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MICROWAVE INDUCED SYNTHESIS OF TRIPHENYL METHANE DERIVATIVES: A SOLVENT FREE PATH OF PREPARATION OF DYES

Bharti Mehta and Shipra Bhardwaj*
Department of Chemistry, M.P. P.G. College, Chittorgarh (Rajasthan)
M.L. Sukhadia University, Udaipur (Raj.)
*E-mail:sidsidsmart@yahoo.co.in

ABSTRACT

Synthesis of Triphenyl Methane derivatives was carried out under Microwave Irradiation. Generally the reaction was found to follow condensation path with removal of water molecules. Formation of product was governed through TLC and was confirmed by spectro photometric studies of compounds the smaller reaction time, carried out in a single pot without using solvent in a domestic Microwave oven provides an alternate pathway for synthesis of these derivatives.

Keywords: - Microwave irradiation, Triphenyl Methane, Condensation, Solvent free, Single pot, TLC .

INTRODUCTION

Microwave irradiation lies in the electromagnetic spectrum in between the radio-waves and infra-red frequencies. As the wave length of Microwave Irradiation is large, these are not capable of ionizing or breaking bonds and thus are considered as heat radiations. Microwave Irradiations are now widely used as non conventional source of energy for synthesis of many organic substances and have variety of applications[1-2]. An organic molecule absorbs the Microwave energy selectively enhancing the rate of reaction. The MORE Chemistry was earlier restricted to the use of high boiling point polar solvents like DMSO, DMF, *etc.* Later few boiling point solvents like Toluene[3] were used but were found to generate serious hazards. Some Microwave Irradiation was carried out on solid support like Alumina clay, Silica-gel, inorganic-oxide *etc*[4].

To overcome the problem in Microwave Synthesis later on solvent free path[5-6] was developed. This dried media Synthesis is now widely used. Varma[7] has carried out solvent free Synthesis of Thioacetones and Thioamides. *Pasha et al*[8] carried out synthesis of Amides from carboxylic acids and urea in presence of Pyridine under Microwave Irradiation. Reid et al[9] carried out Chloro-dehydroxylation of R-CH_2-OH in Microwave in presence of HCl to produce R-CH_2Cl. Several 1-Phenyl 1- thiocarbamoyal and N-substituted thiocarbamoyal -3-(2-furyl)-5-phenyl (2-furyl) - 2-pyrazolines were synthesized by Ozdemir *et al*[10]. Microwave synthesis without use of solvent has following advantages-

1. Possibility of explosion is overruled.
2. Cost of process is less due to absence of expensive solvents having high boiling point
3. The problem of removal of solvent after the completion of reaction is eliminated
4. M.W. synthesis consumes lesser time than traditional method.
5. Yield percentages are higher.
6. Lesser amount of side products and purity of compound are improved

Reactions are carried out in a single pot thus reducing the cost and making the process economically fruitful. Rapidity of the reaction, high yield, feasibility of reaction, incase heating of material, less equipment requirement etc.

Extensive work has been carried out during last few decades in all these directions and a large number of organic compound have been synthesized. Unfortunately the synthesis of coloured compounds has been left untouched. This drew the attention of authors towards the present investigation

EXPERIMENTAL

Compound 1, 2, 3 and 4 are prepared by taking appropriate reagent (alkylated Aniline or Aniline and Benzaldehyde or Chloro-benzaldehyde) which are taken in a beaker and are covered. Then the reactants are heated in a Microwave chamber in presence of concentrated HCl. The corresponding Amine is added first in half of its total quantity. Reaction mixture is irradiated and then in second step remaining half part of the amine is added. Again reaction mixture is irradiated. Obtained viscous liquid is treated with freshly prepared PbO_2 and its excess is removed by adding Na_2SO_4. Then leucobase is obtained. Excess of ammonia solution is added in this solution and dilute HCl is added till the solution becomes acidic. Product is then obtained by evaporation on water-bath. The product is recrystalized with acidified distilled water.

RESULTS AND DISCUSSION

Synthesis of Triphenyl Methane derivatives have been carried out earlier and these are used as colour substances. For synthesis of these compounds via traditional method, it was observed that the time required for synthesis is from several hours to few days. A large set of glassware for synthesis, removal of unreacted part, purification etc. makes the process tedious. For example synthesis requires 24 hours continuous heating and temperature rising gradually from 60º to 140ºC in traditional process.

Now, we have reported a less time consumable, one pot synthesis of some such colour compounds in a domestic microwave oven under solvent free conditions. The physical data of these compounds are given in table. The generalized reaction is as shown(Where, X and Y are given in Table-1).

Spectral Analysis

Compound-1

Elemental Analysis**:** % C = 75.72, % H = 6.85, % Cl = 9.73, % N = 7.68

IR Spectrum: ν_{Max} Nujol (cm^{-1}) 3463.8 (N-H str.in ammonium ion salt), 2992.4 (ArC-H str. in -CH_3), 2368.4 (NH bend in salt of amine), 1592.7 (C-C multiple bond str.), 1383.3 (C-N vib), 1113.1 (ArC-H bending in plane), 670.7 (ArC-H bending out of plane)

NMR Spectrum: δ_H, D_2O TMS, 3.10(N-CH_3, 12H, d) 7.19(Ar-H, 3H, t), 7.30(Ar-H, 4H, d nonbenz.), 7.42(Ar-H, 6H, d benz.)

