

Zn(BH₄)₂/Ac₂O/DOWEX(R)50WX4: A Novel System for Acylation of Aldehydes

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Abstract: The acylation of structurally different aldehydes has been performed by Zn(BH₄)₂/Ac₂O/DOWEX(R)50WX4 as new system within 1-5 min at room temperature with excellent yields of the products (92-97%).

Key Words: Zn(BH₄)₂, Ac₂O, DOWEX(R)50WX4, acylal, *gem*-diacetate, aldehyde.

Resumen: La acilación de aldehídos estructuralmente diferentes ha sido realizada empleando Zn(BH₄)₂/Ac₂O/DOWEX(R)50WX4 como un sistema novedoso, en 1-5 min a temperatura ambiente, con excelentes rendimientos de los productos (92-97%).

Palabras clave: Zn(BH₄)₂, Ac₂O, DOWEX(R)50WX4, acilal, *gem*-diacetato, aldehído.

Introduction

Acylals have been used as starting materials for Diels-Alder [1], Grignard [2a], Barbier [2b], Prins [3], Knoevenagel [4a] and benzoin condensation reactions [4b]. Also, acylals were used in the synthesis of chrysanthemoid acid [5a], sphingofungins E and F [5b] and utilized as cross linking reagents [6] in cellulose and cotton industry. However, the protection of carbonyl functional group of aldehydes is the main goal for the synthesis of acylals, because *gem*-diacetates are stable under critically controlled acidic, neutral and basic conditions [7].

Several reagents or catalysts such as amberlyst-15 [8], envirocat EPZ10 [9], montmorillonite [10], zeolites [11], nafion-H [12], FeSO₄ [13], FeCl₃ [14], AlCl₃ [15], TMSCl-NaI [16], Sc(OTf)₃ [17], I₂ [18], NBS [19], PCl₃ [20], H₂SO₄ [21], Cu(OTf)₂ [22], LiBF₄ [23], H₂NSO₃H [24], InCl₃ [25], (NH₄)₂Ce(NO₃)₆ [26], LiOTf [27], Zn(BF₄)₂ [28], AIPW₁₂O₄₀ [29], ZrCl₄ [30], Bi(NO₃)₃·5H₂O [31], Bi(CF₃SO₃)₃·4H₂O [32], zirconium sulfohenyl phosphonate [33], GaCl₃ [34], GaI₃ [35], sulphated zirconia [36], poly(*N,N'*-dibromo-*N*-ethyl-benzene-1,3-disulfonamide) [PBBS] and *N,N,N',N'*-tetrabromobenzene-1,3-disulfonamide [TBBDA] [37], saccharin sulfonic acid [38], zirconium hydrogen sulfate [39], Fe₂(SO₄)₃·xH₂O [40], erbium triflate [41], alum[KAl(SO₄)₂·12H₂O] [42], sulfated zirconia [43], KHCO₃ [44], H₂SO₄-silica [45], indium tribromide [46], HClO₄-SiO₂ [47], solid lithium perchlorate [48], zinc(II) perchlorate [49] and [Hmim] HSO₄ [50] have been used for synthesis of *gem*-diacetates.

These methods are convenient but have some disadvantages such as long reaction times, harsh reaction conditions, use of strong acids, strictly reaction conditions and moisture sensitivity. Also, several of these catalysts are toxic, unavailable and costly. Thus, the research is still so much interest and we have investigated the acylation of aldehydes in the presence of a reducing agent. So, in this context, we wish to introduce a fast and efficient method for the acylation of a variety of aldehydes to their corresponding *gem*-diacetates using Ac₂O and DOWEX(R)50WX4 in the presence of Zn(BH₄)₂.

Results and discussion

Recently, we have reported that DOWEX(R)50WX4 ion-exchange resin has been used for regioselective synthesis of oximes by NH₂OH·HCl/DOWEX(R)50WX4 system [51], reduction of a variety of carbonyl compounds such as aldehydes, ketones, α -diketones, acylalins and α,β -unsaturated carbonyl compounds to their corresponding alcohols by NaBH₄/DOWEX(R)50WX4 system [52], synthesis of cyanohydrins by NaCN/DOWEX(R)50WX4 [53] and reductive-amination of a variety of aldehydes and anilines by NaBH₄/DOWEX(R)50WX4 [54].

