First Synthesis of 2-(2,4,4-Trimethyl-3,4-dihydro-2*H*-benzo[*h*]chromen-2-yl)-1-naphthol and 3-(2,4,4-Trimethyl-3,4-dihydro-2*H*-benzo[*g*]chromen-2-yl)-2-naphthol from 1- and 2-Naphthol Derivatives

Jose C. J. M. D. S. Menezes,[a] Bikshandarkoil R. Srinivasan,[a] Pallepogu Raghavaiah,[b] Shashikumar K. Paknikar,[c] and Shrivallabh P. Kamat[a]*

[a]Department of Chemistry, Goa University, Taleigao, Goa 403 206 India
[b]School of Chemistry, National Single Crystal X-ray Diffractometer Facility, University of Hyderabad, Hyderabad 500 046, India
[c]R & D Laboratory, Siddharth Chemicals, Kundai Industrial Estate, Kundai, Goa 403 115, India
*E-mail: shrivkamat@yahoo.com

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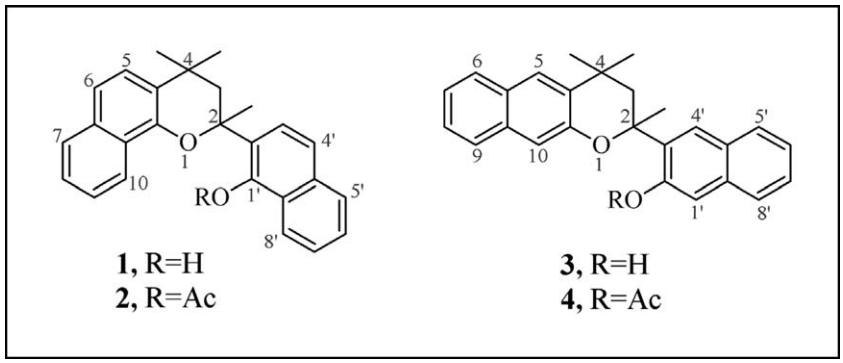

Two new structurally isomeric, 2-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*h*]chromen-2-yl)-1-naphthol (**1**) and 3-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*g*]chromen-2-yl)-2-naphthol (**3**) have been synthesized from 2-acetyl-1-naphthol and ethyl-3-hydroxy-2-naphthoate, respectively, involving Grignard reaction, dehydration of the corresponding tertiary alcohols, and hetero Diels–Alder dimerization. The two benzochromenes (**1** and **3**) have been fully characterized by IR, NMR, and HRESIMS data. Their structures are further supported by crystallography of their corresponding acetates (**2** and **4**).

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INTRODUCTION

Naturally occurring 2-phenylchromans commonly called flavans, characterized by the presence of a benzopyran core, exist widely in the plant kingdom and exhibit many important biological and pharmacological activities [1]. Flavans substituted in the heterocyclic ring (3- and 4-positions, *e.g.*, catechins) are frequently encountered in nature, but natural flavans with methyl substituents at 2- and 4-positions on the heterocyclic ring are comparatively rare [2].

To our knowledge there is only one report on a naturally occurring flavan of this type called inulavosin [2-(2′-hydroxy)-2,4′,4,4,7-penta methyl flavan] isolated [2] from *Inula nervosa* (Compositae) having piscicidal activity [2] and recently discovered as a melanogenesis inhibitor [3]. The flavan inulavosin is a dimer of thymol and was prepared [4] from *m*-cresol and acetone much before its isolation as a natural product. Several synthetic methyl-substituted flavans similar to inulavosin have also been prepared from *o*-cresol [5], *p*-cresol [5–7], resorcinol [7,8], 2,3-dimethyl phenol, and 3,4-dimethyl phenol [7] but so far no such compounds from naphthols have been reported.

In this article, we describe the synthesis of 2-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*h*]chromen-2-yl)-1-naphthol **1** and 3-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*g*]-chromen-2-yl)-2-naphthol **3**, and their acetates (**2** and **4**) from 1- and 2-naphthol derivatives using Grignard reaction followed by dehydration of the corresponding tertiary alcohols and hetero Diels–Alder dimerization. The numbering pattern is given for clarity (Fig. 1).

