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Benzyltriphenylphosphonium Peroxodisulfate $(PhCH_2PPh_3)_2S_2O_8$: a Mild and Inexpensive Reagent for Efficient Oxidation of Organic Compounds under Nonaqueous and Aprotic Conditions

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Benzyltriphenylphosphonium peroxodisulfate is an easily prepared and stable reagent. It could be used as an oxidant under aprotic and nonaqueous conditions in organic solvents. This reagent oxidizes different classes of alcohols to carbonyl compounds, thiols to disulfides, sulfides to sulfoxides, oximes to carbonyl compounds and aromatic amines to azo compounds efficiently. α-Hydroxy carboxylic acids and phenylacetic acids undergo oxidative decarboxylation to produce carbonyl compounds.

The oxidation of organic compounds under nonaqueous and aprotic conditions has found valuable applications in modern organic synthesis. The peroxodisulfate ion is one of the strongest oxidizing agents. The standard redox potential of this ion in aqueous solution is about 2.01 V. The application of peroxodisulfate ion in organic synthesis as an oxidant has been extensively investigated. The typical reactions are: The oxidation of unsaturated aliphatic and arylalkyl alcohols,[1)] alkenes,[2,3)] and thymines;[4,5)] the acetoxylation of methylbenzenes;[6)] the oxidation of amines to Schiff bases[7)] and nitriles;[8)] the conversion of aldehydes and alcohols to nitriles in the presence of ammonia;[9)] the oxidation of allyl alcohols,[10)] nucleic acid bases[11)] and carboxylic acids;[12)] the conversion of electron-rich benzylic hydrocarbons to carbonyl compounds;[13)] the oxidation and fragmentation of phenyl-substituted alcohols and ethers;[14)] and the conversion of aliphatic ketones to diketones.[15)] Most of these reactions are performed in aqueous acidic or basic solutions or in the presence of metal ions such as Ag(I), Fe(II), Ni(II), and Cu(II). Therefore, the introduction of new peroxodisulfate oxidants which work under nonaqueous and aprotic conditions is still in demand.

Tetrakis(pyridine)silver(II) peroxodisulfate[16)] and tetrabutylammonium peroxodisulfate[17)] have also been prepared and used for the oxidation of organic compounds.

We have recently reported on reagents for the oxidative transformation of organic compounds under aprotic conditions in organic solvents.[18)] In this paper we introduce benzyltriphenylphosphonium peroxodisulfate as a new oxidant for the oxidation of organic compounds under nonaqueous and aprotic conditions.

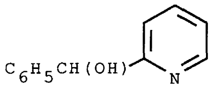
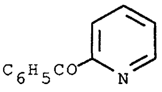
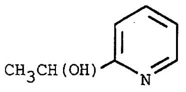
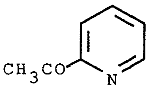
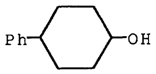
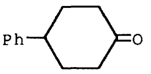
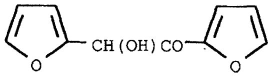
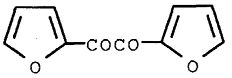
Results and Discussion

Benzyltriphenylphosphonium peroxodisulfate is very easily prepared from an aqueous solution of benzyltriphenylphosphonium chloride and potassium peroxodisulfate at room temperature. This reagent is a stable white powder which could be stored for months without losing its activity. This oxidant is soluble in acetonitrile, chloroform and dichloromethane, and slightly soluble in carbon tetrachloride and ether.

The oxidation of organic compounds with benzyltriphenylphosphonium peroxodisulfate proceeds well in refluxing acetonitrile. Primary and secondary benzylic and saturated alcohols have been oxidized to their corresponding carbonyl compounds in high yields (Table 1, Entries 1—14). Under the same reaction conditions primary and secondary allylic alcohols have been selectively oxidized to their α,β-unsaturated carbonyl compounds without any cleavage of the carbon–carbon double bonds (Table 1, Entries 15—21). In contrast, manganese dioxide, which has been widely used for the oxidation of allylic alcohols, requires a large excess of the reagent and long reaction times for successful oxidations.[19)] α-Hydroxy ketones have been converted to their α-diketones in excellent yields (Table 1, Entries 22—24). Carbon–carbon bond cleavage was not observed in these oxidations. The mildness of the reagent has been shown by the oxidation of thiols to their disulfides in high yields (Table 2, Entries 1—5). This reagent is able to oxidize sulfides to sulfoxides in good yields (Table 2, Entries 6—11). Oximes are converted to the carbonyl compounds with this oxidant in high yields (Table 3, Entries 1—10). Aromatic amines are efficiently converted to their azo compounds (Table 3, Entries 11—19). α-Hydroxy carboxylic acids and phenylacetic acids undergo oxidative decarboxylation as a result of which their corresponding carbonyl compounds are produced in high yields (Table 4).