On the other hand, Zn(BH₄)₂ is the modified borohydride agent which has better solubility in aprotic solvents such as THF, Et₂O and DME. It is unique because of the better coordination ability of Zn²⁺ which is imparting selectivity in hydride-transferring reactions. We have developed the use of Zn(BH₄)₂ under new reducing system such as Zn(BH₄)₂/H₂O [55], Zn(BH₄)₂/C [56], Zn(BH₄)₂/ZrCl₄ [57], Zn(BH₄)₂/Al₂O₃ [58] and Zn(BH₄)₂/2NaCl [59]. In continuing our efforts for the development of using Zn(BH₄)₂ and DOWEX(R)50WX4, herein, we now wish to introduce Zn(BH₄)₂/Ac₂O/DOWEX(R)50WX4 as new convenient system for efficient acylation of aldehydes at room temperature.

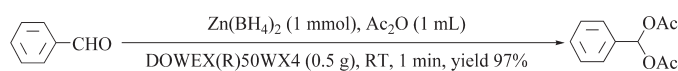
For the selection of appropriate conditions, acylation of benzaldehyde has been selected as model reaction. This reaction was performed with different amounts of Zn(BH₄)₂, Ac₂O and DOWEX(R)50WX4 in different solvents (THF/Ac₂O, Et₂O/Ac₂O, CH₃CN/Ac₂O, EtOAc/Ac₂O, Ac₂O) at room temperature as shown in Table 1.

Experiments show that the reaction was proceeded to give the highest yield in Ac₂O. The optimization reaction conditions showed that the use of 1 molar equivalent of Zn(BH₄)₂ and 0.5 g of DOWEX(R)50WX4 in 1 mL Ac₂O are the best conditions to complete the acylation of benzaldehyde (1 mmol) to *gem*-benzylidiacetate (Table 1, entry 4). Our observation revealed that the acylation was completed within 1 min with 97% yield of product as shown in Scheme 1.

Table 1. The optimization of acylation of benzaldehyde (1 mmol) to 1,1-diacetoxy-1-phenylmethane with Zn(BH₄)₂ and DOWEX(R)50WX4 at RT.

Entry	Zn(BH ₄) ₂ /mmol	DOWEX(R)50WX4/g	Solvent/1 mL	Time/min	Conversion/% ^a
1	0	0.5	EtOAc/Ac ₂ O (0.5:0.5)	30	0
2	0	0.5	Ac ₂ O (1)	30	0
3	1	0.5	EtOAc/Ac ₂ O (1:0)	30	0
4	1	0.5	Ac ₂ O (1)	1	100
5	1	0.5	EtOAc/Ac ₂ O (0.5:0.5)	30	100
6	0.5	0.5	Ac ₂ O (1)	30	100
7	1	0.25	Ac ₂ O (1)	30	100
8	1	0.5	THF/Ac ₂ O (0.5:0.5)	30	100
9	1	0.5	Et ₂ O/Ac ₂ O (0.5:0.5)	30	100
10	1	0.5	CH ₃ CN/Ac ₂ O (0.5:0.5)	30	100

^a Conversions refer to 1,1-diacetoxy-1-phenylmethane and were monitored by TLC (eluent *n*-hexane/EtOAc 9/1).

**Scheme 1.** The acylation of benzaldehyde to *gem*-benzyl diacetate based on optimized reaction conditions.

The efficiency of this protocol was examined by using structurally different aldehydes as shown in Table 2. In this Table, the entries 1-12 are simple aromatic aldehydes and entry 14 is a simple aliphatic aldehyde. Also, the entries 2-8 are aromatic aldehydes with electron-withdrawing groups while the entries 9-11 have electron-donating groups. The entry 13, corresponds to a α,β -unsaturated aldehyde. In this approach, the corresponding acylals were obtained in excellent yields (92-97%) and the reactions have been completed within 1-5 min as shown in Table 2. The products were characterized by the ¹H-chemical shift of the CHs (Table 2, column 6) which appear around 6.8-8.0 ppm as a singlet (1H). Also the C=O stretching frequency in FT-IR spectrum of the products appears around 1748-1764 cm⁻¹ (Table 2, column 7). For more characterization and verification of the products as shown in Table 2 (column 8), the melting points of the products have been measured and were compared with the literature.⁴⁶⁻⁵⁰

