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Reaction of 2-Chloromethylphenols with Enaminones

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Abstract—The reaction of *o*-(chloromethyl)phenols with enamino ketones afforded a series of 3-acyl- and 3-formyl-4*H*-chromenes as a result of cascade transformation including [4+2]-cycloaddition of enamino ketone to *o*-quinone methide generated *in situ* and subsequent elimination of secondary amine.

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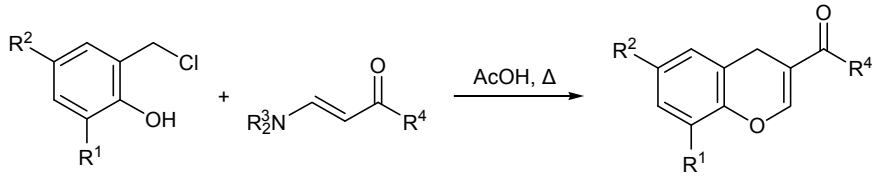
Search for new building blocks for target-oriented synthesis of heterocyclic compounds remains an important problem. In this connection, quite promising is the use of reactive compounds, *o*-quinone methides, in the synthesis and functionalization of heterocycles. *o*-(Chloromethyl)phenols possess a wide synthetic potential as precursors to *o*-quinone methides; however, their ability to readily undergo oligomerization restricts their application in organic synthesis. On the other hand, the presence of strong electron-withdrawing and/or bulky substituents in the molecules of *o*-(chloromethyl)phenols increases their stability, so that it was possible to obtain a series 2,3-dihydrobenzofurans [1, 2], chromans [3], chromeno[2,3-*b*]indoles [4], coumarins [5], indolo[2,1-*b*][1,3]benzoxazines [6–9], and some other heterocyclic systems.

One of the most widespread reactions of *o*-quinone methides is inverse-electron-demand Diels–Alder ([4+2]-cycloaddition) reaction [10, 11] in which the *o*-quinone methide molecule usually acts as heterodiene. As a result, various fused oxygen-containing heterocycles are formed. These reactions provide a convenient and general method of synthesis of chromene and chroman systems. The worth of this approach is obvious since it ensures one-step construction of a fairly complex carbon skeleton consisting of fragments occurring in many natural compounds. In overwhelming cases, electron-rich olefins (vinyl ethers, enamines, vinyl sulfides, etc.) act as dienophiles toward *o*-quinone methides [12].

Enamino ketones belong to "push–pull" olefins [13] in which electron-donating and electron-withdrawing substituents are attached to different carbon atoms of the double bond. The presence of two electrophilic centers in enamino ketone molecules makes them valuable reagents for the synthesis of heterocycles via reactions with binucleophiles [14, 15]. However, the number of reactions where enamino ketones or other push–pull olefins act as dienophiles is quite limited [16–23].

Heating of equimolar mixtures of *o*-(chloromethyl)phenols **1a**–**1d** (precursors to *o*-quinone methides) and enamino ketones **2**–**4** (containing, respectively, a trifluoroacetyl, formyl, or benzoyl group) in boiling acetic acid afforded 44–80% of 3-acyl- and 3-formyl-4*H*-chromenes **5**–**7** (Scheme 1). Initial *o*-(chloro-

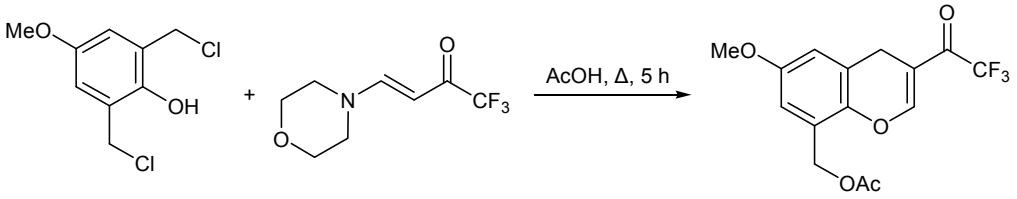
Scheme 1.



1a–1d **2–4** **5a–5d**, **6a**, **6b**, **7a**, **7b**

R^1 = H, R^2 = Ac (**a**); R^1 = 1-Ad, R^2 = Me (**b**); R^1 = H, R^2 = O_2N (**c**); R^1 = R^2 = Br (**d**); **2**, R^3_2N = morpholin-4-yl; **3**, **4**, R^3 = Me; **2**, **5**, R^4 = CF_3; **3**, **6**, R^4 = H; **4**, **7**, R^4 = Ph.

Scheme 2.



