



Polymer supported anionic peroxomolybdenum complexes as new, mild, efficient and versatile oxidants in organic synthesis

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Abstract

Two polymer supported anionic peroxomolybdenum compounds, namely $[\text{Amb}]_2[\text{Mo}_2\text{O}_3(\text{O}_2)_4]$ and $[\text{Amb}][\text{MoO}(\text{O}_2)_2(\text{O}_2\text{C-Ph-NH}_2)]$, are prepared and characterized by conventional methods. Their uses as stoichiometric oxidizing agents for a variety of organic compounds are described. With these reagents alcohols are converted to the corresponding carbonyl compounds. Decarboxylation of α -hydroxy acids proceeds quantitatively. Also thiols are converted to disulfides, hydroquinones to quinones, benzylamines to carbonyl compounds, tertiary amines to the *N*-oxides, phosphines to phosphine oxides, and sulfides to sulfones. Deprotection of oximes and oxidative deprotection of silyl ethers proceed easily. © 1999 Elsevier Science Ltd. All rights reserved.

1. Introduction

A large number of polymeric reagents have been developed for use in organic synthesis [1–4]. In all of these applications, advantage is taken of the insolubility of the polymeric reagent and of its by-product which allows for the easy removal of excess reagent or spent materials from the desired product. Oxidation of organic substrates in aprotic solvents under mild and neutral conditions is important in modern organic synthesis. Therefore, the search for new oxidizing reagents is of interest to synthetic organic chemists. For this purpose a large number of reagents have been developed in recent years with some success [5]. In addition several polymer supported oxidizing agents have also been reported in the literature [6–14]. Use of these polymeric reagents is generally advantageous over their monomeric analogues due to simplification of the reaction work-up, stabilization of unstable reagents and

their selectivity. However most of the monomeric and to a lesser extent polymeric reagents have their own deficiencies and drawbacks, for example, a need to use excess reagent, acidity of the reagent, a need to use the wet reagent, unstability of photosensitivity of the reagent, dangerous procedure for their preparation, long reaction time, and being expensive. Therefore, there still exists a need for further investigation and search for new polymeric oxidizing agents.

Peroxide forms of group VI transition metals, chromium, molybdenum, and tungsten, were reviewed recently [15]. The versatility of these transition metal peroxo complexes as oxidants of organic substrates is well documented [16]. According to the literature, complexes of peroxomolybdenum have been widely used as both stoichiometric and catalytic oxidants in organic chemistry. Stoichiometric oxygen transfer reactions include epoxidation of alkenes [17–20], oxidation of primary and secondary alcohols to aldehydes and ketones, respectively [21–23], oxidation of epoxides to dioxetanes or fragmentation products [24], oxidation of enolates and enol ethers to epoxides and

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dioxatanes [25], oxidation of sulfides and sulfoxides to sulfoxides and sulfones [26,27], oxidation of indoles [28], furans [29], phenacetin [30], organoboranes [31] metal alkyls [32], organopalladium compounds [33] and for transformation of oximes to nitro compounds [34].

In connection to our interest in the preparation and application of polymer supported oxidizing agents [11–14], especially polymeric analogues of metal peroxides [35], we now describe the synthesis of two kinds of anionic forms of peroxo molybdate compounds supported on Amberlyst A-26 anion exchange resin, and their uses for efficient oxidation of a wide variety of organic compounds.

2. Experimental

2.1. General

Chemicals were either prepared in our laboratory or purchased from Fluka, BDH, and Merck chemical companies. Dichloroethane was purified by distillation over P_2O_5 . All yields refer to isolated products, unless otherwise indicated. All oxidation products were known compounds and were identified by comparison of their spectra and physical data with those of the authentic samples. Melting points were determined in open capillaries on a Buchi apparatus. Reaction monitoring was accomplished by GLC on a Shimadzu model GC-8A instrument with a flame-ionization detector using a column of 15% carbowax 20 m chromosorb-w-acid washed 60–80 mesh or by TLC on a silica-gel polygram Silg-uv-254 plate. IR spectra were run on a Perkin Elmer 781 spectrophotometer. The NMR spectra were recorded on a Hitachi R-2413, 60 MHz spectrometer. The amount of molybdenum in the reagents was determined by atomic absorption technique. Estimation of the total peroxide content in each compound was accomplished by redox titration with a standard cerium (IV) solution [36] and also with standard potassium permanganate solution [36].