The ion-exchange resin DOWEX(R)50WX4 is insoluble in Ac₂O. Therefore, the reactions take place under heterogeneous conditions. The influences of DOWEX(R)50WX4 and Zn(BH₄)₂ have been shown in Scheme 2. It seems that SO₃H groups on DOWEX(R)50WX4 (as cation-exchange resin, strong acid) protonate the carbonyl group of aldehyde (Scheme 2, step I). Consequently, it is more susceptible for Ac₂O attack (Scheme 2, step II). Also, the hydride attack from the Zn(BH₄)₂ promotes the formation of hydrogen gas which is slowly liberated *in situ* (Scheme 2, step III).

Also, the acylation of cinnamaldehyde with 1 molar equivalent of Zn(BH₄)₂ and Ac₂O (1 mL) in the presence of 0.5 g of DOWEX(R)50WX4 was carried out and the corresponding cinnamyl acylal was obtained within 1 min with 95% yield at room temperature (Table 2, entry 13).

Our attempt for the preparation of *gem*-diacetates from ketones was not successful using this system. Therefore, this system can act as chemoselective system for the acylation of aldehydes over ketones. Thus, we have performed the acylation of 1 molar equivalent of benzaldehyde in the presence of 1 molar equivalent of acetophenone under optimized reaction conditions (Table 3, entry 4) as shown in Scheme 3.

The chemoselectivity ratio for the acylation of benzaldehyde with respect to acetophenone was 100%. The usefulness of chemoselectivity was further examined by the acylation

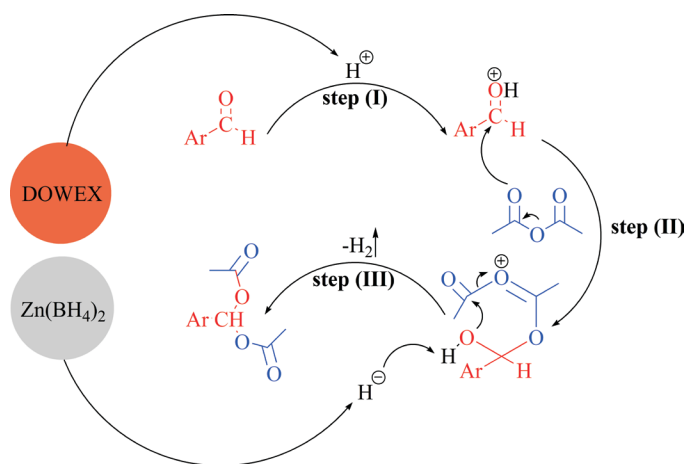
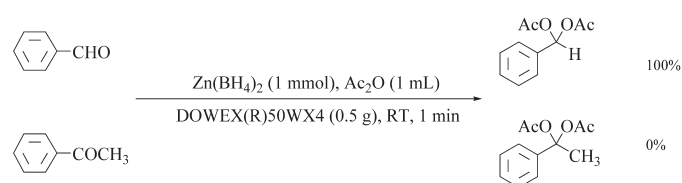
**Scheme 2.** The mechanism for the acylation of aldehydes with Zn(BH₄)₂/Ac₂O/DOWEX(R)50WX4 system.**Scheme 3.** Comparison of the acylation of benzaldehyde and acetophenone based on optimized reaction conditions.

Table 2. The acylation of aldehydes (1 mmol) with Ac₂O (1 mL) and Zn(BH₄)₂ (1 mmol) in the presence of DOWEX(R)50WX4 (0.5 g) at RT.

