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Benzylamine promoted direct C–H arylation of arenes and heteroarenes *via* excitation with heat or light†

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Carbon–halogen bond cleavage in aryl halides through single electron transfer (SET) is a crucial step in radical-based cross-coupling reactions. Accomplishing such cleavage using an organic system without the assistance of any transition metal-based catalyst is highly challenging. In recent years, combining organic molecules and a base has served as a unique system for SET-mediated carbon–halogen bond cleavage. Herein, we report the combination of simple benzylamine and potassium *tert*-butoxide as a super-electron-donor system for SET-mediated cleavage of aryl halides generating reactive aryl radicals, which subsequently react with arenes or heteroarenes and produce biaryl skeletons. The new methodology enables the arylation of arenes and heteroarenes with aryl iodides, or aryl bromides, upon excitation with heat or light. The broad substrate scope, mild reaction conditions and tolerance of common organic functional groups offer an efficient alternative route for direct C–H arylation reactions.

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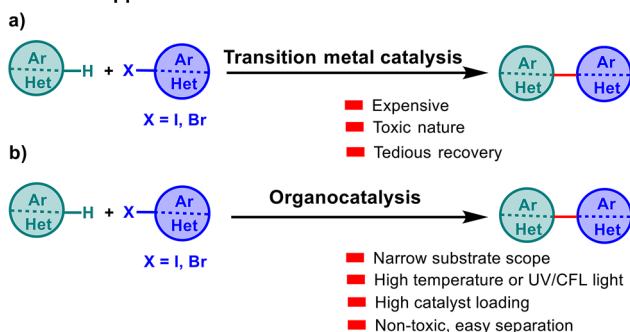
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Introduction

Transition-metal-catalyzed cross coupling reactions of aryl halides (Ar–X) with arenes or heteroarenes C–H bonds are considered as the most powerful and efficient synthetic routes^{1–5} for the construction of ubiquitous biaryl structural motifs found in pharmaceuticals, agrochemicals, and functional materials.^{6–10} However, high economic costs, toxicity, and the difficulty of separating the transition metal catalyst from the final products create obstacles in industrial applications (Scheme 1a).¹¹ Therefore, to overcome these issues, transition metal-free cross-coupling reactions have emerged as an alternative strategy for the direct C–H arylation of arenes or heteroarenes with Ar–X in recent years. To date, a series of organic molecules with nitrogen or oxygen donor atoms, such as DMEDA (*N,N'*-dimethylethane-1,2-diamine),^{12,13} 1,10-phenanthroline,¹⁴ 2,9-diphenyl-1,10-phenanthroline,¹⁵ amino-linked nitrogen heterocyclic carbenes,^{16,17} phenylhydrazine,¹⁸ pyridonepentamer,¹⁹ amino acids,²⁰ 1,2-diamines,²⁰ alcohols,^{20,21} *p*-toluenesulfonylhydrazide,²² *N*-methylanilines,²³ 9-methylamino-phenalen-1-one,²⁴ diketo-piperazines,²⁵ indo-

line,²⁶ *etc.* (Scheme 1b), have been found to promote aryl halide activation under heating conditions (≥ 80 °C) in the presence of a base such as potassium *tert*-butoxide (KO^tBu),

Previous approaches:



Our approaches:



Scheme 1 Comparison of previous approaches with our approach for the C–H arylation of arenes and heteroarenes using aryl or heteroaryl halides.

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producing a reactive aryl radical intermediate through a single electron transfer (SET) mechanism that undergoes substitution reactions with arenes or heteroarenes and forms a carbon-carbon bond. Interestingly, potassium *tert*-butoxide KO^tBu, a common base used in combination with additives for cross-coupling reactions, can alone perform the reaction under relatively harsh reaction conditions when coupling is carried out between Ar-I or Ar-Br and pyridazine or other electron-poor aromatic rings having activated C-H.²⁷ Despite these advancements, the Ar-I coupling partner primarily controls the cross-coupling reactions as it dissociates at a lower potential. However, finding an efficient catalytic system for activating relatively inert Ar-Br or Ar-Cl remains challenging. This is due to the high reduction potential of their C-Br/C-Cl bonds of Ar-Br/Ar-Cl and an increase in their bond strength. To date, only a few organocatalytic cross-couplings using Ar-Br are known.²⁶ The coupling reactions using Ar-Cl are very rare.²⁶ To the best of our knowledge, the reported conventional organocatalytic cross-coupling methods suffer from harsh reaction conditions and limited substrate scope.