1e **2** **5e**

methyl)phenols **1a**–**1d** contained either two additional substituents in both *ortho* and *para* positions relative to the hydroxy group or only one electron-withdrawing group (NO_2 or Ac) in the *para* position. The reaction of 2,6-bis(chloromethyl)-4-methoxyphenol (**1e**) with 1,1,1-trifluoro-4-(morpholin-4-yl)but-3-en-2-one (**2**) was accompanied by nucleophilic replacement of the chlorine atom by acetoxy group (Scheme 2).

The formation of chroman **B** may be regarded as asynchronous but concerted [4+2]-cycloaddition of *o*-quinone methide **A** generated *in situ* to enamino ketone **2**. The primary cycloadduct is unstable in acidic medium and is converted to 4*H*-chromene **3** via elimination of secondary amine molecule (Scheme 3).

The IR spectra of 4*H*-chromenes **5a**–**5e**, **6a**, and **6b** showed a strong absorption band at 1634–1659 cm^{-1} due to stretching vibrations of the C=C double bond in the pyran ring, and the strong band at 1680–1690 cm^{-1} was assigned to carbonyl stretchings. Compounds **6a** and **6b** displayed in the ^1H NMR spectra a singlet at δ 9.45–9.48 ppm due to the aldehyde proton. Protons of the C^4H_2 methylene groups in compounds **5**–**7** resonated as singlets at δ 3.49–3.77 ppm, and the 2-H signal was observed at δ 7.42–8.12 ppm. In the ^{13}C NMR spectra of **5**–**7**, the C^4 signal was located at 20.6–23.0 ppm; the trifluoromethyl carbon atom of **5a**–**5e** gave rise to a quartet at δ_C 116.3–116.7 ppm with a direct C–F coupling constant of 288.9–289.9 Hz, and the C^2 and C=O signals were quartets at δ_C 155.5–157.2 ($^4J_{CF}$ = 5.4 Hz) and 178.9–179.2 ppm ($^2J_{CF}$ = 34.3–35.6 Hz), respectively. The carbonyl carbon atom of chromenes **6a**, **6b**, **7a**, and **7b** resonated at δ_C 189.7–195.5 ppm. The given structures were also confirmed by the DEPT spectra.

Taking into account that the pyran ring in chromenes **5**–**7** can be readily opened by the action of nucleophiles, the obtained compounds attract interest as promising synthetic intermediates [24–26].

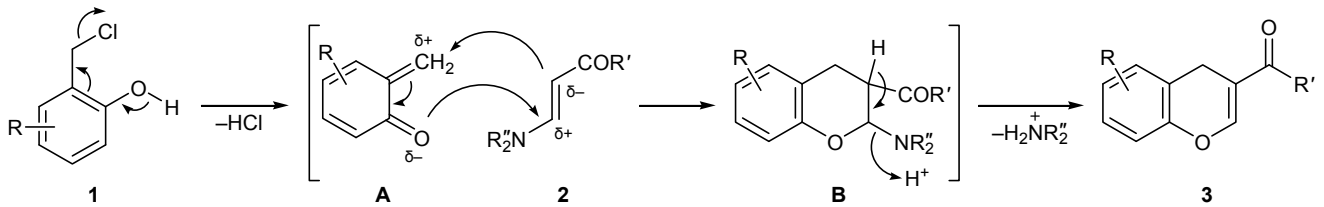
Thus, we have developed a simple one-step procedure for the synthesis of 3-acyl- and 3-formyl-4*H*-chromenes via a cascade process including Diels–Alder reaction of enamino ketones with *o*-quinone methides generated *in situ* and subsequent elimination of secondary amine.

EXPERIMENTAL

The IR spectra of solid compounds were recorded on a Shimadzu IR Affinity-1 spectrometer equipped with a Specac Diamond ATR GS 10800-B accessory. The ^1H and ^{13}C NMR spectra (including DEPT spectra) were measured on a JEOL JNM-ECX400 spectrometer at 400 and 100 MHz, respectively, using $CDCl_3$ or DMSO-d_6 (**5a**) as solvent and tetramethylsilane as internal standard. The elemental analyses were obtained on a Euro Vector EA-3000 automated CHNS analyzer. The melting points were determined in capillaries on a SRS OptiMelt MPA100 automated melting point system. Aluminum plates coated with Kieselgel 60 F-254 (Merck) were used for thin-layer chromatography; spots were detected under UV light or by treatment with iodine vapor.

Compounds 5a–5e (*general procedure*). A mixture of 1.5 mmol of 2-chloromethylphenol **1a**–**1e** and 0.31 g (1.5 mmol) of 1,1,1-trifluoro-4-(morpholin-4-yl)but-3-en-2-one (**2**) in 5 mL of acetic acid was refluxed for 5 h. The solution was cooled to room temperature, and 0.5–1 mL of water was added with

Scheme 3.



