A Green Chemistry Method for the Regeneration of Carbonyl Compounds from Oximes by Using Cupric Chloride Dihydrate as a Recoverable Promoter for Hydrolysis

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Abstract: A mild, efficient, general, and green method for the regeneration of carbonyl compounds from their corresponding oximes is described. Cupric salts promoted hydrolysis of oximes was studied, and the best reaction conditions for the hydrolysis have been found. Carbonyl compounds were obtained in 85–98% yields after the treatment of oximes with 2 molar equivalent of CuCl₂·2H₂O at reflux (around 75 °C) in a mixed solvent of acetonitrile and water (4:1). In addition, cupric salt was readily recovered in an almost quantitative yield via the complete precipitation of Cu(OH)₂·2H₂O.

Key words: carbonyl compounds, oxime, hydrolysis, cupric chloride, green chemistry

Regeneration of carbonyl compounds from the corresponding oximes, that is, deoximation, is a very important reaction,¹ because oximes are often used to protect carbonyl compounds² or as the preferred derivatives for purification and characterization of them.³ Furthermore, when oximes are prepared from noncarbonyl compounds, deoximation provides an alternative approach for the syntheses of aldehydes and ketones.⁴

So far there are four main pathways (Scheme 1) for the deoximation, they include hydrolytic⁵ (method A), reductive⁶ (method B), oxidative⁷ (method C) pathways and transoximation⁸ (method D), respectively.⁹ Due to the relatively hydrolytic stability of oximes, regeneration of carbonyl compounds from oximes via direct hydrolysis (method A) usually needs strong acidic conditions, and thus leads to the damage of acid-sensitive groups and the formation of amides as byproducts by Beckmann rearrangement.¹⁰ Regeneration of carbonyl compounds from oximes via reductive and oxidative pathways (methods B and C) only needs weak acidic conditions, because oximes are reduced or oxidized to form unstable intermediates which are much more susceptible to hydrolysis than oximes themselves. Unfortunately, most of reductive and oxidative methods involve reagents that are often hazardous or very toxic, expensive or not readily available. Moreover, reductive and oxidative pathways often suffer from overreduction and overoxidation of liberated ke-

SYNLETT 2011, No. 7, pp 1028–1032 Advanced online publication: 10.03.2011 DOI: 10.1055/s-0030-1259730; Art ID: W34110ST © Georg Thieme Verlag Stuttgart · New York tones and aldehydes. Regeneration of carbonyl compounds from oximes via exchange with other carbonyl compounds (method D) has also been reported. Oxime exchange with acetone is sluggish,^{8b} but the exchange with levulinic acid,^{8c} pyruvic acid,^{8d} or glyoxylic acid^{8a} is rapid and efficient. However, use of large excess of these oxoacids would certainly retard its wide applications. Herein we would like to report a mild, efficient, general, and green method for the regeneration of carbonyl compounds from the corresponding oximes, in which CuCl₂·2H₂O was used as a recoverable promoter for hydrolysis.



Scheme 1 Main methods for the regeneration of carbonyl compounds from oximes

Although Ragauskas and Jiang have reported a cupric salts catalyzed, ultrasound-promoted dehydration of aldoximes to form nitriles in acetonitrile,¹¹ we thought cupric salts would promote hydrolysis of oximes under aqueous conditions, because the strong coordination ability of copper ion with hydroxylamine would drive the equation (Scheme 2) towards the formation of carbonyl





compounds and a hydroxylamine-coordinated copper complex. With this idea in our mind, and encouraged by Corey's report¹² about the cupric acetate catalyzed facile hydrolysis of *N*,*N*-dimethylhydrazones, we examined copper salts catalyzed hydrolysis of benzophenone oxime (**1a**) under various conditions, the results are summarized in Table 1.

