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ORIGINAL PAPER

Room-temperature copper-catalyzed electrophilic amination of arylcadmium iodides with ketoximes

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Abstract
We started our study by preparation two ketoximes. Later, there were studies to reveal these ketoximes' effects in the electrophilic amination reaction with organocadmium reagents. Primarily, it was observed that arylcadmium iodides could not be reacted with ketoximes at room temperature in the absence of a catalyst. CuCN was a suitable catalyst for this electrophilic amination reaction of arylcadmium iodides and allowed the preparation of functionalized aniline derivatives in good yields under mild reaction conditions. We obtained the results indicated that the yield of primary arylamines was strongly dependent on the steric and electronic effects of organocadmium reagent and amination agent. In the case of both amination reagents, meta-substituted arylamines were obtained in higher yields than para-substituted arylamines. We observed that acetone *O*-(4-chlorophenylsulfonyl)oxime, **1**, as an aminating agent, was more successful than acetone *O*-(2-Naphthylsulfonyl)oxime, **2**, in the synthesis of functionalized arylamines by electrophilic amination of corresponding aryl cadmium iodides. In this method, there is no cadmium release to the environment.

Keywords Electrophilic amination · Organocadmium reagents · Amines · Ketoximes · Catalysis · Organometallic

Introduction

Since amines are used as starting materials both in organic synthesis and in the preparation of important compounds and materials in the industry [1–8]. New studies are constantly being carried out to develop more applicable and highly efficient methods for their synthesis [9].

Therefore, the development of procedures allowing the synthesis of arylamines under mild reaction conditions and in high yields is of significant importance. Although nearly all the methods in the literature let obtaining primary arylamines in good yields, most of them have significant drawbacks such as the requirement of a high temperature, long reaction condition, high pressure, expensive or specific catalysts, and ligands[10–13].

Morondi and co-workers described an iron-catalyzed aromatic C–H amination procedure for the preparation of primary anilines [14]. They managed to obtain various functionalized primary anilines, using MsO-NH_3^+ $^-$OTf (1.5 equiv.) as an aminating agent in the presence of 5 mol% $FeSO_4$ as a catalyst in MeCN/H_2O at room temperature after a 16 h reaction. Amination of electron-poor arenes by this method was performed in HFIP (hexafluoroisopropanol), in the presence of 1 mol% $FeSO_4$ at 60 °C [15]. Jiao and co-workers used 4-NO_2-PhCOONH_3^+ $^-$OTf as an aryl C−H amination agent [16]. The reaction was carried out successfully in the presence of $FeBr_2$ as catalyst (5 mol%) and $AgNTf_2$ as an additive (10 mol%). Cu(OTf)$_2$/1,10-phenanthroline-catalyzed amination of arenes in the presence of CsOH.H_2O in HFIP using hydroxylamine-O-sulfonic acid (H_2N-OSO_3H) as an electrophilic nitrogen source resulted in good yields of primary arylamines [17].

To date, many procedures have been published for the electrophilic amination of organomagnesium, -zinc and -copper reagents with various electrophilic amination agents [18]. Recently, Knochel's group showed that cobalt-catalyzed electrophilic amination of arylzinc pivalates with several anthranil derivatives allowed the formation of corresponding amino-aldehydes and amino-ketones in good to high yields at room temperature after 16 h [19]. Kürti's group used N–H oxaziridines (1.2 equiv.) for the transfer of primary amino group (–NH_2) to arylmagnesium bromides. They conducted this electrophilic amination reaction in

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toluene/THF mixture at 45 or 78 °C. The reactions were completed in 16 h and arylamines were obtained in 16–89% yields [20]. They used N–H oxaziridines for the electrophilic amination of functionalized arylcuprates which prepared by transmetallation of arylmagnesium, -zinc or -lithium reagents with CuCN.2LiCl or CuCl.2LiCl at 78 °C [21].

