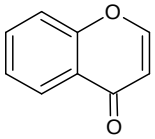
Chapter-IV

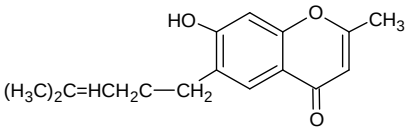
Synthesis of new bischromones linked *via* the 2-aryl ring

The chromone structure is a common heterocyclic framework that is found in numerous natural products exhibiting interesting biological activities.[1-4] The name "chromone" was first used by Bloch and Kostanechi[5] in 1900. This name was chosen because of several naturally occurring compounds like brazilin and hydroxyflavones were colored in nature and known to contain the benzopyran-4-one moiety **4.1**. Chromone was first prepared by Ruhemann and Stapleton[6] by heating 4-oxo-4*H*-1-benzopyran-2-carboxlic acid.

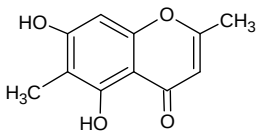


4.1

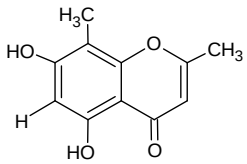
The majority of the chromones that occur in nature contain hydroxyl groups at C-5 and C-7 and a methyl at C-2. First of these to be identified was peucenin **4.2** which was obtained from rhizome of the masterwort *Peucedanum ostruthium*.[7] Eugenitol **4.3** is formed in the cultures of fungal *symbiont Lecanora rupicola*[8] which grows on lichens and its isomer isoeugenitol **4.4** was isolated from the clove plant, *Eugenia caryophyllata*.[9,10]



4.2

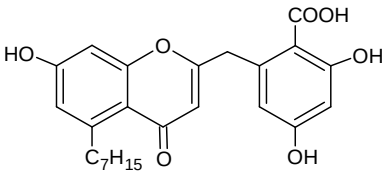


4.3

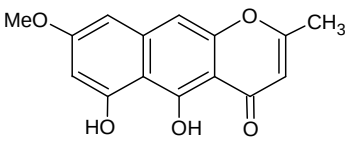


4.4

Siphulin **4.5** and rubrofusarin **4.6** were extracted from *scandinavian lichen*, Siphula ceretites, a chromone carboxylic acid[11] and from a fungus, *Fusarium culmorum*.[12]



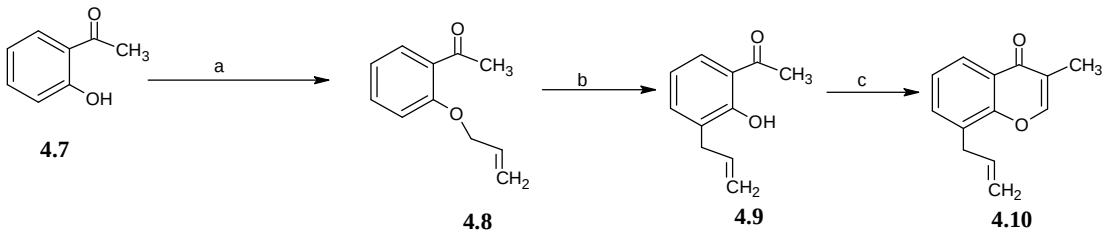
4.5



4.6

Many of the natural and synthetic flavones have been associated with the various biological behaviors such as vasodilatory, diuretic, antioxidant, anticancer, anti HIV, antiviral, anti-inflammatory, antiallergic and bacteriocidal activitities.[13-17] Due to their abundance in plants and low mammalian toxicity, chromone derivatives are also present in large amount in human diet.[18]

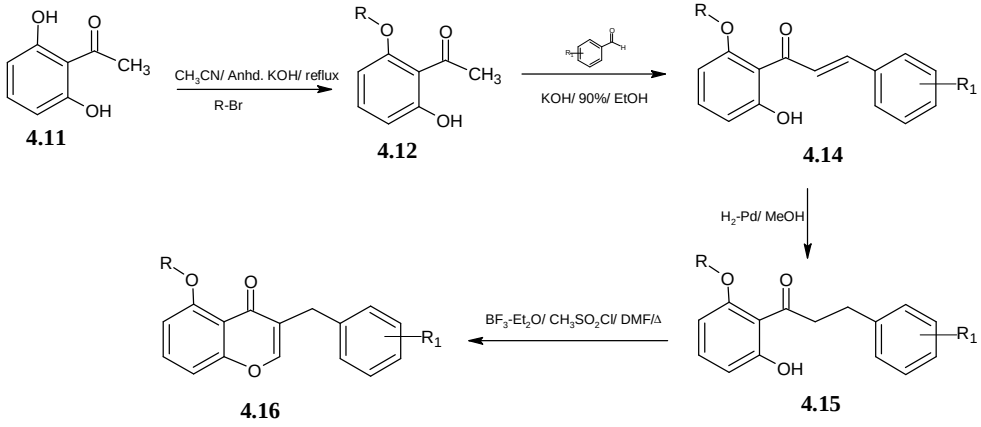
The chromone[19] **4.10** has been prepared starting from the reaction of 2-hydroxyacetophenone **4.7** with allyl bromide to give allyl ether **4.8** which underwent *Claisen rearrangement* to provide 3-allyl-2-hydroxyacetophenone **4.9.** The formylation reaction of later with Vilsmeier Haack reagent (DMF/$POCl_3$) afforded the final compound **4.10 (Scheme-4.1).**



Reaction Conditions: a) CH_2=CH-CH_2Br/ dry acetone/K_2CO_3 ; b) 260°C-270°C; c) (i) $POCl_3$/DMF; (ii) H_2O

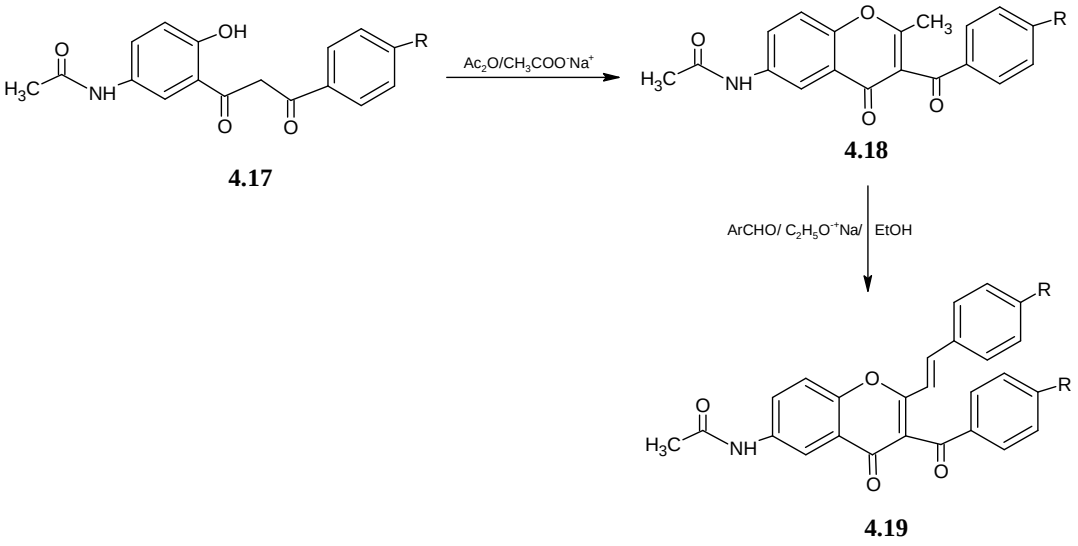
Scheme-4.1

The chalcone **4.14** was obtained[20] from the reaction of 2-hydroxyacetophenone **4.12** with benzaldehyde **4.13** in the presence of KOH/EtOH. The partial reduction of **4.14** with Pd/H_2 resulted in the formation of **4.15** which was finally reacted with N,N-dimethylformamide, methanesulfonyl chloride and boron trifluoride diethyl etherate under refluxing conditions to yield chromone **4.16** as the end product **(Scheme-4.2)**.



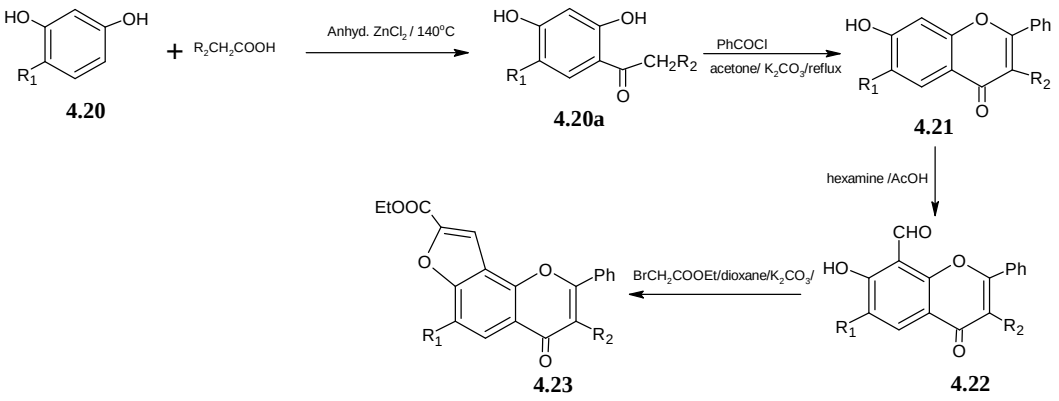
Scheme-4.2

1-(5'-Acetamido-2'-hydroxyphenyl)-3-aryl-1,3-diketones[21] **4.17** have been refluxed under acetic anhydride in presence of anhydrous sodium acetate to obtain 6-acetamido-3-aroyl-2-methyl-chromones **4.18**. The condensation reaction of later with suitable aromatic aldehyde in the presence of NaOEt provided 2-styryl-chromone **4.19** (**Scheme-4.3)**.



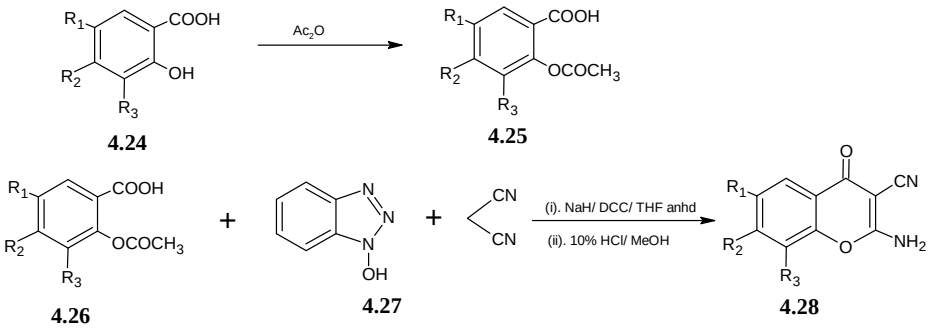
Scheme-4.3

Ethylfuro[2,3-h] chromone-8-carboxylates[22] **4.23** have been realized starting from **4.20** according to the reactions which are shown in **Scheme-4.4**.



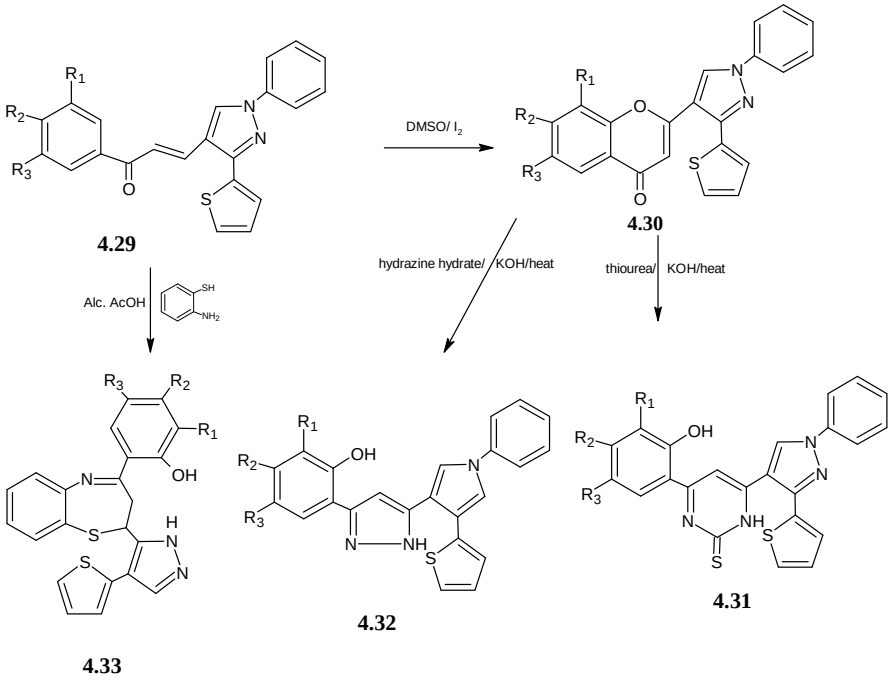
Scheme-4.4

The chromones **4.28** were synthesized[23] from the reaction of *N*-hydroxybenzotriazolyl-acetylsalicylates **4.26** with *N*-hydroxybenzotriazole **4.27** and malononitrile under the reaction conditions which are depicted in **Scheme-4.5**.