On the other hand, the application of visible-light photoredox in organic synthesis has gained increasing interest in recent years.^{28–40} In the field of direct C-H arylation, Rossi's group³¹ showed the arylation of benzene and thiophene using KO^tBu and dimethyl sulfoxide (DMSO) solvents at room temperature (RT) without any additives when a UV-Vis light ($\lambda \geq 350$ nm) source equipped with two Philips HPI-T 400 W lamps (cooled with water) is applied. Su's group used a 24 W household compact fluorescent light bulb (CFL) in a reaction similar to that of the Rossi group and achieved similar results.³² Li's group introduced Ir(ppy)₃, an effective photoredox catalyst for C-H arylation under irradiation with a visible light source.³³ An exciting result was found in 2015 by Yuan's group for visible light induced photocatalytic C-H arylation using a photosensitive complex of KO^tBu and nitrogenous heterocyclic ligands as promoters.³⁴ After that, advancements in a few more methods were made for visible light-mediated photo-induced C-H arylation of arenes and heteroarenes using Ar-X at RT.^{35–40} Based on all these results, we took into consideration that the nitrogenous molecule benzylamine whose N-H proton acidity is between those of the early reported systems of *N*-methylbenzylamine²³ and phenyl hydrazine¹⁸ might show better catalytic activity towards the activation of Ar-X. The first molecule shows poor catalytic activity because it is hard to deprotonate its N-H proton. The second molecule, on the other hand, is easily deprotonated, but the deprotonated form does not have much potential for the SET process. Herein, we have established that the combination of benzylamine (**1**) and KO^tBu acts as a highly efficient promoter for the C-H arylation of arenes and heteroarenes using Ar-X under conventional heating at 90 °C as well as irradiation with visible light (blue) at RT (Scheme 1c). This is an example of a single catalyst for C-H arylation in dual excitation mode. Compared with 2-picoylamine (**2**), 1-naphthylmethylamine (**3**) and diphenylmethanamine (**4**) (Fig. 1), **1** can more efficiently promote the C-H arylation of arenes and heteroarenes using aryl or heteroaryl

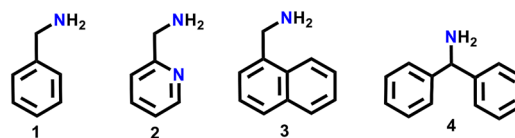


Fig. 1 Different aryl methanamines used as catalysts for screening in the C-H arylation reaction.

halides with a broad substrate scope under identical conditions (Scheme 1c).

Results and discussion

C-H arylation under conventional heating conditions

Our initial studies involved the use of different aryl methanamines (**1–4**, Fig. 1) as promoters for the C-H arylation reaction between the model coupling partners benzene (**5a**) and iodobenzene (**6a**) in the presence of KO^tBu under conventional heating conditions. Upon optimization of several reaction parameters (Scheme S1† and Table 1), 86% isolated yield of phenylbenzene (**7a**) was achieved at 90 °C in the presence of 3 equiv. of KO^tBu, 30 mmol% (0.3 mmol) of benzylamine (**1**, promoter), and 1 mmol of 18-crown-6 additive after 36 h (Table 1, entry 1). Under these conditions (Table 1, entry 1), the performance of other aryl methanamines (**2–4**) is comparatively low (Table 1, entries 2–4). In the absence of **1**, when the C-H arylation was carried out using only the additive 18-crown-6 and base KO^tBu, the yield of the expected product **7a** was <20%, which confirms the importance of **1a** (Table 1, entry 5).