2.2. Preparation of reagent A, $[Amb]_2[Mo_2O_3(O_2)_4]$

A solution of sodium molybdate, $Na_2MoO_4 \cdot 2H_2O$ (5.2 g, 21 mm) in water (100 ml) was placed in an ice-water bath. Treatment with 30% H_2O_2 (10 ml) gave a dark red solution to which dilute hydrochloric acid was added dropwise until the color turned to bright yellow at pH 4–5. The solution was stirred magnetically for 1 h, and to it Amberlyst A-26 (Cl^-) (3.5 g, 15 mm, wet) was added portionwise. The stirring was continued for 10 h. The light yellow resin was filtered and washed several times with water, ether and finally dried over P_2O_5 under vacuum. The IR spectrum

showed bands at 960 cm^{-1} ($Mo = O$), 860 cm^{-1} (O—O) and 760 cm^{-1} (Mo_2O_3 , asy.). The capacity of the reagent based on Mo content, determined by atomic absorption technique, and confirmed by measurement of the peroxide amount determined by redox titration with Ce(IV) or $KMnO_4$, was 0.9 mmol of $Mo_2O_3(O_2)_4$ per gram of dry resin.

2.3. Preparation of Reagent B, $[Amb][MoO(O_2)_2(O_2C-Ph-NH_2)]$





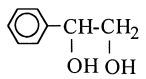
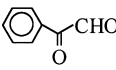




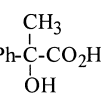
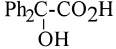
To a suspension of *p*-aminobenzoic acid (2.7 g, 20 mm) in water, Amberlyst A-26 (OH^-) (3.5 g, 15 mm) was added portionwise. The mixture was stirred magnetically for 10 h and then the resin (Amberlyst supported *p*-aminobenzoate) was filtered and washed with acetone and then with water.

Into another beaker, placed in an ice-water bath, $Na_2MoO_4 \cdot 2H_2O$ (3.36 g, 14 mm) was dissolved in water (10 ml), the acidity of solution was adjusted at pH 2 with dilute H_2SO_4 , and H_2O_2 35% (7 ml) was added. To this yellow solution under stirring, the above-mentioned resin was added portionwise, maintaining the acidity of the media at pH 2 with dilute H_2SO_4 . The stirring was continued for 10 h and then yellow-brown resin filtered and washed with water, acetone, then dried over P_2O_5 under vacuum. The IR spectrum showed bands at 3420 cm^{-1} (NH_2), 1700 cm^{-1} (esteric carbonyl) 960 cm^{-1} ($Mo = O$), 860 cm^{-1} (O—O), and 760 cm^{-1} (Mo_2O_3 , asy.). The capacity of the reagent based on Mo content, and confirmed by measurement of peroxide amount was 1.2 mmol of $MoO(O_2)_2(O_2C-Ph-NH_2)$ per gram of dry resin.

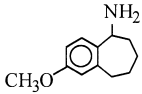
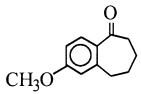
2.4. General procedure for oxidation of alcohols, α -hydroxy acids, thiols, hydroxy phenols, amines, phosphines, sulfides, deoxygenation of oximes and oxidative deprotection of silylethers with reagent A or B

In a round-bottomed flask (25 ml) equipped with a condenser and a magnetic stirrer, a solution of the substrate (1 mm) in 1,2-dichloroethane (10 ml) was prepared. The polymeric reagent A (1 mm) or B (2 mm) was added, and the reaction mixture stirred magnetically while the temperature was kept at 50°C . Progress of the reaction was monitored by TLC (n-hexane:ether, 5:1 as eluent) or GLC. After completion of the reaction, the resin was filtered off and washed with 1,2-dichloroethane. Evaporation of the solvent afforded the pure product in most cases but if necessary the product was further purified by column chromatography on a silica gel column with a non polar solvent as eluent (Table 1 and 2).

