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# Metal Free, Direct and Selective Deoxygenation of α-Hydroxy Carbonyl Compounds: Access to α,α-Diaryl Carbonyl Compounds

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**Abstract:**

An efficient, metal free, direct and selective deoxygenation of α-hydroxy carbonyl compounds is achieved with the aid of catalytic amount of aqueous $HClO_4$ (70%) and triethylsilane as hydride source. A variety of α-hydroxy-α,α-diaryl carbonyl compounds are selectively deoxygenated to give α,α-diaryl carbonyl compounds in good to excellent yields. Intermediacy of α-keto carbenium ion is proposed on the basis of some control experiments and atmospheric pressure chemical ionization mass spectral analysis.

## Introduction

Efficient strategies for the deoxygenation of alcohols have become a cardinal endeavour in organic synthesis, as defunctionalisation rather than functionalization has come to a centre stage due to its extreme importance in "best from the worst" policy.[1] For example, biomass, which is a waste and nuisance (but abundant in –OH due to major carbohydrate content) is getting great thrust as an alternate to fossil based fuels and a clear-cut focus has been laid on the conversion of high-volume, low-value biomass into low-volume, high-value chemicals and fuel.[2] Besides this massive futuristic application, deoxygenation also plays a vital role in the total syntheses of natural products,

pharmaceutically active compounds and their synthetic modifications.[3] Since the pioneer protocol given by Barton-McCombie on radical deoxygenation of alcohols,[4] today's scenario has witnessed deoxygenation of oxygen present in almost all organic functional groups.[5-7] Nevertheless, such deoxygenation reactions have been mostly studied on substrates carrying one type of oxygen functionality. Deoxygenation of hydroxyl and keto group, for example, has been achieved separately using $PdCl_2$ or $InCl_3$ as Lewis acids and hydrosilane as the hydride source (Scheme 1a).[8]

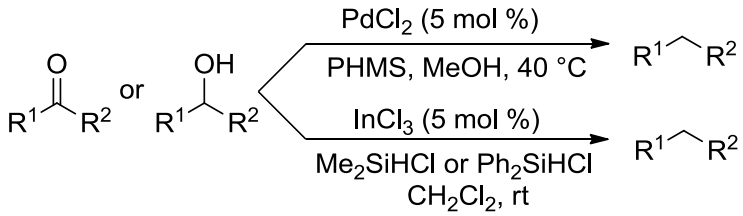
Considering the diverse nature of molecules bearing hydroxyl group, its selective deoxygenation in the presence of other oxygen bearing functional groups (>C=O, –CHO, –COOH, –O–, –COOR etc) becomes an arduous task demanding careful attention. Commendable strides have been achieved in this direction.[9] Presence of another oxygen functionality such as a >C=O moiety at the immediate vicinity of –OH makes the removal much more demanding as there are many naturally occurring compounds and key synthetic intermediates that contain this α-hydroxy carbonyl moiety.[10] Being important, many research groups have achieved such deoxygenations.[11] For example, Speckmeier et al. have reported visible light mediated deoxygenation of O-acetyl benzoin and acyloin derivatives (Scheme 1b, i).[11b] Very recently, in a seminal paper, Yang and Xu have realized iridium catalyzed site selective deoxygenation of a variety of alcohols with a single example of α-keto alcohol.[11a] In all these reports, hydroxyl group is first converted to other functional groups or good leaving groups or activators are required prior to selective deoxygenation.[11] Though selective deoxygenation is achieved, the downside is that a stoichiometric amount of chemical-waste is also generated. In addition, in many of the conversions, environmentally harmful toxic metal derivatives are used as catalyst. The realization of the limitations such as high-cost, environmental pollution and associated toxicity of the metallic reagent has prompted researchers to approach deoxygenation through metal-free pathways and there are

10.1002/ejoc.202000142

significant success stories reported in the current decade.[12] Compain and co-workers have made an important break-through while exploring the use of iodine in the deoxygenation of hydroxyl group present at the carbon bearing electron withdrawing carbonyl group (Scheme 1b, ii).[12b] Selective deoxygenation of α-hydroxy carbonyl compounds has been achieved via the formation of corresponding α-iodo carbonyl intermediates. This iodine mediated deoxygenation of alcohols is not applicable in the case of tertiary α-hydroxy carbonyl compounds.

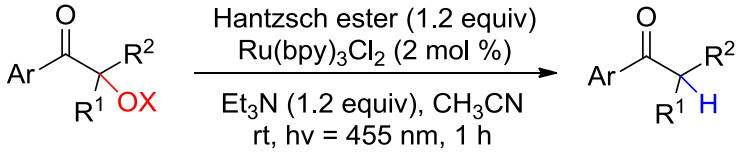
**Scheme 1. Different Approaches Toward Deoxygenation**

a) Well explored deoxygenation of alcohols and ketones[8]



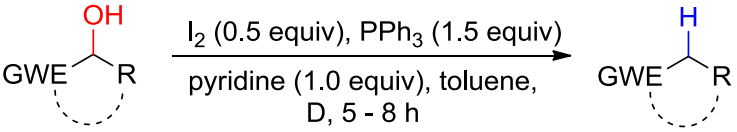

b) Selective deoxygenation of hydroxyl group in the presence of carbonyl group

i) Indirect approach using better leaving groups[11b]



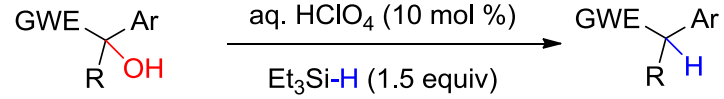

(2°, 3° alcohols)
X = Ac, Bz, Ms, Piv

ii) Indirect approach via corresponding halide[12b]




(1°, 2° alcohols)
EWG = ketone, ester, amide

iii) Direct approach and metal free: This work




3° alcohols
EWG = ketone, acid, ester

Despite these advances, the development of mild, cheaper and greener methods for the direct and selective deoxygenation is highly desirable. Deoxygenation of alcohols containing electron withdrawing group (EWG) at the carbon bearing –OH group is even more challenging when carbocations are the intermediates. This is because EWG tends to destabilize the carbocations. Our previous experience of replacing –OH group with a variety of substituents in tertiary α-hydroxy carbonyl compounds[13] gave us the hope that another

typical Brønsted acid such as $HClO_4$, if used in conjunction with an appropriate hydride source should effect, efficient deoxygenation. If this hypothesis is validated affirmative, then it will provide an efficient strategy, which is metal free to remove –OH group alpha to carbonyl functionality. Thus, herein, we wish to report for the first time a metal-free, direct and selective approach for the deoxygenation of α-hydroxy-α,α-diaryl carbonyl compounds using much cheaper aqueous $HClO_4$ (70%) as catalyst and triethylsilane ($Et_3SiH$) as soluble hydride source (Scheme 1b, iii). There is an added advantage to this strategy. The deoxygenation of –OH group in α-hydroxy-α,α-diaryl carbonyl compounds can furnish α,α-diaryl carbonyl compounds, which are an important class themselves.[14] 1,2,2-Triarylethanones, for example, are very important precursors for the synthesis of dibenzo[*a*,*c*]phenanthridine, analogues of biologically active benzo[*c*]phenanthridine alkaloids.[15] In addition, 1,2,2-triarylethanones are also useful synthons for tamoxifen, the most widely used drug for the treatment of breast cancer.[16] The synthetic methodology that result in α,α-diaryl carbonyl compounds are mostly metal assisted.[14]