RESULTS AND DISCUSSION

We chose to prepare **1** by using a simple approach (Fig. 2). Grignard reaction on 2-acetyl-1-naphthol **5** [9]

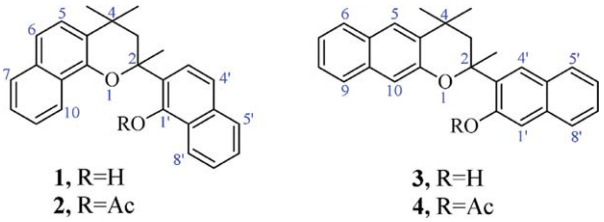

Figure 1. Benzochromenes (**1** and **3**) and their acetates (**2** and **4**). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

afforded 2-(1′-hydroxy-1′-methyl ethyl)-1-naphthol **6** [10] as colorless oil in quantitative yield. Heating **6** with maleic anhydride on water bath gave 2-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*h*]chromen-2-yl)-1-naphthol **1** as viscous oil, which solidified on standing.

IR spectrum of **1** showed a band at 3364 cm^{-1} indicating the presence of hydroxyl group. In its ^1H NMR spectrum, three singlets at δ 1.21, 1.48, and 1.83 integrating for 3H each were assigned to the three methyl groups. Equally spaced doublets at δ 2.17 and 2.63 with $J = 14.5$ Hz were assigned to the geminal protons of C-3 methylene. Its proton decoupled ^{13}C NMR spectrum showed in all 26 carbon signals, which comprised of twenty sp^2 and six sp^3 carbons. The ^{13}C NMR DEPT 135 spectrum indicated that two sp^3 carbons are quaternary and one of which is attached to an oxygen atom (δ 83.2). Among the aromatic signals, the two downfield signals at δ 144.4 and 149.9 are attributed to the oxygen bearing sp^2 carbon atoms. In the HRESIMS data of **1** the presence of a peak at *m*/*z* 369.1853 $[M + H]^+$ indicated the formation of pseudomolecular ion of **1**.

The dehydration of tertiary alcohol **6** using maleic anhydride gives **7**, which dimerizes with its tautomer **7a** to give the benzochromene **1** (Fig. 3). Maleic anhydride was converted to maleic acid by water molecule released during the dehydration reaction and was identified by comparison with an authentic sample (m.p., co-TLC, and IR).

Acetylation of the phenolic hydroxyl in **1** using acetic anhydride and pyridine gave 2-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*h*]chromen-2-yl)-1-naphthyl acetate **2** as a crystalline solid having m.p. 168°C. Single crystal X-ray studies [11] of benzochromene acetate **2** further supported the structure for the benzochromene **1** (Fig. 4).

Dehydration of **6** using *p*-TsOH in refluxing benzene [12] gave two compounds, 7,7-dimethyl-7*H*-diben-

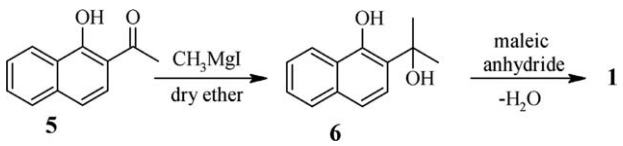

Figure 2. Synthesis of benzochromene **1**.

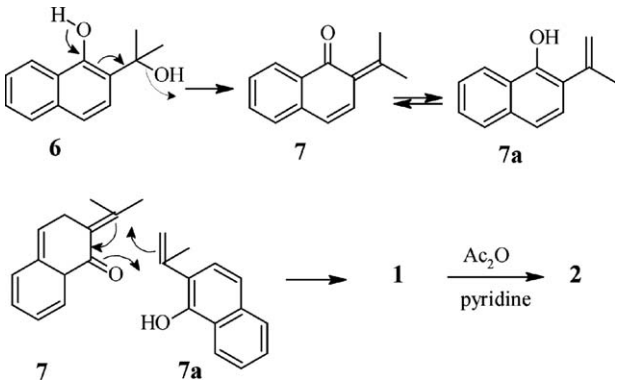

Figure 3. Probable mechanism for the formation of benzochromene **1**.

