Asian Journal of Chemistry; Vol. 37, No. 1 (2025), 100-104

ASIAN JOURNAL OF CHEMISTRY

https://doi.org/10.14233/ajchem.2025.32937

Synthesis of Linear 6-Allylated-7-hydroxycoumarins: *para*-Claisen Rearrangement of 2′-Allylated/Prenylated Derivatives of 4′-Prenyloxy Cinnamic Acid

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Received: 22 October 2024; *Accepted*: 3 December 2024; *Published online*: 31 December 2024; AJC-21856

Linear 6-allylated-7-hydroxycoumarins were synthesized by the *para*-Claisen rearrangement of 4′-prenyloxy cinnamic acid derivatives under refluxing condition in *N*,*N*-diethylaniline. This method eliminates the requirement for an additional Lewis acid, such as boron trifluoride etherate or trichloride, to convert 7-alkoxycoumarins into 7-hydroxycoumarins, unlike alternative techniques.

Keywords: Linear coumarins, Claisen rearrangement, Cleavage, Hydroxycoumarins.

INTRODUCTION

7-Oxygenated coumarins bearing allyl/substituted allyl substituents or its biologically modified forms constitute an important class of naturally occurring oxygen-ring compounds [1-3]. 6-Prenylated derivatives of 7-oxygenated coumarins are particularly useful as intermediates for the synthesis of linear coumarins and biologically active linear furanocoumarins [4]. 7-Allyloxycoumarins provide easy access to angular coumarins *via* 8-allylated products formed by highly regioselective, if not exclusive, *ortho*-Claisen rearrangement due to higher carbon-carbon double bond character of 7-8 bond relative to 6-7 bond. Previously, several attempts have been made for the synthesis of 6-allyllated coumarins by employing 3,4-dihydro-7-allyloxy-coumarins as substrates for Claisen rearrangement [5]. This substantially improves the selectivity in favour of 6-allylated derivatives (linear:angular ~ 1:1). Another elegant approach is to block C-8 position of 7-allyloxycoumarin with bromine thereby directing the allyl migration to C-6 [6]. Thus, thermal rearrangement of 7-allyloxy-8-bromocoumarin afforded 6-allyl-7-hydroxycoumarin as the predominant product. Similar successful attempt has also been made with 7-(1,1-dimethylallyloxy)-8-iodocoumarin in refluxing *N*,*N*-dimethylaniline furnishing 6-prenyl-7-hydroxycoumarin (suberosin) [7].

Direct electrophilic substitution of 7-hydroxycoumarins with *tert.*-allyl alcohols in the presence of Lewis acid catalyst was synthetically less attractive because mixtures of 6- and 8-allylated products are formed in low yields [8]. However, the unfavourable regioselectivity in the Claisen rearrangement of 7-allyloxycoumarins due to the higher carbon-carbon double bond character of 7-8 bond of coumarin moiety thereby favouring the migration of allyl moiety to C-8 was sought to be circumvented by cleaving its α-pyrone ring with sodium methoxide in dry methanol to afford vinylogous coumaric acid derivatives [9]. Thermal rearrangement of 2′-prenyloxy derivatives of 4′-alkoxy coumarates gave, by way of *p*-Claisen rearrangement and subsequent ring closure, 6-prenyl-7-alkoxycoumarins as the major products. The key to success of this protocol is the steric congestion in the dienone intermediate formed by *ortho*-Claisen rearrangement in the first step which, in turn, depends upon substitution of γ-carbon of 2′-allyloxy residue. This steric congestion is the driving force for the subsequent *p*-Claisen rearrangement. However, 6-prenyloxybenzaldehydes have served as substrates for the synthesis of suberosin (6-allyl-7-methoxycoumarin) and linear coumarins by Tandem Claisen rearrangement and Wittig reaction [10].

