Oxidation of Thiol Using Ionic Liquid-Supported Organotelluride as a Recyclable Catalyst

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Abstract: Organotellurium compounds are known to be useful oxidation reagents. For developing a recoverable and reusable reagent, this paper describes the use of an ionic liquid (IL) support for the organotellurium reagent and its application as a recyclable catalyst for thiol oxidation. We have successfully prepared a novel diphenyl telluride derivative **5** bearing an imidazolium hexafluorophosphate group in its structure. It is found that the IL-supported diphenyl telluride **5** efficiently catalyzed the aerobic oxidation of various thiols in [bmim]PF₆ solution under photosensitized conditions to provide the corresponding disulfides in excellent yields. The product can be isolated by simple ether extraction. The IL-supported catalyst **5** remaining in the ionic liquid phase can be reused for five successive runs while retaining high catalytic activity (97% yield even in the fifth run).

Keywords: ionic liquid; recyclable catalyst; organotellurium compound; aerobic oxidation; thiol; photosensitized oxygenation

1. Introduction

Recently, organotellurium oxides have been recognized as useful oxidation reagents [1]. In particular, diaryl telluroxides, tellurones, and aryltellurinic acid derivatives have been identified as versatile and effective oxidants for alcohols, phosphines, thiols, thiocarbonyl compounds, and so on [2–11]. Generally, these oxidation reactions require stoichiometric amounts of organotellurium reagents. From synthetic, economic, and environmental perspectives, the development of a catalytic process is desirable. Herein, we have developed organotelluride-catalyzed oxidation of phosphites to phosphates [12], silanes to silanols [13], and thiols to disulfides [14] while employing aerobic oxygen as a terminal oxidant under photosensitized conditions, where the in situ generation of tellurium oxide species by singlet oxygen oxidation is expected. However, the protocol is not without disadvantages, for instance, the product requires isolation by chromatographic purification, and the organotelluride catalyst is rarely reusable. To overcome these issues, we envisioned to immobilize the organotelluride catalyst on an ionic liquid (IL) support.

IL-supported organic synthesis and catalysis have been extensively studied in recent years [15]. Due to its high polarity, the IL support offers the advantages of easy product isolation and catalyst recycling via simple phase separation. We have previously reported the synthesis of IL-supported 18-crown-6 ether [16], ascorbate-based IL [17], and IL-supported benzyl chloride [18] for Huisgen click chemistry, IL-supported hypervalent iodine reagent [19] for alcohol oxidation, and IL-supported 1,3-dimethylimidazolidin-2-one for halogenation [20]. Herein, we describe the synthesis of IL-supported

diphenyl telluride as a recoverable and reusable oxidation catalyst. The catalytic activity and recyclability of the reagent are evaluated via aerobic oxidation of thiols under photosensitized conditions. The transformation of thiols to disulfides is of interest from the viewpoint of organic and biological processes.

2. Results and Discussion

The IL-supported diphenyl telluride was prepared in the following manner (Scheme 1). Using (4-(hydroxymethyl)phenyl)boronic acid (**1**) as the starting material, (4-(phenyltellanyl)phenyl)methanol (**2**) was produced in 84% yield from the coupling reaction with diphenyl ditelluride. Next, (4-(chloromethyl)phenyl)(phenyl)tellane (**3**) was prepared in 94% yield via halogenation with thionyl chloride followed by basic hydrolysis. Then, 1-methyl-3-(4-(phenyltellanyl)benzyl)-1*H*-imidazol-3-ium chloride (**4**) was obtained in 75% yield by reacting the compound **3** with methyl imidazole. Since this IL-supported telluride **4** was extremely hygroscopic, the anion was subsequently converted to PF_6^- to produce hydrophobic IL-supported diphenyl telluride (**5**) in 96% yield, which was insoluble in low polarity organic solvents and water.

Scheme 1. Synthesis of ionic liquid-supported diphenyl telluride.