Compound-2

Elemental Analysis**:** % C = 77.05, % H = 7.89, % Cl = 8.44, % N = 6.65,

IR Spectrum: ν_{Max} Nujol (cm^{-1}) 3425.1 (ArC-H str. superimposed by –NH str. in ammonium ion salt), 2990.2 (Ar C-H str., -CH_3 C-H str.), 2367.9 (-NH bending in salt of amine), 1628.8 (C-C multiple bond str.), 1272.9 (C-N vib.), 1118.5, 1023.8 (Ar C-H bending in plane), 767.7 (ArC-H, bending out of plane).

NMR Spectrum: δ_H, D_2O TMS, 1.16(-CH_3, 6H, t), 3.68(-CH_2, 4H, d) 7.28(Ar-H, 4H, d nonbenz.), 7.43,(Ar-H, 3H,t), 7.53(Ar-H, 6H,d benz.)

Compound-3

Elemental Analysis**:** % C = 69.17, % H = 6.01, % Cl = 17.79, % N = 7.01

IR Spectrum: ν_{Max} Nujol (cm^{-1}) 3426.8(ArC-H str. superimposed by –NH str. of ammonium ion salt), 2817.2(Ar C-H str.), 2365.3(salt of amine), 1597.0(C-C multiple bond str.), 1384.7(C-N vib), 1352.1(Ar-tert C-N vib.), 1060.2(ArC-H bending in plane), 767.4 (ArC-H bending out of plane), 670.7(C-Cl str.).

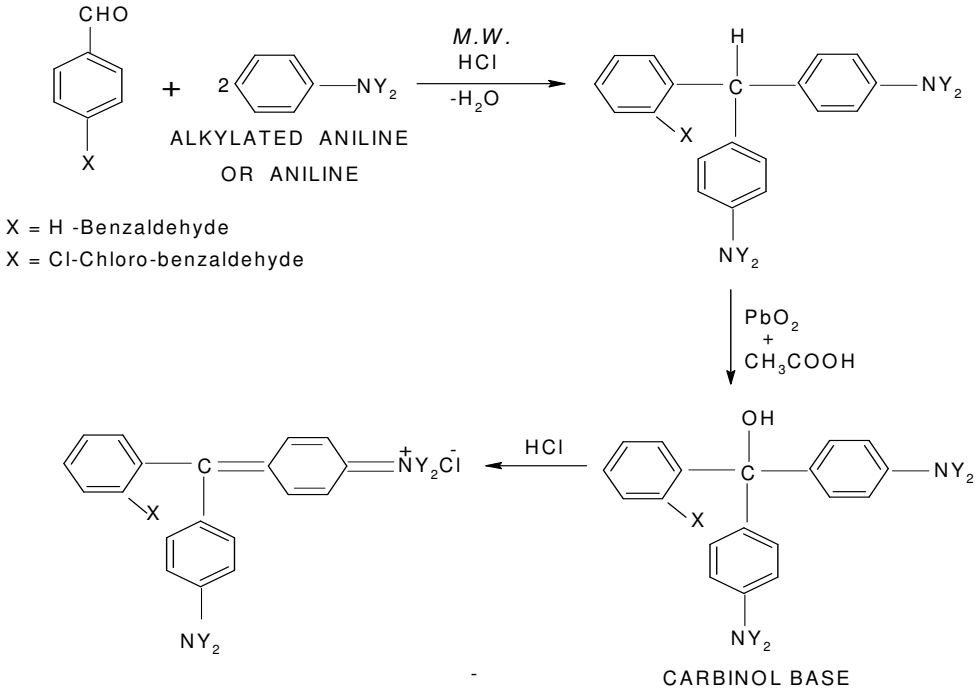
NMR Spectrum: δ_H, D_2O TMS, 3.54(N-CH_3, 12H, t), 4.77(Ar-H, 4H, d, nonbenz.), 7.25(Ar-H, 6H, d, benz.), 7.49(Ar-H, 2H, t)

Compound-4

Elemental Analysis: % C = 73.9, % H = 5.51, % Cl = 11.50, % N = 9.07

IR Spectrum: ν_{Max} Nujol (cm^{-1}), 3433.1(N-H str. pri amine), 3168.4(ArC-H str.), 2365.1(N-H str. in salt of amine), 1629.1(N-H pri. bending), 1508.0(C-C multiple bond str.), 1399.7(C-N vib), 1193.9(Ar C-H bending in plane), 747.4 (Ar C-H bending out of plane).

NMR Spectrum: δ_H, D_2O TMS, 5.74(Ar-NH_2, 4H, d), 7.17(Ar-H, 10H, d) 7.36 (Ar-H, 3H, t).



Scheme-1

Table-1: Physicochemical data of Compounds 1,2,3 and 4

Compd.	-x	-y	Molecular Formula	Mol. wt.	MP (ºC)	MW Intensity	Time (In min's sec.)	λ_{max} (nm)	Yield (gms)	% Yield
1	H	CH_3	$C_{23}H_{25}N_2Cl$	382.94	112	I step- Low II step-Low	10:00. 16:00.	615, 425	13.500	89.76
2	H	C_2H_4	$C_{27}H_{33}N_2Cl$	482.65	210	I step- Low II step-Low	20:00 5:00	625	9.500	62.99
3	Cl	CH_3	$C_{23}H_{24}N_2Cl_2$	399.0	78°	I step- Low II step-Low	4:00 8:00	580	12.500	79.61
4	H	H	$C_{19}H_{17}N_2Cl$	308.5	152	I step- Low II step-Low	8:00 10:00	580	15.600	92

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