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# New, Efficient Synthesis of a-Chloroketones Using SiCl<sub>4</sub>/ Urea-Hydrogen Peroxide or SiCl<sub>4</sub>/ Iodosylbenzene Reagent Systems

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#### NEW, EFFICIENT SYNTHESIS OF α-CHLOROKETONES USING SiCl<sub>4</sub>/UREA-HYDROGEN PEROXIDE OR SiCl<sub>4</sub>/IODOSYLBENZENE REAGENT SYSTEMS

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#### **GRAPHICAL ABSTRACT**



**Abstract** Alkyl aryl ketones on treatment with SiCl<sub>4</sub>/urea–hydrogen peroxide (UHP) or SiCl<sub>4</sub>/iodosylbenzne reagent systems afforded  $\alpha$ -chloroketones in excllent yields, while ketones with higher enol content provide exclusively  $\alpha, \alpha$ -dichloroketones under exceedingly mild conditions. The reaction proceeds via the initial formation of silyl enol ethers. A polarized chlorine intermediate that resulted from the coordination of SiCl<sub>4</sub> with the in situ formed trichlorosilyl hypochlorite Cl<sub>3</sub>SiOCl is thought to be the active chlorinating agent.

Keywords  $\alpha$ -Chloroketones; iodosylbenzene; silyl peroxides; tetrachlorosilane (TCS); urea-hydrogen peroxide (UHP)

#### INTRODUCTION

 $\alpha$ -Chloroketones are reactive and extremely useful intermediates that have several applications in biological and organic synthesis, particularly for the synthesis of natural products and heterocyclic compounds.<sup>[1]</sup> The increase of interest in the chemistry of  $\alpha$ -chloroketones arises from the observation that they are versatile intermediates capable of mediating many different chemical transformations.<sup>[2]</sup> Several useful reagents and procedures have been reported for the synthesis of  $\alpha$ -chloroketones. The direct conversion of ketones to  $\alpha$ -chloroketones can be induced using a variety of chlorinating agents such as N-chloro-succinimide,<sup>[3]</sup>

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tetraethylammonium trichloride,<sup>[4]</sup> trichloromethyl carbinols,<sup>[5]</sup> polymer-supported chlorine,<sup>[6]</sup> and p-toluenesulfonyl chloride.<sup>[7]</sup> Selective  $\alpha$ -chlorination of ketones at the less substituted carbon has been reported via the initial formation of silyl enol ethers as intermediates.<sup>[8]</sup> Copper(II) chloride has been used for the chlorination of unsymmetrical ketones at the more substituted  $\alpha$ -carbon atom.<sup>[9]</sup> The solvent-free  $\alpha$ -chlorination of ketones using [hydroxyl(tosyloxy)iodo]benzene (Koser's reagent) and MgCl<sub>2</sub> under microwave irradiation has also been described.<sup>[10]</sup> Oxidative  $\alpha$ -chlorination of ketones using an aqueous H<sub>2</sub>O<sub>2</sub>/HCl chlorinating system has been recently reported.<sup>[11]</sup> However, some of these methods suffer from drawbacks such as long reaction times, use of hazardous chemicals, and cumbersome workup procedures.

Although recent rapid advances in the methodology of  $\alpha$ -chlorination of ketones have been achieved, their preparation is frequently a troublesome operation, and more efficient and flexible methods are still required.

In continuation of our interest in exploring new synthetic applications of novel tetrachlorosilane (TCS) reagents,<sup>[12–14]</sup> we now report a new mild and inexpensive method for the preparation of  $\alpha$ -chloroketones via the reaction of ketones with TCS in the presence of urea–hydrogen peroxide (UHP), a safe solid substituted with liquid hydrogen peroxide) or iodocylbenzene.

#### **RESULTS AND DISCUSSION**

The reaction of 1-phenylethanone **1a** with a mixture of 2 equivalents of TCS and 1 equivalent of UHP<sup>[15]</sup> in methylene chloride at 0 °C afforded 2-chloro-1-phenylethanone, **2a**, in 88% after usual aqueous workup followed by crystallization. The reaction is very clean, with no side reactions. Indeed, the NMR of the crude product does not show the presence of any impurity.

