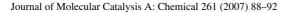


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Iodine-catalyzed synthesis of novel Hantzsch *N*-hydroxyethyl 1,4-dihydropyridines under mild conditions

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Abstract

A mixture of ethanolamine and acetic acid as ethanolammonium acetate and various aldehydes in the presence of methyl acetoacetate were converted to *N*-hydroxyethyl 1,4-dihydropyridines under mild and solvent-free conditions in good to excellent yields. Reactions occurred both in the presence and in the absence of molecular iodine. Molecular iodine catalyzed the described reaction efficiently and also improved the reaction yields. © 2006 Published by Elsevier B.V.

Keywords: Iodine (I₂); Synthesis of 1,4-dihydropyridines; Methyl acetoacetate; N-Hydroxyethyl 1,4-dihydropyridines; Solvent-free; Oxidation of 1,4-dihydropyridines; Aromatization of 1,4-dihydropyridines

In recent years, an increasing interest has been focused on the synthesis of Hantzsch 1,4-dihydropyridines, a class of model compounds of NADH coenzyme, due to the biological pertinence of these compounds to NADH redox process [1,2]. They posses neuroprotective, platet anti-aggregation and antidiabetic activities [3]. 1,4-Dihydropyridines are easily prepared from Hantzsch reaction or its modifications [4–7]. In recent years it was found that drugs such as nifedipine and niguldipine undergo redox processes due to the catalysis of cytochrome P-450 in the liver during their metabolism [8]. 1,4-Dihydropyridines are also well-known compounds as a consequence of their pharmacological profile as the most important calcium channel modulators [9-18]. Some representatives like nifedipine, niguldipine, nicardipine and amlodipine are widely used for treatment of hypertension [19]. Additionally, dihydropyridines are often produced in a synthetic sequence, and have to be oxidized to pyridines [20].

The classical method for the synthesis of 1,4-dihydropyridines is a one-pot condensation of an aldehyde with 1,3dicarbonyl compounds, and ammonia either in acetic acid or refluxing alcohol [21]. Development of an efficient and versatile method for the preparation of 1,4-dihydropyridines is an active ongoing research area and there is scope for further improvement toward synthesis of new derivatives of 1,4-dihydropyridines with milder reaction conditions and improved yields. Although iodine molecule has been used for the synthesis of *N*-unsubstituted 1,4-dihydropyridines [22] but there is no any report about its using for the preparation of *N*-substituted ones. Therefore, we decided to synthesize new *N*-substituted derivatives of 1,4-dihydropyridines by applying a practical and catalytic procedure to overcome the above limitations and provide a clean and easy work-up.

Herein, we wish to report the first example of the synthesis of different kinds of methyl 1-hydroxyethyl-1,4-dihydro-2,6-dimethylpyridine-3,5-dicarboxylates (4) and bis(methyl 1-hydroxyethyl-1,4-dihydro-2,6-dimethylpyridine-3,5-dicarboxylates) (6) under mild and solvent-free conditions (Schemes 1 and 2). Study of the chemistry of 1,4-dihydropyridines due to their biological activity is in the area of our interest. Thus, in continuation of our investigation on the synthesis of 1,4-dihydropyridines [4] and their conversion to the corresponding pyridine derivatives [23] we decided to design some new molecules containing reactive functionalities for further manipulation of 1,4-dihydropyridine moieties (Scheme 3 and

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OMe + RCHO + OH NH₂
$$\xrightarrow{\text{Cat. (X)}}$$
 MeO OMe

 $\begin{array}{c} \text{OMe} \\ \text{CH}_2 \\ \text{CH}_2 \\ \text{CH}_2 \\ \text{OHe} \end{array}$

Scheme 1. Method A: $[X = CH_3COOH \text{ (an equal amount of 3)} + I_2 \text{ (0.038 g, 0.15 mmol)}]$, method B: $[X = CH_3COOH \text{ (an equal amount of 3)}]$, method C: $[X = I_2 \text{ (0.038 g, 0.15 mmol)}]$.

$$H-C=0$$

$$H-C=0$$

$$H-C=0$$

$$H-C=0$$

$$Cat. (X)$$

$$Neat$$

$$40 ^{\circ}C$$

$$COOMe$$

$$CH_2$$

$$C$$

Scheme 2. Method A: $[X = CH_3COOH \text{ (an equal amount of 3)} + I_2 \text{ (0.038 g, 0.15 mmol)}]$ and method B: $[X = CH_3COOH \text{ (an equal amount of 3)}]$.