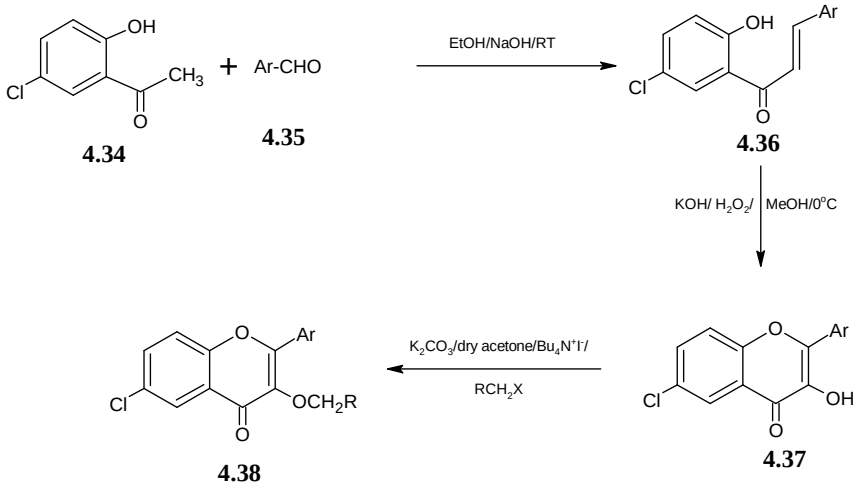
Scheme-4.5

The oxidative cyclization[24] of chalcone **4.29** with DMSO-I_2 yielded chromone **4.30** which was further reacted with thiourea and hydrazine hydrate to provide **4.31** & **4.32** while the cyclocondensation reaction of **4.29** with 2-aminothiophenol provided **4.33** as the end product **(Scheme-4.6)**.



Scheme-4.6

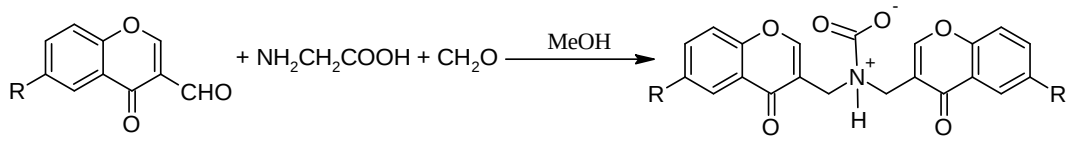
To investigate the photochemical behavior of 3-alkoxychromones,[25-36] compounds **4.38** have been obtained from the alkylation of 3-hydroxychromone **4.37** with suitable alkylating agent. The later were obtained from the oxidative cyclization of chalcone **4.36** under the A. F. O. reaction conditions (KOH/H_2O_2) **(Scheme-4.7)**.



Scheme-4.7

Recently some more studies have been reported upon the synthesis of chromone derivatives.[37,38]

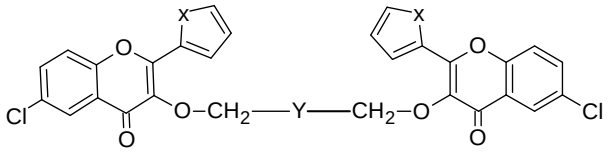
Bischromones are the molecules which are formed by joining two chromones moiety together with the carbon chains of varying lengths and structure. Synthesis of some bischromone have been reported from the deformylative Mannich type reaction of chromone-3-carboxaldehyde **4.39** with □-amino acid **(Scheme-4.8)**.[39]



4.39 **4.40**

Scheme-4.8

Recently, synthesis and photochemical studies of the some bischromones **4.41** have been investigated in our laboratory.[40, 41]

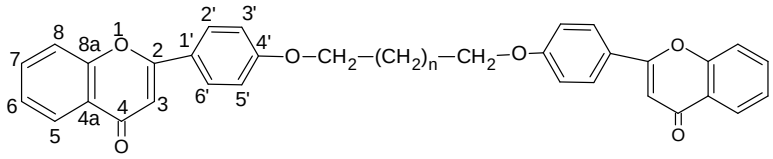


4.41

X = S, O; Y = -C=C-, -C≡C-

It is evident from the above literature survey that chromones and bischromones are associated with the immense synthetic and biological applications. Also very little attention have been paid upon the synthesis of bischromones linked through the 2-aryl ring. These aspects have prompted us to investigate the synthesis and antimicrobial studies of new bischromones **4.50-4.57**.

The compounds **4.50-4.57** needed for the present studies were prepared from the cyclization reaction of bischalcones **4.42-4.49** with iodine by refluxing under DMSO medium. The bischalcones were obtained from the Claisen-Schmidt reaction[42] of *o*-hydroxyacetophenone with dibenzaldehydes **2.48-2.55**. The structures of the prepared compounds were determined from the rigorous analysis of their spectral data (UV-Vis, IR, ^{1}H-NMR, ^{13}C-NMR & ESI-MS). The elemental analysis also confirmed the purity of these compounds.



4.50-4.57

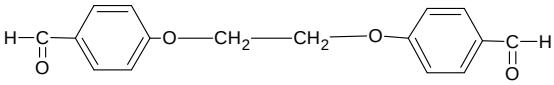
n = 0, 1, 2, 3, 4, 6, 8, 10

Synthesis of bischromone 4.50

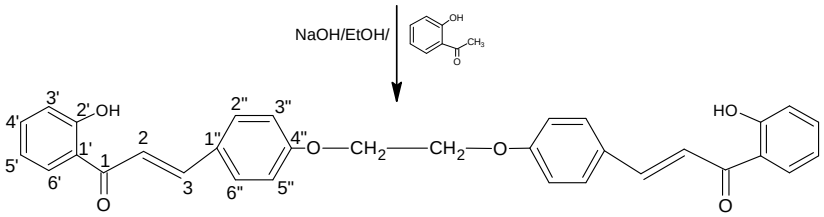
The compound **4.50** was obtained in two steps:

(i). Synthesis of bischalcone 4.42

The dibenzaldehyde **2.48** was reacted with *o*-hydroxyacetophenone in the presence of NaOH/EtOH at room temperature for 12 hrs and the decomposition of resulting reaction mixture into iced-HCl provided a solid product. The crude substance thus obtained was purified by crystallization in $CHCl_3$:MeOH (1:1) to furnish pure compound **4.42** as a yellow solid (70%, m.p. 96-98°C) **(Scheme-4.9)**.



2.48



4.42

Scheme-4.9

The structure of **4.42** became evident from the various spectroscopic data. Its IR spectrum exhibited major absorptions at 3230 (O-H), 2923, 2856 (methylene C-H), 1658 (C=O) and 1598 (C=C). In the ^{1}H-NMR spectrum (400 MHz, DMSO-d_6) of **4.42**, a two protons sharp singlet at δ 12.95 may be ascribed to O***H*** group. The aromatic protons H-6', H-4', H-5', H-2'', 6'', H-3'', 5'' and H-3' appeared as a doublet each at δ 8.56 (2H, J_o=7.8 Hz), 7.98 (2H, J_o=8.1 Hz), 7.60 (2H, J_o=7.5 Hz), 7.80 (4H, J_o=8.8 Hz), 7.11 (4H, J_o=8.8 Hz) and 7.15 (2H, J_o=8.9 Hz) respectively. In this region, two broad doublets centered at δ 7.90 (2H, J_{trans}=15.4 Hz) and 7.78

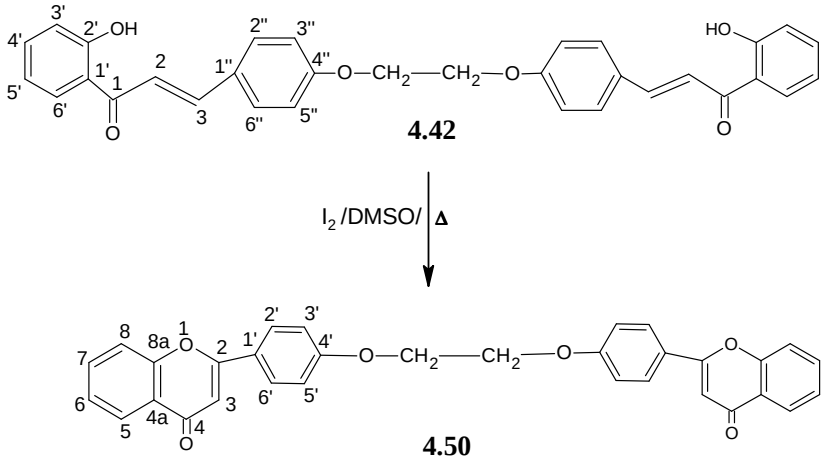
(2H, J_{trans}=15.4 Hz) were denoted by *trans* protons H-3 and H-2 respectively. A singlet at δ 4.24 (4H) may be assigned to intervening chain OC***H₂*** protons.

The ESI-MS spectrum of **4.42** exhibited noticeable ions at m/z 529 (M+Na, 13%), 476 (22%), 475 (100%), 445 (21%) and 426 (8%) which were helpful to describe the structural features of **4.42**. The UV-Vis spectrum of **4.42** had two maxima at 322 and 263 nm which may be ascribed to n→π* and π→π* transitions respectively.

^{13}C-NMR (100 MHz, DMSO-d_6) spectrum of **4.42** was very instrumental to corroborate its proposed structure. Here, presence of C=O group was confirmed by the appearance of a downfield signal at δ 191.87. The carbon atoms C-2', C-4'', C-3 and C-2 were placed at δ 162.76, 157.54, 144.44 and 118.12 respectively. The remaining signals in the aromatic region were found to be resonating at δ 134.32 (C-4'), 128.76 (C-2'', 6''), 123.72 (C-1'), 127.98 (C-6'), 125.21 (C-1''), 121.45 (C-5'), 118.87 (C-3') and 114.52 (C-3'', 5''). Towards the extreme right hand side of the spectrum, a signal was also present at δ 66.22 which could be attributed to O***C***H_2 group.

(ii). Cycilzation of bischalcone 4.42

A mixture of bischalcone **4.42** and iodide in DMSO was refluxed for 6 hrs and the resulting reaction mixture was poured into ice to provide a solid substance. The crude product was crystallized from MeOH to give pure compound **4.50** (52%, m.p. 256-258^{o}C) **(Scheme-4.10)**.



Scheme-4.10

The functional group region of the IR spectrum of **4.50** had strong absorption at 1640 cm^{-1} which is a characteristic band of the pyrone ring. Its ESI-MS spectrum exhibited the heaviest ion at m/z 503 (M+1, 87%) along with other significant ions at m/z 427

(10%), 425 (33%), 385 (100%), 342 (23%), 293 (9%), 249 (28%), 228 (6%) & 176 (39%). These mass fragments were very helpful to confirm the proposed structure of **4.50**. The UV-Vis spectrum of **4.50** had two maxima at 312 & 228 nm due to n→π* and π→π* transitions respectively.

A comparison of the ^{1}H-NMR (400 MHz, DMSO-d_6) spectra of **4.42** and **4.50** shows that resonances present in the former at δ 7.90 (H-3) and 7.78 (H-2) were found missing altogether in the former thereby pronouncing the involvement of these protons in the cyclization reaction. The benzenoid protons produced suitable signals at δ 8.14 (2H, dd, $J_{p,o}$=1.1, 8.6 Hz, H-5), 7.59 (2H, td, $J_{m,o}$=1.8, 8.3 Hz, H-7), 7.50 (2H, d, J_o=8.3 Hz, H-8) and 7.44 (2H, t, J_o=7.7 Hz, H-6). The downfield resonance of the H-5 as compared to other protons may be ascribed to its close proximity to the C=O group of the pyrone moiety. The protons H-2', 6' & H-3', 5' belonging to the *p*-disubstituted benzene ring appeared as a doublet each at δ 7.72 (4H, J_o=8.8 Hz) and 6.93 (4H, J_o=8.8 Hz) respectively. A sharp singlet integrating for two hydrogens at δ 6.74 could be ascribed to pyrone ring proton H-3. A singlet placed in the aliphatic region at δ 4.15 (4H) may be furnished by OC***H*****$_2$** protons.

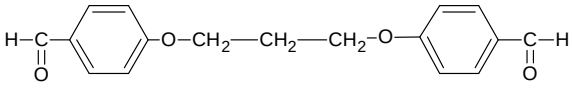
^{13}C-NMR spectrum of **4.50** also proved instrumental to corroborate its carbon framework. The most downfield signal present at δ 178.52 was easily assignable to pyrone ring C=O group and the other downfield signals resonating at δ 168.10, 163.94 and 156.23 could be resulted by C-2, C-8a and C-4' respectively due to their direct bonding to the oxygen atom. The benzenoid ring carbon atoms resonated at δ 133.65 (C-5), 125.16 (C-7), 123.60 (C-6) and 117.98 (C-8) while signals for C-4a and C-3 were resonating at δ 123.92 and 106.25 respectively. The remaining aromatic carbons C-1', C-2', 6' & C-3', 5' generated three signals at δ 128.09, 125.70 and 114.97 respectively. The signal corresponding to O***C***H$_2$ was found to be located at δ 64.50.

Synthesis of bischromone 4.51

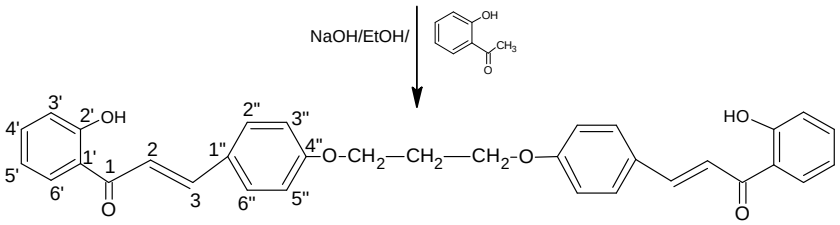
The compound **4.51** was obtained in two steps:

(i). Synthesis of bischalcone 4.43

The dibenzaldehyde **2.49** was reacted with 1,3-dibromopropane under the similar conditions as used earlier for **4.42 (Scheme-4.11)** to yield a pure compound **4.43 (**64%, m.p. 182-184°C).