In an effort to generalize this chlorination methodology, several other 1-phenylethanone derivatives were subjected to the same conditions. The results from these reactions are summarized in Table 1. The chlorination of 1-(4-tolyl) ethanone **1b** provided 2-chloro-1-(4-tolyl) ethanone **2b** in 92% yield. Interestingly,

Compound	$\mathbf{R}^1$	$\mathbb{R}^2$	Time $(h)^{a(b)}$	Yield $(\%)^{a(b)}$	Mp (°C) (lit. mp)
2a	C <sub>6</sub> H <sub>5</sub> - H		1.2 (4.5)	88 (72)	55 (54-55)[11]
2b	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> -	Н	1.0 (4)	92 (75)	57 (56.5-57.5) <sup>[17a]</sup>
2c	4-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub> -	Н	1.0 (4)	93 (76)	98 (98–99) <sup>[17b]</sup>
2d	4-BrC <sub>6</sub> H <sub>4</sub> -	Н	1.0 (4)	92 (75)	$116 (117 - 118)^{[17c]}$
2e	$4-ClC_6H_4-$	Н	1.0 (4)	93 (77)	$101 (101 - 102)^{[17d]}$
2f	$3-NO_2C_6H_4$ -	Н	1.5 (5.5)	85 (70)	96–98 (103) <sup>[17e]</sup>
2g	2-Naphthyl	Н	1.5 (6)	87 (73)	77 (77–79) <sup>[17f]</sup>
2h	C <sub>6</sub> H <sub>5</sub> -	C <sub>6</sub> H <sub>5</sub> -	1.5 (6)	85 (71)	58-60 (61-62) <sup>[17g]</sup>
2i	CH <sub>3</sub> -	-COCH <sub>3</sub>	1.5 (5.5)	76 (66)	Oil

Table 1. Reaction of ketones with TCS/UHP or TCS/PhIO reagent systems

<sup>a</sup>Yields and reaction time using TCS/UHP.

<sup>b</sup>Yields and reaction time using TCS/PhIO.

the ring methyl group was not chlorinated under this condition, thus excluding the radical mechanism. 1-(4-Methoxyphenyl)ethanone 1c, which carries a strong electron-releasing group on the ring, was readily converted into 2-chloro-1-(4-methoxyphenyl)ethanone 2c in 93%. It is concluded from these results that electron-rich aryl groups activate the ketones in this chemoselective electrophilic oxidative chlorination reaction at the  $\alpha$ -carbon without further electrophilic chlorination at the aromatic nucleus. Although electron-poor groups slightly increased the reaction time for the starting material to be consumed, no pronounced effect on the yield of reaction was observed. This was demonstrated from chlorination of 1-(4-bromophenyl) ethanone 1d, 1-(4-chlorophenyl) ethanone 1e, and 1-(3-nitrophenyl) ethanone 1f, which afforded the corresponding  $\alpha$ -chloroketones in 92%, 93%, and 85% yield respectively.

Ketones with more enol content, such as 1-(2-naphthyl) ethanone 1g, 1,2-diphenyl-ethanone 1h, and 2,4-pentanedione 1i, afforded exclusively the  $\alpha,\alpha$ -dichlorinated products in good yields. This presumably may be attributed to the more facile formation of silyl enol ethers from these ketones and their mono-chlorinated products.

$$R^{1} \xrightarrow{O} R^{2} \xrightarrow{\text{SiCl}_{4}/\text{UHP}/0 \, ^{\circ}\text{C}}_{\text{or}} \qquad R^{1} \xrightarrow{Q} R^{2} \times Cl$$

$$1 \text{ a-i} \qquad \text{SiCl}_{4}/\text{PhIO}/0 \, ^{\circ}\text{C} \qquad 2 \text{ a-f}, X = H$$

$$2 \text{ g-i}, X = Cl$$
(1)

$$\operatorname{SiCl}_{4} + \operatorname{PhIO} \quad \underbrace{\operatorname{CH}_{2}\operatorname{Cl}_{2}}_{0 \, {}^{\circ}\operatorname{C}} \quad \operatorname{Cl}_{3}\operatorname{SiOCl} \quad + \operatorname{PhI} \qquad (2)$$

The reaction between TCS and UHP adduct leading to the effective chlorinating agent of ketones is not clear yet. The first step of the reaction could be the formation of bis(trichlorosilyl)peroxide intermediate (A).<sup>[16]</sup> Further reaction of A with

