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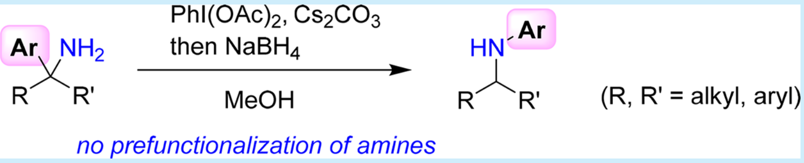
Oxidative Rearrangement of Primary Amines Using $PhI(OAc)_2$ and Cs_2CO_3

Wataru Yamakoshi, Mitsuhiro Arisawa, and Kenichi Murai*

Graduate School of Pharmaceutical Sciences, Osaka University, 1-6 Yamada-oka, Suita, Osaka 565-0871, Japan

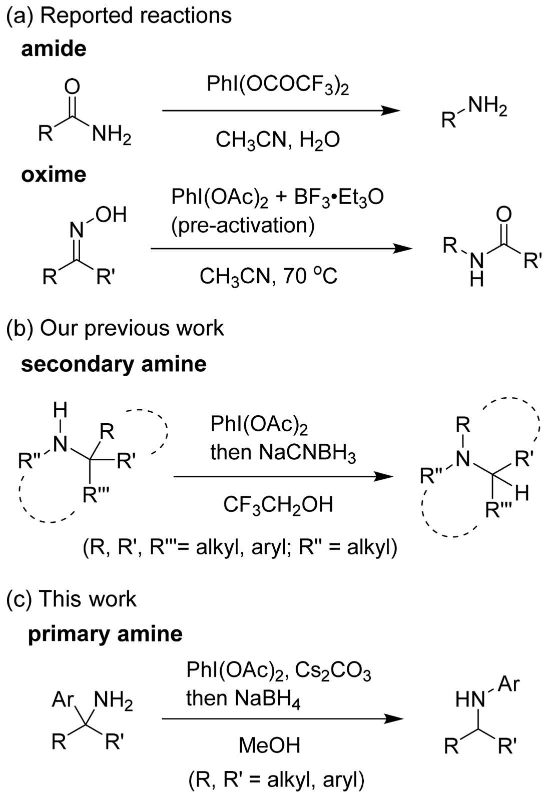
S Supporting Information



ABSTRACT: An oxidative rearrangement of primary amines mediated by a hypervalent iodine(III) reagent is herein reported. The combination of $PhI(OAc)_2$ and Cs_2CO_3 proves highly efficient at inducing the direct 1,2-C to N migration of primary amines, which can be applied to the preparation of both acyclic and cyclic amines. A mechanistic study shows that the rearrangement proceeds via a concerted mechanism.

Among the transformations of nitrogen-containing compounds, processes involving 1,2-C to N migration are useful tools for organic synthesis because they fundamentally alter the molecular skeletons.[1] For example, the Hofmann, Schmidt, Beckmann, Curtius, and Stieglitz rearrangements are all well-known reactions whose utility has been proved by their application to the synthesis of various molecules.[2] Considering the usefulness of these transformations, the development of new methods for 1,2-C to N migration is still an important source of new options in organic synthesis. Recently, hypervalent iodine(III) reagents[3] have proven useful for various oxidative rearrangements because of their electrophilicity and ability to act as leaving groups as well as their environmentally friendly behavior.[4−6] In regard to 1,2-C to N migration, the reaction of amides with hypervalent iodine(III) reagents, that is, Hofmann rearrangement, is established as an important synthetic tool for converting amides into the corresponding amines and their derivatives (Scheme 1a).[7] Catalytic versions of hypervalent iodine(III) reagent-induced Hofmann rearrangements have also been developed in the past decade.[8] However, until recently the applications of these reagents in 1,2-C to N migration have been limited to amides, and the analogous rearrangements employing other nitrogen-containing functional groups have not been well explored. Recently, Beckmann rearrangement of a ketoxime using $PhI(OAc)_2$ combined with $BF_3{\cdot}Et_2O$ was reported by Maegawa and Miki.[9] Alternatively, we have recently reported the 1,2-C to N migration of secondary amines (Stieglitz rearrangement) (Scheme 1b).[10] By using $PhI(OAc)_2$ in CF_3CH_2OH, the oxidative rearrangements directly proceeded with a range of amine compounds. Compared with the classic Stieglitz rearrangement, which employs azides, hydroxylamine derivatives, and *N*-chloroamines,[2,11] this method could provide a more straightforward synthetic strategy in which amines can be used directly as substrates.

