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Allyltriphenylphosphonium Peroxodisulfate (CH₂=CHCH₂PPh₃)₂S₂O₈: An Efficient and Convenient Reagent for the Oxidation of Alcohols and Silyl and THP-Ethers Under Nonaqueous Conditions

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Allyltriphenylphosphonium Peroxodisulfate (CH₂==CHCH₂PPh₃)₂S₂O₈: An Efficient and Convenient Reagent for the Oxidation of Alcohols and Silyl and THP-Ethers Under Nonaqueous Conditions

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ABSTRACT

The preparation of allyltriphenylphosphonium peroxodisulfate (ATPPD) as a new and efficient reagent for the oxidation of primary and secondary alcohols, trimethylsilyl and tetrahydropyranyl (THP) ethers to their corresponding carbonyl compounds in refluxing acetonitrile with 1.1:1 molar equivalents of the oxidant is described.

Key Words: Oxidation; Allyltriphenylphosphonium peroxodisulfate; Trimethylsilyl ethers; Tetrahydropyranyl ethers.

1173

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INTRODUCTION

1174

The peroxodisulfate ion is one of the strongest oxidizing agent. The investigations^[1] on application of peroxidisulfate ion in organic synthesis as an oxidant were performed in aqueous acidic or basic solution or in the presence of transition metal ions. In recent decades much attention has been given to the synthesis and application of new peroxodisulfate compounds such as tetrakis(pyridine)silver(II) peroxodisulfate,^[2] tetrabutylammonium peroxodisulfate,^[3] benzyltriphenyl-phosphonium peroxodisulfate,^[4] and *bis*(1-benzyl-3,5,7-1-azoniatricyclo-[3.3.1.1^{3,7}]decane) peroxodisulfate^[5] which were used for the oxidation of organic compounds under nonaqueous conditions.

The protection of functional group is an important process in the synthesis of complex organic molecules.^[6] Alcohols are typically very reactive due to their acidity, and are often protected for organic synthesis. One way to protect hydroxyl group is to convert it to the corresponding silylether. Direct oxidation of trimethylsilyl ethers to the corresponding carbonyl compounds has found considerable attention during recent years.^[7–18] However, some of the reported methods show limitations such as the requirement for aqueous reaction condition,^[8,11] use of expensive reagents,^[13,17,18] long reaction time,^[10,12] low yields of the products,^[16] and tedious workup.^[11] Therefore, introduction of new methods and inexpensive reagents for such functional group transformation is still in demand.

On the other hand, tetrahydropyranyl (THP) group is one of the most frequently used groups to protect alcohols and phenols.^[6,19] Owing to the outstanding stability of THP ether towards strongly basic media, organometallic reagents, reductive and oxidative compounds, 3,4,-dihydro-2H-pyrane is still a reagent of choice for protection of the alcohol group in peptide, nucleotide, and carbohydrate chemistry. Since direct transformation of THP ethers to their carbonyl compounds is rare in the literature,^[20–25] introduction of new method is desirable.

RESULTS AND DISCUSSION

In this communication we wish to report that allyltriphenylphosphonium peroxodisulfate (ATPPD) a stable white solid is able to perform oxidation of alcohols, THP and trimethylsilyl ethers in acetonitrile at reflux temperature. This reagent is easily prepared from an aqueous solution of allyltriphenylphosphonium bromide^[26] and potassium peroxodisulfate at room temperature. This oxidant is soluble in acetonitrile, DMSO, DMF and slightly soluble in acetone and insoluble in carbon tetrachloride, dichloromethane, chloroform, and ether. Structure of this reagent was confirmed by NMR, IR spectroscopy, and elemental analysis.





A wide variety of primary and secondary benzylic alcohols oxidized to the corresponding carbonyl compounds using ATPPD in refluxing acetonitrile with 1.15 : 1 molar ratio of oxidant to substrate in high yields (Table 1, entries 1–11). Under the same reaction conditions allylic alcohol has been selectively oxidized to its α,β -unsaturated carbonyl compound without any cleavage of the carbon–carbon double bond (Table 1, entry 12). α -Hydroxy ketone has been converted to its α -diketone in moderate yield (Table 1, entry 15), even on longer periods.

