A New Route for the Synthesis of Flavanones from 2-Methoxybenzoic Acids

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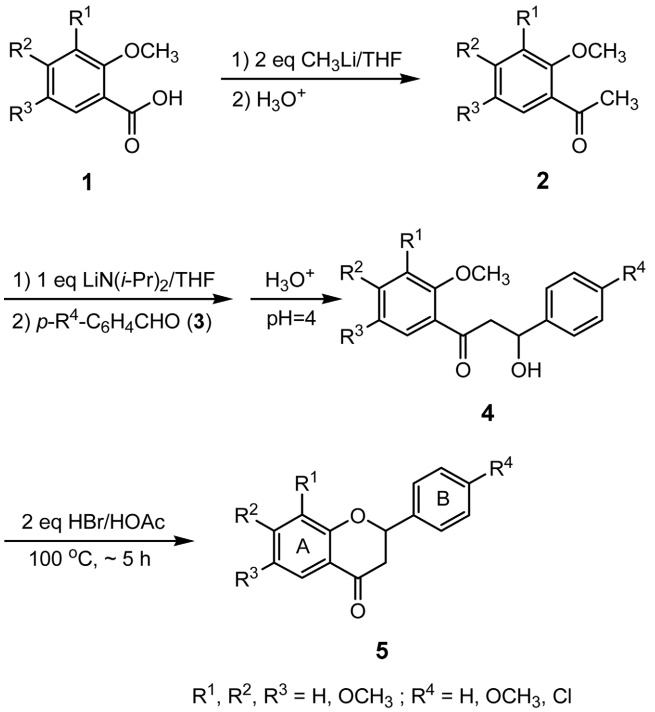
The flavanones (2-phenylchroman-4-ones) are mainly found from plants of the family Leguminosae, Compositae, and Moraceae[1] and have attracted a good deal of attention because they possess pharmacological activities, such as antioxidant effect, toxicity to several bacteria, and inhibition of hormone-dependent proliferation of cancer cells.[2] The most widely used route to flavanones is the oxidative cyclization of 2'-hydroxychalcones which are prepared from 2'-hydroxyacetophenones and benzaldehydes with 2 equiv of base using various reagents.[3] The reaction of 2'-hydroxychalcones with palladium(II) acetate,[4a] Co(salpr) under oxygen atmosphere,[4b] and potassium ferricyanide using phase transfer catalysis[4c] leads to the formation of flavanones, but the yields are low to moderate. Although 2'-hydroxychalcones may be also cyclized to flavanones with acidic reagents such as silica gel,[5a] H_2SO_4 in methanol,[5b] CF_3COOH,[5c] and polyphosphoric acid[5d] or basic reagents such as pyridine[6a] and DBU in microwave irradiation,[6b] the most employed methods require for prolonged reaction time at high temperature. Alternatively flavanones are prepared from the oxidative cyclization of cinnamic acids and phenols with polyphosphoric acid[7] or 2'-hydroxyacetophenones and benzaldehydes with piperidine[8] in one step, but the yields of the former are very low and the latter are not applicable for flavanones.

However, the preparation of 2'-hydroxychalcones from 2'-hydroxyacetophenones and benzaldehydes has underwent troublesomeness because they are always cyclized to flavanones partially during their synthesis.[9] The synthetic strategy to avoid this undesirable reaction is to prepare β-hydroxyketones as precursors of flavanones from 2'-hydroxyacetophenones and benzaldehydes. For example, the reaction of 2'-hydroxyacetophenones and acetaldehyde with LDA affords 1-(2'-hydroxyphenyl)-1-oxo-butan-3-ols, which are cyclized with HCl in methanol,[10a] HMPT,[10b] and H_2SO_4 in acetic acid[10c] to give the corresponding chroman-4-ones, but there are no reports on the preparation of flavanones with 2-substituted phenyl groups. The use of 2'-methoxyacetophenones can also avoid the undesirable cyclization of chalcones to flavanones and furthermore they are generally cheaper than 2'-hydroxyacetophenones. In this paper we report that flavanones can be synthesized *via* 1-(2'-methoxyphenyl)-1-oxo-propan-3-phenyl-3-ols from 2'-methoxyacetophenones as a new synthetic route.

