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Ferric Hydrogensulfate as Effective and Recyclable Catalyst for Mild Dithioacetalization of Aldehydes and Ketones

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Ferric hydrogensulfate has been found to be an extremely efficient and recyclable heterogeneous catalyst for dithioacetalization reactions. Carbonyl compounds have been successfully converted into their corresponding dithiolanes and dithianes derivatives in excellent yields at ambient conditions. The synthetic simple procedures reported in this paper constitute an exceptionally mild procedure for selective carbonyl protection.

Keywords 1,3-dithiane; 1,3-dithiolane; dithioacetalization; $\text{Fe}(\text{HSO}_4)_3$; heterogeneous catalyst

INTRODUCTION

Thioacetalization of carbonyl compounds plays an important role in organic synthesis.¹ Since the introduction of 1,3-dithianes as nucleophilic acylating reagents by Corey and Seebach, dithioacetals have become widely used tools for C–C bond formation.² Furthermore, the stability exhibited by cyclic and acyclic dithioacetal under acidic and basic conditions has led to their synthetic utility as carbonyl protecting groups and as intermediates for the conversion of carbonyl compounds to their parent hydrocarbons.^{3,4} Several Brønsted and Lewis acid catalysts such as: $\text{Sc}(\text{OTf})_3$,⁵ InCl_3 ,⁶ iodine supported on natural phosphate⁷ CoCl_2 ,⁸ NiCl_2 ,⁹ NBS ,¹⁰ $\text{In}(\text{OTf})_3$,¹¹ $\text{MoO}_2(\text{acac})_2$,¹² $[\text{bmim}]\text{HSO}_4$,¹³ *p*-dodecyl benzenesulfonic acid (DBSA),¹⁴ TiCl_4 ,¹⁵ LaCl_3 ,¹⁶ hydrobromic

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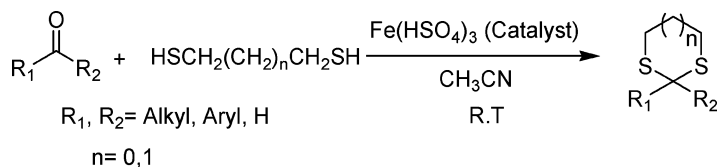
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acid,¹⁷ ZnCl_2 ,¹⁸ $\text{Y}(\text{OTf})_3$ ¹⁹ Iron(III) fluoride,²⁰ and $\text{VO}(\text{OTf})_2$ ²¹ have been used in thioacetalization reactions. Many of these methods require long reaction times, reflux conditions, stoichiometric amounts of catalyst, unwanted side products and show low selectivity when applied to the mixtures of aldehydes and ketones. Although some Lewis acid catalysts have been reported to show chemoselectivity, these catalysts are destroyed in the work-up procedure and cannot be recovered and reused.¹⁴ Some methods failed to protect deactivated aromatic substrates. Therefore, there is still a need to develop a simple and efficient method for dithioacetalization of carbonyl groups and chemoselective protection of aldehydes. In this research, ferric (III) hydrogensulfate as the solid heterogeneous acid catalyst shows special mention and also can be recovered and reused again. This catalyst is safe, easy to handle, environmentally benign, presents fewer disposal problems and is stable in reaction media. Ferric hydrogensulfate has been used in some organic reactions, such as Schmidt reaction of ketones to amides²² and Friedel-Crafts acylation of alkoxy benzenes by aliphatic anhydrides.²³

RESULTS AND DISCUSSION

Recent progress in the catalysis by Brønsted and Lewis acids is worthy of note. Among these, heterogeneous catalysts have been attracting much attention, because they offer a viable approach to the preparation of invaluable organic compounds.²⁴ These catalysts have been used in high demand in terms of minimizing waste generation side product and provide environmentally benign processes.

In continuation of our interest to develop protection and deprotection of carbonyl groups,²⁵ herein, we wish to report an efficient method for the dithioacetalization of carbonyl compounds in good to high yields, as well as the chemoselective protection of various carbonyl compounds by employing $\text{Fe}(\text{HSO}_4)_3$ as active heterogeneous catalyst (Scheme 1). We believe that our method is very simple, mild, rapid and new to the literature.