A noteworthy advantage of this reagent lies in its ability to selective oxidation of benzylic hydroxyl group in the presence of aliphatic hydroxyl group. In order to evaluate the selectivity of this reagent, the following competitive reaction was carried out [Eq. (1)].

$$Ph_{2}CHOH + Cyclohexanol \xrightarrow[CH_{3}CN/Reflux(20 \text{ min})]{} Ph_{2}CO + Cyclohexanol (unchanged)$$
(1)

Yield^b Time Entry Substrate Product^a (min) (%) 1 C₆H₅CH₂OH C₆H₅CHO 20 95 2 p-ClC₆H₄CH₂OH p-ClC₆H₄CHO 15 84 3 o-ClC₆H₄CH₂OH o-ClC₆H₄CHO 20 86 4 p-CH₃C₆H₄CH₂OH p-CH₃C₆H₄CHO 20 94 5 o-CH₃C₆H₄CHO 20 92 o-CH₃C₆H₄CH₂OH 6 p-CH₃OC₆H₄CH₂OH p-CH₃OC₆H₄CHO 15 92 7 o-CH₃OC₆H₄CHO 20 91 o-CH3OC6H4CH2OH 95 8 $(C_6H_5)_2CO$ 20 (C₆H₅)₂CHOH 9 p-ClC₆H₄CH(OH)C₆H₅ p-ClC₆H₄COC₆H₅ 30 87 10 20 83 C₆H₅CH(OH)CH₃ C₆H₅COCH₃ 11 30 81 p-ClC₆H₄CH(OH)CH₃ p-ClC₆H₄COCH₃ 12 C₆H₅CH=CHCH₂OH C₆H₅CH=CHCHO 35 86 40 13 C₆H₅CH(OH)CH₂CH₃ C₆H₅COCH₂CH₃ 80 60 30 14 C₆H₅CH₂CH₂OH C₆H₅CH₂CHO C₆H₅COCOC₆H₅ 60 50 15 C₆H₅COCH(OH)C₆H₅ CH≡CCH₂OH CH≡CCH₂OH 60 16 17 CH₃(CH₂)₆CH₂OH CH₃(CH₂)₆CH₂OH 60 18 C₆H₁₁OH C₆H₁₁OH 60

Table 1. Oxidation of alcohols with ATPPD in refluxing CH₃CN.

^aProducts were characterized by comparison of their physical and spectra data with authentic samples.

^bYields refer to isolated products.

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As shown in Eq. (1), when an equimolar amount of benzhydrol and cyclohexanol was treated with one molar ratio of reagent only benzylic alcohol was selectively oxidized and cyclohexanol remained unchanged.

1176

In the oxidation of alcohols the overoxidation of products to the corresponding carboxylic acids was not at all observed.

In addition, we also report oxidative deprotection of benzylic trimethylsilyl and THP ethers with this reagent under mild conditions (Sch. 1).

Similar to oxidation of alcohols benzylic trimethylsilyl and THP ethers are efficiently converted to the corresponding carbonyl compounds using ATPPD in refluxing acetonitrile using 1.15 : 1 molar ratio of oxidant to substrate. The yields are high to excellent (Tables 2 and 3 entries 1–11). Under the reaction condition allylic trimethylsilyl and THP ethers have been selectively oxidized to their α , β -unsaturated carbonyl compounds without any cleavage of the carbon–carbon double bonds (Tables 2 and 3 entry 12). α -Hydroxy trimethylsilyl and THP ethers, have been converted to their α -diketone in high yield (Tables 2 and 3 entry 15).

ATPPD cannot oxidize aliphatic trimethylsilyl and THP ethers. This chemoselectivity was examined by the following competitive reactions [Eqs. (2) and (3)].

$$o-CH_3OC_6H_4CH_2OSiMe_3 + n-Octan-OSiMe_3 \xrightarrow{\text{AIPPD}} o-CH_3OC_6H_4CHO + n-Octan-OSiMe_3 \qquad (2)$$

$$p\text{-ClC}_{6}H_{4}CH_{2}OTHP + HC \equiv CCH_{2}OTHP \xrightarrow{\text{AIPPD}} p\text{-ClC}_{6}H_{4}CHO + HC \equiv CCH_{2}OTHP \qquad (3)$$

As shown in above equations, when an equimolar amount of benzylic trimethylsilyl or THP ethers and aliphatic trimethylsilyl or THP ethers were treated with one molar ratio of reagent, only benzylic trimethylsilyl or THP ethers were selectively oxidized.

