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$AlCl_3.6H_2O$ as a Catalyst for Simple and Efficient Synthesis of *gem*-Dihydroperoxides from Ketones and Aldehydes using Aqueous H_2O_2

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$AlCl_3.6H_2O$ was explored as an efficient catalyst for the synthesis of *gem*-dihydroperoxides (DHPs) from ketones and aldehydes. The reactions took place within a short period of time using (30%) aqueous H_2O_2 as a "green" oxidant in acetonitrile under neutral conditions at room temperature to afford the products in high yields.

Keywords: *gem*-Dihydroperoxides, Aluminium chloride hexahydrate, $AlCl_3.6H_2O$, Ketones, Aldehydes, Aqueous H_2O_2

INTRODUCTION

gem-Dihydroperoxides (DHPs) are stable and versatile derivatives of ketones and aldehydes [1]. The rapidly increasing interest in *gem*-dihydroperoxides stems from their relevance to peroxidic antimalarial drugs [2-7], and the presence of *gem*-peroxy linkage in *gem*-peroxyketals as a salient structural feature [8-10], shared by many known antimalarial cyclic organic peroxides [11-21]. Also, these compounds have been used as useful precursors in the synthesis of different peroxides including tetraoxanes [22-27], and their analogues such as silatetraoxanes [28], spirobisperoxyketals [29] and 1,2,4,5-tetraoxacycloalkanes [30]. Very recently, *gem*-DHPs have found applications as initiators in polymerization reactions [31,32], as reagents for epoxidation of *α,β*-unsaturated ketones [33], and oxidation of different organic molecules [34] such as sulfides [35,36]. Recently, *gem*-dihydroperoxides have been reported as effective reagents for enantioselective epoxidation of 2-substituted 1,4-naphthoquinones [37].

Synthetic methods to synthesize *gem*-dihydroperoxides have been reviewed [1]. Common routes include (i) ozonolysis of ketone enol ethers or *α*-olefines in the presence of aqueous H_2O_2 [8,30,38]; (ii) treatment of ketals and ketones with H_2O_2 utilizing acid catalysts such as tungstic acid [14], BF_3/Et_2O [16,39], and HCl [26,40], albeit 4-*tert*-butylcyclohexyl-1,1-dihydroperoxide has been synthesized under neutral conditions catalyzed by methyltrioxorhenium ($MeReO_3$) in trifluoroethanol [2], and (iii) peroxidation of ketones in an acidic solvent, *e.g.* AcOH [12,41], or hydroperoxide rearrangement of bicyclic alcohols [42]. However, these methods suffer from certain drawbacks such as longer reaction times, use of concentrated H_2O_2 and excess acid, non-recoverable catalysts, low yields, poor selectivity of ozonolysis and the presence of ozone-sensitive groups in the substrates.

A more general way has been reported for the synthesis of *gem*-DHPs from ketones and aromatic aldehydes using aqueous H_2O_2 (30%) and iodine as the catalyst in acetonitrile [19,43]. Das *et al*. [18] reported an efficient method for the synthesis of *gem*-DHPs from ketones and aldehydes by aqueous H_2O_2 (50%) under catalytic effect of ceric ammonium nitrate (CAN). For primary *gem*-DHPs this method is, however, limited to electron-rich aromatic aldehydes. A number of other recently reported synthetic routes to *gem*-DHPs include the use of 50% H_2O_2 solution under acid catalysis in THF [13], and rhenium(VII) oxide in acetonitrile

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[20]. Moreover, silicon-supported sodium hydrogen sulfate [15], phosphomolybdic acid [44], and camphorsulfonic acid [17], have been reported for the synthesis of primary *gem*-DHPs from ketones and aldehydes.