Table 1 Optimization study for the C-H arylation of benzene (**5a**) with iodobenzene (**6a**)^a

Entry	Promoter (mol%)	18-Crown-6 (equiv.)	KO ^t Bu (equiv.)	T (°C)	Time (h)	Yield ^b (%)
1	1 (30)	1	3	90	36	86
2	2 (30)	1	3	90	36	67
3	3 (30)	1	3	90	36	78
4	4 (30)	1	3	90	36	74
5	—	1	3	90	36	<20
6	1 (30)	1	—	90	36	—
7	1 (30)	—	3	90	36	69
8	1 (20)	1	3	90	36	71
9	1 (30)	1	2	90	36	75
10	1 (30)	0.5	3	90	36	82
11	1 (30)	1	3	80	36	80
12	1 (30)	1	3	90	30	82
13	1 (40)	1	3	90	36	86
14	1 (30)	2	3	90	36	87

^a The reactions of benzene (**5a**, 1.5 mL) with iodobenzene (**6a**, 1 mmol) were performed in sealed tubes in the presence of promoters **1–4** (0 to 0.3 mmol), KO^tBu base (0–3 mmol), and additive 18-crown-6 (0–1 mmol) under inert and dry conditions. ^b Isolated yield.

No reaction was observed without the base KO^tBu (Table 1, entry 6). It was found that the additive 18-crown-6 plays a significant role in the yield of the product (Table 1, entry 7). Furthermore, we tried to improve the reaction conditions and yield of the reaction by varying the loading of promoter 1, amount of KO^tBu, amount of 18-crown-6, temperature and time, but no further significant improvement was noted (Table 1, entries 8–14). A kinetics study at different time intervals (5, 10, 15, 20, 25, 30, 35 and 40 h) shows the product evolution over time, indicating that within 35 h, the reaction reaches near completion (Fig. S1, ESI†).

Under our optimized conditions (Table 1, entry 1), the product phenylbenzene, **7a**, was formed selectively, and no biarylation was found. The optimized conditions were further applied for the arylation of benzene, **5a**, with different aryl halides (**6**) (Ar–X) such as aryl iodides, aryl bromides, heteroaryl iodides and heteroaryl bromides, which led to the desired product **7b–k** with a good to excellent yield (64–92%, Table 2).

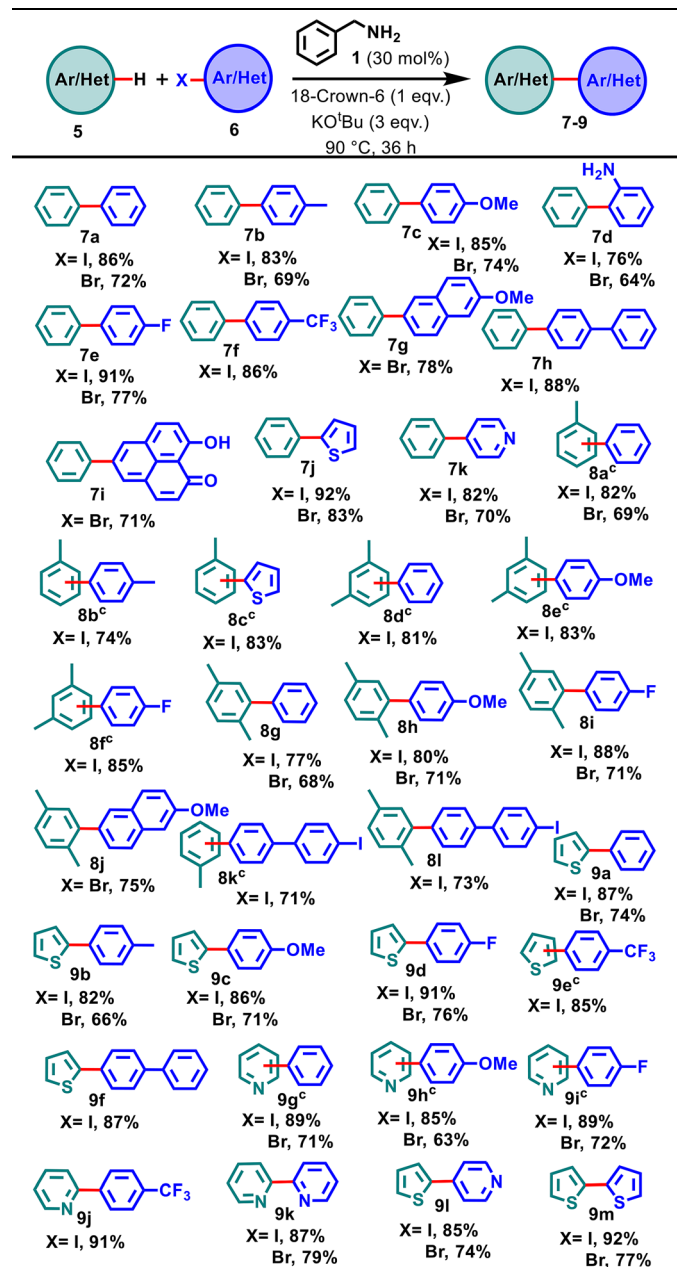
After the successful arylation of benzene, we moved to the arylation of more electronically rich systems such as toluene and xylene using different Ar–X (X = I and Br) under optimized conditions and isolated the corresponding monoarylated products **8a–l** with good yields (68–88%, Table 2). Therefore, the benzylamine promoter works well because it can arylate electronically rich arenes like toluene and xylenes at 90 °C.