We were interested in the *p*-Claisen rearrangement based approach of L.M. Harwood using coumaric ester derivatives and set out to find appropriate conditions, which would provide entry to 6-allylated-7-hydroxycoumarins, since several natural linear coumarins are derived from 6-prenyl-7-hydroxycoumarin (demethylsuberosin) [11]. Also, the free hydroxy group at 7-position can be allylated/prenylated and the ethers so obtained could be further subjected to Claisen rearrangement conditions

to provide 6,8-dialkylated coumarins, which are fairly well represented in nature [1-4]. Earlier attempts to cleave 6-prenylated-7-methoxycoumarins with Lewis acids, such as BCl_3 to prepare demethylsuberosin was unsuccessful because liberated hydroxy group at C-7 underwent concomitant ring closure with prenyl group at *ortho*-position to give furocoumarins [12,13]. Our attention was drawn to the reported cleavage of 7-prenyloxycoumarins to 7-hydroxycoumarins under thermal rearrangement conditions [14]. We envisaged that coumaric acid or ester derivatives prepared by cleavage of α-pyrone ring of 7-prenyloxycoumarins with NaOMe followed by sequential allylation/prenylation of 2′-hydroxy group so formed and *p*-Claisen rearrangement might provide an easy entry to 6-allylated/prenylated 7-hydroxycoumarins. We were also interested to explore the possibility of competitive *ortho*-Claisen rearrangement of coumaric acids that are substituted at C-4′ with prenyloxy moiety. *para*-Claisen rearrangement of 4′-prenyloxy-2′-allyloxycoumaric acids *is hitherto* unstudied and might provide 6-allylated 7-hydroxycoumarins in case *p*-Claisen rearrangement involving 2′-allyloxy ether and ring closure to coumarin precedes thermal cleavage of 7-prenyloxycoumarin so formed. To investigate the feasibility of this plan, three 4′-prenyloxycinnamic acids bearing substituted allyloxy/prenyloxy side chains at 2′position *viz.* **4**, **7** are synthesized and then subjected to thermal Claisen rearrangement.

EXPERIMENTAL

Prenylation of 7-hydroxycoumarin with prenyl bromide: 1-Bromo-3-methyl-2-butene (1.5 g, 10 mmol) was added to dry acetone solution (200 mL) of 7-hydroxycoumarin (**1**) (1.5 g, 9.25 mmol). It was refluxed on water bath with anhydrous K_2CO_3 (6 g) for 10 h. The reaction mixture was cooled, filtered and the solvent was removed. The residual mass was extracted with ether (3 × 20 mL). The combined ethereal extract was washed with 2% NaOH solution to remove any unreacted starting material. The residue, after removal of ether, was chromatographed over silica gel (60 g) using chloroform-light petrol (1:1) as eluent to give a pale yellow solid. It was crystallized from chloroform-light petrol mixture to yield 7-prenyloxycoumarin (**2**) (1.74 g, 82%), m.p.: 75-77 ºC (lit. [15] 77-78 ºC).

7-Prenyloxycoumarin (2): FTIR (KBr, ν_{max}, cm^{-1}): 3046, 2978, 2916, 2854, 1726, 1615, 1505, 1469, 1453, 1402, 1391, 1372, 1354, 1284, 1235, 1199, 1157, 1130, 1011, 988; ^{1}H NMR ($CDCl_3$) δ ppm: 1.76, 1.80 (each 3H, s, CH=C**Me**$_2$), 4.57 (2H, d, *J* = 6.8 Hz, OCH_2), 5.46-5.48 (1H, m, C**H**=CMe_2), 6.24 (1H, d, *J* = 9.4 Hz, H-3), 6.82 (1H, dd, *J* = 8.4, 2.1 Hz, H-6), 6.84 (1H, d, *J* = 2.1 Hz, H-8), 7.35 (1H, d, *J* = 8.4 Hz, H-5), 7.63 (1H, d, *J* = 9.4 Hz, H-4); ^{13}C NMR ($CDCl_3$) δ_C ppm: 162.21 (C-7), 161.80 (C-2), 155.96 (C-8a), 143.59 (C-4), 128.82 (C-5), 118.69 (**C**H=CMe_2), 113.34 (C-6), 113.07 (C-4a), 112.53 (C-3), 101.64 (C-8), 65.51 (OCH_2), 25.96 (Me), 18.41 (Me); MS *m/z* (%) 230 (12, M^+), 163 (100); Anal. calcd. (found) % for $C_{14}H_{14}O_3$: C, 73.04 (73.01); H, 6.08 (6.10).

