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Selective oxidation of aromatic primary alcohols to aldehydes using molybdenum acetylide oxo-peroxo complex as catalyst

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ABSTRACT

Selective oxidation of various aromatic alcohols to aldehydes has been carried out with very high conversion (90%) and selectivity (90%) for aldehydes using cyclopentadienyl molybdenum acetylide complex, $CpMo(CO)_3(C{\equiv}CPh)$ (**1**) as catalyst and hydrogen peroxide as environmentally benign oxidant. Water-soluble Mo acetylide oxo-peroxo species is formed in situ after reaction of **1** with aqueous hydrogen peroxide during the course of reaction as catalytically active species. Interestingly even though the catalyst is homogeneous it could be recycled very easily by separating the products in organic phase and catalyst in aqueous phase using separating funnel. Even after five recycles no appreciable loss in alcohol conversion and aldehyde selectivity was observed.

Oxidation of alcohols to aldehydes and ketones is one of the most important transformations in organic synthesis.[1] In particular; the oxidation of primary alcohols to aldehydes is important since they find wide applications as intermediates in fine chemicals particularly for perfume industry.[2,3] Traditionally, the oxidation of alcohols is carried out using stoichiometric inorganic oxidants such as permanganate,[4] bromate,[5] or Cr(VI) based reagents which generates large amount of heavy metal waste.[6] Several transition metal-based homogeneous systems such as palladium,[7] ruthenium,[8] manganese,[9] tungsten,[10] rhenium,[11] copper,[12,13] and iron[14] have also been reported. However, mixtures of the organic substrates, products, solvents, and molecular oxygen are well known for being explosion hazards in many cases. Also, some of the oxidation reactions have to be performed under severe conditions, such as high temperature and high oxygen pressure. In many cases the reactions are carried out in environmentally undesired solvents, typically chlorinated hydrocarbons. Due to these limitations, there is significant interest in the development of environmentally benign and safe catalytic route for oxidation of alcohols using dioxygen or hydrogen peroxide as an oxidant and preferably without solvent.[15] Ley et al. have reported oxidation of alcoholic group in azadirachtin derivative to carbonyl group using Dess–Martin periodinane as stoichiometric reagent.[16] They have also used a mixture of immobilized oxidizing reagents such as polystyrene-supported TEMPO, resin-bound chlorite and buffered with immobilized hydrogen phosphate in aqueous acetonitrile containing KBr and sodium hypochlorite, in one pot, for oxidation of alcohols to carboxylic acids, with excellent yields (>90%) of acids. Among the early transition-metal complexes, molybdenum(VI) complexes have functional as well as structural similarity with molybdo-enzymes and have the ability to catalyze a variety of oxidation reactions.[17] Various molybdenum organometallic complexes and oxides are known to be very good homogeneous and heterogeneous catalysts for the oxidation/epoxidation reactions.[18,19] Molybdenum carbonyl complexes with different ligands such as halides,[20] N-containing ligands,[21] and cyclopentadienyl; η^5-C_5R_5; R = H, Me, and Ph[22] are mainly used for epoxidation of a variety of olefins. Recently, we have used molybdenum acetylide complex, $CpMo(CO)_3(C{\equiv}CPh)$; Cp = η^5-C_5H_5 (**1**) as an efficient oxidation catalyst for *cis*-dihydroxylation of various olefins as well as for selective N-oxidation of amines to nitrosocompounds.[23] In continuation of our efforts to investigate the catalytic oxidation of a variety of organic substrates, oxidation of aromatic alcohols has been studied using catalyst **1** and hydrogen peroxide as an oxidant. To our knowledge there are no reports in the literature on the use of the above-mentioned complex for oxidation of alcohol. Here we report the use of complex **1** for selective oxidation of aromatic primary alcohols to aldehydes.

Molybdenum acetylide complex, $CpMo(CO)_3(-C{\equiv}CPh)$ was prepared as per literature method.[24] The oxidant used was hydrogen peroxide and hence to confirm the stability of the catalyst and to study the nature of catalytically active species formed under the reaction conditions, catalyst **1** was treated with H_2O_2 and was

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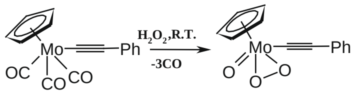
characterized by FTIR. On treatment of the complex **1** with 30% aqueous hydrogen peroxide, corresponding oxo-peroxo Mo(VI) acetylide complex is formed after loss of carbonyl ligands (Scheme 1).[23] This in situ generated Mo-oxo-peroxo acetylide complex was used for oxidation of aromatic primary alcohols with hydrogen peroxide as an oxidant.