Initially, we investigated the catalytic oxidation of thiol using IL-supported diphenyl telluride in various ILs employing thiophenol as the model substrate. An IL solution of the thiol, IL-supported catalyst, and rose bengal as a photosensitizer were stirred in an open flask and irradiated with a 500-W halogen lamp. After 3 h, the produced diphenyl disulfide was isolated by extracting with diethyl ether. The yields are compiled in Table 1. The catalytic activities of the IL-supported diphenyl tellurides **4** and **5** were similar to or higher than that of free diphenyl telluride (Entry 7), whereas the reaction was significantly retarded in the absence of the catalyst (Entry 8). The presence of oxygen is also essential for this transformation. In fact, the reaction under nitrogen atmosphere resulted in significant yield reduction (Entry 9). Although the reaction in [bmim](CF_3SO_2) $_2\text{N}$ and [bmim]MeSO $_4$ reached completion, the isolated yields were slightly lowered owing to the phase separation problems (Entries 1 and 2). The best result was obtained using a hydrophobic IL, [bmim]PF $_6$, as a solvent (Entry 3). Diphenyl disulfide was also isolated in quantitative yields in Entries 4 and 6, however, the IL [bmim]BF $_4$ and the IL-supported catalyst **4** were unsuitable for reuse because of their hygroscopic nature.

Although the active species for this oxidation reaction could not be identified at the present stage, a possible catalytic cycle was proposed according to our previous paper (Figure 1) [14]. Namely, singlet oxygen oxidation of the telluride catalyst **5** gave the corresponding telluroxide (and/or tellurone), which underwent a nucleophilic attack by thiol to afford an adduct **A**. Then, the adduct **A** reacted with another thiol to give the disulfide along with regeneration of the catalyst **5**.

Table 2. Catalytic oxidation of various thiols using IL-supported diphenyl telluride **5** ^a.

$\text{R-SH} \xrightarrow[\text{Rose bengal, [bmim]PF}_6, \text{O}_2 \text{ as an air, hv, 3h}]{\text{IL-supported catalyst } \mathbf{5}} \text{R-S-S-R}$			
Entry	Substrate	Product	Yield (%) ^b
1			quant.
2			94
3			73
4			98
5			quant.
6			91
7			quant.
8			quant.
9			71
10			quant.

^a Condition: thiol (0.5 mmol), **5** (0.1 mmol), rose bengal (0.025 mmol), [bmim]PF₆ (5 mL), irradiated with a 500-W halogen lamp under aerobic conditions, 3 h; ^b isolated yield.

Run	1 (fresh)	2	3	4	5
Yield (%) ^a	quant.	quant.	98	97	97

^a Isolated yield.

Figure 2. Recycling experiment of IL-supported diphenyl telluride catalyst **5**. Condition: thiol (1 mmol), tellurium catalyst (0.2 mmol), rose bengal (0.05 mmol), solvent (10 mL), irradiated with a 500-W halogen lamp under aerobic conditions, 3 h.

3. Materials and Methods

3.1. General

All reagents and solvents were commercially sourced and of reagent grade and were used without purification. The reactions were monitored using aluminum thin layer chromatography plates with silica gel 60 F254 (Merck, (Darmstadt, Germany)). Column chromatography was performed using silica gel 60 (Kanto Chemical, Japan, Tokyo). ^1H , ^{13}C , and ^{125}Te NMR spectra were measured on a Bruker Advance DRX 500 (^1H : 500 MHz, ^{13}C : 125 MHz, ^{125}Te : 159 MHz spectrometer. All chemical shifts are reported in parts per million (ppm) relative to TMS (0 ppm for ^1H), CHCl_3 (77 ppm, for ^{13}C), DMSO (39 ppm for ^{13}C), and PhTeTePh (419 ppm in CDCl_3 , 422 ppm in DMSO for ^{125}Te). Mass analyses were performed using a JEOL AccuTOF LC-plus JMS-T100LP spectrometer (Japan, Tokyo).