Next, we also applied our optimized reaction conditions (Table 1, entry 1) to the arylation of heteroarenes such as pyridine and thiophene using different Ar–X (X = I and Br) and found good to excellent yields (63–92%, Table 2, **9a–m**). The formation of selectively C2-arylated products (**9a–d**, **9f**, and **9l–m**) occurs during thiophene C–H arylation under the current catalytic conditions with the exception of **9e**. However, C–H arylation of pyridine results in the formation of both C2- and C3-arylated products (**9g–i**) and can be distinguished easily by column chromatography. The regioisomers of bi-heteroaryls have great applications in coordination compounds as well. The formation of several regioisomers was also observed for the C–H arylation reaction of toluene and *m*-xylene (**8a–f**, Table 2) in the current methodology. Efforts were also made towards biarylation using aryl bisiodide; unfortunately, only monoarylation was observed (**8k–l**, Table 2). Next, as biaryl systems have great applications in pharmaceuticals, agrochemicals, and functional materials, a gram-scale synthesis of **7a** was also carried out, resulting in 78% yield (Scheme S2, ESI†).

C–H arylation under blue light irradiation

When we added benzylamine, **1**, to the base KO^tBu in benzene or THF, we saw that the surface of KO^tBu changes from white to red at room temperature (RT). This indicates that the mono-deprotonation of benzylamine to **1a** takes place at the surface of KO^tBu. To support this color change due to the deprotonation of benzylamine, we have performed DFT calculations of **1** and **1a** (Fig. 2). The calculated band gaps between the HOMO and the LUMO in **1** and **1a** indicate that the excitation wavelengths could be 205 and 589 nm, respectively (Fig. 2). The 205 nm wavelength falls in the UV region, whereas 589 nm

Table 2 1-Promoted C–H arylation of arenes and heteroarenes using aryl or heteroaryl halides under heating conditions^{a,b,c}



^a Reaction conditions: arene (1.5 mL), aryl halide (1 mmol), promoter **1** (0.3 mmol), KO^tBu (3 mmol), and 18-crown-6 (1 mmol) for 36 h at 90 °C under inert conditions. ^b Isolated yield. ^c Mixture of isomers (*o*, *m*, *p*, etc.).

falls in the blue light region. Therefore, **1** will be a colorless species as expected; however, the color of **1a** would be red if it absorbs 589 nm light. The predicted red color of **1a** matches exactly with the experimentally observed color of **1a**. As **1a** absorbs blue light, an electron will jump from the HOMO to a higher energy level LUMO (excited state, **1a***); therefore, **1a*** will have higher reducing power than native **1a** and may dissociate the aryl halide (Ar–X, X = I and Br) through a SET