Entry	Aldehydes	Products	Time/min	Yields/% ^a	δ CH/ppm ^b	$\bar{\nu}$ C=O/cm ⁻¹ ^b	m.p./ °C ^b
1			1	97	7.69	1761	44-45
2			1	94	7.62	1764	94-95
3			1	93	7.54	1760	52-53
4			1	96	7.62	1760	83-84
5			1	96	7.78	1761	64-65
6			1	97	7.67	1763	99-101
7			1	92	7.73	1759	124-125
8			1	95	7.74	1760	66-67
9			5	95	7.61	1748	65-66
10			5	92	8.03	1761	69-70
11			2	93	7.66	1759	80-81
12			1	95	7.70	1763	52-53
13			1	95	7.38	1762	83-85
14			1	93	6.81	1755	oil

^a Yields refer to isolated pure products. ^b The obtained data were compared with the literature.⁴⁶⁻⁵⁰**Table 3.** Competitive acylation of aldehydes (1 mmol) and ketones (1 mmol) with Zn(BH₄)₂ (1 mmol)/Ac₂O (1 mL)/DOWEX(R)50WX4 (0.5 g) system at RT.

Entry	Substrate 1	Substrate 2	Molar Ratio ^a	Time/min	Conversion 1/Conversion 2/% ^b
1			1:1	1	100:0
2			1:1	1	100:0
3			1:1	1	100:0
4			1:1	1	100:0
5			1:1	1	100:0

^a Molar Ratio as: Substrate 1/Substrate 2. ^b Conversions refer to TLC monitoring (eluent; *n*-hexane/EtOAc: 9/1).

of benzaldehyde in the presence of other ketones as shown in Table 3.

The reusability of the catalyst has been checked by using recovered DOWEX(R)50WX4 for the acylation of benzaldehyde under optimized reaction conditions (Table 1, entry 4). We have observed that the recovered DOWEX(R)50WX4 is not convenient for a second run without regeneration. However, after regeneration of DOWEX(R)50WX4 (stirred with HCl 5-10% for 30-60 min and then washed with distilled water), the acylation reaction has been carried out as well as the first run as shown in Table 4.

Experimental

General. All substrates and reagents were purchased from commercial sources with the best quality and used without further purification. IR spectra were recorded on Perkin-Elmer FT-IR RXI and ¹H NMR spectra were determined in a 300 MHz Bruker spectrometer. The products were characterized by their ¹H NMR, ¹³C NMR or IR spectra and by comparison with authentic samples (melting points). All yields referred to isolated pure products. ¹H NMR was applied for the purity determination of products and TLC for reaction monitoring over silica gel 60 F₂₅₄ aluminum sheets.

The acylation of benzaldehyde with Zn(BH₄)₂/DOWEX(R)50WX4/Ac₂O system. A typical procedure

In a round-bottom flask (5 mL) equipped with a magnetic stirrer, a mixture of benzaldehyde (0.106 g, 1 mmol), Ac₂O (1 mL) and DOWEX(R)50WX4 (0.5 g) was treated with Zn(BH₄)₂ (0.095 g, 1 mmol). The resulting reaction mixture was stirred at room temperature. After completion of the reaction within 1 min, the catalyst was filtered and washed with ethyl acetate (15 mL). The combined organic layers were washed with saturated NaHCO₃ solution (3 × 10 mL), water (10 mL) and then dried over anhydrous Na₂SO₄. The solvent was removed on a rotary evaporator under reduced pressure to give 1,1-diacetoxy-1-phenylmethane (0.201 g, 97% yield). The product was characterized by ¹H-NMR, ¹³C-NMR and FT-IR spectroscopy.

Spectral data for selected compounds

1,1-Diacetoxy-1-phenylmethane (Table 1, entry 1): ¹H NMR (CDCl₃): δ 2.14 (s, 6H), 7.41-7.43 (Ar, 3H), 7.52-7.55 (Ar,

2H), 7.69 (s, 1H); ¹³C NMR (CDCl₃): δ 20.85, 89.67, 126.65, 128.58, 129.74, 135.40, 169.79; IR (KBr) ν = 3021, 1761, 1373, 1243, 1216, 1009, 767 cm⁻¹.

1,1-Diacetoxy-1-(4-bromophenyl)methane (Table 1, entry 2): ¹H NMR (CDCl₃): δ 2.11 (s, 6H), 7.37-7.40 (Ar, 2H), 7.51-7.54 (Ar, 2H), 7.62 (s, 1H); ¹³C NMR (CDCl₃): δ 20.78, 89.05, 123.90, 128.40, 131.76, 134.46, 168.64; IR (KBr) ν = 3025, 1764, 1373, 1236, 1208, 1070, 1012, 758 cm⁻¹.