2'-Methoxyacetophenones **2** were readily prepared by the treatment of 2-methoxybenzoic acids **1** with 2 equiv of methyllithium in THF for 0.5-2 h at −78 °C in 88-93% yields according to our previous method.[11] The key intermediates, 1-(2'-methoxyphenyl)-1-oxo-propan-3-phenyl-3-ols **4**, as precursors of flavanones **5** were prepared by the treatment of the lithium enolate of **2** with benzaldehydes (Scheme 1). To a yellowish solution of the lithium enolate, generated from 2'-methoxyacetophenones and 1 equiv of LDA in THF for 2 h at −20 °C, were added benzaldehydes at −78 °C. After being stirred for 1 h between −78 °C and −40 °C, the mixture was carefully acidified with 0.1 N-HCl up to pH=4. The mixture was separated by extraction and purified by recrystallization and/or silica gel chromatography in EtOAc/*n*-hexane to afford **4** in high yields (77-92%). The reaction proceeded well regardless of the kind of substituents (methoxy, chloro) on both 2'-methoxyacetophenones and benzaldehydes without the formation of 2'-methoxychalcones under the present reaction conditions.

The cyclization of **4** proceeded *via* the dehydration of the β-hydroxyl group and the successive conjugate addition of the phenolic OH which are generated from the cleavage of 2'-methoxy group. The initial reaction of 1-(2'-methoxyphenyl)-1-oxo-propan-3-(4'-chlorophenyl)-3-ol with 2 equiv of 48% HBr in glacial acetic acid afforded the corresponding 2'-methoxychalcone as a major product after 7 h at room temperature together with the corresponding 2'-hydroxychalcone. The reaction of 1-(2'-methoxyphenyl)-1-oxo-propan-3-(4'-chlorophenyl)-3-ol with 2 equiv of 48% HBr in methanol also produced only the corresponding 2'-methoxychalcone in 95% yield after 4 h at 100 °C. However, the cyclization of 1-(2'-methoxyphenyl)-1-oxo-propan-3-(4'-chlorophenyl)-3-ol was successfully accomplished by heating with 2 equiv of 48% HBr in glacial acetic acid and 4'-chloroflavanone was obtained in 71% yield. The use of 2 equiv of 47% HI was also effective, but the yield of 4'-chloroflavanone was decreased to 58%.

As shown in Table 1, various flavanones were synthesized in high yields by this method. The reaction worked well both for the methoxy substituent (**5d**-**5j**) on the A-ring and the methoxy (**5c**, **5h**) or chloro substituent (**5b**, **5e**, **5g**, **5j**) on the B-ring of **5**. During the cyclization 6 or 7-methoxy group of A-ring and 4'-methoxy group of B-ring were not cleaved under the present reaction conditions. Thus, the present



Scheme 1

method provides some advantages over the previous methods with respect to (i) the avoidance of isomerization between 2'-methoxychalcones and flavanones using 2'-methoxy protective group (ii) the cheapness of 2'-methoxyacetophenones over 2'-hydroxyacetophenones in general (iii) the use of 1 equiv of LDA for the preparation of **4** (iv) the high yield synthesis of **5**.

Experimental Section

Preparation of 1-(2'-methoxyphenyl)-1-oxo-propan-3-phenyl-3-ol (General procedure). To a solution of 2'-methoxyacetophenone (600.7 mg, 4.0 mmol) in THF (12 mL) was added lithium diisopropylamide (2.0 M, 2.1 mL, 4.2 mmol) at −20 °C under argon atmosphere and stirred for 2 h. The temperature of the mixture was then lowered to −78 °C and a solution of benzaldehyde (424.5 mg, 4.0 mmol) in THF (6 mL) was added. After being stirred for 1 h between −78 °C and −40 °C, the mixture was acidified with 0.1 N HCl up to pH 4. After evaporation of THF, the mixture was poured into sat. NH_4Cl (30 mL) and the aqueous phase was extracted with dichloromethane (3 × 25 mL). The combined organic phases were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography using 30% EtOAc/*n*-hexane to give 1-(2'-methoxyphenyl)-1-oxo-propan-3-phenyl-3-ol (922.7 mg, 90%). M.p. 72-73 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (dd, J_1 = 7.8 Hz, J_2 = 1.8 Hz, 1H), 7.25-7.52 (m, 6H), 6.95-7.04 (m, 2H), 5.92 (d, J = 6.3 Hz, 1H), 3.86 (s, 3H), 3.66 (d, J = 3.0 Hz, 1H), 3.47 (dd, J_1 = 18.0 Hz, J_2 = 2.9 Hz, 1H), 3.35 (dd, J_1 = 18.0 Hz, J_2 = 9.3 Hz, 1H); FT-IR (KBr) 3479 (OH), 3029, 2942, 1666 (C=O), 1596, 1464, 1245, 1022, 759, 700 cm^{-1}; Ms *m/z* (%) 150 (18), 136 (9), 135 (100), 105 (3), 92 (14), 77 (29).

