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Full Paper

Zinc Hydrogensulfate as an Efficient Catalyst for Preparation of *β*-Amido Carbonyl Compounds

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A one-pot and efficient three-component condensation of benzaldehyde derivatives, enolizable ketones or ketoesters, acetyl chloride, and acetonitrile or benzonitrile under ambient conditions in the presence of zinc hydrogensulfate for the synthesis of *β*-amido carbonyl compounds is described. The simple experimental procedure, high to excellent yields of products and preparation of diastereoselective *β*-acetamido ketoesters are strong features of the presented method

Keywords multi-component reaction, zinc hydrogensulfate, metal hydrogensulfate, *β*-amido carbonyl compound, diastereoselectivity

Introduction

Multi-component reaction (MCR) plays an important role in combinatorial chemistry because of its ability to synthesize small drug-like molecules with several degrees of structural diversity easily in a single procedural step and also avoiding complicated purification operations.[1]

β-Acetamido ketones and esters are valuable building blocks for a number of biologically and pharmaceutically bioactive important compounds.[2,3] They are easily converted to 1,3-amino alcohols, which are precursors for the synthesis of several antibiotics such as nikkomycins or neopolyoxins.[4,5]

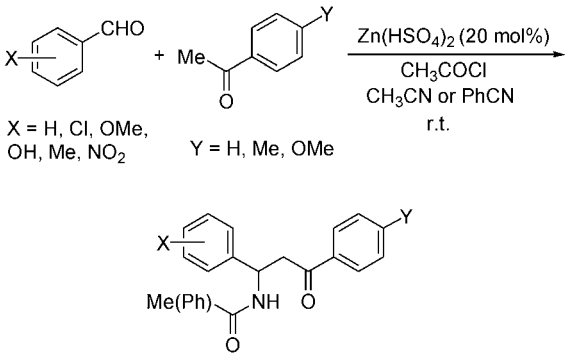
The common procedure for the synthesis of the acetamide ketone compounds is the Dakin-West reaction,[6] which involves the condensation of *α*-amino acids with acetic anhydride in the presence of suitable base,[6] and these reactions produce *α*-acetamido ketones through an intermediate azalactone.[6d]

Iqbal *et al*. reported a multi-component reaction for the synthesis of *β*-acetamido ketones through the condensation of acetophenone, benzaldehydes and acetyl chloride in acetonitrile in the presence of cobalt(II) chloride[7,8] or montmorillonite K-10 clay[9] as catalyst. Other catalysts such as silica sulfuric acid,[10] $BiCl_3$ generated from BiOCl,[11] $ZrOCl_2 \cdot 8H_2O$,[12] heteropoly acid,[13] $H_3PW_{12}O_{40}$,[14] sulfuric acid absorbed on silica gel,[15] $Sc(OTf)_3$,[16] $K_5CoW_{12}O_{40} \cdot 3H_2O$,[17] $CeCl_3 \cdot 7H_2O$,[18] ZnO,[19] iodine,[20] Amberlyst-15,[21] sulfated zirconia,[22] iron(III) chloride,[23] *p*-TSA,[24] $Fe(HSO_4)_3$,[25] $Mg(HSO_4)_2$,[26] $HClO_4$-SiO_2,[27] PCl_3[28] and $ZrCl_4$[29] have been used for the mentioned transformation.

Zinc hydrogensulfate was prepared from the reaction of anhydrous zinc chloride with concentrated sulfuric acid.[30] This catalyst has been used in some organic reactions, such as synthesis of bis(indolyl)methanes[30a] and preparation of 3,4-dihydropyrimidine-2(1*H*)-one.[30b]

Considering the above facts and also in extension of our previous studies on acidic catalyst in organic reactions,[31] we now reported a new, simple, and effective procedure for the one-pot synthesis of *β*-amido carbonyl compound via a multi-component condensation reaction from benzaldehydes, enolizable ketones or ketoesters, acetyl chloride, and acetonitrile or benzonitrile in the presence of zinc hydrogensulfate as catalyst at room temperature (Scheme 1).

Scheme 1



Experimental

All reagents were purchased from Merck and Aldrich and used without further purification. $Zn(HSO_4)_2$ was prepared according to the reported literature.[30] All yields refer to isolated products after purification. Products were characterized by comparison with authentic samples and by spectroscopy data (IR, ^{1}H NMR).

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The NMR spectra were recorded on a Bruker Avance DPX 500 MHz instrument in DMSO-d_6 relative to TMS. Mass spectra were recorded on an Agilent technologies 5973 network mass selective detector (MSD) operating at an ionization potential of 70 eV. Elemental analyses for C, H and N were performed using a Heraeus CHN-O-rapid analyzer. Melting points were determined in open capillaries with a Buchi 510 melting point apparatus. TLC was performed on silica-gel polygram SIL G/UV 254 plates.

