

## SYNTHESIS OF NEW CONDENSATION PRODUCT OF HYDROXY COUMARIN

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### Abstract

A Series of new coumarin derivatives (1-8) was synthesized by condensation of 4-hydroxy coumarin with appropriate chloro compounds by using simple, efficient and generally applicable method. The structures of all compounds were confirmed by elemental analysis and spectral data.

**Introduction:** 4-hydroxy coumarin and their derivatives have been reported to exhibit a broad spectrum of biological activities, such as analgesic<sup>1</sup>, anti- arthritis<sup>2</sup>, anti-inflammatory<sup>3</sup>, anti-pyretic<sup>4</sup>, anti-bacterial<sup>5</sup>, anti-viral<sup>6</sup>, anti- cancer<sup>7</sup>, and anti-microbial activity<sup>8</sup>. A number of comparative pharmacological investigations of 4-hydroxy coumarin derivatives have shown good anti-coagulant activity combined with low side effect and little toxicity<sup>9</sup>. In synthesis, structure modification and very wide variety of biological activities of 4-hydroxy coumarin have been reported in many research papers<sup>10,11,12,13,14</sup>. On the basis of above all properties of 4-hydroxy coumarin and its derivatives, we decide to synthesize new derivatives of 4-hydroxy coumarin by well known method<sup>15</sup>.

Fig. 1

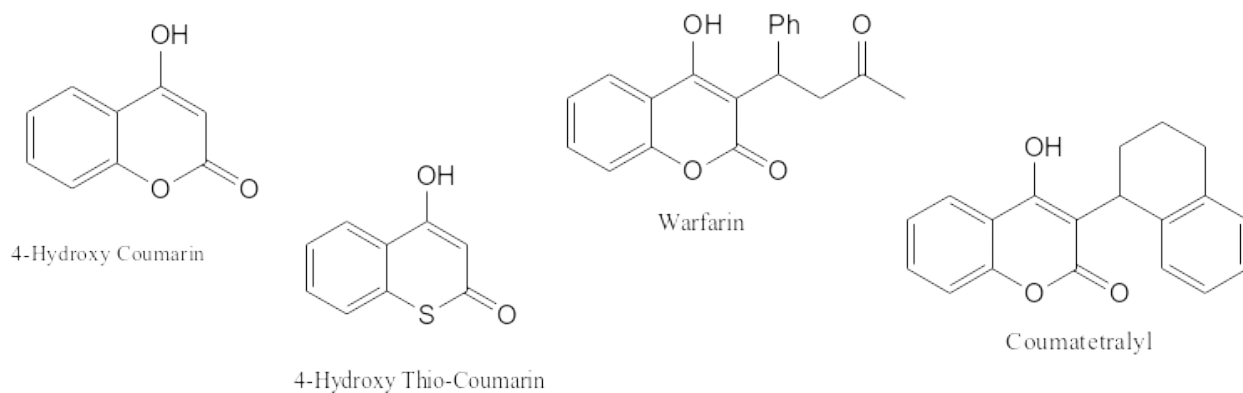
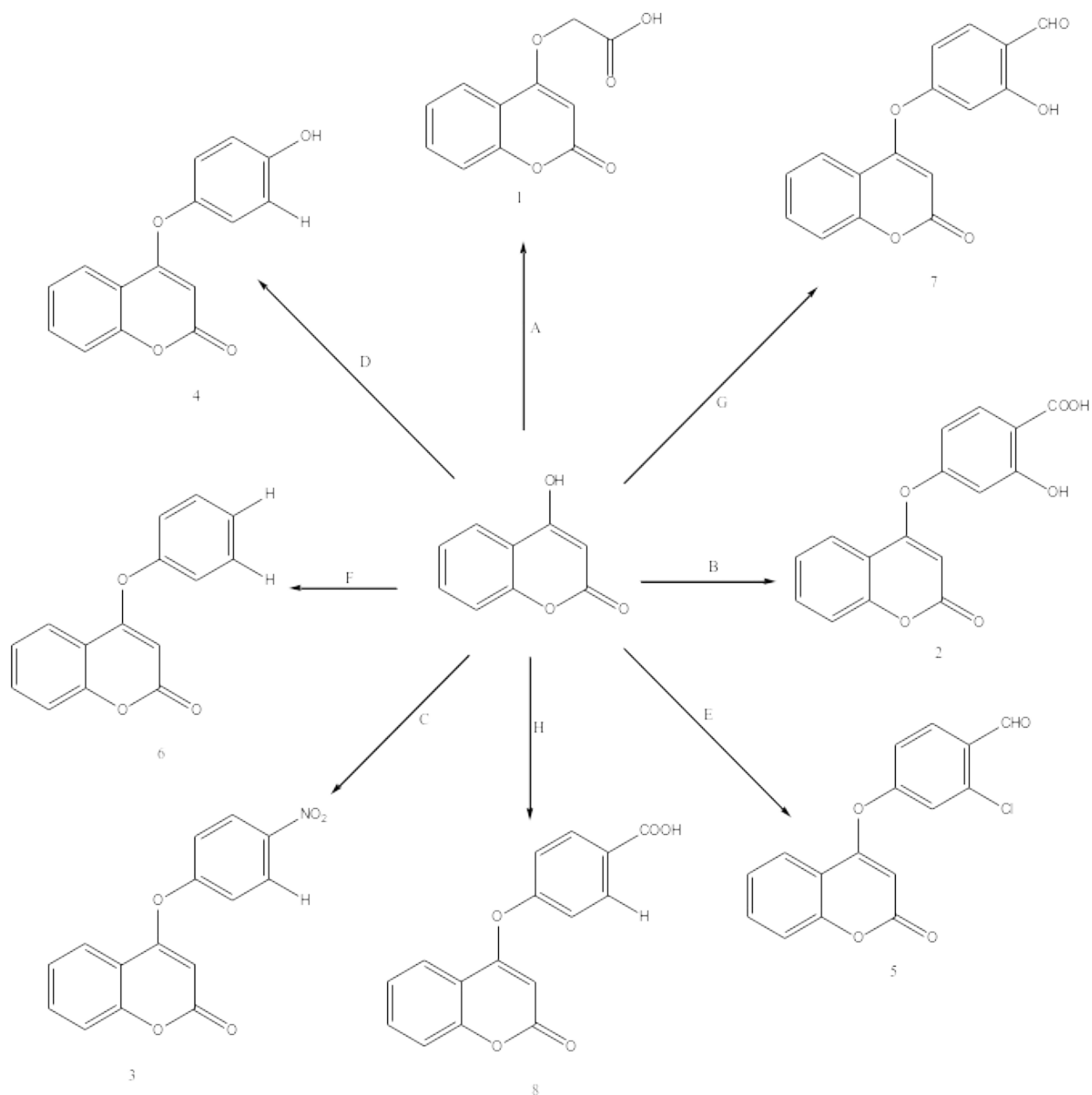