**Results and Discussion**

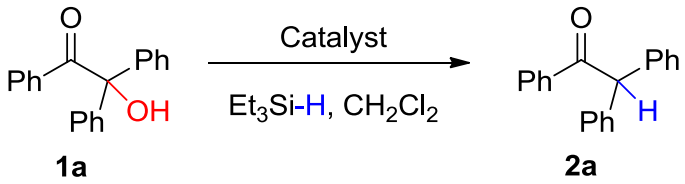
Our careful choice of a substrate to test the above mentioned hypothesis resulted in 2-hydroxy-1,2,2-triphenylethanone (**1a**, Table 1). **1a** has the essential requisites, the requited α-hydroxy ketone moiety, but devoid of any influencing electron withdrawing group (EWG) or electron donating group (EDG) attached to the phenyl ring(s). The lead to select $InBr_3$ and aqueous $HClO_4$ (70%) is based on the previous reports.[13b,17] A solution of α-hydroxy ketone **1a** in $CH_2Cl_2$ containing $InBr_3$ (10 mol %) and $Et_3SiH$ (1.5 equiv) was stirred at room temperature (Table 1, entry 1). After usual work up, the product was purified by flash chromatography. To our delight, the isolated compound was identified as 1,2,2-triphenylethanone (**2a**) in which selective deoxygenation of –OH took place, retaining >C=O. Very similar and highly desired metal free deoxygenation of **1a** to **2a** was also achieved with

10.1002/ejoc.202000142

the use of aq. $HClO_4$ (20 mol %) as the catalyst in 55% yield (Table 1, entry 2). The reaction conditions were then optimized for the deoxygenation of 2-hydroxy-1,2,2-triphenylethanone (**1a**) using both $InBr_3$ (entries 3-5) and $HClO_4$ (entries 6-10) as metal and metal free catalyst respectively by varying mol% of the catalyst, temperature, reaction time etc. (Table 1, optimal conditions are shown in bold font).

10.1002/ejoc.202000142

**Table 1. Optimization of the Reaction Conditions**




**1a** **2a**

| Entry[a] | Catalyst (mol %) | $Et_3Si$-H (equiv) | Temperature (°C) | Time | Yield[b] (%) |
|---|---|---|---|---|---|
| 1 | $InBr_3$ (10) | 1.5 | 25 | 40 min | 93 |
| 2[c] | $HClO_4$ (20) | 1.5 | 25 | 2 h | 55 |
| 3 | $InBr_3$ (10) | 1.5 | 40 | 10 min | 95 |
| 4 | $InBr_3$ (5) | 1.5 | 25 | 1.5 h | 88 |
| 5 | $InBr_3$ (5) | 1.5 | 40 | 30 min | 94 |
| 6[c] | $HClO_4$ (20) | 2.0 | 25 | 2 h | 74 |
| 7[c] | $HClO_4$ (20) | 3.0 | 25 | 2 h | 85 |
| 8[c] | $HClO_4$ (10) | 2.0 | 25 | 2 h | 27 |
| **9[c]** | **$HClO_4$ (10)** | **1.5** | **40** | **2.5 h** | **96** |
| 10[c] | $HClO_4$ (10) | 2.0 | 40 | 1.5 h | 96 |

[a] Reactions were performed on 0.35 mmol of 2-hydroxy-1,2,2-triphenylethanone (**1a**) in $CH_2Cl_2$ (3 mL). [b] Isolated yields. [c] Aqueous $HClO_4$ (70%) is used.

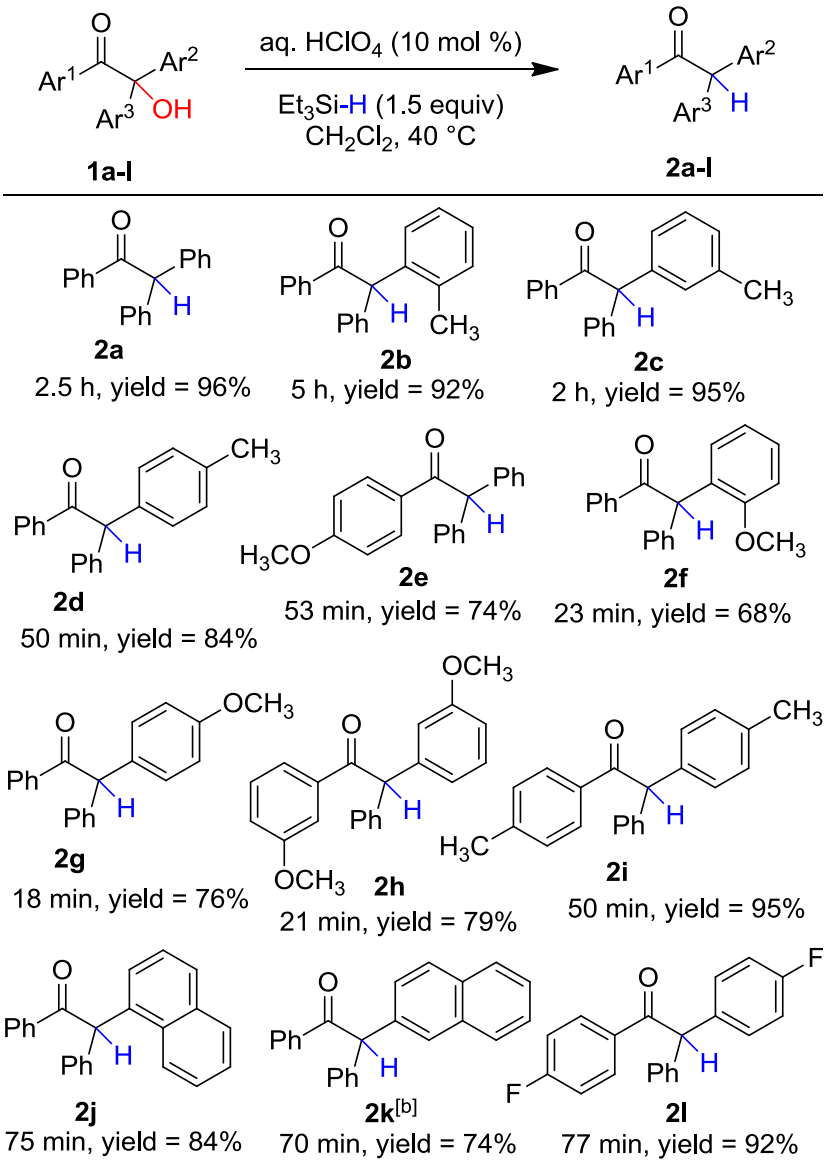
Conversion of the α-hydroxy ketone **1a** to **2a** in excellent yield (96%) using aq. $HClO_4$ (70%) can offer a convenient metal free pathway to generate these family of triarylketones through deoxygenation if the reaction is amenable (i.e. feasibility) and sustainable (yield) for