In Table 5 some of the results of our experiments are

Table 1. Oxidation of Hydroxy Compounds with $(PhCH_2PPh_3)_2S_2O_8$ in Refluxing CH_3CN

Entry	Substrate	Product	Time (h)	Yield (%)[a)]	Mp or bp (°C)[20,21]
1	$C_6H_5CH_2OH$	C_6H_5CHO	0.5	100	176—178
2	$4\text{-}CH_3OC_6H_4CH_2OH$	$4\text{-}CH_3OC_6H_4CHO$	0.5	95	248
3	$4\text{-}ClC_6H_4CH_2OH$	$4\text{-}ClC_6H_4CHO$	0.5	92	47—50
4	$4\text{-}BrC_6H_4CH_2OH$	$4\text{-}BrC_6H_4CHO$	0.5	93	55—58
5	$4\text{-}NO_2C_6H_4CH_2OH$	$4\text{-}NO_2C_6H_4CHO$	1	80	105—108
6	$C_6H_5CH(OH)C_6H_5$	$C_6H_5COC_6H_5$	0.5	98	49—51
7	$4\text{-}ClC_6H_4CH(OH)CH_3$	$4\text{-}ClC_6H_4COCH_3$	0.75	98	230—232
8	$C_6H_5CH(OH)CH_2C_6H_5$	$C_6H_5COCH_2C_6H_5$	0.75	96	55—56
9	$4\text{-}BrC_6H_4CH(OH)CH_2Br$	$4\text{-}BrC_6H_4COCH_2Br$	0.5	98	108—110
10	$4\text{-}NO_2C_6H_4CH(OH)C_6H_5$	$4\text{-}NO_2C_6H_4COC_6H_5$	1	86	136—138
11			0.5	92	42—44
12			0.5	90	188—189
13			0.75	92	78—80
14	$C_6H_5CH_2CH(OH)CH_3$	$C_6H_5CH_2COCH_3$	0.6	95	235
15	$C_6H_5CH{=}CHCH(OH)C_6H_5$	$C_6H_5CH{=}CHCOC_6H_5$	0.75	88	57—58
16	$4\text{-}CH_3C_6H_4CH(OH)CH{=}CHC_6H_5$	$4\text{-}CH_3C_6H_4COCH{=}CHC_6H_5$	1	89	77—78
17	$4\text{-}CH_3OC_6H_4CH{=}CHCH(OH)C_6H_5$	$4\text{-}CH_3OC_6H_4CH{=}CHCOC_6H_5$	0.75	83	72—74
18	$3\text{-}NO_2C_6H_4CH{=}CHCH(OH)C_6H_5$	$3\text{-}NO_2C_6H_4CH{=}CHCOC_6H_5$	1	82	145—146
19	$C_6H_5CH{=}CHCH(OH)CH_3$	$C_6H_5CH{=}CHCOCH_3$	0.75	88	260—262
20	$C_6H_5CH{=}CHCH_2OH$	$C_6H_5CH{=}CHCHO$	0.75	85	248
21	$4\text{-}NO_2C_6H_4CH{=}CHCH(OH)C_6H_5$	$4\text{-}NO_2C_6H_4CH{=}CHCOC_6H_5$	1	80	158—160
22	$C_6H_5CH(OH)COC_6H_5$	$C_6H_5COCOC_6H_5$	1	95	94—95
23	$4\text{-}CH_3OC_6H_4CH(OH)COC_6H_4(4\text{-}OCH_3)$	$4\text{-}CH_3OC_6H_4COCOC_6H_4(4\text{-}OCH_3)$	1	92	132—134
24			1	90	163—165

a) Substrate/Oxidant (1 : 1).

compared with some of those reported with tetrakis(pyridine)silver(II) peroxodisulfate.[16b)] The results show that the rates of the reaction and the yields are usually higher with benzyltriphenylphosphonium peroxodisulfate than those reported with tetrakis(pyridine)silver(II) peroxodisulfate; also, the ratio of the substrate to the oxidant for benzyltriphenylphosphonium peroxodisulfate is lower. It must be pointed out that benzyltriphenylphosphonium peroxodisulfate is cheaper than tetrakis(pyridine)silver(II) peroxodisulfate; this fact is of great importance from an economical point of view.