Table 1. Preparation of Flavanones from 2-Methoxybenzoic Acids

Products	R^1	R^2	R^3	R^4	Isolated yield, %[a]
5a	H	H	H	H	55
5b	H	H	H	Cl	57
5c	H	H	H	OCH_3	38
5d	H	OCH_3	H	H	62
5e	H	OCH_3	H	Cl	45
5f	H	H	OCH_3	H	54
5g	H	H	OCH_3	Cl	46
5h	H	H	OCH_3	OCH_3	47
5i	OCH_3	OCH_3	H	H	41
5j	OCH_3	OCH_3	H	Cl	42

[a]Overall yields of three steps from the starting 2-methoxybenzoic acids.

Preparation of flavanone 5a (General procedure). A solution of 1-(2'-methoxyphenyl)-1-oxo-propan-3-phenyl-3-ol (768.9 mg, 3.0 mmol) and hydrobromic acid (48 wt. % in H_2O, 679 μL, 6.0 mmol) in glacial acetic acid (9 mL) was heated to 100 °C for 5 h. After evaporation of the solvent, the mixture was poured into sat. $NaHCO_3$ (30 mL) and the aqueous phase was extracted with dichloromethane (3 × 20 mL). The combined organic phases were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was recrystallized from 95% *n*-hexane/EtOAc to give **5a** (444.0 mg, 66%) as a colorless solid. M.p. 76-77 °C (lit.[5a] 76 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.94 (dd, J_1 = 8.1 Hz, J_2 = 1.7 Hz, 1H), 7.37-7.55 (m, 6H), 7.04-7.09 (m, 2H), 5.50 (dd, J_1 = 13.2 Hz, J_2 = 3.0 Hz, 1H), 3.10 (dd, J_1 = 16.9 Hz, J_2 = 13.2 Hz, 1H), 2.90 (dd, J_1 = 16.9 Hz, J_2 = 3.0 Hz, 1H); FT-IR (KBr) 3005, 2961, 1690 (C=O), 1605, 1463, 1252, 1027, 764 cm^{-1}; ^{13}C NMR (75 MHz, $CDCl_3$) δ 192.4, 162.0, 139.1, 136.6, 129.3, 129.2, 127.5, 126.6, 122.0, 121.3, 118.5, 80.0, 45.1; Ms *m/z* (%) 224 (M^+, 100), 223 (96), 147 (59), 120 (97), 105 (39), 92 (52), 77 (17).

4'-Chloroflavanone (5b). M.p. 85 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 (dd, J_1 = 7.7 Hz, J_2 = 1.4 Hz, 1H), 7.50-7.53 (m, 1H), 7.38-7.44 (m, 4H), 7.04-7.10 (m, 2H), 5.47 (dd, J_1 = 13.0 Hz, J_2 = 3.2 Hz, 1H), 3.05 (dd, J_1 = 16.9 Hz, J_2 = 13.0 Hz, 1H), 2.88 (dd, J_1 = 16.9 Hz, J_2 = 3.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.5, 161.3, 137.3, 136.3, 134.6, 129.1, 127.5, 127.1, 121.8, 120.9, 118.1, 78.8, 44.6; FT-IR (KBr) 3033, 2900, 1696 (C=O), 1601, 1471, 1229, 1015, 818 cm^{-1}; Ms *m/z* (%) 260 (M^++2, 21), 259 (18), 258 (M^+, 63), 257 (56), 147 (43), 120 (100), 92 (58), 77 (13).

