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New Catalytic Methods for the Preparation of Acetals from Alcohols and Aldehydes

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New Catalytic Methods for the Preparation of Acetals from Alcohols and Aldehydes

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Abstract: The acetalization of aldehydes has been studied with ethyleneglycol, 3-chloro-1,2-propanediol, 1,2-propanediol, and 1,2,3-propanetriol using resin-D_{72} catalyst in high yields under reflux and water separator conditions. The reaction is simple, efficient, and chemoselective and does not involve any other additive.

Keywords: Acetalization, aldehyde, catalyst

INTRODUCTION

Acetals[1,2] are important in synthetic carbohydrate[3–5] and steroid[6] chemistry. In the pharmaceutical,[7] phytopharmaceutical, fragrance,[8] and lacquer[9] industries, acetals are used as intermediates and end products.[10] Acetalization is one of the widely used synthetic methods for protecting aldehydes and

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ketones in the course of the preparation of a variety of multifunctional complex organic molecules.[11] So far, several methods have been developed for this purpose. Generally, acetalization reactions are carried out by treatment of carbonyl compounds with alcohols in the presence of an acid catalyst and proceed with azeotropic removal of water, using Dean–Stark apparatus. Previously, the catalysts used in the acetalization reaction were generally protonic acids, Lewis acids, and a number of transitional metal complexes including Rh, Pd, and Pt.[12–15] Although good results were obtained, the separation of the product from the catalyst system after the reaction was still difficult, and the noble metal catalysts used were quite expensive and usually unstable.[16] Therefore, the development of a catalytic system that is stable, easily separable, and reusable has long been awaited, and the increasing demand for safe industrial processes requires and implementation of ecofriendly, solid, benign catalysts for value-added acid-catalyzed reactions. We are surprise to find, however, resin-D_{72}, a sort of strongly acidic macroporous adsorption cation exchanger–sulfonic acid functionality resin, has little been used as a catalyst in this context. In this article, we report our preliminary findings that this simple, inexpensive, efficient, recyclable, and highly ecofriendly acid catalyst can be successfully used in the acetalization reactions.

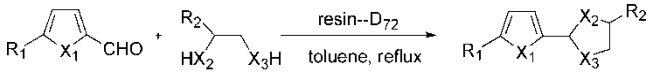
RESULTS AND DISCUSSION

To evaluate the influence of resin-D_{72} on the acetalization reactions, we examined the reaction of furfural with 1,2-propanediol in toluene in the presence of resin-D_{72}, using a water separator. Good results were obtained. Cation-exchange resin-D_{72} is a copolymer of divinylbenzene (DVB) and styrene; sulfonic acid is the active site (Brønsted acidity), which is the same as Amberlyst-15.[17,18] Resin-D_{72}, however, is a sort of commercial domestic product. Unlike Amberlyst-15 (U.S.), it can be obtained easily and cheaply. Unlike dry HCl, corrosive acid HF, saturated H_2SO_4, and *p*-TsOH, it can be recycled, does not erode the apparatus, and separates the products easily, and the reaction does not involve any other additive.

We also successfully carried out the resin-D_{72}-induced acetalization reactions of other aldehydes with glycols or thiols, as shown in Table 1. From the results of Table 1, it was observed that the use of resin-D_{72} as an acid catalyst gives a high yield.

In conclusion, we have demonstrated that the rather cheap, readily available, and user-friendly resin-D_{72} can be used as a new, simple, and efficient acid catalyst for the acetalization reactions. It does not involve additives, and the catalyst can be recycled without loss of activity. In addition, its activity is even superior to that of the most widely used catalyst, *p*-TsOH. Further investigation in optimizing the reaction conditions is being actively pursed in our laboratory.