10.1002/ejoc.202000142

a wide range of substrates. To test this scope, a variety of α-hydroxy ketones **1b-l** were treated under the optimized reaction conditions using aq. $HClO_4$ (10 mol %) and $Et_3SiH$ (1.5 equiv) in $CH_2Cl_2$ to give 1,2,2-triarylethanones in good to excellent yields (Scheme 2). The formation of the by-product, 2,3-diphenylnaphtho[*1,2-b*]furan (**3**) from **1k** needs a mention here (see experimental section and page S2 of the Supporting Information). Such derivatives are known and can result from intramolecular rearrangement (4π cyclization) of α-keto carbenium ion.[18] Indeed, the isolation of **3** (even though in minor yield), indirectly sheds light upon the proposed mechanism involving the formation of α-carbonyl carbenium ion (*vide infra*).[18] It is worth to note that the substrate **1l** carrying electron withdrawing fluorine also deoxygenated to give the desired product **2l** in excellent yield.

10.1002/ejoc.202000142

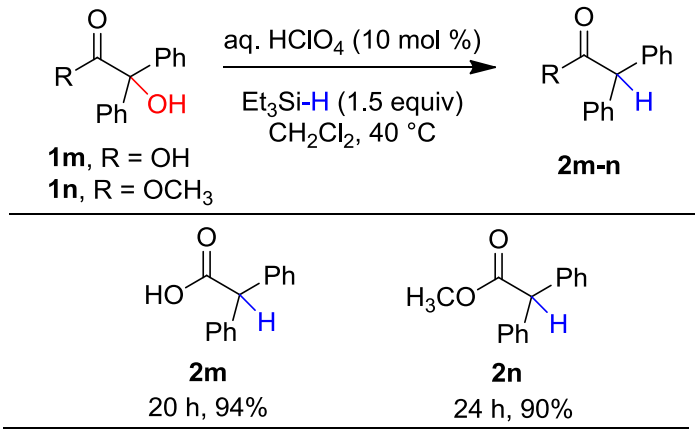
**Scheme 2. Deoxygenation of Various α-Hydroxy Ketones**[a]




[a] Reactions were performed on 0.35-0.66 mmol scale of 2-hydroxy-1,2,2-triarylylethanone (**1a-l**) in $CH_2Cl_2$ (3 mL) using aq. $HClO_4$ (70%). [b] 2,3-diphenylnaphtho[*1,2-b*]furan (**3**, 7%) was also formed in this case.

Extension of this metal free selective deoxygenation to other representative examples such as acids and esters was also undertaken to showcase the utility of the reaction to related α-hydroxy carbonyl compounds (Scheme 3). α-Hydroxy acid such as benzilic acid (**1m**) and α-hydroxy ester such as methyl benzilate (**1n**) are typical and can serve as model compounds as they lack any extra EWG or EDG moieties that can impart secondary influences. Both gave desired deoxygenated product in excellent yield under the optimized reaction conditions. The scope of selective deoxygenation, therefore, can be extended to α-hydroxy acids and α-hydroxy esters as well.

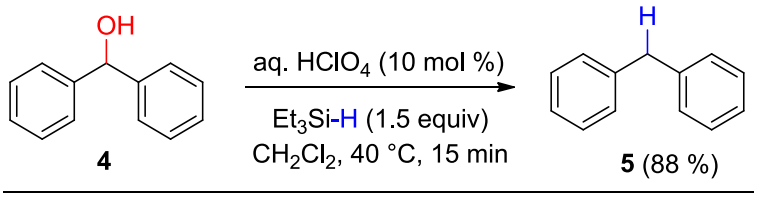
**Scheme 3. Deoxygenation of α-Hydroxy Acid and α-Hydroxy Ester**




To understand the mechanistic necessities of the deoxygenation of α-hydroxy ketone, our model compound **1a** (phenyl benzoin) can be bifurcated to a diphenyl methanol moiety and a bonded benzoyl group. A series of probing questions can be raised, such as; *a)* does the reaction happen without the presence of this electron withdrawing benzoyl moiety? *b)* what would the consequences of alkyl/phenyl substitution at C-OH moiety? etc. To answer the first question, reaction of 1,1-diphenylmethanol (**4**) with $Et_3SiH$ (1.5 equiv) and aq. $HClO_4$ (10 mol %) in $CH_2Cl_2$ was performed and it gave 1,1-diphenylmethane (**5**) within 15 minutes in 88% yield (Scheme 4). This experiment suggests that the presence of an electron withdrawing keto group at the carbon bearing -OH group in **1a** has no (at least very minimal) role in

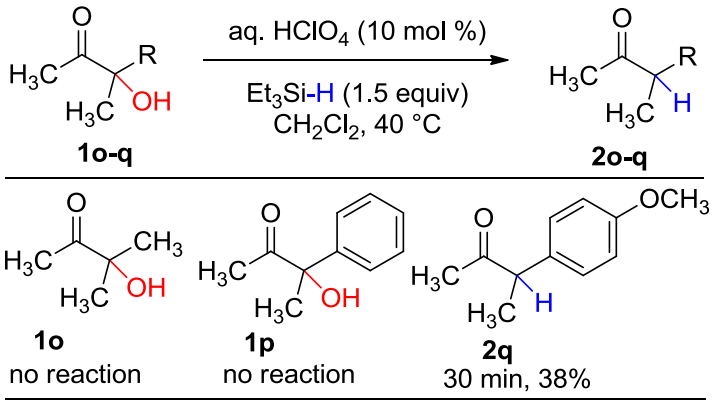
realizing deoxygenation. Similarly, in order to explore the prerequisite of two phenyl groups at the carbon bearing –OH, deoxygenation of simple benzoin under identical reaction conditions was carried out. TLC of this reaction showed a number of spots and isolation of any deoxygenated product did not succeed. This demarcates the difference in the reactivity pattern of benzoin and phenyl benzoin (**1a**), and the decisive role played by the second phenyl moiety in the deoxygenation of α-hydroxy-α,α-diaryl carbonyl compounds. Mechanistically, the α-carbonyl carbenium ion gets more stability due the presence of an additional electron donating phenyl group.

