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NOTE

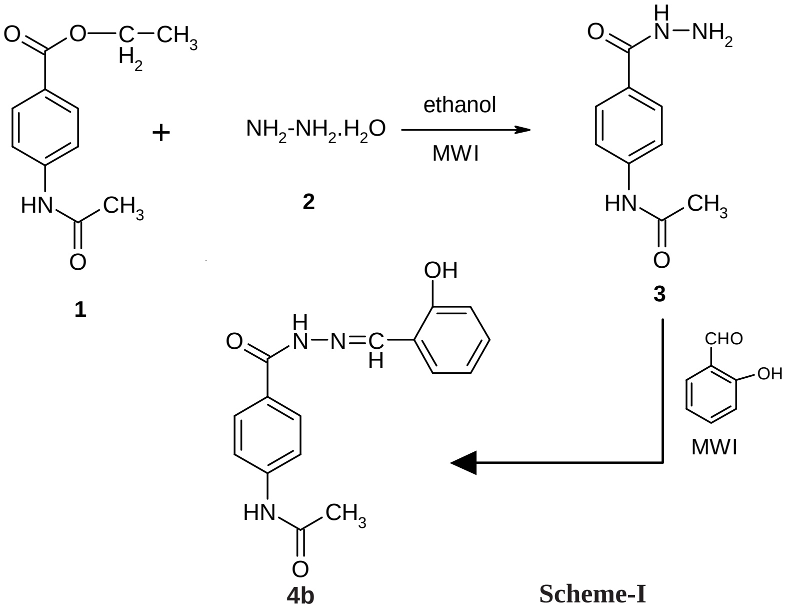
Microwave Assisted Synthesis of Some New Schiff's Bases

ELJO P. CHACKO* and GOPAL KRISHNA RAO
Department of Pharmaceutical Chemistry
Al-Ameen College of Pharmacy, Bangalore-560027, India
E-mail: eljo_chacko@yahoo.co.in

In present work, the microwave irradiation induced, synthesis of N-(4-hydrazinocarbonyl phenyl)acetamide with different aryl aldehyde yields, N-[4-(substituted benzylidine)-hydrazino carbony phenyl]acetamide has been carried out.

Key Words: Microwave synthesis, N-[4-(substituted benzylidine)hydrazino carbony phenyl]acetamide.

Schiff bases exhibit good antimicrobial activity and an array of biological applications such as fungicidal activity[1], herbicides[2], neoplasm inhibitors[3], antiviral[4], anticonvulsants[5], anticancer[6], and also plant growth regulators[7]. As part of our ongoing research towards the non-traditional approach to the experimental set-up of organic reactions, the concept of Microwave Induced Organic Reaction Enhancement (MORE) chemistry[8], under the framework of Green Chemistry[9] we have developed an environmental benign synthesis of hydrazide which on condensation with different aryl aldehyde gives different Schiff bases.



Scheme-I

Melting points of the compounds were determined in open capillary and are uncorrected. The infrared spectra were recorded on Shimadzu-8700 spectrophotometer as KBr disc. ^{1}H NMR (400 MHz) spectra were recorded in chloroform-d_6 in Amx-400 using TMS as internal standard.

Procedure for preparation of N-(4-Hydrazinocarbonyl phenyl) acetamide (3): In 250 mL beaker, N-acetyl *p*-ethylbenzoate (**1**) (0.048 mol) was taken to this hydrazine hydrate (**2**) (0.144 mol) was added and mixed. To this mixture ethanol was added and stirred well. This mixture was allowed for microwave irradiation for 3 min. Then the excess of ethanol was distilled off and left over night to get solid material **3**, washed with excess of cold water, filtered and recrystallized. Yield 77%; m.p. 260°; IR (KBr, cm^{-1}): 3340, 1606.