2.49



4.43

Scheme-4.11

IR spectrum of **4.43** showed noticeable bands at 3217 (O-H), 2928, 2853 (methylene C-H), 1597 (C=C) & 1658 (C=O). Its ^{1}H-NMR spectrum (400 MHz, DMSO-d_6) produced two doublets at δ 7.88 (2H) and 7.77 (2H) which may be assigned to H-3 and H-2 protons and the coupling constant of $J_{3,2}$=15.6 Hz between these hydrogens suggests their *trans* relationship. The signal for O***H*** group was observed at δ 12.89 as a D_2O exchange sharp singlet while the protons H-6', H-4', H-5' & H-3' were present at δ 8.52 (2H, d, J_o=7.6 Hz), 7.95 (2H, d, J_o=7.4 Hz), 7.67 (2H, d, J_o=7.2 Hz) and 7.11 (2H, d, J_o=6.8 Hz) respectively. The remaining aromatic hydrogens (H-2'', 6'' & H-3'', 5'') also resulted suitable resonances at the appropriate positions (vide experimental). Towards the upfield region, two signals centered at δ 4.21 (4H, t, J_{vic}=6.2 Hz) and 2.25 (2H, quintet, J_{vic}=6.2 Hz) were easily assigned to OC***H₂***CH_2 and OCH_2C***H₂*** group protons respectively. The UV-Vis spectrum of **4.43** had two maxima at 316 and 254 nm which may be provided by n→π* and π→π* transitions respectively.

ESI-MS spectrum of **4.43** corroborated its structures which showed heaviest ion and base peak at m/z 543 (M+Na, 35%) and 475 (100%) respectively and its mass fragmentation pattern has been shown in **Chart-1 (**vide experimental).

The carbon framework of **4.43** was corroborated from its ^{13}C-NMR (100 MHz, DMSO-d_6) spectral data which had significant signals at δ 191.80 (C=O), 162.62 (C-2'), 157.48 (C-4''), 144.40 (C-3) and 118.00 (C-2). Other recognizable resonances in the aromatic region were found to be present at δ 134.27 (C-4'), 128.74 (C-2'', 6''), 127.95 (C-6'), 125.18 (C-1''), 123.64 (C-1'), 121.41 (C-5'), 118.85 (C-3') and 114.49 (C-3'', 5''). The intervening chain could provide two signals at δ 68.78 (O***C***H_2CH_2) and 28.17 (OCH_2***C***H_2).

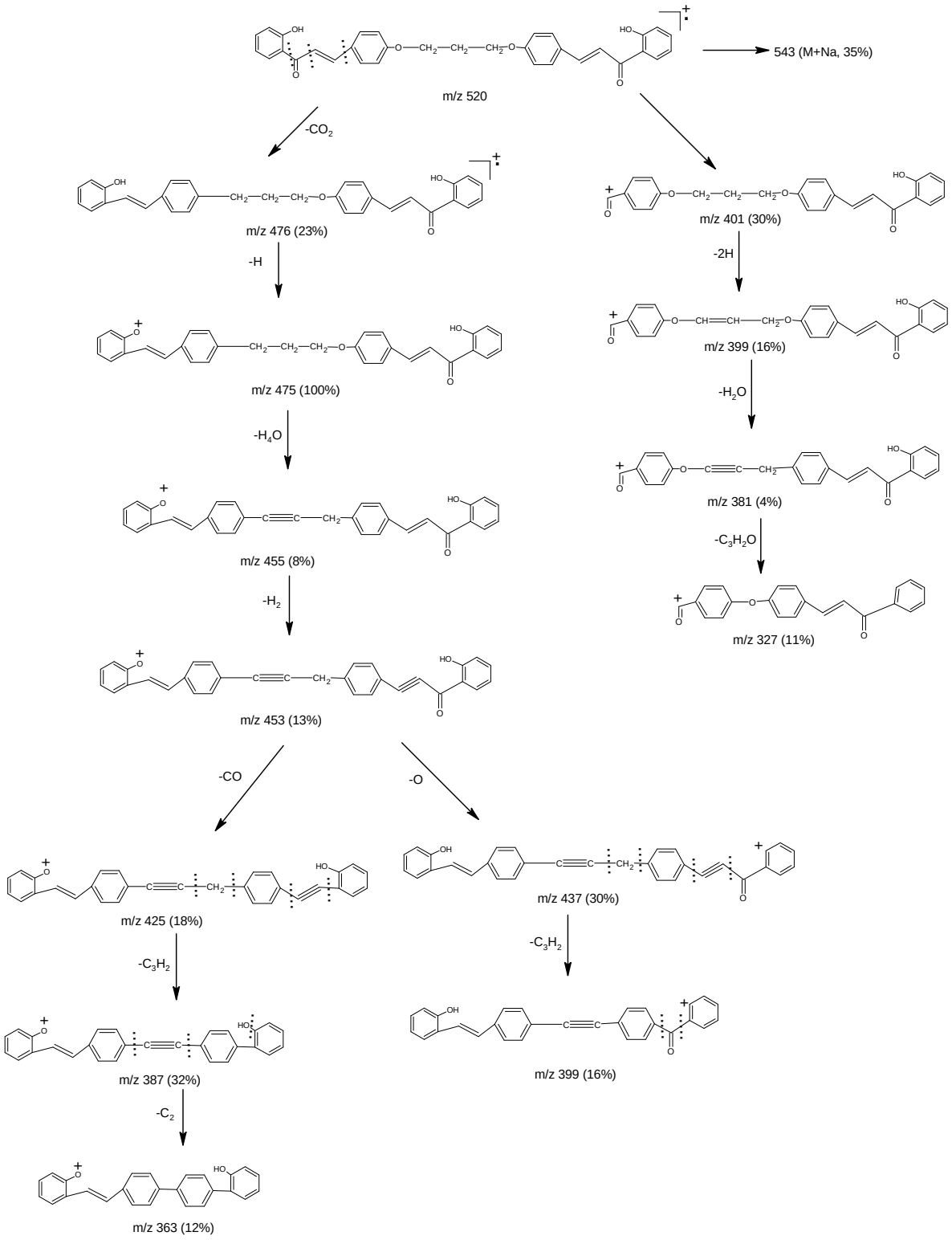
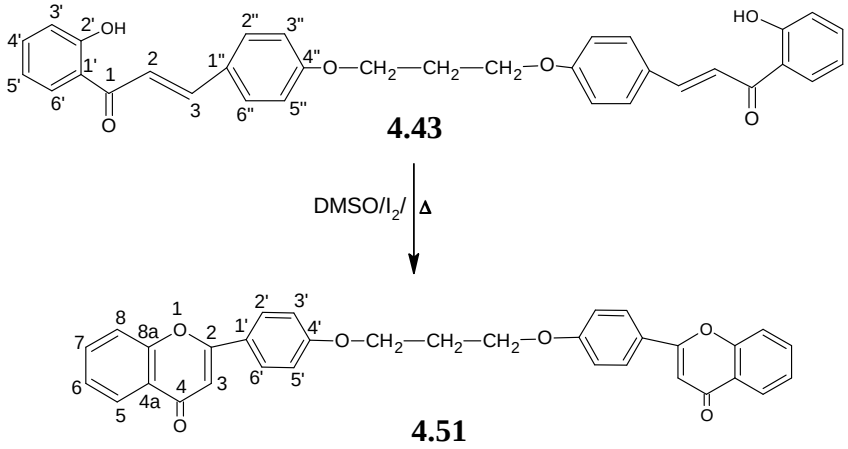


Chart-1

(ii). Cyclization of bischalcone 4.43

The compound **4.51** (61%, m.p. 210-212^{o}C) was prepared from the reaction of **4.43** with iodine **(Scheme-4.12)** under the similar conditions as used earlier for **4.50**.



Scheme-4.12

The IR spectrum of **4.51 (Plate-50)** had strong absorption at 1641 cm^{-1} due to C=O group of the pyrone moiety. The prominent ions in its ESI-MS spectrum **(Plate-53)** were observed at m/z 539 (M+Na, 100%) and 517 (M+1, 23%) and its mass fragmentation pattern has been depicted in **Chart-2** which fully supported the structure of **4.51**.

The salient feature of the ^{1}H-NMR (400 MHz, DMSO-d_6) spectrum of **4.51 (Plate-51)** was the appearance of a two protons sharp singlet at δ 6.69 (H-3) which confirms the formation of the pyrone ring during the cylization reaction. The signals due to benzenoid ring protons H-5, H-7, H-8 and H-6 could appear as a doublet of doublet at δ 8.14 (2H, $J_{p,o}$=1.0, 8.7 Hz), as a doublet at δ 7.62 (2H, J_o=8.6 Hz), as a doublet at δ 7.48 (2H, J_o=8.4 Hz) and as a doublet at δ 7.34 (2H, J_o=7.9 Hz) respectively. Two more doublets placed at δ 7.82 (4H, J_o=8.9 Hz) and 6.98 (4H, J_o=8.9 Hz) were generated by the protons H-2', 6' & H-3', 5' respectively. The hydrogens belonging to OC***H₂***CH_2 and OCH_2C***H₂*** group were resonating at δ 4.21 (4H, t, J_o=6.0 Hz) and 2.29 (2H, t, J_{vic}=6.0 Hz) respectively.

In the ^{13}C-NMR (100 MHz, DMSO-d_6) spectrum of **4.51 (Plate-52)**, four downfield signals present at δ 178.05, 168.00, 163.94 & 156.10 could be allotted to C=O, C-2, C-8a & C-4' respectively. The benzenoid ring carbons C-5, C-7, C-6 & C-8 were very well located at δ 133.65, 125.16, 123.92 & 117.98 respectively. The *p*-disubstituted benzene ring furnished suitable resonances at δ 128.09 (C-1'), 125.70 (C-2', 6') & 114.97 (C-3', 5') while a signal at δ 106.25 was assignable to C-3. Two signals

present in the upfield region at δ 67.50 & 25.78 may be represented by O***C***H_2CH_2 & OCH_2***C***H_2 group respectively. UV-Vis spectrum of **4.51** had two maxima at 310 and 225 nm which may be ascribed to n→π* and π→π* transitions respectively.

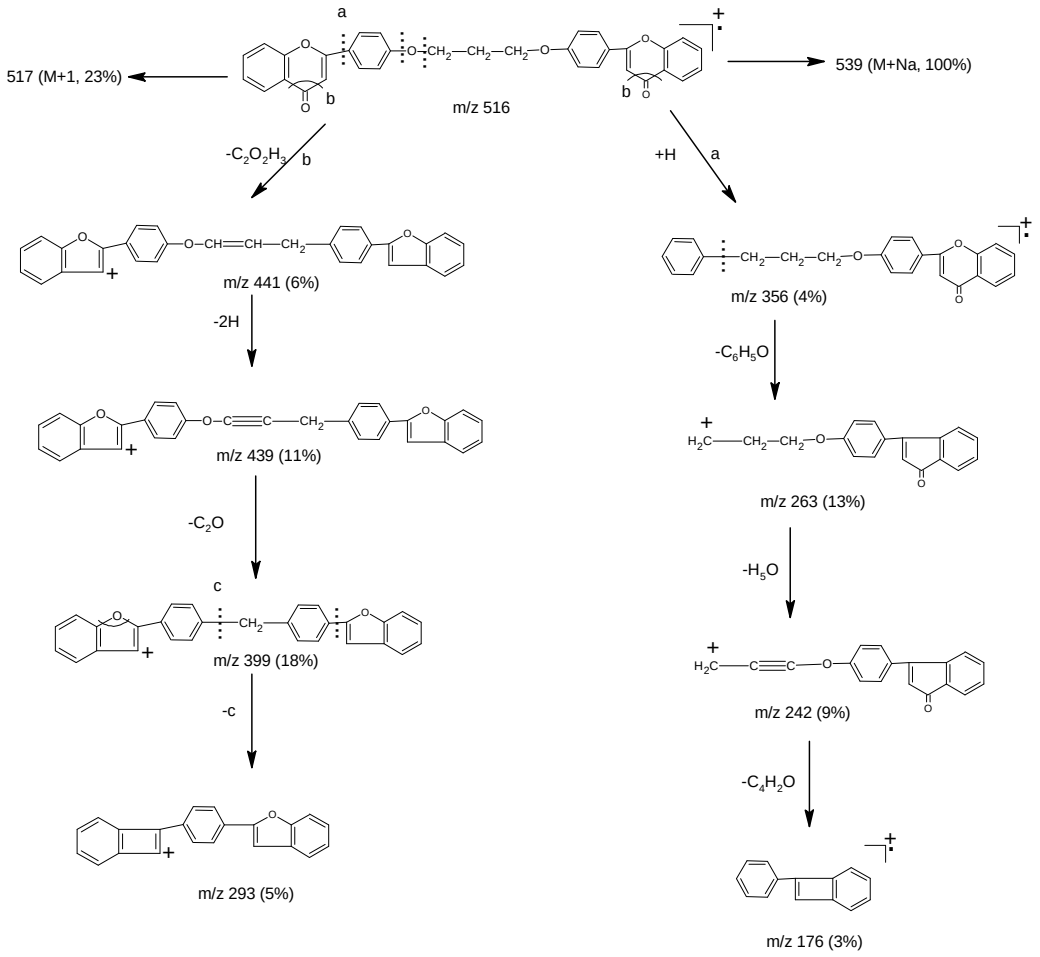


Chart-2

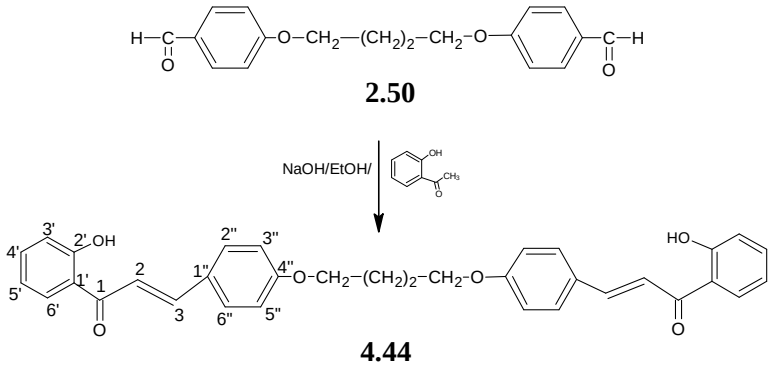
Synthesis of bischromone 4.52

The compound **4.52** was obtained in two steps:

(i). Synthesis of bischalcone 4.44

The compound **4.44** (69%, m.p. 178-180°C) was obtained from the reaction of **2.50** with 1,4-dibromobutane under the similar conditions as used earlier for **4.42** **(Scheme-4.13)**.