Preparation of zinc(II) hydrogensulfate

A 50 mL suction flask was equipped with a dropping funnel. The gas outlet was connected to a vacuum system through an alkaline solution trap. Anhydrous zinc chloride (10 mmol) was charged into the flask and concentrated sulfuric acid 98% (20 mmol) was added dropwise over a period of 30 min at room temperature. HCl evolved immediately. After completion of the addition, the mixture was shaken for 30 min at 100 ℃, while the residual HCl was eliminated by suction. Finally, a white solid $Zn(HSO_4)_2$ was obtained.[30]

Typical experimental procedure for the one-pot preparation of *N*-[1-(2,3-dimethoxyphenyl)-3-oxo-3-phenylpropyl]acetamide (Table 2, Entry 12) A mixture of 2,3-dimethoxybenzaldehyde (1 mmol), acetophenone (1 mmol), acetyl chloride (0.5 mL) and acetonitrile (2 mL) in the presence of zinc hydrogensulfate (0.05 g, 20 mol%) was stirred at room temperature in the appropriate time (Table 2). The progress of the reaction was followed by TLC. After completion of the reaction, acetonitrile was evaporated from the reaction mixture. The reaction residue was poured into 50 mL of ice-water. The solid residue was isolated and dissolved in dichloromethane (5 mL). Silica-gel (2 g) was added to the solution and then evaporation of the solvent afforded a resorbed material, which was purified by column chromatography [petroleum ether (60—80 ℃)/ethyl acetate (*V*/*V*, 4/1)]. Eluent solvent was evaporated under reduced pressure to give the desired pure *N*-[1-(2,3-dimethoxyphenyl)-3-oxo-3-phenylpropyl]acetamide in 85% yield (Table 2, Entry 12). ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.99 (s, 3H), 3.40 (dd, J=6.0, 15.7 Hz, 1H), 3.55 (dd, J=6.6, 15.7 Hz, 1H), 3.85 (s, 3H), 3.97 (s, 3H), 5.73 (dd, J=6.4, 14.3 Hz, 1H), 6.80—6.85 (m, 3H), 6.98 (t, J=8.0 Hz, 1H), 7.43 (t, J=7.7 Hz, 2H), 7.54 (t, J=7.4 Hz, 1H), 7.95 (d, J=7.6 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 23.4, 43.7, 47.1, 55.7, 60.7, 111.7, 119.5, 124.1, 128.3, 128.6, 133.4, 134.5, 136.5, 146.2, 152.6, 169.2, 198.8; IR (KBr) ν: 3257, 3069, 2937, 1685, 1639, 1560, 1480, 1449, 1372, 1282, 1220, 1084, 1052, 1003, 756, 690 cm^{-1}; MS (70 eV) *m/z* (%): 327.2 (M^+, 10), 284.3 (100), 237.2 (15), 208.2 (22), 180.2 (21), 166.2 (74), 105.2 (59), 77.2 (31), 51.2 (5), 43.2 (2). Anal. calcd for $C_{19}H_{21}NO_4$ C 69.71, H 6.47, N 4.28; found C 69.55, H 6.40, N 4.35.

The desired pure products were characterized by comparison of their physical data with those of known β-amido carbonyl compounds.[7-29]

Results and discussion

First, we prepared *N*-(3-oxo-1,3-diphenylpropyl)-acetamide from the reaction of benzaldehyde, acetophenone, acetyl chloride and acetonitrile (reactant as well as solvent) in the presence of ZnO, $ZnCl_2$, $Zn(OTf)_2$ and $Zn(HSO_4)_2$ as catalyst (Table 1). The results in Table 1 showed that amongst these catalysts, $Zn(HSO_4)_2$ was the catalyst of choice in terms of time and yield. Next, we optimized the amount of $Zn(HSO_4)_2$ in the mentioned reaction (Table 1), which was found to be 20 mol%.

Table 1 Preparation of *N*-(3-oxo-1,3-diphenylpropyl)acetamide[a]

Entry	Catalyst	Amount of catalyst/mol%	Time/h	Yield[b]/%
1	None	—	24	trace
2	ZnO	20	24	30
3	$Zn(OTf)_2$	10	30	60
4	$ZnCl_2$	20	24	30
5	$Zn(HSO_4)_2$	20	4	84
6	$Zn(HSO_4)_2$	40	2.5	89
7	$Zn(HSO_4)_2$	30	3.5	86
8	$Zn(HSO_4)_2$	10	7.5	68
9	$Zn(HSO_4)_2$	5	12	40

[a] The effect of various catalysts and amount of $Zn(HSO_4)_2$ was studied. [b] Yields refer to the isolated pure products.