Table 1Optimization of Reaction Conditions for the Conversion ofBenzophenone Oxime (1a) into Benzophenone (2a)

Entry	Reagent (2 equiv)	Solvent (4:1)	Temp (°C)	Time (h)	Yield (%) ^a
1	CuCl ₂ ·2H ₂ O	MeCN-H ₂ O	75 ^b	2	98
2	$CuCl_2 \cdot 2H_2O$	DMF-H ₂ O	80	12	80
3	$CuCl_2 \cdot 2H_2O$	DMSO-H ₂ O	80	10	95
4	$CuCl_2 \cdot 2H_2O$	Me ₂ CO-H ₂ O	54 ^b	12	30 ^c
5	$CuCl_2 \cdot 2H_2O$	EtOH-H ₂ O	72 ^b	12	90
6	$CuCl_2 \cdot 2H_2O$	THF-H ₂ O	64 ^b	12	25°
7	$CuCl_2 \cdot 2H_2O$	<i>i</i> -PrOH–H ₂ O	78 ^b	12	80
8	CuSO ₄	MeCN-H ₂ O	75 ^b	12	86
9	Cu(OAc) ₂ ·H ₂ O	MeCN-H ₂ O	75 ^b	12	82
10	CuBr ₂ ·2H ₂ O	MeCN-H ₂ O	75 ^b	4	93
11	CuCl	MeCN-H ₂ O	75 ^b	12	67°
12	CuBr	MeCN-H ₂ O	75 ^b	12	69°
13	FeCl ₃ ·6H ₂ O	MeCN-H ₂ O	75 ^b	12	85
14	NiCl ₂	MeCN-H ₂ O	75 ^b	12	40 ^c
15	CoCl ₂ ·6H ₂ O	MeCN-H ₂ O	75 ^b	12	45°
16	ZnCl ₂	MeCN-H ₂ O	75 ^b	12	60 ^c
17	Ni(OAc) ₂ ·4H ₂ O	MeCN-H ₂ O	75 ^b	12	2 ^c
18	Co(OAc)2·4H2O	MeCN-H ₂ O	75 ^b	12	10 ^c

^a Isolated yield.

^b Reflux.

 $^{\rm c}$ 30–95% of compound 1a was recovered.

As can been seen from Table 1, the impact of various metal salts on the hydrolysis of oxime 1a in aqueous acetonitrile (entries 1 and 8–18) have been investigated. The metal salts used in our test included cupric chloride dihydrate, cupric sulfate, cupric acetate dihydrate, cupric bromide dihydrate, cuprous chloride, cuprous bromide, ferric chloride hexahydrate, nickel chloride, cobalt chloride hexahydrate, zinc chloride, nickel acetate tetrahydrate, and cobalt acetate tetrahydrate. When 2 mol equivalents of cupric chloride dihydrate or cupric bromide dihydrate were used, the hydrolysis took place smoothly in aqueous acetonitrile, affording benzophenone (2a) in excellent yields (Table 1, entries 1 and 10). Considering cupric chloride dihydrate is much cheaper than cupric bromide dihydrate for the study infra. Solvent effect on the hydrolysis has also been examined. Compared with several other aqueous solvents (Table 1, entries 2–7), aqueous acetonitrile (MeCN– $H_2O = 4:1$) was found to be the best solvent for the hydrolysis (Table 1, entry 1).

We then attempted the CuCl₂·2H₂O-promoted hydrolysis of various oximes in aqueous acetonitrile. As shown in Table 2, a total of 22 oximes (1a–v) were tried, either aldoximes or ketoximes underwent smooth hydrolysis when they were treated with 2 mol equivalents of CuCl₂·2H₂O in aqueous acetonitrile at reflux (75 °C), corresponding carbonyl compounds 2a-v could be obtained in high yields in all instances.¹³ Acid-sensitive groups, such as ester and acetal, kept intact during the hydrolysis (Table 2, entries 2 and 6–8). An α , β -unsaturated aldehyde could be obtained in an excellent yield from the conjugate oxime (Table 2, entry 14). Although hydrolysis of oximes with a dithiane group gave dicarbonyl compounds in high yields (Table 2, entries 18 and 19) due to simultaneous removal of the dithiane group by cupric chloride during the hydrolysis,¹⁴ the sulfide moieties survived after the hydrolysis under the same conditions (Table 2, entries 20 and 21).

Table 2 $CuCl_2 \cdot 2H_2O$ -Promoted Regeneration of Carbonyl Compounds from Various Oximes^a



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 Table 2
 CuCl₂·2H₂O-Promoted Regeneration of Carbonyl Compounds from Various Oximes^a (continued)



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 Table 2
 CuCl₂·2H₂O-Promoted Regeneration of Carbonyl Compounds from Various Oximes^a (continued)



^a Reaction conditions: CuCl₂·2H₂O (2 equiv), refluxing (75 °C) in a mixed solvent of MeCN and H₂O (MeCN-H₂O = 4:1). ^b Isolated yield.