1 **A** **2** **B** **3**

stirring. If a solid separated, it was filtered off and washed with 70% aqueous acetic acid. Otherwise, an additional 15 mL of cold water was added, and the precipitate was filtered off, washed with cold water, dried in air, and purified by recrystallization from methanol.

1-(6-Acetyl-4*H*-chromen-3-yl)-2,2,2-trifluoroethanone (5a). Yield 65%, light yellow crystals, mp 120–122°C. IR spectrum, ν, cm^{-1}: 1682 (C=O), 1634 ($C=C_{pyran}$), 1580, 1493, 1425, 1360, 1277, 1231, 1196, 1175, 1132, 1111, 941, 870, 837, 735. ^1H NMR spectrum, δ, ppm: 2.51 s (3H, CH_3), 3.56 s (2H, CH_2), 7.18 d (1H, 8-H, ^3J = 8.5 Hz), 7.80 d.d (1H, 7-H, ^3J = 8.5, ^4J = 2.1 Hz), 7.87 d (1H, 5-H, ^4J = 2.1 Hz), 8.10 s (1H, 2-H). ^{13}C NMR spectrum, δ_C, ppm: 21.4 (CH_2), 27.1 (CH_3), 110.9, 116.6 q (CF_3, $^1J_{CF}$ = 288.9 Hz), 117.4 (CH), 120.0, 129.2 (CH), 131.1 (CH), 134.7, 152.1, 157.2 q (C^2, $^4J_{CF}$ = 5.4 Hz), 178.9 q (C=O, $^2J_{CF}$ = 34.3 Hz), 196.9 (C=O). Found, %: C 57.88; H 3.29. $C_{13}H_9F_3O_3$. Calculated, %: C 57.79; H 3.36.

1-[8-(Adamantan-1-yl)-6-methyl-4*H*-chromen-3-yl]-2,2,2-trifluoroethanone (5b). Yield 80%, light yellow crystals, mp 134–136°C. IR spectrum, ν, cm^{-1}: 2905, 2886, 2855 (CH_{Ad}), 1686 (C=O), 1634 ($C=C_{pyran}$), 1593, 1454, 1354, 1314, 1279, 1227, 1200, 1169, 1134, 1103, 989, 924, 868, 860, 741, 721. ^1H NMR spectrum, δ, ppm: 1.78 br.s (6H, CH_2, Ad), 2.06 br.s (6H, CH_2, Ad), 2.09 br.s (3H, CH, Ad), 2.28 s (3H, CH_3), 3.54 s (2H, CH_2), 6.80 d (1H, H_{arom}, ^4J = 1.5 Hz), 6.94 d (1H, H_{arom}, ^4J = 1.5 Hz), 7.90 s (1H, 2-H). ^{13}C NMR spectrum, δ_C, ppm: 21.1 (CH_3), 21.9 (CH_2), 29.0 (3C, CH_{Ad}), 36.9 (Ad), 37.0 (3C, CH_2, Ad), 41.0 (3C, CH_2, Ad), 110.0, 116.7 q (CF_3, $^1J_{CF}$ = 289.9 Hz), 119.1, 126.7 (CH), 127.9 (CH), 134.9, 138.1, 146.0, 156.0 q (C^2, $^4J_{CF}$ = 5.4 Hz), 179.2 q (C=O, $^2J_{CF}$ = 35.0 Hz). Found, %: C 70.29; H 6.10. $C_{22}H_{23}F_3O_2$. Calculated, %: C 70.20; H 6.16.

2,2,2-Trifluoro-1-(6-nitro-4*H*-chromen-3-yl)-ethanone (5c). Yield 63%, light yellow crystals, mp 141–143°C. IR spectrum, ν, cm^{-1}: 3113, 1690 (C=O), 1639 ($C=C_{pyran}$), 1578, 1522 (NO_2), 1479, 1335 (NO_2), 1238, 1217, 1207, 1194, 1167, 1134, 1117, 1086, 949, 920, 887, 847, 810, 748, 733. ^1H NMR spectrum, δ, ppm: 3.70 s (2H, CH_2), 7.17 d (1H, 8-H, ^3J = 9.6 Hz), 7.82 s (1H), 8.10–8.12 m (2H). ^{13}C NMR spectrum, δ_C, ppm: 21.8 (CH_2), 110.4, 116.3 q (CF_3, $^1J_{CF}$ = 288.9 Hz), 118.0 (CH), 120.6, 124.3 (CH), 125.7 (CH), 145.0, 153.2, 155.5 q (C^2, $^4J_{CF}$ = 5.4 Hz), 178.9 q (C=O, $^2J_{CF}$ = 35.3 Hz). Found, %: C 48.25; H 2.26; N 5.06. $C_{11}H_6F_3NO_4$. Calculated, %: C 48.37; H 2.21; N 5.13.