Table 1. Resin-D_{72}-induced acetalization reactions



1 **2** **3**

Entry	X_1	X_2	X_3	R_1	R_2	Cat.	Time (h)	Product	Yield (%)[a]
1	O	O	O	H	CH_3	D_{72}	5	**3a**	85
2	O	O	O	H	CH_2Cl	D_{72}	5	**3b**	82
3	O	S	O	H	H	D_{72}	5	**3c**	78
4	O	S	S	H	H	D_{72}	5	**3d**	93
5	O	O	O	H	CH_2OH	D_{72}	5	**3e**	90
6	O	O	O	CH_3	H	D_{72}	5	**3f**	87
7	O	O	O	CH_3	CH_3	D_{72}	5	**3g**	83
8	O	O	O	CH_3	CH_2Cl	D_{72}	5	**3h**	73
9	O	S	O	CH_3	H	D_{72}	5	**3i**	78
10	O	S	S	CH_3	H	D_{72}	5	**3j**	90
11	S	O	O	H	H	D_{72}	10	**3k**	89
12	S	O	O	H	CH_3	D_{72}	10	**3l**	85
13	S	O	O	H	CH_2Cl	D_{72}	10	**3m**	80
14	S	S	O	H	H	D_{72}	10	**3n**	84
15	S	S	S	H	H	D_{72}	10	**3o**	91

[a]Isolated yield based on aldehyde.

EXPERIMENTAL

The IR spectra were determined on a Bio-Rad Win-IR infrared spectrometer by dispersing samples in KBr disks. ^{1}H NMR spectra were measured on a Bruker DPX300 NMR spectrometer with $CDCl_3$ as solvent. Resin-D_{72} was purchased from the Chemical Plant of Nankai University. Analytical thin-layer chromatography (TLC) was performed on precoated plates purchased from Qingdao Haiyang Chemical Co., Ltd. (silica GF254) using UV light. Partial products are already reported in the literature.[19–21] Partial commercial available organic compounds were used with further purification, and solvents for the reactions were dried on 4-Å molecular sieves before use.

General Procedure

In a general reaction procedure, a mixture of carbonyl compounds (10 mmol); protecting groups glycols or thiols such as ethyleneglycol, 3-chloro-1,2--propanediol, 1,2-propanediol, 1,2,3-propanetriol, and 1-hydroxyethane-2-thiol (11 mmol) and resin-D_{72} 10% (w/w) in toluene (25 mL) was refluxed for 5–10 h, using a water separator. After the reaction was complete (TLC), the

resin-D_{72} was separated by filtration and could be reused for the next run after simple treatment. The toluene was removed under reduced pressure. The product was obtained by distilling the residue under oil-pump reduced pressure.

2-Furan-2-yl-4-methyl-[1,3]dioxolane (3a). This compound was prepared in the same manner as that described previously. This pure product (**3a**, isomers) is a colorless liquid: 69–70°C/1.73 KPa, $C_8H_{10}O_3$. IR (neat): ν (cm^{-1}) 3124, 2980, 2883, 1604, 1503, 1377, 1357, 1254, 1225, 1156, 1095, 1070, 1067, 746. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.32 (d, $J = 6.12$ Hz, 3H, OCHCH_3), 3.50 (m, 1H, OCH$_a$H$_b$), 4.09 (m, 1H, OCH_aH$_b$), 4.29 (m, 1H, OCHCH$_3$), 5.89 (s, 1H, CCHO$_2$), 6.34 (m, 1H, CHCHO), 6.42 (**m**, 1H, CHCO), 7.41 (m, 1H, CHCHO); 1.38 (d, $J = 6.03$ Hz, 3H, OCHCH_3), 3.60 (m, 1H, OCH$_a$H$_b$), 4.25 (m, 1H, OCH_aH$_b$), 4.42 (m, 1H, CHCH$_3$O), 6.02 (s, 1H, CCHO$_2$), 6.34 (m, 1H, CHCHO), 6.47 (**m**, 1H, CHCO), 7.41 (m, 1H, CHCHO) ppm.

4-Chloromethyl-2-furan-2-yl-[1,3]dioxolane (3b). This compound was prepared in the same manner as that described previously. This pure product (**3b**, isomers) is a pale yellow liquid: 129–131°C/1.86 KPa, $C_8H_9ClO_3$. IR (neat): ν (cm^{-1}) 3126, 2958, 2889, 1606, 1507, 1361, 1257, 1232, 1154, 1094, 1013, 960, 747. ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.56 (m, 1H, CH$_a$H$_b$Cl), 3.67 (m, 1H, CH_aH$_b$Cl), 3.94 (m, 1H, OCH$_a$H$_b$), 4.12 (m, 1H, OCH_aH$_b$), 4.40 (m, 1H, OCHCH$_2$Cl), 5.91 (s, 1H, CCHO$_2$), 6.35 (m, 1H, CHCHCO), 6.43 (**m**, 1H, CHCO), 7.42 (m, 1H, CHCHO); 3.60 (m, 1H, CH$_a$H$_b$Cl), 3.69 (m, 1H, CH_aH$_b$Cl), 4.10 (m, 1H, OCH$_a$H$_b$), 4.28 (m, 1H, OCH_aH$_b$), 4.52 (m, 1H, OCHCH$_2$Cl), 6.07 (s, 1H, CCHO$_2$), 6.37 (m, 1H, CHCHCO), 6.49 (**m**, 1H, CHCO), 7.42 (m, 1H, CHCHO) ppm.