Scheme 1. Rearrangement Reaction with Hypervalent Iodine Reagent



During our ongoing study of the hypervalent iodine(III) reagent-mediated oxidative rearrangement of amines, we

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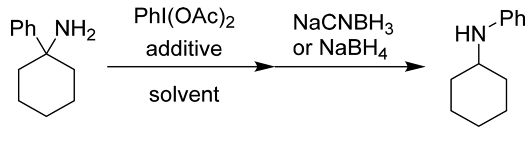
noticed that secondary and primary amines showed a quite different reactivity: Although tritylamine was applicable in our previously reported reaction, the conditions used for secondary amines were not very effective for rearrangement of other primary amines (Table 1, entry 1). Taking note of this, we then re-investigated the reaction of primary amines.[12] Herein, we report our recent success in finding that the combined use of $PhI(OAc)_2$ with Cs_2CO_3 efficiently induces the rearrangement of primary amines (Scheme 1c). Because of the easy handling and environmentally benign nature of hypervalent iodine reagents, the method is more practical than the previously reported 4-nitrobenzenesulfonyl peroxide- or $Pb(OAc)_4$-mediated Stieglitz rearrangements, which also use amines directly.[13]

Table 1. Study of Reaction Conditions[a]



1a **2a**

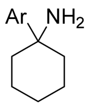
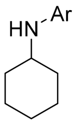
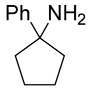
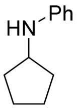
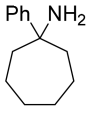
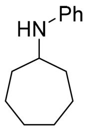
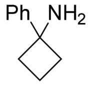
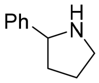
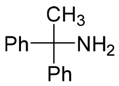
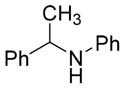
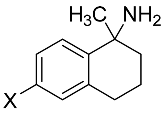
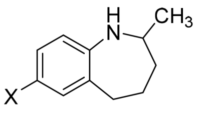
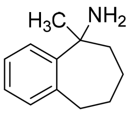
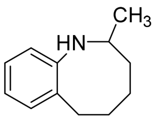
entry	solvent	additive (equiv)	yield (%)[b]
1[c]	CF_3CH_2OH	none	33
2[c]	CF_3CH_2OH	K_2CO_3 (5.2)	41
3[d]	MeOH	K_2CO_3 (5.2)	69
4[d]	EtOH	K_2CO_3 (5.2)	61
5[d]	CH_3CN	K_2CO_3 (5.2)	35
6[d]	CH_2Cl_2	K_2CO_3 (5.2)	N.D.[e]
7[d]	MeOH	Na_2CO_3 (5.2)	74
8[d]	MeOH	Cs_2CO_3 (5.2)	82
9[d]	MeOH	pyridine (5.2)	trace
10[d]	MeOH	Et_3N (5.2)	20
11[d]	MeOH	Cs_2CO_3 (3.9)	79
12[d]	MeOH	Cs_2CO_3 (2.6)	90
13[d]	MeOH	Cs_2CO_3 (1.3)	65

[a] Reaction conditions: $PhI(OAc)_2$ (1.3 equiv) and additive in solvent (0.05 M) at 0 °C to rt then $NaCNBH_3$ or $NaBH_4$ (5.0 equiv) at 0 °C to rt. [b] Isolated yield. [c] Reductant: $NaCNBH_3$ [d] Reductant: $NaBH_4$. [e] Not detected on TLC.