1,1-Diacetoxy-1-(4-nitrophenyl)methane (Table 1, entry 7): ¹H NMR (CDCl₃): δ 2.11 (s, 6H), 7.70 (Ar, 2H), 7.73 (s, 1H), 8.27 (Ar, 2H); ¹³C NMR (CDCl₃): δ 20.74, 88.30, 123.84, 127.86, 141.87, 148.61, 158.55; IR (KBr) ν = 3033, 1759, 1367, 1239, 1208, 1067, 1007, 812 cm⁻¹.

1,1-Diacetoxy-1-(4-methoxyphenyl)methane (Table 1, entry 9): ¹H NMR (CDCl₃): δ 2.06 (s, 6H), 3.75 (s, 3H), 6.70 (Ar, 2H), 7.43 (Ar, 2H), 7.61 (s, 1H); ¹³C NMR (CDCl₃): δ 20.47, 55.08, 89.63, 113.79, 128.01, 131.86, 160.49, 168.72; IR (KBr) ν = 3018, 1748, 1600, 1367, 1259, 1160, 1027, 833 cm⁻¹.

1,1-Diacetoxy-1-(2-methoxyphenyl)methane (Table 1, entry 10): ¹H NMR (CDCl₃): δ 2.10 (s, 6H), 3.82 (s, 3H), 6.90 (Ar, 1H), 6.98 (Ar, 1H), 7.34 (Ar, 1H), 7.49 (Ar, 1H), 8.03 (s, 1H); ¹³C NMR (CDCl₃): δ 20.69, 55.52, 85.54, 110.88, 120.37, 123.72, 126.78, 130.84, 156.89, 168.44; IR (KBr) ν = 3016, 1761, 1602, 1372, 1220, 1005, 764 cm⁻¹.

1,1-Diacetoxy-1-(4-methylphenyl)methane (Table 1, entry 11): ¹H NMR (CDCl₃): δ 2.12 (s, 6H), 2.38 (s, 3H), 7.23 (Ar, 2H), 7.43 (Ar, 2H), 7.66 (s, 1H); ¹³C NMR (CDCl₃): δ 20.86, 21.28, 89.77, 126.60, 129.25, 132.59, 139.78, 168.80; IR (KBr) ν = 3033, 1759, 1367, 1239, 1067, 1039, 812 cm⁻¹.

1,1-Diacetoxy-1-(cinnamyl)methane (Table 1, entry 13): ¹H NMR (CDCl₃): 2.13 (s, 6H), 6.22 (dd, *J* = 15 Hz, 6 Hz, 1H), 6.82 (d, *J* = 15 Hz, 1H), 7.27-7.44 (m, 5H), 7.37 (d, *J* = 6 Hz, 1H); ¹³C NMR (CDCl₃): δ 20.90, 89.71, 121.65, 127.00, 128.66, 128.83, 135.09, 135.60, 168.70; IR (KBr) ν = 3024, 1762, 1373, 1243, 1216, 1001, 756, cm⁻¹.

Conclusions

In this research, we have shown that Zn(BH₄)₂/DOWEX(R)50WX4/Ac₂O system is convenient method for the acylation of a variety of aldehydes to their corresponding *gem*-diacetates in excellent yields. The acylation reactions were carried out with 1 molar equivalents of Zn(BH₄)₂ and Ac₂O (1 mL) in the presence of 0.5 g DOWEX(R)50WX4 at room temperature. High efficiency, shorter reaction times

Table 4. Reusability of DOWEX(R)50WX4 in the preparation of 1,1-diacetoxy-1-phenylmethane from benzaldehyde under optimized conditions (Table 1, Entry 4).

Yields ^c /%	Conversion ^b /100%	Time/min ^a	Run Number	Entry
97	100	1	1	1
30	100<	30	2	2
95	100	1	—	3 ^d

^a It is the highest time when the reaction ends or not further progress.

^b Conversion refer to TLC monitoring (eluent; *n*-hexane/EtOAc: 9/1); ^c Yields refer to isolated pure products. ^d Regeneration by HCl (5-10%).

and easy work-up make to this new protocol attractive for the acylation of aldehydes. Therefore, this new system could be a useful addition to the present methodologies.

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