



SONOCHEMICAL SYNTHESIS OF SOME SCHIFF BASES

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ABSTRACT

Some new Schiff bases have been synthesized using ultrasonic waves. The structural confirmation was done by IR and MASS spectral data. It is observed that using ultrasonic waves, the reaction time was reduced and % yield of the products was increased. Further, the reaction can be carried out at room temperature by using minimum amount of catalyst than that used in conventional synthetic method.

Key words : Schiff bases, Sonochemistry, Ultrasound.

INTRODUCTION

The use of ultrasound in chemistry –Sonochemistry- has grown spectacularly in recent years¹. Sonochemistry is chemistry assisted or enhanced by the physical or chemical effects of acoustic/ ultrasound cavitations in a chemical process. Ultrasound waves are known for their wide applications in various fields like life sciences, medical, sonar, cleaning, electronics, agriculture, oceanography, material science²⁻⁹ etc. Further, it is known to have physical and chemical effects on substances due to which these ultrasonic waves have been used for the synthesis of many substances¹⁰⁻¹⁶.

In the present work, synthesis of some new Schiff bases has been done using ultrasonic waves. The results are compared with those observed by traditional conventional method. It is observed that sonochemical reactions are better than conventional methods in many ways.

EXPERIMENTAL

The following compounds have been synthesized:

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Us-1 : *N*-{(1*E*)-[4-(Dimethylamino) phenyl] methylene}-*N*-(4-fluorophenyl) amine

Us-2 : *N*-(3-Chloro-4-fluorophenyl)-*N*-{(1*E*)-[4-(dimethylamino) phenyl] methylene} amine

Us-3 : *N*-{(1*E*)-[4-(Dimethylamino) phenyl] methylene}-*N*-(4-methoxyphenyl) amine

Us-4 : *N*-{(1*E*)-[4-(Dimethylamino)phenyl]methylene}-*N*-(4-methylphenyl)amine

Us-5 : *N*-{(1*E*)-[4-(Dimethylamino)phenyl]methylene}-*N*-(4-nitrophenyl)amine

Us-6 : *N*-(4-Chlorophenyl)-*N*-{(1*E*)-[4-(Dimethylamino)phenyl]methylene} amine

Us-7 : *N*-{(1*E*)-[4-(Dimethylamino)phenyl]methylene}-*N*-(2-methylphenyl)amine

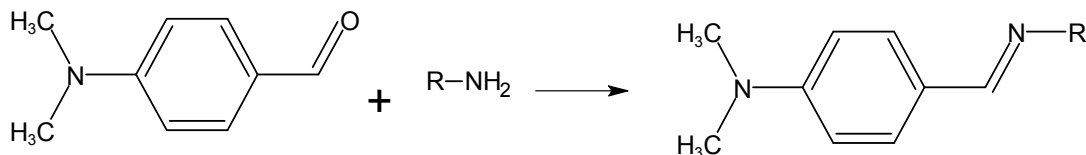
Us-8 : *N*-benzyl-*N*-{(1*E*)-[4-(Dimethylamino)phenyl]methylene} amine

Us-9 : *N*-{(1*E*)-[4-(Dimethylamino)phenyl]methylene}-*N*-(2, 4-dimethylphenyl) amine

Us-10 : *N*-(2, 5-Dichlorophenyl)-*N*-{(1*E*)-[4-(Dimethylamino)phenyl]methylene} amine

Synthesis

A mixture of 4-(dimethylamino) benzaldehyde (0.005 M), desired aromatic amine (0.005 M) and one drop of glacial acetic acid (as catalyst) was kept in the cell of single frequency (2 MHz) ultrasonic interferometer (Model No. M-81). The temperature of the mixture was maintained at 25°C. The cell was turn on and the reaction was allowed to take place for desired period. After the completion of reaction, it was poured into crushed ice and product was isolated and crystallized in ethyl alcohol. The reaction time varies with each reaction. Purity of the compounds was checked by TLC and characterization was done by Infra red spectra. The R_f values for all the Schiff bases are given in Table 1.



where R are different for different Schiff bases.

RESULTS AND DISCUSSION

Table 1 shows the physical constants of synthesized compounds.