Thus, we continued preparation of β-amido ketones in an optimum model experiment: benzaldehydes (1 equiv.), enolizable ketones (1 equiv.), acetyl chloride (0.5 mL), acetonitrile, reactant as well as solvent (2 mL) or benzonitrile (2 mL) in the presence of zinc hydrogensulfate (0.05 g, 20 mol%) under ambient conditions (Table 2).

As shown in Table 2, aromatic aldehydes and acetophenone derivatives with both electron-withdrawing and electron-donating substituents afforded the corresponding β-amido ketones without the formation of any side products, in high to excellent yields (Table 2, Entries 1—20). Phenolic —OH groups under present reaction conditions were converted to acetate (—OAc) groups (Table 2, Entries 14, 15 and 18).

Under the optimized reaction conditions, by using benzonitrile in place of acetonitrile, aldehydes were transformed to their corresponding β-benzamido ketones in high yields (Table 2, Entries 2, 5, 7 and 9).

We also studied the multi-component reaction of aromatic aldehydes, other enolizable ketones (methyl acetoacetate, ethyl acetoacetate and propiophenone) and acetonitrile in the presence of acetyl chloride and zinc

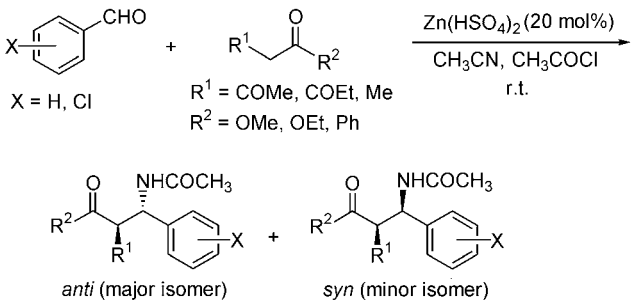
Table 2 Preparation of *β*-amido ketones catalyzed by $Zn(HSO_4)_2$ at room temperature

Entry	Aldehyde	Enolizable ketone[a]	Nitrile	Time/h (Yield[b]/%)	m.p. (Lit. m.p.)/℃
1	Benzaldehyde	H	CH_3CN	4 (84)	102—104 (103—105)[13]
2	Benzaldehyde	H	PhCN	4 (78)	153—156 (153—154)[23]
3	Benzaldehyde	4-OMe	CH_3CN	4 (83)	129—131 (130)[11]
4	4-Chlorobenzaldehyde	H	CH_3CN	3.5 (79)	149—151 (149—150)[12]
5	4-Chlorobenzaldehyde	H	PhCN	4 (70)	179—182 (180—182)[23]
6	4-Nitrobenzaldehyde	H	CH_3CN	5.5 (81)	141—144 (153)[23]
7	4-Nitrobenzaldehyde	H	PhCN	6 (75)	142—145 (142—144)[23]
8	3-Nitrobenzaldehyde	H	CH_3CN	5 (86)	135—137 (139—140)[12]
9	3-Nitrobenzaldehyde	H	PhCN	5.5 (78)	192—195 (194—195)[12]
10	Cinnamaldehyde	H	CH_3CN	3 (80)	120—122 (120—121)[23]
11	4-Methoxybenzaldehyde	H	CH_3CN	3.5 (76)	108—111 (110—112)[12]
12	2,3-Dimethoxybenzaldehyde	H	CH_3CN	4 (85)	117—119 (117—119)[27]
13	2,3-Dimethoxybenzaldehyde	4-Me	CH_3CN	3.5 (88)	108—111 (108—110)[26]
14	2-Methoxy-4-hydroxybenzaldehyde	H	CH_3CN	3 (83)	128—130 (129—132)[26]
15	2-Methoxy-4-hydroxybenzaldehyde	4-Me	CH_3CN	2.5 (89)	90—93 (89—93)[27]
16	2,5-Dimethoxybenzaldehyde	H	CH_3CN	3.5 (85)	151—154 (153—155)[26]
17	4-Methylbenzaldehyde	H	CH_3CN	3.5 (74)	111—114 (112)[12]
18	4-Hydroxybenzaldehyde	H	CH_3CN	2.5 (68)	120—122 (120—122)[26]
19	3-Nitrobenzaldehyde	4-Me	CH_3CN	6 (75)	94—97 (94—97)[27]
20	4-Methylbenzaldehyde	4-Me	CH_3CN	4.5 (86)	103—105 (103—105)[26]

[a] Enolizable ketones refer to acetophenone derivatives according to Scheme 1. [b] Yield refers to the isolated pure products; the *β*-amido ketones were prepared from the reactions of benzaldehydes and enolizable ketones in the presence of aceyl chloride, and nitriles.

hydrogensulfate (20 mol %) as catalyst (Scheme 2).