Organocadmium reagents have been mostly used for the synthesis of ketones from active substrates such as acid halides or acid anhydrides. In addition, they have been employed as alkylation reagents in the regioselective alkylation of quinines [22, 23]. But they have a high functional group tolerance and if can be increased their reactivity they can be used for the synthesis of functionalized organic compounds.

Up to now, the Daşkapan group was developed successful procedures for electrophilic amination of organomagnesium, organozinc, and organocopper reagents [24–29]. The studies in the literature about electrophilic amination of organocadmium reagents were published by our group [30, 34].

Experimental section

General

All reactions involving arylcadmium iodide reagents were performed in flame-dried glassware with standard syringe/cannula techniques under an atmosphere of dry, oxygen-free argon [31]. Melting points were determined on a Gallencamp capillary melting point. NMR spectra were recorded on a Bruker DRX-400 high-performance digital FT-NMR spectrometer. All chemical shifts were given in ppm downfield from tetramethylsilane (TMS).

THF was freshly distilled from the solution of sodium–benzophenone under dry argon and kept over molecular sieves (4 Å 4–8 mesh) and under argon atmosphere. Copper (I) cyanide was purified prior to use and kept under a dry argon atmosphere [32].

Organomagnesium bromides were prepared in THF by conventional standard methods and their concentrations were determined by the method of Watson and Eastham [33]. Aryl bromides (Sigma-Aldrich) were in high purities and used without any further purification.

General procedure for CuCN-Catalyzed electrophilic amination of arylcadmium iodides with ketoximes

A solution of CdI_2 (0.7324 g, 2 mmol) in anhydrous THF (5 mL) was cooled to 10 °C under argon atmosphere and 2 mmol of arylmagnesium bromide in THF was added dropwise via syringe. The reaction mixture was stirred for an additional 10 min, the cooling bath was removed, and the resulting white suspension was allowed to warm to room temperature. To this mixture, CuCN (0.0089 g, 0.01 mmol), and a solution of **1** or **2** (0.247 g or 0.263 g, 1 mmol) in dry THF (2 mL) were added. The reaction mixture was stirred at room temperature for 30 min and then worked up by the addition of conc. HCl with stirring at room temperature for 24 h. The aqueous phase was washed with diethyl ether, made basic with conc. NaOH ($Cd(OH)_2$ precipitated as a white solid) and the free amine was extracted with diethyl ether (3 × 50 ml). The organic layer was dried over Na_2SO_4, and the solvent was evaporated and the crude product was converted to its N-benzoyl derivative by reaction with benzoyl chloride in the presence of NaOH. The product was recrystallized from ethanol–water (4:1). Benzamide derivatives were identified from their melting points ^{1}H NMR and ^{13}C NMR analysis.

General procedure for the preparation of acetone *O*-(4-Chlorophenylsulfonyl)oxime (1) and acetone *O*-(2-Naphthylsulfonyl)oxime (2)

Acetone O-(4-Chlorophenylsulfonyl)oxime (**1**) and acetone O-(2-Naphthylsulfonyl)oxime (**2**) were prepared according to the method made by Korkmaz [34]. Acetonoxime (13.70 mmol), DMF (4 mL), and triethylamine (1.9 mL) were placed into 100-mL flask. This flask was immersed into the ice bath and set up on a magnetic stirrer. Then, 4-chlorophenylsulfonyl chloride (13.70 mmol) was attentively added piece by piece keeping stirring in 5–10 min. The resulted reaction mixture stirred for 1 h, keeping in ice bath. Then white solid formed by gently diluting the reaction mixture by adding 50 mL of ice water. This crude product was filtered off under vacua and dried in a desiccator. The dry white solid was crystallized from hexane-benzene (5: 1) mixture (1.962 g, 58%, mp 89–92 °C); ^{1}H NMR spectrum ($CDCl_3$, ppm): 7.91–7.89 (d, 2H, Ar–C-H), 7.51–7.49 (d, 2H, Ar–C-H), 1.98 (s, 3H, CH_3), 1.92 (s, 3H, CH_3). ^{13}C NMR spectrum ($CDCl_3$ olacak) δ(ppm): 165.36 (R^1R^2C=N), 140.46, 134.33, 130.22, 129.24 (Ar–C), 21.62, 16.96 (R–C).