zo[*c*,*h*]xanthene **8** and benzochromene **1** (Fig. 5). The benzoxanthene **8** was obtained as silvery white flakes (m.p. 180°C). GCMS showed molecular ion peak at *m*/*z* 310.1 $[M^+]$ and a base peak at *m*/*z* 295 $[M - 15]^+$ because of the doubly benzylic ion formed by the loss of one methyl group. Benzoxanthene **8**, prepared by the reaction of 1-naphthol with acetone in presence of $POCl_3$, is reported in literature [13] but with no spectroscopic data. Hence it was characterized by its spectral data and is included in the Experimental section. The identity of **8** was further confirmed by its independent synthesis from 1-naphthol and acetone in the presence of *p*-TsOH [14].

Grignard reaction on ethyl-3-hydroxy-2-naphthoate (**9**) [15] gave 3-(1′-hydroxy-1′-methyl ethyl)-2-naphthol (**10**) [16] as solid in 98% yield. Compound **10** is reported in literature [16] but with no spectroscopic

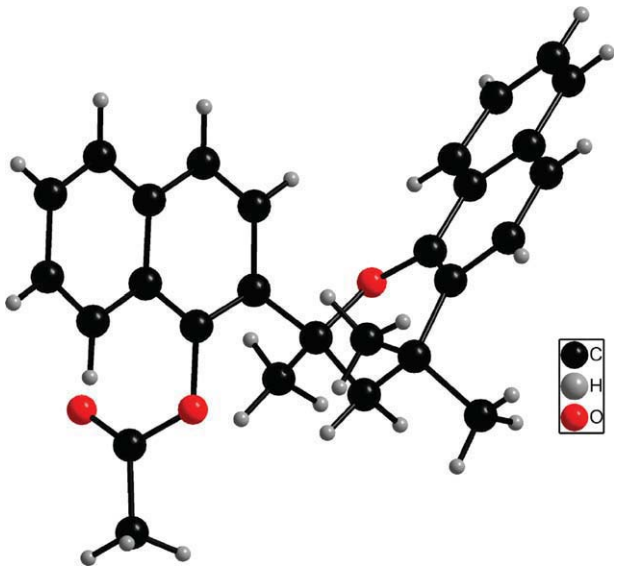

Figure 4. Crystallographic structure of benzochromene acetate **2**. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

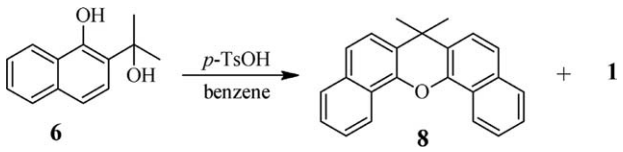

Figure 5. Dehydration of alcohol **6** with *p*-TsOH.

data. Therefore, it was characterized by detail NMR and MS data. The GCMS data of **10** showed a peak at *m*/*z* 184 $[M - 18]^+$ indicating loss of a water molecule. Heating **10** with maleic anhydride on a water bath gave 3-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*g*]chromen-2-yl)-2-naphthol **3** as viscous oil which solidified on standing (Fig. 6).

The IR spectrum of **3** showed a band at 3504 cm^{-1} indicating the presence of a hydroxyl group. In its ^{1}H-NMR spectrum, three singlets at δ 1.31, 1.59, and 1.88 integrating for 3H each were assigned to the three methyl groups. Equally spaced doublets at δ 2.30 and 2.89 with $J =$ 14.4 Hz were assigned to the geminal protons of the C-3 methylene. The proton decoupled ^{13}C-NMR spectrum of **3** showed in all 26 carbon signals comprising of 20 sp^2 and 6 sp^3 carbons. Furthermore, the ^{13}C-NMR DEPT 135 spectrum indicated that, two sp^3 carbons are quaternary, and one of which is attached to an oxygen atom (δ 81.7). The two downfield signals at δ 149.7 and 152.7 are attributed to the two oxygen bearing sp^2 carbon atoms. In the HRESIMS data of **3**, the presence of a peak at *m*/*z* 369.1851 $[M + H]^+$ indicated the formation of a pseudomolecular ion of **3**. The ^{1}H, ^{13}C, and HRESIMS data of **3** were similar to that observed for **1**, indicating that the benzochromenes **1** and **3** are structural isomers. Moreover, the mechanism of the formation of the benzochromene **3** is also similar to that of the formation of the benzochromene **1**.