Benzyl alcohol was first examined as a model substrate with H_2O_2 (30%) as oxidant. Considering the solubility of the molybdenum-oxo-peroxo species in water, the oxidation reaction was initially carried out without additional solvent in the presence of a catalytic amount of **1**,[25] (Scheme 2, Table 1). As 30% hydrogen peroxide is used as oxidant the remaining 70% water acts as a solvent. The reaction mixture was allowed to reflux at 80 °C in the presence of 1 mol % catalyst for 8 h. The progress of the reaction was monitored by GC. A high conversion of 86% with very high selectivity (92%) for aldehyde was obtained (Table 1, entry 1). Very high turn over number (TON = moles of benzaldehyde formed per mole of the catalyst) of 396 was obtained for benzaldehyde in one cycle. To improve the conversion and selectivity, various other solvents were tried and to our surprise, the conversions and selectivity were considerably lower (entries 2–5) compared to those obtained without additional solvent.

The influence of temperature on benzyl alcohol conversion and product selectivity was examined (Table 2). When reaction temperature was increased gradually from rt to 80 °C, the rate of the reaction increased. Even up to 40 °C, no reaction took place and at 60 °C only 40% conversion was obtained. At 80 °C highest conversion (86%) was obtained after 8 h.

One major advantage with catalyst **1** is the recyclability of the catalyst in spite of being homogeneous complex. After addition of hydrogen peroxide to complex **1,** the in situ formed oxo-peroxo Mo acetylide complex is soluble in aqueous hydrogen peroxide and hence forms miscible phase with alcohol. As the reaction proceeds and the product aldehydes are formed, it forms separate organic phase and hence the catalyst recycle becomes very easy for this system. After completion of the reaction the organic phase (aldehyde) was easily separated using separating funnel and the fresh charge of alcohol was added for the next recycle. The catalyst was recycled efficiently for five cycles (Table 3). The conversion and aldehyde selectivity did not decrease significantly even after five recycles. Hence very easy recycle of this homogeneous catalyst is one of the major achievements in this work.

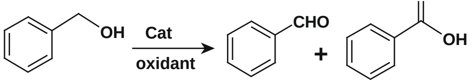
After optimization of the reaction parameters, wider applicability of catalyst 1 was tested for oxidation of a variety of substituted aromatic primary alcohols and the results are summarized in Table 4. In case of *p*-methyl benzyl alcohol best results were obtained with 90% conversion and 90% selectivity for aldehyde. Activated and electron-rich alcohols such as *p*-methyl benzyl alcohol (entry 2) and *p*-methoxy benzyl alcohol (entry 3) gave high conversion and selectivity for aldehyde compounds.



1

1a

Scheme 1. In situ generation of oxo-peroxo Mo(VI) acetylide complex.



Scheme 2. Oxidation of aromatic alcohol.

Table 1
Benzyl alcohol oxidation in various solvents[a]

Entry	Solvent	Conv (%)	Selectivity (%)		TON[b]
			Aldehyde	Acid	
1	No solvent[c]	86	92	8	395.6
2	Acetonitrile	45.2	85.2	14.8	38.5
3	*t*-Butanol	22.6	80.5	19.5	18.1
4	Methanol	17.4	45.9	54.1	8
5	Chlorobenzene	23.8	86.9	13.1	20.7

[a] Reaction conditions: benzyl alcohol—0.01 mole; 30% H_2O_2—0.02 mole, solvent—10 g, catalyst **1**—0.1 mmol; time 8 h; temp 80 °C.
[b] TON for aldehyde = moles of aldehyde formed per mole of catalyst.
[c] Benzyl alcohol 0.05 mol, 30% H_2O_2 0.1 mol.

Table 2
Effect of temperature on benzyl alcohol oxidation[a]

Entry	Temperature (°C)	Conv (%)	Selectivity (%)	
			Aldehyde	Acid
1	RT	0	0	0
2	40	0	0	0
3	60	40	100	0
4	80	86	92	8

[a] Reaction conditions: benzyl alcohol—0.05 mole; 30% H_2O_2—0.1 mole, catalyst **1**—0.1 mmol; time 8 h.