 $CH_2Cl_2$ Cl<sub>3</sub>Si<sub>O</sub>SiCl<sub>3</sub> 1. 2 SiCl<sub>4</sub> +  $H_2O_2$ -NH<sub>2</sub>CONH<sub>2</sub> NH<sub>2</sub>CONH<sub>2</sub> + 2HCl (A) Cl<sub>3</sub>Si\_O\_O\_SiCl<sub>3</sub> 2. Cl<sub>3</sub>SiOCl Cl<sub>3</sub>SiOH **(B)** (A) Cl SiCl<sub>4</sub> 3. Cl<sub>3</sub>SiOCl Cl<sub>3</sub>Si−Ó→SiCl<sub>4</sub> **(B)** (C) Cl<sub>3</sub>Si.  $+ \operatorname{Cl}_3 Si - \stackrel{\stackrel{\stackrel{\stackrel{\scriptstyle}}{O}}{\longrightarrow}}{SiCl_4} \longrightarrow \begin{array}{c} O \\ R^1 \stackrel{\stackrel{\stackrel{\scriptstyle}}{\longrightarrow}}{R^2} + \end{array}$ R1 Cl<sub>3</sub>SiOSiCl<sub>3</sub> + SiCl<sub>4</sub> 4. (C) **(D)** 

Scheme 1. Oxidative  $\alpha$ -chlorination of ketones using TCS-UHP reagent system.

liberated hydrogen chloride produces the trichlorsilyl hypochlorite (**B**). The coordination of **B** with TCS produces the ionized chlorine intermediate (**C**). Electrophilic attack of **C** on the in situ formed silyl enol ethers (**D**) finally furnishes the corresponding  $\alpha$ -chloroketones (Scheme 1).

To get more information about the sequence of this reaction, we conducted the reaction of ketones with TCS, using iodosylbenzene as an oxidizing agent substitute of UHP. Under this condition, the ketones **1a–i** afforded the same products as in the case of TCS/UHP reagent, but the yields were lower and the reaction time was longer (Table 1). A trichlorosilyl hypochlorite intermediate is also thought to be formed in this reaction [Eq. (2)] subsequently; the reaction proceeded in the same manner shown in Scheme 1. The structures of  $\alpha$ -chloroketones have been confirmed using elemental and spectroscopic analyses (*cf.* Table 2).