Acetylation of the phenolic hydroxyl in **3** gave 3-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*g*]chromen-2-yl)-2-naphthyl acetate **4** as greenish crystalline solid (m.p. 136°C), which on single crystal X-ray diffraction studies [11] provided further support to the structure of benzochromene **3** (Fig. 7). Dehydration of **10** using *p*-TsOH in refluxing benzene [12] gave benzochromene **3** as the sole product.

EXPERIMENTAL

Melting points were determined in open capillary tubes and are uncorrected. UV spectra were recorded on a Shimadzu UV-2450 UV–visible spectrophotometer. IR spectra were recorded on a Shimadzu (IR Prestige-21) FTIR spectrophotometer in the range 4000–400 cm^{-1} as KBr diluted pellets in the solid state or as film in NaCl plates. NMR spectra were recorded on a Bruker WT 300 MHz FTNMR spectrophotometer with TMS as the internal standard. The multiplicities of carbon signals were obtained from a distortionless enhancement by polarization transfer (DEPT-135). Coupling constants (*J*) are expressed in Hz. Diethyl ether was distilled and stored over sodium wire.

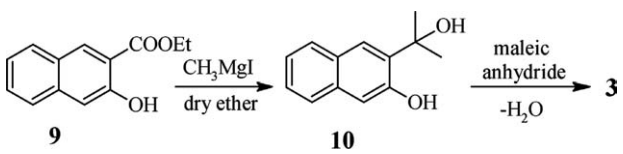

Figure 6. Synthesis of benzochromene **3**.

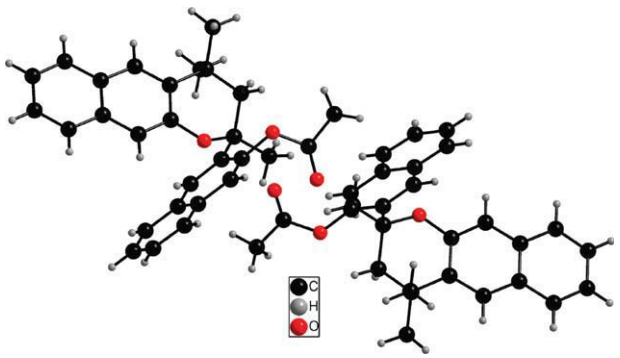

Figure 7. Crystallographic structure of benzochromene acetate **4**. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

2-(1′-Hydroxy-1′-methyl ethyl)-1-naphthol (6). Grignard reagent was prepared by dropwise addition of methyl iodide (1.7 mL, 3.82 g, 27 mmol) in ether (5 mL) to a stirred suspension of magnesium metal (0.65 g, 27 mmol) in ether (5 mL) containing a crystal of I_2. After addition was complete (30 min), the mixture was refluxed for additional 30 min. The Grignard reagent was cooled in ice bath and to this a solution of **5** (2.0 g, 11 mmol) in ether (5 mL) was added dropwise with vigorous stirring. After addition was completed, the resulting mixture was refluxed for 2 h, cooled, and added to a saturated solution of NH_4Cl in crushed ice. This was extracted with ether, washed with water, brine, and dried over anhydrous Na_2SO_4. Evaporation of the ether afforded 2-(1′-hydroxy-1′-methyl ethyl)-1-naphthol **6** as oil in quantitative yield; IR: 3520, 3258, 1574, 1464, 1385, 1304, 1107, 955, 876 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.63 (s, 6H, 2 × CH_3), 2.49 (s, 1H, 1′ OH), 7.01 (d, 1H, H-4, J = 8.7 Hz), 7.25 (d, 1H, H-3, J = 8.7 Hz), 7.37–8.25 (m, 4H, H-5, 6, 7, 8), 9.78 (s, 1H, OH).