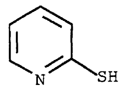
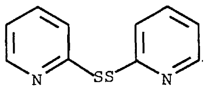
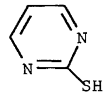
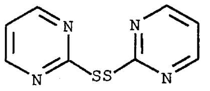
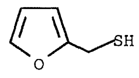
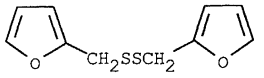
Experimental

General: Chemicals were either prepared in our laboratories or purchased from Fluka or Merck Chemical Companies. The oxidation products were characterized by a comparison of their spectral and physical data with authentic samples. The yields refer to isolated products. The melting points were determined using a Mettler FP5 apparatus. Infrared (IR) spectra were obtained on a Philips PU9716 spectrophotometer. ^1H and ^{13}C NMR spectra were recorded on Bruker AW 80 MHz and Bruker AvanceDPX-250 MHz spectrometers in $CDCl_3$ and $(CD_3)_2SO$ solvents, respectively. An elemental analysis was performed by Research Institute of Petroleum Industry, Tehran-Iran. A GLC analysis was performed with a Shimadzu 16A Gas Chromatograph with a flame-ionization detector and Carbowax 20M/Chromosorb-W acid washed 60—80 mesh.

Preparation of Benzyltriphenylphosphonium Peroxodisulfate. To an aqueous solution of benzyltriphenylphosphonium chloride (8.63 g, 22 mmol; 75 ml of H_2O) was added a solution of potassium peroxodisulfate (3 g, 11 mmol) in water (60 ml); the mixture was stirred at room temperature for 15 min. The resulting white solid product was filtered, washed with cooled distilled water (20 ml) and dried in a desiccator under a vacuum over calcium chloride, yield 9.5 g, 96%, mp 180—182 °C. ^1H NMR (80 MHz) $\delta = 4.7$ (d, $J = 25.6$ Hz, 2H, $C\underline{H}_2$–P), 6.87—7.93 (m, 20 H); ^{13}C NMR (250 MHz) $\delta = 117.3$ (d, $J = 85.7$ Hz, P-$\underline{C}H_2$), 127.2, 127.7, 128.1, 129.4, 129.6, 130.2, 133.3, 133.5, 134.5; IR (KBr) 1290, 1260, 1092, 1058, 700, 685, 590, 554 cm^{-1}. Found: C, 66.7; H, 4.9; S, 7.35%. Calcd for $C_{50}H_{44}O_8P_2S_2$: C, 66.8; H, 4.9; S, 7.13%. Found: S_2O_8, 21.3%. Calcd: 21.38%.[22)]

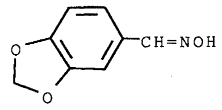
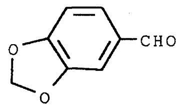
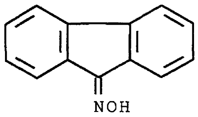
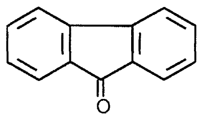
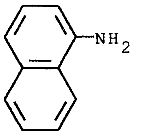
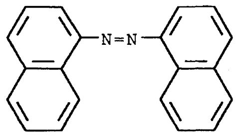
Oxidation of Organic Compounds with Benzyltriphenylphosphonium Peroxodisulfate; General Procedure. In a round-bottom flask (50 ml) equipped with a condenser and a magnetic stirrer, a solution of organic compound (1 mmol) in acetonitrile (10—

Table 2. Oxidation of Thiols to Disulfides and Sulfides to Sulfoxides with $(PhCH_2PPh_3)_2S_2O_8$ in Refluxing CH_3CN