Procedure for preparation of N-[4-(hydroxybenzylidine)-hydrazino carbonyl phenyl]acetamide (4b) (Scheme-I: In 250 mL beaker, N-(4-hydrazinocarbonyl phenyl)acetamide (**3**) (0.051 mol), were added 2-hydroxy benzaldehyde (0.051 mol), a pinch of anhydrous zinc chloride and ethanol were mixed. This beaker was covered with a stem less glass funnel and was microwave irradiated for 2 min. The excess of ethanol was distilled off. The residue was poured in to crushed ice where, the product **4b** separated out and recrystallized from suitable solvent. Yield 87 %; m.p. 62°; IR (KBr, cm^{-1}): 3298, 1656, 1602; ^{1}H NMR ($CDCl_3$): 4.4 (s, 1H), 8.6 (s, 1H); λ_{max}: 238. Similarly, the other Schiff bases were synthesized (**4a-j**) and their physical data are reported in Table-1.

TABLE-1
PHYSICAL DATA OF N-[4-(SUBSTITUTED BENZYLIDINE)HYDRAZINO CARBONYL PHENYL]ACETAMIDE (**4a-j**)

Compound	R	m.f. (m.w.)	m.p. (ºC)	Yield (%)
4a	2-Cl	$C_{16}H_{14}O_2N_3Cl$ (315)	240	87
4b	2-OH	$C_{16}H_{15}O_3N_3$ (297)	62	87
4c	4-OCH_3	$C_{17}H_{17}O_2N_3$ (311)	243	89
4d	4-OH	$C_{16}H_{15}N_3O_3$ (297)	250	87
4e	H	$C_{16}H_{15}O_2N_3$ (281)	277	91
4f	H	$C_{18}H_{17}O_2N_3$ (307)	245	83
4g	4-Cl	$C_{16}H_{14}O_2N_3Cl$ (315)	240	87
4h	2-NO_2	$C_{16}H_{14}O_4N_4$ (326)	198	85
4i	4-$N(CH_3)$	$C_{18}H_{20}N_4O_2$ (324)	183	85
4j	3,4-(OCH_3)	$C_{18}H_{19}N_3O_3$ (325)	180	86

In present work, the compound **3** reacts with different aryl aldehyde under microwave irradiation for 2 min to give different Schiff bases **4a-j**.

The Schiff bases **4a-j** were obtained in good yields (Table-1). Structure of the resulting Schiff bases **4a-j** are conformed by spectral data. The IR spectra exhibit stretching frequency of (C=N) at 1602 cm^{-1} for compounds **4b**, **4c**, **4d**, *etc*. The ^{1}H NMR of **4b** shows δ 8.6 due to (N=CH).

The compounds **4a-j** were confirmed by the following IR (KBr, cm^{-1}), UV spectra. **(4a)** IR (KBr, cm^{-1}) 3247 ν(NH), 1641 ν(C=O), 1600 ν(C=N), 759 ν(C-Cl); λ_{max}: 306. **(4b)** IR (KBr, cm^{-1}) 3298 ν(NH), 1656 ν(C=O), 1602 ν(C=N); λ_{max}: 238. **(4c)** IR (KBr, cm^{-1}) 3284 ν(NH), 1645 ν(C=O), 1602 ν(C=N); λ_{max}: 316. **(4d)** IR (KBr, cm^{-1}) 3988 ν(NH), 1712 ν(C=O), 1602 ν(C=N); λ_{max}: 320. **(4e)** IR (KBr, cm^{-1}) 3301 ν(NH), 1651 ν(C=O), 1602 ν(C=N); λ_{max}: 298. **(4f)** IR (KBr, cm^{-1}) 3264 ν(NH), 1640 ν(C=O), 1602 ν(C=N); λ_{max}: 321. **(4g)** IR (KBr, cm^{-1}) 3220 ν(NH), 1631 ν(C=O), 1598 ν(C=N), 761 ν(C-Cl); λ_{max}: 324. **(4h)** IR (KBr, cm^{-1}) 3219 ν(NH), 1631 ν(C=O), 1596 ν(C=N); λ_{max}: 306. **(4i)** IR (KBr, cm^{-1}) 3336 ν(NH), 1602 ν(C=O); λ_{max}: 349. **(4j)** IR (KBr, cm^{-1}) 3338 ν(NH), 1638 ν(C=O), 1604 ν(C=N); λ_{max}: 328.

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