2-(2,4,4-Trimethyl-3,4-dihydro-2*H*-benzo[*h*]chromen-2-yl)-1-naphthol (1). A mixture of **6** (0.662 g, 3.27 mmol) and maleic anhydride (0.482 g, 4.9 mmol) was heated on a water bath for 1 h. Reaction mixture was cooled, diluted with water, and extracted with ether. The organic extract was washed with saturated $NaHCO_3$, water and dried over anhydrous Na_2SO_4. Evaporation of the ether afforded 2-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*h*]chromen-2-yl)-1-naphthol **1** as oil. Purification by silica gel column chromatography using benzene and petroleum ether (1:1) as eluant gave pure **1** (0.6 g, 99.5%), which solidified on standing, m.p. 68–70°C; UV (MeOH): 296, 235, 213 nm; IR: 3364, 2963, 1574, 1387, 1074, 806, 748, 677 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.21 (s, 3H, CH_3), 1.48 (s, 3H, CH_3), 1.83 (s, 3H, CH_3), 2.17 (d, 1H, H-3, J = 14.5 Hz), 2.63 (d, 1H, H-3, J = 14.5 Hz), 7.17–7.47 (m, 8H, Ar-H), 7.68–7.76 (m, 2H, Ar-H), 8.19 (d, 2H, Ar-H, J = 9 Hz),

9.26 (s, 1H, OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 27.3 (CH_3), 31.5 (CH_3), 32.9 (CH_3), 48.5 (C-3), 75.5 (C-4), 83.2 (C-2), 119.1 (C-4′), 121.2 (C-6), 121.8 (C-7′), 122.3 (C-2′), 122.5 (C-9), 124.2 (C-6′), 124.4 (C-8), 125.3 (C-5′), 125.7 (C-4a, 10a, 8′a), 126.1 (C-3′), 126.2 (C-7), 126.5 (C-5), 127.0 (C-10), 127.7 (C-8′), 133.1 (C-4′a), 133.8 (C-6a), 144.4 (C-1a), 149.9 (C-1′); HRESIMS: *m*/*z* $[M + H]^+$ Found: 369.1853, Calcd. for $C_{26}H_{25}O_2$: 369.1849.

2-(2,4,4-Trimethyl-3,4-dihydro-2*H*-benzo[*h*]chromen-2-yl)-1-naphthyl acetate (2). A mixture of benzochromene **1** (0.3 g, 0.8 mmol), acetic anhydride (4 mL), and pyridine (2 mL) was heated on a water bath for 5 min and kept overnight. The reaction mixture was poured over crushed ice to which few drops of conc. HCl were added, stirred, and extracted with ether. The ethereal layer was washed with water, brine, and dried over anhydrous Na_2SO_4. Evaporation of ether afforded 2-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*h*]chromen-2-yl)-1-naphthyl acetate **2** (0.27 g, 80.8%) as colorless solid; m.p. 168°C (hexane); IR: 1769 (CO), 1385, 1366, 1198, 1082, 810, 750 cm^{-1}.