**Scheme 4. Deoxygenation of 1,1-Diphenylmethanol**



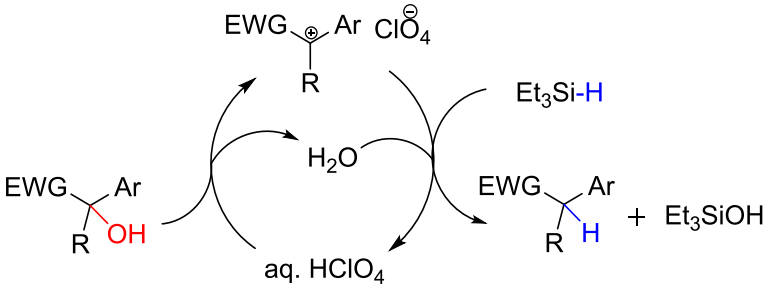

To cross-check the deciding influence of the phenyl group, three α-hydroxy ketones **1o-q** with alkyl groups have been subjected to deoxygenation reactions under the prevailing reaction conditions (Scheme 5). α-Hydroxy ketones **1o** and **1p** failed to react, and in the case of **1q** having methoxy group at para position, deoxygenation did occur, though with poor yield. A combined analysis of the results of benzoin, phenyl benzoin and scheme 5 clearly indicates that subtle electronic factors play crucial role in driving the deoxygenation reaction and α-carbonyl carbenium ions become progressively stable as more EDGs are bonded to the C-OH moiety.

**Scheme 5. Deoxygenation of α-Hydroxy Ketones Bearing Alkyl Groups**



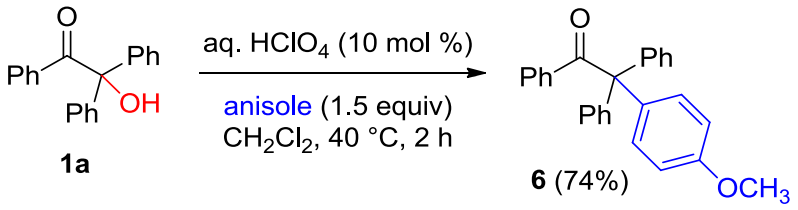

Consolidating different aspects of the deoxygenation reactions described above, intermediacy of the α-carbonyl carbenium ion in the catalytic cycle is proposed (Scheme 6). Interaction of $HClO_4$ with the –OH group leads the formation of α-carbonyl carbenium ion and the elimination of a water molecule. The carbenium ion then reacts with the hydride source ($Et_3SiH$) to give deoxygenated product. This step will regenerate the $HClO_4$ catalyst to complete the catalytic cycle. It is noteworthy that such an α-carbonyl carbenium ion had been observed by reacting 2-hydroxy-1,2,2-triphenylethanone with chlorosulphonic acid at -60 °C in $CDCl_3$ or $CD_2Cl_2$.[18b]

**Scheme 6. Proposed Reaction Mechanism**



We performed two additional experiments to prove the intermediacy of α-carbonyl carbenium ion. Firstly, the deoxygenation reaction did not happen in the absence of aq. $HClO_4$ catalyst. Secondly, the trapping of the carbenium ion is possible if an arene is used as a nucleophile. The reaction of 2-hydroxy-1,2,2-triphenylethanone (**1a**) with anisole occurred smoothly in the presence of aq. $HClO_4$ to give 2-(4-methoxyphenyl)-1,2,2-triphenylethanone (**6**, Scheme 7). This reaction is similar to the Friedel-Crafts arylation reaction of alcohols occurring through carbocation under acidic conditions.[19] Isolation of **3** as rearangement product during deoxygenation of α-hydroxy ketone **1k** also suggests the intermediacy of α-carbonyl carbenium ion.

**Scheme 7. Friedel-Crafts Reaction of 1a With Anisole**




Based on the nature of the substrate, it was considered logical to utilize atmospheric pressure chemical ionization mass spectrometry (APCI-MS) to gather further support to the intermediacy of α-carbonyl carbenium ion. The APCI mass spectra of representative α-hydroxy ketones **1a, 1e, 1g, 1j** showed two prominent base peaks corresponding to two different fragment ions **A** and **B** (Table 2). Fragment **B** is base peak for **1a** and **1e**, and **A** is for the later two (see the Supporting Information). Occurrence of different base-peaks is due to the difference in the stability of the respective carbocations that depends upon the nature of aryl groups and substituents present; nevertheless, the formation of the α-keto carbenium ion is vouched in both cases.

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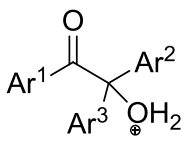
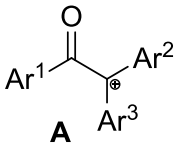
**Table 2. APCI Mass Spectral Fragments of 2-Hydroxy-1,2,2-triarylethanones**

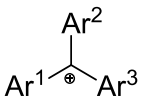



m/z = MH+



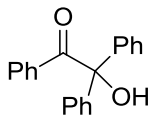
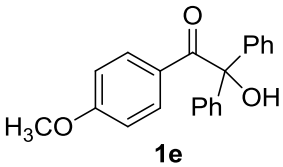
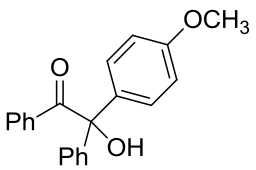
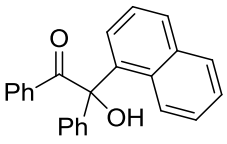

**A**

m/z = $MH^+$-$H_2O$




**B**

m/z = $MH^+$-$H_2O$-CO

| α-Hydroxy Ketone | M | $MH^+$ m/z | **A** m/z (RA)[a] | **B** m/z (RA) |
|---|---|---|---|---|
|  **1a** | 288 | 289 | 271(93) | 243(100) |
|  **1e** | 318 | 319 | 301(41) | 273(100) |
|  **1g** | 318 | 319 | 301(100) | 273(12) |
|  **1j** | 338 | 339 | 321(100) | 293(48) |

[a] RA = Relative abundance in %.