EXPERIMENTAL

Solvents, reagents, and chemical materials were obtained from Aldrich and Merck chemical companies and purified prior to use. Melting points were determined in open capillary tubes in a Stuart SMP_3 apparatus and uncorrected. Nuclear magnetic resonance spectra were recorded on JEOL FX 90Q using tetramethylsilane (TMS) as an internal standard. Infrared spectra were recorded on a PerkinElmer GX FT IR spectrometer (KBr pellets).

Caution: Although we did not encounter any problem with these reactions, peroxidic compounds are potentially explosive and should be handled with some caution; all reactions should be carried out behind a safety shield inside a fume hood and transition metal salts or heating should be avoided.

General Procedure for the Synthesis of *gem*-Dihydroperoxides 2

A mixture of carbonyl substrates **1** or **3** (1 mmol), 30% aqueous H_2O_2 (3 ml) and $AlCl_3.6H_2O$ (0.1 mmol) in MeCN (4 ml) was stirred at room temperature for 3-10 h (Table 1). After completion of the reaction (TLC), the mixture was diluted with water (5 ml) and extracted with EtOAc (3 × 5 ml). The combined organic layer was washed with saturated aqueous sodium bicarbonate solution (3 ml), dried over anhydrous Na_2SO_4 and concentrated in vacuo. The residue was purified by silica-packed column chromatography (hexane-EtOAc) to afford pure *gem*-dihydroperoxides **2** (or hydroxyl-hydroperoxides **4**) (Table 1). The products were characterized on the basis of their physical properties and spectral (^1H, ^{13}C NMR and IR) data which were in accord with those reported in the literature.

Spectral Data of New Compounds

2,2-Dihydroperoxy-1,7,7-trimethyl-bicyclo[2.2.1] heptane (2g). Colorless oil; IR (KBr, cm^{-1}): 3473 (O-H stretching of hydroperoxy groups), 2973, 2853, 1589, 1448, 1385, 1268, 1146, 1017; ^1H NMR (FT-90 MHz, $CDCl_3$, δ (ppm)): 4.45 (2H, brs, -OOH), 2.46-0.72 (16H, m, aliph-H); ^{13}C NMR (22.5 MHz, $CDCl_3$, δ (ppm)): 114.2, 52.0, 45.4, 39.5, 37.0, 24.6, 23.5, 21.0, 8.6; Anal. Calcd. for $C_{10}H_{18}O_4$: C, 59.40; H, 8.91%. Found: C, 59.34; H, 8.87; FABMS: *m/z* 225 $[M+Na]^+$.

3-Bromo-2,2-dihydroperoxy-1,7,7-trimethyl-bicyclo [2.2.1]heptane (2h). Colorless solid; m.p.: 68-70 ºC; IR (KBr, cm^{-1}): 3410 (O-H stretching of hydroperoxy groups), 2956, 2853, 1618, 1478, 1384, 1316, 1205, 1034, 910, 768; ^1H NMR (FT-90 MHz, $CDCl_3$, δ (ppm)): 4.56 (2H, brs, -OOH), 2.27-0.91 (15H, m, aliph-H); ^{13}C NMR (22.5 MHz, $CDCl_3$, δ (ppm)): 120.3, 54.0, 46.8, 40.0, 38.3, 26.6, 24.7, 22.4, 9.6; Anal. Calcd. for $C_{10}H_{17}BrO_4$: C, 42.70; H, 6.05%. Found: C, 42.63; H, 5.97%; FABMS: *m/z*: 304 $[M+Na]^+$.

Methyl-(naphthalen-1-yl)-1,1-dihydroperoxide (2k). Colorless oil; IR (KBr, cm^{-1}): 3324, 3052 (O-H stretching of hydroperoxy groups), 2922, 2853, 1594, 1573, 1508, 1461, 1356, 1279, 1240, 1192, 1128, 941, 863, 802, 775, 591; ^1H NMR (FT-90 MHz, $CDCl_3$, δ (ppm)): 8.83-8.75 (2H, brs, -OOH), 8.10-7.20 (7H, m, Ar-H), 2.66 (3H, s, CH_3); ^{13}C NMR (22.5 MHz, $CDCl_3$, δ (ppm)): 136.0, 134.4, 131.1, 129.9, 127.8, 126.6, 125.5, 123.0, 107.0, 20.5; Anal. Calcd. for $C_{12}H_{12}O_4$: C, 65.45; H, 5.45%. Found: C, 65.42; H, 5.40%; FABMS: *m/z* 243 $[M+Na]^+$.