Scheme 2



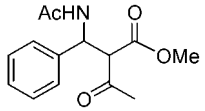
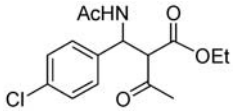
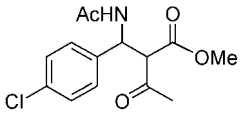
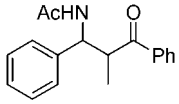
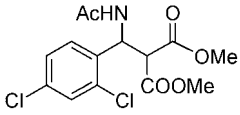
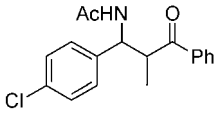
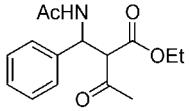
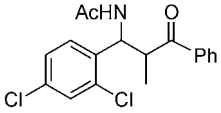
In all cases, mixtures of *syn* and *anti* diastereomers were obtained. The result showed that the diastereoselectivity depended upon the nature of the reactants (Table 3). The amount of these *syn* and *anti* products was determined by ^{1}H NMR spectra, since the coupling constant between H-2 and H-3 is 6—9 Hz for an *anti* isomer, while 2—5 Hz for a *syn* isomer.[7-9,24]

As it was shown in Table 3, an *anti* diastereomer was found to be the major product. Methyl acetoacetate and ethyl acetoacetate afforded the corresponding *β*-acetamido ketoesters in good yields and diastereoselectivity. The *syn*/*anti* ratio was determined from ^{1}H NMR spectrum of crude reaction mixture.

The probable catalytic mechanistic pathway for preparation of *β*-acetamido ketone as a typical example may be depicted as shown in Scheme 3. However operation process chart (OPC) of the catalyst in this work is unknown, but on the basis of previously reported mechanism for applying of $CoCl_2$[8] and BiOCl[11] to the preparation of *β*-amido ketones using aldehydes, enolizable ketones, acetonitrile and our observation in the course of reaction, we suggested that aldehydes react with acetyl chloride in acetonitrile in the presence of a catalytic amount of $Zn(HSO_4)_2$ to afford *α*-chloroacetate **I**. Acetonitrile is incorporated in the intermediate **I** and formed **II**. Acetate migration and coupling with the acetophenone enolate derivatives are similar to those proposed by Iqbal *et al.*[8] in a $CoCl_2$-catalyzed reaction. We confirmed the suggested mechanism using benzoyl chloride instead of acetyl chloride in the same reaction conditions. Benzoic acid was obtained as by-product in place of acetic acid.

To show the merit of the present work in comparison with reported results in the literature, we compared results of zinc hydrogensulfate with BiOCl,[11] $ZrOCl_2 \cdot 8H_2O$,[12] $CeCl_3 \cdot 7H_2O$,[18] ZnO,[19] iodine,[20] $Fe(HSO_4)_3$,[25] and $Mg(HSO_4)_2$[26] in the synthesis of *β*-acetamido ketone derivatives (Table 4). The results show that zinc hydrogensulfate promotes the reactions more effectively than BiOCl, $ZrOCl_2 \cdot 8H_2O$, $CeCl_3 \cdot 7H_2O$, ZnO and iodine with respect to reaction time. Reactions in the presence of $Fe(HSO_4)_3$ required reflux conditions but

Table 3 Diastereoselective preparation of *β*-acetamido ketoesters and *β*-acetamido ketones using $Zn(HSO_4)_2$ at room temperature

Entry	Product	Time/h (Yield[a]/%)	*Syn anti* ratio[b]/%	Entry	Product	Time/h (Yield[a]/%)	*Syn anti* ratio[b]/%
1		5.5 (81)	24 (76)	5		6 (80)	19 (81)
2		5.5 (84)	30 (70)	6		5 (79)	37 (63)
3		5.5 (82)	20 (80)	7		5 (81)	40 (60)
4		6 (79)	17 (83)	8		5 (84)	31 (69)

[a] Yields are reported after aqueous work-up; [b] Ratio obtained from ^{1}H NMR of the crude reaction mixture.