In conclusion, we report that ATPPD is easily prepared as a stable powder which can be kept for months without loosing its activity. In all reactions 1-1.15 equivalents of the oxidant were consumed which shows the efficiency of the reagent and advantage of this oxidant in comparison with some other peroxodisulfate derivatives. Mildness of reaction conditions, short reaction

$$R_1R_2CHOX \xrightarrow[CH_3CN/Reflux]{CH_3CN/Reflux} R_1R_2CO$$

Scheme 1.

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Table 2. Oxidation of trimethylsilyl ethers with ATPPD in refluxing CH₃CN.

Entry	Substrate	Product ^a	Time (min)	Yield ^b (%)
1	C ₆ H ₅ CH ₂ OSiMe ₃	C ₆ H ₅ CHO	15	95
2	p-ClC ₆ H ₄ CH ₂ OSiMe ₃	<i>p</i> -ClC ₆ H ₄ CHO	20	93
3	o-ClC ₆ H ₄ CH ₂ OSiMe ₃	o-ClC ₆ H ₄ CHO	20	94
4	p-CH ₃ C ₆ H ₄ CH ₂ OSiMe ₃	p-CH ₃ C ₆ H ₄ CHO	15	96
5	o-CH ₃ C ₆ H ₄ CH ₂ OSiMe ₃	o-CH ₃ C ₆ H ₄ CHO	15	98
6	p-CH ₃ OC ₆ H ₄ CH ₂ OSiMe ₃	<i>p</i> -CH ₃ OC ₆ H ₄ CHO	15	97
7	o-CH ₃ OC ₆ H ₄ CH ₂ OSiMe ₃	o-CH ₃ OC ₆ H ₄ CHO	15	95
8	(C ₆ H ₅) ₂ CHOSiMe ₃	$(C_6H_5)_2CHO$	20	92
9	p-ClC ₆ H ₄ CH(OSiMe ₃)C ₆ H ₅	p-ClC ₆ H ₄ COC ₆ H ₅	25	86
10	C ₆ H ₅ CH(OSiMe ₃)CH ₃	C ₆ H ₅ COCH ₃	20	88
11	p-ClC ₆ H ₄ CH(OSiMe ₃)CH ₃	p-ClC ₆ H ₄ COCH ₃	30	83
12	C ₆ H ₅ CH=CHCH ₂ OSiMe ₃	C ₆ H ₅ CH=CHCHO	25	84
13	C ₆ H ₅ CH(OSiMe ₃)CH ₂ CH ₃	C ₆ H ₅ COCH ₂ CH ₃	30	82
14	C ₆ H ₅ CH ₂ CH ₂ OSiMe ₃	C ₆ H ₅ CH ₂ CHO	35	85
15	C ₆ H ₅ COCH(OsiMe ₃)C ₆ H ₅	C ₆ H ₅ COCOC ₆ H ₅	30	85
16	CH≡CCH ₂ OSiMe ₃	CH≡CCH ₂ OSiMe ₃	60	
17	CH ₃ (CH ₂) ₆ CH ₂ OSiMe ₃	CH ₃ (CH ₂) ₆ CH ₂ OSiMe	60	
18	C ₆ H ₁₁ OSiMe ₃	C ₆ H ₁₁ OSiMe ₃	60	

^aProducts were characterized by comparison of their physical and spectra data with authentic samples.

^bYields refer to isolated products.

time, ease of work-up procedure, stability of the reagent, and high yield of products are among the other advantages of this method which make ATPPD a benchtop and versatile reagent for the oxidation of alcohols, trimethylsilyl and THP ethers to the corresponding carbonyl compounds.

EXPERIMENTAL

IR spectra were determined on a shimadazu model 8300. ¹H NMR spectra were recorded with a bruker advance DPX 250 MHz spectrometer. Solvent was freshly distilled. Trimethylsilyl ethers and THP ethers were prepared according to the literature procedure.^[27,28]

Preparation of Allyltriphenylphosphonium Peroxodisulphate

To an aqueous solution of allyltriphenylphosphonium bromide^[26] (24 g, 0.062 mol; in 100 mL of mild hot water) was added a solution of potassium