Benzoyl-phenyl-1,1-dihydroperoxide (2n). Colorless solid; m.p.: 118-120 ºC; IR (KBr, cm^{-1}): 3427, 3071 (O-H stretching of hydroperoxy groups), 2841, 2699, 2559, 1688 (C=O streching), 1603, 1583, 1454 (C=C stretching of aromatic ring), 1384, 1292, 1128, 1072, 1026, 934, 809, 700, 667; ^1H NMR (FT-90 MHz, $CDCl_3$, δ (ppm)): 8.81 (2H, brs, -OOH), 8.10-7.95 (4H, m, Ar-H), 7.50-7.10 (6H, m, Ar-H); Anal. Calcd. for $C_{14}H_{12}O_5$: C, 64.61; H, 4.61%. Found: C, 64.54; H, 4.57%; FABMS: *m/z* 283 $[M+Na]^+$.

(4-Bromophenyl)methylene-1,1-dihydroperoxide (4q). Colorless solid; m.p.: 88-90 ºC; IR (KBr, cm^{-1}): 3426, 3085 (O-H stretching of hydroperoxy groups), 2909, 1608, 1528, 1411 (C=C stretching of aromatic ring), 1353, 1236, 1195, 1083, 973, 856, 828, 753, 706, 601; ^1H NMR (FT-90 MHz, $CDCl_3$, δ (ppm)): 9.96 (2H, brs, -OOH), 7.90-7.00 (4H, m, Ar-H), 6.26 (1H, s, CH); ^{13}C NMR (22.5 MHz, $CDCl_3$, δ (ppm)): 141.0, 132.5, 130.5, 120.0, 111.0; Anal. Calcd. for $C_7H_7BrO_4$: C, 35.74; H, 2.97%. Found: C, 35.72; H, 2.94%; FABMS: *m/z* 258 $[M+Na]^+$.

Table 1. Effects of Catalyst and Solvent in the Synthesis of *gem*-DHPs[a]

Entry	Ketone 1/Aldehyde 3	Catalyst	Solvent	Time (h)	Yield (%)[b]
1	3-Pentanone	$AlCl_3.6H_2O$	CH_3CN	3	96
2	3-Pentanone	$AlCl_3.6H_2O$	CH_2Cl_2	5	82
3	3-Pentanone	$AlCl_3.6H_2O$	Et_2O	8	60
4	3-Pentanone	$AlCl_3.6H_2O$	AcOEt	5	87
5	3-Pentanone	$SbCl_3$	CH_3CN	8	48
6	3-Pentanone	CeO_2	CH_3CN	10	45
7	3-Pentanone	$CrCl_3.6H_2O$	CH_3CN	8	75
8	3-Pentanone	KF-Al_2O_3	CH_3CN	10	Trace
9	Cyclohexanone	$AlCl_3.6H_2O$	CH_3CN	3	98
10	Cyclohexanone	$SbCl_3$	CH_3CN	7	55
11	Cyclohexanone	CeO_2	CH_3CN	8	50
12	Cyclohexanone	$CrCl_3.6H_2O$	CH_3CN	6	70
13	Cyclohexanone	KF-Al_2O_3	CH_3CN	10	Trace
14	Acetophenone	$AlCl_3.6H_2O$	CH_3CN	8	56
15	Acetophenone	$SbCl_3$	CH_3CN	12	23
16	Acetophenone	CeO_2	CH_3CN	12	15
17	Acetophenone	$CrCl_3.6H_2O$	CH_3CN	10	28
18	Acetophenone	KF-Al_2O_3	CH_3CN	20	0
19	Benzaldehyde	$AlCl_3.6H_2O$	CH_3CN	8	67
20	Benzaldehyde	$SbCl_3$	CH_3CN	15	32
21	Benzaldehyde	CeO_2	CH_3CN	15	15
22	Benzaldehyde	$CrCl_3.6H_2O$	CH_3CN	12	22
23	Benzaldehyde	KF-Al_2O_3	CH_3CN	20	0