Aceton *O*-(2-naphthylsulphonyl)oxime (**2**) was synthesized following the same procedure (2.161 g, 60%, mp 92–95 °C); ^{1}H NMR spectrum ($CDCl_3$, ppm): 8.56 (s, 1H, naphthyl-H), 7.93–7.90 (m, 4H, naphthyl -H), 7.68–7.59 (m, 2H, naphthyl -H), 1.98 (s, 3H, CH_3), 1.89 (s, 3H, CH_3). ^{13}C NMR spectrum ($CDCl_3$, ppm): 165.10 (R^1R^2C=N), 135.35, 132,80, 131.93, 130.70, 129.44, 129.30, 129.13, 127.93, 127.58, 123.32 (naphthyl-C), 21.64, 16.95 (R–C).

Results and discussion

In this manuscript, the results of our works on the reaction of arylcadmium iodides with ketoximes in the presence of CuCN as a catalyst at room temperature are given.

According to our knowledge, these results will be the first data about electrophilic amination of arylcadmium iodides with ketoximes.

We used two novel ketoximes, acetone *O*-(4-chlorophenylsulfonyl)oxime, **1**, and acetone *O*-(2-Naphthylsulfonyl) oxime, **2,** as amination reagents. We prepared and purified these compounds according to the methods described in the literature [35]. Arylcadmium iodides were prepared by transmetallation of the corresponding arylmagnesium bromides with CdI_2 in THF and controlled by Gilman's color test [36]. Amination reaction was performed by cannulation of THF solution of **1** or **2** into organocadmium reagent.

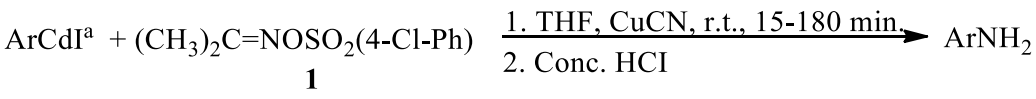
Arylamines as the final products were removed from the reaction mixture as their benzamide derivatives and these known compounds were identified from their melting points, ^{1}H NMR, and ^{13}C NMR analysis [37–42]. Cadmium was removed from the reaction mixture as its white-colored salt ($Cd(OH)_2$) during the reaction mixture's basification of after hydrolysis with conc. HCl. Thereby cadmium was not thrown as waste to the environment.

Since we aimed to develop an easy applicable procedure for the electrophilic amination of arylcadmium iodides with ketoximes, we conducted our works at room temperature.

Firstly, we reacted phenyl cadmium iodide with ketoxime under catalyst-free conditions and we observed that the reaction failed (Table 1, entries 1 and 2). Then we decided to perform the reaction in the presence of a catalyst. For this reason, we used 2.5–10 mol% CuCN as a catalyst and continued the reactions for 30 min. We observed that 5 mol% of CuCN needed for the best arylamine yield. Chancing the reaction time as 15 min or 60 min led to a decrease in the yield (entry 7 and 8).

After determination of optimal reaction conditions using amination reaction of phenylcadmium iodide as a model reaction, we prepared various functionalized arylcadmium reagents and reacted them with **1** under these reaction conditions to show the scope of the method. As is seen in Table 1, when aryl ring has a good electron-donating group, electrophilic amination reaction resulted in high yield (entry 11) but in the presence of strong electron-withdrawing group lower yield of amine was obtained (entry 15). The results obtained from reaction of functionalized arylcadmium reagents showed that arylcadmium reagent gave a selective reaction with **1**. For example, in the presence of an electron-withdrawing substituent, *meta*-substituted aniline was obtained in higher yield compared to *para*-substituted

