## Letter

# 2-Hydroxylation of 1,3-Diketones with Atmospheric Oxygen

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**Abstract** An efficient method for the 2-hydroxylation of 1,3-diketones by using atmospheric oxygen as an oxidant under transition-metal-free condition is described. The protocol has the advantages of using an inexpensive and stable oxidant, producing high yields, and requiring ecofriendly conditions.

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Key words diketones, hydroxylations, oxidations, green chemistry

2-Hydroxy-1,3-dicarbonyl compounds are widely used as synthetic intermediates, and the 2-hydroxy-1,3-dicarbonyl group is a common structural motif in many natural products and pharmaceuticals.<sup>1</sup> The most convenient synthetic route to 2-hydroxy-1,3-dicarbonyl compounds is the direct oxidation of the corresponding 1,3-dicarbonyl compounds with various oxidants, such as *m*-chloroperbenzoic acid,<sup>2</sup> dimethyldioxirane,<sup>3</sup> oxaziridines,<sup>4</sup> cumene hydroperoxide,<sup>5</sup> or nitrosobenzene.<sup>6</sup> However, such oxidants have disadvantages in terms of their instability and high cost. With respect to the synthesis of 2-hydroxy-1,3-diketones, Labat and Meunier reported a peroxidase route for the hydroxylation of dimedone (5,5-dimethylcyclohexane-1,3-dione) with hydroperoxide as an oxidant.<sup>7</sup> Suzuki and coworkers reported a two-step method involving the hydroxylation of isoxalium salts by using sodium hypochlorite as an oxidant.<sup>8</sup> Recently, oxidation by molecular oxygen catalyzed by transition-metal catalysts (manganese,<sup>9</sup> cerium,<sup>10</sup> or palladium<sup>11</sup>) or by diiodine<sup>12</sup> has been used to synthesize 2-hydroxy-1,3-dicarbonyl compounds. From an economic and ecological point of view, the oxidation of dicarbonyl compounds by air is optimal. However, these reactions are still limited by the restricted scope of the substrates, such as  $\beta$ -keto esters. A heavy-metal catalyst loading of at least 5 mol% is still required, which can cause environmental pollution. Therefore the exploration of the reaction using air as an oxidant without heavy metals and the enlargement of the substrate scope are still necessary. Here we report a 2hydroxylation of 1,3-diketones by using air as an oxidant in the absence of heavy metals.

Initially, the required 1,3-diarylpropane-1,3-diones **1** were prepared by the reaction of methyl benzoates with acetophenones in the presence of sodium methoxide in xylene (Scheme 1).



Scheme 1 Synthesis of 1,3-diarylpropane-1,3-diones

In the investigations on the 2-hydroxylation of 1,3-diketones, 1,3-diphenylpropane-1,3-dione (1a) was selected as a substrate. The reaction was conducted by using air as an oxidant under various conditions involving a range of catalysts, bases, and solvents. In the absence of a catalyst, no 2hydroxy-1,3-diphenylpropane-1,3-dione (2a) was obtained (Table 1, entry 1). The reaction also failed in the presence of the transition-metal catalyst copper(I) iodide (entry 2). Fortunately, further research showed that several alkali metal halides, such as sodium iodide, potassium iodide, potassium bromide, and potassium chloride were efficient catalysts for the reaction, giving **2a** in good yields (entries 3–6). In particular, potassium fluoride, cesium fluoride, calcium fluoride, and tetrabutylammonium fluoride trihydrate gave the hydroxy compound 2a in high yields (entries 7–10). Of these catalysts, potassium fluoride afforded the best yield (entry 7). The choice of base also had an effect on the reaction of dione 1a. Product 2a was not obtained in the absence of a base (entry 13). When calcium hydroxide, sodium tert-butoxide, or sodium hydroxide was used as the

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base (entries 7, 11–12), the best results were obtained with sodium hydroxide (entry 7). The solvents also played an important role in the reaction of dione **1a**. The reaction in aqueous dimethyl sulfoxide gave the desired product **2a** in good yield (entry 15). Aqueous *N*,*N*-dimethylformamide was also an effective solvent for the reaction, giving the hydroxylated product in high yield (entry 7). Note that water is indispensable for the reaction; in the absence of water, product **2a** was not obtained (entry 14). Other solvents that were tested (ethanol, 1,4-dioxane, acetonitrile, toluene, tetrahydrofuran, and dichloromethane) did not give an efficient reaction.