Methanolysis of 7-prenyloxycoumarin (2): Metallic sodium (5.2 g) was carefully dissolved in dry methanol (75 mL) in a 100 mL three-necked round bottomed flask and refluxed till sodium completely reacts (1.5 h). 7-Prenyloxycoumarin (**2**) (1.5 g, 6.52 mmol) was then added to this solution and refluxed for 7 h without nitrogen cover. It was then cooled to room temperature, poured into ice-cold water (50 g) and acidified with 6N HCl followed by cooling. Extraction with ethyl acetate (3 × 40 mL), drying with anhydrous sodium sulfate and concentration under reduced pressure gave the crude product. It was purified by column chromatography over silica gel (40 g) using chloroform-methanol (95:5) as eluent to give compound **3** as a white crystalline solid (1.22 g, 76%), m.p.: 162 ºC.

2′-Hydroxy-4′-(3,3-dimethylallyloxy)cinnamic acid (3): FTIR (KBr, ν_{max}, cm^{-1}): 3420, 2925, 2854, 2721, 2611, 1760, 1670, 1595, 1513, 1464, 1432, 1330, 1307, 1273, 1235, 1195, 1091, 980; ^{1}H NMR ($CDCl_3$) δ ppm: 1.75, 1.81 (each 3H, s, CH=C**Me**$_2$), 4.56 (2H, d, *J* = 6.9 Hz, O**CH**$_2$), 5.36 (1H, t, *J* = 7 Hz, OCH_2C**H**=CMe_2), 6.45 (1H, d, *J* = 2.4 Hz, H-3′), 6.52 (1H, dd, *J* = 8, 2.4 Hz, H-5′), 6.53 (1H, d, *J* = 17 Hz, CH=C**H**CO_2H), 7.40 (1H, d, *J* = 8 Hz, H-6′), 7.95 (1H, d, 17 Hz, C**H**=$CHCO_2H$); MS *m/z* (%) 249 (100, M+H^+), 248 (22, M^+), 220 (15, M^+-CO), 161 (58); Anal. calcd. (found) % for $C_{14}H_{16}O_4$: C, 67.74 (67.75); H, 6.45 (6.43).

Alkylation of compound 3 with 1-chloro-2-butene: 2′-Hydroxy-4′-(3,3-dimethyl-allyloxy)cinnamic acid (**3**) (1 g, 4.03 mmol) dissolved in 100 mL dry acetone was added to 1-chloro-2-butene (407 mg, 4.5 mmol) in round bottom flask *via* syringe followed by the addition of anhydrous K_2CO_3 (5 g) and NaI (50 mg). The mixture was refluxed on a water bath for 12 h. Usual workup and chromatographic purification over silica gel gave compound **4** (767 mg, 63%) as viscous liquid from the chloroform-methanol (98:2) eluates in addition to a small amount of **2** (167 mg, 18%).

2′-(3-Methylallyloxy)-4′-prenyloxycinnamic acid (4): FTIR (neat, ν_{max}, cm^{-1}): 3340, 3025, 2919, 1708, 1625, 1604, 1571, 1502, 1435, 1378, 1320, 1298, 1261, 1159, 1111, 1002, 966; ^{1}H NMR ($CDCl_3$) δ ppm: 1.73 (3H, d, *J* = 6 Hz, CH=CH**Me**), 1.75, 1.81 (each 3H, s, CH=C**Me**$_2$), 4.38 (2H, d, *J* = 6.5 Hz, O**CH**$_2$CH=CMe_2), 4.45 (2H, d, *J* = 6 Hz, O**CH**$_2$CH=CHMe), 5.50 (1H, t, *J* = 6.5 Hz, C**H**=CMe_2), 5.52 (2H, m, C**H**=C**H**Me), 6.48-6.50 (2H, m, H-3′ and H-5′), 6.51 (1H, d, *J* = 16 Hz, CH=C**H**CO_2H), 7.42 (1H, d, *J* = 8.4 Hz, H-6′), 7.92 (1H, d, *J* = 16 Hz, C**H**=$CHCO_2H$); MS *m/z* (%) 302 (52, M^+), 161 (100); Anal. calcd. (found) % for $C_{18}H_{22}O_4$: C, 71.52 (71.53); H, 7.28 (7.26).

