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COMPARISION OF DIFFERENT WAYS OF METHOD OF PREPARATIONS OF SIMPLE SCHIFF BASE

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Abstract

In this paper we propose the synthesis of (E)-4-methyl-N-(3,4,5-trimethoxybenzylidene) benzenamine in different ways and compare of the way of synthesize it. As a result, microwave irradiation is the simple way to synthesis this Schiff base. As Schiff bases have wide applications in food industry, dye industry, analytical chemistry, catalysis, fungicidal, agrochemical and biological activities.

Keywords: *Synthesis, Schiff base, Compare*

Introduction

Schiff bases are versatile ligands which are synthesized from thecondensation of an amino compound with carbonyl compounds. Schiff basesderived from an amino and carbonylcompound are an important class of ligands thatcoordinate to metal ions via azomethine nitrogen and have been studied extensively .In azomethinederivatives, the C=N linkage is essential for biologicalactivity, several azomethines were reported to possess remarkable antibacterial, antifungal, anticancer and diuretic activities. Recent discovery has shown that Schiff bases can act as antioxidants also.

Oximes react with metal ions to give characteristic coloured complexes which can be quantitatively extracted into organic solvents.Generally carbonyl monoximesgive coloured complexes with metal ions. A large number of oximes are used as spectrophotometric regents in analytical chemistry. They are applied to trace determination of metal ions invarious materials. Apart from their analytical applications, oximes haverecently found commercial applications in hydrometallurgy of transition metals .

For the sake of convenience to compare the result, we choose the simple material (3,4,5-trimethoxybenzaldehyde and p-toluidine) to synthesize simple Schiff base.

Scheme.1 synthesis of Schiff base

Experimental

Melting points were uncorrected and were measured with micro-melting point apparatus XT-4. IR spectra (KBr) were obtained on a Thermo Nicolet Nexus 470 FT-IR spectrometer. 1H NMR spectra were determined on a Varian Mecurry 300 spectrometer using CDCl3 as solvent andtetramethylsilane (TMS) as internal reference. Microwave irradiation was carried out with commercial LG domestic microwave oven (1000W). All reagents were commercially available.

General procedure for the preparation of Schiff base ((*E*)-4-methyl-N- (3,4,5-trimethoxybenzylidene) benzenamine)

Way 1:

A mixture of p-toluidine (0.107g, 1mmol), 3,4,5-trimethoxybenzaldehyde(0.196g, 1mmol), neutral alumina(1g) and dichloromethane(2ml) in conical flask was introduced into the microwave oven and irradiated for 4min (output power at 20%). After cooling, the solid was recrystallized from ethylacetate/petroleum ether to provide (0.242g, 85%) of the title compound as a white lamellar crystal.

Way2:

A solution of 3,4,5-trimethoxybenzaldehyde(1g, 5.09mmol) in benzene (10mL) was added dropwise in a solution of p-toluidine (0.54g, 5.09mmol) in benzene (5mL). The mixture was heated in reflux temperature, until no water appear(monitor with a Barrett distilling receiver). The solvent was removed in vacuo, and the residual was recrystallized from EtOAc to obtain the title compound (1.05g, 72%) as a white lamellar crystal.

Way3:

To a stirred solution of 3,4,5-trimethoxybenzaldehyde (1g, 5.09mmol) and **7**(0.54g, 5.09mmol) in 10ml DCM, anhydrous MgSO4 was added. The reaction mixture was stirred 2 hours at room temperature. The resulting mixture was filtered through a sintered glass funnel with the aid of two 2ml portions of DCM, and then the filtrate was concentrated under reduced pressure by rotary evaporation at room temperature to afford yellow oil. The residual was dissolved in ethanol heated in an 80°C water bath while hot water was added with stirring. The resulting solution was allowed to cool to room temperature and then was cooled in an ice-water bath for 2 hr. Filtration provide the title compound (1.09 g, 75%) as white lamellar crystal.

Results and Discussion

Compared with way 2 and way 3, way 1 has a great virtue. It is very suit for industrial manufacture which consumes the least time to finish the synthesis of Schiff base. Microwave irradiation synthesis is not only use the least time, but also has the greatest yield. From the table.1, we can know clearly that microwaveirradiation is the simple way to synthesis this Schiff base. Microwave irradiation is becoming an increasingly popular method of heating which replaces the classical one because it proves to be a clean, cheap, and convenient method. Often, it affords higher yields and results in shorter reaction time. This method of heating has been extended to almost all areas of organic chemistry [7].

Table.1 The compare of three way of synthesis of Schiff base

Entry	Way Reaction condition	Time	Yield
1	microwave irradiation	4min	82%
2	reflux over	7h	70%
3	rt stir	4h	73%

Melting Point: 91–93ºC.

TLC: Rf(silica; ethyl acetate: petroleum ether, 1:4) 0.40.IR (KBrcm-1): 2954，2934，2835，1624，1558，1506，1460，1330，1127，1003.

1H-NMR (300 MHz, CDCl3): δ= 2.35 (s, 3H, -CH3). 3.90(s, 9H,-(OCH3)3), 7.11(s, 6H, PhH). 8.31 (s1H, N=CH).

References:

A. D. Garnovskii, ZhurnalNeorganichesskojkhimii, ***43(9)****, 1491-1500 (1998).*
M. Sonmez, I. Berber and E. A. Kba, Eurpean. J. Med. Chem., ***41(1)****, 101-105 (2006).*
E. A. Liberta and D. Xwest, Inorganic Chim. Acta, ***363(1)****, 157-162 (2010).*
M. A. Jasim and A. Karawi, Transition Metal Chemistry, ***34(8)****, 891-897 (2009).*
Sm. S. Alam and D. U. Lee, Chem. Crystallogr, ***10****, 1007-1027 (2011).*
J. Singh and P. Singh, Int. Scholarly Res. Netwrk, 10(54), 1-6 (2012).
N. Raman et al., J. ChilianChem, Soc., ***53****, doi.:10.4067/S0717-97072008000100025 (2008).*
M.S. Suresh and V. P. Rakash, Int. J. the Physical Sci., ***5(14)****, 2203-2211 (2010).*
M. S. Suresh and V. P. Rakash, E. J. Chem., ***8(3)****, 1408-1416 (2011).*
T. S. Wong, N. Wu, D. Roccatano and U. Schaneberg, J. Biolecutar Screening, ***10(3)****, 1- 6 (2005).*
A. Earnshaw, Introduction to Magnetochemistry, Academic Press, London (1980).
M. M. Mashaly, Z. H. Abd – Elwahab, A. A. Faheim, J. Chinese. Chem. Soc., ***51****, 901-915 (2004).*
N. Raman, Y. P. Raja and A. K. Samy, Proct. Indian Acad., ***113(3)****, 183-189 (2001).*
A. M. Ali, M. A. Hada and M. S. Mohammad, J. Kufa Chem. Sci., ***2(1)****, 64-71 (2011).*