Fig. 1). Therefore, ethanolamine was used for the synthesis of necessary precursors **4** and **6**.

A wide range of methyl 1-hydroxyethyl-1,4-dihydro-2,6-dimethylpyridine-3,5-dicarboxylates (4) were synthesized from a combination of methyl acetoacetate (1), an aromatic aldehyde (2) ethanolamine (3) and acetic acid as ethanol ammonium acetate under mild and solvent-free conditions. The reactions were completed within 1.5–5 h (Method B, Table 1, Schemes 1 and 2). Methyl 1-hydroxyethyl-1,4-dihydro-2,6-dimethylpyridine-3,5-dicarboxylates were also synthesized by using the above mentioned materials and also applying a catalytic amount of iodine molecule (I₂) under the same conditions with decreasing the reaction times from 1.5–5 h to 0.75–3.5 h (method A, Table 1 and Schemes 1 and 2). Although reactions occurred both in the presence and in the absence of molec-

ular iodine but I₂ catalyzed the described reaction efficiently and also improved the reactions yields (Table 1, method A). For showing the catalytic role of iodine molecule two reactions were run in the presence of I₂ solely and without any using of acetic acid (method C, Scheme 1, Table 1, entries **4f** and **4j**). As shown in the table, times and yields of reactions were not seriously changed but purification of crude products of the method A was more easier than methods B and C. Reaction did not occur in the absence of both of acetic acid and I₂, therefore one of them is necessary. The crude products were isolated by precipitation upon addition of cold aqueous NaHCO₃ (pH 8–9) to the reaction mixture followed by vigorous shaking and decanting. Thus methyl 1-hydroxyethyl-1,4-dihydro-2,6-dimethylpyridine-3,5-dicarboxylates (**4**) obtained with good to high yields and purities. Bis (methyl 1-hydroxyethyl-1,4-

Scheme 3.

Fig. 1. Suggested complexes molecules with multi 1,4-dihydopyridine moieties which may be prepared.

dihydro-2,6-dimethylpyridine-3,5-dicarboxylate) (6) was also prepared under the same conditions (Scheme 2 and Table 1), which is an excellent precursor for the synthesis of new complex molecules (Fig. 1). We sure that selective oxidation of hydroxy group of *N*-hydroxyethyl moiety of 4 and 6 may be occurred by choosing a suitable oxidizing agent (Scheme 3) because oxidation of similar functional group with same moieties has been

Table 1 Synthesis of methyl 1-hydroxyethyl-1,4-dihydro-2,6-dimethylpyridine-3,5-dicarboxylate 4 under solvent-free conditions at 40 °C with a combination of methyl acetoacetate (1) (3 mmol), aldehyde (2) (1 mmol), ethanolamine (3) (1.5 mmol), HOAc (1.5 mmol), molecular iodine [I $_2$ (0.038 g, 0.15 mmol)] as a catalyst (method A) and in the absence of molecular iodine catalyst (method B)

Product	R	Time (h)		Yield ^a (%)	
		A	B (C) ^b	A	B (C) ^b
4a	2-Br-C ₆ H ₄	1.5	2.5	98	80
4b	$3-Br-C_6H_4$	1.5	2	97	82
4c	4 -Br– C_6H_4	1	2	98	75
4d	$2-C1-C_6H_4$	1.25	1.5	89	72
4e	$3-Cl-C_6H_4$	0.75	1.5	93	65
4f	$4-Cl-C_6H_4$	0.75	2 (0.75)	85	60 (84)
4g	$2-NO_2-C_6H_4$	1	4	91	73
4h	$3-NO_2-C_6H_4$	0.75	3	90	69
4i	$4-NO_2-C_6H_4$	2	4	92	79
4j	2-Furyl	3	4.25(3)	89	73 (89)
4k	2-Thienyl	3.5	5	85	64
6	Terephtaldialdehyde	1.5	2	89	75

^a Crude isolated yields.

reported in the literature [24]. To the best of our knowledge, oxidation of *N*-substituted 1,4-dihydropyridines into the corresponding *N*-substituted pyridines may be occurred under force full conditions [23]. Therefore synthesis of **7** and its bis analogous let us achieving to the designed new complex molecules with multi 1,4-dihydropyridine moieties in future.