Thermal rearrangement of compound 4: Compound **4** (500 mg, 1.65 mmol) was refluxed with N,N-diethylaniline (2 mL) for 2.5 h in a 10 mL round bottomed flask. After disappearance of the starting material (TLC monitoring), the reaction mixture was cooled to room temperature and poured into ice-cold water, neutralized with ice-cold 6N HCl and extracted with ether (3 × 10 mL). The combined organic extract was washed with water (10 mL), brine (10 mL) and dried over anhydrous Na_2SO_4. Concentration of the solution and chromatographic purification over silica gel (20 g) gave compound **5** (171 mg, 48%) and compound **6** (57 mg, 16%) from chloroform eluates. 7-Hydroxycoumarin (**1**) (27 mg, 10%) was also isolated from chloroform-methanol (95:5) eluates.

6-(3-Methylbut-2-enyl)-7-hydroxycoumarin (5): FTIR (neat, ν_{max}, cm^{-1}): 3423, 3045, 3027, 2945, 1723, 1657, 1606,

1565, 1487, 1452, 1401, 1303, 1259, 1230, 1176, 1125, 1108, 930; ^{1}H NMR ($CDCl_3$) δ ppm: 1.73 (1H, d, *J* = 6.2 Hz, CH=CH**Me**), 3.74 (2H, d, *J* = 7.2 Hz, 1′-CH_2). 5.20-5.34 (2H, m, C**H**=C**H**Me), 6.30 (1H, d, *J* = 9.6 Hz, H-3), 6.90 (1H, s, H-8), 7.20 (1H, s, H-5), 7.68 (1H, d, *J* = 9.6 Hz, H-4); MS *m/z* (%) 216 (52, M^+), 201 (12, M^+-Me), 188 (22, M^+-CO), 161 (*m/z* 188-CO-H); Anal. calcd. (found) % for $C_{13}H_{12}O_3$: C, 72.22 (72.20); H, 5.56 (5.54).

8-Methyl-7,8-dihydropyrano[3,2-*g*]benzopyran-2-one (6): FTIR (neat, ν_{max}, cm^{-1}): 3067, 3023, 2975, 1720, 1657, 1610, 1548, 1490, 1430, 1342, 1305, 1278, 1239, 1108, 1065, 1009, 968; ^{1}H NMR ($CDCl_3$) δ ppm: 1.42 (3H, d, *J* = 6.8 Hz, -OCH**Me**), 1.82 and 2.84 (each 2H, m, -C**H**$_2$C**H**$_2$), 4.02 (1H, m, -OC**H**Me), 6.18 (1H, d, *J* = 9.6 Hz, H-3), 6.70 (1H, s, H-8), 7.15 (1H, s, H-5), 7.60 (1H, d, *J* = 9.6 Hz, H-4); MS *m/z* (%) 216 (75.2, M^+), 201 (15.4, M^+-CH_3), 188 (10.8, M^+-CO), 161 [15.4, (*m/z* 188 + H^+) – CO]; Anal. calcd. (found) % for $C_{13}H_{12}O_3$: C, 72.22 (72.21); H, 5.56 (5.55).

Alkylation of compound 3 with 3-chloro-2-methyl-1-propene: To a solution of 2′-hydroxy-4′-(3,3-dimethylallyloxy)cinnamic acid (**3**) (1.2 g, 4.83 mmol) in dry acetone (100 mL) was added 3-chloro-2-methyl-1-propene (470 mg, 5.2 mmol) and K_2CO_3 (6 g) followed by NaI (50 mg) under nitrogen atmosphere. The resulting mixture was refluxed on a water bath for 10 h. Usual work-up and chromatographic purification over silica gel (60 g) using chloroform-light petrol (1:1) as eluent afforded compound **7** (906 mg, 62%) as a viscous liquid and a small amount of compound **2** (222 mg, 20%).

2′-(3′-Methylallyloxy)-4′-prenyloxycinnamic acid (7): FTIR (liquid film, ν_{max}, cm^{-1}): 3081, 2974, 2917, 2858, 1623, 1607, 1574, 1500, 1454, 1441, 1381, 1316, 1291, 1262, 1175, 1141, 1113, 1021, 899; ^{1}H NMR ($CDCl_3$) δ ppm: 1.70 (3H, s, C(**Me**)=CH_2), 1.76, 1.80 (each 3H, s, CH=C**Me**$_2$), 4.95 (2H, d, *J* = 7 Hz, O**CH**$_2$CHCMe$_2$), 4.96 (2H, s, OCH_2C(Me)=**CH**$_2$), 5.08 (2H, bs, O**CH**$_2$C(Me)=CH_2), 5.47 (1H, m, **CH**=CMe_2), 6.42 (1H, d, *J* = 17 Hz, CH=C**H**CO_2H), 6.45-6.50 (2H, m, H-3′and H-5′), 7.42 (1H, d, *J* = 8.4 Hz, H-6′), 7.95 (1H, d, *J* = 17 Hz, C**H**=CHCO_2H).