IR spectrum of **4.44** showed major bands at 3212 (O-H), 2932, 2845 (methylene C-H), 1600 (C=C) and 1659 (C=O). Its ^{1}H-NMR spectrum (400 MHz, DMSO-d_6) had the characteristics doublets of the *trans* hydrogens H-3 & H-2 at δ 7.84 (2H, J_{trans}=15.1 Hz) and 7.75 (2H, J_{trans}=15.1 Hz) respectively.



2.50

4.44

Scheme-4.13

The hydrogens H-6', H-4', H-5' and H-3' appeared as doublet each at δ 8.40 (2H, J_o=7.7 Hz), 7.94 (2H, J_o=7.3 Hz), 7.65 (2H, J_o=7.6 Hz) and 7.06 (2H, J_o=6.8 Hz) respectively while O***H*** group could provide a D_2O exchangeable two protons singlet at δ 12.93. A triplet present at δ 4.04 (4H, J_{vic}=6.1 Hz) and a quintet centered at δ 1.92 (4H, J_{vic}=6.1 Hz) were assignable to the intervening chain OC***H₂***CH_2 and OCH_2C***H₂*** group hydrogens respectively. Two maxima were also observed in the UV-Vis spectrum of **4.44** at 319 & 264 nm due to the n→π* and π→π* respectively.

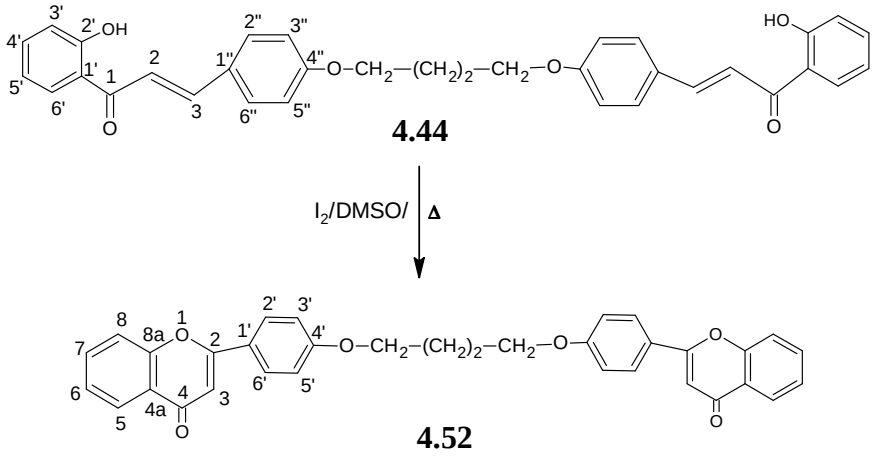
ESI-MS spectrum of **4.44** exhibited noticeable ions at m/z 535 (M+1, 27%), 490 (44%), 489 (100%) and 469 (59%) and its mass fragmentation pattern was found to be similar as shown in **Chart-1** (vide experimental).

In the ^{13}C-NMR (100 MHz, DMSO-d_6) spectrum of **4.44**, important signals were resonating at δ 191.82 (C=O), 157.53 (C-4''), 144.42 (C-3) and 118.02 (C-2). The internal chain could produce two resonances at δ 67.80 (O***C***H_2CH_2) and 27.88 (OCH_2***C***H_2). The carbon atoms of the C-1 and C-3 phenyl rings were also found to be placed at the expected δ values (vide experimental).

(ii). Cyclization of bischalcone 4.44

The compound **4.52** (60%, m.p. 138-140^{o}C) was prepared **(Scheme-4.14)** from the reaction of **4.44** with iodine under the similar conditions as used earlier for **4.50**.

The ESI-MS spectrum of **4.52** also confirmed the proposed expression which exhibited (M+Na) ion at m/z 553 which is also the base peak and its mass fragmentation pattern was found to be similar as shown in **Chart-2** (vide experimental).



Scheme-4.14

The compound **4.52** in its IR spectrum exhibited strong absorption at 1638 cm^{-1} which may be assigned to C=O group of the benzopyrone ring. The significant feature of its ^{1}H-NMR (400 MHz, DMSO-d_6) spectrum was the presence of a singlet at δ 6.74 (2H) which may be given by H-3. The benzenoid ring hydrogens H-5, H-7, H-8 and H-6 were found to be centered at δ 8.13 (2H, dd, $J_{p,o}$=1.0, 8.6 Hz), 7.73 (2H, td, $J_{m,o}$=1.9, 8.6 Hz), 7.44 (2H, d, J_o=8.4 Hz) and 7.06 (2H, d, J_o=7.9 Hz) respectively. The hydrogens H-2', 6' and H-3', 5' provided two doublets at δ 7.94 (4H, J_o=8.9 Hz) and 6.92 (4H, J_o=8.9 Hz). The signals of the OC***H*****$_2$**CH_2 & OCH_2C***H*****$_2$** group protons were resonating at δ 4.15 (4H, t, J_{vic}=5.8 Hz) and 2.03 (4H, quintet, J_{vic}=5.8 Hz) respectively.

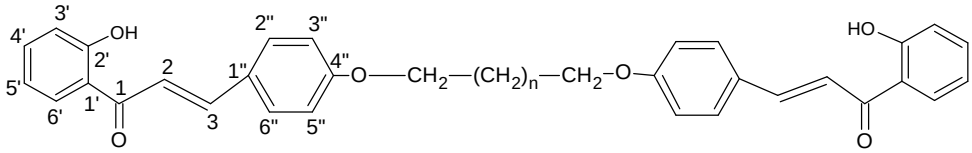
The presence of pyrone ring in **4.52** was confirmed by the appearance of three signals in its ^{13}C-NMR spectrum (100 MHz, DMSO-d_6) at δ 178.30 (C=O), 168.15 (C-2) and 105.68 (C-3) respectively. The carbon atoms C-8a, C-5, C-7, C-4a, C-6 and C-8 resulted six signals at δ 163.82, 133.74, 125.11, 123.79, 123.73 and 118.11 respectively. Additionally, two signals were also found in the aliphatic region at δ 67.37 (O***C***H_2CH_2) & 25.70 (OCH_2***C***H_2) while the signals due to the 2-phenyl ring carbons (C-1', 2', 3', 4', 5', 6') were resonating at the usual positions (vide experimental).

UV-Vis spectrum of **4.52** had two maxima at 316 and 229 nm which may be assigned to n→π* and π→π* transitions respectively. The structural features fully lent support to the propose structure.

Synthesis of bischalcones 4.45-4.49

The compounds **4.45-4.49** were obtained in good yields from the Claisen-Schmidt reactions of dibenzaldehydes **2.51-2.55** with *o*-hydroxyacetophenones under the

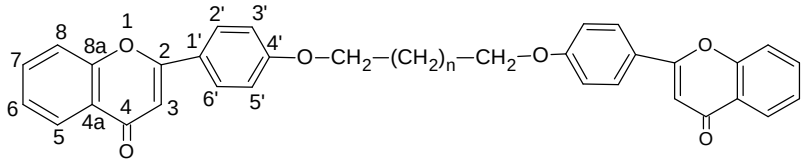
similar conditions as used earlier for **4.42-4.44**. The characteristics spectral data of these compounds have been presented in **Table-1**.



4.45 (n=3), 4.46 (n=4), 4.47 (n=6), 4.48 (n=8), 4.49 (n=10)

Synthesis of bischromones 4.53-4.57

The compounds **4.53-4.57** were prepared from the cyclization reactions of the bischalcones **4.45-4.49** with I_2 under the similar reaction conditions as used earlier for **4.50-4.52**. The physical and characteristics spectral features of these products have been provided in **Table-2**.



4.53 (n=3), **4.54 (n=4)**, **4.55 (n=6)**, **4.56 (n=8)**, **4.57 (n=10)**

Antimicrobial evaluation of 4.42-4.49 & **4.50-4.57**

The newly prepared compounds were screened for their *in vitro* antibacterial and antifungal activity against five bacterial strains namely *Klubsellia pneumoniae* (MTCC 3384), *Pseudomonas aeruginosa* (MTCC 424), *Escherichia coli* (MTCC 443), *Staphylococcus aureus* (MTCC 96), *Bacillius subtilis* (MTCC 441) and four fungi strains *Aspergillius janus* (MTCC 2751), *Aspergillius niger* (MTCC 281), *Aspergillius flavus* (MTCC 277) & *Pencillium glabrum* (MTCC 4951). MIC of these compounds were determined by using serial tube dilution method[43] and these analysis were performed by following the similar procedures as described on page no. 43,44 (**Chapter-IIa**). Amoxicillin and Fluconazole were used as reference drugs for the antibacterial and antifungal activities respectively. The observed minimum inhibitory concentrations (MIC-μg/ml) are given in **Table-3** & **Figure-1 (4.42-4.49)** and **Table-4** & **Figure-2 (4.50-4.57)**.

It is evident from **Table-3** that the compounds **4.43**, **4.45** & **4.47** exhibited significant activity against *Pseudomonas aeruginosa* at MIC of 12.5 μg/ml and the compounds **4.42**, **4.47**, **4.48** & **4.49** were found to have similar activity against *Bacillius subtilis*, *Escherichia coli*, *Klubsellia pneumoniae* and *Aspergillius janus* respectively.

Table-1: Physical and characteristics spectral data of bischalcones 4.45-4.49

Compd.	m.p (°C)	Yield (%)	IR ($\upsilon_{max}cm^{-1}$)	^{1}H-NMR(δ)		^{13}C-NMR(δ)			ESI-MS (m/z)
			C=O	H-3	H-2	C=O	C-3	C-2	
4.45	142-144	65	1657	7.79	7.76	191.84	144.36	118.06	571 $(M+Na)^+$
4.46	120-122	63	1650	7.82	7.75	191.70	144.30	118.21	585 $(M+Na)^+$
4.47	128-130	70	1647	7.81	7.72	190.18	144.01	118.01	591 $(M+1)^+$
4.48	140-142	66	1652	7.83	7.73	190.28	144.11	118.00	641 $(M+Na)^+$
4.49	108-110	72	1654	7.78	7.70	190.19	143.0	118.07	647 $(M+1)^+$

Table-2: Physical and characteristics spectral data of bischromones 4.53-4.57

Compd.	m.p. (°C)	Yield (%)	IR ($\upsilon_{max}cm^{-1}$)	^{1}H-NMR (δ)		^{13}C-NMR (δ)			ESI-MS (m/z)
			C=O	H-8	H-3	C=O	C-2	C-3	
4.53	238-240	57	1643	7.42	6.70	178.18	168.13	105.74	567 $(M+Na)^+$
4.54	124-126	58	1646	7.57	6.72	178.25	168.19	105.96	581 $(M+Na)^+$
4.55	180-182	56	1642	7.46	6.73	177.68	167.90	104.79	587 $(M+1)^+$
4.56	136-138	51	1640	7.40	6.67	177.56	167.53	104.50	615 $(M+1)^+$
4.57	220-222	53	1645	7.43	6.71	177.60	167.48	104.38	643 $(M+1)^+$

.

Table-3. *In vitro* MIC (µg/ml) of bischalcones 4.42-4.49

Compound	Gram negative bacteria			Gram positive bacteria		Fungi			
	Escherichia coli	*Klubssila pneumoniae*	*Pseudomonas aeruginosa*	*Staphylococcus aureus*	*Bacillius subtilis*	*Aspergillus janus*	*Pencilluim glabrum*	*Aspergillus niger*	*Aspergillius flavus*
4.42	25	50	25	25	**12.5**	50	25	50	50
4.43	25	50	**12.5**	25	25	25	25	50	25
4.44	25	50	50	25	25	50	50	25	**12.5**
4.45	25	50	**12.5**	**6.25**	25	25	25	25	50
4.46	25	100	25	25	25	25	50	**12.5**	50
4.47	**12.5**	50	**12.5**	25	**6.25**	25	50	50	**12.5**
4.48	25	**12.5**	25	25	25	50	50	50	**12.5**
4.49	25	50	25	25	25	**12.5**	25	**12.5**	**12.5**
Amoxicillin	3.12	3.12	3.12	3.12	3.12	--	--	--	--
Fluconazole	--	--	--	--	--	6.25	3.12	3.12	6.25

Table-4. *In vitro* MIC (µg/ml) of bischromones 4.50-4.57

Compound	Gram (-ve) bacteria			Gram (+ve) bacteria		Fungi			
	Escherichia coli	*Klubssila pneumoniae*	*Pseudomonas aeruginosa*	*Staphylococcus aureus*	*Bacillius subtilis*	*Aspergillus janus*	*Pencilluim glabrum*	*Aspergillus niger*	*Aspergillius flavus*
4.50	50	50	25	25	50	25	50	50	50
4.51	50	50	25	25	25	50	50	25	50
4.52	25	25	25	**12.5**	50	25	50	50	50
4.53	**12.5**	25	**12.5**	**12.5**	25	**12.5**	25	25	50
4.54	25	**12.5**	50	**6.25**	25	**12.5**	**12.5**	25	50
4.55	**12.5**	25	**12.5**	**12.5**	50	25	**12.5**	**12.5**	**12.5**
4.56	25	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	25	**12.5**	25
4.57	**12.5**	25	**12.5**	25	**12.5**	**12.5**	**12.5**	**12.5**	25
Amoxicillin	3.12	3.12	3.12	3.12	3.12	--	--	--	--
Fluconazole	--	--	--	--	--	6.25	3.12	3.12	6.25