Table 3
Catalyst-recycle studies[a]

Entry	Run	Conv (%)	Selectivity (%)	
			Aldehyde	Acid
1	0	86	92	8
2	1	85.8	93	7
3	2	85.3	93	7
4	3	84.9	92	8
5	4	84.6	92	8
6	5	84.2	92	8

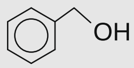
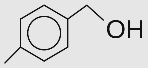
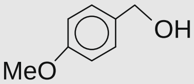
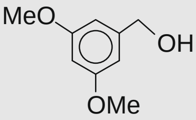
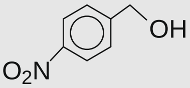
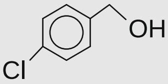
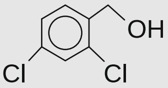
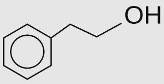
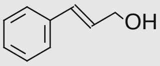
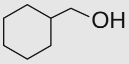
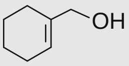
[a] Reaction conditions: benzyl alcohol—0.05 mole; 30% H_2O_2—0.1 mole, catalyst **1**—0.1 mmol; time 8 h.

Lower conversion (60%) was obtained for benzyl alcohol with electron-withdrawing substituents such as *p*-nitro, *p*-chloro, and 2,4-dicholoro benzyl alcohols (entries 5–7). Additionally to test the oxidation of alcohol in presence of other functional groups such as double bond, cinnamyl alcohol oxidation was tested (entry 9). In cinnamyl alcohol, alcohol group was selectively oxidized without oxidation of double bond with 70% conversion and 75% selectivity for aldehyde. The lower selectivity for aldehyde may be due to presence of double bond that makes further oxidation of aldehyde to acid more favorable. When non-aromatic alcohols such as cyclohexanemethanol and cyclohexenemethanol (entries 10 and 11) were oxidized, 82% conversion with more than 85% selectivity for aldehyde was obtained. However when aliphatic alcohols such as *n*-propanol, *n*-butanol, and *n*-octanol were oxidized using catalyst **1**, no reaction took place.

Uemira et al. described the palladium-catalyzed oxidation of alcohols to aldehydes and ketones using a 5 mol % $Pd(OAc)_2$, pyridine and 3 Å molecular sieves (MS3A) under atmospheric pressure of oxygen with 100% conversion and 92% selectivity. This system has given very high efficiency though using high loading of expensive Pd catalyst and needs an additive.[7]

Sheldon et al. have used various copper salts in combination with different N-containing ligands, TEMPO, and a base as co-catalyst, for oxidation of a series of primary alcohols to aldehydes

Table 4
Oxidation of different alcohols[a]

Entry	Substrate	% conv	Selectivity (%)		TON	Yield (%)
			Aldehyde	Acid		
1		86	92	8	396	79
2		90	87	13	391	78
3		90	90	10	392	90
4		83	85	15	352	71
5		60	88	12	264	53
6		65	91	9	296	62
7		78	90	10	343	70
8		72	82	18	295	59
9		70	75	25	262	52
10		82	88	12	361	72
11		82	85	15	348	68

[a] Reaction conditions: benzyl alcohol—0.05 mole; 30% H_2O_2—0.1 mole, catalyst **1**—0.01 mmol; time 8 h.