Table 1
Oxidation of different organic compounds with reagents A and B^a

Substrate	Product ^d	Reagent A ^b	Reagent B ^c
		Yield % (time (h))	Yield % (time (h))
Benzyl alcohol	Benzaldehyde	96(3)	96(4.5)
2-Naphthalenemethanol	2-Naphthenele carboxaldehyde	96(3.1)	97(4.5)
9-Anthracene methanol	9-Anthracene carboxaldehyde	97(3.1)	97(3.5)
<i>p</i> -Nitrobenzyl alcohol	<i>p</i> -Nitrobenzaldehyde	97(4)	95(5)
Piperanol	Piperanal	95(3)	96(4.5)
1-Phenyl ethanol	Acetophenone	90(8.5)	96(7)
Benzhydrol	Benzophenone	90(9)	95(7.5)
		89(10)	92(8)
Ph-CH ₂ -CH ₂ -CH ₂ -CH ₂ -OH	Ph-CH ₂ -CH ₂ -CH ₂ -CHO	80(15)	90(10)
1-Heptanol	1-Heptanone	80(15)	90(10)
2-Octanol	2-Octanone	81(16)	91(10)
Borneol	Camphor	85(16)	89(11)
		82(16)	92(10)
Benzoin	Benzil	90(7.5)	93(8)
Anisoin	Anisil	92(7)	94(7.8)
Furoin	Furil	90(7.1)	92(7.5)
1,2-Diphenylethyleneglycol	Benzaldehyde	7(6.1)	10(6)
	Benzil	82(6.1)	85(6)
	Benzaldehyde	9(5.8)	5(6)
		80(5.8)	81(6)
Cionamyl alcohol	Cinamaldehyde	90(6)	97(4)
		87(8)	92(7)
		88(8)	91(7.1)
CH ₂ =CH-CH ₂ OH	CH ₂ =CH-CHO	86(8.5)	88(8)
<i>o</i> -Hydroquinone	<i>o</i> -Benzoquinone	97(2)	96(2)
<i>p</i> -Hydroquinone	<i>p</i> -Benzoquinone	99(1.7)	98(1.9)
2- <i>t</i> -Butyl, <i>p</i> -hydroquinone	2- <i>t</i> -Butyl <i>p</i> -benzoquinone	96(2)	98(2)
1,4-dihydroxy naphthalene	1,4-Naphthoquinone	98(1.9)	97(2)
Ph-CH(OH)-CO ₂ H			
	Benzaldehyde	99(2.5)	98(2.9)
Ph ₂ C(OH)-CO ₂ H			
	Acetophenone	97(2.6)	94(2.5)
	Banzophenone	98(2.9)	94(2.5)

(continued on next page)

Ph ₃ P	Ph ₃ PO	99(0.4)	99(0.5)
Ph ₂ MeP	Ph ₂ Me ₂ PO	98(0.4)	98(0.5)
Bu ₃ P	Bu ₃ PO	97(0.6)	98(0.6)
Ph ₂ S	Ph ₂ SO ₂	93(4)	92(4.8)
(PhCH ₂) ₂ S	(PhCH ₂) ₂ SO ₂	92(4)	90(4.5)
Bu ₂ S	Bu ₂ SO ₂	90(5)	91(5.2)
PhMeS	PhMeSO ₂	93(4.3)	93(4.5)
α -Toluene thiol	Benzyl disulfide	93(2)	92(2.2)
Furfuryl mercaptane	Furyl disulfide	94(2.2)	90(2.1)
Thiophenol	Phenyl disulfide	92(2.1)	93(2.4)
Cyclohexane thiol	Cyclohexyldisulfide	92(2.5)	92(2.6)
Benzyl amine	Benzaldehyde	93(5)	92(5.1)
		91(5.5)	91(5.7)
Triethyl amine	Triethyl amine oxide	100(0.8)	100(0.8)
<i>N,N'</i> -Dimethyl aniline	<i>N,N'</i> -Dimethyl aniline oxide	100(1)	100(1)

^a Reaction performed in 1,2-dichloroethane at 50°C.

