

GREEN ITINERARY FOR EFFICIENT SYNTHESIS OF NEWFANGLED SCHIFF BASES DERIVATIVES DERIVED FROM 1-[(2-HYDROXY PHENYL)(PHENYL)METHYL]THIOUREA

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Abstract

Microwave heating has emerged as a powerful technique to promote a variety of chemical reactions. Microwave reactions under solvent-free conditions are attractive in offering reduced pollution, low cost and offer high yield together with simplicity in processing and handling. In the present study Schiff's bases are synthesized by the conventional as well as by microwave irradiation. Excellent yield within short reaction time is obtained using microwave irradiation along with other advantages like mild reaction condition, non-hazardous and safer environmental conditions. The effects of temperature, reactant molar ratio, and microwave power variation on yield are observed.

Keywords: Microwave Irradiation, Schiff Bases, Conventional Method

Introduction

Microwave heating is the non-conventional energy source which replaces the classical one because it proves to be a clean, cheap, and convenient method. Microwave heating^{1,2} has been shown to lead to dramatically reduce reaction times³, increased product yields and enhanced product purities by reducing unwanted side reactions compared to the conventional method of heating^{4,5}. This technique has rapidly gained acceptance as a valuable tool for accelerating drug discovery and development processes.

Microwave irradiation has been also applied to carry out synthesis in open vessel, using organic solvents such as ethanol, N,N-Dimethylformamide etc as energy transfer media which absorb microwave energy efficiently through dipole rotation. Schiff bases are important intermediates for the synthesis of various bioactive products.

On the other hand, they are fundamental materials for synthesis of various Schiffbase ligands which are used as chiral auxiliaries in asymmetric synthesis.

EXPERIMENTAL PROCEDURE

Materials and Method

All chemicals used were of analytical grade and purchased from Merck, India. Fourier transform infrared spectra were registered on a SHIMADZU (8300, Kyoto, Japan) infrared spectrophotometer, using KBr discs. Proton nuclear magnetic resonance (1 H-NMR) spectra were obtained at room temperature with Bruker equipment using TMS as an internal standard in dimethyl sulfoxide (DMSO). Melting points were recorded using Gallenkamp melting point apparatus and were uncorrected.

The purity of the compounds was checked on silica-gel-coated aluminium plates. Microwave irradiations were carried out in microwave synthesizer⁹.

Method

The synthesis of new Schiff bases was achieved by cycloaddition of aromatic aldehydes (10 mmol) with newly synthesized amines (10 mmol) in presence of catalytic amount of glacial acetic acid under microwave irradiation. The same compounds were also synthesized using conventional approach. A comparative study in terms of yield and reaction

period 1 has been reported using conventional method. The reaction carried out using conventional method required about 1.0–2.0 hr, while microwave irradiation method required only 2.0–3.0 min. All the compounds synthesized were characterized by element analysis, IR, NMR and Mass data.

¹H NMR (CDCl₃) δ: 8.63 (s, H, OH), 2.34 (s, 2H, NH₂), 1.32 (d, H, NH), 7.01 (H, CH). MS:m/z: 399.02.

1-[(2,4-dihydroxy phenyl)(phenyl) methyl]-3-[(E)-(4-hydroxy-3-methoxy phenyl) methylidene]

Brown, M.p. 170-172^oC, IR (KBr, cm⁻¹): 3448.18, 1571.88 cm⁻¹; 1602.74 cm⁻¹; 1286.43, 838.54, 758.17 ¹H NMR (400MHz, CDCl₃): δ=11.02 (1H, S), 7.99 (1H, S), 7.40 (2H, d), 7.38 (2H, d), 7.22 (2H, d), 7.02 (1H, d), 6.95 (1H, t). M/S: 418.05.

RESULT AND DISCUSSION

Structure of all the synthesized derivatives have been established on the basis of their consistent IR and ¹H-NMR spectral analysis^{6,7,8}. The synthesized derivatives showed the presence of hydroxyl, methoxy, methyl and chloro functional groups along with the presence of aromatic ring which was also evident in the ¹H-NMR spectra.

Yield of these azomethine compounds were recorded and have been summarized in Table-1.

All compounds are colored, stable in air. The solids do not melt sharply and undergo decomposition. These are insoluble in water and soluble in organic solvents such as DMF, DMSO giving respective colors to the solutions. All compounds gave satisfactory elemental analysis. Values are in the close agreement with the values calculated for expected molecular formulae assigned to these complexes

Product Characterization data

1-[(3-chloro-2-hydroxyphenyl)(phenyl)methyl]-3-[(E)-(4-hydroxy-3-methoxyphenyl) methylidene]

White Solid, M.p. 96-98^oC, IR (KBr, cm⁻¹): 3157.00, 1618.01, 1262.91, 1438.10, 1132.75, 741.71.; ¹H NMR (400MHz, CDCl₃): δ=12.96 (1H, S), 7.68 (1H, S), 7.40 (2H, d), 7.28 (1H, m), 7.04 (1H, t), 6.95 (3H, m). M/S: 420.15

1-[(2-hydroxy-3-methylphenyl)(phenyl)methyl]-3-[(E)-(4-hydroxy-3-methoxyphenyl) methylidene]

Golden brown, M.p. 70-72^oC

IR (KBr, cm⁻¹): 3448.18, 1511.28, 1635.83, 838.54, 758.17 ¹H NMR (400MHz, CDCl₃): δ=11.02 (1H, S), 7.99 (1H, S), 7.40 (2H, d), 7.38 (2H, d), 7.22 (2H, d), 7.02 (1H, d), 6.95 (1H, t). M/S: 428.05.

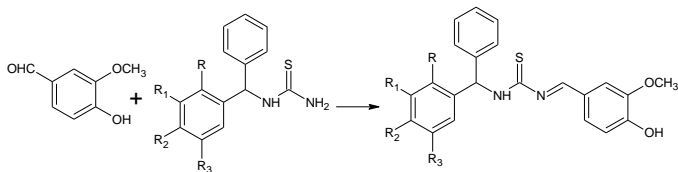
1-[(1-hydroxynaphthalen-2-yl)(phenyl)methyl]-3-[(E)-(4-hydroxy-3-methoxyphenyl) methylidene]

Color: brick red, M.P: 110^oC, IR (vmax, cm⁻¹): 3746.81, 3370.19, 3250.53, 1158., 1201.05, 1076.08, 624.96

Table 1: Analytical Data of the Synthesized Compounds

Compound	Molecular formula	Molecular weight	Elemental anal.	Yield
1.	C ₂₂ H ₁₉ Cl N ₂ O ₃ S	426.915	C(61.89%) H(4.49%) Cl(8.30%) N(6.56%) O(11.24%) S(7.51%)	78%
2	C ₂₂ H ₂₀ N ₂ O ₄ S	408.4702	C(64.69%) H(4.94%) N(6.86%) O(15.67%) S(7.85%)	77.21%
3	C ₂₆ H ₂₄ N ₂ O ₃ S	444.54536	C(70.25%) H(5.44%) N(6.30%) O(10.80%)	72.19%

			%) S(7.21%)	
4.	C₂₂H₁₉Cl N₂O₄	410.85026	C(64.31 %) H(4.66%) Cl(8.63%) N(6.82%) O(15.58 %)	77.8%



R= OH, NH₂ ; R₁ =H, Cl ; R₂= OH,CH₃,H ; R₄= Cl, H, OH

CONCLUSION

In conclusion, we have synthesized a number of various Schiff Base derivative. The reaction conditions were established and found to be reproducible.

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