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NOTE

Synthesis and Antibacterial Activity of Some Chalcones and 1,4-Oxazipine Derivatives

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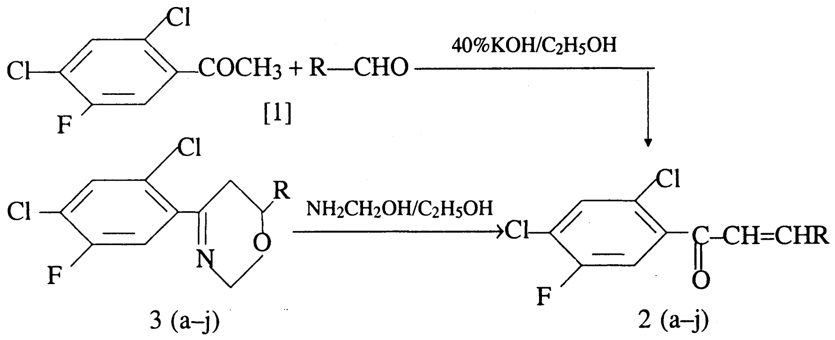
Some new 1,4-oxazipine derivatives have been prepared by condensation of 2′,4′-dichloro-5′-fluorochalcones with 2-amino ethanol in ethanol. The structures of the products have been characterised by elemental analysis and IR spectral studies. The products have been screened for antibacterial activity against *Staphylococcus aureus* and *Escherichia coli.*

Several workers have reported the synthesis of 1,4-oxazipine derivatives by the reaction of α,β-unsaturated carbonyl compounds with 2-amino ethanol under a variety of experimental conditions[1, 2]. In the present investigation, we report the reaction of 2,4-dichloro-5-fluoro acetophenone (1) with various substituted aldehydes to yield the corresponding 2′,4′-dichloro-5′-fluorochalcones [2(a–j)]. Chalcones [2(a–j)] react with 2-amino ethanol in ethanol to give the corresponding 1,4-oxazipine derivatives [3(a–j)]. (Scheme-I) (Table-1).

The compounds were secreened for antibacterial activity against gram positive bacteria, *i.e.*, *Staphylococcus aureus* and gram-negative bacteria, *i.e.*, *Escherichia coli* using cup-plate method[3]. The compounds were tested at the concentration of 50 μg/mL in DMF for antibacterial activity and the antibacterial activity of All the compounds was compared with the known antibiotics, *viz.*, chloramphenicol and ampicillin at the same concentration. All compounds show mild activity against both the bacteria.



Scheme-1

All melting points were taken in open capillary tubes and are uncorrected.

Purity of the compounds was checked by TLC. IR spectra in KBr discs were recorded on a Perkin-Elmer infracord-377 spectrophotometer. All compounds gave satisfactory elemental analysis.

TABLE-1
PHYSICAL DATA OF COMPOUNDS 2 AND 3

	R	m.f.	m.p. (°C)
2a	phenyl	$C_{15}H_9OCl_2F$	84
2b	2-chloro phenyl	$C_{15}H_8OCl_3F$	106
2c	4-chloro phenyl	$C_{15}H_8OCl_3F$	114
2d	4-methyl phenyl	$C_{16}H_{11}OCl_2F$	108
2e	4-N,N-dimethyl phenyl	$C_{17}H_{14}NOCl_2F$	112
2f	4-methoxyl phenyl	$C_{16}H_{11}O_2Cl_2F$	93
2g	3,4,5-trimethoxy phenyl	$C_{18}H_{15}O_4Cl_2F$	103
2h	2-nitro phenyl	$C_{15}H_8NO_3Cl_2F$	102
2i	2-furyl	$C_{13}H_7O_2Cl_2F$	63
2j	2-thienyl	$C_{13}H_7OCl_2FS$	85
3a	phenyl	$C_{17}H_{14}NOCl_2F$	157
3b	2-chloro phenyl	$C_{17}H_{13}NOCl_3F$	166
3c	4-chloro phenyl	$C_{17}H_{13}NOCl_3F$	173
3d	4-methoxy phenyl	$C_{18}H_{16}NOCl_2F$	161
3e	4-N,N-dimethyl phenyl	$C_{19}H_{19}N_2OCl_2F$	159
3f	4-methoxy phenyl	$C_{18}H_{16}NO_2Cl_2F$	137
3g	3,4,5-trimethoxy phenyl	$C_{20}H_{20}NO_4Cl_2F$	146
3h	2-nitro phenyl	$C_{17}H_{13}N_2O_3Cl_2F$	125
3i	2-furyl	$C_{15}H_{12}NO_2Cl_2F$	102
3j	2-thienyl	$C_{15}H_{12}NOSCl_2F$	115

General method for the preparation of 2′,4′-dichloro-5′-fluorochalcones [2(a–j)].

A mixture of 2,4-dichloro-5-fluoro acetophenone (1) (0.01 mole) and aryl aldehydes (0.01 mole) were stirred in ethanol (30 mL) and to it aqueous solution of potassium hydroxide (40%, 15 mL) was added. The mixture was kept overnight at room temperature. The colour of the reaction mixture changed from yellow to orange. The content was then poured over crushed ice and acidified with dilute hydrochloric acid. The solid separated was filtered, washed with distilled water, dried and crystallized from glacial acetic acid to give [2(a–j)].

$\nu_{max}(cm^{-1})$: 1675–1640 $\nu(C{=}O)$; 980–960 $\nu(CH{=}CH)$; 795–720 $\nu(C{-}Cl)$; 1250—1100 cm^{-1} $\nu(C{-}F)$

General method for the preparation of 7-(2′,4′-dichloro-5′-fluorophene-1′-yl)-5-substituted phenyl-2,3,6,7-tetrahydro-1,4-oxazipine derivatives [3(a–j)].

A mixture of 2′,4′-dichloro-5′-fluorochalcone [2(a–j)](0.01 mole) and 2-amino ethanol (0.01 mole) in absolute ethanol (20 mL) was refluxed on a water bath for 4 h. The excess of solvent was removed and the residue was poured into crushed ice. The solid thus separated was filtered, washed with cold water, dried and crystallized from glacial acetic acid to give [3(a–j)].

$\nu_{max}(cm^{-1})$: 1620–1610 ν(C=N); 1075–1050 ν(C–O—C stretching of cyclic ether); 760–700 ν(C—Cl); 1250–1100 cm^{-1} ν(C—F).

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