^b Molar ratio of oxidant to substrate 1/1.

^c Molar ratio of oxidant to substrate 2/1.

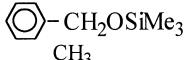
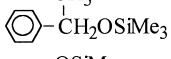
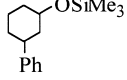

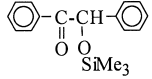
^d Products were pure by TLC and CG analysis and were identified by comparison of their spectra and physical data with those of the authentic samples.

2.5. Regeneration of spent reagents A or B

To an aqueous solution of mixture of nitric acid and hydrochloric acid (2 N, 50 ml, 20/80 ratio), the spent

reagent A or B (1 g) was added and the mixture was warmed and stirred for 2 h. The resin was filtered and washed successively with a solution of hydrochloric acid (2 N) and finally with water. This Cl⁻ for of

Table 2
Deoxygenation of Oximes and Oxidative Deprotection of Silyethers^a

Substrate	Product ^d	Reagent A ^b	Reagent B ^c
		Yield % (time (h))	Yield % (time (h))
Benzaldehyde oxime	Benzaldehyde	90(4)	98(4.5)
9-Anthracene carboxaldehyde oxime	9-Anthracene carboxaldehyde	89(4.1)	91(4.3)
<i>p</i> -Chlorobenzaldehyde oxime	<i>p</i> -Chlorobenzaldehyde	92(4.4)	92(4.5)
Camphoroxime	Camphor	90(4.9)	91(5)
	Benzaldehyde	96(4)	97(4.1)
	Acetophenone	96(4)	95(4.1)
		94(15.5)	93(16.6)
	Benzil	91(7.6)	92(8)

^a Reaction performed in 1,2-dichloroethane at 50°C.

^b Molar ratio of oxidant to substrate 1/1.

^c Molar ratio of oxidant to substrate 2/1.

^d Products were pure by TLC and GC analysis and were identified by comparison of their spectra and physical data with those of the authentic samples.

Amberlyst A-26 was then used for the preparation of reagent A or B with activities the same as those of the original reagents.

3. Results and discussion

A review of the literature data shows that anionic analogues of peroxo molybdenum compounds show superior reactivity for oxidation reactions than neutral ones. Griffith et al. [37] have studied the relative reactivities of different kinds of anionic peroxomolybdenum compounds as stoichiometric oxidants of alcoholic functional groups. They claimed that the dianion, bimetallic complex of $[\text{Mo}_2\text{O}_3(\text{O}_2)_4]^{2-}$ (I) was the most powerful one. Peroxomolybdenum compounds which contain different kinds of ligands have also been reported and used as oxidants. The activity of these compounds is in part controlled by the co-ligand, and it has been found that even “minor” modification of the ligand occupancy can change the properties of the complex dramatically. DiFuria et al. [38] have studied the relative efficiency of different kinds of peroxomolybdenum complexes as oxidants of the alcoholic functional groups. They claimed that anionic types of these compounds containing electron releasing groups, as ligands, were the best. In other words every factor that increased anionic character of oxidant resulted in an increase in its efficiency. They suggested anion $[\text{MoO}(\text{O}_2)_2(\text{O}_2\text{C-Ph-NH}_2)]^-$ (II) as the most effective one. Considering the above facts, two anions (I and II) were chosen and in order to add advantages of polymer-supported reagents to their potential abilities, their polymeric forms were synthesized and studied. Reagent A was prepared easily by immersion of Amberlyst-A-26 (Cl^-) into an aqueous solution of $\text{Na}_2[\text{Mo}_2\text{O}_3(\text{O}_2)_4(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$ which was in turn prepared from sodium molybdate and hydrogen peroxide at pH 4.5.

Reagent B was prepared by immersion of *p*-aminobenzoate supported Amberlyst (prepared from Amberlyst (OH^-) and *p*-aminobenzoic acid) into a solution of sodium molybdate and hydrogen peroxide at pH 2.