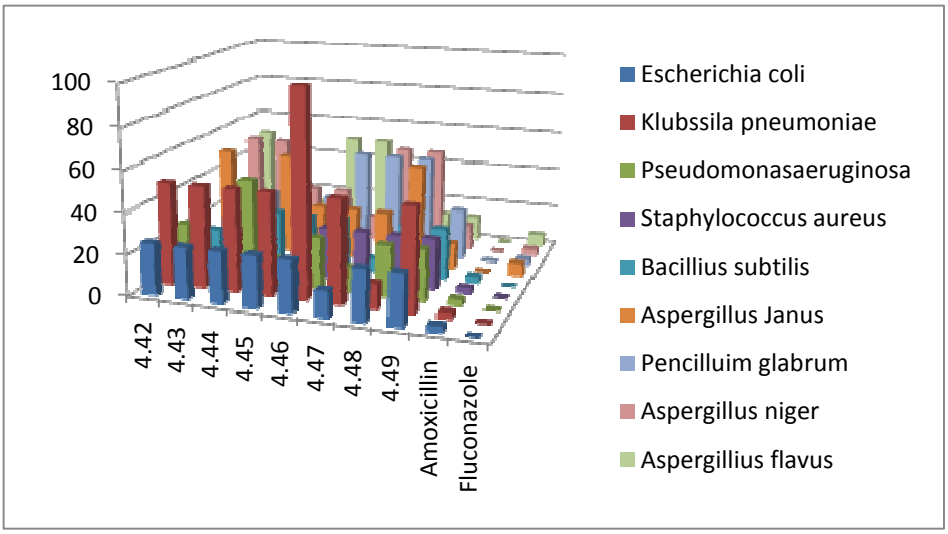


Figure-1. ***In vitro*** **MIC (µg/ml) of bischalcones 4.42-4.49**

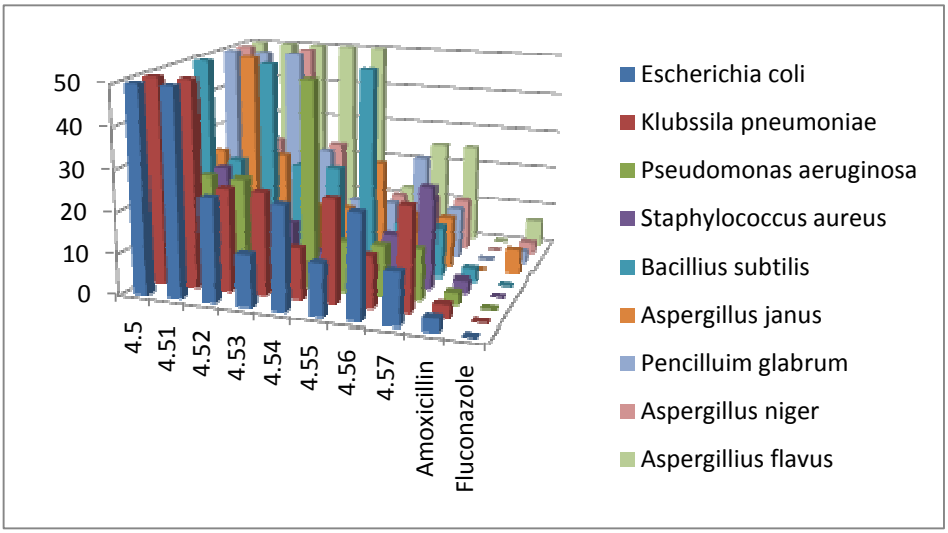


Figure-2. ***In vitro*** **MIC (µg/ml) of bischromones 4.50-4.57**

The compounds **4.45** & **4.47** had MIC of 6.25 µg/ml against *Staphylococcus aureus* & *Bacillius subtilis* respectively and compounds **4.44**, **4.47, 4.48**, **4.49** and **4.46** showed MIC of 12.5 µg/ml against the strains *Aspergillius flavus* and *Aspergillius niger* respectively.

Table-4 describes that bischromones **4.53** and **4.55** provided noticeable activity (MIC-12.5 µg/ml) against *Escherichia coli*, *Pseudomonas aeruginosa*, *Staphylococcus aureus*, *Aspergillius janus* and *Escherichia coli*, *Pseudomonas aeruginosa*, *Staphylococcus aureus*, *Pencillium glabrum*, *Aspergillius niger*, *Aspergillius flavus* respectively. The compounds **4.56** and **4.57** could exhibit the

MIC of similar order against *Klubsellia pneumoniae*, *Pseudomonas aeruginosa*, *Staphylococcus aureus*, *Bacillus subtilis*, *Aspergillius janus*, *Aspergillius niger* and *Escherichia coli*, *Pseudomonas aeruginosa*, *Bacillius subtilis*, *Aspergillius janus*, *Pencillium glabrum* & *Aspergillius niger* respectively. Among the studied compounds, **4.54** was found to be most active (MIC-6.25 μg/ml) against the gram positive bacterial strain *Staphylococcus aureus*.

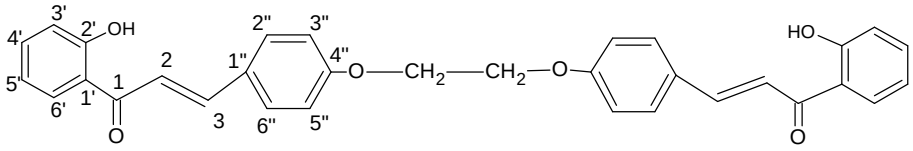
The comparison of **Table-3** and **Table-4** shows that bischromones **4.50-4.57** were found to be better antimicrobial agents than bischalcones **4.42-4.49**.

It may be concluded that present study describes the general and efficient method for the synthesis of new bischromones linked through the 2-phenyl ring under the normal conditions. The length of the intermediate spacer had significant effect upon the antimicrobial behavior of bischromones i.e. the compounds linked through the longer chains were found to be better antimicrobial agents than their shorter chain derivatives.

Experimental

Synthesis of (2*E*,2'*E*)-3,3'-(4,4'-(ethane-1,2-diylbis(oxy))bis(4,1-phenylene))bis(1-(2-hydroxyphenyl)prop-2-en-1-one 4.42

A mixture of *o*-hydroxyacetophenone (0.4634 g, 0.0034 mol), dibenzaldehyde **2.48** (1.0 g, 0.001687 mol) and NaOH (0.01 mol) in EtOH (25.0 ml) was stirred for 12 hrs at room temperature. The reaction was monitored by TLC. After the completion of reaction, the resulting mixture was poured into iced-HCl to provide a crude substance which was crystallized from CH_3OH:$CHCl_3$ (1:1) to yield a pure compound **4.42**.

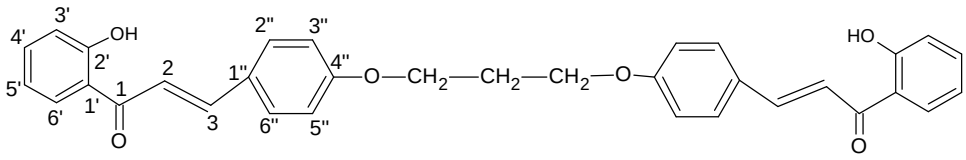


4.42

4.42: Yellow solid; Yield 70%; m.p.: 96-98^oC. UV-Vis (MeOH) λ_{max}(nm): 322, 263; IR (KBr) cm^{-1} 3230 (O-H), 2923, 2856 (methylene C-H), 1658 (C=O), 1598 (C=C); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 12.95 (2H, s, O***H***), 8.56 (2H, d, J_o=7.8 Hz, H-6'), 7.98 (2H, d, J_o=8.1 Hz, H-4'), 7.90 (2H, d, J_{trans}=15.4 Hz, H-3), 7.80 (4H, d, J_o=8.8 Hz, H-2'', 6''), 7.78 (2H, d, J_{trans}=15.4 Hz, H-2), 7.60 (2H, d, J_o=7.5 Hz, H-5'), 7.15 (2H, d, J_o=8.9 Hz, H-3'), 7.11 (4H, d, J_o=8.8 Hz, H-3'', 5''), 4.24 (4H, s, OC***H***$_2$); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 191.87 (C=O), 162.76 (C-2'), 157.54 (C-4''), 144.44 (C-3), 134.32 (C-4'), 128.76 (C-2'', 6''), 127.98 (C-6'), 125.21 (C-1''), 123.72 (C-1'), 121.45 (C-5'), 118.87 (C-3'), 118.12 (C-2), 114.52 (C-3'', 5''), 66.22 (O***C***H$_2$); MS(ESI): m/z 529 (M+Na, 13%), 476 (22%), 475 (100%), 445 (21%), 437 (33%), 426 (8%), 418 (30%), 400 (22%), 399 (52%), 397 (56%), 373 (49%), 344 (13%), 69 (12%). Anal. Calc. for $C_{32}O_6H_{26}$: Calc. C, 75.88 %; H, 5.14 %; Found: C, 76.18 %; H, 5.16 %.

Synthesis of (2*E*,2'*E*)-3,3'-(4,4'-(propane-1,3-diylbis(oxy))bis(4,1-phenylene))bis(1-(2-hydroxyphenyl)prop-2-en-1-one 4.43

The compound **4.43** was synthesized from the reaction of dibenzaldehyde **2.49** (1.0 g, 0.0016611 mol) with *o*-hydroxyacetophenone (0.4523 g, 0.003322 mol) under the similar conditions as used earlier for **4.42**.

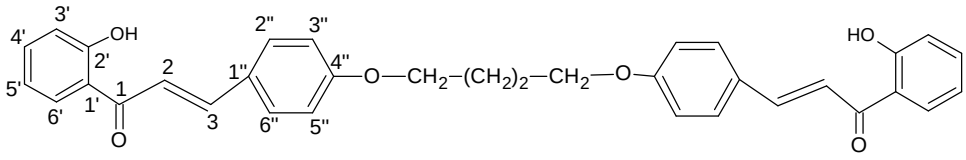


4.43

4.43: Yellow solid; Yield 64%; m.p.: 182-184^{o}C. UV-Vis (MeOH) λ_{max}(nm): 316, 254; IR (KBr) cm^{-1} 3217 (O-H), 2928, 2853 (methylene C-H), 1658 (C=O), 1597 (C=C); ^{1}H-NMR (400 MHz, DMSO-*d*$_6$): δ 12.89 (2H, s, O***H***), 8.52 (2H, d, J_o=7.6 Hz, H-6'), 7.95 (2H, d, J_o=7.4 Hz, H-4'), 7.88 (2H, d, J_{trans}=15.6 Hz, H-3), 7.77 (2H, d, J_{trans}=15.6 Hz, H-2), 7.70 (4H, d, J_o=8.7 Hz, H-2'', 6''), 7.67 (2H, d, J_o=7.2 Hz, H-5'), 7.11 (2H, d, J_o=6.8 Hz, H-3'), 7.03 (4H, d, J_o=8.7 Hz, H-3'', 5''), 4.21 (4H, t, J_{vic}=6.2 Hz, OC***H*****$_2$**CH$_2$), 2.25 (2H, quintet, J_{vic}=6.2 Hz, OCH$_2$C***H*****$_2$**); ^{13}C-NMR (100 MHz, DMSO-*d*$_6$): δ 191.80 (C=O), 162.62 (C-2'), 157.48 (C-4''), 144.40 (C-3), 134.27 (C-4'), 128.74 (C-2'', 6''), 127.95 (C-6'), 125.18 (C-1''), 123.64 (C-1'), 121.41 (C-5'), 118.85 (C-3'), 118.00 (C-2), 114.49 (C-3'', 5''), 68.78 (O***C***H$_2$CH$_2$), 28.17 (OCH$_2$***C***H$_2$); MS(ESI): m/z 543 (M+Na, 35%), 476 (23%), 475 (100%), 455 (8%), 453 (13%), 437 (30%), 425 (18%), 401 (30%), 399 (16%), 387 (32%), 381 (4%), 363 (12%), 327 (11%). Anal. Calc. for $C_{33}O_6H_{28}$: Calc. C, 76.15 %; H, 5.38 %; Found: C, 76.45 %; H, 5.40 %.

Synthesis of (2*E*,2'*E*)-3,3'-(4,4'-(butane-1,4-diylbis(oxy))bis(4,1-phenylene))bis(1-(2-hydroxyphenyl)prop-2-en-1-one 4.44

The compound **4.44** was prepared from the reaction of dibenzaldehyde **2.50** (1.0 g, 0.001623 mol) with *o*-hydroxyacetophenone (0.4420 g, 0.00324 mol) under the same conditions as used for **4.42**.



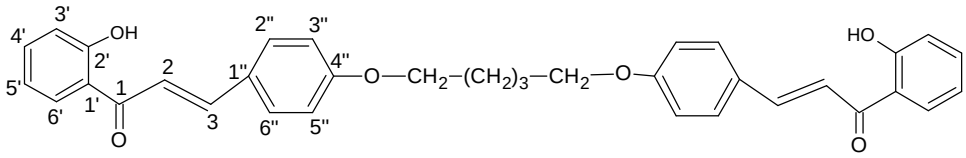
4.44

4.44: Yellow solid; Yield 69%; m.p.: 178-180^{o}C. UV-Vis (MeOH) λ_{max}(nm): 319, 264; IR (KBr) cm^{-1} 3212 (O-H), 2932, 2845 (methylene C-H), 1659 (C=O), 1600 (C=C); ^{1}H-NMR (400 MHz, DMSO-*d*$_6$): δ 12.93 (2H, s, O***H***), 8.40 (2H, d, J_o=7.7 Hz, H-6'), 7.94 (2H, d, J_o=7.3 Hz, H-4'), 7.84 (2H, d, J_{trans}=15.1 Hz, H-3), 7.75 (2H, d, J_{trans}=15.1 Hz, H-2), 7.71 (4H, d, J_o=8.6 Hz, H-2'', 6''), 7.65 (2H, d, J_o=7.6 Hz,

H-5'), 7.06 (2H, d, J_o=6.8 Hz, H-3'), 6.99 (4H, d, J_o=8.6 Hz, H-3'', 5''), 4.04 (4H, t, J_{vic}=6.1 Hz, OC***H₂***CH_2), 1.92 (4H, quintet, J_{vic}=6.1 Hz, OCH_2C***H₂***); ^{13}C-NMR (100 MHz, DMSO-*d₆*): δ 191.82 (C=O), 162.64 (C-2'), 157.53 (C-4''), 144.42 (C-3), 134.30 (C-4'), 128.77 (C-2'', 6''), 127.92 (C-6'), 125.12 (C-1''), 123.70 (C-1'), 121.48 (C-5'), 118.70 (C-3'), 118.02 (C-2), 114.58 (C-3'', 5''), 67.80 (O***C***H_2CH_2), 27.88 (OCH_2***C***H_2); MS(ESI): m/z 535 (M+1, 27%), 490 (44%), 489 (100%), 469 (59%), 467 (10%), 451 (64%), 439 (19%), 416 (71%), 415 (87%), 413 (80%), 399 (60%), 395 (7%), 387 (29%), 363 (31%), 326 (14%). Anal. Calc. for $C_{34}O_6H_{30}$: Calc. C, 76.40 %; H, 5.61 %; Found: C, 76.70 %; H, 5.63 %.