In conclusion, synthesis of new derivatives of 1,4-dihydropyridines with a reactive functionality, simple work-up, and high yield makes this method an attractive methodology. We believe that the present methodology addresses the current devise toward green chemistry due to high yields and atomic economy, and fewer reagents. Meanwhile, the new 1,4-dihydropyridine derivatives that reported in this article has hydroxyethyl moiety, which are excellent precursors for the synthesis of new molecules containing of multi 1,4-dihydropyridine moieties.

1. Experimental

1.1. General

Chemicals were purchased from Fluka, Merck and Aldrich chemical companies. Yields refer to isolated crude products. The products were characterized by their spectroscopic data (IR, ¹H NMR and MS) and microanalysis.

1.2. Synthesis of methyl 1-hydroxyethyl-1,4-(2-bromophenyl)dihydro-2,6-dimethylpyridine-3,5-dicarboxylate (4a), a typical procedure

Methylacetoacetate 1 (0.348 g, 3 mmol) was added to a stirred mixture of 2-bromobenzaldehyde (2a) (0.185 g, 1 mmol), a mixture of ethanol amine 3 and acetic acid as

 $[^]b$ Method C: synthesis of methyl 1-hydroxyethyl-1,4-dihydro-2,6-dimethyl-pyridine-3,5-dicarboxylate (4) under solvent-free conditions at 40 $^\circ\text{C}$ with a combination of methyl acetoacetate (1) (3 mmol), aldehyde (2) (1 mmol), ethanolamine (3) (1.5 mmol), molecular iodine (0.038 g, 0.15 mmol) as a catalyst in the absence of acetic acid.

ethanolammonium acetate [HOCH₂CH₂NH₃⁺OAc⁻ (0.181 g, 1.5 mmol)] at 40 °C for 2.5 h. Progress of the reaction was followed by TLC. After completion of the reaction, cold aqueous NaHCO₃ (pH 8–9) (20 mL) was added to the reaction mixture followed by vigorous shaking and decanting. The residue was dissolved in dichloromethane and anhydrous Na₂SO₄ (3 g) was added. Solvent was removed after filtration and methyl 1-hydroxyethyl-1,4-dihydro-2,6-(2-bromophenyl) dimethylpyridine-3,5-dicarboxylate (4a) was obtained in 80% yield. Recrystallization of crude product in a mixture of ethanol–water gave highly pure product.

1.3. Synthesis of methyl 1-hydroxyethyl-1,4-(aryl)dihydro-2,6-dimethylpyridine-3,5-dicarboxylate in the presence of I_2 as a catalyst: a typical procedure

A similar procedure for running the reactions in the presence of I_2 was used. So that I_2 (0.038 g, 0.15 mmol) was added in the first step together with other reactants in a reaction vessel.

1.4. Spectral and microanalysis data of 1-hydroxyethyl-1,4-(aryl)dihydro-2,6-dimethylpyridine-3,5-dicarboxylates

4a—mp: 201–202 °C, IR (KBr): (3448, 3279, 2950, 1739, 1578, 1282, 1213). ¹H NMR (90 MHz, CDCl₃), δ (ppm) 1.8 (s, 6H), 2.1 (s, 1H), 2.44 (t, 2H), 3.1 (t, 2H), 3.7 (s, 6H), 5.0 (s, 1H), 7.1–7.5 (m, 4H). MS (*m/z*): 425. Anal. Calcd.: C, 53.7; H, 5.1; N, 3.3; Found: C, 53.1; H, 5.1; N, 3.1.

4b—mp: 174–175 °C, IR (KBr): (3500, 2951, 1743, 1652, 1435, 1361). 1 H NMR (90 MHz, CDCl₃), δ (ppm): 1.7 (s, 6H), 2.2 (s, 1H), 2.7 (t, 2H), 3.3 (t, 2H), 3.9 (s, 6H), 5.1 (s, 1H), 7.1–7.4 (m, 4H). MS (m/z): 424.

4c—mp: 185–186 °C, IR (KBr): (3507, 2948, 1736, 1663, 1489, 1161). ¹H NMR (90 MHz, CDCl₃),δ (ppm): 1.7 (s, 6H), 2.2 (s, 1H), 2.7 (t, 2H), 3.1 (t, 2H), 3.7 (s, 6H), 5.1 (s, 1H), 7.1 (d, 2H), 7.4 (d, 2H). MS (m/z): 424.