Scheme 3

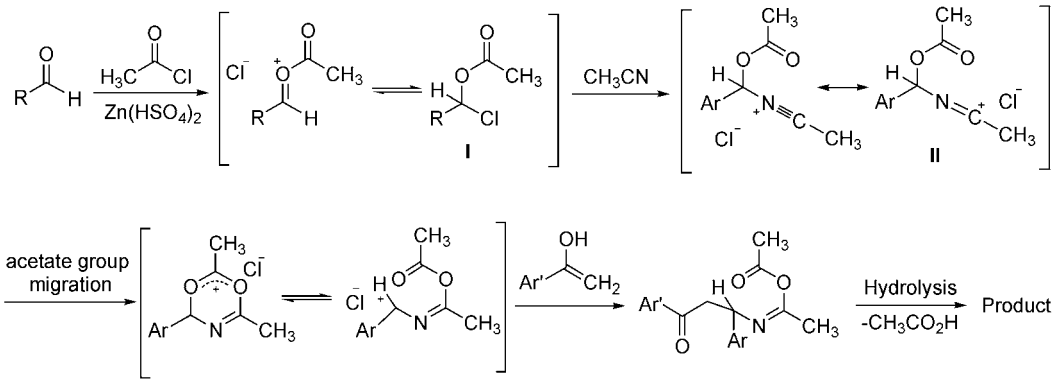
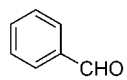
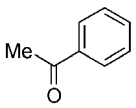
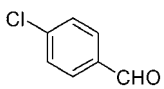
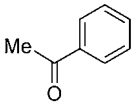
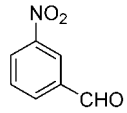
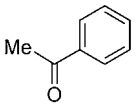


Table 4 Comparison result of zinc hydrogensulfate with BiOCl,[11] $ZrOCl_2•8H_2O$,[12] $CeCl_3•7H_2O$,[18] ZnO,[19] iodine,[20] $Fe(HSO_4)_3$[25] and $Mg(HSO_4)_2$[26] in the synthesis of *β*-acetamido ketones

Entry	Aldehyde	Enolizable ketone	Catalyst	*n*(aldehyde)/*n*(enolizable ketone) (catalyst/mol%)	Time/h	Yield/%
1			BiOCl	1/1 (20 mol%)	7	92
			$ZrOCl_2•8H_2O$	1/1 (20 mol%)	5	90
			$CeCl_3•7H_2O$	1/1 (10 mol%)	7	96
			ZnO	1/1 (50 mol%)	6	90
			I_2	1/1 (10 mol%)	4.5	85
			$Fe(HSO_4)_3$	1/1 (25 mol%)	50 min	93
			$Mg(HSO_4)_2$	1/1 (20 mol%)	2.5	89
			$Zn(HSO_4)_2$	1/1 (20 mol%)	4	84
2			BiOCl	1/1 (20 mol%)	10	80
			$ZrOCl_2•8H_2O$	1/1 (20 mol%)	8	91
			$CeCl_3•7H_2O$	1/1 (10 mol%)	6	98
			ZnO	1/1 (50 mol%)	5.5	92

Continued

Entry	Aldehyde	Enolizable ketone	Catalyst	*n*(aldehyde)/*n*(enolizable ketone) (catalyst/mol%)	Time/h	Yield/%
			I_2	1/1 (10 mol%)	4.5	85
			$Fe(HSO_4)_3$	1/1 (25 mol%)	1.5	94
			$Mg(HSO_4)_2$	1/1 (20 mol%	2	92
			$Zn(HSO_4)_2$	1/1 (20 mol%)	3.5	79
			BiOCl	1/1 (20 mol%)	8	91
			$ZrOCl_2•8H_2O$	1/1 (20 mol%)	6	92
3			$CeCl_3•7H_2O$	1/1 (10 mol%)	10	90
			ZnO	1/1 (50 mol%)	5	90
			I_2	1/1 (10 mol%)	4	85
			$Fe(HSO_4)_3$	1/1 (25 mol%)	2	90
			$Mg(HSO_4)_2$	1/1 (20 mol%)	4	84
			$Zn(HSO_4)_2$	1/1 (20 mol%)	5	86

$Zn(HSO_4)_2$ and $Mg(HSO_4)_2$ catalyses were performed in CH_3CN at room temperature.

Conclusion

In summary, we have demonstrated a new and important catalytic activity of zinc hydrogensulfate as an inexpensive, commercially available catalyst for the synthesis of *β*-amido ketones and ketoesters in high to excellent yields. The simple experimental procedure combined with the easy work-up, preparation of highly diastereoselective *β*-acetamido ketoesters, mild and ambient conditions are advantages of the presented method.

Acknowledgments

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