Entry	Substrate	Product	Time (h)	Yield (%)[a]	Mp or bp (°C)[20]
1	C_6H_5SH	$C_6H_5SSC_6H_5$	0.5	92	58—60
2	$4\text{-}NO_2C_6H_4SH$	$(4\text{-}NO_2C_6H_4S)_2$	0.75	88	184—186
3			0.75	95	56—58
4			0.75	95	142—145
5			0.75	86	112—115/0.5 mmHg[b]
6	$(C_6H_5CH_2)_2S$	$(C_6H_5CH_2)_2SO$	2	70	133—135
7	$C_6H_5SCH_2C_6H_5$	$C_6H_5S(O)CH_2C_6H_5$	1.5	72	122—124
8	$4\text{-}NO_2C_6H_4SC_6H_5$	$4\text{-}NO_2C_6H_4S(O)C_6H_5$	2.5	60	107—108
9	$C_6H_5CH_2SC_4H_9$	$C_6H_5CH_2S(O)C_4H_9$	2	65	62—64
10	$C_6H_5SCH_3$	$C_6H_5S(O)CH_3$	1.5	67	139—141/14 mmHg[b]
11	$C_6H_5SC_4H_9$	$C_6H_5S(O)C_4H_9$	2	65	102—104

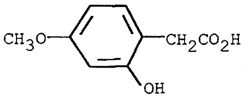
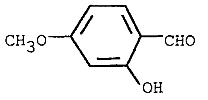
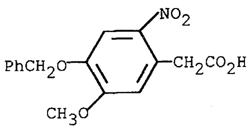
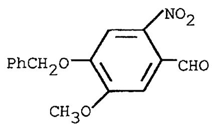
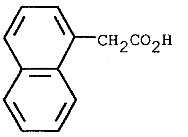
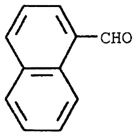
a) Thiol/Oxidant (1 : 1), Sulfide/Oxidant (2 : 1). b) 1 mmHg = 133.322 Pa.

Table 3. Oxidation of Oximes and Amines to Carbonyl and Azo Compounds with $(PhCH_2PPh_3)_2S_2P_8$ in Refluxing CH_3CN

Entry	Substrate	Product	Time (h)	Yield (%)[a]	Mp or bp (°C)[20]
1	$C_6H_5CH{=}NOH$	C_6H_5CHO	0.5	90	176—178
2	$4\text{-}NO_2C_6H_4CH{=}NOH$	$4\text{-}NO_2C_6H_4CHO$	0.75	75	105—108
3	$3\text{-}NO_2C_6H_4CH{=}NOH$	$3\text{-}NO_2C_6H_4CHO$	1	90	57—59
4			1	78	264
5	$C_6H_5C({=}NOH)CH_3$	$C_6H_5COCH_3$	0.75	96	202
6	$(C_6H_5)_2C{=}NOH$	$C_6H_5COC_6H_5$	0.75	98	49—51
7	$4\text{-}ClC_6H_4C({=}NOH)CH_3$	$4\text{-}ClC_6H_4COCH_3$	0.5	96	230—232
8	$C_6H_5CH{=}CHC({=}NOH)CH_3$	$C_6H_5CH{=}CHCOCH_3$	0.75	90	260—262
9	$C_6H_5CH{=}CHCH{=}NOH$	$C_6H_5CH{=}CHCHO$	0.5	93	248
10			0.5	88	82—85
11	$3\text{-}CH_3OC_6H_4NH_2$	$3\text{-}CH_3OC_6H_4N{=}NC_6H_4OCH_3(3-)$	1.25	85	73—75
12	$4\text{-}CH_3C_6H_4NH_2$	$4\text{-}CH_3C_6H_4N{=}NC_6H_4CH_3(4-)$	1.5	96	138—140
13	$2\text{-}CH_3C_6H_4NH_2$	$2\text{-}CH_3C_6H_4N{=}NC_6H_4CH_3(2-)$	1.5	93	52—54
14	$4\text{-}EtC_6H_4NH_2$	$4\text{-}EtC_6H_4N{=}NC_6H_4Et(4-)$	1.5	92	62—63
15	$4\text{-}NO_2C_6H_4NH_2$	$4\text{-}NO_2C_6H_4N{=}NC_6H_4NO_2(4-)$	1.5	55	220—222
16	$4\text{-}ClC_6H_4NH_2$	$4\text{-}ClC_6H_4N{=}NC_6H_4Cl(4-)$	1.5	80	220—221
17	$3\text{-}NO_2C_6H_4NH_2$	$3\text{-}NO_2C_6H_4N{=}NC_6H_4NO_2(3-)$	1.5	60	148—150
18	$C_6H_5NH_2$	$C_6H_5N{=}NC_6H_5$	1.25	95	68—69
19			1.25	85	188—190

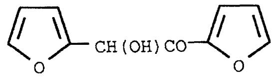
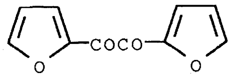
a) Substrate/Oxidant (1 : 1).