Table I : Characterization data of compounds prepared.

Compounds	Mol. For	MP(OC)	Yield(%)
1	$C_{11}H_8O_5$	240	82
2	$C_{16}H_{10}O_6$	352	75
3	$C_{15}H_9NO_5$	-	70
4	$C_{15}H_{10}O_4$	265	81
5	$C_{16}H_9ClO_4$	260	80
6	$C_{15}H_{10}O_3$	152	90
7	$C_{16}H_{10}O_5$	332	85
8	$C_{16}H_{10}O_5$	335	77

Scheme I



Where,

A = Cl-ACOH, B= Para-Cl-Salicylic Acid, C= Para Cl-Nitro Benzene, D= Para Cl-Phenol  
E= 2,4-Di-Cl-benzaldehyde, F= Cl-Benzene, G= 4-Cl-Salicylaldehyde, H= 4-Cl-Benzoic Acid



## Experimental

Melting points were determined in open capillary tubes and are uncorrected. The FT-IR spectra were recorded in KBr pellets on a Shimadzu FT-IR spectrophotometer.  $^1\text{H-NMR}$  spectra were recorded on a 200 MHz by using TMS as an internal standard. Chemical shift expressed in  $\delta$  ppm. All compounds gave satisfactory C, H, N analysis. The physical characterization data are presented in table-I.

### Synthesis of 1-8

A mixture of compounds 4-hydroxy coumarin (0.01 mol), and corresponding chloro compounds (0.02 mol), and 10 % NaOH (20 ml) in ethanol (30 ml) was refluxed on a water bath for 3 hours. The reaction mixture was cooled and poured onto crushed ice. The solid obtained was filtered, dried and recrystallized from a suitable solvent to afford the desired product.