SCHEME 1

TABLE I Conversion of Benzaldehyde (1mmol) to 2-Phenyl-1,3-dithiane Using 1,3-Propanedithiol (1.05 mmol) in the Presence of Different Amounts of Solid $\text{Fe}(\text{HSO}_4)_3$ as a Catalyst under a Variety of Solvents (2 mL) at Room Temperature

Entry	Solvent	Catalyst (mol %)	Time (min)	GC Yield (%)
1	Chloroform	10	25	100
2	Dichloromethane	10	20	100
3	Ethylacetate	10	15	100
4	n-Hexane	10	40	100
5	Diethyl ether	10	20	100
6	Acetonitrile	10	2	100
7	Acetonitrile	20	Immediately	100
8	Acetonitrile	5	8	96
9	Acetonitrile	2	20	90
10	Acetonitrile	1	60	85
11	Acetonitrile	0	24 h	—

First, we tried to convert benzaldehyde (1 mmol) to its corresponding 2-phenyl-1,3-dithiane with various amounts of catalyst in the presence of various solvents at room temperature (Table I). The results in Table I show that amongst these solvents, acetonitrile was the solvent of choice in terms of time and product yield and the quantity of $\text{Fe}(\text{HSO}_4)_3$ was chosen to be 10 mol%.

Next, we prepared a range of cyclic dithioacetals under the following reaction conditions: aldehydes or ketones (1 eq), 1,3-propanedithiol or 1,2-ethanedithiol (1.05 eq) and acetonitrile (as solvent, 2 mL) in the presence of $\text{Fe}(\text{HSO}_4)_3$ (10 mol%) (Table II).

As shown in Table II, several different aliphatic, aromatic aldehydes with both electron-withdrawing and donating substituents produced corresponding cyclic dithioacetal without the formation of any side products, in high to excellent yields within a few minutes at room temperature (Table II, entries 1–12). The reaction was performed with benzaldehyde containing withdrawing as well as electron donating groups, but benzaldehydes with electron-donating groups are generally more reactive than their corresponding benzaldehydes with electron-withdrawing groups and provided the desired product at short reaction time with excellent yield (Table II, entries 3–10). As reported in the literature,²⁶ this observation clearly shows that the reactivity of benzaldehydes in thioacetalization reaction is more strongly affected by the electronic factors. We also performed dithioacetalization reactions with aromatic and aliphatic ketones at room temperature (Table II, entries 13–19). The reaction of acetophenone with 1,3-propanedithiol

TABLE II Conversion of Aldehydes and Ketones to the Corresponding 1,3-Dithiane and 1,3-Dithiolane Using $\text{Fe}(\text{HSO}_4)_3$ as Catalyst in Acetonitrile as a Solvent at Room Temperature









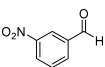
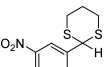
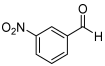
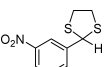
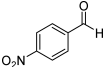
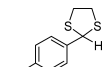
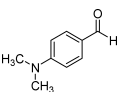
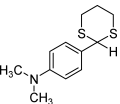
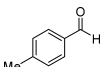

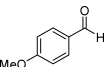
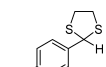
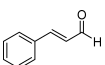
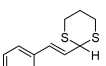
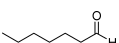
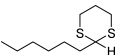










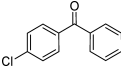
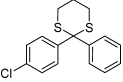


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3			7	95
4			10	96
5			20	94
6			24	91
7			24	93
8			7	93
9			5	95
10			3	97
11			2	89
12			4	97
13			10	94 ^c
14			9	99

TABLE II Conversion of Aldehydes and Ketones to the Corresponding 1,3-Dithiane and 1,3-Dithiolane Using $\text{Fe}(\text{HSO}_4)_3$ as s Catalyst in Acetonitrile as a Solvent at Room Temperature (Continued)

Entry	Substrate	Product	Time (min)	Yield ^a (%)
15			10	96
16			10	97
17			10	90
18			8	91
19			24h	0.0