Table 4. Oxidative Decarboxylation of α-Hydroxy Carboxylic and Phenylacetic Acids with $(PhCH_2PPh_3)_2S_2O_8$ in Refluxing CH_3CN

Entry	Substrate	Product	Time (h)	Yield (%)[a]	Mp or bp (°C)[20]
1	$(C_6H_5)_2CHCO_2H$	$C_6H_5COC_6H_5$	0.5	85	48—50
2	$C_6H_5CH_2CO_2H$	C_6H_5CHO	0.5	85	176—178
3	$4\text{-}HOC_6H_4CH_2CO_2H$	$4\text{-}HOC_6H_4CHO$	0.5	92	117—119
4	$C_6H_5CH(OH)CO_2H$	C_6H_5CHO	1	82	176—178
5			0.5	90	41—43
6			1	75	133
7			0.5	88	160—161/15 mmHg

Substrate/Oxidant (1 : 1).

Table 5. Comparison of Oxidation of some Organic Compounds with $(PhCH_2PPh_3)_2S_2O_8$ and $(Py)_4AgS_2O_8$

Entry	Substrate	Product	Yield (%) (molar equiv, h)	
			$(PhCH_2PPh_3)_2S_2O_8$	$(Py)_4AgS_2O_8$
1	$C_6H_5CH_2OH$	C_6H_5CHO	100 (1, 0.5)	100 (1.5, 0.75)
2	$4\text{-}NO_2C_6H_4CH_2OH$	$4\text{-}NO_2C_6H_4CHO$	80 (1, 1)	60 (3, 3)
3	$4\text{-}CH_3OC_6H_4CH_2OH$	$4\text{-}CH_3OC_6H_4CHO$	95 (1, 0.5)	65 (3, 3)
4			90 (1, 1)	55 (3, 3)
5	$C_6H_5CH{=}CHCH_2OH$	$C_6H_5CH{=}CHCHO$	85 (1, 0.75)	80 (3, 6)
6	$C_6H_5CH_2CO_2H$	C_6H_5CHO	85 (1, 0.5)	45 (3, 0.75)
7	$C_6H_5CH(OH)CO_2H$	C_6H_5CHO	82 (1, 1)	50 (3, 2)
8	$(C_6H_5)_2CHCO_2H$	$C_6H_5COC_6H_5$	85 (1, 0.5)	95 (3, 0.25)

15 ml) was prepared. Benzyltriphenylphosphoniun peroxodisulfate (1—2 mmol) was added to the solution, and the reaction mixture was stirred under reflux conditions for 0.5—2.5 h. The progress of the reaction was monitored by GLC or TLC (eluent: CCl_4/Et_2O). The reaction mixture was cooled to room temperature and filtered. The solid material was washed with acetonitrile (20 ml). The filtrate was evaporated on a rotary evaporator, and the resulting crude material was purified on a silica-gel plate or silica-gel column with an appropriate eluent. Pure products were obtained in 55—100% yields (Tables 1, 2, 3, and 4).

Oxidation of Diphenylmethanol to Benzophenone as a Typical Procedure for Hydroxyl Group Oxidation. In a round-bottom flask (50 ml) equipped with a condenser and a magnetic stirrer, a solution of diphenylmethanol (0.184 g, 1 mmol) in acetonitrile (15 ml) was prepared. Benzyltriphenylphosphonium peroxodisulfate (0.898 g, 1 mmol) was added to the solution and the mixture was stirred under reflux conditions for 0.5 h. The progress of the reaction was monitored by TLC (eluent: CCl_4/Et_2O: 3/1). The reaction mixture was cooled to room temperature and filtered. The solid material was washed with acetonitrile (20 ml). The combined filtrates were evaporated, and the resulting crude material was purified on a silica-gel plate (eluent: CCl_4/Et_2O: 3/1) to afford pure benzophenone; yield 0.178 g, 98%, mp 48—50 °C, lit,[20] 49—51 °C (Table 1).

