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Synthesis of some Chromene derivatives

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Abstract : In the present study the synthesis of chromene derivatives **3a-3d** and **4a-4c** have been reported by the reaction of Salicylaldehyde with 2-cyclohexen-1-one/ 2-cyclopenten-1-one in the presence of 1,4-diazabicyclo[2.2.2]octane (DABCO). The synthesized compounds have been characterized by TLC, Elemental analysis, IR and ^{1}H-NMR Spectroscopy. This condensation method offers an alternative route for the novel synthesis of chromenes in reasonable yields making the process more economic than other conventional methods.

Key Words : Chromene Salicylaldehydes,2-Cyclohexen-1-one/Cyclopenten-1-one,DABSCO.

Introduction

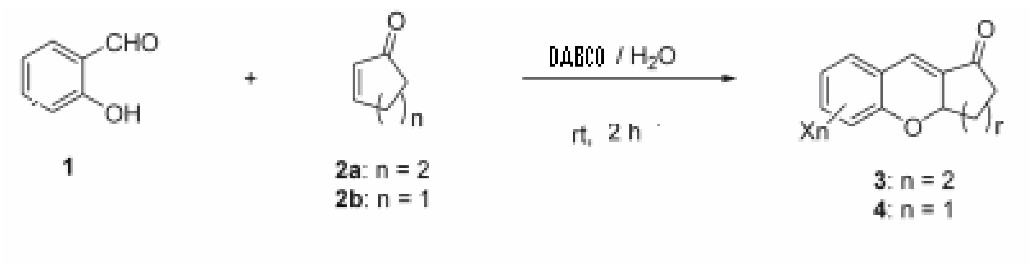
2H-1-Benzopyrans (2H-Chromenes) are important intermediates in the synthesis of many natural products and medicinal agents[1-4]. Some chromene derivatives might prove useful synthetic intermediates for the synthesis of certain naturally occurring substances, such as Miroestrol. Also, in a study of the photochemistry of some structurally related unsaturated ketones, the chromenes were needed. Thus, various synthetic methods for the formation of these compounds have been reported but the isolated yields were very low. Literature preparations of 2H-1-benzopyrans generally give low to moderate yields of product[5-8] and this prompted us to investigate improved procedures toward these compounds.

It has been reported[9-25] that polyfunctional vinylic compounds can be prepared by using 1,4-diazabicyclo[2.2.2]octane (**DABCO**) as a catalyst by coupling various aldehydes with monofunctional vinylic substrates. Accidently we undertook an investigation to prepare chromene derivatives. We have successfully established the utility of DABCO for heterocyclic synthesis[9,13,21,25]. In this paper, we wish to report a practical and highly efficient procedure for preparing 2H-1-chromene derivatives. The structures of compounds **3a-3d** and **4a-4c** were deduced from their elemental analyses, IR , ^{1}H and ^{13}C -NMR spectra.

Experimental

Melting points were determined in an open capillary tube with a Buchi melting point apparatus and are uncorrected. Elemental analyses were carried out using Perkin-Elmer 240C CHN-analyzer. IR spectra were recorded on a FT-IR spectrophotometer.^{1}H- NMR spectra was run in (DMSO-d_6) solvent at 200 MHz on a NMR spectrophotometers(chemical shifts in δ ppm).

Scheme:1



General Procedure: To a mixture of Salicylaldehyde (0.61 g, 5Mm) in water (5mL) and 20 mol% of 1,4-diazabicyclo[2.2.2]octane followed by 10 mM of 2-cyclohexen-1-one/ 2-cyclopenten-1-one added and the stirring continued at room temperature for 2h. Then the reaction mixture was acidified with 5 mL of concentrated HCl and the lower layer was separated, washing the water layer with dichloromethane (DCM). The organic layer was dried and, after vaccum distillation, provided the desired chromenes **3a-3d** and **4a-4c** in reasonable yields, Table 1, Scheme 1. In conclusion, this condensation method offers an alternative route for the novel synthesis of chromenes in reasonable yields.

Acknowledgements

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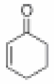
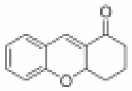
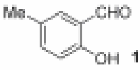
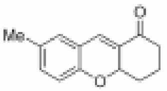
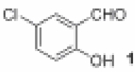
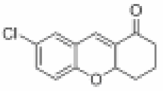
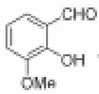
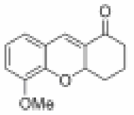
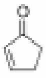
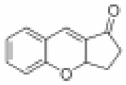
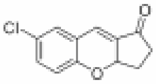
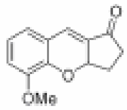
Table 1: Synthesis of Chromene derivatives [a,b,c,d]

S.No.	Salicylaldehydes	substrate	Products	Yield (%)
1	 1a	 2a	 3a (135-136)	56
2	 1b	2a	 3b (142-144)	54
3	 1c	2a	 3c (145-146)	60
4	 1d	2a	 3d (134-135)	52
5	1a	 2b	 4a (113-115)	64
6	1c	2b	 4b (150-151)	60
7	1d	2b	 4c (151-152)	58

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[a] All reactions were carried out in 5 mM scale of Salicylaldehyde in water and 20 mol% DABCO followed by 10 mM of the corresponding substrates at room temperature for 2h.

[b] Satisfactory spectral data IR, ^{1}H (200 MHz) NMR and elemental analysis were obtained.

[c] M.p (^{0}C) was written in paranthesis.

[d] Isolated yields after column chromatography (silicagel, 1% ethyl acetate in hexane).

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