^aYields refer to the pure isolated products. All known products have been reported previously in the literature and were characterized by comparison of IR and ^1H NMR spectra with authentic samples.^{5–21};


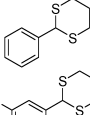
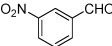
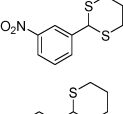

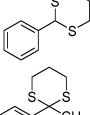



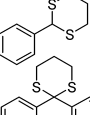
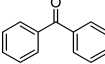
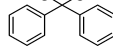
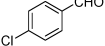
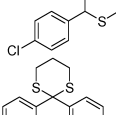
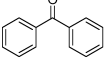
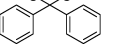
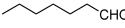
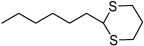


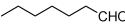
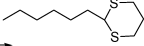
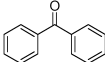
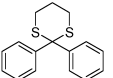
^bIsolated yield after third cycle with reused catalyst. ^cThe reaction was performed at 50°C.

was not completed after 24 h at room temperature (24 h, 60% GC yield), thus we performed this reaction at 50°C in oil bath (10 min, 100% GC yield) (Table II, entry 13). The condensation reaction of 9H-fluoren-9-one with dithiols was not successful in the reaction conditions (Table II, Entry 19).

In a typical experiment, after the reaction was completed, in the work-up stage, acetonitrile was evaporated from the reaction mixture. The reaction mixture was combined with chloroform, the ferric hydrogensulfate as catalyst was isolated from the reaction mixture by simple filtration. The purified catalyst was achieved by washing the solid residue catalyst by ethyl acetate followed by drying in an oven at 100°C for 30 min. In every experiment, more than 98% of the ferric hydrogensulfate was easily recovered from the reaction mixture. Catalytic activity of the recovered catalyst was tested and showed activity same as ferric hydrogensulfate that it was used in the first time.

In order to show the ability of ferric hydrogensulfate to act as selective catalyst, we studied dithioacetalization of aldehydes and ketones with 1,3-propanedithiol (Table III). By this method, benzaldehyde was

TABLE III Selective Dithioacetalization of Different Aldehydes and Ketones in the Presence $\text{Fe}(\text{HSO}_4)_3$ as Catalyst Using 1,3-Propanedithiol

Entry	Carbonyl compound	Product	Time (min)	Yield ^{a,b} (%)
1			10	90
				10
2			10	100
				0
3			10	100
				0
4			10	100
				0
5			10	100
				0
6			10	100
				0

^a GC yield using n-octane as internal standard. ^bThe molar ratio of substrate1/su-
bstyrate2 /1,3-propanedithiol/ $\text{Fe}(\text{HSO}_4)_3$ was chosen 1/1/1.05/0.1.

protected in the presence of 3-nitrobenzaldehyde with the ration of 90/10 (Table III, entry 1). Benzaldehyde in the presence of acetophenone and benzophenone was exclusively protected (Table III, entries 2, 3). Heptanal was quantitatively converted to its S, S-cyclic acetal, whereas acetophenone and benzophenone remained intact (Table III, entries 5, 6). Thus, the excellent chemoselectivity observed for dithioacetalization of aldehydes and ketones make the presented method useful contribution in organic synthesis.

In conclusion, we have demonstrated that ferric hydrogensulfate is a new, efficient, mild and heterogeneous catalyst for dithioacetalization of various aldehydes and ketones at room temperature. This simple and efficient method is a chemoselective procedure for protection of aldehydes in the presence of ketones. This synthetic mythology is important from an environmental point of view and economic considerations, because it produces little waste. Ferric hydrogensulfate has excellent activity and can be recovered from reaction mixtures and reused. Greener conditions, simple work-up procedure, high yields and short reaction times are other advantages of this method.

EXPERIMENTAL

All reagents were purchased from Merck and Aldrich and used without further purification. Ferric hydrogensulfate was prepared according to the reported procedure.^{22,23} All yields refer to isolated products after purification. Products were characterized by comparison of spectroscopic data (IR, ¹H NMR spectra) with authentic samples. The NMR spectra were recorded on a Bruker Avance DEX 400 MHz instrument. IR spectra were recorded on a Perkin-Elmer 781 spectrophotometer. TLC was performed on Silica-gel polygram SIL G/UV 254 plates.