Synthesis of (2*E*,2'*E*)-3,3'-(4,4'-(pentane-1,5-diylbis(oxy))bis(4,1-phenylene))bis(1-(2-hydroxyphenyl)prop-2-en-1-one 4.45

The compound **4.45** was synthesized from the reaction of dibenzaldehyde **2.51** (1.0 g, 0.0015873 mol) with *o*-hydroxyacetophenone (0.43222 g, 0.0031746 mol) under the similar conditions as used for **4.42**.

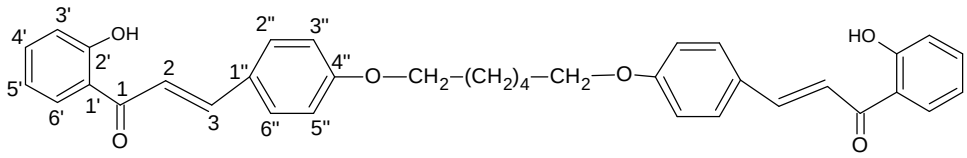


4.45

4.45: Yellow solid; Yield 65%; m.p.: 142-144^{o}C. UV-Vis (MeOH) λ_{max}(nm): 322, 254; IR (KBr) cm^{-1} 3219 (O-H), 2941, 2835 (methylene C-H), 1657 (C=O), 1599 (C=C); ^{1}H-NMR (400 MHz, DMSO-*d₆*): δ 12.90 (2H, s, O***H***), 8.35 (2H, d, J_o=7.8 Hz, H-6'), 7.86 (2H, d, J_o=7.5 Hz, H-4'), 7.79 (2H, d, J_{trans}=15.0 Hz, H-3), 7.76 (2H, d, J_{trans}=15.0 Hz, H-2), 7.70 (4H, d, J_o=8.7 Hz, H-2'', 6''), 7.61 (2H, d, J_o=7.4 Hz, H-5'), 7.04 (2H, d, J_o=7.8 Hz, H-3'), 6.88 (4H, d, J_o=8.7 Hz, H-3'', 5''), 4.00 (4H, t, J_{vic}=6.3 Hz, OC***H₂***CH_2CH_2), 1.83 (4H, quintet, J_{vic}=6.3 Hz, OCH_2C***H₂***CH_2), 1.63 (2H, quintet, J_{vic}=6.3 Hz, OCH_2CH_2C***H₂***); ^{13}C-NMR (100 MHz, DMSO-*d₆*): δ 191.84 (C=O), 162.63 (C-2'), 157.45 (C-4''), 144.36 (C-3), 134.21 (C-4'), 128.70 (C-2'', 6''), 127.88 (C-6'), 125.08 (C-1''), 123.61 (C-1'), 121.37 (C-5'), 118.65 (C-3'), 118.06 (C-2), 114.40 (C-3'', 5''), 66.88 (O***C***$H_2CH_2CH_2$), 27.17 (OCH_2***C***H_2CH_2), 21.60 (OCH_2CH_2***C***H_2); MS(ESI): m/z 571 (M+Na, 81%), 504 (8%), 503 (89%), 483 (17%), 481 (24%), 465 (32%), 453 (40%), 430 (18%), 402 (74%), 399 (61%), 387 (33%), 363 (12%). Anal. Calc. for $C_{35}O_6H_{32}$: Calc. C, 76.64 %; H, 5.83 %; Found: C, 76.95 %; H, 5.85 %.

Synthesis of (2*E*,2'*E*)-3,3'-(4,4'-(hexane-1,6-diylbis(oxy))bis(4,1-phenylene))bis(1-(2-hydroxyphenyl)prop-2-en-1-one 4.46

The compound **4.46** was obtained from the reaction of dibenzaldehyde **2.52** (1.0 g, 0.001552 mol) with *o*-hydroxyacetophenone (0.4228 g, 0.00310559 mol) under the similar conditions as described earlier for **4.42**.

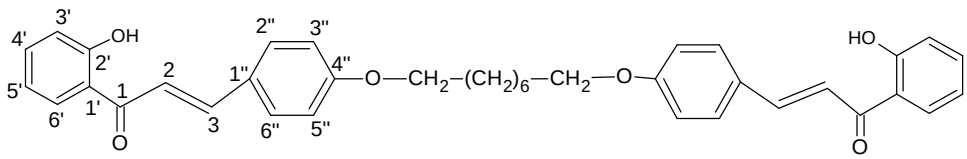


4.46

4.46: Orange solid; Yield 63%; m.p.: 120-122^{o}C. UV-Vis (MeOH) λ_{max}(nm): 317, 259; IR (KBr) cm^{-1} 3225 (O-H), 2954, 2838 (methylene C-H), 1650 (C=O), 1599 (C=C); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 12.89 (2H, s, O***H***), 8.13 (2H, d, J_o=7.9 Hz, H-6'), 7.88 (2H, s, H-4'), 7.82 (2H, d, J_{trans}=15.3 Hz, H-3), 7.75 (2H, d, J_{trans}=15.3 Hz, H-2), 7.72 (4H, d, J_o=8.9 Hz, H-2'', 6''), 7.50 (2H, t, J_o=7.5 Hz, H-5'), 7.01 (2H, d, J_o=7.7 Hz, H-3'), 6.96 (4H, d, J_o=8.9 Hz, H-3'', 5''), 4.06 (4H, t, J_{vic}=6.4 Hz, OC***H₂***CH_2CH_2), 1.80 (4H, quintet, J_{vic}=6.4 Hz, OCH_2C***H₂***CH_2), 1.46 (4H, quintet, J_{vic}=6.4 Hz, OCH_2CH_2C***H₂***); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 191.70 (C=O), 162.58 (C-2'), 157.34 (C-4''), 144.30 (C-3), 134.18 (C-4'), 128.64 (C-2'', 6''), 127.74 (C-6'), 125.13 (C-1''), 123.55 (C-1'), 121.40 (C-5'), 118.68 (C-3'), 118.21 (C-2), 114.13 (C-3'', 5''), 67.39 (O***C***$H_2CH_2CH_2$), 28.77 (OCH_2***C***H_2CH_2), 24.80 (OCH_2CH_2***C***H_2); MS(ESI): m/z 585 (M+Na, 100%), 518 (78%), 517 (72%), 497 (60%), 495 (11%), 479 (44%), 467 (16%), 429 (19%), 402 (38%), 399 (69%), 387 (23%), 375 (58%), 363 (77%). Anal. Calc. For $C_{36}O_6H_{34}$: Calc. C, 76.86 %; H, 6.40 %; Found: C, 77.17 %; H, 6.43 %.

Synthesis of (2*E*,2'*E*)-3,3'-(4,4'-(octane-1,8-diylbis(oxy))bis(4,1-phenylene))bis(1-(2-hydroxyphenyl)prop-2-en-1-one 4.47

The compound **4.47** was prepared from the reaction of dibenzaldehyde **2.53** (1.0 g, 0.0014881 mol) with *o*-hydroxyacetophenone (0.4052 g, 0.002976 mol) under the same conditions as used above for **4.42**.

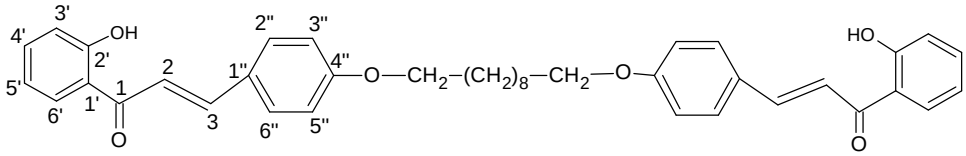


4.47

4.47: Brown solid; Yield 70%; m.p.: 128-130^{o}C. UV-Vis (MeOH) λ_{max}(nm): 320, 260; IR (KBr) cm^{-1} 3218 (O-H), 2972, 2850 (methylene C-H), 1647 (C=O), 1602 (C=C); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 12.92 (2H, s, O***H***), 8.20 (2H, d, J_o=7.8 Hz, H-6'), 7.86 (2H, d, J_o=7.6 Hz, H-4'), 7.81 (2H, d, J_{trans}=15.0 Hz, H-3), 7.72 (2H, d, J_{trans}=15.0 Hz, H-2), 7.63 (4H, d, J_o=8.6 Hz, H-2'', 6''), 7.58 (2H, t, J_o=7.5 Hz, H-5'), 7.03 (2H, d, J_o=8.8 Hz, H-3'), 6.95 (4H, d, J_o=8.6 Hz, H-3'', 5''), 3.95 (4H, t, J_{vic}=6.4 Hz, OC***H$_2$***CH$_2$CH$_2$CH$_2$), 1.80 (4H, quintet, J_{vic}=6.3 Hz, OCH$_2$C***H$_2$***CH$_2$CH$_2$), 1.49 (4H, m, OCH$_2$CH$_2$C***H$_2$***CH$_2$), 1.41 (4H, m, OCH$_2$CH$_2$CH$_2$C***H$_2$***); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 190.18 (C=O), 161.40 (C-2'), 156.11 (C-4''), 144.01 (C-3), 133.07 (C-4'), 128.13 (C-2'', 6''), 127.53 (C-6'), 124.31 (C-1''), 122.16 (C-1'), 120.06 (C-5'), 118.01 (C-2), 117.86 (C-3'), 113.63 (C-3'', 5''), 68.40 (O***C***H$_2$CH$_2$CH$_2$CH$_2$), 28.32 (OCH$_2$***C***H$_2$CH$_2$CH$_2$), 28.00 (OCH$_2$CH$_2$***C***H$_2$CH$_2$), 24.85 (OCH$_2$CH$_2$CH$_2$***C***H$_2$); MS(ESI): m/z 591 (M+1, 40%), 546 (13%), 545 (47%), 525 (30%), 523 (16%), 507 (28%), 495 (70%), 457 (73%), 427 (20%), 415 (61%), 375 (54%), 363 (58%). Anal. Calc. for $C_{38}O_6H_{38}$: Calc. C, 76.25 %; H, 6.44 %; Found: C, 76.55 %; H, 6.47 %.

Synthesis of (2*E*,2'*E*)-3,3'-(4,4'-(decane-1,10-diylbis(oxy))bis(4,1-phenylene))bis(1-(2-hydroxyphenyl)prop-2-en-1-one 4.48

The compound **4.48** was synthesized by reacting dibenzaldehyde **2.54** (1.0 g, 0.0014286 mol) with *o*-hydroxyacetophenone (0.1945 g, 0.0028 mol) under the similar conditions as described earlier for **4.42**.



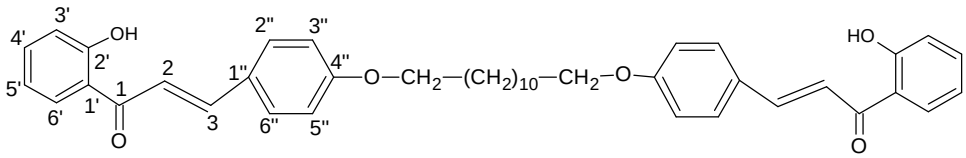
4.48

4.48: Yellow solid; Yield 66%; m.p.: 140-142^{o}C. UV-Vis (MeOH) λ_{max}(nm): 327, 252; IR (KBr) cm^{-1} 3216 (O-H), 2932, 2851 (methylene C-H), 1652 (C=O), 1601 (C=C); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 12.90 (2H, s, O***H***), 8.15 (2H, d, J_o=7.9 Hz, H-6'), 7.93 (2H, d, J_o=6.8 Hz, H-4'), 7.83 (2H, d, J_{trans}=15.2 Hz, H-3), 7.73 (2H, d, J_{trans}=15.2 Hz, H-2), 7.70 (4H, d, J_o=8.9 Hz, H-2'', 6''), 7.50 (2H, t, J_o=7.5 Hz, H-5'), 7.08 (2H, d, J_o=7.6 Hz, H-3'), 6.96 (4H, d, J_o=8.9 Hz, H-3'', 5''), 3.90 (4H, t, J_{vic}=6.4 Hz, OC***H$_2$***CH$_2$CH$_2$CH$_2$CH$_2$), 1.70 (4H, quintet, J_{vic}=6.4 Hz, OCH$_2$C***H$_2$***CH$_2$CH$_2$CH$_2$), 1.32 (4H, m, OCH$_2$CH$_2$C***H$_2$***CH$_2$CH$_2$), 1.24 (4H, m,

$OCH_2CH_2CH_2C\boldsymbol{H_2}CH_2$), 1.20 (4H, m, $OCH_2CH_2CH_2CH_2C\boldsymbol{H_2}$); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 190.28 (C=O), 161.20 (C-2'), 156.13 (C-4''), 144.11 (C-3), 133.00 (C-4'), 128.27 (C-2'', 6''), 127.60 (C-6'), 124.13 (C-1''), 122.20 (C-1'), 120.17 (C-5'), 118.00 (C-2), 117.66 (C-3'), 113.59 (C-3'', 5''), 67.90 ($O\boldsymbol{C}H_2CH_2CH_2CH_2CH_2$), 28.62 ($OCH_2\boldsymbol{C}H_2CH_2CH_2CH_2$), 28.24 ($OCH_2CH_2\boldsymbol{C}H_2CH_2CH_2$), 28.13 ($OCH_2CH_2CH_2\boldsymbol{C}H_2CH_2$), 25.12 ($OCH_2CH_2CH_2CH_2\boldsymbol{C}H_2$); MS(ESI): m/z 641 (M+Na, 100%), 574 (82%), 573 (17%), 553 (28%), 551 (44%), 523 (13%), 499 (64%), 497 (60%), 479 (29%), 363 (9%), 326 (68%), 310 (7%). Anal. Calc. for $C_{40}O_6H_{42}$: Calc. C, 77.66 %; H, 6.79 %; Found: C, 77.97 %; H, 6.82 %.