Thermal rearrangement of compound 7: To compound **7** (700 mg, 2.31 mmol) was added N,N-diethylaniline (2 mL) in a 5 mL round bottomed flask and the mixture was refluxed for 2 h. It was then cooled and poured into ice (10 g) and neutralized to pH 6 with 6N HCl. It was then extracted with chloroform (3 × 15 mL) and the combined organic extract was washed with brine (15 mL) and dried over anhydrous sodium sulfate. Removal of the solvent gave a crude gummy mass, which was purified by preparative TLC using chloroform-methanol (98:2) as eluent to afford compound **8** (300 mg, 60%), m.p.: 168 ºC along with a small amount of **9** (75 mg, 15%).

6-(2-Methylprop-2-enyl)-7-hydroxycoumarin (8): FTIR (KBr, ν_{max}, cm^{-1}): 3420, 3084, 3034, 2916, 2835, 1726, 1703, 1609, 1564, 1435, 1409, 1386, 1364, 1303, 1256, 1207, 1172, 1038, 1006, 984; ^{1}H NMR ($CDCl_3$) δ ppm: 1.74 (3H, s, CH_2=C**Me**), 3.86 (2H, s, **CH**$_2$C(Me)=CH_2), 5.02 (2H, bs, -CMe=**CH**$_2$), 6.25 (1H, d, *J* = 9.6 Hz, H-3), 6.81 (1H, s, H-8), 7.35 (1H, s, H-5), 7.78 (1H, d, *J* = 9.6 Hz, H-4); MS *m/z* (%) 216 (62.8, M^+), 188 (21, M^+-CO), 189 (24), 161 (100); Anal. calcd. (found) % for $C_{13}H_{12}O_3$: C, 72.23; H, 5.56. Found: C, 72.21; H, 5.53.

2′,2′-Dimethylfuranocoumarin (9) or [7,7-dimethyl-6,7-dihydrofuro[3,2-*g*]benzo[1] pyran-2-one]: FTIR (KBr, ν_{max}, cm^{-1}): 3068, 3042, 2957, 2846, 1734, 1608, 1589, 1474, 1424, 1386, 1368, 1302, 1230, 1218, 1185, 1103, 1006, 960; ^{1}H NMR ($CDCl_3$) δ ppm: 1.42 (6H, s, 2 × 2′-Me), 2.84 (2H, s, CMe_2**CH**$_2$), 6.20 (1H, d, *J* = 9.6 Hz, H-3), 6.70 (1H, s, H-8), 7.22 (1H, s, H-5), 7.64 (1H, d, *J* = 9.6 Hz, H-4); MS *m/z* 216 (78, M^+); Anal. calcd. (found) % for $C_{13}H_{12}O_3$: C, 72.22 972.23); H, 5.56 (5.54).