[a]Conditions: ketone and aldehyde (1 mmol), solvent (4 ml), catalyst (0.1 mmol), 30% aq. H_2O_2 (3 ml), reactions are carried out at rt. [b]Isolated yields.

(4-Flourophenyl)methylene-1,1-dihydroperoxide (4s). Colorless solid; m.p.: 110-112 ºC; IR (KBr, cm^{-1}): 3464, 3082 (O-H stretching of hydroperoxy groups), 2905, 1625, 1601, 1564, 1453 (C=C stretching of aromatic ring), 1353, 1303, 1071, 1025, 844, 720, 685; ^{1}H NMR (FT-90 MHz, $CDCl_3$, δ (ppm)): 9.21 (2H, brs, -OOH), 8.14-7.14 (4H, m, Ar-H), 6.14 (1H, s, CH); ^{13}C NMR (22.5 MHz, $CDCl_3$, δ (ppm)): 161.62, 137.0, 128.5, 118.5, 112.0; Anal. Calcd. for $C_7H_7FO_4$: C, 48.27; H, 4.02%. Found: C, 48.23; H, 3.97%; FABMS: *m/z* 197 $[M+Na]^+$.

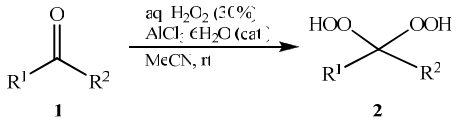
(2-Methoxyphenyl)methylene-1,1-dihydroperoxide (4u). Colorless oil; IR (KBr, cm^{-1}): 3226, 3085 (O-H stretching of hydroperoxy groups), 2853, 1647, 1603, 1493, 1465 (C=C stretching of aromatic ring), 1372, 1245, 1177, 1017, 844, 758; ^{1}H NMR (FT-90 MHz, $CDCl_3$, δ (ppm)): 9.35 (2H, brs, -OOH), 8.18-6.84 (4H, m, Ar-H), 6.04 (1H, s, CH), 4.05 (3H, s, OCH_3); ^{13}C NMR (22.5 MHz, $CDCl_3$, δ (ppm)): 157.4, 139.1, 128.5, 123.0, 118.5, 102.0, 57.5; Anal. Calcd. for $C_8H_{10}O_5$: C, 51.61; H, 5.37%. Found: C, 51.57; H, 5.34%; FABMS: *m/z* 209 $[M+Na]^+$.

RESULTS AND DISSCUTION

In our ongoing research for making developments in the synthesis of *gem*-DHPs [45], and their applications in various transformations [36,46,47], herein, we introduce $AlCl_3.6H_2O$ as another new and highly efficient catalyst to effect the synthesis of *gem*-DHPs from ketones and aldehydes employing aqueous H_2O_2 (30%) at room temperature.

The effects of catalysts and solvents were investigated in order to obtain suitable reaction conditions in terms of yield and reaction time for the conversion of the ketones and

aldehydes into their corresponding DHPs. Various catalysts and solvents were examined using 3-pentanone, cyclohexanone, acetophenone, and benzaldehyde as test compounds in aqueous H_2O_2 (30%) at room temperature. The obtained results are summarized in Table 1. As could be seen in this table, the best results emerged when $AlCl_3.6H_2O$ (10 mol%) was employed as a catalyst using MeCN as the solvent of choice. It appears that, the solvent polarity plays important role in the synthesis of DHPs. The other catalysts such as $SbCl_3$, CeO_2 and $CrCl_3.6H_2O$ resulted in moderate to low yields while KF-Al_2O_3 was found quite unsuitable for this synthesis.