4'-Methoxyflavanone (5c). M.p. 97 °C (lit.[4c] 98 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 (dd, J_1 = 8.1 Hz, J_2 = 1.8 Hz, 1H), 7.48-7.53 (m, 1H), 7.42 (d, J = 8.6 Hz, 2H), 7.03-7.08 (m, 2H), 6.96 (d, J = 8.6 Hz, 2H), 5.44 (dd, J_1 = 13.2 Hz, J_2 = 2.8 Hz, 1H), 3.84 (s, 3H), 3.12 (dd, J_1 = 16.8 Hz, J_2 = 13.2 Hz, 1H), 2.87 (dd, J_1 = 16.8 Hz, J_2 = 2.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 192.7, 162.0, 160.4, 136.6, 131.2, 128.1, 127.4, 121.9, 121.3, 118.5, 114.6, 79.8, 55.8, 44.9; FT-IR (KBr) 3084, 2964, 1693 (C=O), 1604, 1463, 1263,

1028, 829 cm^{-1}; Ms *m/z* (%) 254 (M^+, 54), 253 (43), 134 (100), 147 (6), 121 (27), 92 (11).

7-Methoxyflavanone (5d). M.p. 91-92 °C (lit.[4a] 90-91 °C); ^1H NMR (300 MHz, $CDCl_3$) δ 7.88 (d, J = 8.8 Hz, 1H), 7.36-7.50 (m, 5H), 6.62 (dd, J_1 = 8.8 Hz, J_2 = 2.3 Hz, 1H), 6.51 (d, J = 2.3 Hz, 1H), 5.48 (dd, J_1 = 13.2 Hz, J_2 = 3.0 Hz, 1H), 3.84 (s, 3H), 3.05 (dd, J_1 = 16.9 Hz, J_2 = 13.2 Hz, 1H), 2.83 (dd, J_1 = 16.9 Hz, J_2 = 3.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.0, 166.6, 163.9, 139.2, 129.3, 129.2, 126.6 (overlapped), 115.2, 110.7, 101.3, 80.4, 56.1, 44.7; FT-IR (KBr) 3065, 2967, 1682 (C=O), 1606, 1442, 1257, 1023, 764, 699 cm^{-1}; Ms *m/z* (%) 254 (M^+, 45), 253 (37), 147 (7), 134 (100), 121 (26), 92 (11).

4'-Chloro-7-methoxyflavanone (5e). M.p. 120-121 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, J = 8.8 Hz, 1H), 7.37-7.48 (m, 4H), 6.63 (dd, J_1 = 8.8 Hz, J_2 = 2.3 Hz, 1H), 6.50 (d, J = 2.3 Hz, 1H), 5.45 (dd, J_1 = 12.9 Hz, J_2 = 3.2 Hz, 1H), 3.85 (s, 3H), 2.99 (dd, J_1 = 16.8 Hz, J_2 = 12.9 Hz, 1H), 2.82 (dd, J_1 = 16.8 Hz, J_2 = 3.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.5, 166.6, 163.7, 137.7, 135.0, 129.4, 129.2, 127.9, 115.2, 110.8, 101.3, 79.6, 56.1, 44.6; FT-IR (KBr) 3091, 2965, 1680 (C=O), 1608, 1492, 1258, 1057, 830 cm^{-1}; Ms *m/z* (%) 290 (M^++2, 33), 288 (M^+, 100), 177 (64), 150 (86), 122 (38), 107 (20).

6-Methoxyflavanone (5f). M.p. 141-142 °C (lit.[4a] 140-142 °C); ^1H NMR (300 MHz, $CDCl_3$) δ 7.38-7.51 (m, 5H), 7.36 (d, J = 3.1 Hz, 1H), 7.13 (dd, J_1 = 9.0 Hz, J_2 = 3.1 Hz, 1H), 7.00 (d, J = 9.0 Hz, 1H), 5.45 (dd, J_1 = 13.3 Hz, J_2 = 3.0 Hz, 1H), 3.82 (s, 3H), 3.08 (dd, J_1 = 17.0 Hz, J_2 = 13.3 Hz, 1H), 2.88 (dd, J_1 = 17.0 Hz, J_2 = 3.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 192.5, 156.7, 154.6, 139.2, 129.2, 129.1, 126.5, 125.8, 121.2, 119.8, 107.7, 80.1, 56.2, 45.0; FT-IR (KBr) 3034, 2959, 1673 (C=O), 1615, 1484, 1460, 1283, 1034, 769, 697 cm^{-1}; Ms *m/z* (%) 254 (M^+, 62), 253 (13), 177 (15), 150 (100), 135 (10), 107 (18).