2-Furan-2-yl-[1,3]oxathiolane (3c). This compound was prepared in the same manner as that described previously. This pure product (**3c**) is a yellow liquid: 85–88 °C/1.33 KPa, $C_7H_8O_2S$. IR (neat): ν (cm^{-1}) 3121, 2942, 2875, 1723, 1501, 1232, 1152, 1055, 1013, 968, 743. ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.20 (m, 2H, SCH_2CH$_2$), 4.15 (m, 2H, OCH$_2CH_2$), 6.11 (s, 1H, CHSO), 6.33 (m, 1H, CHCO), 6.45 (m, 1H, CHCHO), 7.42 (m, 1H, CHCHO) ppm.

2-[1,3]Dithiolan-2-yl-furan (3d). This compound was prepared in the same manner as that described previously. This pure product (**3d**) is an aurantium liquid: 118–120 °C/0.66 KPa, $C_7H_8OS_2$. IR (neat): ν (cm^{-1}) 3117, 2925, 1585, 1499, 1421, 1276, 1171, 1147, 1010, 936, 851, 739. ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.26–3.33 (m, 2H, SCH_2CH$_2$S), 3.38–3.46 (m, 2H, SCH$_2CH_2$S), 5.62 (s, 1H, CHS$_2$), 6.27–6.30 (m, 2H, OCH$CHCH$C), 7.36 (m, 1H, OCHCHCH) ppm.

(2-Furan-2-yl-[1,3]dioxolan-4-yl)-methanol (3e). This compound was prepared in the same manner as that described previously. This pure product (**3e**, isomers) is a colorless liquid: 162–164°C/1.20 KPa, $C_8H_{10}O_4$.

IR (neat): ν (cm^{-1}) 3437, 3126, 2931, 2889, 1723, 1504, 1363, 1230, 1155, 1097, 884, 749. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.45 (s, 1H, *OH*), 3.63–3.68 (m, 5H, CH_2CHCH_2OH), 5.90 (s, 1H, CHO_2), 6.36 (m, 1H, OCH*CH*CHC), 6.45 (m, 1H, OCHCH*CH*C), 7.43 (m, 1H, O*CH*CHCHC), 2.46 (s, 1H, *OH*), 4.04–4.23 (m, 5H, CH_2CHCH_2OH), 6.04 (s, 1H, CHO_2), 6.37 (m, 1H, OCH*CH*CHC), 6.50 (m, 1H, OCHCH*CH*C), 7.45 (m, 1H, O*CH*CHCHC) ppm.

2-(5-Methyl-furan-2-yl)-[1,3]dioxolane (3f). This compound was prepared in the same manner as that described previously. This pure product (**3f**) is a colorless liquid: 74–75°C/0.80 KPa, $C_8H_{10}O_3$. IR (neat): ν (cm^{-1}) 3111, 2955, 2890, 1726, 1616, 1565, 1383, 1349, 1219, 1110, 945, 791. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.30 (s, 3H, CH_3CO), 3.97–4.05 (m, 2H, OCH_2CH_2O), 4.12–4.16 (m, 2H, OCH_2CH_2O), 5.84 (s, 1H, CHO_2), 5.93 (m, 1H, CH_3CCHCH), 6.33 (d, $J = 3.12$ Hz, 1H, CH_3CCHCH) ppm.