Preparation of Ferric Hydrogensulfate²²

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 ferric chloride (10 mmol) was charged into the flask and concentrated sulfuric acid 98% (30 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°C, while the residual HCl was eliminated by suction. Finally, a pale-brown solid Fe(HSO₄)₃ was obtained.²²

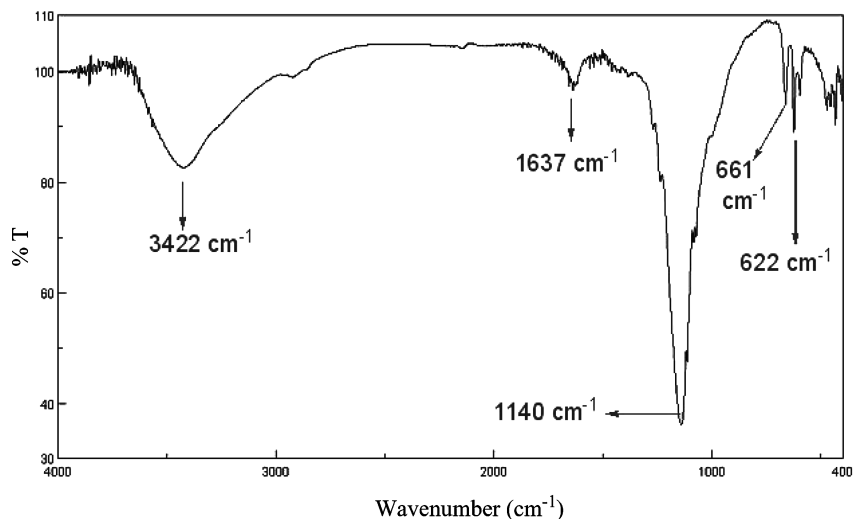


FIGURE 1 FT-IR spectrum of $\text{Fe}(\text{HSO}_4)_3$.

FT-IR Spectrum of $\text{Fe}(\text{HSO}_4)_3$

The FT-IR spectrum of the catalyst was shown in Figure 1. The catalyst is solid and solid-state IR spectrum was recorded using the KBr disk technique. The spectrum shows a broad OH stretching absorption around 3500 and 3100 cm^{-1} . For sulfuric acid functional group in $\text{Fe}(\text{HSO}_4)_3$, the FTIR absorption of the O=S=O stretching modes lies in 1113 cm^{-1} , and that of the S—O stretching mode lies in 600–700 cm^{-1} .

General Procedure for the Dithioacetalization of Carbonyl Compounds

To a mixture of a carbonyl compound (1 mmol) and 1,3-propanedithiol or 1,2-ethanedithiol (1.05 mmol) in acetonitrile (2 mL) was added 10 mol% of $\text{Fe}(\text{HSO}_4)_3$. The reaction mixture was stirred at room temperature for time indicated in Table II. The progress of the reaction was monitored by TLC or GC. After completion of the reaction, acetonitrile was evaporated from the reaction mixture. The reaction mixture was combined with chloroform and filtered. The catalyst washed with chloroform and recovered catalyst can be used again. The organic layer was extracted with 10% sodium hydroxide solution (10 ml) followed by water (10 ml) and brine solution (5 ml). Organic layer dried with anhydrous CaCl_2 and filtered, solvent evaporated under reduced pressure and the pure product was obtained. The desired pure product(s)

was characterized by comparison of their physical data with those of known the dithioacetals.^{5–21} The spectral data of some representative dithioacetals are given below.

2-Phenyl-1,3-dithiane (Table II, entry 1)

¹H NMR (CDCl₃, 400 MHz): δ = 1.92–1.96 (1H, m), 2.15–2.17 (1H, m), 2.88–3.10 (4H, m), 5.17 (1H, s), 7.29–7.48 (5H, m) ppm; IR (KBr, cm⁻¹): 3063, 3022, 2929, 2894, 1685, 1598, 1487, 1445, 1429, 1276, 1240, 1158, 1071, 845, 698.