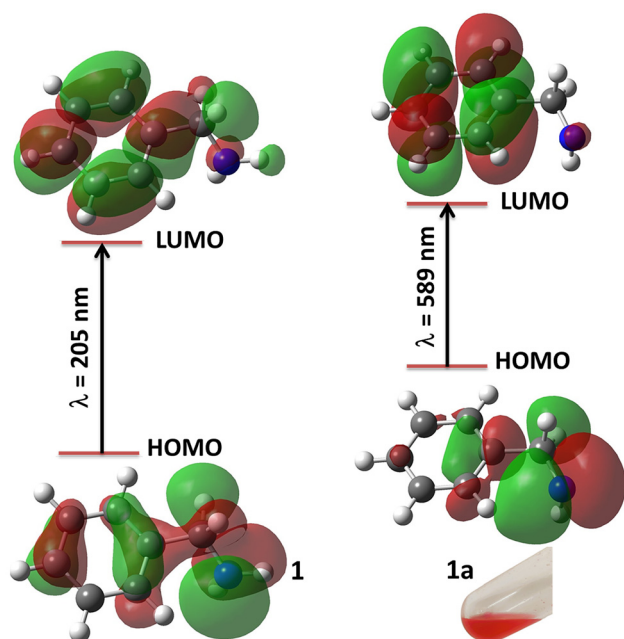


Fig. 2 DFT calculated HOMO and LUMO of **1** (left) and **1a** (right).

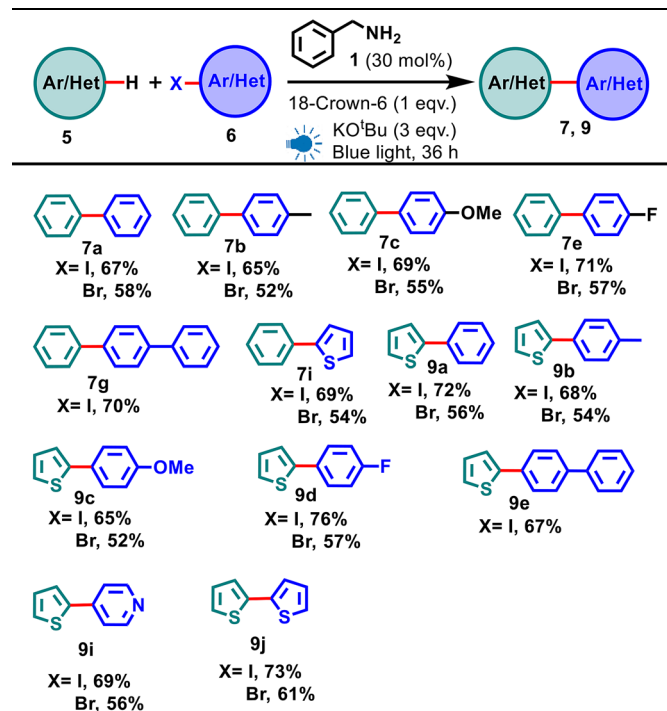
mediated process. In the current C–H arylation reaction (Table 1, entry 1), instead of heat, we have used blue light as an external stimulator and found sufficient yields of the desired arylated product **7a**. Next, this blue light induced C–H arylation of different arenes and heteroarenes using **1** was further tested and to our delight, in many cases the corresponding C–H arylated products were isolated in fair yields (52–76%, Table 3).