## Conclusion

In conclusion, we have uncovered a direct, selective and metal free method for the deoxygenation of α-hydroxy-α,α-diaryl carbonyl compounds to obtain an important class of α,α-diaryl carbonyl compounds using $Et_3SiH$ as a soluble hydride source and catalytic amount of aqueous $HClO_4$ (70%). Use of much cheaper aq. $HClO_4$ proves to be effective and environment friendly. The presence of two aryl groups at carbon bearing hydroxyl group plays a crucial role in the selective deoxygenation. The intermediacy of α-keto carbenium ion has been proposed for the reaction and is supported by the APCI mass spectral data of few 2-hydroxy-1,2,2-triarylethanones.

## EXPERIMENTAL SECTION

**General Information.** Melting points were recorded in open capillaries and are uncorrected. IR spectra (in wavenumber) were recorded with Thermo Scientific (NICOLET IS 50) FT-IR spectrophotometer. $^1H$ NMR spectra were recorded in $CDCl_3$ with a Bruker Avance II 400 and Bruker Avance NEO 500 NMR spectrometer operating at 400 MHz and 500 MHz respectively. Proton-decoupled $^{13}C$ NMR spectra were also recorded in $CDCl_3$ with a Bruker Avance II 400 and Bruker Avance NEO 500 NMR spectrometer operating at 100.53 MHz and 125.76 MHz respectively. Chemical shifts (δ) are reported in parts per million downfield from tetramethylsilane as an internal standard. Coupling constant (J) values are given in hertz (Hz). Multiplicity of the signal is defined as ‘s’ (singlet), ‘d’ (doublet), ‘t’ (triplet), ‘q’ (quartet), ‘m’ (multiplet). Atmospheric pressure chemical ionization (APCI) mass spectra were obtained with Finnigan Mat LCQ mass spectrometer equipped with an APCI source. High resolution mass spectra (HRMS) were obtained with a Waters Alliance 2795 Q-ToF Micromass spectrometer running under Mass Lynx version 4.0 software and equipped with an ESI source. TLC analyses were performed using aluminum backed plates pre-coated with

silica gel containing fluorescent material and examining them under UV light. Flash chromatography was performed by using 40–63 μm silica gel (230–400 mesh) and applying nitrogen pressure from the top of the column.[20]

**General experimental procedure for the deoxygenation of α-hydroxy ketones.** A solution of α-hydroxy ketones **1a-l, 1o-q** (1.0 equiv.) in $CH_2Cl_2$ (3 mL) and $Et_3SiH$ (1.5 equiv.) were placed in a 25 mL two neck round bottom flask (RBF) fitted with a condenser. Aqueous $HClO_4$ (70%, 10 mol%) was added to this reaction mixture. The contents were stirred at 40 °C and progress of the reaction was monitored by TLC analysis. After stirring the contents for appropriate time period, the product was extracted with $CH_2Cl_2$ (3×15 mL), washed with 10% $Na_2CO_3$ and dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure and purification was done by flash chromatography using n-hexane and ethyl acetate (EtOAc) as eluent.

**1,2,2-Triphenylethanone (2a).**[14a] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2a** was obtained as a white solid (90 mg, 96%) from **1a** (100 mg, 0.35 mmol). $R_f$ = 0.40 (n-hexane/EtOAc 9:1); Mp 136-137 °C; $^1$H NMR ($CDCl_3$, 400 MHz): δ = 5.95 (s, 1H; -CH), 7.14-7.25 (m, 10H; ArH), 7.30 (t, J = 7.0 Hz, 2H; ArH), 7.39 (tt, J = 1.8, 1.2 Hz, 1H; ArH), 7.91ppm (dd, J = 7.1, 1.4 Hz, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 100.53 MHz): δ = 59.4, 127.2, 128.6, 128.7, 129.0, 129.1, 133.1, 136.8, 139.1, 198.2 ppm; IR (neat): 3088, 3035, 3002, 2921, 1677 (CO), 1593, 1576, 1492, 1446, 1283, 1203 $cm^{-1}$.

**2-(2-Methylphenyl)-1,2-diphenylethanone (2b).**[16] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2b** was obtained as a colourless oil (175 mg, 92%) from **1b** (200 mg, 0.66 mmol). $R_f$ = 0.52 (n-hexane/EtOAc 9:1); $^1$H NMR ($CDCl_3$, 500 MHz): δ = 2.32 (s, 3H; $CH_3$), 6.12 (s, 1H; -CH),

7.04 (dd, J = 7.7, 1.5 Hz, 1H; ArH), 7.11 (dt, J = 7.2, 1.0 Hz, 1H; ArH), 7.16 (dt, J = 7.5, 1.5 Hz, 1H; ArH), 7.20 (m, 3H; ArH), 7.25 (tt, J = 5.0, 1.0 Hz, 1H; ArH), 7.31 (tt, J = 7.5, 1.0 Hz, 2H; ArH), 7.37 (m, 2H; ArH), 7.47 (tt, J = 5.5, 1 Hz, 1H; ArH), 7.92 ppm (dd, J = 8.5, 1.0 Hz, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 20.0, 56.6, 126.2, 127.1, 127.3, 128.5, 128.6, 128.7, 128.8, 129.6, 130.8, 132.9, 135.8, 136.7, 137.5, 137.8, 198.5 ppm; IR (neat): 3064, 3027, 1683 (CO), 1593, 1580, 1491, 1446, 1274, 1203 $cm^{-1}$.

**2-(3-Methylphenyl)-1,2-diphenylethanone (2c).**[16] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2c** was obtained as a white solid (180 mg, 95%) from **1c** (200 mg, 0.66 mmol). $R_f$ = 0.50 (n-hexane/EtOAc 9:1); Mp 86-87 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 2.29 (s, 3H; $CH_3$), 5.99 (s,1H; -CH), 7.03-7.09 (m, 3H; ArH), 7.17-7.31 (m, 6H; ArH), 7.37 (t, J = 6.5 Hz, 2H; ArH), 7.47 (tt, J = 6.5, 1.0 Hz, 1H; ArH), 7.99 ppm (dd, J = 8.5, 1.0 Hz, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 21.5, 59.4, 126.2, 127.1, 128.0, 128.6, 128.7, 129.0, 129.1, 129.8, 133.0, 136.9, 138.4, 138.9, 139.2, 198.3 ppm; IR (neat): 3060, 3023, 2917, 1682 (CO), 1605, 1576, 1495, 1446, 1274, 1200, 1184 $cm^{-1}$.