Dehydration of 6 with *p*-TsOH, formation of 7,7-dimethyl-7*H*-dibenzo[*c,h*]xanthene (8) and benzochromene (1). A mixture of **6** (0.516 g, 2.5 mmol) and catalytic amount of *p*-TsOH (0.05 g) was heated under reflux in benzene (10 mL) for 7 h (monitored by TLC). The reaction mixture was cooled, diluted with water, and extracted with ether. The organic extracts were washed with saturated $NaHCO_3$, water, and dried over anhydrous Na_2SO_4. Evaporation of ether gave crude oil (0.529 g). TLC of the oil in benzene showed two spots, which were separated by silica gel column chromatography. Elution with petroleum ether gave silvery white flakes of 7,7-dimethyl-7*H*-dibenzo[*c,h*]xanthene **8** (0.09 g, 22.7%); m.p. 180°C (hexane) (Ref. [13] 186°C); IR: 2966, 1566, 1393, 1371, 1211, 1111, 808, 746, 677 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.80 (s, 6H, 2 × CH_3), 7.55–7.67 (m, 8H, Ar-H), 7.85 (d, 2H, H-5, 9, J = 8.1 Hz), 8.64 (d, 2H, H-6, 8, J = 8.1 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ 33.2 (CH_3 × 2), 34.1 (C-7), 121.8 (C-5, 9), 122.7 (C-2, 12), 123.1 (C-6a, 8a), 124.2 (C-3, 11), 124.3 (C-1a, 13a), 126.0 (C-4, 10), 126.2 (C-6, 8), 127.4 (C-1, 13), 133.1 (C-4a, 9a), 144.2 (C-14a, 14b); GCMS: *m*/*z* 310.1 $[M]^+$, 295. Further elution with benzene:petroleum ether (3:7) gave **1** (0.233 g, 49.6%) as oil and found to be identical with that obtained earlier.

7,7-Dimethyl-7*H*-dibenzo[*c,h*]xanthene (8). To a mixture of acetone (0.25 mL, 3.5 mmol) and distilled 1-naphthol (1.0 g, 6.9 mmol), *p*-TsOH (13.3 mg, 0.07 mmol) was added and stirred at 125°C for 1 h. The progress of the reaction was monitored by TLC. The reaction mixture was cooled, diluted with water, and extracted with CH_2Cl_2. The organic layer was washed with dilute NaOH, water, and dried over anhydrous Na_2SO_4. Evaporation of the solvent gave crude solid (1.08 g). Purification through a small bed of silica using petroleum ether as eluant gave **8** (0.766 g, 71.2%) as silvery white flakes, m.p. 180°C (hexane) (Ref. [13] 186°C).

3-(1′-Hydroxy-1′-methyl ethyl)-2-naphthol (10). Grignard reagent was prepared by dropwise addition of methyl iodide (6.2 mL, 14.2 g, 0.1 mol) in ether (10 mL) to a stirred suspension of magnesium metal (2.4 g, 0.1 mol) in ether (10 mL) containing a crystal of I_2. After addition was complete (30 min), the mixture was refluxed for additional 30 min. The Grignard reagent was cooled in ice bath and to this a solution of **9** (2.14 g, 0.01 mol) in ether (10 mL) was added dropwise with vigorous stirring. After addition was complete, the resulting mixture was refluxed for 5 h, cooled and added to a saturated solution of NH_4Cl in crushed ice. This was extracted with ether, washed with water, brine and dried over anhydrous Na_2SO_4. Evaporation of the ether afforded 3-(1′-hydroxy-1′-methyl ethyl)-2-naphthol **10** (1.96 g, 98%) as solid; m.p. 140°C (benzene) (Ref. [16] 140°C); IR: 3418, 2982, 1634, 1599, 1369, 1240, 1199, 947, 874, 748 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.77 (s, 6H, 2 × CH_3), 2.56 (bs, 1H, 1′ OH), 7.23 (s, 1H, H-1), 7.25–7.39 (m, 2H, H-6,7), 7.56 (s, 1H, H-4), 7.65 (d, 1H, H-8, J = 8.3 Hz), 7.70 (d, 1H, H-5, J = 8.3 Hz), 8.97 (bs, 1H, OH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 30.3 (C-2′, 3′), 75.9 (C-1′), 111.9 (C-1), 123.5 (C-6), 124.5 (C-7), 125.9 (C-4), 126.4 (C-8), 127.8 (C-5), 128.0 (C-3), 133.4 (C-4a), 134.4 (C-8a), 153.9 (C-2); GCMS: *m*/*z* 184 $[M-18]^+$.

