



Hydrodehalogenation

Regioselective Hydrodehalogenation of Aromatic α - and β -Halo carbonyl Compounds by Cul in Isopropanol

Iram Parveen,^[a] Danish Khan,^[a] and Naseem Ahmed*^[a]

Abstract: An operationally efficient and regioselective hydrodehalogenation methodology of aromatic α - and β -halo carbonyl compounds has been developed using Cul in isopropanol at 90 °C under basic condition. The catalytic system effectively dehalogenates chloride, bromide, and iodide groups and afforded high yield (up to 97 %) as carbonyl compounds. The methodology is environmentally friendly and demonstrates excellent tolerance to a broad range of electronically rich and poor substituents.

Introduction

In the past decades, copper salts are used as innocuous, inexpensive and mild catalyst in diverse organic syntheses and has been found to be vastly effective for the carbon-carbon and Cheteroatoms (X = N, O, S, P, Se, etc.) bond formations.^[1] Organic halides are widely used as starting material and intermediates in numerous chemical transformations, and their effective C-X (X = Br, Cl, I) bond cleavage has been actively studied. Still, a substantial progress is needed towards the development of easy dehalogenation protocols, to eliminate halogenated pollutants and for their other synthetic applicabilities.^[2,3] To date, set of different catalysts and reagents have been employed for the same. Traditionally, for hydride source molecular H₂ is used for dehalogenation of halides with Pd/C-Et₃N^[4] and other palladium catalysts,^[5] palladium and rhodium nanoparticles.^[6,7] Different photocatalyzed methodologies are also been documented for the dehalogenation reactions with alcohols, amine and water as hydrogen transferring agents under light irradiation conditions.[8-14]

Strikingly, some alternative hydride source for carrying out the dehalogenation reactions with different transition metals have also been described, such as palladium with the usage of polymethylhydrosiloxane (PMHS) or sodium formate,^[15] Palladium, Rhodium and Iridium complexes with bidentate ligand involving silane,^[16] Pd^{II} through cyclic amine,^[17] Pt^{II} with hydrosilanes,^[18] Palladium involving DMF,^[19] Ruthenium(II) phosphine complex, Rh-complex, palladium by phosphine and carbene ligand and Ni–Al clusters with alcohol,^[20] Palladium and Nickel include alkoxides,^[21] halogen–metal exchange reaction,^[22] Pd/C contain hydrazine hydrochloride,^[23] Indium-catalyzed dehalogenation of halo hetero-arene in water,^[24] Pd⁰ and iron

https://www.iitr.ac.in/departments/CY/pages/People+Faculty+nasemfcy.htm Supporting information and ORCID(s) from the author(s) for this article are available on the WWW under https://doi.org/10.1002/ejoc.201801385. with Grignard reagents through intramolecular β -hydride elimination of aryl halide,^[25] cobalt porphyrin in the presence of

KOH,^[26] transition metals-catalyzed via metal hydride,^[27] KHSO₄-Na₂SO₃ system,^[28] Pd/C with ammonium formate in methanol^[29] and NaBH₄ with TMEDA^[30] are usually accessible for the reductive dehalogenation transformation. Recently, Xia et al. described a Ru-catalyzed dehalogenation protocol for the hydrodehalgenation of organic halides.^[31] MacArthur at al. developed an inexpansive method for the hydrodehalogenation of aryl chlorides and aryl bromides using copper catalyst in the presence of diamine ligand.^[32] These methods are allied with some of the serious drawbacks such as use of expensive catalyst, requirement of phosphine, carbene or other auxiliary ligands, which are either air sensitive, toxic or difficult to prepare and used only for Br and I groups. Hence, expansion of an innovative catalytic process for the selectively ortho-hydrodehalogenation is highly desirable. Herein, we report a novel Cu-catalyzed protocol for the regioselectively hydro dehalogenation of β -halo-chalcone, α -and β -halo-flavone and different α -aryl halides using isopropanol as a hydrogen donor solvent under milder reaction conditions.