Cupric chloride dihydrate might serve as a coordinating reagent rather than an oxidant in the hydrolysis, because cuprous chloride could also promote the hydrolysis of oxime **1a** albeit in a moderate yield (Table 1, entry 11), and no oxidation of the sulfide groups of oximes occurred through the hydrolysis (Table 2, entries 20 and 21).

Two mol equivalents of cupric chloride dihydrate were necessary for the above-described deoximation, when less than 2 equivalents of cupric chloride dihydrate were used, the yields of carbonyl compounds would be diminished. Cupric chloride was washed into an aqueous solution during the workup procedure.¹³ However, from a green chemistry point of view,¹⁵ cupric salt should be recovered herein to prevent any possible environmental pollution. Fortunately, the cupric salt could be readily recovered in an almost quantitative yield just by adding 2.2 mol equivalents of NaOH into the above aqueous extract to allow

Cu(OH)₂·2H₂O completely precipitated as a bluish solid.¹⁶ Moreover, when the above Cu(OH)₂·2H₂O was converted into CuCl₂·2H₂O, it could be reused for the hydrolysis of oximes without any observable difference from the commercial one.¹⁷

In conclusion, a mild, efficient, and general method for the regeneration of carbonyl compounds from their corresponding oximes has been developed. Some merits such as simple manipulation, mild reaction conditions, high yields, wild scope of application, cheapness of cupric chloride dihydrate, as well as the complete recovery of the cupric salt might allow this green method to be very useful for the regeneration of ketones and aldehydes from various ketoximes and aldoximes.

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- (13) Typical Procedure for the CuCl₂·2H₂O-Promoted Regeneration of Carbonyl Compounds from Various Oximes

Oxime **1a** (1.01 g, 5.12 mmol) was dissolved in MeCN (20 mL), CuCl₂·2H₂O (1.73 g, 10.15 mmol) and H₂O (5 mL) were added. When the suspension was heated to reflux, the mixture became a bluish clear solution. The resulting reaction solution was then stirred at reflux (75 °C) for around 2 h and monitored by TLC (EtOAc–hexane, 1:6). After the reaction was complete, the solvents were removed by vacuum distillation. The residue was partitioned between EtOAc (50 mL) and H₂O (30 mL), the organic and aqueous phases were separated. Organic phase was washed with brine (5 mL) and dried over anhyd MgSO₄. Concentration of the organic solution gave crude product, which was purified by flash chromatography to afford benzophenone (**2a**, 0.914 g, 5.02 mmol) in 98% yield. To the above-mentioned

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aqueous phase was added an aq solution of NaOH (11.0 mL, 2 M, 22.00 mmol). After vigorous stirring for 1 h, the bluish solid was collected on a Buchner funnel by suction. After being dried in a warm air at around 50 °C for 12 h until the weight of the solid kept constant, $Cu(OH)_2 \cdot 2H_2O$ (1.34 g, 10.03 mmol) was recovered in 99% yield.

Spectral analysis showed that compounds **2a–e,i–l,n–s,v** obtained from the above hydrolysis are identical with the commercially available authentic samples. Characterization data of compounds **2f–h,m,t,u** are as follows:

Compound **2f**: ¹H NMR (400 MHz, CDCl₃): δ = 3.89 (s, 3 H), 7.35 (d, *J* = 7.9 Hz, 1 H), 7.48–7.57 (m, 4 H), 7.62–7.69 (m, 1 H), 8.18–8.24 (m, 2 H), 9.98 (s, 1 H). MS (EI): *m/z* (%) = 256 (4) [M⁺], 217 (18), 182 (2), 155 (2), 105 (100), 77 (17). IR (KBr): v = 3005, 2885, 1735, 1680, 1600, 1505, 1450, 1400, 1255, 1190, 1140, 1120, 1060, 1025, 850, 805, 730, 700 cm⁻¹.