3.2. (4-(Hydroxymethyl)Phenyl)(Phenyl)Telluride (2)

A solution of diphenyl ditelluride (81.9 mg, 0.200 mmol), (4-hydroxymethyl)phenylboronic acid (**1**) (66.9 mg, 0.440 mmol), 1,10-phenanthroline· H_2O (2.20 mg, 12.0 μmol), and CuSO_4 (19.0 mg, 12.0 μmol) in ethanol (0.6 mL) was stirred at room temperature for 1 min. Then, a 5% Na_2CO_3 aqueous solution (0.1 mL) was added to the solution and the mixture was stirred at room temperature for 5 h. The progress of the reaction was monitored by ^1H NMR spectroscopy. After the reaction was completed, the mixture was dried over MgSO_4 , and the solvent was evaporated. The residue was purified via dry column chromatography to produce product **2** (0.104 g, 84%) as a colorless solid. Mp 62–63 °C; ^1H -NMR (500 MHz, CDCl_3): δ = 7.69–7.67 (m, 4H), J = 6, 4H, 7.21–7.29 (m, 5H), 4.66 (s, 2H), 1.80 (br, 1H); ^{13}C -NMR (125 MHz, CDCl_3): δ = 140.7, 138.3, 137.9, 129.5, 128.1, 127.9, 114.7, 113.7, 65.0; ^{125}Te -NMR (159 MHz, CDCl_3): δ = 690.7; HRMS (APCI): m/z $[\text{M}-\text{OH}]^+$ calcd. for $\text{C}_{13}\text{H}_{11}\text{Te}$: 296.9917, found: 296.9874.

3.3. (4-(Chloromethyl)Phenyl)(Phenyl)Telluride (3)

To a solution of (4-(hydroxymethyl)phenyl)(phenyl)telluride (**2**) (2.32 g, 7.45 mmol) and triethylamine (1.50 mL, 10.4 mmol) in CH_2Cl_2 (4.6 mL), a solution of thionyl chloride (2.41 mL, 33.5 mmol) in CH_2Cl_2 (7.4 mL) was added dropwise at 0 °C. The resulting mixture was stirred at room temperature for 1 h. Then CH_2Cl_2 was removed by evaporation, THF (36.9 mL) and a 2 M NaOH solution (25.2 mL) were added, and the mixture was stirred at room temperature for 19 h. After the reaction was completed, the mixture was evaporated and extracted with ethyl acetate. The organic layer was washed with water, dried over MgSO_4 , and evaporated. The residue was purified via column chromatography to yield product **3** (2.32 g, 94%) as a yellow oil. ^1H -NMR (500 MHz, CDCl_3): δ = 7.72 (d, J = 8.0, 2H), 7.63 (d, J = 8.0, 2H), 7.33–7.21 (m, 5H), 4.55 (s, 2H); ^{13}C -NMR (125 MHz, CDCl_3): δ = 138.5, 137.8, 137.1, 129.6, 128.1 (overlapped), 115.3, 114.3, 45.9; ^{125}Te -NMR (159 MHz, CDCl_3): δ = 690.6; HRMS (APCI): m/z $[\text{M}-\text{Cl}]^+$ calcd. for $\text{C}_{13}\text{H}_{11}\text{Te}$: 296.9917, found: 296.9951.