1-(6,8-Dibromo-4*H*-chromen-3-yl)-2,2,2-trifluoroethanone (5d). Yield 44%, colorless crystals, mp 114–116°C. IR spectrum, ν, cm^{-1}: 3071, 1689 (C=O), 1634 ($C=C_{pyran}$), 1593, 1558, 1452, 1435, 1244, 1179, 1167, 1146, 939, 932, 868, 853, 754, 735, 704. ^1H NMR spectrum, δ, ppm: 3.60 s (2H, CH_2), 7.25 d (1H, H_{arom}, ^4J = 2.3 Hz), 7.58 d (1H, H_{arom}, ^4J = 2.3 Hz), 7.86 s (1H, 2-H). ^{13}C NMR spectrum, δ_C, ppm: 22.0 (CH_2), 110.4, 112.2, 116.3 q (CF_3, $^1J_{CF}$ = 288.9 Hz), 118.2, 122.5, 131.5 (CH), 134.7 (CH), 145.4, 155.8 q (C^2, $^4J_{CF}$ = 5.4 Hz), 178.9 q (C=O, $^2J_{CF}$ = 35.6 Hz). Found, %: C 34.34; H 1.27. $C_{11}H_5Br_2F_3O_2$. Calculated, %: C 34.23; H 1.31.

[6-Methoxy-3-(2,2,2-trifluoroacetyl)-4*H*-chromen-8-yl]methyl acetate (5e). Yield 57%, light yellow crystals, mp 113–115°C. IR spectrum, ν, cm^{-1}: 3094, 1748 (C=O, ester), 1686 (3-C=O), 1638 ($C=C_{pyran}$), 1620, 1597, 1478, 1385, 1256, 1211, 1186, 1167, 1138, 1074, 1040, 1015, 974, 932, 872, 858, 737. ^1H NMR spectrum, δ, ppm: 2.11 s (3H, CH_3), 3.57 s (2H, CH_2), 3.78 s (3H, CH_3O), 5.14 s (2H, CH_2OAc), 6.62 d (1H, H_{arom}, ^4J = 2.8 Hz), 6.78 d (1H, H_{arom}, ^4J = 2.8 Hz), 7.85 s (1H, 2-H). ^{13}C NMR spectrum, δ_C, ppm: 21.0 (CH_3), 22.1 (CH_2), 55.7 (CH_2), 60.7 (CH_3O), 109.6, 113.8 (CH), 114.5 (CH), 116.5 q (CF_3, $^1J_{CF}$ = 288.9 Hz), 120.3, 125.5, 140.9, 156.4 q (C^2, $^4J_{CF}$ = 5.4 Hz), 156.8, 170.7 (C=O), 179.2 q (C=O, $^2J_{CF}$ = 35.3 Hz). Found, %: C 54.65; H 4.06. $C_{15}H_{13}F_3O_5$. Calculated, %: C 54.55; H 3.97.

Compounds 6a, 6b, 7a, and 7b (*general procedure*). A mixture of 1.5 mmol of 2-(chloromethyl)-phenol **1a** or **1b** and 1.5 mmol of enaminone **3** or **4** in 5 mL of acetic acid was refluxed for 1 h. The solvent was distilled under reduced pressure, and the residue was recrystallized from isopropyl alcohol.

6-Acetyl-4*H*-chromene-3-carbaldehyde (6a). Yield 56%, colorless crystals, mp 133–135°C. IR spectrum, ν, cm^{-1}: 1680 (C=O), 1672 (C=O), 1645 ($C=C_{pyran}$), 1578, 1495, 1435, 1423, 1362, 1275, 1233, 1190, 1175, 1150, 1113, 966, 899, 826, 773. ^1H NMR spectrum, δ, ppm: 2.57 s (3H, CH_3), 3.58 s (2H, CH_2), 7.06 d (1H, 8-H, ^3J = 8.5 Hz), 7.38 s (1H, 5-H), 7.78–7.81 m (2H, 2-H, 7-H), 9.48 s (1H, CHO). ^{13}C NMR spectrum, δ_C, ppm: 20.6 (CH_2), 26.6 (CH_3), 117.3 (CH), 118.5, 119.9, 128.8 (CH), 130.8 (CH), 134.3, 153.6, 158.5 (CH), 189.7 (CHO), 196.6 (C=O). Found, %: C 71.35; H 4.89. $C_{12}H_{10}O_3$. Calculated, %: C 71.28; H 4.98.