4-Methyl-2-(5-methyl-furan-2-yl)-[1,3]dioxolane (3g). This compound was prepared in the same manner as that described previously. This pure product (**3g**, isomers) is a yellow liquid: 97–99°C/0.80 KPa, $C_9H_{12}O_3$. IR (neat): ν (cm^{-1}) 3438, 2981, 2938, 1724, 1678, 1565, 1521, 1382, 1187, 967, 794. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.32 (d, $J = 6.12$ Hz, 3H, $OCHCH_3$), 2.30 (s, 3H, CH_3CO), 3.50 (m, 1H, OCH_aH_b), 4.09 (m, 1H, OCH_aH_b), 4.28 (m, 1H, $OCHCH_3$), 5.82 (s, 1H, $CCHO_2$), 5.93 [m, 1H, $CHC(CH_3)O$], 6.30 (d, $J = 3.09$ Hz, 1H, *CH*CO), 1.39 (d, $J = 6.06$ Hz, 3H, $OCHCH_3$), 2.30 (s, 3H, CH_3CO), 3.61 (m, 1H, OCH_aH_b), 4.23 (m, 1H, OCH_aH_b), 4.42 (m, 1H, $OCHCH_3$), 5.95 (s, 1H, $CCHO_2$), 5.91 [m, 1H, $CHC(CH_3)O$], 6.35 (d, $J = 3.12$ Hz, 1H, *CH*CO) ppm.

4-Chloromethyl-2-(5-methyl-furan-2-yl)-[1,3]dioxolane (3h). This compound was prepared in the same manner as that described previously. This pure product (**3h**, isomers) is a pale yellow liquid: 129–131°C/1.86 KPa, $C_9H_{11}ClO_3$. IR (neat): ν (cm^{-1}) 3492, 2957, 2887, 1728, 1565, 1431, 1351, 1220, 1174, 1093, 1021, 946, 792. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.29 (m, 3H, CH_3C), 3.56 (m, 1H, CH_aH_bCl), 3.64 (m, 1H, CH_aH_bCl), 3.93 (m, 1H, OCH_aH_b), 4.12 (m, 1H, OCH_aH_b), 4.30 (m, 1H, $OCHCH_2Cl$), 5.84 (s, 1H, $CCHO_2$), 5.93 (m, 1H, *CH*CHCO), 6.32 (**m**, 1H, *CH*CO); 2.30 (m, 3H, CH_3C), 3.60 (m, 1H, CH_aH_bCl), 3.67 (m, 1H, CH_aH_bCl), 4.09 (m, 1H, OCH_aH_b), 4.30 (m, 1H, OCH_aH_b), 4.52 (m, 1H, $OCHCH_2Cl$), 5.99 (s, 1H, $CCHO_2$), 5.95 (m, 1H, *CH*CHCO), 6.38 (**m**, 1H, *CH*CO) ppm.

2-(5-Methyl-furan-2-yl)-[1,3]oxathiolane (3i). This compound was prepared in the same manner as that described previously. This pure product (**3i**) is an aurantium liquid: 120–121 °C/1.20 KPa, $C_8H_{10}O_2S$. IR (neat): ν (cm^{-1}) 2943, 2873, 1559, 1385, 1222, 1162, 1056, 1021, 966, 947, 788. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.29 (s, 3H, CH_3CO), 3.07–3.27 (m, 2H, SCH_2CH_2), 3.98–4.34 (m, 2H, OCH_2CH_2), 6.05 (s, 1H, *CH*SO), 5.92 [m, 1H, $CHC(CH_3)O$], 6.34 (m, 1H, *CH*CO) ppm.

2-[1,3]Dithiolan-2-yl-5-,ethyl-furan (3j). This compound was prepared in the same manner as that described previously. This pure product (**3j**) is an aurantium liquid: 132–133 °C/0.80 KPa, $C_8H_{10}OS_2$. IR (neat): ν (cm^{-1}) 3105, 2923, 1607, 1556, 1422, 1277, 1217, 1121, 1021, 997, 965, 789. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.27 (s, 3H, CH_3CO), 3.26–3.34 (m, 2H, SCH_2CH_2S), 3.38–3.46 (m, 2H, SCH_2CH_2S), 5.59 (s, 1H, CHS_2), 5.86 (m, 1H, CH_3CCHCH), 6.17 (d, 1H, CH_3CCHCH) ppm.

2-Thiophen-2-yl-[1,3]dioxolane (3k). This compound was prepared in the same manner as that described previously. This pure product (**3k**) is a colorless liquid: 88–89 °C/0.66 KPa, $C_7H_8O_2S$. IR (neat): ν (cm^{-1}) 3106, 2969, 2889, 1674, 1545, 1442, 1391, 1331, 1086, 943, 838, 712. ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.96–4.15 (m, 4H, OCH_2CH_2O), 6.11 (s, 1H, CHO_2), 6.98 (m, 1H, CHCHCHC), 7.16 (m, 1H, CHCHCHC), 7.3 (m, 1H, SCHCHCHC) ppm.