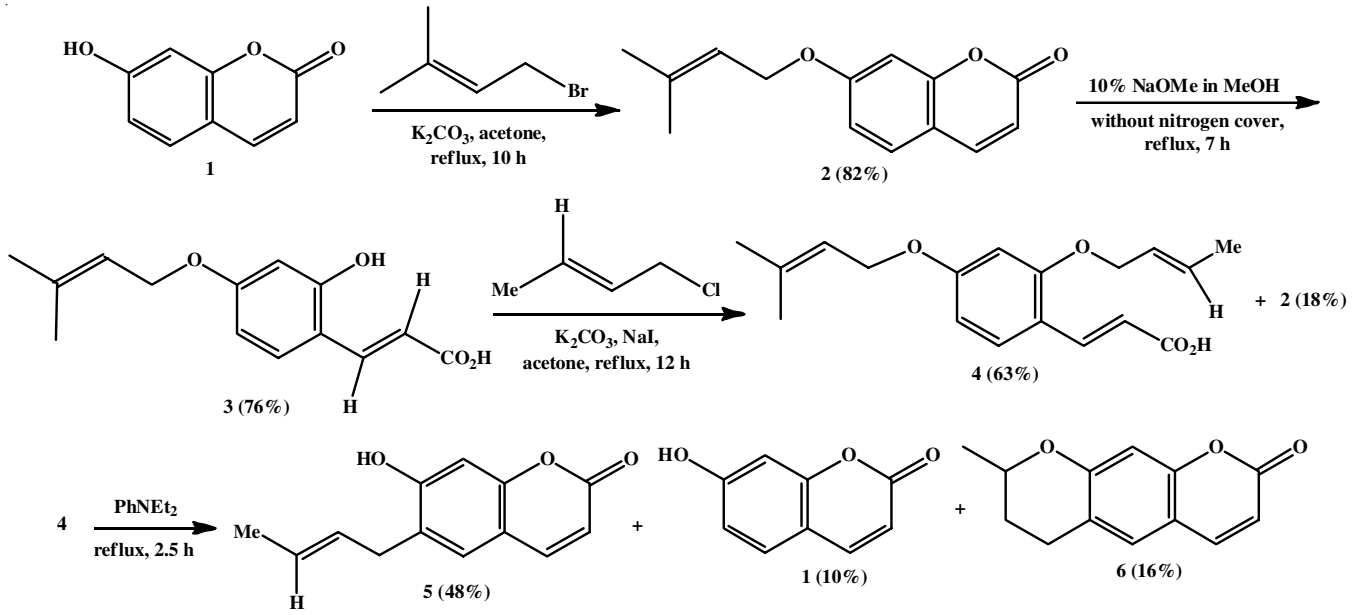
RESULTS AND DISCUSSION

7-Hydroxycoumarin (**1**) was prenylated with 3-methylbut-2-enyl bromide (prenyl bromide) in the presence of anhydrous K_2CO_3 in refluxing acetone (10 h) to give 7-(3,3-dimethylallyloxy)coumarin (**2**) which was refluxed with 10% sodium methoxide in dried methanol without nitrogen atmosphere. 2′-Hydroxy-4′-(3,3-dimethylallyloxy)cinnamic acid (**3**) was isolated in a yield of 76% from this cleavage reaction. Compound **3** was alkylated with 4-chloro-2-butene in refluxing acetone in the presence of anhydrous K_2CO_3 and NaI (Finkelstein condition) to afford the expected ether **4** as a viscous liquid (yield: 63%) along with some 7-prenyloxycoumarin (**2**) as a minor product (18%) (**Scheme-I**).

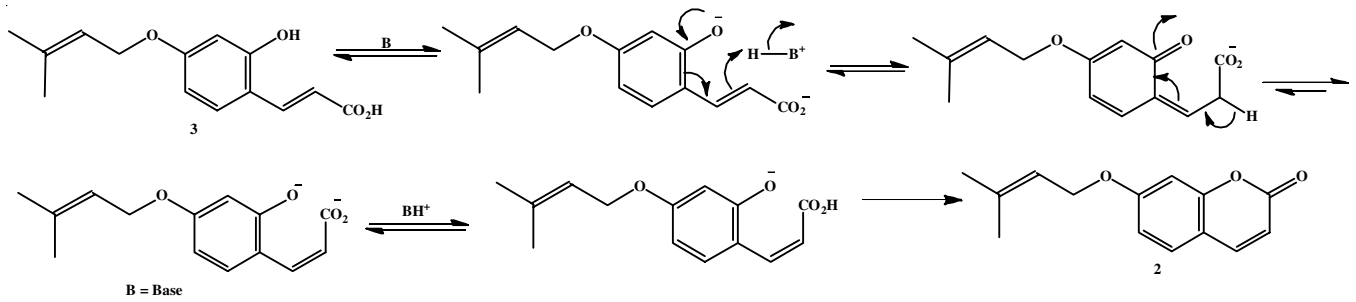
The formation of compound **2** was not anticipated, since it required *trans* to *cis*-isomerization of the α,β-unsaturated acid prior to ring closure to the coumarin moiety. The presence of 2′-phenolic hydroxy group seems to be the key to this base-catalyzed isomerization of the α,β-double bond as depicted in **Scheme-II**.