IR spectra of these reagents show the characteristic peaks of $\text{Mo}=\text{O}$, $\text{O}-\text{O}$, and $\text{Mo}_2\text{O}(\nu_{\text{asy}})$ at 960, 860 and 760 cm^{-1} , respectively, for both reagents and esteric CO and NH_2 at 1700 and 3420 cm^{-1} , respectively, for reagent B. The determination of the amounts of molybdenum via atomic absorption technique and peroxide contents via redox titration confirmed the proposed structure of reagents A and B. Prior to oxidation reactions, the conditions of reactions, including the kind of solvent, reaction temperature and the ratio of oxidant to substrate, were optimized for a given substrate (anisyl alcohol). Therefore, 1,2-dichloro-

ethane was used as the solvent, 50°C as reaction temperature, and ratio of oxidant to substrate of 1/1 and 2/1 were chosen for reagents A and B, respectively.

Oxidation of different classes of organic compounds were investigated with reagents A and B. As shown in Table 1, benzylic alcohols were converted to the corresponding carbonyl compounds in excellent yields. Overoxidation of primary benzylic alcohols to carboxylic acids, which is common for some other reagents, especially when metal peroxides are prepared in situ by addition of hydrogen peroxide to a suitable metal precursor [39–41], was not observed with these reagents. Primary and secondary saturated alcohols were converted to their corresponding carbonyl compounds in high yields. According to these results, reagent A and B were more efficient for oxidation of benzylic alcohols than saturated alcohols. Acyloins were converted to their corresponding α -diketones with these oxidizing agents in high yields. Carbon–carbon bond cleavage, which is a common side reaction with many reagents, was not observed with these reagents. One of the interesting properties of reagent A and B was their ability to convert 1,2 dihydroxy compounds to their α -diketones contrasting to the results obtained by many other reagents which cleave the carbon–carbon bonds to give the corresponding aldehydes or acids. Oxidation of α - β -unsaturated alcohols to corresponding carbonyl compounds without disturbing the carbon–carbon double bond was performed with both reagents in high yields. Hydroquinones were transformed to quinones very easily in short times and high yields. Oxidative decarboxylation of α -hydroxycarboxylic acids to carbonyl compound was performed by reagents A and B very easily. Phosphines were converted to phosphine oxides quantitatively in very short times. Oxidation of sulfides with ratio of oxidant to substrates equal to 1/1 and 2/1 for reagents A and B, respectively, gave a mixture of sulfoxides and sulfones (70/30 ratio). However with using one molar excess of reagents, i.e. 2/1 and 3/1 for reagents A and B, respectively, sulfones were obtained exclusively. Oxidative coupling of thiols to disulfides were performed in high yields using reagents A and B. Benzylic amines were converted to corresponding carbonyl compounds very easily in high yields. However, in the case of primary aromatic amines, a mixture of unidentified products was obtained and the desired azo coupling products were not observed. Tertiary amines were transformed to corresponding amine oxides very rapidly in high yields. Polycyclic aromatic compounds were converted to quinones in low yields, and using additional reagents was not effective.

Oximes and silyl ethers are important protecting groups for carbonyl compounds [42] and alcohols [43], respectively. Deoximation of different aldoximes and ketoximes and oxidative deprotection of silyl ethers

with reagents A and B afforded the corresponding carbonyl compounds in excellent yields (Table 2).

The regeneration of spent reagents A and B was also investigated. The spent reagents A and B were regenerated easily by acid treatment and washing with water. The resulting Amberlyst (Cl⁻) resin was used directly for preparation of reagent A, or after treatment with NaOH solution and transformation to Amberlyst (OH⁻), for preparation of reagent B. No noticeable reduction in reactivity of reagents for oxidation reactions was observed when regenerated resins were used.

In conclusion, reagents A and B were introduced as new polymeric analogues of anionic peroxomolybdate compounds. Using these reagents many functional group transformations are reported here for the first time in metal peroxide categories. In addition these reagents offer at least one of the following advantages: preparation of the reagents is neither difficult nor dangerous; they are stable and non hygroscopic; they usually provide high yields of products; the reaction medium is neutral, which is suitable for oxidation of acid labile compounds; the molar ratio of oxidant to reactant is reasonable; they are regenerable; and finally, because of the heterogeneous nature of the reagents, the reaction work-ups are very easy.

Acknowledgements

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