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 Table 1
 Optimization of the Reaction Conditions for the 2-Hydroxylation of Dione 1a<sup>a</sup>

EntryCatalystBaseSolventTime (h)Yieldb ( $\chi$ )1-NaOHDMF-H2O (24:1)2402CulNaOHDMF-H2O (24:1)2403NaINaOHDMF-H2O (24:1)12754KINaOHDMF-H2O (24:1)12805KBrNaOHDMF-H2O (24:1)12846KCINaOHDMF-H2O (24:1)12877KFNaOHDMF-H2O (24:1)12958CsFNaOHDMF-H2O (24:1)12939CaF2NaOHDMF-H2O (24:1)128910TBAF·3H2ONaOHDMF-H2O (24:1)127812KFCa(OH)2DMF-H2O (24:1)128913KF-DMF-H2O (24:1)24014KFNaOHDMF24015KFNaOHDMSO-H2O (24:1)1270	Ĺ	1a	cataly air, s 120 °C	st, base olvent C, 12 h	0 0H 2a	$\bigcirc$
1-NaOHDMF- $H_2O$ (24:1)2402CulNaOHDMF- $H_2O$ (24:1)2403NalNaOHDMF- $H_2O$ (24:1)12754KINaOHDMF- $H_2O$ (24:1)12805KBrNaOHDMF- $H_2O$ (24:1)12846KClNaOHDMF- $H_2O$ (24:1)12877KFNaOHDMF- $H_2O$ (24:1)12958CsFNaOHDMF- $H_2O$ (24:1)12939CaF2NaOHDMF- $H_2O$ (24:1)128910TBAF· $3H_2O$ NaOHDMF- $H_2O$ (24:1)129011KFCa(OH)2DMF- $H_2O$ (24:1)128913KF-DMF- $H_2O$ (24:1)24014KFNaOHDMF24015KFNaOHDMSO- $H_2O$ (24:1)1270	Entry	Catalyst	Base	Solvent	Time (h)	Yield <sup>⊾</sup> (%)
2         Cul         NaOH         DMF-H <sub>2</sub> O (24:1)         24         0           3         Nal         NaOH         DMF-H <sub>2</sub> O (24:1)         12         75           4         KI         NaOH         DMF-H <sub>2</sub> O (24:1)         12         80           5         KBr         NaOH         DMF-H <sub>2</sub> O (24:1)         12         84           6         KCI         NaOH         DMF-H <sub>2</sub> O (24:1)         12         87           7         KF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         95           8         CsF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           10         TBAF·3H <sub>2</sub> O         NaOH         DMF-H <sub>2</sub> O (24:1)         12         90           11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         78           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0	1	-	NaOH	DMF-H <sub>2</sub> O (24:1)	24	0
3         Nal         NaOH         DMF-H <sub>2</sub> O (24:1)         12         75           4         KI         NaOH         DMF-H <sub>2</sub> O (24:1)         12         80           5         KBr         NaOH         DMF-H <sub>2</sub> O (24:1)         12         84           6         KCl         NaOH         DMF-H <sub>2</sub> O (24:1)         12         87           7         KF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         95           8         CsF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         89           10         TBAF·3H <sub>2</sub> O         NaOH         DMF-H <sub>2</sub> O (24:1)         12         90           11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         78           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	2	Cul	NaOH	DMF-H <sub>2</sub> O (24:1)	24	0
4         KI         NaOH         DMF-H <sub>2</sub> O (24:1)         12         80           5         KBr         NaOH         DMF-H <sub>2</sub> O (24:1)         12         84           6         KCl         NaOH         DMF-H <sub>2</sub> O (24:1)         12         87           7         KF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         95           8         CsF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         99           10         TBAF·3H <sub>2</sub> O         NaOH         DMF-H <sub>2</sub> O (24:1)         12         90           11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         78           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	3	Nal	NaOH	DMF-H <sub>2</sub> O (24:1)	12	75
5         KBr         NaOH         DMF-H <sub>2</sub> O (24:1)         12         84           6         KCl         NaOH         DMF-H <sub>2</sub> O (24:1)         12         87           7         KF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         95           8         CsF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         89           10         TBAF·3H <sub>2</sub> O         NaOH         DMF-H <sub>2</sub> O (24:1)         12         90           11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         89           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         12         0           14         KF         NaOH         DMF         24         0           15         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	4	KI	NaOH	DMF-H <sub>2</sub> O (24:1)	12	80
6         KCl         NaOH         DMF-H <sub>2</sub> O (24:1)         12         87           7         KF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         95           8         CsF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         89           10         TBAF·3H <sub>2</sub> O         NaOH         DMF-H <sub>2</sub> O (24:1)         12         90           11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         78           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMFO-H <sub>2</sub> O (24:1)         12         70	5	KBr	NaOH	DMF-H <sub>2</sub> O (24:1)	12	84
7         KF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         95           8         CsF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         89           10         TBAF·3H <sub>2</sub> O         NaOH         DMF-H <sub>2</sub> O (24:1)         12         90           11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         78           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMF         24         0           15         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	6	KCl	NaOH	DMF-H <sub>2</sub> O (24:1)	12	87
8         CsF         NaOH         DMF-H <sub>2</sub> O (24:1)         12         93           9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         89           10         TBAF·3H <sub>2</sub> O         NaOH         DMF-H <sub>2</sub> O (24:1)         12         90           11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         78           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	7	KF	NaOH	DMF-H <sub>2</sub> O (24:1)	12	95
9         CaF <sub>2</sub> NaOH         DMF-H <sub>2</sub> O (24:1)         12         89           10         TBAF·3H <sub>2</sub> O         NaOH         DMF-H <sub>2</sub> O (24:1)         12         90           11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         78           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMFO-H <sub>2</sub> O (24:1)         12         70	8	CsF	NaOH	DMF-H <sub>2</sub> O (24:1)	12	93
10         TBAF·3H <sub>2</sub> O         NaOH         DMF-H <sub>2</sub> O (24:1)         12         90           11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         78           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	9	$CaF_2$	NaOH	DMF-H <sub>2</sub> O (24:1)	12	89
11         KF         Ca(OH) <sub>2</sub> DMF-H <sub>2</sub> O (24:1)         12         78           12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMF         24         0           15         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	10	TBAF-3H <sub>2</sub> O	NaOH	DMF-H <sub>2</sub> O (24:1)	12	90
12         KF         t-BuONa         DMF-H <sub>2</sub> O (24:1)         12         89           13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMF         24         0           15         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	11	KF	Ca(OH) <sub>2</sub>	DMF-H <sub>2</sub> O (24:1)	12	78
13         KF         -         DMF-H <sub>2</sub> O (24:1)         24         0           14         KF         NaOH         DMF         24         0           15         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	12	KF	t-BuONa	DMF-H <sub>2</sub> O (24:1)	12	89
14         KF         NaOH         DMF         24         0           15         KF         NaOH         DMSO-H <sub>2</sub> O (24:1)         12         70	13	KF	-	DMF-H <sub>2</sub> O (24:1)	24	0
15 KF NaOH DMSO-H <sub>2</sub> O (24:1) 12 70	14	KF	NaOH	DMF	24	0
	15	KF	NaOH	DMSO-H <sub>2</sub> O (24:1)	12	70