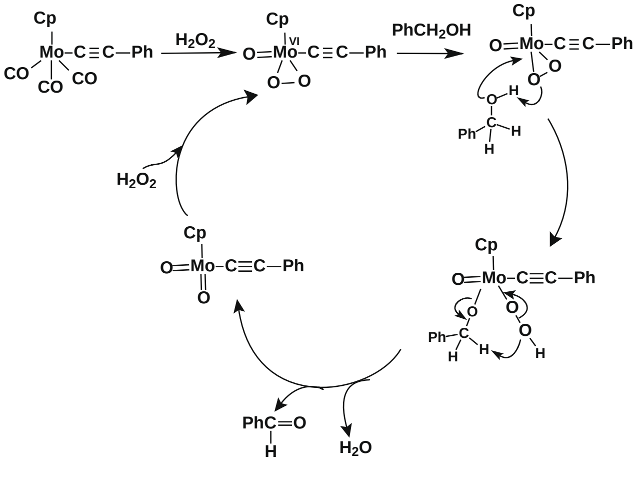
using air as oxidant under very mild reaction conditions. In case of benzyl acohol 100% conversion was obtained in 2.5 h whereas 61% conversion was obtained in 24 h for octanol. Even for 100% alcohol conversion the TON obtained in this case was ~20.[12] Ragauskas et al. have also used copper catalyst for oxidation of primary as well as secondary alcohols. A multi-component system consisting of $Cu(ClO_4)_2$, TEMPO, TMDP as well as DABCO has been used for room temperature oxidation of alcohols to carbonyl compounds using oxygen as oxidant with good to excellent yields. The conversions ranged from 98% to 11% for various alcohols with corresponding reaction times varying from 2 to 24 h, respectively. The maximum TON obtained for this system was 25. This catalyst system was recycled three times; however during the recycle 8 to 17% decrease in the conversion was observed due to leaching of copper, hence it is not a very efficient recyclable catalytic system.[24,13] Both the copper-catalyzed alcohol oxidations need a multi-component system involving co-catalyst, base, expensive reagent TEMPO along with an oxidant whereas the present study involves a simple reaction system with catalyst and hydrogen peroxide as oxidant apart from substrate alcohol. The TON in the present system is very high; more than 10-fold (262–396) compared to those in both the copper systems (~25). Minisci et al. have reported oxidation of various benzylic alcohols to aldehydes and ketones using molecular oxygen as oxidizing agent and *N*-hydroxyphthalimide (NHPI) and Co(II) salts as catalytic system at room temperature with very high conversion (75–100%) and selectivity (>90%) for aldehydes as well as oxidation of primary and secondary alcohols (benzylic and aliphatic) to aldehydes or ketones using Mn(II)–Co(II) or Mn(II)–Cu(II) nitrates in combination with TEMPO in acetic acid solvent using air or oxygen as oxidant with >95% yield.[26,27]

Hou et al. described the use of Fe-coordinated polymers for oxidation of alcohols and the result showed excellent selectivity, though the recycle of the catalyst is not very efficient. Martın et al. have carried out aerobic oxidation of secondary and benzylic alcohols with very high efficiency using binary catalyst system $Fe(NO_3)_3$–$FeBr_3$ under air at room temperature with good yield (75%).[14]

Mo-oxo-peroxo complex with quinone as ligand has been used by Bhattacharya et al. for oxidation of alcohols as well as sulfides with varying TONs ranging from 84 to 1780 for various alcohols using oxygen, hydrogen peroxide, or mixture of oxygen and H_2O_2 as oxidant. Though very small amount of the catalyst is used in this paper, no recycle is reported for the catalytic system.[19]

The mechanism for the oxidation of various alcohols to aldehydes using catalyst **1** has been proposed (Scheme 3) by comparison with the literature reports.[28,29] Molybdenum acetylide complex **1** on treatment with H_2O_2 forms corresponding oxo-peroxo complex which was well characterized before.[23a] When alcohol molecule approaches the catalyst the Mo-peroxo ring opens by abstracting alcohol –OH proton by peroxo moiety. The carbon proton abstraction followed by liberation of water molecule and aldehyde leads to the formation of Mo-dioxo complex which after further treatment with H_2O_2 regenerates the Mo-oxo-peroxo species.

The results show that molybdenum acetylide complex **1** is very efficient catalyst for the oxidation of a variety of primary aromatic alcohols to aldehydes with maximum selectivity. It can very well tolerate electron-rich as well as electron-withdrawing substituents on aromatic ring. Water soluble nature of the catalytically active species, oxo-peroxo molybdenum acetylide moiety and formation of organic phase of the product (aldehyde) have enabled very easy and efficient recycle of this homogeneous catalyst even up to five



Scheme 3. Possible mechanism of alcohol oxidation.

recycles without appreciable loss in conversion and aldehyde selectivity. This system has a number of advantages compared to the literature reports, such as simple catalyst system, high TON as well as recyclability of the catalyst.

