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Synthesis of Some Novel Ketimines

Vikas Borgaonkar
Shri Siddheshwar Mahavidyalaya, Majalgaon, MS, India
Email - vikas.borgaonkar@rediffmail.com

Abstract: *- As imines occupy main place amongst pharmaceutically important compounds, bioactive ketimines were synthesized by the condensation of hydroxy aromatic ketones and aromatic amines using solvent free microwave irradiation method. This method provides advantages such as environmental friendliness, short reaction time with excellent yield.*

Keywords: *Ketimines, Microwave irradiation method.*

1. INTRODUCTION:

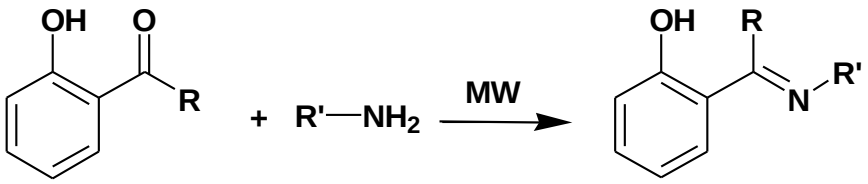
The imine group present in organic compounds is crucial for their biological behaviour (1,2). The biological activities of imines have attracted organic and medicinal researchers from many years. Recent studies on biological evaluation of imines revealed some to be antimalarial (3), anticancer (4), antimicrobial (5), anti-HIV (6), nematicidal (7), antifungal (8), antituberculosis (9), antidepressant and anti-inflammatory (10). Microwave-assisted synthesis remarkably decreases the time necessary to carry out reaction. Thus, microwave-mediated organic reactions take place more rapidly, safely, with high yields and making industrially important organic synthesis more eco-friendly (11,12). From above review, imines are expected to broaden biological activity profile. The present piece of work has been undertaken to study the synthesis of ketimines.

2. MATERIALS AND METHODS:

Melting points were determined in an open capillary tube and are uncorrected. All chemicals are commercially available and used without further purification. The purity was confirmed by TLC.

3. GENERAL PROCEDURE:

A mixture of 2- hydroxy propiophenone (0.01 mole) and aromatic amines (0.01 mole) along with 3-4 drops of glacial acetic acid was irradiated for appropriate time (Table 1) in domestic microwave oven. The progress of the reaction was monitored by TLC. After completion of reaction it was cooled and 25 ml ice-cold water was added. The reaction mixture was extracted with ethyl acetate (3 x 10 ml), organic layer was separated, dried over anhydrous sodium sulphate and evaporated to dryness. The crude product was recrystallized by ethanol to obtain pure crystals of desired compound (3a-e).



1 2 (3a-e)

Table-1: Physical and Analytical data of ketimines

Comp.	R'	Mol. Formula	Yield (%)	M.P. °C	Time (Sec)	Elemental analysis (%) Calculated (Found)			
						C	H	N	Cl
3a	C_6H_5	$C_{15}H_{15}NO$	70	121	480	79.97 (79.90)	6.71 (6.65)	6.22 (6.66)	-

3b	C_6H_4-CH_3	$C_{16}H_{17}NO$	75	85	420	80.30 (80.55)	7.16 (7.13)	5.85 (5.01)	-
3c	C_6H_4Cl	$C_{15}H_{14}ClNO$	65	80	450	69.36 (69.65)	5.43 (5.16)	5.39 (5.01)	13.65 (13.78)
3d	C_6H_4-OCH_3	$C_{16}H_{17}NO_2$	69	83	300	75.27 (75.57)	6.71 (6.50)	5.49 (5.19)	-
3e	CH_2-C_6H_5	$C_{16}H_{17}NO$	72	70	480	80.30 (80.19)	7.16 (7.20)	5.85 (5.75)	-

R= $-C_2H_5$

Spectra of sample compounds:

2-(1-Phenylimino-propyl)-phenol (3a)

IR (KBr): 3385 (OH), 1630 (C=N), 1535(C=C) cm^{-1}; 1HNMR (CDCl3):- δ 2.80 (q, 2H), 1.20 (t, 3H), 6.50-7.30 (m, 9H), 13.25 (s, 1H, -OH) ppm.

2-(1-p-Tolylimino-propyl)-phenol (3b)

IR (KBr): 3330 (OH), 1625 (C=N), 1585(C=C) cm^{-1}; 1HNMR (CDCl3):- δ 2.85 (q, 2H), 1.25 (t, 3H), 6.70-7.30 (m, 9H), 14.50 (s, 1H, -OH) ppm.

4. RESULTS AND DISCUSSION:

The ketimines were synthesized by solvent free microwave irradiation method. Synthesized compounds were reliable with their chemical structures. The characteristic IR band positions provided important sign for the formation of the ketimines. The reaction product was confirmed by disappearance of NH_2 absorption band at 3210-3336 cm^{-1} and appearance of band at 1590-1641 cm^{-1} for >C=N stretch vibrations which confirms condensation of carbonyl with amino group. Absorption at near 1500-1592 cm^{-1} is due to >C=C< aromatic stretch, absorption at 3300- 3480 cm^{-1} is due to (2-OH) hydroxyl group. In addition confirmation for the formation of ketimines was obtained from the ^1H NMR spectra, Signal at δ 1.20-1.24 for $-CH_2$-CH_3 and quartet at δ 2.70-3.15 for $-CH_2$-CH_3 is observed in all compounds. The appearance of multiplets at δ 6.50–8.15 was due to aromatic protons. Common signal appearing at δ 13.00- 16.00 is due to 2-OH group in all the compounds.

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