Scheme 1

To develop the scope of the reaction, we determined to extend this reaction to a variety of other cyclic and acyclic aliphatic and aromatic ketones **1a-n** and aldehydes **1o-v** under the optimized conditions (rt, 0.1 mol% catalyst, in MeCN) (Scheme 1). The experimental results are summarized in Table 2. The *gem*-dihydroperoxides **2a-k** corresponding to the

Table 2. Synthesis of *gem*-Dihydroperoxides with $AlCl_3.6H_2O$ (cat.)/30% aq. H_2O_2[a,b]

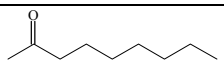
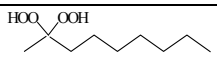
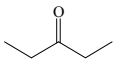
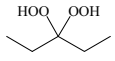
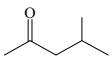
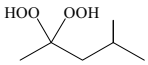
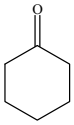
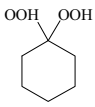
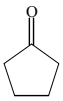
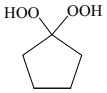
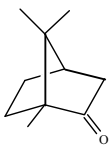
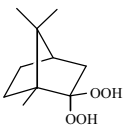
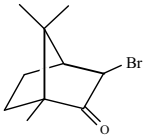
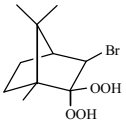
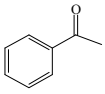
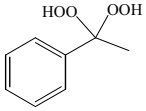
Entry	Ketone **1**/Aldehyde **3**	Product[c] **2** or **4**	Time (h)	Yield (%)[d]	Ref.
a			4 (5)	97 (95)	[43]
b			3 (4)	96 (93)	[18]
c			3.5 (4)	95 (92)	[18]
d			3 (5)	98 (90)	[15]
e			3 (4)	97 (94)	[44]
f			3 (4)	93 (90)	[20]
g			4	68	new
h			5	58	new
i			8 (11)	56 (45)	[18]

Table 2. Continued

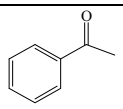
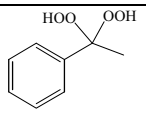
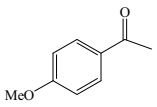
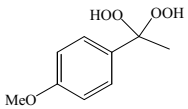
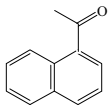
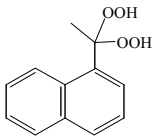
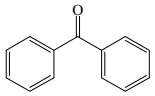
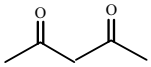
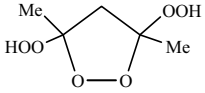
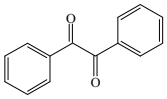
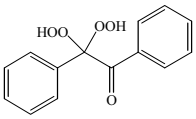
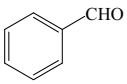
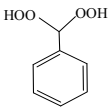
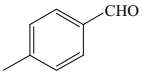
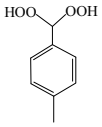
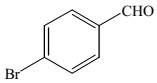
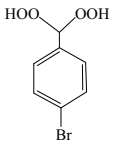
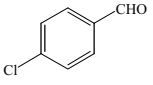
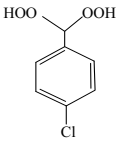
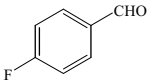
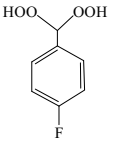
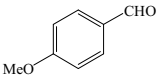
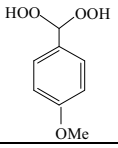
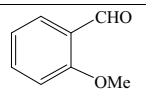
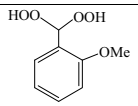
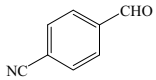
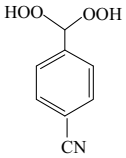
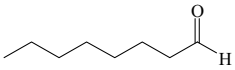
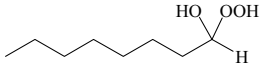
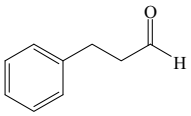
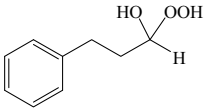
i			8 (11)	56 (45)	[18]
j			7.5 (9)	82 (62)	[19]
k			4	95	new
l		-	12	-	-
m			10	87	[36]
n			12	75	new
o			8 (12)	67 (50)	[18]
p			10 (12)	64 (52)	[43]
q			9	85	new
r			8 (10)	80 (75)	[43]
s			11	84	new
t			8.5 (10)	78 (60)	[44]

