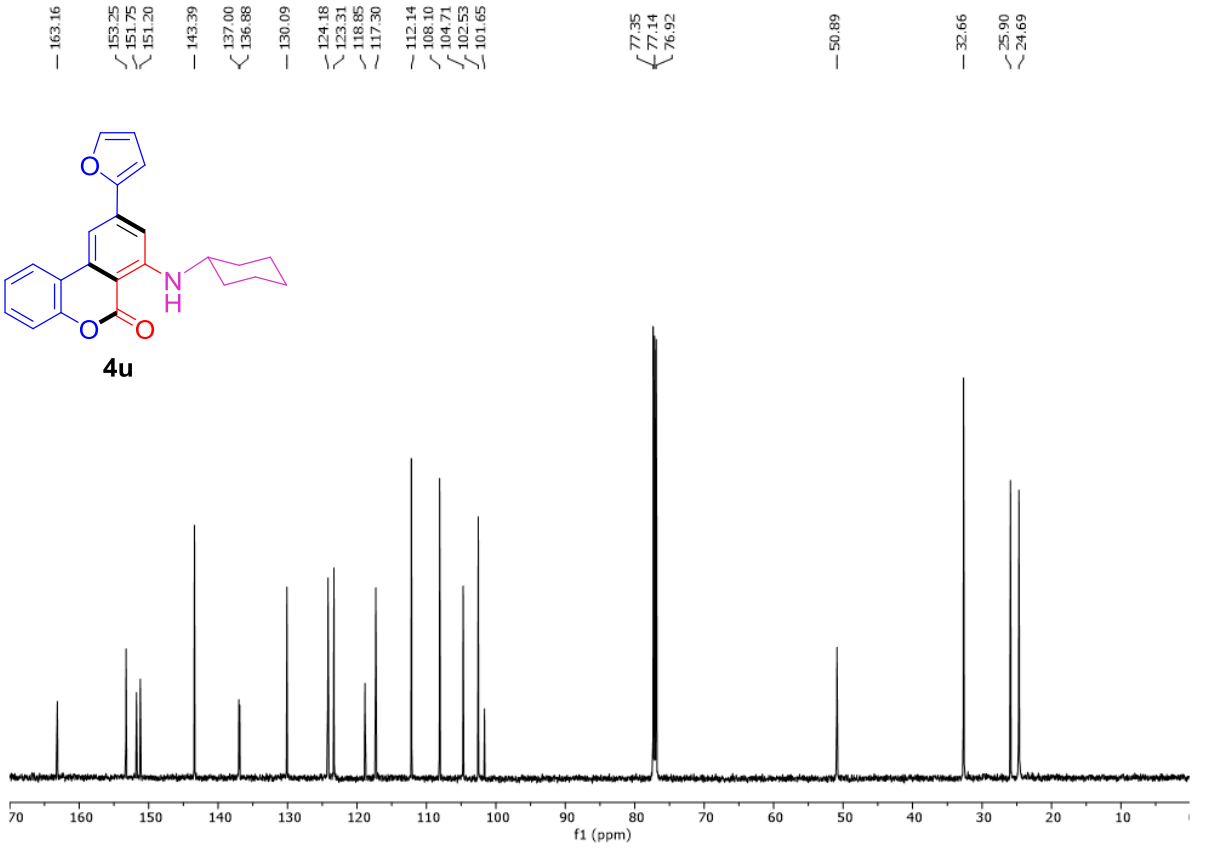
1.38
4.09
1.30
f1 (ppm)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77.14
76.92
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32.66
25.90
24.69
O
N
H
O
O
4u
f1 (ppm)

12. References

1. E. Sankari Devi, T. Pavithra, A. Tamilselvi, S. Nagarajan, V. Sridharan, and C. Uma Maheswari., *Org. Lett.* **2020**, *22*, 9, 3576–3580.
2. B. S. Vachan, A. Ramesh, M. Karuppasamy, I. Muthukrishnan, S. Nagarajan, C. J. Men´endez, C. U. Maheswari and V. Sridharan, *RSC Adv.* **2019**, *9*, 32946–32953.
3. S. Yaragorla and R. Dada, *ACS Omega*., **2017**, *2*, 4859−4869.
4. Andrew P. Dicks, *Curr. Opin. Green Sustain. Chem*., 2018, **13**, 27–31.