2-Phenyl-1,3-dithiolane (Table II, Entry 2)

¹H NMR (CDCl₃, 400 MHz): δ = 2.32 (1H, s), 3.29–3.52 (4H, m), 5.62 (1H, s), 7.10 (2H, d, J = 6.5 Hz), 7.40 (2H, d, J = 6.5 Hz) ppm; IR (KBr, cm⁻¹): 3004, 1510, 1437, 1411, 1277, 1177, 1165, 830, 777.

3> 2-(4-Chlorophenyl)-1,3-dithiane (Table II, entry 3)

¹H NMR (CDCl₃, 400 MHz): δ = 1.89–1.94 (1H, m), 2.87–2.94 (2H, m), 3.00–3.09 (2H, m), 2.87–3.09 (4H, m), 5.13 (1H, s), 7.30 (2H, d, J = 9.0 Hz), 7.40 (2H, d, J = 9.0 Hz) ppm; IR (KBr, cm⁻¹): 3081, 3036, 2901, 1912, 1490, 1425, 1276, 1174, 1115, 1088, 1016, 885, 854, 819, 788, 761, 675, 513.

2-(3-Nitrophenyl)-1,3-dithiane (Table II, entry 5)

¹H NMR (400 MHz, CDCl₃): δ = 1.75–2.05 (m, 1H), 2.10–2.25 (m, 1H), 2.80–3.15 (m, 4H) 5.20 (s, 1H), 7.49 (1H, t, J = 8 Hz), 7.77 (1H, d, J = 8 Hz), 8.13 (1H, d, J = 8 Hz), 8.31 (s, 1H) ppm; IR (KBr, cm⁻¹): 3059, 2942, 1533, 1477, 1440, 1353, 1269, 1186, 1086, 931, 877, 815, 774, 728, 696, 447.

2-(4-N,N-dimethylphenyl)-1,3-dithiane (Table II, entry 8)

¹H NMR (CDCl₃, 400 MHz): δ = 1.88–1.91(1H, m), 2.12–2.17 (1H, m), 2.87–3.08 (4H, m), 2.94 (6H, s), 5.11 (1H, s), 6.67 (2H, d, J = 8.8 Hz), 7.33(2H, d, J = 8.8Hz) ppm; IR (KBr, cm⁻¹): 3066, 2949, 2898, 2801, 1608, 1520, 1349, 765.

2-(2-Phenyl ethenyl)- 1,3-dithiane (Table II, entry 11)

¹H NMR (CDCl₃, 400 MHz): δ = 1.89–1.95 (1H, m), 2.11–2.16 (1H, m), 2.83–2.99 (4H, m), 4.82 (1H, d, J = 7.6 Hz), 6.26 (1H, dd, J = 16.0 and 7.6 Hz), 6.76 (1H, d, J = 16.0 Hz), 7.30–7.40 (5H, m) ppm; IR (KBr, cm⁻¹): 3066, 2941, 1677, 1649, 1616, 1579, 1496, 1451, 1424, 1275, 1246, 1206, 1172, 1044, 964, 911, 869, 767, 896, 677, 509.

2-Methyl-2-phenyl-1,3-dithiane (Table II, entry 13)

¹H NMR (CDCl₃, 400 MHz): δ = 1.80 (1H, s), 1.94–1.98 (2H, m), 2.72–2.75 (4H, m), 7.20–7.40 (3H, m), 7.94–7.96 (2H, m) ppm; IR (KBr, cm⁻¹): 3059, 2905, 2362, 1645, 1489, 1446, 1370, 1511, 1278, 1254, 1181, 1067, 1029, 909, 869, 766, 701, 625, 554.

1,5-Dithiaspiro[5.5] undecane (Table II, entry 14)

¹H NMR (CDCl₃, 400 MHz): δ = 1.25–1.45 (2H, m), 1.58–1.66 (4H, m), 1.96–2.00 (6H, m), 2.80–2.82 (4H, m) ppm; IR (KBr, cm⁻¹): 2936, 2851, 1645, 1443, 1352, 1317, 1275, 1243, 1190, 1132, 1011, 824, 769, 559.

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