3.4. 1-Methyl-3-(4-(Phenyltellanyl)Benzyl)-1H-Imidazol-3-Ium Chloride (4)

A solution of phenyl(4-chloromethylphenyl)tellane (**3**) (2.32 g, 7.02 mmol) and *N*-methylimidazole (0.692 g, 8.43 mmol) in CH_3CN (63.6 mL) was heated to reflux for 19 h. Then, the mixture was evaporated, and the residue was purified via column chromatography to yield product **4** (2.18 g, 75%) as a yellow oil. ^1H -NMR (500 MHz, $\text{DMSO}-d_6$): δ = 9.42 (s, 1H), 7.84 (s, 1H), 7.75 (s, 1H), 7.71 (d, J = 8.0, 2H), 7.66 (d, J = 8.0, 2H), 7.37–7.27 (m, 5H), 5.45 (s, 2H), 3.86 (s, 3H); ^{13}C -NMR (125 MHz, $\text{DMSO}-d_6$): δ = 138.7, 137.9, 137.24, 135.0, 130.3, 129.9, 128.7, 124.8, 122.8, 116.6, 115.0, 51.8, 36.3; ^{125}Te -NMR (159 MHz, $\text{DMSO}-d_6$): δ = 705.3; HRMS (APCI): m/z M^+ calcd. for $\text{C}_{17}\text{H}_{17}\text{N}_2\text{Te}$: 379.0448, found: 379.0462.

3.5. 1-Methyl-3-(4-(Phenyltellanyl)Benzyl)-1*H*-Imidazol-3-ium Hexafluorophosphate (5)

A solution of 1-methyl-3-(4-(phenyltellanyl)benzyl)-1*H*-imidazol-3-ium chloride (4) (2.16 g, 5.24 mmol) and KPF₆ (0.965 g, 5.24 mmol) in methanol (10 mL) was stirred at 30 °C for 17 h. Then, the mixture was filtered, and the solution was dried over MgSO₄ and evaporated to yield product 5 (2.63 g, 96%) as a yellow oil. ¹H-NMR (500 MHz, DMSO-*d*₆): δ = 9.18 (s, 1H), 7.77–7.65 (m, 6H), 7.38–7.26 (m, 5H), 5.39 (s, 2H), 3.84 (s, 3H); ¹³C-NMR (125 MHz, DMSO-*d*₆): δ = 138.7, 137.9, 137.2, 134.9, 130.3, 129.9, 128.7, 124.5, 122.8, 116.6, 115.0, 51.9, 36.3; ¹²⁵Te-NMR (159 MHz, DMSO-*d*₆): δ = 707.0; HRMS (APCI): *m/z* M⁺ calcd. for C₁₇H₁₇N₂Te: 379.0448, found: 379.0491.

3.6. Oxidation of Thiols

Typically, [bmim]PF₆ solution (10 mL) of thiophenol (0.110 g, 1.00 mmol), IL-supported diphenyl telluride 5 (0.104 g, 0.200 mmol), and rose bengal (50.9 mg, 0.0500 mmol) were vigorously stirred in an open flask and irradiated using a 500-W halogen lamp for 3 h. The resulting mixture was extracted with diethyl ether and evaporated to yield diphenyl disulfide (0.114 g, quant.). The catalytic oxidation of other thiols was similarly conducted and the products were identified by comparison of physical and spectral data with published values.

3.6.1. Diphenyl Disulfide

Pale pink solids, mp 55–56 °C (mp 59–61 °C [14]); ¹H-NMR (500 MHz, CDCl₃): δ = 7.50 (d, *J* = 8.5, 4H), 7.30 (t, *J* = 7.8, 4H), 7.26 (t, *J* = 8.5, 2H); ¹³C-NMR (125 MHz, CDCl₃): δ = 137.0, 129.1, 127.5, 127.2.

3.6.2. Didodecyl Disulfide

Pale pink solids, mp 33–40 °C (mp 31–32 °C [14]); ¹H-NMR (500 MHz, CDCl₃): δ = 2.70 (t, *J* = 7.5, 4H), 1.68 (quin., *J* = 7.5, 4H), 1.39 (m, 4H), 1.26 (br, 32H), 0.88 (t, *J* = 6.8, 6H); ¹³C NMR (125 MHz, CDCl₃): δ = 39.20, 31.90, 29.68, 29.66, 29.60, 29.50, 29.40, 29.27, 29.25, 28.60, 22.70, 14.10.

