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Note

Synthesis of 2-(2-phenylethyl)chromones

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2-(2-Phenylethyl)chromones have been obtained by selective reduction of double bond of the styryl group in 2-styrylchromones involving catalytic hydrogen transfer reaction using ammonium formate in the presence of Pd-C.

Keywords: Phenylethylchromones, styrylchromones, catalytic hydrogen atom, Pd-C

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2-(2-Phenylethyl)chromones, their dimers and trimers constitute a small group of naturally occurring compounds and have been isolated from 'Agalwood, Jinko'[1,2]. These compounds contribute to a pleasant lasting odour when Agalwood is burnt[3] and also show interesting medicinal properties.

In order to synthesise more effective dimeric compounds, we required 2-(2-phenylethyl) chromones. In an attempt to synthesise these compounds by reduction of double bond in 2-styrylchromones using catalytic hydrogenation, led the required compounds in 15-20% yield only after purification of the mixture using column chromatography. Similarly, reduction with sodium borohydride resulted in mixture of compounds.

We now report a simple method for the selective reduction of double bond of styryl group in 2-styrylchromones involving hydrogen transfer reaction using metallic catalyst.

2-Styrylchromones required for the present work were obtained from 2-hydroxybenzoylcinnamoyl methanes[4]. 2-Styrylchromone was refluxed with ammonium formate in the presence of activated Pd-C (10%) in dry methanol to give a colourless compound **1** (mp 90°C) in 60% yield after purification of the residue. Compound **1** in its ^{1}H NMR showed a multiplet at δ 2.50-3.10 for four protons (CH_2-CH_2) in the side chain, a singlet at δ 6.10 for H-3 proton, a multiplet at δ 7.20-8.00 for eight aromatic protons and a doublet at δ 8.10; (J = 9.0 Hz) for H-5 proton. Based on these data, the compound **1** was assigned the structure 2-(2-phenylethyl) chromone.

Following similar procedures differently substituted 2-(2-phenylethyl)chromones **1-5** were obtained from corresponding 2-styrylchromones and their structures were confirmed by ^{1}H NMR and C, H analysis (**Table I**).

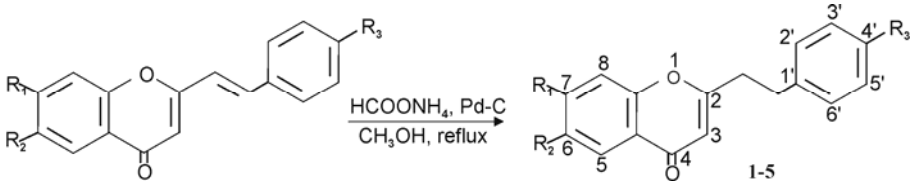

Table I — Characterization data of compounds **1-5**

Compd	R_1	R_2	R_3	Mol.formula	Yield (%)	m.p. (lit m.p.) °C	Found % (Calcd) C	Found % (Calcd) H	^{1}H NMR (δ, ppm)
1	H	H	H	$C_{17}H_{14}O_2$	60	90-91	81.82 (81.63	5.48 5.60)	2.50-3.10 (m, 4H, CH_2CH_2), 6.10 (s, 1H, H-3), 7.20-8.00 (m, 8H, H-6, H-7, H-8 & C_6H_5), 8.10 (d, J = 9.0 Hz, 1H, H-5)
2	H	CH_3	H	$C_{18}H_{16}O_2$	62	65-67	81.76 (81.81	6.00 6.06)	2.37 (s, 3H, CH_3), 2.57-3.15 (m, 4H, CH_2CH_2), 6.15 (s, 1H, H-3) 7.00-7.50 (m, 7H, H-7, H-8 & C_6H_5), 7.92 (s, 1H, H-5)

— *Contd*

Table I — Characterization data of compounds **1-5** — *Contd*

Compd	R_1	R_2	R_3	Mol.formula	Yield (%)	m.p. (lit m.p.) ^oC	Found % (Calcd) C	Found % (Calcd) H	^1H NMR (δ, ppm)
3	H	OCH_3	H	$C_{18}H_{16}O_3$	50	84-85	77.10 (77.14	5.60 5.71)	2.70-3.00 (m, 4H, CH_2CH_2), 3.60 (s, 3H, OCH_3), 6.15 (s, 1H, H-3), 7.30 (m, 7H, H-7, H-8, & C_6H_5), 7.70 (s, 1H, H-5)
4	OCH_3	H	H	$C_{18}H_{16}O_3$	55	96-97	76.95 (77.14	5.62 5.71)	2.64-3.20 (m, 4H, CH_2CH_2), 3.80 (s, 3H, OCH_3), 6.20 (s, 1H, H-3), 6.70(s, 1H, H-8), 6.90 (d, *J* = 9Hz, 1H, H-6), 7.30 (s, 5H, C_6H_5), 8.10 (d, *J* = 9Hz, 1H, H-5)
5	H	CH_3	OCH_3	$C_{19}H_{18}O_3$	60	150-52 (151)[5]	77.52 (77.55	6.04; 6.12)	2.30 (s, 3H, CH_3), 2.70-3.00 (m, 4H, CH_2CH_2), 3.70 (s, 3H, OCH_3), 5.85 (s, 1H, H-3), 6.60 (d, *J* = 9Hz, 2H, H-3' & H-5'), 6.70-6.90 (m, 4H, H-7, H-8, H-2' & H-6'), 7.60 (s, 1H, H-5)

Experimental Section

2-(2-Phenylethyl)chromones. To a solution of 2-styrylchromones (0.4g) in methanol (20 mL) was added ammonium formate (0.8g) and activated Pd-C (10%, 0.1g). The mixture was refluxed on a water-bath for 1 hr. Completion of the reaction was monitored by TLC. The reaction mixture was filtered to remove Pd-C and filterate was concentrated under reduced pressure. Ice cold water was added to the residue and extracted with ether. The ether was removed and the residue after drying in vacuum over phosphorous pentoxide was crystallised from ether-pet. ether to give 2-(2-phenylethyl) chromone as colourless solid.

References

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