<sup>a</sup> Reaction conditions: **1a** (1 mmol), catalyst (0.1 mmol), base (0.2 mmol), solvent (5 mL), 120 °C, air.

<sup>b</sup> Isolated yield.

On the basis of these promising findings, a series of 1,3diketones **1** were examined in the hydroxylation reaction to give the corresponding 2-hydroxy-1,3-diketones **2** by using potassium fluoride as the catalyst and sodium hydroxide as the base in aqueous *N*,*N*-dimethylformamide under air (Table 2).<sup>13</sup> 1,3-Diketones bearing either electron-donating (methyl, methoxy) groups or electron-withdrawing (fluoro, chloro, bromo) groups on the aromatic rings participated in the reaction (Table 2, entries 2–12). However, the former gave the desired 2-hydroxy-1,3-diketones in slightly higher yields than the latter. 1,3-Diketones bearing heteroaryl groups, such as thienyl or furyl, also reacted efficiently (entries 13 and 14). Unfortunately, attempts to perform 2-hydroxylation of aliphatic 1,3-diketones such as pentane-2,4-dione (**10**,  $R^1 = R^2 = Me$ ; entry 15), were unsuccessful, possibly because, unlike aromatic 1,3-diketones, aliphatic 1,3-diketones cannot form stable conjugate enol intermediates during the reactions, making the hydroxylation more difficult.

