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Synthesis of 2-(α-Naphthyl)-Halogenochromones and Study of their Toxicity to Fish

S. V. KUBERKAR* AND Y. B. VIBHUTE

Department of Chemistry, Yeshwant Mahavidyalaya Nanded–431 602, India

2-(α-Naphthyl)-halogenochromones were prepared by condensing 1-naphthaldehyde and 2-methoxy-1-naphthaldehyde with 3- and/or 5-halogeno substituted hydroxy acetophenone. The intermediate chalcones were obtained. These chalcones were treated with selenium dioxide in isoamyl alcohol and as a result of oxidative cyclization, 2-(α-naphthyl)-halogenochromones were isolated. The structures have been confirmed on the basis of elemental analysis and IR spectral data. All these altogether new chromones were tested for their toxicity to fish.

INTRODUCTION

Flavones[1–4] and several naturally occurring flavonoids[5] have been reported to possess antimicrobial activity. 2-(α-Naphthyl) chromone substituted at 6,8 positions by bromine have been found to possess antimicrobial activity[4]. Since wide range of biological activities are reported in flavones, the synthesis of some flavones have been made and were tested for their biological activity.

EXPERIMENTAL

Ten halogeno chalcones[6] were prepared using Claisen-Schmidt method by condensing 3-chloro-2-hydroxy, 5-chloro-2-hydroxy, 3,5 dichloro-2-hydroxy, 5-bromo-2-hydroxy and 3,5-diiodo-2-hydroxy acetophenones independently with 1-naphthaldehyde and 2-methoxy-1-naphthaldehyde respectively in the presence of concentrated alcoholic potassium hydroxide solution. 2-(α-Naphthyl)-halogenochromones were then prepared by the oxidative cyclization of these halogeno chalcones with selenium dioxide. This method was selected on account of the mild reaction conditions and also because it gives the chromone, free from any side product, Halogeno chalcone on heating with selenium dioxide in isoamyl alcohol at 135–140°C for sixteen hrs gave a product which gave yellow coloration with Wison test[7] and no colour with ferric chloride solution. The purity of all the compounds was checked by TLC. The structures have been confirmed on the basis of elemental analysis and IR Spectra. All these chromones showed absorption bands in the range of 1640–1655 cm^{-1} due to carbonyl group. This band is in agreement with those observed by Shaw and Simpson[8] in case of flavones.

The basic static acute toxicity test method[9] was employed for testing

TABLE 1

ANALYTICAL DATA AND TOXICITY TO FISH ACTIVITY

Compound No.	Compounds	M.pt.°C	Crystallised from	Molecular formula	Analysis %		Loss of equilibrium (min)	Death (min)
					Found	Required		
1.	6,8-Dichloro-2-(α-Naphthyl) -Chromone	180	Ethanol	$C_{19}H_{10}O_2Cl_2$	C, 66.65 H, 2.71	C, 66.86 H, 2.93	38	69
2.	6,8-Dichloro-2-(2-methoxy-α-naphthyl)-chromone	203	Ethanol	$C_{20}H_{12}O_3Cl_2$	C, 64.52 H, 3.32	C, 64.69 H, 3.23	29	52
3.	6,8-Diiodo-2-(α-naphthyl)-chromone	201	Acetic acid	$C_{19}H_{10}O_2I_2$	C, 43.35 H, 1.74	C, 43.51 H, 1.90	21	42
4.	6,8-Diiodo-2-(2-methoxy-α-naphthyl)-chromone	213	Benzene	$C_{20}H_{12}O_3I_2$	C, 43.11 H, 2.13	C, 43.32 H, 2.16	17	39
5.	6-Bromo-2-(α-naphthyl) chromone	75	Ethanol	$C_{19}H_{11}O_2Br$	C, 64.74 H, 2.98	C, 64.95 H, 3.13	92	115
6.	6-Bromo-2-(2-methoxy-α-naphthyl)-chromone	176	Ethanol	$C_{20}H_{13}O_3Br$	C, 62.75 H, 3.26	C, 62.99 H, 3.41	95	120
7.	8-Chloro-2-(α-naphthyl)-chromone	170	Ethanol	$C_{19}H_{11}O_2Cl$	C, 74.35 H, 3.38	C, 74.50 H, 3.59	102	148
8.	8-Chloro-2-(2-methoxy-α-naphthyl)-chromone	182	Ethanol	$C_{20}H_{13}O_3Cl$	C, 71.28 H, 3.62	C, 71.42 H, 3.86	108	159
9.	6-Chloro-2-(α-naphthyl)-chromone	85	Ethanol	$C_{19}H_{11}O_2Cl$	C, 74.36 H, 3.48	C, 74.50 H, 3.59	82	112
10.	6-Chloro-2-(2-methoxy-α-naphthyl)-chromone	92	Ethanol	$C_{20}H_{13}O_3Cl$	C, 71.32 H, 3.72	C, 71.42 H, 3.86	62	102

*All melting points are uncorrected.

the chromones for their toxicity to fish. The method was followed as closely as possible without any alterations.

2-(Naphthyl)-Halogenochromone

Chalcone (1.0 g), selenium dioxide (1.5 g) and isomyl alcohol (15 ml) were refluxed on an oil bath at 135–140°C for 16 hours. On cooling the reaction mixture, product was separated which on crystallization gave crystalline needles. The melting point and analytical data are given in Table 1.

RESULTS AND DISCUSSION

Toxicity to Fish

The fresh water (Barbus ticto) was selected as a test animal for determining the toxic effect of the chromones. The fishes were collected from Godavari river near Nanded. They were about 1–1.5" in length and weighed about 2–3 g on an average. Before carrying out experiment, fishes were maintained in large glass aquarium filled with aerated water for 24 hrs at laboratory conditions.

Dioxane solution of the substance under investigation was added (25 mg of each compound dissolved in 1 ml of dioxane) to one litre of water in a glass through and after mixing well, five fishes were introduced into it. Their movements and the time at which the fish lost their balance and overturned and the time of death were recorded. The average of five individual values (which did not differ very greatly) was obtained.

The control experiment conducted by using dioxane showed that a concentration of 1% is harmless to the fish over a period of 24 hrs. Loss of equilibrium and the time of death B. ticto are represented in Table 1. More or less all the compounds tested were found to be toxic to fish. Further work regarding correlation of toxicity to fish with that of antibacterial activity of these compounds is under study.

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