We began our study on the rearrangement of primary amines using 1-phenylcyclohexylamine (**1a**) as a model substrate (Table 1). As described above, the use of $PhI(OAc)_2$ in CF_3CH_2OH was not effective for **1a** and only afforded 33% yield of corresponding amine **2a** (entry 1). Varying the reaction conditions by using different solvents, such as MeOH, CH_3CN, CH_2Cl_2, and DMF as well as hypervalent iodine reagents such as $4\text{-}FC_6H_4(OAc)_2$, $4\text{-}NO_2C_6H_4(OAc)_2$, $PhI(OCOCF_3)_2$, Koser's reagents,[14] and 1-acetoxy-1,2-benziodoxol-3-(1*H*)-one, failed to improve the efficiency of the reaction (for details see Supporting Information (SI)). However, we found that the yield was slightly improved by adding K_2CO_3 (entry 2). Moreover, the use of K_2CO_3 in MeOH greatly improved the yield (69%, entry 3). It should be noted that in this work, the products were typically obtained as the amine form via a one-pot reduction of the imine generated by rearrangement reaction with $NaCNBH_3$ or $NaBH_4$ (for details see footnote of Table 1). Encouraged by these results, we further investigated the effects of base and solvent used for the reaction. A survey of several solvents revealed that, when adding a base, MeOH was a better solvent choice than CF_3CH_2OH, EtOH, CH_3CN, and CH_2Cl_2 (entries 2–6). Furthermore, a survey of various base additives revealed that the use of Cs_2CO_3 gave a superior result (entry 8). The efficiency observed with Cs_2CO_3 can probably be attributed to its complete solubility in MeOH, in contrast to the partial solubility of K_2CO_3 and Na_2CO_3. The use of organic bases such as pyridine or triethylamine decreased the yield (entries 9,10). Regarding the amount of Cs_2CO_3, 2.6 equiv relative to the substrate was needed to obtain good results (entry 12). Therefore, the optimal reaction conditions found for the rearrangement of primary amines involved treatment with $PhI(OAc)_2$ (1.3 equiv) in MeOH in the presence of Cs_2CO_3 (2.6 equiv).[15]

With the optimized conditions established, we proceeded to investigate the scope of the reaction (Table 2). As shown in

Table 2. Generality of Rearrangement Reaction[a]

entry	substrate	product[b]
		
1	**1a** (Ar = Ph)	**2a** (Ar = Ph) 90%
2	**1b** (Ar = 4-MeC_6H_4)	**2b** (Ar = 4-MeC_6H_4) 69%
3	**1c** (Ar = 4-ClC_6H_4)	**2c** (Ar = 4-ClC_6H_4) 71%
4	**1d** (Ar = 4-$MeOC_6H_4$)	**2d** (Ar = 4-$MeOC_6H_4$) 70%
5	 **1e**	 **2e** 79%
6	 **1f**	 **2f** 89% (84%)[c]
7	 **1g**	 **2g** 66%
8	 **1h**	 **2h** 92%
		
9	**1i** (X = H)	**2i** (X = H): 82%
10	**1j** (X = Cl)	**2j** (X = Cl): 77%
11	**1k** (X = MeO)	**2k** (X = MeO): 87%
12[d]	**1l** (X = CO_2Me)	**2l** (X = CO_2Me): 48%
13[d]	**1m** (X = CF_3)	**2m** (X = CF_3): 40%
14	 **1n**	 **2n** 95%

[a] Reaction conditions: **1** (0.15–0.2 mmol), $PhI(OAc)_2$ (1.3 equiv), and Cs_2CO_3 (2.6 equiv) in MeOH (0.05 M) at 0 °C to rt (12 h) then $NaBH_4$ (5.0 equiv) at 0 °C to rt (1–7 h, see SI). [b] Isolated yield. [c] 1.0 mmol of **1f** was used. [d] Reaction time for rearrangement: 24 h.