Compound **4** was heated with N,N-diethylaniline for 2.5 h under reflux to give a mixture of three products *viz.* **1**, **5** and **6** which could be separated by column chromatography over silica gel (**Scheme-I**). The early fractions eluted with benzene-ethyl acetate (80:20) yielded a nonhydroxylic coumarin **6** in 16% yield. Elution with chloroform-methanol (98:2) yielded two hydroxycoumarins *viz*. **5** and **1** successively in 48% and 10% yields, respectively. The most polar solid, m.p.: 224-225 °C that was isolated from later fractions was found to be identical with an authentic sample of 7-hydroxycoumarin in all respects (mixed m.p., co-TLC and superimposable IR).

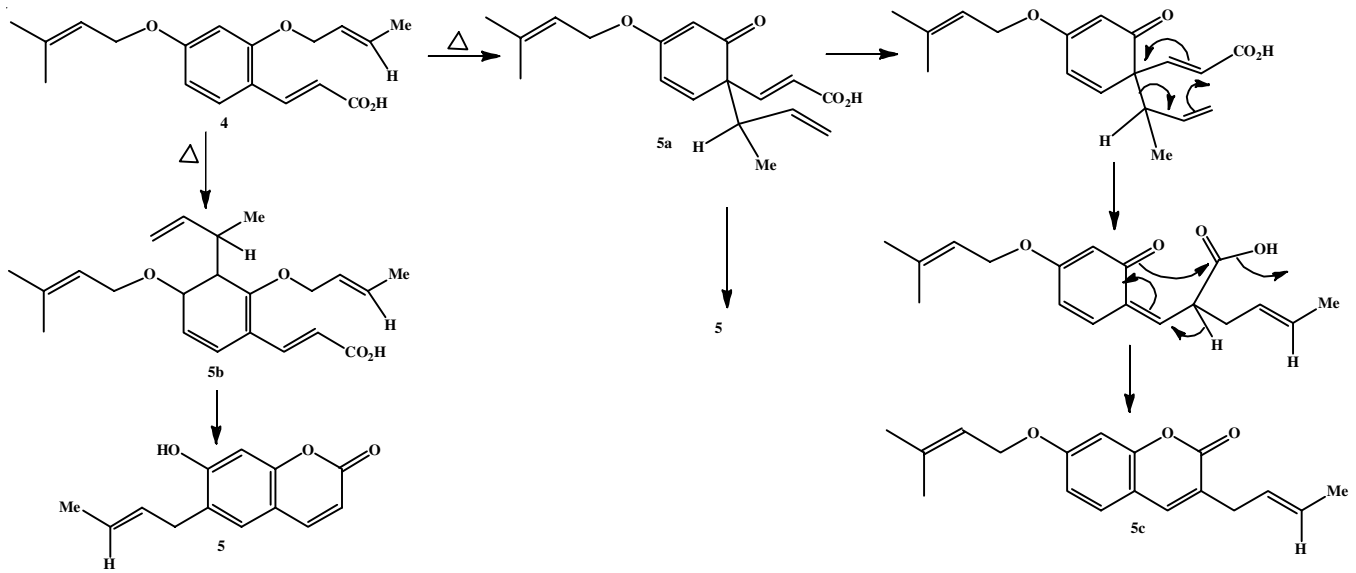
The formation of compound **5** may be rationalized by *para*-Claisen rearrangement of 3′-methylallyl moiety at 2′position of coumaric acid followed by ring closure to coumarin and concomitant cleavage of the resulting 7-prenyloxycoumarin. On the other hand, 7-hydroxycoumarin is formed by simultaneous cleavage of both 3-methylallyloxy and prenyloxy group at 2′- and 4′-positions, respectively prior to Claisen rearrangement followed by ring closure. Pyranocoumarin (**6**) is presumably formed by 6-endo-trig ring closure of hydroxycoumarin (**5**). *para*-Claisen rearrangement of compound **4** leading to compound **5** might occur by way of intermediates **5a** and **5b** (**Scheme-III**). Significantly, **5a** may also give rise to 3-allylated coumarin **5c**, which was not isolated. The alternative sterically congested intermediate **5b** formed by initial *ortho* rearrangement to 3′-position might also afford compound **5**. Strong steric interference of pendant 4′-prenyloxy moiety with the alkyl



Scheme-I: Synthesis of 5 from 7-hydroxycoumarin (**1**)