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References and notes

1. (a) March, J. *Advanced Organic Chemistry: Reactions, Mechanisms, and Structure*; Wiley: New York, 1992; (b) Sheldon, R. A.; Kochi, J. K. *Metal-Catalyzed Oxidation of Organic Compounds*; Academic Press: New York, 1981; (c) Brink, G. T.; Arends, I. W. C. E.; Sheldon, R. A. *Science* **2000**, *47*, 1636.
2. (a) Enache, D. I.; Edwards, J. K.; Landon, P.; Solsona-Espriu, B.; Carley, A. F.; Herzing, A. A.; Watanabe, M.; Kiely, C. J.; Knight, D. W.; Hutchings, G. J. *Science* **2006**, *311*, 362; (b) Beller, M.; Bolm, C. *Transition Metals for Organic Synthesis*, 2nd ed.; Verlag GmbH & Co. KGaA: Weinheim, Germany, 2004.
3. (a) Pillai, U. R.; Sahle-Demessie, E. *Appl. Catal. Gen* **2003**, *245*, 103; (b) Hudlicky, M. *Oxidations in Organic Chemistry*; American Chemical Society: Washington, DC, 1990.
4. Menger, F. M.; Lee, C. *Tetrahedron Lett.* **1981**, *22*, 1655.
5. Lee, C. K.; Koo, B. S.; Lee, Y. S.; Cho, H.; Lee, K. K.-J. *Bull. Korean Chem. Soc.* **2002**, *23*, 1667.
6. For a general review on chromium oxidations, see for example: Cainelli, G.; Cardillo, G. *Chromium Oxidants in Organic Chemistry*; Springer: Berlin, 1984; Ley, S. V.; Madin, A.. In *Comprehensive Organic Synthesis*; Trost, B. M., Fleming, I., Ley, S. V., Eds.; Pergamon: Oxford, 1991; Vol. 7, p 251.
7. (a) Seddon, K. R.; Stark, A. *Green Chem.* **2002**, *4*, 119; (b) Ganchegui, B.; Bouquillon, S.; Henin, F.; Muzart, J. *Tetrahedron Lett* **2002**, *43*, 6641; (c) Muzart, J. *Tetrahedron* **2003**, *59*, 5789; (d) Ganchegui, B.; Bouquillon, S.; Henin, F.; Muzart, J. *J. Mol. Catal. A: Chem.* **2004**, *214*, 65; (e) Hardacre, C.; Mullan, E. A.; Rooney, D. W.; Thompson, J. M. *J. Catal.* **2005**, *232*, 355; (f) Nishimura, T.; Onoue, T.; Ohe, K.; Uemura, S. *Tetrahedron Lett.* **1998**, *39*, 6011; (g) Nagata, H.; Ogasawara, K. *Tetrahedron Lett.* **1999**, *40*, 6617; (h) ten Brink, G.-J.; Arends, I. W. C. E.; Sheldon, R. A. *Science* **2000**, *287*, 1636; (i) Besson, M.; Callezot, P. *Catal. Today* **2000**, *57*, 127; (j) Nishimura, T.; Kakiuchi, N.; Inoue, M.; Uemura, S. *Chem. Commun.* **2000**, 1245; (k) Kakiuchi, N.; Maeda, Y.; Nishimura, T.; Uemura, S. *J. Org. Chem.* **2001**, *66*, 6620; (l) Peterson, K. P.; Larock, R. C. *J. Org. Chem.* **1998**, *63*, 3185.
8. (a) Wolfson, A.; Wuyts, S.; De, V. D.; Vankelecom, I. F. J.; Jacobs, P. A. *Tetrahedron Lett.* **2002**, *43*, 8107; (b) Tang, W. M.; Li, C. J. *Acta Chim. Sini.* **2004**, *62*, 742; (c) Roberto, F. D. S.; Jairton, D.; Jeane, E. D. *J. Braz. Chem. Soc.* **2006**, *17*, 48; (d) Shi, F.; Tse, M.; Beller, M. *Chem. Asian J.* **2007**, *2*, 411.
9. (a) Chhikara, B. S.; Tehlan, S.; Kumar, A. *Synlett* **2005**, 63; (b) Chhikara, B. S.; Chandra, R.; Tandon, V. *J. Catal.* **2005**, *230*, 436.
10. Bianchini, G.; Crucianelli, M.; de Angelis, F.; Neri, V.; Saladino, R. *Tetrahedron Lett.* **2005**, *46*, 2427.
11. Kumar, A.; Jain, N.; Chauhan, S. M. S. *Synlett* **2007**, 411.
12. Gamez, P.; Arends, I. W. C. E.; Sheldon, R. A.; Reedijk, J. *Adv. Synth. Catal.* **2004**, *346*, 805.
13. Jiang, N.; Ragauskas, A. J. *J. Org. Chem.* **2006**, *71*, 7087.