Synthesis of (2*E*,2'*E*)-3,3'-(4,4'-(dodecane-1,12-diylbis(oxy))bis(4,1-phenylene))bis(1-(2-hydroxyphenyl)prop-2-en-1-one 4.49

The compound **4.49** was synthesized by reacting dibenzaldehyde **2.55** (1.0 g, 0.0013 mol) with *o*-hydroxyacetophenone (0.1870 g, 0.0026 mol) under the similar conditions as described earlier for **4.42**.



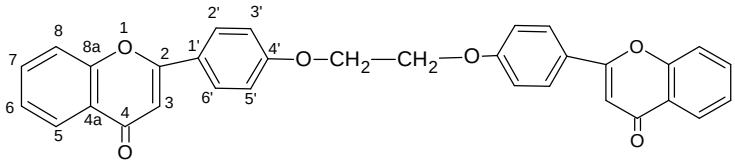
4.49

4.49: Brown solid; Yield 72%; m.p.: 108-110^{o}C. UV-Vis (MeOH) λ_{max}(nm): 312, 263; IR (KBr) cm^{-1} 3211 (O-H), 2937, 2849 (methylene C-H), 1654 (C=O), 1600 (C=C); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 12.85 (2H, s, O***H***), 8.00 (2H, d, J_o=7.9 Hz, H-6'), 7.90 (2H, d, J_o=8.6 Hz, H-4'), 7.78 (2H, d, J_{trans}=15.3 Hz, H-3), 7.70 (2H, d, J_{trans}=15.3 Hz, H-2), 7.58 (4H, d, J_o=8.9 Hz, H-2'', 6''), 7.44 (2H, t, J_o=7.5 Hz, H-5'), 7.06 (2H, d, J_o=7.8 Hz, H-3'), 6.90 (4H, d, J_o=8.9 Hz, H-3'', 5''), 3.99 (4H, t, J_{vic}=6.4 Hz, $OC\boldsymbol{H_2}CH_2CH_2CH_2CH_2CH_2$), 2.78 (4H, quintet, J_{vic}=6.4 Hz, $OCH_2C\boldsymbol{H_2}CH_2CH_2CH_2CH_2$), 2.13 (4H, m, $OCH_2CH_2C\boldsymbol{H_2}CH_2CH_2CH_2$), 1.45 (4H, m, $OCH_2CH_2CH_2C\boldsymbol{H_2}CH_2CH_2$), 1.30 (8H, m, $OCH_2CH_2CH_2CH_2C\boldsymbol{H_2}C\boldsymbol{H_2}$); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 190.19 (C=O), 161.12 (C-2'), 156.02 (C-4''), 143.0 (C-3), 133.27 (C-4'), 128.03 (C-2'', 6''), 127.26 (C-6'), 124.25 (C-1''), 122.03 (C-1'), 120.0 (C-5'), 118.07 (C-2), 117.52 (C-3'), 113.48 (C-3'', 5''), 67.80 ($O\boldsymbol{C}H_2CH_2CH_2CH_2CH_2CH_2$), 28.60 ($OCH_2\boldsymbol{C}H_2CH_2CH_2CH_2CH_2$), 28.38 ($OCH_2CH_2\boldsymbol{C}H_2CH_2CH_2CH_2$), 28.00 ($OCH_2CH_2CH_2\boldsymbol{C}H_2CH_2CH_2$), 27.87

($OCH_2CH_2CH_2CH_2\boldsymbol{C}H_2CH_2$), 25.23 ($OCH_2CH_2CH_2CH_2CH_2\boldsymbol{C}H_2$); MS(ESI): m/z 647 (M+1, 40%), 602 (61%), 601 (100%), 581 (45%), 579 (39%), 551 (12%), 527 (30%), 525 (68%), 513 (18%), 507 (80%), 452 (7%), 363 (28%), 326 (10%), 298 (35%), 222 (46%), 206 (91%). Anal. Calc. for $C_{42}O_6H_{46}$: Calc. C, 78.01 %; H, 7.12 %; Found: C, 78.32 %; H, 7.15 %.

Synthesis of 2,2'-(4,4'-(ethane-1,2-diylbis(oxy))bis(4,1-phenylene))bis(4*H*-chromen-4-one) 4.50

A mixture of bischalcone **4.42** (1.0 g, 0.001976 mol) and iodide (1.25 g, 0.0049 mol) in DMSO (20 ml) was refluxed for 4 hrs. The progress of reaction was monitored by TLC. After the completion of reaction, the reaction mixture was poured into ice to obtain a solid substance. The crude product thus obtained was crystallized from MeOH to obtain a pure compound **4.50**.

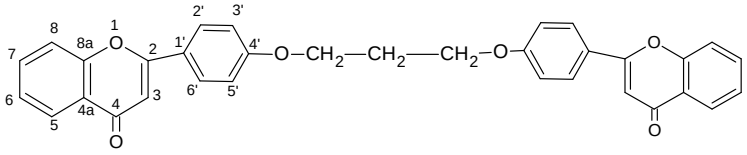


4.50

4.50: Brown solid; Yield 52%; m.p.: 256-258^oC. UV-Vis (MeOH) λ_{max}(nm): 312, 228; IR (KBr) cm^{-1} 2960, 2852 (methylene C-H), 1640 (C=O); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 8.14 (2H, dd, $J_{p,o}$=1.1, 8.6 Hz, H-5), 7.72 (4H, d, J_o=8.8 Hz, H-2', 6'), 7.59 (2H, td, $J_{m,o}$=1.8, 8.3 Hz, H-7), 7.50 (2H, d, J_o=8.3 Hz, H-8), 7.44 (2H, t, J_o=7.7 Hz, H-6), 6.93 (4H, d, J_o=8.8 Hz, H-3', 5'), 6.74 (2H, s, H-3), 4.15 (4H, s, $OC\boldsymbol{H_2}$); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 178.52 (C=O), 168.10 (C-2), 163.94 (C-8a), 156.23 (C-4'), 133.65 (C-5), 128.09 (C-1'), 125.70 (C-2', 6'), 125.16 (C-7), 123.92 (C-4a), 123.60 (C-6), 117.98 (C-8), 114.97 (C-3', 5'), 106.25 (C-3), 64.50 ($O\boldsymbol{C}H_2$); MS(ESI): m/z 503 (M+1, 87%), 427 (10%), 425 (33%), 385 (100%), 342 (23%), 293 (9%), 249 (28%), 228 (6%), 176 (39%). Anal. Calc. for $C_{32}O_6H_{22}$: Calc. C, 76.49 %; H, 4.38 %; Found: C, 76.79 %; H, 4.40 %.

Synthesis of 2,2'-(4,4'-(propane-1,3-diylbis(oxy))bis(4,1-phenylene))bis(4*H*-chromen-4-one) 4.51

The compound **4.51** was synthesized by reacting bischalcone **4.33** (1.0 g, 0.001923 mol) with iodine (1.21 g, 0.00480 mol) under the similar conditions as described earlier for **4.50**.

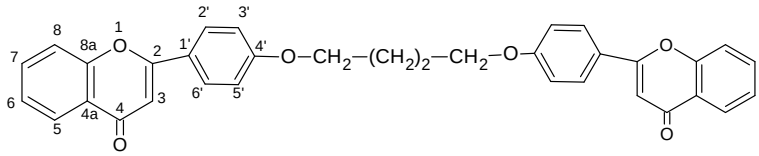


4.51

4.51: Brown solid; Yield 61%; m.p.: 210-212^{o}C. UV-Vis (MeOH) λ_{max}(nm): 310, 225; IR (KBr) cm^{-1} 2945, 2883 (methylene C-H), 1641 (C=O); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 8.14 (2H, dd, $J_{p,o}$=1.0, 8.7 Hz, H-5), 7.82 (4H, d, J_o=8.9 Hz, H-2', 6'), 7.62 (2H, d, J_o=8.6 Hz, H-7), 7.48 (2H, d, J_o=8.4 Hz, H-8), 7.34 (2H, d, J_o=7.9 Hz, H-6), 6.98 (4H, d, J_o=8.9 Hz, H-3', 5'), 6.69 (2H, s, H-3), 4.21 (4H, t, J_o=6.0 Hz, OC***H₂***CH$_2$), 2.29 (2H, t, J_{vic}=6.0 Hz, OCH$_2$C***H₂***); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 178.05 (C=O), 168.00 (C-2), 163.94 (C-8a), 156.10 (C-4'), 133.65 (C-5), 128.09 (C-1'), 125.70 (C-2', 6'), 125.16 (C-7), 124.21 (C-4a), 123.92 (C-6), 117.98 (C-8), 114.97 (C-3', 5'), 106.25 (C-3), 67.50 (O***C***H$_2$CH$_2$), 25.78 (OCH$_2$***C***H$_2$); MS(ESI): m/z 539 (M+Na, 100%), 517 (M+1, 23%), 441 (6%), 439 (11%), 399 (18%), 356 (4%), 293 (5%), 263 (13%), 242 (9%), 176 (3%). Anal. Calc. for $C_{33}O_6H_{24}$: Calc. C, 76.74 %; H, 4.65 %; Found: C, 77.05 %; H, 4.67 %.

Synthesis of 2,2'-(4,4'-(butane-1,4-diylbis(oxy))bis(4,1-phenylene))bis(4*H*-chromen-4-one) 4.52

The compound **4.52** was obtained by reacting bischalcone **4.44** (1.0 g, 0.001872 mol) with iodine (1.18 g, 0.00468 mol) under the similar conditions as described earlier for **4.50**.



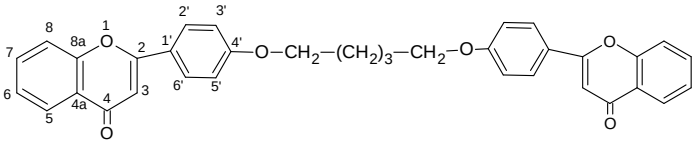
4.52

4.52: Brown solid; Yield 60%; m.p.: 138-140^{o}C. UV-Vis (MeOH) λ_{max}(nm): 316, 229; IR (KBr) cm^{-1} 2938, 2822 (methylene C-H), 1638 (C=O); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 8.13 (2H, dd, $J_{p,o}$=1.0, 8.6 Hz, H-5), 7.94 (4H, d, J_o=8.9 Hz, H-2', 6'), 7.73 (2H, td, $J_{m,o}$=1.9, 8.6 Hz, H-7), 7.44 (2H, d, J_o=8.4 Hz, H-8), 7.06 (2H, d, J_o=7.9 Hz, H-6), 6.92 (4H, d, J_o=8.9 Hz, H-3', 5'), 6.74 (2H, s, H-3), 4.15 (4H, t, J_{vic}=5.8 Hz, OC***H₂***CH$_2$), 2.03 (4H, quintet, J_{vic}=5.8 Hz, OCH$_2$C***H₂***); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 178.30 (C=O), 168.15 (C-2), 163.82 (C-8a), 156.00 (C-4'), 133.74 (C-5), 128.00 (C-1'), 125.58 (C-2', 6'), 125.11 (C-7), 123.79 (C-4a), 123.73

(C-6), 118.11 (C-8), 113.95 (C-3', 5'), 105.68 (C-3), 67.37 (O***C***H_2CH_2), 25.70 (OCH_2***C***H_2); MS(ESI): m/z 553 (M+Na, 100%), 455 (43%), 453 (36%), 413 (10%), 370 (20%), 307 (79%), 293 (50%), 277 (41%), 256 (30%), 176 (34%). Anal. Calc. for $C_{34}O_6H_{26}$: Calc. C, 76.98 %; H, 4.90 %; Found: C, 77.29 %; H, 4.92 %.

Synthesis of 2,2'-(4,4'-(pentane-1,5-diylbis(oxy))bis(4,1-phenylene))bis(4*H*-chromen-4-one) 4.53

The compound **4.53** was prepared by reacting bischalcone **4.45** (1.0 g, 0.001824 mol) with iodine (1.154 g, 0.0045 mol) under the similar conditions as used earlier for **4.50**.