8-(Adamantan-1-yl)-6-methyl-4*H*-chromene-3-carbaldehyde (6b). Yield 60%, colorless crystals, mp 196–198°C. IR spectrum, ν, cm^{-1}: 2903, 2849 (CH_{Ad}), 1680 (C=O), 1659 ($C=C_{pyran}$), 1593, 1450, 1435, 1398, 1342, 1315, 1273, 1200, 1190, 1190, 1175, 989, 876, 860, 797, 770, 716. ^1H NMR spectrum, δ, ppm: 1.78 br.s (6H, CH_2, Ad), 2.08 br.s (9H, CH, CH_2, Ad), 2.27 s (3H, CH_3), 3.49 s (2H, CH_2), 6.79 s (1H, H_{arom}), 6.92 s (1H, H_{arom}), 7.45 s (1H, 2-H), 9.45 s (1H, CHO). ^{13}C NMR spectrum, δ_C, ppm: 20.9 (CH_2), 21.1 (CH_3), 29.1 (3C, CH, Ad), 36.97 (Ad), 37.03 (3C, CH_2, Ad), 41.0 (3C, CH_2, Ad), 117.9, 119.5, 126.5 (CH), 128.2 (CH), 134.3, 138.1, 147.2, 158.7 (CH), 190.2 (CHO). Found, %: C 81.86; H 7.75. $C_{21}H_{24}O_2$. Calculated, %: C 81.78; H 7.84.

1-(3-Benzoyl-4*H*-chromen-6-yl)ethanone (7a). Yield 78%, colorless crystals, mp 167–169°C. IR spectrum, ν, cm^{-1}: 3055, 1682 (C=O), 1641, 1620, 1612, 1578, 1493, 1445, 1385, 1360, 1331, 1298, 1273, 1256, 1234, 1173, 1163, 1123, 924, 908, 860, 820, 702, 692. ^1H NMR spectrum, δ, ppm: 2.58 s (3H, CH_3), 3.77 s (2H, CH_2), 7.02 d (1H, H_{arom}, ^3J = 8.5 Hz), 7.34 s (1H, H_{arom}), 7.43–7.47 m (2H, H_{arom}), 7.51–7.56 m (1H, H_{arom}), 7.63 s (1H, 2-H), 7.64 d (1H, H_{arom}, ^3J = 8.5 Hz), 7.79 d.d (1H, H_{arom}, ^3J = 8.5, ^4J = 1.8 Hz), 7.82 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 22.7 (CH_2), 26.6 (CH_3), 115.4, 116.9 (CH), 120.4, 128.55 (2C, CH), 128.59 (CH), 128.8 (2C, CH), 130.7 (CH), 131.8 (CH), 134.0, 138.3, 153.3, 154.6 (CH), 194.9 (C=O), 196.7 (C=O). Found, %: C 77.78; H 5.00. $C_{18}H_{14}O_3$. Calculated, %: C 77.68; H 5.07.

[8-(Adamantan-1-yl)-6-methyl-4*H*-chromen-3-yl]phenylmethanone (7b). Yield 72%, colorless crystals, mp 140–142°C. IR spectrum, ν, cm^{-1}: 2911, 2897, 2878, 2849 (CH_{Ad}), 1647 (C=O), 1628 ($C=C_{pyran}$), 1593, 1447, 1393, 1317, 1290, 1206, 1179, 1103, 989, 926, 866, 849, 725, 702, 683. ^1H NMR spectrum, δ, ppm: 1.74 br.s (6H, CH_2, Ad), 2.05 br.s (9H, CH_2, CH, Ad), 2.29 s (3H, CH_3), 3.69 s (2H, CH_2), 6.85 s (1H, H_{arom}), 6.91 s (1H, H_{arom}), 7.42 s (1H, 2-H), 7.44–7.48 m (2H, H_{arom}), 7.50–7.55 m (1H, Ph), 7.61–7.65 m (2H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 21.2 (CH_3), 23.0 (CH_2), 29.1 (3C, CH, Ad), 36.9 (Ad), 37.0 (3C, CH_2, Ad), 41.0 (3C, CH_2, Ad), 115.1, 120.2, 126.3 (CH), 128.1 (CH), 128.5 (2C, CH), 128.7 (2C, CH), 131.3 (CH), 134.0, 137.8, 139.0, 146.7, 155.1 (CH), 195.5 (C=O). Found, %: C 84.26; H 7.41. $C_{27}H_{28}O_2$. Calculated, %: C 84.34; H 7.34.

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