4d—mp: 199–200 °C, IR (KBr): (3448, 2953, 1739, 1663, 1579, 1289, 1177). 1 H NMR (90 MHz, CDCl₃), δ (ppm): 1.8 (s, 6H), 2 (s, 1H), 2.8 (t, 2H), 3.1 (t, 2H), 3.7 (s, 6H), 5 (s, 1H), 7–7.3 (m, 4H). MS (*m/z*): 380. Anal, Calcd.: C, 60.0; H, 6.2; N, 3.6; Found: C, 59.3;H, 5.8; N, 3.8.

4e—mp:178–179 °C, IR (KBr): (3486, 2952, 1740, 1663, 1597, 1247). ¹H NMR (90 MHz, CDCl₃), δ (ppm): 1.7 (s, 6H), 2.2 (s, 1H), 2.7 (t, 2H), 3.3 (t, 2H), 3.7 (s, 6H), 5.1 (s, 1H), 7.1–7.5 (m, 4H). MS (m/z): 380.

4f—mp: 183–184 °C, IR (KBr): (3506, 2953, 1732, 1660, 1493, 1379). 1 H NMR (90 MHz, CDCl₃), δ (ppm): 1.7 (s, 6H), 2 (s, 1H), 2.7 (t, 2H), 3.4 (t, 2H), 3.7 (s, 6H), 5 (s, 1H), 7.2 (d, 2H), 7.4 (d, 2H). MS (*m/z*): 380.

4g—mp:211–212 °C, IR(KBr): (3464, 2926, 1739, 1665, 1455, 1293). ¹H NMR (90 MHz, CDCl₃), δ (ppm): 1.8 (s, 6H), 2 (s, 1H), 2.7 (t, 2H), 3.4 (t, 2H), 3.7 (s, 6H), 5 (s, 1H), 7.3–7.7 (m, 4H). MS (*m/z*): 391. Anal. Calcd.: C, 58.4; H, 5.6; N, 7.1; Found: C, 57.3; H, 5.5; N, 6.9.

4h—mp: 198–199 °C, IR (KBr): (3520, 2964, 1736, 1663, 1530, 1351). ¹H NMR (90 MHz, CDCl₃), δ (ppm): 1.7 (s, 6H),

2.1 (s, 1H), 2.7 (t, 2H), 3.2 (t, 2H), 3.7 (s, 6H), 5.1 (s, 1H), 7.5–7.8 (m, 4H). MS (*m/z*): 391.

4i—mp:185–186 °C, IR (KBr): (3290, 2953, 1735, 1650, 1582, 1353). ¹H NMR (90 MHz, CDCl₃), δ (ppm): 1.7 (s, 6H), 2.1 (s, 1H), 2.7 (t, 2H), 3.5 (t, 2H), 3.7 (s, 6H), 5 (s, 1H), 7.3–7.5 (d, 2H), 7.6–7.9 (d, 2H). MS (m/z): 390.

4j—mp: 115–116 °C, IR (KBr): (3426, 2954, 1738, 1660, 1575, 1226). H NMR (90 MHz, CDCl₃), δ (ppm): 1.7 (s, 6H), 2 (s, 1H), 2.6 (t, 2H), 3.3 (t, 2H), 3.7 (s, 6H), 5.1 (s, 1H), 6–6.8 (m, 3H). MS (m/z): 336. Anal. Calcd.: C, 60.7; H, 6.5; N, 4.1; Found: C, 60.6; H, 6.2; N, 4.2.

4k—mp: 132–133 °C, IR (KBr): (3432, 2953, 1736, 1661, 1575, 1282). ¹H NMR (90 MHz, CDCl₃), δ (ppm): 1.7 (s, 6H), 2 (s, 1H), 2.6 (t, 2H), 3.3 (t, 2H), 3.7 (s, 6H), 5.1 (s, 1H), 6.6–7 (m, 3H). MS (*m*/*z*): 351. Anal. Calcd.: C, 57.9; H, 6.2; N, 3.9; Found: C, 56.8; H, 5.8; N, 4.2.

6—mp: 227–228 °C, IR (KBr): (3406, 2956, 1739, 1662, 1436, 1248). ¹H NMR (90 MHz, CDCl₃), δ (ppm): 1.55 (s, 12H), 2 (s, 2H), 2.8 (t, 4H), 3.3 (t, 4H), 3.5 (s, 12H), 4.9 (s, 2H), 7.2 (s, 4H). MS (*m*/*z*): 609.

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