#### Table 2 Scope of the 1,3-Diketones

R <sup>1</sup> R <sup>1</sup> , R <sup>2</sup>	0 0 R <sup>2</sup> 1 = aryl, heteroaryl	KF, NaOH air, DMF-H <sub>2</sub> O, 120 °C	;, 12 h R <sup>1</sup>	$R^1 \xrightarrow{O}_{OH} R^2$	
Entry	<b>R</b> <sup>1</sup>	R <sup>2</sup>	Product	Yield (%)	
1	Ph	Ph	2a	95	
2	Ph	4-Tol	2b	93	
3	Ph	4-MeOC <sub>6</sub> H <sub>4</sub>	2c	90	
4	4-Tol	4-Tol	2d	89	
5	4-Tol	4-MeOC <sub>6</sub> H <sub>4</sub>	2e	85	
6	Ph	3-MeOC <sub>6</sub> H <sub>4</sub>	2f	87	
7	Ph	$4-FC_6H_4$	2g	82	
8	Ph	$4-ClC_6H_4$	2h	85	
9	Ph	$4-BrC_6H_4$	2i	82	
10	4-Tol	$4-FC_6H_4$	2j	80	
11	4-Tol	$4-ClC_6H_4$	2k	82	
12	Ph	$3-BrC_6H_4$	21	80	
13	Ph	2-thienyl	2m	81	
14	Ph	2-furyl	2n	80	
15	Me	Me	20	0	

The following plausible mechanism is proposed for the 2-hydroxylation of 1,3-diketones (Scheme 2). First, the 1,3-diketone **1** is converted into an enol isomer **A** by hydrogen bonding to the fluoride ion of potassium fluoride. Enol **A** is oxidized by air to form the epoxide intermediate **B**, which undergoes nucleophilic attack by a hydroxy ion to give intermediate **C** through ring-opening of the epoxide moiety. Intermediate **C** easily yields the final product **2** by loss of fluoride and hydroxy ions.

In conclusion, novel method has been developed for the 2-hydroxylation of 1,3-diketones by using atmospheric oxygen as an oxidant, potassium fluoride as a catalyst, and sodium hydroxide as a base under transition-metal-free conditions. The advantages of this protocol are its use of an inexpensive and stable oxidant, its high yield, and its ecofriendly conditions. 2865

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## **Supporting Information**

Supporting information for this article is available online at http://dx.doi.org/10.1055/s-0035-1560647.

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- (13) 2-Hydroxy-1,3-diketones 2; General Procedure

A mixture of 1,3-diketone **1** (1.0 mmol), KF (0.1 mmol), and NaOH (0.2 mmol) in DMF (4.8 mL) and H<sub>2</sub>O (0.2 mL) was stirred at 120 °C under air for 12 h. When the reaction was complete (TLC), the mixture was cooled to r.t., diluted with EtOAc, and washed with sat. aq Na<sub>2</sub>CO<sub>3</sub>. The resulting organic phase was dried (MgSO<sub>4</sub>) and concentrated under reduced pressure. The residue was purified by column chromatography [silica gel, PE– EtOAc (20:1)]. Analytical data for representative products are given below.

#### 2-Hydroxy-1,3-diphenylpropane-1,3-dione (2a)

White solid; yield: 95%; mp 92–94 °C. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.99 (d, *J* = 7.8 Hz, 4 H, ArH), 7.47–7.55 (m, 6 H, ArH), 6.86 (s, 1 H, CH). <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>):  $\delta$  = 185.7, 135.5, 132.4, 126.7, 127.1, 93.1. Anal. Calcd for C<sub>15</sub>H<sub>12</sub>O<sub>3</sub>: C, 74.99; H, 5.03. Found: C, 74.86; H, 5.05.

#### 1-(2-Furyl)-2-hydroxy-3-phenylpropane-1,3-dione (2n)

White solid; yield: 80%; mp 76–78 °C. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.96 (d, *J* = 7.2 Hz, 2 H, ArH), 7.47–7.62 (m, 4 H, ArH and FuH), 7.23–7.27 (m, 2 H, ArH and FuH), 6.77 (s, 1 H, CH). <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>):  $\delta$  = 182.5, 177.7, 151.0, 146.1, 134.7, 132.4, 128.7, 127.0, 115.8, 112.7, 92.7. Anal. Calcd for C<sub>13</sub>H<sub>10</sub>O<sub>4</sub>: C, 67.82; H, 4.38. Found: C, 67.74; H, 4.39.