Table 1 CuCN-catalyzed electrophilic amination of ArCdI with **1**



Entry	Ar	CuCN (%)	Time (min.)	Yield[b] (%)
1	C_6H_5	0	30	–
2	C_6H_5	0	180	–
3	C_6H_5	7.5	30	50
4	C_6H_5	5	30	73
5	C_6H_5	10	30	41
6	C_6H_5	2.5	30	65
7	C_6H_5	5	15	61
8	C_6H_5	5	60	55
9	4-ClC_6H_4	5	30	58
10	3-ClC_6H_4	5	30	70
11	4-$CH_3SC_6H_4$	5	30	73
12	3,5-$Cl_2C_6H_3$	5	30	48
13	4-$MeOC_6H_4$	5	30	59
14	3-$MeOC_6H_4$	5	30	85
15	4-FC_6H_4	5	30	64
16	4-$CH_3C_6H_4$	5	30	60
17	4-Cl-2-MeC_6H_3	5	30	64
18	3,4,5-$(MeO)_3C_6H_2$	5	30	51

[a]ArCdI/**1** = 2

[b]Yield of amines was isolated as their N-benzoyl derivatives, and these known compounds were identified from their melting point, ^{1}H NMR, and ^{13}C NMR analysis [38–42]

aniline (entry 9 and 10). Similar results were obtained from the reaction of the electron-donating group substituted arylcadmium reagents (entry 13 and 14). While two electron-withdrawing group substituted arylcadmium gave the corresponding amine in low yield (entry 12), electrophilic amination reaction of one electron-withdrawing and one electron-donating group substituted arylcadmium reagent with **1** resulted in a good yield (entry 17).

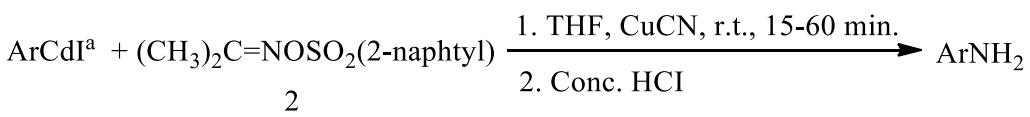
Unfortunately, acetone *O*-(2-Naphthylsulfonyl)oxime, **2**, as an electrophilic nitrogen source, has not been as successful as **1** in the electrophilic amination of aryl cadmium reagents. Besides, in the presence of electron-withdrawing group, there was not observed any meta-para selectivity (Table 2, entries 8 and 9), in the case of electron-donating substituent a low selectivity in favor of meta-position was observed (Entries 12 and 13). According to these results, the use of **1** for the formation of aryl C-N bond employing arylcadmium iodides as the organometallic reagent is more suitable than **2** (Scheme 1).

Scheme 2 shows the proposed mechanism for CuCN-catalyzed electrophilic amination of arylcadmium iodides with ketoximes. A mechanism for the CuCN-catalyzed electrophilic amination of arylcadmium iodides has been proposed using the literature data of organo-copper compounds and copper-catalyzed binding of organozinc reagents, and explanations have been made considering these mechanisms [43]. Arylcadmium iodides react with CuCN catalyst to form catalytic amount of arylcyanocuprate, which is the real catalyst. $ArCuCN^-$, as a nucleophile, attacks on the electrophilic nitrogen of ketoxime to form a Cu-III complex (Scheme 2, **a**, oxidative addition).

The rate-determining step can prefer oxidative addition or the reductive elimination step in the mechanism depending on the reaction conditions, the nucleophile structure, and the electrophile. Furthermore, both the electronic effects and steric effects of these structures are effective in selecting the rate-determining step [44, 45]. In the case of the amination of diarylcadmium containing a functional group, it is not possible to give a precise sequence of activities. Steric hindrance and electronegativity can be evaluated where they are performed under the same reaction conditions. Thus, for the arylcadmium iodide reagents having the same functional group at different positions, the rate-determining step can be estimated as the oxidative addition step or the reductive elimination step thanks to the amine yields obtained.