**2-(4-Methylphenyl)-1,2-diphenylethanone (2d).**[16] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2d** was obtained as a white solid (160 mg, 84%) from **1d** (200 mg, 0.66 mmol). $R_f$ = 0.50 (n-hexane/EtOAc 9:1); Mp 95-97 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 2.29 (s, 3H; $CH_3$), 5.99 (s, 1H; -CH), 7.17 (d, J = 8.0, 2H; ArH), 7.15 (d, J = 8.0, 2H; ArH), 7.21-7.31 (m, 5H; ArH), 7.38 (t, J = 7.5 Hz, 2H; ArH), 7.47 (tt, J = 6.5, 1.0 Hz, 1H; ArH), 7.99 ppm (dd, J = 8.5, 1.0 Hz, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 21.0, 59.1, 127.0, 128.6, 128.7, 128.9, 129.0, 129.1, 129.5, 132.9, 136.0, 136.8, 136.9, 139.3, 198.3 ppm; IR (neat): 3064, 3027, 2917, 1679 (CO), 1593, 1492, 1446, 1282, 1202 $cm^{-1}$.

**1-(4-Methoxyphenyl)-2,2-diphenylethanone (2e).**[14a] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2e** was obtained as a white solid (70 mg, 74%) from **1e** (100 mg, 0.31 mmol). $R_f$ = 0.37 (n-hexane/EtOAc 9:1); Mp 123-124 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 3.81 (s, 3H; $OCH_3$), 5.99 (s, 1H; -CH), 6.87 (td, J = 6.5, 2.5 Hz, 2H; ArH), 7.21-7.32 (m, 10H; ArH), 7.99 ppm (dd, J = 7.0, 2.0 Hz, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 55.4, 59.1, 113.8, 127.0, 128.7, 129.1, 129.8, 131.3, 139.4, 163.4, 196.7 ppm; IR (neat): 3064, 3031, 2978, 2900, 1673 (CO), 1594, 1493, 1452, 1311, 1266, 1211, 1154 $cm^{-1}$.

**2-(2-Methoxyphenyl)-1,2-diphenylethanone (2f).**[16] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2f** was obtained as a white solid (130 mg, 68%) from **1f** (200 mg, 0.63 mmol). $R_f$ = 0.48 (n-hexane/EtOAc 9:1); Mp 117-118 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 3.72 (s, 3H; $OCH_3$), 6.33 (s, 1H; -CH), 6.83-6.86 (m, 2H; ArH), 6.92 (dd, J = 6.5, 1.5 Hz, 1H; ArH), 7.19-7.21 (m, 1H; ArH), 7.22-7.25 (m, 1H; ArH), 7.29-7.32 (m, 4H; ArH), 7.34-7.37 (m, 2H; ArH), 7.44 (tt, J = 6.0, 1.5 Hz, 1H; ArH), 7.99 ppm (dd, J = 8.5, 1.0 Hz, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 53.1, 55.4, 110.4, 120.6, 127.1, 128.3, 128.4, 128.67, 128.70, 129.7, 132.6, 137.1, 137.6, 156.3, 198.8 ppm; IR (neat): 064, 2933, 2835, 1678 (CO), 1596, 1489, 1465, 1446, 1307, 1244, 1165, 1108 $cm^{-1}$.

**2-(4-Methoxyphenyl)-1,2-diphenylethanone (2g).**[14a] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2g** was obtained as a white solid (145 mg, 76%) from **1g** (200 mg, 0.63 mmol). $R_f$ = 0.42 (n-hexane/EtOAc 9:1); Mp 86-87 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 3.75 (s, 3H; $OCH_3$), 5.98 (s, 1H; -CH), 6.84 (dd, J = 6.5, 1.5 Hz, 2H; ArH), 7.18 (dd, J = 6.5, 1.5 Hz, 2H; ArH), 7.22-7.26 (m, 3H; ArH), 7.30 (t, J = 7.5 Hz, 2H; ArH), 7.38 (t, J = 7.5 Hz, 2H; ArH), 7.48 (t,

J = 7.0 Hz, 1H; ArH), 7.99 ppm (dd, J = 8.5, 1.0 Hz, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 55.2, 58.6, 114.1, 127.0, 128.58, 128.67, 128.93, 129.03, 130.2, 131.1, 133.0, 136.8, 139.5, 158.6, 198.5 ppm; IR (neat): 3027, 2962, 2896, 2839, 1685 (CO), 1597, 1511, 1445, 1255, 1169 $cm^{-1}$.

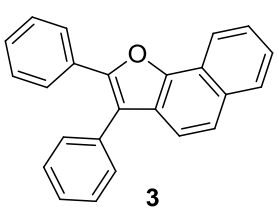
**1,2-bis(3-Methoxyphenyl)-2-phenylethanone (2h).** Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2h** was obtained as a colourless oil (150 mg, 79%) from **1h** (200 mg, 0.57 mmol). $R_f$ = 0.44 (n-hexane/EtOAc 9:1); $^1$H NMR ($CDCl_3$, 500 MHz): δ = 3.74 (s, 3H; -$OCH_3$), 3.79 (s, 3H; -$OCH_3$), 5.98 (s, 1H; -CH), 6.77-6.79 (m, 1H; ArH), 6.81-6.82 (m, 1H; ArH), 6.86 (d, J = 7.5 Hz, 1H; ArH), 7.03-7.06 (m, 1H; ArH), 7.21-7.32 (m, 7H; ArH), 7.52-7.53 (m, 1H; ArH), 7.57 ppm (dd, J = 7.0, 2.0 Hz, 1H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 55.2, 55.3, 59.5, 112.4, 113.3, 115.0, 119.5, 121.54, 121.57, 127.1, 128.7, 129.1, 129.6, 129.7, 138.2, 138.9, 140.5, 159.7, 159.8, 197.9 ppm; IR (neat): 3060, 3002, 2937, 2839, 1682 (CO), 1594, 1580, 1486, 1451, 1426, 1253, 1193, 1160 $cm^{-1}$; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{22}H_{21}O_3$: 333.1486; found: 333.1414.