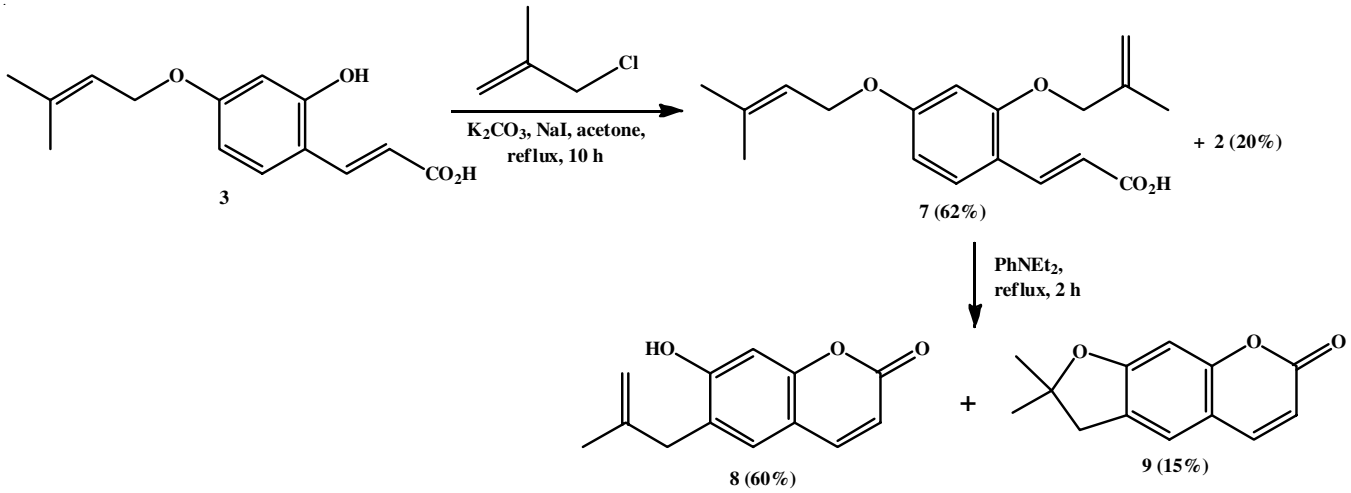
Scheme-II: Base catalyzed isomerization reaction controlled by phnolic hydroxy group



Scheme-III: *para*-Claisen rearrangement of **4** yielding **5**

residue at C-3′ makes this intermediate unstable and provides the driving force for further rearrangement to compound **5**. This intermediate **5b** is not expected to give 3-allylated coumarin.

Encouraged by the success of this exploratory experiment leading to 6-allylated 7-hydroxycoumarin, we alkylated coumaric acid **3** with 3-chloro-2-methyl-prop-1-ene in the presence of anhydrous K_2CO_3 and NaI in refluxing dry acetone to give a viscous liquid **7** (62% yield). Significantly, 7-prenyloxycoumarin (**2**) was also formed during the allylation in 20% yield, as in the previous case. Refluxing compound **7** in N,N-



Scheme-IV: Synthesis of 6-allyl substituted coumarin **8**

diethylaniline for 2 h gave a linear furanocoumarin **9** in 15% yield and a predominant polar solid product (60% yield), which was identified as 6-(2-methylprop-2-enyl)-7-hydroxycoumarin **8** (**Scheme-IV**).

Conclusion

In conclusion, a synthetic route for 6-prenylated/substituted allylated-7-hydroxycoumarins utilizing thermal cleavage of 4′-prenyloxy moiety and *para*-Claisen rearrangement of 2′-allyloxy/prenyloxy moiety of 4′-prenylated derivatives of 2′-allylated/prenylated coumaric acid is developed. 2′-Prenyloxy or similar allyloxy moieties undergo *p*-Claisen rearrangement in preference to 4′-prenyloxy moiety, which after ring closure undergo thermal cleavage to generate hydroxy group at 7-position of the coumarins. This factor is the key to the success of this protocol. The advantageous feature of this method is that no separate Lewis acid like boron trichloride or boron trifluoride etherate is required for converting 7-alkoxycoumarins to 7-hydroxycoumarins, as done in the previous methods.

ACKNOWLEDGEMENTS

The author thanks Dr. Nemai C. Ganguly, Ex-Professor of Chemistry, University of Kalyani, Kalyanim India and the Department of Chemistry, Sarojini Naidu College for Women, Kolkata, India for their constant help and encouragement.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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