It seems that this step occurs more easily when **1** is used as an electrophilic amination agent for the aryl C-N bond formation employing arylcadmium iodides as an organometallic

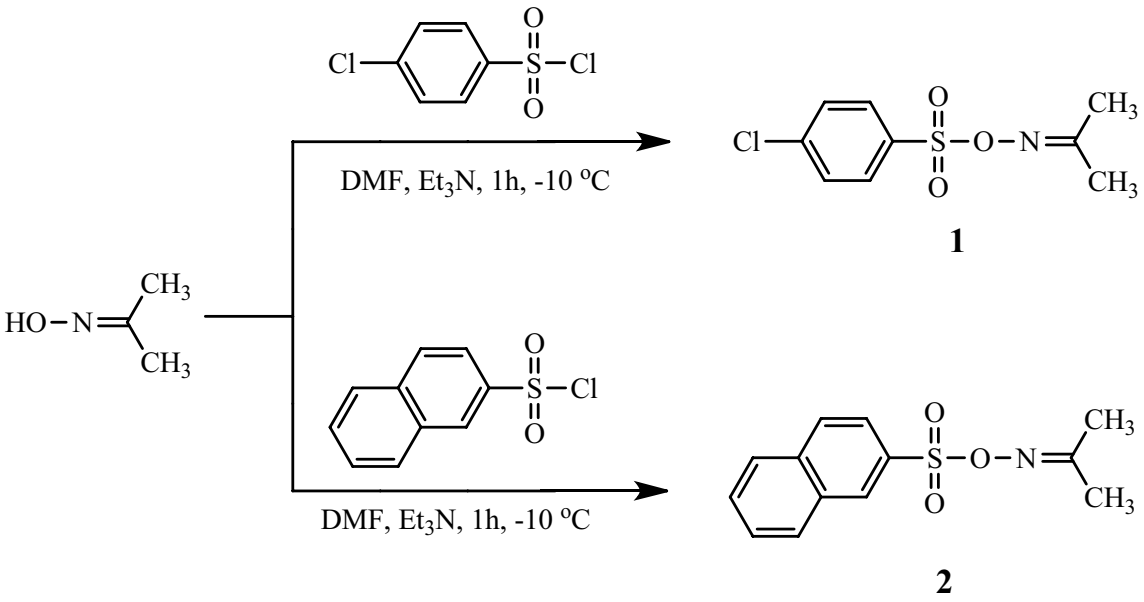
Table 2 CuCN-catalyzed electrophilic amination of ArCdI with **2**



Entry	Ar	CuCN (%)	Time (min.)	Yield[b] (%)
1	C_6H_5	10	30	33
2	C_6H_5	7.5	30	32
3	C_6H_5	5	30	49
4	C_6H_5	2.5	30	61
5	C_6H_5	1.25	30	34
6	C_6H_5	2.5	15	56
7	C_6H_5	2.5	60	42
8	4-ClC_6H_4	2.5	30	52
9	3-ClC_6H_4	2.5	30	56
10	4-$CH_3SC_6H_4$	2.5	30	55
11	3,5-$Cl_2C_6H_3$	2.5	30	52
12	4-$MeOC_6H_4$	2.5	30	39
13	3-$MeOC_6H_4$	2.5	30	50
14	4-FC_6H_4	2.5	30	63
15	4-$CH_3C_6H_4$	2.5	30	52
16	4-Cl-2-MeC_6H_3	2.5	30	53
17	3,4,5-$(MeO)_3C_6H_2$	2.5	30	22

[a]ArCdI/**2** = 2

[b]Yield of amines was isolated as their N-benzoyl derivatives, and these known compounds were identified from their melting point, ^{1}H NMR, and ^{13}C NMR analysis[38–42]



Scheme 1 Preparation of ketoximes

reagent. Because naphthylsulfonyiloxy group is bulkier than 4-chlorophenylsulfonyloxy group, and this leads to the need less energy for the formation of Cu-III complex during the reaction of **1** with arylcadmiums, compared to the formation by reaction of **2**. *meta*-selectivity is interpreted as: the *meta*-structure is more rounded than *para*-structure and thus facilitates reductive elimination (Scheme 2, **b**) by creating a steric effect in the Cu-III complex. The lack of a significant selectivity in the amination reaction of **2** is due to the fact that the bulky naphthylsulfonyloxy group facilitates reductive elimination (Scheme 2, **b**).