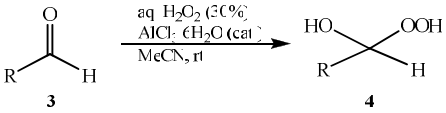
Table 2. Continued

u			10 (10)	75 (60)	new
v			10 (11)	82 (70)	[45]
w			4 (5)	93 (90)	[48]
x			5 (6)	95 (88)	[48]

[a]Conditions: ketone and aldehyde (1 mmol), acetonitrile (4 ml), $AlCl_3.6H_2O$ (0.1 mmol), 30% aq. H_2O_2 (3 ml), reactions are carried out at rt. [b]The yields and reaction times obtained under similar conditions using $SnCl_2.3H_2O$ catalyst [45] are quoted in the parentheses. [c]The structures of the products were established from their physical properties and spectral (^1H, ^{13}C NMR and MS) analysis and compared with the literature. [d]Isolated yield.

ketones afforded high to excellent yields (56-98%) within 3-8 h (Table 2). However, under the same reaction condition, no conversion to *gem*-DHP was observed for benzophenone **1k** and it was recovered almost intact after 12 h. This, possibly, can be accounted for by the strong resonance stabilization and steric effects exerted by the presence of two phenyl groups. In this reaction, acetylacetone **1m** gave *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (87%) [36], while only one of the two carbonyl groups present in dibenzoyl **1n** underwent conversion to corresponding dihydroperoxide **2n** (75%). Similarly, the aromatic aldehydes **1o-v** were converted to their corresponding *gem*-DHPs **2o-v** in (64-85%) yield (Table 1).

As we previously noted [45], and is also reported by Rieche [48] and others [49], this method proved unsuitable for converting aliphatic aldehydes such as octanal **3w** and hydrocinnamaldehyde **3x** into their corresponding *gem*-DHPs under the same reaction conditions which transferred benzaldehyde into *gem*-DHP. Instead, these compounds were both converted into their corresponding hydroxyl-hydroperoxides **4w** and **4x** respectively (Scheme 2, Table 1),



Scheme 2

that is, the addition of just one molecule of hydrogen peroxide to the carbonyl group has occurred.

As shown in Table 2, $AlCl_3.6H_2O$ appears to catalyze these reactions more efficiently to furnish the respective *gem*-DHPs in considerably higher yields and shorter reaction times than $SnCl_2.3H_2O$ catalyst reported in our previous work [45]. Moreover, $AlCl_3.6H_2O$ is believed to be environmentally more friendly and benign in comparison with $SnCl_2.3H_2O$ and many other previously reported catalysts.

CONCLUSIONS

In summary, a new, efficient and cheaply available catalyst $AlCl_3.6H_2O$ has been explored to effect the synthesis of *gem*-

dihydroperoxides from aliphatic and aromatic ketones and aldehydes using aqueous H_2O_2 (30%) in acetonitrile at room temperature. The attractive features of this new approach are: the readily available and non-toxic catalyst, the high yields, mild reaction condition and the operational simplicity of the procedure.

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