	Mol. formula (M <sub>r</sub> )	Analysis (%) calcd. (found)		Spectral data: <sup>1</sup> H NMR [CDCl <sub>3</sub> /	
Compound		С	Н	(KBr, cm <sup>-1</sup> ); mass $(m/z)$	
2a	C <sub>8</sub> H <sub>7</sub> ClO (154.59)	62.15 (62.29)	4.56 (4.41)	IR: 3061, 2951, 1695, 1596, 1449, 1000, 748. MS: m/z (%): 154 (M <sup>+</sup> , 10), 105 (100), 77 (88).	
2b	C <sub>9</sub> H <sub>9</sub> ClO (168.62)	64.11 (63.98)	5.38 (5.13)	<sup>1</sup> H NMR: $\delta = 7.93 - 7.44$ (m, 5H), 4.7 (s, 2H). IR: 3057, 2960, 1698, 1592, 1449, 984, 756. <sup>1</sup> H NMR: $\delta = 7.46$ (d, 2H, $J = 7.5$ Hz), 7.01 (d, 2H, J = 7.5 Hz), 4.63 (s, 2H), 2.35 (s, 3H)	
2c	C <sub>9</sub> H <sub>9</sub> ClO <sub>2</sub> (184.62)	58.55 (58.43)	4.91 (4.82)	IR: 3065, 2955, 1700, 1593, 988, 756. <sup>1</sup> H NMR: $\delta = 7.85$ (d, 2H, $J = 7.5$ Hz), 7.43 (d, 2H, J = 7.5 Hz), 4.68 (s, 2H) 3.93 (s, 3H)	
2d	C <sub>8</sub> H <sub>6</sub> BrClO (233.49)	41.15 (41.23)	2.59 (2.68)	IR: 2998, 2952, 1695, 1584, 1449, 1006, 695. <sup>1</sup> H NMR: $\delta = 7.83$ (d, 2H, $J = 7.5$ Hz), 7.63 (d, 2H $I = 7.5$ Hz) 4 61 (s, 2H)	
2e	C <sub>8</sub> H <sub>6</sub> Cl <sub>2</sub> O (189.04)	50.83 (50.66)	3.20 (3.37)	IR: 2998, 2952, 1694, 1590, 1449, 999, 782. MS: m/z (%): 188 (M <sup>+</sup> , 3.4), 139 (100), 111 (43.6). <sup>1</sup> H NMR: $\delta = 7.88$ (d, 2H, $J = 7.5$ Hz), 7.45 (d, 2H, $J = 7.5$ Hz) 4 65 (s, 2H)	
2f	C <sub>8</sub> H <sub>6</sub> ClNO <sub>3</sub> (199.59)	48.14 (47.93)	3.03 (3.25)	IR: 2998, 3089, 1691, 1614, 1526, 1349, 1110, 819, 741, 675. <sup>1</sup> H NMR: $\delta = 8.77$ (s, 1H), 8.6 (d, 1H, $J = 7.6$ ), 8.3 (d, 1H, $J = 7.6$ ), 7.68 (m, 1H), 4.71 (s, 2H).	
2g	C <sub>12</sub> H <sub>8</sub> Cl <sub>2</sub> O (239.10)	60.28 (60.43)	3.37 (3.29)	IR: 3057, 2944, 1701, 1621, 1591, 1464, 924, 740. MS: $m/z$ (%): 238 (M <sup>+</sup> , 7.3), 204 (3.4), 155 (100), 127 (68.4). <sup>1</sup> H NMR: $\delta$ = 8.63 (s, 1H), 8.12–7.58 (m, 6H), 6.84 (s, 1H).	
2h	C <sub>14</sub> H <sub>10</sub> Cl <sub>2</sub> O (265.13)	63.42 (63.35)	3.80 (3.56)	IR: 3070, 3031, 2951, 1694, 1595, 995, 758. MS: m/z (%): 230 (M <sup>+</sup> -Cl, 1.0), 229 (4), 105 (100), 77 (43.6). <sup>1</sup> H NMR: $\delta = 8.15-7.29$ (m, 10H).	
2i				IR: 3020, 2951, 1716, 1617, 1397, 1267, 1035, 912. <sup>1</sup> H NMR: $\delta = 2.28$ (s, 6H). <sup>13</sup> C NMR: $\delta = 23.86$ , 108.08, 188.91.	

Table 2. Analytical and spectral data of  $\alpha$ -chloroketones 2a-2i

#### CONCLUSION

In conclusion, we have presented a very cheap, chemoselective, fast, safe, and ecofriendly procedure for chlorniation of ketones at the  $\alpha$ -carbon without any trace of nuclear chlorination at the aromatic ring. Using TCS as chlorine source in the presence of easily prepared and easily handled oxidizing agents available in large amounts such as UHP or iodosylbenzene offers useful new chlorinating agents, which can be used for chlorination of different organic compounds as alternative for the existing methods.

#### **EXPERIMENTAL**

Melting points were determined in open capillary tubes and are uncorrected. Infrared (IR) spectra were recorded on Thermo Nicolet nexus 470 Fourier transform (FT)–IR and Mattson 5000 FT-IR spectrophotometer with only selected absorptions being recorded. Absorption maxima were recorded in centimeters<sup>-1</sup>. Nuclear magnetic resonance (NMR) spectra were run on a <sup>1</sup>H NMR Varian-Mercury (200-MHz) FT-NMR spectrometer. Spectra were measured using CDCl<sub>3</sub> as solvent with chemical shifts quoted in parts per million ( $\delta$  ppm) using tetramethylsilane (TMS) as internal standard. The mass spectra (MS) were recorded on a GC-MS QP-1000 EX Schmiadzu (Japan) mass spectrometer. Elemental analyses were carried out at the Microanalytical Unit, Faculty of Science, Cairo University.

#### **General Procedure**

In a typical procedure, TCS (1.2 ml, 10 mmol) was added to a mixture of ketone (5 mmol) and UHP (0.47 g, 5 mmol) or iodosylbenzene (1.05 g, 5 mmol) in anhydrous methylene chloride (15 ml). This mixture was stirred with exclusion of moisture at 0 °C until the ketone was consumed (monitoring by thin-layer chromatography). The mixture was then poured into ice-cooled water (50 ml) and extracted with chloroform (2  $\times$  50 ml). The extracts were dried over anhydrous sodium sulfate and evaporated under reduced pressure. The residue was purified by crystallization from diethyl ether to give pure products (*cf.* Table 1).

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