Oxidation of Benzenethiol to Diphenyl Disulfide as a Typical Procedure for the Oxidation of Thiols to the Corresponding Disulfides. To a solution of benzenethiol (0.110 g, 1 mmol) in acetonitrile (10 ml) in a round-bottom flask (50 ml) equipped with a condenser and a magnetic stirrer, the oxidant (0.898 g, 1 mmol) was added. The reaction mixture was stirred magnetically under reflux conditions for 0.5 h. The progress of the reaction was monitored by TLC (eluent: CCl_4/Et_2O: 4/1). The reaction mixture was cooled to room temperature and filtered. The solid material was washed with acetonitrile (20 ml). The combined filtrates were evaporated on a rotary evaporator. The resulting crude material was purified on a silica-gel plate (eluent: CCl_4/Et_2O: 4/1) to afford pure diphenyl disulfide, yield 0.101 g, 92%, mp 58—60 °C, lit,[20] 58—60 °C (Table 2).

Oxidation of Dibenzyl Sulfide to Dibenzyl Sulfoxide as a Typical Procedure for the Conversion of Sulfides to Sulfoxides. In a round-bottom flask (50 ml) equipped with a condenser and a magnetic stirrer, a solution of dibenzyl sulfide (0.214 g, 1 mmol) in acetonitrile (15 ml) was prepared. Benzyltriphenylphosphonium peroxodisulfate (1.796 g, 2 mmol) was added to the solution and the mixture was stirred magnetically under reflux conditions for 2 h. The progress of the reaction was monitored by TLC (eluent: CCl_4/Et_2O: 4/1). The reaction mixture was cooled to room temperature and filtered. The solid material was washed with acetonitrile (20 ml). Work-up was performed as mentioned in the general procedure. Pure dibenzyl sulfoxide was obtained; yield 0.161 g, 70%, mp 132—134 °C, lit,[20] 133—135 °C (Table 2).

Oxidation of 3-Nitrobenzaldehyde Oxime to 3-Nitrobenzaldehyde as a Typical Procedure for the Conversion of Oximes to the Carbonyl Compounds. In a round-bottom flask (50 ml) equipped with a condenser and a magnetic stirrer, a solution of 3-nitrobenzaldehyde oxime (0.166 g, 1 mmol) in acetonitrile (15 ml) was prepared. Benzyltriphenylphosphonium peroxodisulfate (0.898 g, 1 mmol) was added to the solution and the reaction mixture was stirred under reflux conditions for 1 h. The progress of the reaction was monitored by TLC (eluent: CCl_4/Et_2O: 3/1). The reaction mixture was cooled to room temperature and filtered. A solid material was washed with acetonitrile (20 ml). The filtrates were combined together and evaporated. The resulting crude material was purified on a silica-gel plate with an appropriate eluent. Pure 3-nitrobenzaldehyde was obtained; yield 0.136 g, 90%, mp 56—58 °C, lit,[20] 57—59 °C (Table 3).

Oxidation of Aniline to Azobenzene as a Typical Procedure for the Conversion of Aromatic Amines to Azo Compounds. A solution of aniline (0.093 g, 1 mmol) in acetonitrile (10 ml) in a round-bottom flask (50 ml) was prepared. To the resulting solution, the oxidant (0.898 g, 1 mmol) was added and the reaction mixture was stirred magnetically under reflux conditions for 1.25 h. The progress of the reaction was monitored by TLC (eluent: CCl_4/Et_2O: 3/1). The reaction mixture was cooled to room temperature and filtered. The solid material was washed with acetonitrile (20 ml). The filtrate was evaporated and the resulting crude material was purified on a silica-gel plate with an appropriate eluent. Pure azobenzene was obtained; yield 0.086 g, 95%, mp 67—68 °C, lit,[20] 68—69 °C (Table 3).

Oxidation of Diphenylacetic Acid to Benzophenone as a Typical Procedure for the Oxidative Decarboxylation. In a round-bottom flask (50 ml) equipped with a condenser and a magnetic stirrer, a solution of diphenylacetic acid (0.212 g, 1 mmol) in acetonitrile (15 ml) was prepared. Benzyltriphenylphosphonium peroxodisulfate (0.898 g, 1 mmol) was added to the solution and refluxed for 0.5 h. The progress of the reaction was monitored by TLC (eluent: CCl_4/Et_2O: 3/1). The reaction mixture was cooled to room temperature and filtered. The solid material was washed with acetonitrile (20 ml). The filtrates were combined together and evaporated. The resulting crude material was purified on a silica-gel plate with an appropriate eluent. Evaporation of the solvent afforded pure benzophenone; yield 0.155 g, 85%, mp 48—50 °C, lit,[20] 49—50 °C (Table 4).

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