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Entry	Substrate	Product ^a	Time (min)	Yield ^b (%)
1	C ₆ H ₅ CH ₂ OTHP	C ₆ H ₅ CHO	20	96
2	p-ClC ₆ H ₄ CH ₂ OTHP	p-ClC ₆ H ₄ CHO	20	90
3	o-ClC ₆ H ₄ CH ₂ OTHP	o-ClC ₆ H ₄ CHO	20	91
4	p-CH ₃ C ₆ H ₄ CH ₂ OTHP	p-CH ₃ C ₆ H ₄ CHO	15	93
5	o-CH ₃ C ₆ H ₄ CH ₂ OTHP	o-CH ₃ C ₆ H ₄ CHO	15	94
6	p-CH ₃ OC ₆ H ₄ CH ₂ OTHP	p-CH ₃ OC ₆ H ₄ CHO	15	97
7	o-CH ₃ OC ₆ H ₄ CH ₂ OTHP	o-CH ₃ OC ₆ H ₄ CHO	20	96
8	$(C_6H_5)_2$ CHOTHP	$(C_6H_5)_2CO$	20	90
9	p-ClC ₆ H ₄ CH(OTHP)C ₆ H ₅	p-ClC ₆ H ₄ COC ₆ H ₅	25	86
10	C ₆ H ₅ CH(OTHP)CH ₃	C ₆ H ₅ COCH ₃	20	87
11	p-ClC ₆ H ₄ CH(OTHP)CH ₃	p-ClC ₆ H ₄ COCH ₃	25	85
12	C ₆ H ₅ CH=CHCH ₂ OTHP	C ₆ H ₅ CH=CHCHO	35	83
13	C ₆ H ₅ CH(OTHP)CH ₂ CH ₃	C ₆ H ₅ COCH ₂ CH ₃	30	80
14	C ₆ H ₅ CH ₂ CH ₂ OTHP	C ₆ H ₅ CH ₂ CHO	60	40
15	C ₆ H ₅ COCH(OTHP)C ₆ H ₅	C ₆ H ₅ COCOC ₆ H ₅	40	80
16	CH≡CCH ₂ OTHP	CH≡CCHO	60	
17	CH ₃ (CH ₂) ₆ CH ₂ OTHP	CH ₃ (CH ₂) ₆ CHO	60	_
18	C ₆ H ₁₁ OTHP	Cyclohexanon	60	_

Table 3. Oxidation of THP ethers with ATPPS in refluxing CH₃CN.

^aProducts were characterized by comparison of their physical and spectra data with authentic samples.

^bYields refer to isolated products.

1178

peroxodisulfate (8.38 g, 0.031 mol; in 170 mL of H₂O) and the mixture was stirred at room temperature for 3 hr. The resulting white solid product was filtered, washed with distilled water (50 mL) and dried in a desiccator under vaccum over phosphorus pentoxide to give product.(23 gr., 92%). M.p. 150-152°C

$$2(CH_2 = CHCH_2(Ph)_3P)^+Br^- + K_2S_2O_8 \xrightarrow[rt/3 hr]{water}$$
$$(CH_2 = CHCH_2(Ph)_3P)_2S_2O_8 + 2 \text{ KBr}$$

¹H NMR (250 MHz) $\delta = 7.85$ (m, 15H, (C₆H₅)₃P), 5.72–5.75 (m, 1H, CH2==CH-), 5.35-5.41 (m, 2H, CH2==CH-), 4.56-4.65 (m, 2H, -CH2-P(Ph)3); IR (KBr) 3010, 2900, 1400, 1300, and 1100 (-O-SO2-O-), 1000, 925, 850, 710, 675. Found: C, 63%; H, 4.9%; S, 7.9% Calc for C₄₂H₄₀P₂S₂O₈: C, 63.14%; H, 5.04%; S, 8.02%.





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General Procedure for Oxidation of Alcohols, THP and Silyl Ethers

Substrate (1 mmol) was dissolved in acetonitrile (10 mL) and oxidant (1.15 mmol) was added to it. The mixture was refluxed for specified time (Tables 1–3), until entire substrate was disappeared. The progress of reaction was monitored by TLC (eluent: carbon tetrachloride/ethyl acetate 20:4 for alcohols and 20:1 for trimethylsilylether and THP ethers). The reaction mixture was allowed to cool to room temperature and filtered. The solid material was washed with acetonitrile (20 mL) and combined filtrates were evaporated on a rotary evaporator.

The crude material was purified on a silica gel plate to afford pure carbonyl compound. Products were separated and were identified by the comparison of their m.p., IR, NMR spectra, and b.p., with those reported for the authentic samples.

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