3.6.3. Dicyclohexyl Disulfide

Orange oil; ¹H-NMR (500 MHz, CDCl₃): δ = 2.67–2.68 (m, 2H), 2.03–2.05 (m, 4H), 1.77–1.79 (m, 4H), 1.56–1.62 (m, 2H), 1.25–1.31 (m, 10H); ¹³C NMR (125 MHz, CDCl₃): δ = 50.00, 32.90, 26.10, 25.70.

3.6.4. Di-Tert-Butyl Disulfide

Pink oil; ¹H-NMR (500 MHz, CDCl₃): δ = 1.31 (s, 18H); ¹³C NMR (125 MHz, CDCl₃): δ = 46.20, 30.60.

3.6.5. 2-Hydroxyethyl Disulfide

Pink oil; ¹H-NMR (500 MHz, CDCl₃): δ = 3.92 (t, *J* = 5.8, 4H), 2.89 (t, *J* = 5.8, 4H); ¹³C NMR (125 MHz, CDCl₃): δ = 60.40, 41.20.

3.6.6. Dimethyl 3,3'-Dithiodipropionate

Pink oil; ¹H-NMR (500 MHz, CDCl₃): δ = 3.71 (s, 6H), 2.93 (t, *J* = 7.0, 4H), 2.76 (t, *J* = 7.3, 4H); ¹³C NMR (125 MHz, CDCl₃): δ = 172.2, 51.90, 33.90, 33.10 [21].

3.6.7. Dibenzyl Disulfide

Colorless solids, mp 65–70 °C (mp 70–71 °C [14]); ¹H-NMR (500 MHz, CDCl₃): δ = 7.29–7.33 (m, 4H), 7.26–7.28 (m, 4H), 7.22–7.24 (m, 2H), 3.59 (s, 1H); ¹³C NMR (125 MHz, CDCl₃): δ = 137.40, 129.47, 128.53, 127.48, 43.300.

3.6.8. 1,2-Bis(4-Nitrophenyl)Disulfane

Yellow solids, mp 168–173 °C (mp 178–180 °C [22]); ¹H-NMR (500 MHz, CDCl₃): δ = 8.20 (d, *J* = 9 Hz, 4H), 7.61 (d, *J* = 9 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃): δ = 147.30, 144.38, 126.70, 124.79.

3.6.9. 1,2-Bis(4-Chlorophenyl)Disulfane

Colorless solids, mp 63–65 °C (mp 65–66 °C [14]); ¹H-NMR (500 MHz, CDCl₃): δ = 7.39 (d, *J* = 8.5 Hz, 4H), 7.27 (d, *J* = 8.5 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃): δ = 135.44, 133.94, 131.08.

3.6.10. 4,4'-Dipyridyl Disulfide

Colorless solids, mp 59–64 °C (mp 72–74 °C [23]); ¹H-NMR (500 MHz, CDCl₃): δ = 8.56–8.50 (m, 4H), 7.46–7.24 (m, 4H); ¹³C NMR (125 MHz, CDCl₃): δ = 150.4, 149.9, 121.8.

4. Conclusions

We demonstrated the synthesis of the IL-supported organotelluride reagent and its application as a recyclable catalyst for thiol oxidation. A novel diphenyl telluride catalyst **5** carrying an imidazolium hexafluorophosphate moiety as an IL support was successfully synthesized. The synthesis was very easy, requiring only four steps. The IL-supported diphenyl telluride **5** was found to be an efficient catalyst for aerobic oxidation of various thiols under photosensitized conditions, affording the corresponding disulfides in excellent yields. Although the oxidation proceeds smoothly in various IL solutions, [bmim]PF₆ is best suited for efficient phase separation. The produced disulfides can be isolated and purified via simple extraction with diethyl ether. After removal of the product, the resulting IL solution containing IL-supported catalyst (**5**) and rose bengal can be reused at least four times without a considerable loss in catalytic activity (97% yield in the fifth run).

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