14. (a) Han, H.; Zhang, S.; Hou, H.; Fan, Y.; Zhu, Y. *Eur. J. Inorg. Chem.* **2006**, 1594; (b) Martın, S. E.; Suarez, D. F. *Tetrahedron Lett.* **2002**, *43*, 4475.
15. Veitch, G. V.; Boyer, A.; Ley, S. V. *Angew. Chem., Int. Ed.* **2008**, *47*, 9402.
16. Yasuda, K.; Ley, S. V. *J. Chem. Soc., Perkin Trans. 1* **2002**, 1024.
17. (a) Khanbabaee, K. K. *Ann. Chem.* **1993**, 905; (b) Krohn, K.; Bruggmann, K.; Doring, D.; Jones, P. G. *Chem. Ber.* **1992**, *125*, 2439; (c) Krohn, K.; Rieger, H.; Bruggmann, K. *Synthesis* **1990**, 1141; (d) Patnaik, Pradyot *Handbook of Inorganic Chemicals*; McGraw Hill, 2003.
18. Manyar, H. G.; Chaure, G. S.; Kumar, A. *J. Mol. Catal. A* **2006**, *243*, 244.
19. Maiti, S. K.; Abdul Malik, K. M.; Bhattacharyya, R. *Inorg. Chem. Commun.* **2004**, *7*, 823.
20. Abrantes, M.; Santos, A. M.; Mink, J.; Kuhn, F. E.; Romao, C. C. *Organometallics* **2003**, *22*, 2112.
21. Grivani, G.; Tangestaninejad, S.; Habibi, M. H.; Mirkhani, V. *Catal. Commun.* **2005**, *6*, 375.
22. Martins, A. M.; Romao, C. C.; Abrantes, M. M.; Azevedo, C.; Cui, J.; Dias, A. R.; Duarte, M. T.; Lemos, M. A.; Lourenco, T.; Poli, R. *Organometallics* **2005**, *24*, 2582.
23. (a) Biradar, A. V.; Sathe, B. R.; Dongare, M. K.; Umbarkar, S. B. *J. Mol. Catal. A* **2008**, *285*, 111; (b) Biradar, A. V.; Kotbagi, T. V.; Dongare, M. K.; Umbarkar, S. B. *Tetrahedron Lett.* **2008**, *49*, 3616.
24. Bruce, M. I.; Humphrey, M. G.; Matisons, J. G.; Roy, S. K.; Swincer, A. G. *Aust. J. Chem.* **1984**, *37*, 1955. A mixture of $CpMo(CO)_3Cl$ (2.0 g, 0.0071 mol), ($H–C\equiv CPh$) (1.05 g, 0.010 mol), and catalytic amount of CuI (5 mg) was stirred at room temperature in diethyl amine (50 mL) for 15 min. The solvent was removed in vacuum and purified by column chromatography on silica gel using hexane/dichloromethane 80:20 v/v mixture as solvent system. Yield of $CpMo(CO)_3(–C\equiv CPh)$ = 1.5 g, 68.1% based on $CpMo(CO)_3Cl$. The compound was confirmed using FTIR spectroscopy. $\nu_{(CO)}$, 1940, 2031 cm^{-1}; $\nu_{(C\equiv C)}$, 2102 cm^{-1}. NMR in $CDCl_3$: δ (^1H); (s, 5H, Cp), 7.15–7.38 (m, 5H, Ph); δ (^{13}C); 92.93 (Cp), 87.85. 126.01, 127.0, 129.39, 130.8, 130.87 ($C\equiv CPh$); 222.4, 238.78 (CO).
25. In a typical experiment, benzyl alcohol (5.41 g, 0.05 mol), **1** (34.5 mg, 0.01 mmol), and 30% H_2O_2 (10.02 g, 0.1 mol) were added to a two-necked 50 mL round-bottomed flask. The reaction mixture was vigorously stirred (750 rpm) at 80 °C for 8 h. After the reaction, a two-layer mixture was obtained (organic phase/water phase), The reaction was monitored using GC (Agilent 6890 Gas Chromatograph equipped with a HP-5 dimethyl polysiloxane (60 m length, 0.25 mm internal diameter, 0.25 μm film thickness) with flame ionization detector. Products were confirmed using GC–MS (Model GC Agilent 6890 N with HP5 MS 30 m capillary column, MS Agilent 5973 Network MSD.
26. Minisci, F.; Punta, C.; Recupero, F.; Fontana, F.; Pedulli, G. F. *Chem. Commun.* **2002**, 688.
27. Cecchetto, A.; Fontana, F.; Minisci, F.; Recupero, F. *Tetrahedron Lett.* **2001**, *42*, 6651.
28. Hida, T.; Nogusa, H. *Tetrahedron* **2009**, *65*, 270.
29. Maiti, S. K.; Banerjee, S.; Mukharjee, A. K.; Malik, K. M. A.; Bhattacharyya, R. *New. J. Chem.* **2005**, *29*, 554.