Results and Discussion

Our initial optimization commenced with the identification of proper catalyst and appropriate reaction conditions. A model reaction was performed with 3-bromoflavone (**1**) using 10 mol-% of copper iodide in the presence of formic acid as hydrogen donating solvent with K_2CO_3 at r.t. to 90 °C for 24 h (Table 1, entry 1). Unfortunately, the reaction failed to yield the desired product. Further, the usage of acetic acid or TFA was also found ineffective (Table 1, entries 2 and 3). To our delight the formation of hydrodehalogenated product flavone (**2b**) was obtained in 36 % and 16 % yield respectively, when used HCOOH/Et₃N (7:3 and 1:1 molar ratio) under similar conditions (Table 1, entries 4 and 5). Gratifyingly, application of isopropanol as a hydride source and 3 mol-% of Cul offered the desired product in 38 % yield (Table 1, entry 6). Under this condition, the reac-

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Table 1. Optimization of reaction conditions for the reaction 3-bromoflavane 1.^[a]



Entry	Catalyst	Additive	Solvent	Temp.	Time	Yield ^[g]
	(10 1101-70)	(5 equiv.)		[C]	[11]	[70]
1	Cul	K ₂ CO ₃	HCOOH	r.t. to 90	24	NR
2	Cul	K ₂ CO ₃	CH₃COOH	r.t. to 90	24	NR
3	Cul	K ₂ CO ₃	CF₃COOH	r.t. to 90	24	NR
4	Cul	-	HCOOH/	r.t. to 90	12–24	36
			Et ₃ N (7:3)			
5	Cul	-	HCOOH/	r.t. to 90	24	16
			Et ₃ N (1:1)			
6 ^[b]	Cul	K ₂ CO ₃	<i>i</i> PrOH	90	24	38
7 ^[c]	Cul	K ₂ CO ₃	<i>i</i> PrOH	90	24	52
8 ^[d]	Cul	K ₂ CO ₃	<i>i</i> PrOH	90	24	71
9 ^[e]	Cul	K ₂ CO ₃	<i>i</i> Pr OH	90	12	92
10 ^[f]	Cul	K ₂ CO ₃	<i>i</i> PrOH	90	24	91
11	-	-	<i>i</i> PrOH	r.t. to 90	24	NR
12	Cul	-	<i>i</i> PrOH	90	24	NR
13	CuBr	K ₂ CO ₃	<i>i</i> PrOH	90	12	75
14	CuCl	K ₂ CO ₃	<i>i</i> PrOH	90	12	64
15	Cul	K ₂ CO ₃	EtOH	90	36	NR
16	Cul	K ₂ CO ₃	MeOH	90	36	NR
17	Cul	K ₂ CO ₃	<i>t</i> BuOH	90	12	NR
18	Cul	Na ₂ CO ₃	<i>i</i> PrOH	90	12	82
19	Cul	KOH	<i>i</i> PrOH	90	12	79
20	Cul	NaOAc	<i>i</i> PrOH	90	12	75
21	Cul	Cs ₂ CO ₃	<i>i</i> PrOH	90	12	92
22 ^[f]	Cul	K ₂ CO ₃	iPrOH	90	12	89

[a] Reaction conditions unless specified otherwise: 1 (1.0 mmol), catalyst, additive (3 equiv.), solvent as hydrogen donor (3 mL) stirred at 90 °C for 12–24 h. [b] Cul (3 mol-%). [c] Cul (5 mol-%). [d] Cul (7 mol-%). [e] Cul (10 mol-%). [f] Cul (15 mol-%). [g] Under N_2 .

tion showed incomplete conversion of starting material even after 24 h. Wherein, a noticeable betterment in the isolated yield up to 71 % was noticed with the gradual increase in the amount of catalysts loading from 3 mol-% to 7 mol-% at 90 °C for 24 h (Table 1, entries 7 & 8) with the trace amount of starting material. Delightfully, the employment of 10 mol-% catalyst has offered the desired product in best yield of 92 %, with reducing the reaction time up to 12 h (Table 1, entry 9) with complete conversion. Moreover, further increment in the catalyst loading has not aid any amelioration in the yield and time (Table 1, entry 10). Notably, the reaction failed to proceed at all in the absence of catalyst and base, signifying the crucial importance of catalyst and base for the described transformations (Table 1, entries 11 & 12). The use of other copper salts such as CuBr and CuCl, resulted in the lower isolated yield, i.e. 75 % and 64 % respectively under similar reaction condition (Table 1, entries 13 and 14). Use of other hydrogen donating solvents such as; EtOH, MeOH and tBuOH failed in flourishing the desired product (2b) because of tertiary butanol do not have hydrogen on α -carbon atom (Table 1, entries 15, 16 and 17).