**1,2-bis(4-Methylphenyl)-2-phenylethanone (2i).**[16] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2i** was obtained as a white solid (180 mg, 95%) from **1i** (200 mg, 0.63 mmol). $R_f$ = 0.46 (n-hexane/EtOAc 9:1); Mp 64-65 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 2.28 (s, 3H; $CH_3$), 2.34 (s, 3H; $CH_3$), 5.97 (s, 1H; -CH), 7.11 (d, J = 8.0 Hz, 2H; ArH), 7.14-7.18 (m, 4H; ArH), 7.21-7.23 (m, 1H; ArH), 7.24-7.31 (m, 4H; ArH), 7.89 ppm (d, J = 8.0 Hz, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 21.0, 21.6, 58.9, 127.0, 128.6, 129.0, 129.08, 129.09, 129.3, 129.4, 134.3, 136.2, 136.7, 139.5, 143.8, 197.9 ppm; IR (neat): 3027, 2921, 1678 (CO), 1603, 1511, 1450, 1406, 1276, 1201, 1176 $cm^{-1}$.

**2-(Naphthalen-1-yl)-1,2-diphenylethanone (2j).**[14a] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2j** was obtained as a white solid (160 mg, 84%) from **1j** (200 mg, 0.59 mmol). $R_f$ = 0.57 (n-hexane/EtOAc 9:1); Mp 110-111 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 6.73 (s, 1H; -CH), 7.20-7.38 (m, 9H; ArH), 7.45-7.49 (m, 3H; ArH), 7.77 (d, J = 8.0 Hz, 1H; ArH), 7.86-7.87 (m, 1H; ArH), 7.96-7.98 (m, 2H; ArH), 7.99-8.01 ppm (m, 1H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 55.9, 123.2, 125.4, 125.8, 126.7, 127.1, 127.3, 128.1, 128.7, 128.79, 128.9, 129.1, 129.6, 131.3, 133.1, 134.2, 135.0, 136.6, 138.1, 198.3 ppm; IR (neat): 3060, 3035, 1682 (CO), 1597, 1449, 1291, 1210, 1177 $cm^{-1}$.

**2-(Naphthalen-2-yl)-1,2-diphenylethanone (2k).** A solution of α-hydroxy ketone **1k** (200 mg, 0.59 mmol) in $CH_2Cl_2$ (3 mL) and $Et_3SiH$ (1.5 equiv.) were placed in a 25 mL round bottom flask (RBF). Aqueous $HClO_4$ (70%, 10 mol%) was added to this reaction mixture. The contents were stirred at 40 °C. After stirring the contents for 70 minute, the product was extracted with $CH_2Cl_2$ (3×15 mL), washed with 10% $Na_2CO_3$ and dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure. The purification was done by flash chromatography (n-hexane/EtOAc 9:1) to obtain the title compound **2k** and a by-product 2,3-Diphenylnaphtho[*1,2-b*]furan (**3**).

**2-(Naphthalen-2-yl)-1,2-diphenylethanone (2k).**[21] White solid (141 mg, 74%); $R_f$ = 0.60 (n-hexane/EtOAc 9:1); Mp 105-106 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 6.19 (s, 1H; -CH), 7.24-7.44 (m, 10H; ArH), 7.48 (tt, J = 7.5, 1.5 Hz, 1H; ArH), 7.69-7.80 (m, 4H; ArH), 8.02-8.04 (m, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 59.5, 126.0, 126.1, 127.2, 127.3, 127.6, 127.8, 127.9, 128.5, 128.6, 128.7, 129.0, 129.3, 132.5, 133.1, 133.4, 136.6, 136.8, 139.0, 198.3 ppm; IR (neat): 3056, 2925, 1686 (CO), 1593, 1499, 1445, 1360, 1282, 1198 $cm^{-1}$.



3

**2,3-Diphenylnaphtho[*1,2-b*]furan (3).**[22] Light yellow solid (14 mg, 7%); $R_f$ = 0.80 (n-hexane/EtOAc 9:1); Mp 97-98 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 7.29 (tt, J = 7.5, 1.5 Hz, 1H; ArH), 7.33-7.36 (m, 2H; ArH), 7.43 (tt, J = 7.5, 1.5 Hz, 1H; ArH), 7.48-7.52 (m, 3H; ArH), 7.55-7.57 (m, 3H; ArH), 7.62 (dt, J = 8.0, 1 Hz, 1H; ArH), 7.58 (d, J = 8.5 Hz, 1H; ArH), 7.73-7.75 (m, 2H; ArH), 7.93 (d, J = 8 Hz, 1H; ArH), 8.43 ppm (d, J = 8 Hz, 1H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 118.5, 118.8, 120.2, 121.2, 123.6, 125.3, 125.6, 126.4, 126.8, 127.7, 128.1, 128.4, 128.5, 129.0, 129.9, 130.9, 131.8, 133.0, 149.6, 150.0 ppm; IR (neat): 3056, 3032, 2938, 1638, 1597, 1577, 1446, 1375, 1274, 1197, 1160, 1068 $cm^{-1}$; HRMS (ESI) m/z $[M + H]^+$ calcd for $C_{24}H_{17}O$: 321.1274; found: 321.1225.

**1,2-bis(2-Fluorophenyl)-2-phenylethanone (2l).** Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2l** was obtained as a white solid (175 mg, 92%) from **1l** (200 mg, 0.62 mmol). $R_f$ = 0.62 (n-hexane/EtOAc 9:1); Mp 105-106 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 5.95 (s, 1H; -CH), 7.00 (tt, J = 8.5, 2.0 Hz, 2H; ArH), 7.05-7.08 (m, 2H; ArH), 7.19-7.27 (m, 5H; ArH), 7.31-7.34 (m, 2H; ArH), 7.99-8.02 ppm (m, 2H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 58.6, 115.5 (d, J = 21.2 Hz), 115.7 (d, J = 21.2 Hz), 127.4, 128.92, 128.96, 130.7 (d, J = 7.5 Hz), 131.7 (d, J = 8.8 Hz), 132.9 (d, J = 3.8 Hz), 134.7 (d, 2.5 Hz), 138.7, 162.0 (d, J = 255.3 Hz), 165.7 (d, J = 246.5 Hz), 196.5 ppm; IR (neat): 3031, 1681 (CO), 1592, 1506, 1413, 1211, 1159 $cm^{-1}$; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{20}H_{15}F_2O$: 309.1086; found: 309.1104.