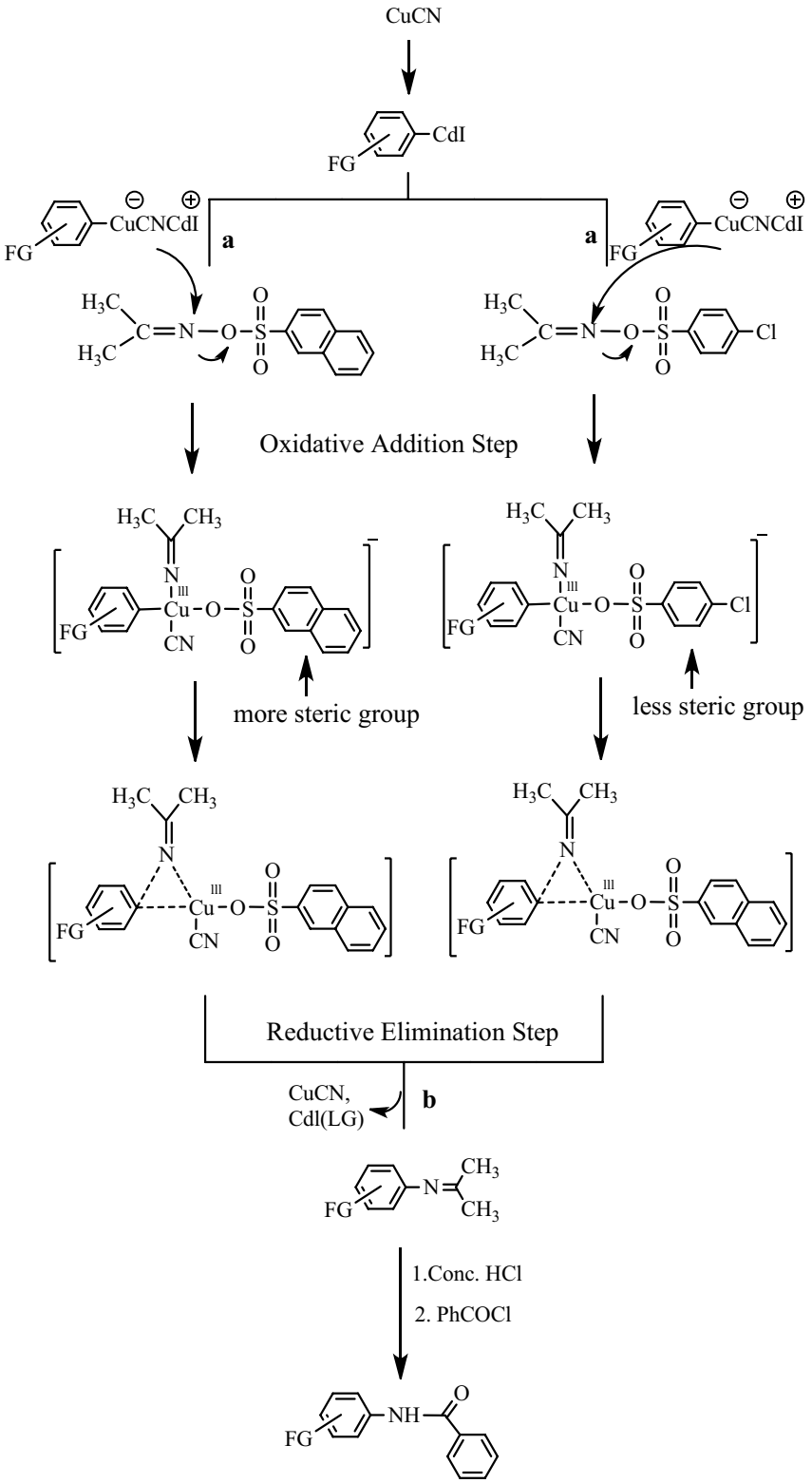
One of the aims of our study was to determine the efficacy of acetone O-(4-Chlorophenylsulfonyl)oxime and acetone O-(2-Naphthylsulfonyl)oxime molecules with arylcadmium iodide reagents with the electrophilic amination reactions. There are naphthyl and 4-chlorophenylsülphonate structures as leaving groups in these molecular structures. The naphthyl group is a larger group than the 4-chlorophenyl structure. It means that the naphthyl group has a more significant steric effect on the reaction mechanism. Owing to the steric effect of the naphthyl group, it thought that the complex formed of the oxidative addition step became unstable. Thus, it was predicted that unstable complex formation in the oxidative addition step reduced the arylamine yield. It has been observed that the experimental yields agree with the theoretical concepts considered. On the other hand, the effectiveness of the leaving group 4-chlorophenylsulfonate was considered. Since this structure is linear, it was thought that it had a less steric effect in the oxidative addition step compared to the naphthyl group. So, it was thought that higher arylamine yields would be obtained when 4-chlorophenylsulfonate was used as leaving group. It was determined that the experimental data were in accordance with the theoretical data explained.