We have also conducted study of different bases (additives) which showed that the yield of the product **2b** decreased dramatically (Table 1, entries 18 to 20). But in case of Cs_2CO_3 similar results were obtained. Among the reactions, the best result was

achieved by reaction in *i*PrOH solvent with K_2CO_3 as the additive, which afforded **2b** in 92 % yield (Table 1, entry 9). Under nitrogen, the reaction of **1** afforded slightly less yield (89 %) of the product **2b** under similar conditions (Table 1, entry 22). It might be that the aerial oxygen is helpful in forward reaction (oxidation).

With the optimal reaction condition in hand, substrate scope of the dehalogenation reaction was also attempted with simple α -bromo carbonyl derivatives such as α -halogenated benzaldehyde, benzamide, acetophenone, benzoic acid and esters underwent reduction effectively to get 91–97 % yields (Scheme 1).



Scheme 1. Substrate scope of α -halo derivative. Reaction conditions: **1aa** (1.0 mmol), catalyst, (10 mol-%), additive (3 equiv.) Isopropanol (3 mL) heated at 90 °C for 12 h.







Scheme 2. Substrate scope of 3-bromo flavone. Reaction conditions: 1 (1.0 mmol), catalyst, (10 mol-%), additive (3 equiv.), solvent as hydrogen donor i.e. Isopropanol (3 mL) stirred at 90 °C for 12 h.



Scheme 3. Substrate scope of β' -halochalcone. Reaction conditions: **3** (1.0 mmol), catalyst, (10 mol-%), additive (3 equiv.), solvent as hydrogen donor i.e. Isopropanol (3 mL) stirred at 90 °C for 12 h.





After α -halo carbonyls derivatives, we described the substrate scope for a series of 3-bromo flavone (α -halo carbonyl compounds) bearing different electron withdrawing and electron donating substituents (Scheme 2). As expected, the reduction of the C-I bond is slightly more efficient in comparison to C-Br and C-Cl bonds yielding the desired product 2a in 95 %, 92 % and 86 % yields respectively. Without substituents, the presence of halogen group (F, Br and Cl) on the 2-phenyl ring has not shown much alteration in yields of 2b-2e. Seminally, the presence of methoxy, dimethoxy, trimethoxy functional groups have also not shown noticeable variation, offering the described product 2f-2h in 81-84 % vields. However, nitro group containing derivatives showcased sluggish reactivity, resulting in the formation of targeted product **2i** in comparatively lower yields. Gratifyingly, the methodology was also successful with 3-bromo-2-(furan-2-yl)-4H-chromen-4-one, offering 2j in 82 % yield.

To extend the substrate scope of the present methodology, we explored the reaction with β' -halo chalcone derivatives, bearing different electron donating and withdrawing groups. Under the present condition, the reaction has not shown any noticeable impact of electronic environments, offering the respective products **3a**–**3i** in seminal yields (88–92 %) (Scheme 3). Moreover, 2-halo indoline chalcone was also successfully dehalogenated under the current reaction conditions, affording product **3j** in 82 % yield (Scheme 4).



Scheme 4. Substrate scope of 2-haloIndoline Chalcone.

It is noteworthy that the methodology showcased excellent selectivity in reducing the halogen functionalities, specifically reduction of α - and β -halo functional groups over the other positions.

Furthermore, the scope of the methodology was expanded by without and with substituted 4-chloro-3-acetyl-4-chlorocoumarin (Scheme 5). It was noted that, 4-chloro-3-acetyl-4chlorocoumarin bearing electron donating group like OH and OMe and without groups (**4a**–**4c**) offered smoothly the corresponding products in 89 % to 85 % yields respectively. Wherein other derivatives containing Br, di-Cl, and NO₂ functional groups (**4d**–**4f**) for example fused 4-cholro-3-acetyl-4-chlorocoumarin contentedly produces the same result, albeit in slight lesser yield, i.e., **77** %.