3-(2,4,4-Trimethyl-3,4-dihydro-2*H*-benzo[*g*]chromen-2-yl)-2-naphthol (3). A mixture of **10** (0.2 g, 0.9 mmol) and maleic anhydride (0.11 g, 1.48 mmol) was heated on a water bath for 1 h. Reaction mixture was cooled, diluted with water, and extracted with ether. The organic extract was washed with saturated $NaHCO_3$, water and dried over anhydrous Na_2SO_4. Evaporation of the ether afforded 3-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzo[*g*]chromen-2-yl)-2-naphthol **3** as oil. Purification by silica gel column chromatography using benzene and petroleum ether (1:1) as eluant gave pure **3** (0.153 g, 84%), which solidified on standing, m.p. 82–84°C; UV (MeOH): 333, 320, 278, 269, 244 nm; IR: 3504, 2961, 1634, 1504, 1444, 1339, 1252, 1165, 1060, 947, 868, 744 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.31 (s, 3H, CH_3), 1.59 (s, 3H, CH_3), 1.88 (s, 3H, CH_3), 2.30 (d, 1H, H-3, J = 14.4 Hz), 2.89 (d, 1H, H-3, J = 14.4 Hz), 3.50 (s, 1H, OH), 7.24–7.76 (m, 12H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 28.9 (CH_3), 32.6 (CH_3), 32.8 (CH_3), 47.5 (C-3), 77.2 (C-4), 81.7 (C-2), 111.9 (C-1′), 113.0 (C-10), 123.5 (C-6′), 124.1 (C-7), 125.6 (C-7′), 125.7 (C-8), 125.9 (C-4′), 126.2 (C-5), 126.3 (C-8′), 126.4 (C-9), 127.4 (C-5′), 127.9 (C-6), 128.2 (C-3′), 129.9 (C-4a), 132.7 (C-4′a), 133.2 (C-5a), 134.1 (C-8′a), 134.3 (C-9a), 149.7 (C-10a), 152.7 (C-2′); HRESIMS: *m*/*z* $[M + H]^+$ Found: 369.1851, Calcd. for $C_{26}H_{25}O_2$: 369.1849.

3-(2,4,4-Trimethyl-3,4-dihydro-2*H*-benzo[*g*]chromen-2-yl)-2-naphthol (3) by *p*-TsOH method. A mixture of **10** (0.812 g, 4.0 mmol) and catalytic amount of *p*-TsOH (0.05 g) was heated under reflux in benzene (10 mL) for 4 h (monitored by TLC). The reaction mixture was cooled, diluted with water and extracted with ether. The organic extracts were washed with saturated $NaHCO_3$, water and dried over anhydrous Na_2SO_4. Evaporation of the solvent gave **3** as oil (0.706 g, 95.5%) identical (Co-TLC, IR, NMR) with **3** obtained by maleic anhydride method.

3-(2,4,4-Trimethyl-3,4-dihydro-2*H*-benzo[*g*]chromen-2-yl)-2-naphthyl acetate (4). A mixture of benzochromene **3** (0.475 g, 1.29 mmol), acetic anhydride (8 mL) and pyridine (2 mL) was heated on a water bath for 5 min and kept overnight. The reaction mixture was poured over crushed ice to which few drops of cone. HCl were added, stirred and extracted with ether. The ethereal layer was washed with water, brine and dried over anhydrous Na_2SO_4. Evaporation of ether afforded 3-(2,4,4-trimethyl-3,4-dihydro-2*H*-benzd[*g*]chromen-2-yl)-2-naphthyl acetate **4** (0.497 g, 94%) as greenish crystals, mp 136°C (hexane); IR: 2970, 1757, 1633, 1599, 1500, 1442, 1369, 1255, 1199, 1037, 950 cm^{-1}.

CONCLUSIONS

A simple and convenient synthesis of two new substituted benzochromenes **1** and **3** has been achieved. They have been fully characterized by IR, NMR, HRESIMS data, and supported by crystallographic structures of their corresponding acetates (**2** and **4**).

NOTE ADDED IN PROOF

This article was corrected following its initial online publication. Bikshandarkoil R. Srinivasan and Pallepogu Raghavaiah are now included as coauthors; Professor Shrivallabh P. Kamat is now designated as corresponding author; the affiliation of Professor S. K. Paknikar has been updated; Figures 2, 5, and 6 have been revised; a description of the spectral data for compound **3** has been included; and experimental details for compounds **3** and **4** have been included.

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