Mechanism

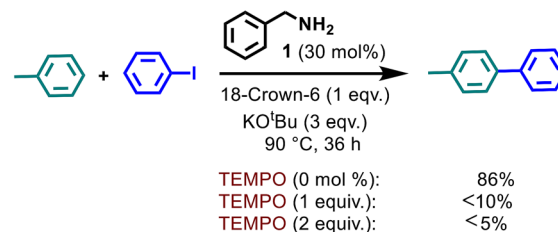
The earlier report indicated that the organocatalytic coupling reactions of aryl halides with arenes usually happen through a radical mechanism.^{12–26,31–40} The main radical is an aryl radical that is produced from an aryl halide through a single electron transfer (SET) process. To confirm this hypothesis, the model reaction (Table 1, entry 1) was conducted in the presence of a radical scavenger, TEMPO, and it was found that it has a great influence on the rate of the reaction (Scheme 2), and the yield of the product indicates the involvement of radical species.⁴¹ The analysis of the TEMPO trapped radical (Scheme S3†) confirms the involvement of an aryl radical. In order to understand the rate determining step, a competition experiment was carried out between C₆H₆ and C₆D₆ (Scheme 3). The kinetic isotope effect (KIE) value for this reaction is almost close to 1 (Fig. S5†), which indicates that the C–H bond cleavage of the arene is not the rate determining step.

Based on all the above information, a SET-mediated plausible mechanistic path has been proposed for the C–H arylation of the model arene substrate benzene using aryl halides (Scheme 4). At first, 18-crown-6 increases KO^tBu's basicity by encapsulating the K⁺ ion in it. The increased basicity of KO^tBu deprotonates the benzylamine, forming species **1a**. The **1a** has

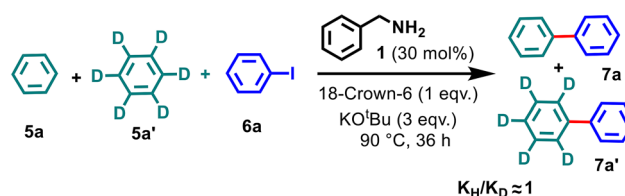
Table 3 1-promoted C–H arylation of arenes and heteroarenes using aryl or heteroaryl halides under irradiation with light^{a,b}



^a Reaction conditions: arene (1.5 mL), aryl halide (1 mmol), promoter **1** (0.3 mmol), KO^tBu (3 mmol), and 18-crown-6 (1 mmol) for 36 h under an inert atmosphere and blue light irradiation. ^b Isolated yield.

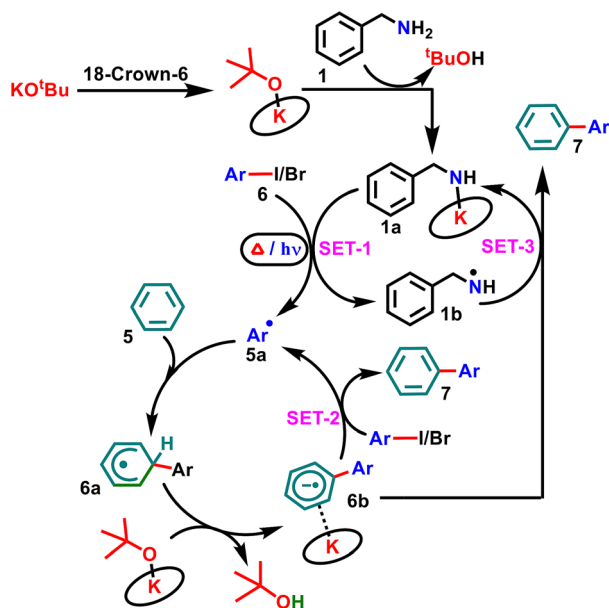


Scheme 2 1-Promoted C–H arylation of toluene in the presence of TEMPO.



Scheme 3 Kinetic isotope effect experiment.

strong reducing power compared to the corresponding 18-crown-6 coordinated KO^tBu and donates an electron (SET-1) to the aryl halide (**6**) under excitation with heat or light. SET-1 leads to C–halogen bond cleavage, resulting in the formation of the key radical species Ar[•]. The formation of Ar[•] has been



Scheme 4 Proposed mechanistic cycle for **1** catalyzed C–H arylation.

confirmed by trapping it with TEMPO, followed by characterization using NMR and mass spectrometry (Fig. S2–4†). Next, in the reaction of Ar[•] with the arene π -system, an arylated radical, **6a**, is formed, and following this, the 18-crown-6 coordinated activated KO^tBu deprotonates a C–H proton from **6a**, resulting in the biaryl radical anion species **6b**. An electron transfer from **6b** to the aryl halide (SET-2) or to radical species **1b** (SET-3) leads to the regeneration of the key radical Ar[•] or the active catalyst **1a**, and the desired biaryl product **7**.