1: IR (KBr  $\text{cm}^{-1}$ ): 1110 (C-O-C Str), 1738 (C=O Acidic gr Str), 2937 (C-H Alk Str), 3062 (C-H Ar Str).

NMR ( $\text{CDCl}_3$   $\delta$  ppm): 6.9-7.30 (m, 4H, Ar-H), 5.07 (s, 1H, ethylene), 4.89 (s, 2H, methylene), 11.05 (s, 1H, O-H)

MS m/z: 220 (bp), 221 (M+1).

Elemental Analysis: C, 60.01; H, 3.68; O, 36.08.

2: IR (KBr  $\text{cm}^{-1}$ ): 1100 (C-O-C Str), 1737 (C=O Acidic gr Str), 3055 (C-H Ar Str), 3360 (O-H broad Str).

NMR ( $\text{CDCl}_3$   $\delta$  ppm): 6.5-7.80 (m, 7H, Ar-H), 5.62 (s, 1H, ethylene), 5.02 (s, 1H, O-H), 10.9 (s, 1H, COO-H)

MS m/z: 298.0 (bp), 299 (M+1), 300 (M+2).

Elemental Analysis: C, 64.50; H, 3.4; O, 32.02.

3: IR (KBr  $\text{cm}^{-1}$ ): 1310 (C-O-C Str), 1610 (Ar-NO<sub>2</sub> str), 1735 (lactone C=O Str), 3066 (C-H Ar Str).

NMR ( $\text{CDCl}_3$   $\delta$  ppm): 7.02-7.99 (m, 8H, Ar-H), 5.62 (s, 1H, ethylene),



MS m/z: 283 (bp), 284(M+1), 285.

Elemental Analysis: C,63.6; H,3.30; O,28.30.

4:IR (KBr  $\text{cm}^{-1}$ ): 860,844,829 (sub. Benzene Str), 1710 (C=O Str), 3066 (C-H ArStr) , 3366 (O-H broad Str).

NMR ( $\text{CDCl}_3\delta$  ppm): 6.5-7.30(m,8H,Ar-H), 5.61(s,1H,ethylene), 5.01(s,1H,O-H),

MS m/z: 254 (bp), 255(M+1), 256.

Elemental Analysis: C, 71.01; H, 4.01; O,25.0.

5:IR (KBr  $\text{cm}^{-1}$ ):559 (Ar-C-ClStr), 1734 ( lactone C=O Str), 1705 (C=O Str) , 1050 (C-O-C Str).

NMR ( $\text{CDCl}_3\delta$  ppm): 6.8-7.60(m,7H,Ar-H), 5.60 (s,1H,ethylene), 10.20(s,1H,CHO).

MS m/z: 300(bp), 301 ,302.

Elemental Analysis: C,64.0; H,3.0; O,21.3.

6:IR (KBr  $\text{cm}^{-1}$ ): 1210 (C-O-C Str), 1730 ( lactone C=O Str), 3056 (C-H ArStr).

NMR ( $\text{CDCl}_3\delta$  ppm):6.5-7.29 (m,9H,Ar-H), 5.5 (s,1H,ethylene).

MS m/z:238 (bp),239, 240.

Elemental Analysis: C, 76.02; H,4.20; O,20.10.

7:IR (KBr  $\text{cm}^{-1}$ ): 3059 (C-H ArStr), 1090 (C-O-C Str), 3350 (O-H Str broad), 1710 (C=O Str).

NMR ( $\text{CDCl}_3\delta$  ppm): 6.4-7.30(m,7H,Ar-H), 6.3 (s,1H,ethylene), 5.01(s,1H,O-H) , 10.30(s,1H,CHO).

MS m/z: 282 (bp), 283,284.

Elemental Analysis: C,68.0; H,3.6; O,28.02.

8:IR (KBr  $\text{cm}^{-1}$ ): 1152 (C-O-C Str), 1708 ( lactone C=O Str), 1738 (C=O Acidic gr Str).

NMR ( $\text{CDCl}_3\delta$  ppm): 6.9-7.9 (m,8H,Ar-H), 5.65 (s,1H,ethylene), 10.98 (s,1H,COO-H) .

MS m/z: 282(bp),283,284.



Elemental Analysis: C,67.8; H,3.60; O,28.09.

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