4-Methyl-2-thiophen-2-yl-[1,3]dioxolane (3l). This compound was prepared in the same manner as that described previously. This pure product (**3l**, isomers) is a colorless liquid: 131–133°C/0.66 KPa, $C_8H_{10}O_2S$. IR (neat): ν (cm^{-1}) 3107, 2977, 2932, 2880, 1545, 1444, 1382, 1330, 1212, 1089, 1002, 971, 837, 711. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.33 (d, 3H, $J = 5.97$ Hz, OCH_aCH_3), 3.50 (m, 1H, OCH_aH_b), 4.10 (m, 1H, OCH_aH_b), 4.33 (m, 1H, $OCHCH_3$), 6.10 (s, 1H, $CCHO_2$), 6.98 (m, 1H, CHCHO), 7.16 (**m**, 1H, CHCS), 7.33 (m, 1H, CHCHO), 1.39 (d, $J = 5.94$ Hz, 3H, $OCHCH_3$), 3.62 (m, 1H, OCH_aH_b), 4.32 (m, 1H, OCH_aH_b), 4.42 (m, 1H, $OCHCH_3$), 6.24 (s, 1H, $CCHO_2$), 7.00 (m, 1H, CHCHO), 7.18 (**m**, 1H, CHCS), 7.35 (m, 1H, CHCHO) ppm.

4-Chloromethyl-2-yl-[1,3]dioxolane (3m). This compound was prepared in the same manner as that described previously. This pure product (**3m**, isomer) is a pale yellow liquid: 110–112 °C/0.40 KPa, $C_8H_9ClO_2S$. IR (neat): ν (cm^{-1}) 3108, 2955, 2886, 1545, 1442, 1394, 1332, 1214, 958, 844, 713. ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.53–3.72 (m, 2H, CH_aH_bCl), 3.93 (m, 1H, OCH_aH_b), 4.12 (m, 1H, OCH_aH_b), 4.32 (m, 1H, $OCHCH_2Cl$), 6.11 (s, 1H, $CCHO_2$), 6.99 (m, 1H, CHCHCS), 7.18 (d, $J = 0.873$ Hz 1H, CHCS), 7.35 (m, 1H, CHCHS), 3.53–3.72 (m, 2H, CH_aH_bCl), 4.14 (m, 1H, OCH_aH_b), 4.30 (m, 1H, OCH_aH_b), 4.50 (m, 1H, $OCHCH_2Cl$), 6.28 (s, 1H, $CCHO_2$), 7.00 (m, 1H, CHCHCS), 7.20 (d, $J = 0.927$ Hz 1H, CHCS), 7.36 (m, 1H, CHCHS) ppm.

2-Thiophen-2-yl-[1,3]oxathiolane (3n). This compound was prepared in the same manner as that described previously. This pure product (**3n**) is a pale yellow liquid: 108–110°C/0.40 KPa, $C_7H_8OS_2$. IR (neat): ν (cm^{-1}) 3104, 2974, 2941, 2872, 1708, 1438, 1372, 1315, 1266, 1057, 963, 855, 709. ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.17 (m, 1H, $SCH_aH_bCH_2$), 3.30 (m, 1H, $SCH_aH_bCH_2$), 3.98 (m, 1H, $OCH_aH_bCH_2$), 4.41 (m, 1H, $OCH_aH_bCH_2$), 5.31 (s, 1H, CHSO), 6.95 (m, 1H, CHCS), 7.14 (m, 1H, CHCHS), 7.31 (m, 1H, CHCHS) ppm.

2-Thiophen-2-yl-[1,3]dithiolane (3o). This compound was prepared in the same manner as that described previously. This pure product (**3o**) is a colorless liquid: 131–133 °C/0.40 KPa, $C_7H_8S_3$. IR (neat): ν (cm^{-1}) 3101, 3070, 2922, 1430, 1270, 1221, 1118, 1085, 978, 853, 747. ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.28–3.38 (m, 2H, S*CH*$_2$CH$_2$S), 3.43–3.52 (m, 2H, SCH$_2$*CH*$_2$S), 5.92 (s, 1H, *CH*S$_2$), 6.87–6.89 (m, 1H, SCHCH*CH*C), 7.06 (m, 1H, SCH*CH*CHC), 7.23 (m, 1H, S*CH*CHCH) ppm.

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