**2,2-Diphenylethanoic acid (2m)**.[23] A solution of α-hydroxy acid **1m** (500 mg, 2.19 mmol) in $CH_2Cl_2$ (3 mL) and $Et_3SiH$ (1.5 equiv.) were placed in a 25 mL two neck RBF fitted with a condenser. Aqueous $HClO_4$ (70%, 10 mol%) was added to this reaction mixture. The contents were stirred at 40 °C and progress of the reaction was monitored by TLC analysis. After

stirring the contents for 20 h, the product was extracted with $CH_2Cl_2$ (3×15 mL) and dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure and purification was done by flash chromatography (n-hexane/EtOAc 8:2) to give the title compound **2m** as a white solid in 94% yield (435 mg). $R_f$ = 0.60 (n-hexane/EtOAc 7:3); Mp 152-153 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 4.94 (s, 1H; -CH), 7.14-7.17 (m, 2H; ArH), 7.19-7.23 (m, 8H; ArH), 10.93 ppm (s, 1H; -COOH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 57.1, 127.6, 128.75, 128.76, 137.9, 179.0 ppm; IR (neat): 3076, 3019, 2900, 2815, 2700, 1698 (CO), 1495, 1413, 1315, 1221 cm$^{-1}$.

**Methyl 2,2-diphenylacetate (2n)**.[24] A solution of α-hydroxy ester **1n** (500 mg, 2.06 mmol) in $CH_2Cl_2$ (3 mL) and $Et_3SiH$ (1.5 equiv.) were placed in a 25 mL two neck RBF fitted with a condenser. Aqueous $HClO_4$ (70%, 10 mol%) was added to this reaction mixture. The contents were stirred at 40 °C and progress of the reaction was monitored by TLC analysis. After stirring the contents for 24 h, the product was extracted with $CH_2Cl_2$ (3×15 mL), washed with 10% $Na_2CO_3$, and dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure and purification was done by flash chromatography (n-hexane/EtOAc 9:1) to give the title compound **2n** as a white solid in 90% yield (420 mg). $R_f$ = 0.68 (n-hexane/EtOAc 9:1); Mp 63-64 °C; $^1$H NMR ($CDCl_3$, 500 MHz): δ = 3.66 (s, 3H; -$CH_3$), 5.02 (s, 1H; -CH), 7.19-7.22 (m, 2H; ArH), 7.25-7.30 ppm (m, 8H; ArH); $^{13}$C NMR ($CDCl_3$, 125.76 MHz): δ = 52.4, 57.1, 127.4, 128.73, 128.74, 138.8, 173.0 ppm; IR (neat): 3092, 3068, 3023, 2998, 2949, 1722 (CO), 1605, 1491, 1454, 1428, 1359, 1194 cm$^{-1}$.

**3-(4-Methoxyphenyl)butan-2-one (2q)**.[25] Following the general procedure and purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **2q** was obtained as a colourless liquid (70 mg, 38%) from **1q** (200 mg, 1.03 mmol). $R_f$ = 0.48 (n-hexane/EtOAc 9:1); $^1$H NMR ($CDCl_3$, 400 MHz): δ=1.36 (d, J = 7.0 Hz, 3H; -$CH_3$), 2.03 (s, 3H; -$CH_3$), 3.69

(q, J = 7.0 Hz, -1H; CH), 3.79 (s, 3H; -$OCH_3$), 6.86-6.88 (m, 2H; ArH), 7.11-7.14 ppm (m, 2H; ArH); $^{13}C$ NMR ($CDCl_3$, 100.53 MHz): δ = 17.2, 28.2, 52.8, 55.2, 114.3, 128.8, 132.6, 158.7, 209.2 ppm; IR (neat): 2974, 2937, 2835, 1708 (CO), 1605, 1508, 1454, 1353, 1301, 1244, 1177 $cm^{-1}$.

**1,1-Diphenyl methane (5).**[26] Following the general procedure and purification by flash chromatography (n-hexane), the title compound **5** was obtained as a colourless liquid (160 mg, 88%) from **4** (200 mg, 1.08 mmol). $R_f$ = 0.86 (n-hexane); $^1H$ NMR ($CDCl_3$, 500 MHz): δ = 3.97 (s, 2H; $CH_2$), 7.17-7.20 (m, 6H; ArH), 7.25-7.28 ppm (m, 4H; ArH); IR (neat): 3035, 2962, 2904, 2876, 1605, 1503, 1454, 1241, 1069 $cm^{-1}$.

**2-(4-Methoxyphenyl)-1,2,2-triphenylethenone (6).**[13a] A solution of α-hydroxy ketone **1a** (200 mg, 0.69 mmol) in $CH_2Cl_2$ (3 mL) and anisole (1.5 equivalents) were placed in a 25 mL two neck RBF fitted with a condenser. Aqueous $HClO_4$ (70%, 10 mol%) was added to this reaction mixture. The contents were stirred at 40 °C and progress of the reaction was monitored by TLC analysis. After stirring the contents for 2 h, the product was extracted with $CH_2Cl_2$ (3×15 mL), washed with 10% $Na_2CO_3$ and dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure. After purification by flash chromatography (n-hexane/EtOAc 9:1), the title compound **6** was obtained as a white solid in 74% yield (195 mg). $R_f$ = 0.54 (n-hexane/EtOAc 9:1); Mp 138-140 °C; $^1H$ NMR ($CDCl_3$, 500 MHz): δ = 3.75 (s, 3H; $OCH_3$), 6.77 (m, 2H; ArH), 7.12-7.14 (m, 4H; ArH), 7.15-7.24 (m, 9H; ArH), 7.25-7.29 (m, 2H; ArH), 7.66 ppm (dd, J = 8.0, 1.5 Hz, 2H; ArH); IR (neat): 3056, 3031, 2949, 2839, 1675(CO), 1609, 1581, 1507, 1443, 1299, 1250, 1214, 1180 $cm^{-1}$.

## Supporting Information

$^1H$ NMR spectra and $^{13}C$ NMR spectra of final products. APCI mass spectra of **1a**, **1e**, **1g** and **1j** (PDF).

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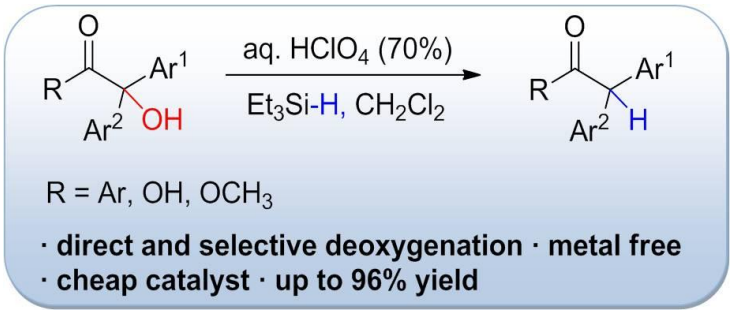
**Table of Contents**




A variety of α-hydroxy-α,α-diaryl carbonyl compounds are selectively deoxygenated to give an important class of α,α-diaryl carbonyl compounds using catalytic amount of aqueous $HClO_4$ (70%) and triethylsilane as hydride source.

Key topic: Deoxygenation