Compound **2g**: ¹H NMR (400 MHz, CDCl₃): $\delta = 1.30$ (t, J = 7.1 Hz, 3 H), 4.30 (q, J = 7.1 Hz, 2 H), 4.71 (s, 2 H), 7.02 (d, J = 6.9 Hz, 2 H), 7.85 (d, J = 6.9 Hz, 2 H), 9.90 (s, 1 H). IR (neat): v = 2980, 2835, 2770, 1755, 1690, 1600, 1510, 1440, 1380, 1310, 1280, 1205, 1160, 1080, 1025, 835, 715, 610 cm⁻¹. HRMS (EI): m/z calcd for C₁₁H₁₂O₄ [M⁺]: 208.0736; found: 208.0730.

Compound **2h**: ¹H NMR (400 MHz, CDCl₃): $\delta = 1.30$ (t, J = 7.1 Hz, 3 H), 3.96 (s, 1 H), 4.28 (q, J = 7.1 Hz, 2 H), 4.79 (s, 2 H), 6.84 (d, J = 8.1 Hz, 1 H), 7.41–7.46 (m, 2 H), 9.87 (s, 1 H). IR (KBr): v = 2980, 2940, 2910, 1750, 1680, 1590, 1510, 1470, 1430, 1395, 1270, 1200, 1140, 1070, 1030, 870, 810, 780, 735, 640 cm⁻¹. HRMS (EI): m/z calcd for $C_{12}H_{14}O_5$ [M⁺]: 238.0841; found: 238.0839.

Compound **2m**: ¹H NMR (400 MHz, CDCl₃): δ = 7.52 (dd, J_1 = 1.6 Hz, J_2 = 8.6 Hz, 1 H), 7.67 (d, J = 1.6 Hz, 1 H), 8.29

(d, J = 8.6 Hz, 1 H), 10.07 (s, 1 H), 10.58 (s, 1 H). IR (KBr): v = 3255, 3085, 2870, 1700, 1620, 1585, 1530, 1480, 1450, 1315, 1255, 1165, 1140, 1080, 985, 850, 760, 705, 540 cm⁻¹. MS (EI): m/z (%) = 167 (100) [M⁺], 166 (44), 151 (1), 136 (3), 119 (7), 109 (4), 92 (3), 81 (3), 63 (6). Compound **2t**: ¹H NMR (400 MHz, CDCl₃): δ = 2.28 (s, 3 H), 3.50–3.69 (m, 2 H), 4.88 (dd, J_1 = 6.1 Hz, J_2 = 8.1 Hz, 1 H), 7.03 (d, J = 7.9 Hz, 2 H), 7.13–7.26 (m, 5 H), 7.28–7.35

(m, 2 H), 7.37–7.44 (m, 2 H) 7.48–7.55 (m, 1 H), 7.82–7.89 (m, 2 H). MS (EI): m/z (%) = 332(4) [M⁺], 209 (5), 179 (2), 123 (13), 105 (100), 91 (6), 77 (59). IR (KBr): v = 3035, 2920, 1685, 1595, 1490, 1450, 1420, 1335, 1220, 980, 810, 745, 700, 685, 550 cm⁻¹. Compound **2u**: ¹H NMR (400 MHz, CDCl₃): δ = 2.05 (s, 3 H), 2.29 (s, 3 H), 2.92–3.08 (m, 2 H), 4.63 (dd, J_1 = 6.8 Hz, J_2 = 7.8 Hz, 1 H), 7.03 (d, J = 8.0 Hz, 2 H), 7.18 (d, J = 8.0 Hz, 2 H), 7.19–7.26 (m, 5 H). MS (EI): m/z (%) = 270 (32) [M⁺], 213 (2), 147 (43), 124 (100), 103 (5), 91 (12), 77 (7), 43 (43). IR (neat): v = 3030, 2940, 1720, 1490, 1455, 1410, 1360, 1150, 1020, 810, 700, 535, 500 cm⁻¹.

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- (17) Conversion of Cu(OH)₂·2H₂O into CuCl₂·2H₂O The above Cu(OH)₂·2H₂O was first heated at 150 °C for around 6 h, and the resulting brown anhyd CuO was then treated with 2.2 molar equivalent of aq HCl at reflux for 2 h. Removal of H₂O under vacuum at 45 °C gave blue crystalline CuCl₂·2H₂O in a nearly quantitative yield.

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