4'-Chloro-6-methoxyflavanone (5g). M.p. 115-116 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.39-7.44 (m, 4H), 7.37 (d, J = 3.2 Hz, 1H), 7.15 (dd, J_1 = 9.0 Hz, J_2 = 3.2 Hz, 1H), 7.01 (d, J = 9.0 Hz, 1H), 5.44 (dd, J_1 = 13.0 Hz, J_2 = 3.2 Hz, 1H), 3.84 (s, 3H), 3.04 (dd, J_1 = 16.9 Hz, J_2 = 13.0 Hz, 1H), 2.88 (dd, J_1 = 16.9 Hz, J_2 = 3.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 192.0, 156.4, 154.7, 137.8, 134.9, 129.4, 127.9, 125.9, 121.1, 119.8, 107.8, 79.3, 56.2, 44.9; FT-IR (KBr) 3070, 2996, 1685 (C=O), 1617, 1487, 1281, 1063, 826 cm^{-1}; Ms *m/z* (%) 290 (M^++2, 13), 288 (M^+, 37), 177 (6), 150 (100), 135 (8), 107 (15).

4',6-Dimethoxyflavanone (5h). M.p. 157-158 °C (lit.[4a] 159-160 °C); ^1H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, J = 8.7 Hz, 2H), 7.35 (d, J = 3.2 Hz, 1H), 7.12 (dd, J_1 = 9.0 Hz, J_2 = 3.2 Hz, 1H), 6.97 (d, J = 9.0 Hz, 1H), 6.96 (d, J = 8.7 Hz, 2H), 5.39 (dd, J_1 = 13.3 Hz, J_2 = 2.9 Hz, 1H), 3.84 (s, 3H), 3.82 (s, 3H), 3.09 (d, J_1 = 16.9 Hz, J_2 = 13.3 Hz, 1H), 2.85 (dd, J_1 = 16.9 Hz, J_2 = 2.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 192.3, 159.9, 156.4, 154.2, 130.9, 127.7, 125.4, 120.7, 119.4, 114.2, 107.3, 79.5, 55.8, 55.4, 44.4; FT-IR (KBr) 3004, 2969, 1682 (C=O), 1612, 1486, 1276, 1027, 826 cm^{-1}; Ms *m/z* (%) 284 (M^+, 83), 150 (86), 139 (19), 134 (100), 107 (17).

7,8-Dimethoxyflavanone (5i). M.p. 114 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, J = 8.9 Hz, 1H), 7.38-7.52 (m, 5H), 6.68 (d, J = 8.9 Hz, 1H), 5.52 (dd, J_1 = 12.4 Hz, J_2 = 3.3 Hz, 1H), 3.94 (s, 3H), 3.88 (s, 3H), 3.05 (dd, J_1 = 16.9 Hz, J_2 = 12.4 Hz, 1H), 2.90 (dd, J_1 = 16.9 Hz, J_2 = 3.3 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.3, 159.2, 155.8, 139.3, 137.4, 129.2, 129.0, 126.4, 123.3, 116.6, 106.1, 80.3, 61.5, 56.7, 44.8; FT-IR (KBr) 3065, 2969, 1685 (C=O), 1598, 1451, 1286, 1089, 763, 700 cm^{-1}; Ms *m/z* (%) 284 (M^+, 100), 207 (18), 180 (43), 152 (79), 151 (24).

4'-Chloro-7,8-dimethoxyflavanone (5j). M.p. 115-116 °C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, J = 8.9 Hz, 1H), 7.38-7.46 (m, 4H), 6.68 (d, J = 8.9 Hz, 1H), 5.50 (dd, J_1 = 12.0 Hz, J_2 = 3.6 Hz, 1H), 3.94 (s, 3H), 3.87 (s, 3H), 3.00 (dd, J_1 = 16.7 Hz, J_2 = 12.0 Hz, 1H), 2.88 (dd, J_1 = 16.7 Hz, J_2 = 3.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.5, 158.9, 155.1, 137.4, 137.0, 134.5, 129.0, 127.5, 123.0, 116.1, 105.9, 79.1, 61.1, 56.3, 44.3; FT-IR (KBr) 2936, 1684 (C=O), 1598, 1453, 1287, 1088, 802 cm^{-1}; Ms *m/z* (%) 320 (M^++2, 24), 318 (M^+, 73), 207 (16), 180 (73), 152 (100), 151 (34), 137 (35).

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