Notes

Chalcones. XIX : Potential Germicides Derived from 2-Acetonaphthones

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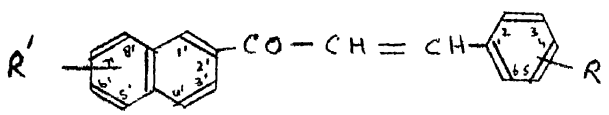
ARYL styryl ketones have exhibited potent and effective germicidal[2-4,13], fungicidal[5-7,13-16,20], bactericidal[14,22-24,26-28], sedative[18], analgesic[18], antihistaminic[29], carcinogenic[8], antifertility[12,23], cardiovascular, etc. activity. The utility of chalcones as antibacterial and antifungal agent was first reported by Schraufstatter[1,5] and Eaton and Davies[8]. These compounds have since been utilized as disease germs killer. Earlier studies on chalcones were mostly employed on phenyl groups[19,21,24] but very few naphthyl substituents were so used by Misra[2,9,26] *et al.* Detailed studies on new 2-naphthyl. substituted series were, therefore, undertaken and fifteen new 2-naphthyl substituted chalcones synthesised. Simultaneously screening of the germicidal activity was made by Agar-cup method to see their usefulness as potential germicides.

Naphthyl chalcones were prepared by the condensation of 2-acetyl naphthalene and 1-methoxy-2-acetyl naphthalene with various aryl aldehyde according to the method of Misra[7,10] *et al.* The interaction of 2-acetyl naphthalene with various benzaldehydes (Table 1) gave well defined crystalline compounds in very good yields, under normal conditions except in case of 1-naphthaldehyde. The poor yield in case of 2-naphthyl 5 : 6-benzo styryl ketone may be attributed due to steric factors or other side reactions. The chalcones 1-methoxy-2-acetyl naphthalene series were designed at 30°–50° with various benzaldehydes (Table 1) in fair yields. In the condensation of 1-methoxy-2-acetonaphthone with vanillin poor yield was obtained which may be due to resinification of vanillin with concentrated alkali solution at elevated temperatures.

The identity of the compounds was established by halochromism with conc. H_2SO_4, elemental analyses and to ascertain the position of α,β-unsaturated $>C=O$ group in the chalcones a few infra-red absorption spectra were recorded[16,21]. The compounds analysed satisfactorily for C, H, and N and results were within $\pm 0.05\%$ of the theoretical value.

TABLE 1—PHYSICAL PROPERTIES AND ANALYTICAL DATA ON 2-NAPHTHYLCHALCONES



R′	R	Yield %	Colour and crystal form	M.P. °C	ν_{max} CO	Dia. of zone of inhib. in mm.	Halochromism with Conc. H_2SO_4
H	H	69	light yellow plates	104	—	6	Red
H	34-(O–CH_2–O)	71	yellow plates	143	1680	5	Blood red
H	2-OH-3-OMe+**	57	canary yellow needles	153	1663	–	Dark red
H	5-NO_2-4-OH-3-OMe	73	yellow plates	170	1670	6	Cherry red
H	2-Br	81	yellow globules	155	1685	10	Blood red
H	26-Cl_2	86	yellow needles	180	1672	9	Dark red
H	5-Br-3 : 4-$(OCH_3)_2$**	72	dull yellow plates	151	—	9	Rosy pink
H	5 : 6-benzo	51	light shining plates	162	—	–	Violet
H	3-Br	67	yellow globules	137d	—	7	Brown
1′-OMe	2-Me	78	yellow rods	101	1665	–	Blood red
1′-OMe	3-Me	68	yellow plates	110	—	6	Red
1′-OMe	4-Me	76	shining plates	108	—	–	Dark red
1′-OMe	2,3-(OMe_2)**	65	yellow plates	146	1675	–	Cherry red
1′-OMe	3 : 4-$(OMe)_2$	77	yellow needles	133	—	–	Brown
1′-OMe	4-OH-3-OMe	45	yellow plates	116	1682	7	Blood red
Benzoic Acid						12	

* All melting points are uncorrected.
** Crystallised from ethylacetate.

The germicidal activity was screened against *S. aureus* gram positive and *E. coli* gram negative bacteria by using agar-cup method. The strength

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of the test solution was 100 to 150 μg/ml and the inocula for the purpose were 24 hr old and were prepared from stationary culture and the results were standardised against benzoic acid. No encouraging results were obtained.

Experimental

The aryl styryl ketones recorded in Table 1 have been prepared by adopting the method of Misra[9] *et al*. One typical example is cited below.

Saturated solution of sodium in methanol (5–10 ml) was added dropwise with constant stirring to an aldehyde free alcoholic solution of 2-acetyl naphthalene and 2-bromo benzaldehyde (0.005 mol). After 24 hr the dark coloured reaction mixture was kept in refrigerator and separated yellow coloured plates were purified by recrystallisation from alcohol. Finally the purity was checked by TLC as reported earlier[11].

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Potentiometric Study of Copper(II) Complexes of L-Hydroxyproline

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AMINO acids which form stable complexes have analytical importance in separation of transition metals, and rare earths[1]. A study of these complexes is also important in biological chemistry, in that the accumulation of sufficient data on amino acid complexes with metal ions may contribute to a better understanding of the types of linkages involved in metal-protein interactions. There are scanty references[2–5] on the chelate forming tendency of hydroxyproline. In the present communication, the formation of copper(II) chelate with hydroxyproline was studied using Bjerrum's and Calvin's method as modified by Irving and Rossotti[6]. The stepwise stability constants of the metal chelate were determined by Rossotti and Rossotti procedure.

Experimental

Materials. Copper nitrate (B.D.H., AnalaR) was used without further purification. 0.01*M* stock solution of the copper nitrate was standardised by titration with EDTA (AnalaR, disodium salt) using PAN indicator[7]. L-hydroxyproline (E. Merck), sodium hydroxide (E. Merck) and nitric acid (AnalaR) were used. *p*H measurements were done by ELICO *p*-Meter model LI-12, using a glass-calomel electrode assembly at 30°. The *p*H-meter was standardised with buffer solution prepared from buffer tablets (B.D.H.) for *p*H 4.0 and *p*H 9.2 at 30°. All solutions were prepared in conductivity water.

Titration Procedure. Mixture containing (a) acid (10 ml of 0.5*M* potassium nitrate and 5 ml of 0.01*M* nitric acid), (b) ligand (mixture (a) and 10 ml of 0.025*M* hydroxyproline), (c) complex (mixture (b) and 5 ml of 0.01*M* $Cu(NO_3)_2$ solution) was taken, and total volume made upto 50 ml. The ionic strength was maintained at 0.1 by KNO_3 solution. Mixture a, b and c were separately titrated with a standard carbonate free 0.2*N* KOH solution delivered from micro burette.