Chinese Chemical Letters Vol. 12, No. 11, pp 957 – 958, 2001
http://www.imm.ac.cn/journal/ccl.html

A Facile Synthetic Approach to Two Chalcones

Zhen Ting DU, Shao Bai LI*

National Laboratory of Applied Organic Chemistry,
Institute of Organic Chemistry, Lanzhou University, Lanzhou 730000

Abstract: A facile synthetic route for two chalcone analogues was developed. The key step was selective deprotection of MOM in aryl methyl ether **6** by silica gel.

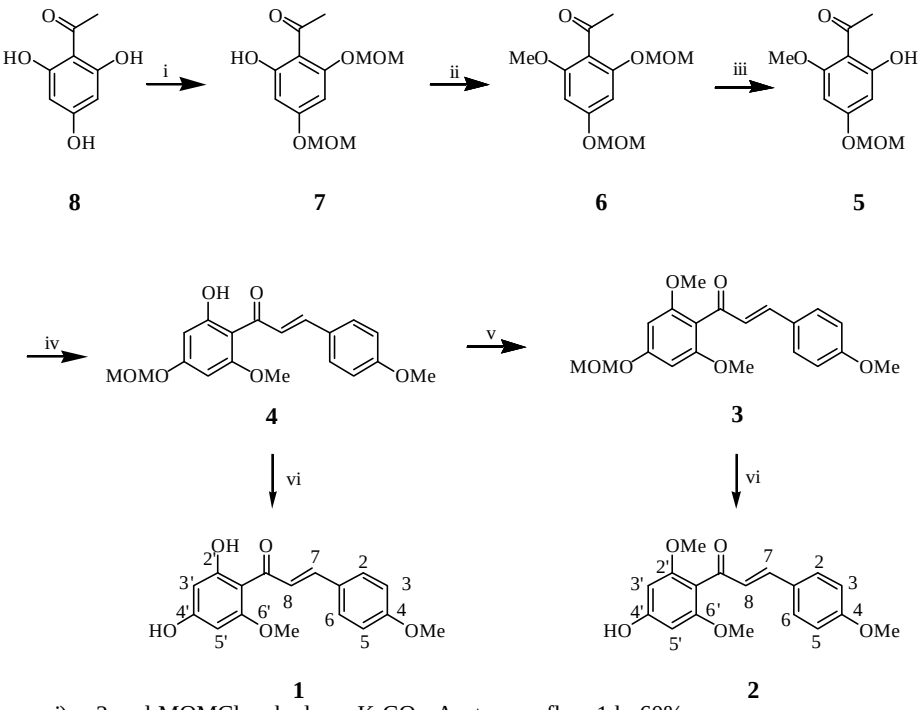
Keywords: Chalcone, synthesis.

Some *Vitex* species have long been used in folk remedies due to their excellent antibiotic activities[1]. Chalcones also have good activities such as immunological action[2]. 2′, 4′-dihydroxy-4, 6′-dimethoxy chalcone **1** and 4′-hydroxy-4, 2′, 6′-trimethoxy chalcone **2** were natural products which were firstly separated from the aerial parts of *Vitex leptobotrys* in North Vietnam by Trinh[3]. To our knowledge, no synthetic method about these compounds was reported. Herein we report a convenient synthetic route for these compounds. The spectroscopy data of synthetic **1** and **2** were identical with those of the natural products.

2, 4, 6-trihydroxyacetobenzene **8** was used as starting material. Two moles MOMCl and K_2CO_3 were used to protect the 2, 4 hydroxy groups affording the 2,4 dimethoxymethyl 6-hydroxy phenylacetone **7** in 60% yield. **7** was methylated by Me_2SO_4 in acetone to give **6** in 97% yield, which was extracted with ether, dried over $MgSO_4$. The solvent was evaporated in *vacuo*, some amount of silica gel was added to the syrup as mild acetic medium to deprotect MOM group at 2 position. The deprotection of MOM group at 2-position proceed easily due to the electron withdrawing effect of neighboring acetic group while the protecting group MOM at 4-position is not easy to deprotect. In the result **5** was obtained through chromatography. **5** was condensed with anisaldehyde in KOH-H_2O-EtOH solution to give **4**, **4** was methylated by the phase-transfer method[4] to obtain **3**, **4** and **3** were deprotected the MOM in acidic CH_3OH to give **1** and **2**[5,6].

* E-mail: bsli@lzu.edu.cn

Scheme 1



i) 2 mol MOMCl, anhydrous K_2CO_3, Acetone, reflux, 1 h; 60%;
ii) Me_2SO_4, K_2CO_3, Acetone, reflux, 1 h; 97%;
iii) Silica Gel, (75 μm) chromatography; 97%;
iv) anisaldehyde, KOH, EtOH, H_2O; 86%;
v) NaOH, H_2O, $(n\text{-}C_4H_9)_4NI$, Me_2SO_4; 98%;
vi) 3 mol/L HCl, MeOH, reflux; 98%.

References and notes

1. *Dictionary of the Traditional Chinese Medicines*, 1st. Ed., Peoples Health Publisher, Beijing, **1977**, p.1130.
2. V. S. Parmar, S. C. Jain, K. B. Bisht, *et al*., *Indian. J. Chem. Sect. B*, **1998**, *37B*, 628.
3. T. T. Trinh, R. Andrea, R. Helmut, V. S. Tran, A. Gunter, *Phytochemistry*, **1998**, *49*, 2603.
4. A. McKillop, J. C. Fiaud, R. P. Hug, *Tetrahydron*, **1998**, *30*, 1397.
5. **1**, mp 168-170°C (lit[1], 170-172°C), ^{1}HNMR (200MHz, $CDCl_3$, δ *ppm*): 3.87 (s, 3H, OCH_3-6′), 3.90 (s, 3H, OCH_3-4), 5.95 (d, 1H, *J*=2.4Hz, H-3′), 6.02 (d, 2H, *J*=2.4Hz, H-5′), 6.93 (dd, 2H, *J*=8.0Hz, 2.0Hz, H-3, 5), 7.56 (dd, 2H, *J*=8.0, 2.0Hz, H-2, 6), 7.78 (s, 2H, H-7, 8)[6], 14.25 (s, 1H, OH-2′); EIMS (*m/z*): 300 (M^+, 50), 44 (100). ESI positive: M+H=315.1227, (calcd.: 315.1234). **2**, mp 200-202℃, (lit[1], 208-210°C), ^{1}HNMR (200MHz, $CDCl_3$, δ *ppm*): 3.73 (s, 6H, OCH_3-2′, 6′), 3.84 (s, 3H, OCH_3-4), 6.10 (s, 2H, H-3′, 5′), 6.85 (d, 1H, *J*=16Hz, H-8), 6.89 (d, 2H, *J*=8.4Hz, 2.2Hz, H-3, 5), 7.34 (d, 1H, *J*=16Hz, H-7), 7.48 (d, 2H, *J*=8.4Hz, 2.2Hz, H-2, 6); EIMS (*m/z*): 314 (M^+, 32), 299 (26), 286 (100). ESI positive: M+H=301.1065, (calcd.: 301.1064).
6. Measured in Me_2CO-d_6: δ 7.91 (d, 1H, *J*=15.6Hz, H-7), δ 7.74 (d, 1H, *J*=15.6Hz, H-8).

Received 1 February, 2001
Revised 3 September, 2001