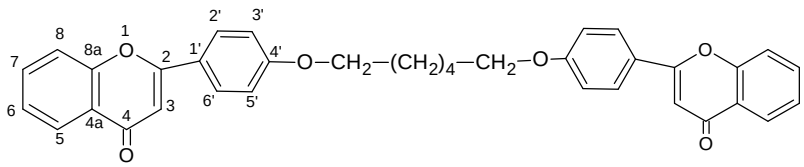


4.53

4.53: Brown solid; Yield 57%; m.p.: 238-240^{o}C. UV-Vis (MeOH) λ_{max}(nm): 309, 220; IR (KBr) cm^{-1} 2944, 2832 (methylene C-H), 1643 (C=O); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 8.16 (2H, dd, $J_{p,o}$=1.1, 8.7 Hz, H-5), 7.78 (4H, d, J_o=8.9 Hz, H-2', 6'), 7.76 (2H, td, $J_{m,o}$=1.9, 8.6 Hz, H-7), 7.42 (2H, d, J_o=8.4 Hz, H-8), 7.26 (2H, t, J_o=7.9 Hz, H-6), 6.96 (4H, d, J_o=8.9 Hz, H-3', 5'), 6.70 (2H, s, H-3), 3.98 (4H, t, J_{vic}=5.6 Hz, OC***H₂***CH_2CH_2), 1.98 (4H, quintet, J_{vic}=5.6 Hz, OCH_2C***H₂***CH_2), 1.60 (2H, quintet, J_{vic}=5.0 Hz, OCH_2CH_2C***H₂***); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 178.18 (C=O), 168.13 (C-2), 163.79 (C-8a), 156.29 (C-4'), 133.69 (C-5), 128.13 (C-1'), 125.40 (C-2', 6'), 125.14 (C-7), 123.81 (C-4a), 123.64 (C-6), 118.16 (C-8), 113.90 (C-3', 5'), 105.74 (C-3), 67.30 (O***C***$H_2CH_2CH_2$), 27.10 (OCH_2***C***H_2CH_2), 22.78 (OCH_2CH_2***C***H_2); MS(ESI): m/z 567 (M+Na, 47%), 469 (11%), 467 (19%), 427 (23%), 384 (53%), 293 (46%), 291 (100%), 270 (62%), 176 (78%). Anal. Calc. for $C_{35}O_6H_{28}$: Calc. C, 77.20 %; H, 5.14 %; Found: C, 77.51 %; H, 5.16 %.

Synthesis of 2,2'-(4,4'-(hexane-1,6-diylbis(oxy))bis(4,1-phenylene))bis(4*H*-chromen-4-one) 4.54

The compound **4.54** was synthesized by reacting bischalcone **4.46** (1.0 g, 0.001779 mol) with iodine (1.125 g, 0.00445 mol) under the similar conditions as described earlier for **4.50**.

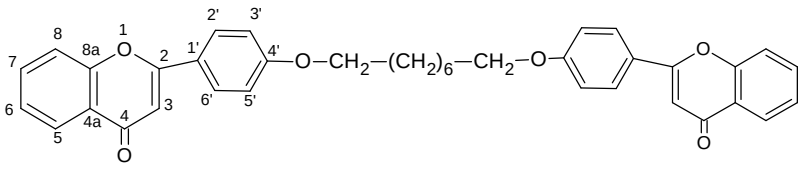


4.54

4.54: Brown solid; Yield 58%; m.p.: 124-126^{o}C. UV-Vis (MeOH) λ_{max}(nm): 314, 228; IR (KBr) cm^{-1} 2972, 2850 (methylene C-H), 1646 (C=O); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 8.11 (2H, dd, $J_{p,o}$=1.0, 8.6 Hz, H-5), 7.85 (4H, d, J_o=8.9 Hz, H-2', 6'), 7.65 (2H, td, $J_{m,o}$=1.8, 8.6 Hz, H-7), 7.57 (2H, d, J_o=8.2 Hz, H-8), 7.30 (2H, t, J_o=7.9 Hz, H-6), 6.94 (4H, d, J_o=8.9 Hz, H-3', 5'), 6.72 (2H, s, H-3), 3.93 (4H, t, J_{vic}=6.0 Hz, OC***H₂***CH_2CH_2), 1.75 (4H, quintet, J_{vic}=6.0 Hz, OCH_2C***H₂***CH_2), 1.48 (4H, quintet, J_{vic}=6.0 Hz, OCH_2CH_2C***H₂***); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 178.25 (C=O), 168.19 (C-2), 163.88 (C-8a), 156.00 (C-4'), 133.70 (C-5), 128.23 (C-1'), 125.54 (C-2', 6'), 125.10 (C-7), 123.86 (C-4a), 123.70 (C-6), 118.25 (C-8), 114.16 (C-3', 5'), 105.96 (C-3), 67.3 (O***C***$H_2CH_2CH_2$), 27.13 (OCH_2***C***H_2CH_2), 24.16 (OCH_2CH_2***C***H_2); MS(ESI): m/z 581 (M+Na, 17%), 483 (27%), 481 (38%), 457 (14%), 398 (100%), 305 (3%), 293 (49%), 284 (76%), 176 (24%). Anal. Calc. for $C_{36}O_6H_{30}$: Calc. C, 77.41 %; H, 5.37 %; Found: C, 77.72 %; H, 5.39 %.

Synthesis of 2,2'-(4,4'-(octane-1,8-diylbis(oxy))bis(4,1-phenylene))bis(4*H*-chromen-4-one) 4.55

The compound **4.55** was obtained by reacting bischalcone **4.47** (1.0 g, 0.00169 mol) with iodine (1.072 g, 0.004237 mol) under the similar conditions as described earlier for **4.50**.



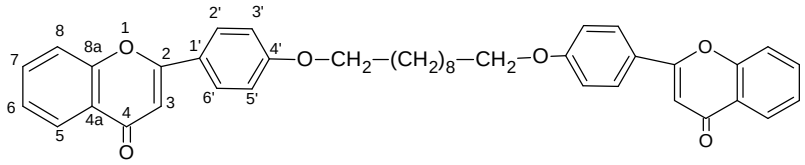
4.55

4.55: Brown solid; Yield 56%; m.p.: 180-182^{o}C. UV-Vis (MeOH) λ_{max}(nm): 313, 220; IR (KBr) cm^{-1} 2934, 2843 (methylene C-H), 1642 (C=O); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 8.18 (2H, dd, $J_{p,o}$=1.0, 8.5 Hz, H-5), 7.90 (4H, d, J_o=8.8 Hz, H-2', 6'), 7.75 (2H, td, $J_{m,o}$=1.9, 8.6 Hz, H-7), 7.46 (2H, d, J_o=8.3 Hz, H-8), 7.00 (2H, t, J_o=7.8 Hz, H-6), 6.93 (4H, d, J_o=8.9 Hz, H-3', 5'), 6.73 (2H, s, H-3), 3.90 (4H, t, J_{vic}=6.2 Hz, OC***H₂***$CH_2CH_2CH_2$), 1.73 (4H, quintet, J_{vic}=6.2 Hz, OCH_2C***H₂***CH_2CH_2), 1.40 (4H, m, OCH_2CH_2C***H₂***CH_2), 1.38 (4H, m, $OCH_2CH_2CH_2C$***H₂***); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 177.68 (C=O), 167.90 (C-2), 162.81 (C-8a), 155.29 (C-4'), 132.58 (C-5), 126.26 (C-1'), 124.20 (C-2', 6'), 124.18 (C-7), 123.82 (C-6), 122.41 (C-4a), 117.60 (C-8), 113.68 (C-3', 5'), 104.79 (C-3), 67.3 (O***C***$H_2CH_2CH_2CH_2$), 27.19 (OCH_2***C***$H_2CH_2CH_2$), 27.00 (OCH_2CH_2***C***H_2CH_2), 25.82 ($OCH_2CH_2CH_2$***C***H_2);

MS(ESI): m/z 587 (M+1, 26%), 511 (40%), 509 (100%), 485 (28%), 426 (49%), 333 (10%), 312 (14%), 293 (69%), 176 (23%). Anal. Calc. for $C_{38}O_6H_{34}$: Calc. C, 77.81 %; H, 5.80 %; Found: C, 78.12 %; H, 5.82 %.

Synthesis of 2,2'-(4,4'-(decane-1,10-diylbis(oxy))bis(4,1-phenylene))bis(4*H*-chromen-4-one) 4.56

The compound **4.56** was prepared by reacting bischalcone **4.48** (1.0 g, 0.001618 mol) with iodine (1.023 g, 0.004045 mol) under the similar conditions as described earlier for **4.50**.

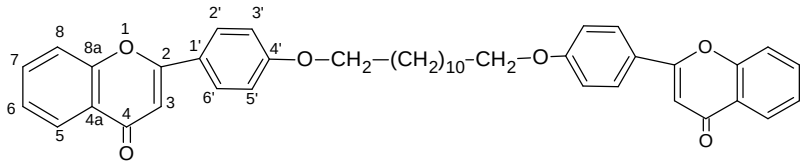


4.56

4.56: Brown solid; Yield 51%; m.p.: 136-138^{o}C. UV-Vis (MeOH) λ_{max}(nm): 313, 252; IR (KBr) cm^{-1} 2960, 2823 (methylene C-H), 1640 (C=O); ^{1}H-NMR (400 MHz, DMSO-*d*$_6$): δ 8.10 (2H, dd, $J_{p,o}$=1.1, 8.8 Hz, H-5), 7.92 (4H, d, J_o=8.6 Hz, H-2', 6'), 7.73 (2H, td, $J_{m,o}$=1.8, 8.6 Hz, H-7), 7.40 (2H, d, J_o=8.2 Hz, H-8), 7.02 (2H, t, J_o=7.7 Hz, H-6), 6.90 (4H, d, J_o=8.6 Hz, H-3', 5'), 6.67 (2H, s, H-3), 3.90 (4H, t, J_{vic}=6.0 Hz, OC***H*****$_2$**$CH_2CH_2CH_2CH_2$), 1.70 (4H, quintet, J_{vic}=6.0 Hz, OCH_2C***H*****$_2$**$CH_2CH_2CH_2$), 1.39 (4H, quintet, J_{vic}=6.0 Hz, OCH_2CH_2C***H*****$_2$**CH_2CH_2), 1.28 (4H, m, $OCH_2CH_2CH_2C$***H*****$_2$**CH_2), 1.20 (4H, m, $OCH_2CH_2CH_2CH_2C$***H*****$_2$**); ^{13}C-NMR (100 MHz, DMSO-*d*$_6$): δ 177.56 (C=O), 167.53 (C-2), 162.74 (C-8a), 155.10 (C-4'), 132.60 (C-5), 126.50 (C-1'), 124.63 (C-7), 124.52 (C-2', 6'), 123.87 (C-6), 122.84 (C-4a), 117.90 (C-8), 113.59 (C-3', 5'), 104.50 (C-3), 67.04 (O***C***$H_2CH_2CH_2CH_2CH_2$), 28.76 (OCH_2***C***$H_2CH_2CH_2CH_2$), 28.45 (OCH_2CH_2***C***$H_2CH_2CH_2$), 28.00 ($OCH_2CH_2CH_2$***C***H_2CH_2), 24.81 ($OCH_2CH_2CH_2CH_2$***C***H_2); MS(ESI): m/z 615 (M+1, 36%), 539 (10%), 537 (86%), 513 (54%), 454 (100%), 361 (17%), 340 (69%), 293 (18%), 217 (27%), 201 (77%), 176 (17%). Anal. Calc. for $C_{40}O_6H_{38}$: Calc. C, 78.17 %; H, 6.18 %; Found: C, 78.46 %; H, 6.20 %.

Synthesis of 2,2'-(4,4'-(dodecane-1,12-diylbis(oxy))bis(4,1-phenylene))bis(4*H*-chromen-4-one) 4.57

The compound **4.57** was synthesized by reacting bischalcone **4.49** (1.0 g, 0.001547 mol) with iodine (0.9791 g, 0.00386 mol) under the similar conditions as described earlier for **4.50**.



4.57

4.57: Brown solid; Yield 53%; m.p.: 220-222^{o}C. UV-Vis (MeOH) λ_{max}(nm): 315, 257; IR (KBr) cm^{-1} 2919, 2834 (methylene C-H), 1645 (C=O); ^{1}H-NMR (400 MHz, DMSO-d_6): δ 8.13 (2H, dd, $J_{p,o}$=1.1, 8.5 Hz, H-5), 7.80 (4H, d, J_{o}=8.7 Hz, H-2', 6'), 7.60 (2H, td, $J_{m,o}$=1.9, 8.6 Hz, H-7), 7.43 (2H, d, J_{o}=8.3 Hz, H-8), 7.28 (2H, t, J_{o}=7.9 Hz, H-6), 6.92 (4H, d, J_{o}=8.7 Hz, H-3', 5'), 6.71 (2H, s, H-3), 3.88 (4H, t, J_{vic}=6.0 Hz, OC***H*****$_2$**$CH_2CH_2CH_2CH_2CH_2$), 1.65 (4H, quintet, J_{vic}=6.4 Hz, OCH_2C***H*****$_2$**$CH_2CH_2CH_2CH_2$), 1.32 (4H, quintet, J_{vic}=6.0 Hz, CH_2CH_2C***H*****$_2$**$CH_2CH_2CH_2$), 1.22 (4H, m, $OCH_2CH_2CH_2C$***H*****$_2$**CH_2CH_2), 1.18 (8H, m, $OCH_2CH_2CH_2CH_2C$***H*****$_2$**C***H*****$_2$**); ^{13}C-NMR (100 MHz, DMSO-d_6): δ 177.60 (C=O), 167.48 (C-2), 162.88 (C-8a), 155.32 (C-4'), 132.13 (C-5), 126.63 (C-1'), 124.67 C-2', 6'), 124.52 (C-7), 123.80 (C-6), 122.69 (C-4a), 117.40 (C-8), 113.70 (C-3', 5'), 104.38 (C-3), 68.10 (O***C***$H_2CH_2CH_2CH_2CH_2CH_2$), 28.38 (OCH_2***C***$H_2CH_2CH_2CH_2CH_2$), 28.15 (OCH_2CH_2***C***$H_2CH_2CH_2CH_2$), 27.90 ($OCH_2CH_2CH_2$***C***$H_2CH_2CH_2$), 27.34 ($OCH_2CH_2CH_2CH_2$***C***H_2CH_2), 24.64 ($OCH_2CH_2CH_2CH_2CH_2$***C***H_2); MS(ESI): m/z 643 (M+1, 100%), 567 (10%), 565 (22%), 541 (76%), 482 (46%), 389 (49%), 340 (57%), 293 (69%), 217 (15%), 201 (19%). Anal. Calc. for $C_{42}O_6H_{42}$: Calc. C, 78.50 %; H, 6.54 %; Found: C, 78.81 %; H, 6.57 %.

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