Conclusions

In the present work, we have established a transition-metal-free, cost-effective synthetic method for the C–H arylation of arenes and heteroarenes using commercially available benzylamine. A series of C–H arylation reactions using benzylamine as a promoter were carried out by combining unactivated arenes or heteroarenes with aryl or heteroaryl iodides/bromides under external stimuli, such as heating or light irradiation, which in turn enabled the formation of several aryl–aryl, aryl–heteroaryl and heteroaryl–heteroaryl structural motifs. We believe that easy access to biaryl products would help to elucidate them in various research fields, including organic synthesis, medicinal chemistry, and materials sciences.

Experimental section

Materials and methods

All the reagents and solvents were commercially available and were used as received. When necessary, we dried the solvents and arenes or heteroarenes using standard protocols and

stored them under a nitrogen atmosphere before use. All the reactions were carried out under an inert atmosphere. TLC was performed to monitor the reactions using Merck silica gel 60 F-254 plates, and the detection of compounds was done under UV light. Flash column chromatography (FCC) was performed using a Merck silica gel 100–200 mesh to purify the products. The solvents were removed using a rotary evaporator under reduced pressure. ¹H and ¹³C{¹H} NMR spectra were recorded on an Ultrashield Bruker Avance 400 MHz spectrometer equipped with a 5 mm probe. All resonances are reported relative to TMS. Spectra were calibrated relative to the solvent's residual proton and carbon chemical shifts: DMSO-d₆ ($\delta = 2.50$ for ¹H NMR and $\delta = 39.52$ for ¹³C NMR). Coupling constants (*J*) are reported in Hz. All the gas chromatography-mass spectrometry (GC-MS) analyses were carried out on an AGILENT 6890 spectrometer.

General procedure for **1** promoted C–H arylation of arenes and heteroarenes under heating conditions

In a pressure tube, KO^tBu (336 mg, 3 mmol, 3.0 equiv.), promoter **1** (32 mg, 30 mol%) and arene or heteroarene (1.5 mL) were added under inert conditions. An instant colour change from colorless to red was observed. After that, aryl/heteroaryl halide (1 mmol) and 18-crown-6 (264 mg, 1 mmol) were added, and then the resulting mixture was stirred for 36 h at 90 °C under an inert atmosphere. Next, the reaction mixture was cooled to room temperature and quenched with water (4 mL). After quenching, the arylated products were extracted with 30 mL (3 × 10 mL) of ethyl acetate and the combined organic part was dried over anhydrous sodium sulfate. The organic part was evaporated under reduced pressure leading to a crude arylated product that was further purified by column chromatography on silica gel using hexane as the eluent to get the pure desired arylated product.

General procedure for **1** promoted C–H arylation of arenes and heteroarenes under light irradiation

The **1** promoted C–H arylation of arenes and heteroarenes using light as an external stimulator was performed by following an identical reaction scale, procedure and reaction time mentioned in the above general procedure. This method employed two 7 W blue lamps as a light source, rather than heating at 90 °C. All the products obtained in this process were purified by the above-mentioned technique.

Author contributions

Souvik Chatterjee: conceptualization, formal analysis, methodology, and investigation; Sabnam Azmi: conceptualization, formal analysis, methodology, and investigation; Nilaj Bandopadhyay: formal analysis and investigation; Krishnendu Paramanik: formal analysis and investigation; Gayetri Sarkar: formal analysis, investigation, and visualization; Bhaskar Biswas: conceptualization, writing – reviewing and editing, and supervision and Hari Sankar Das: conceptualization, writing – reviewing and editing, and supervision.

Data availability

Data will be available on request.

Experimental information such as *etc.* are given in ESI.†

Conflicts of interest

There are no conflicts to declare.

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