Table 1 : Physical data of synthesized compounds

Code No.	R	Molecular formula	Mol. weight	Yield (%)	M. P. (°C)	R _f	Reaction time (hrs)
Us-1	4-F-C ₆ H ₄	C ₁₅ H ₁₅ FN ₂	242.29	86	187	0.89	2.00
Us-2	3-Cl-4-F-C ₆ H ₃	C ₁₅ H ₁₄ ClFN ₂	276.74	52	198	0.92	2.00
Us-3	4-OCH ₃ -C ₆ H ₄	C ₁₆ H ₁₈ N ₂ O	254.33	87	180	0.52	2.00
Us-4	4-CH ₃ -C ₆ H ₄	C ₁₆ H ₁₈ N ₂	238.33	86	167	0.81	0.45
Us-5	4-NO ₂ -C ₆ H ₄	C ₁₆ H ₁₈ N ₃ O ₂	269.30	55	211	0.69	2.00
Us-6	4-Cl-C ₆ H ₄	C ₁₅ H ₁₅ ClN ₂	258.75	74	217	0.74	2.00
Us-7	2-CH ₃ -C ₆ H ₄	C ₁₆ H ₁₈ N ₂	238.33	84	173	0.88	2.15
Us-8	-CH ₂ -C ₆ H ₅	C ₁₆ H ₁₈ N ₂	238.33	88	154	0.66	2.00
Us-9	2,4-CH ₃ -C ₆ H ₃	C ₁₇ H ₂₀ N ₂	252.35	91	169	0.84	1.30
Us-10	2,5-Cl-C ₆ H ₃	C ₁₅ H ₁₄ Cl ₂ N ₂	293.19	85	181	0.68	2.00

IR spectral Data: (KBr, cm⁻¹)

Us-1 : (-C=N-) 1604, (C-H sym) 2814, (Aromatic) 1546, (Amine) 3390, (C-H asym) 1436.6, (C-H sym) 1366.5

Us-2 : (-C=N-) 1603, (C-Hsym) 2811.1, (Aromatic) 1547, (Amine) 3403.2, (C-H asym) 1419.8, (C-H sym) 1368.4

Us-3 : (-C=N-) 1607.6, (C-H sym) 2809.1, (Aromatic) 1553.6, (Amine) 3362.7, (C-H asym) 1412, (C-H sym) 1361.7

Us-4 : (-C=N-) 1613, (C-H sym) 2914.2, (Aromatic) 1502, (Amine) 3397.4, (C-H asym) 1436.9, (C-H sym) 1371.9

Us-5 : (-C=N-) 1598.9, (C-H sym) 2914.2, (Aromatic) 1536.1, (Amine) 3330.8, (C-H asym) 1415.7, (C-H sym) 1310.5

Us-6 : (-C=N-) 1603, (C-H sym) 2903.6, (Aromatic) 1551, (Amine) 3410.9, (C-H asym) 1401, (C-H sym) 1367.4

Us-7 : (-C=N-) 1604, (C-H sym) 2901, (Aromatic) 1550.7, (Amine) 3719.6, (C-H asym) 1448.2, (C-H sym) 1308.6

Us-8 : (-C=N-) 1589, (C-H sym) 2810, (Aromatic) 1527, (Amine) 3399, (C-H asym) 1431.1, (C-H sym) 1370

Us-9 : (-C=N-) 1593.1, (C-H sym) 2808.2, (Aromatic) 1553.1, (Amine) 3419.5, (C-H asym) 1432, (C-H sym) 1361.7

Us-10 : (-C=N-) 1609, (C-H sym) 2906, (Aromatic) 1539, (Amine) 3398.1, (C-H asym) 1423.6, (C-H sym) 1365.3

The % yield and reaction time are compared with those of conventional method. It is observed that by using ultrasonic waves, reaction time is reduced to about two hours. In some cases, reaction was complete in about 30 minutes. By conventional method, time required to complete such reaction is usually about 12-15 hours at about 70-80° C whereas by using ultrasonic waves, reactions were carried out at room temperature.

Further, the amount of catalyst was reduced to one fifth than conventional method. In addition to above mentioned advantages, it is observed that yield is increased about one and half times more.

Thus, by using ultrasonic waves, one can synthesize the compounds in good yield at room temperature. Further, minimum reaction time and minimum amount of catalyst is required for the synthesis.

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