The reaction conditions of the previously performed with acetone O- (Mesitylenesulfonyl)oxime were determined as 5% CuCN catalysis at 60 min [30]. In the study of the acetone O-(4-Chlorophenylsulfonyl)oxime, the time was reduced to half (30 min) using the same catalyst ratio. Moreover, arylamine yields were observed almost the same in some reagents (Tables 1, Experiments 11 and 15). Some of them, the yields were obtained with higher than before the studied (Table 1, Experiments 10 and 14). Furthermore, di(4-Chloro-2-methylphenyl)cadmium and di(3,4,5-tri(methoxy)phenyl) cadmium reagents were tested in this study (Table 1, Experiments 17 and 18).

Conclusion

In this work, the ketoximes (**1** and **2**) were prepared and tested in the CuCN-catalyzed electrophilic amination reaction of aryl cadmium iodides, as the aminating agent. The obtained results are the first results about electrophilic amination of arylcadmium iodides with ketoximes. While **2** gave functionalized anilines in moderate yields, **1** was found to be a successful aminating agent for the synthesis of arylamines by electrophilic amination of arylcadmium iodides, where cadmium is not released into the environment. Especially, when looking at the results obtained from this study, it was determined that the structure of each amination reagent affects the reductive elimination and oxidative addition

Scheme 2 Proposed mechanism for the CuCN-catalyzed electrophilic amination of arylcadmium iodides with ketoximes



FG : 4-ClC_6H_4, 3-ClC_6H_4, 4-$MeOC_6H_4$, 3-$MeOC_6H_4$, 4-MeC_6H_4, 2-Cl-4-MeC_6H_3, 4-$MeSC_6H_4$, 3,5-$(Cl)_2C_6H_3$, 4-FC_6H_4, 3,4,5-$(MeO)_3C_6H_2$.

LG : -OSO_2-2-$C_{10}H_7$, -OSO_2(4-ClC_6H_4)

steps. Thus, it has been determined that the structure of the amination reagent has an effect on the yield using different functional arylcadmium reagents. As a result, it has been observed that it can be achieved by changing the structure of the amination reagent to synthesize the desired functional arylamine in the best yield.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s13738-021-02254-4.

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Declarations

Conflıct of ınterest The author declares no conflict of interest, financial or otherwise.

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