In order to demonstrate the reaction pathway via hydrogen transfer reaction, we performed the control experiments using deuterated isopropanol with different substrates (Scheme 6).



Scheme 6. Controlled Experiment.







Scheme 5. Substrate scope of 3-acetyl-4-chlorocoumarin. Reaction conditions: 4 (1.0 mmol), catalyst, (10 mol-%), additive (3 equiv.), Solvent as hydrogen donor i.e. Isopropanol (3 mL) stirred at 90 °C for 12 h.







Scheme 8. Proposed mechanism.

Deuteration of the product instead of hydrogenation provides a transparent support for the involvement of isopropanol in this transformation. All deuterated compounds were confirmed by HRMS analysis (see supporting information). To extend our work, we also studied the possible reactions with aryl halide and p-bromo acetophenone under the same reaction conditions, but no product was detected and the starting materials were isolated. This experiment indicates that chelating group (carbonyl) is necessary for the reaction (Scheme 7).

Based on previous literature reports,^[14,33] a plausible mechanism for the copper catalyzed hydrodehalogenation reaction is proposed in Scheme 8. Where Cu^I iodide initially undergoes oxidative addition with halide to generate a Cu^{II} complex intermediate **A**. Subsequently, by mean of base isopropanol afforded isopropyl oxide ligand which on nucleophilic substitution of bromo group gave intermediate **A** or **B** or Cu^{III}-hydride complex, i.e. intermediate **C**. Followed by hydride transfer from isopropyl oxide and removal of CuBr, generating acetone, which was confirmed by GC–MS. Eventually, intermediate **B** or **C** undergoes reductive elimination to produce the targeted product with the regeneration of catalyst which continues the cycle.^[34]

Conclusions

In summary, we have described an easy, efficient and simple hydrodehalogenation protocol of α - and β -carbonyl compounds, under copper iodide catalytic condition. This method is featured with cheap, commercially and easily available copper iodide catalyst with isopropanol turning both as solvent and hydrogen source without any additional ligand, with broader substrate scope.

Experimental Section

General Information: All the required chemicals were purchased from Avra, Himedia and Sigma Aldrich. Thin layer chromatography was performed on 0.25 mm silica gel plates (60F–254) using UV light as the visualizing agent. Silica gel (100–200 mesh) was used for column chromatography. Melting points were determined on a capillary point apparatus equipped with a digital thermometer and are uncorrected. Nuclear magnetic resonance spectra were recorded on Jeol resonance ECX400MHz and Bruker Spectro spin DPX 500 MHz spectrometer, and chemical shifts are reported in δ units,

parts per million (ppm), relative to residual chloroform (7.26 ppm) in the deuterated solvent or with tetramethylsilane (TMS, δ = 0.00 ppm) as the internal standard. ¹³C NMR spectra were referenced to CDCl₃ (δ = 77.00 ppm, the middle peak). Coupling constants were expressed in Hz. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, m = multiplet. High resolution mass spectra were recorded with a micro TOF-Q analyzer spectrometer by using the electro-spray mode. IR spectra of the compounds were recorded on Thermo Nicolet FT-IR spectrometer with and are expressed as wave number (cm⁻¹).

General Procedure: A round-bottomed flask was charged with 3bromo substrate (1 mmol), potassium carbonate (3 equivalent) in isopropanol (3 mL), with copper iodide (10 mol-%). The reaction mixture was stirred at 90 °C until complete consumption of the starting material as detected by TLC. After completion of the reaction, the reaction mixture was cooled to ambient temperature and extract with ethyl acetate. The residue was purified via column chromatography with excellent yield (**2a–4g**).

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An efficient and regioselective hydrodehalogenation of aromatic α - and β halo carbonyl compounds has been developed using Cul in isopropanol under basic condition. This reaction system effectively reduces chloride, bromide and iodide groups and af-

fords high yield (up to 97 %) as carbonyl compounds. The method is environmentally friendly and demonstrates excellent tolerance to a broad range of electronically rich and poor substituents.

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