Table 2, several electronically different 1-arylcyclohexylamines underwent the rearrangement efficiently, including those bearing 4-MeC_6H_4, 4-ClC_6H_4, and 4-$MeOC_6H_4$ (entries 2–4).[16] It should be noted that despite the risk of electron-rich aromatic groups such as methoxybenzene being oxidized by hypervalent iodine reagents, compound **1d** successfully proceeded in the reaction. Cyclopentylamine **1e** and cycloheptylamine **1f** also gave phenyl group-migrated products **2e** and **2f** ,respectively, while cyclobutylamine **1g** gave ring-expanded pyrrolidine **2g**, probably because of the ring strain of the cyclobutane ring (entries 5–7). Acyclic compounds were tolerant, and compound **2h** was obtained selectively from 1,1-diphenylethylamine (**1h**) (entry 8).[17] Finally, we applied the method to the synthesis of cyclic amines via a ring expansion reaction. By using tetraline **1i** as a substrate, benzoazepine **2i** was obtained in good yield (entry 9). The method was also well applicable to the substrates **1j** and **1k** with a chloro or a methoxy group on the benzene ring (entries 10 and 11). The substrates **1l** and **1m** having an electron-withdrawing group such as an ester or a trifluoromethyl group also gave the corresponding benzoazepine **2l** and **2m** (entries 12 and 13).[18] Furthermore, eight-membered ring benzoazocine **2n** was obtained in high yield from benzosuberan **1n** (entry 14).

The reaction mechanism was then studied. We first conducted the reaction of the secondary amine **3** to investigate the reactivity difference between primary and secondary amines (Scheme 2, (i). The secondary amine **3** was found not to undergo rearrangement under the standard conditions using $PhI(OAc)_2$ and Cs_2CO_3 in MeOH, suggesting that a primary amine structure was essential for the rearrangement. The reactions were also conducted in the presence of radical scavengers, because in our previous report on the oxidative rearrangement of secondary amines with $PhI(OAc)_2$ and CF_3CH_2OH, we observed that the reaction proceeded via a radical mechanism. We therefore explored the effect of adding 9,10-dihydroanthracene and galvinoxyl to the reaction of **1a**. In each case, the rearrangement proceeded without significant decrease in efficiency, and the desired products were obtained in good yields (Scheme 2, (ii)). From these results, a radical mechanism could be excluded. We also studied the reaction of compound **1a** treated with only Cs_2CO_3 in MeOH, because solely base-induced rearrangement of tritylamines to imines has been reported by Theodorou.[19] However, in our case, the rearrangement did not proceed at all. Therefore, the possibility that the rearrangement could have been mediated solely by Cs_2CO_3 was also excluded. Lastly, a series of reactions using monosubstituted tritylamines **4a**–**e** were conducted to investigate the migratory aptitude (M.A.) of the rearrangement (Table 3).[20] We reacted **4** under the rearrangement conditions, producing a crude mixture of **5** and **6**, then subjected this mixture to acid hydrolysis and analyzed the ratio of benzophenone **7** and substituted benzophenone **8** in the product. This approach was chosen as the product ratio was easier to analyze than that of the rearrangement itself. The migratory aptitudes of the substituted phenyl groups (labeled Ar in Table 3), relative to a migratory aptitude of 1 for the phenyl group, were calculated by the following formula: M.A. = 2(% benzophenone (**7**))/% substituted benzophenone **8**. As shown in Table 3, the migratory aptitudes obtained were as follows: 4-$MeOC_6H_4$, >100; 4-MeC_6H_4, 8.2; 4-FC_6H_4, 1.36; 4-ClC_6H_4, 0.72, showing a strong effect of the electron-rich/-deficient nature of the substituted phenyl groups. This tendency is similar to the common trend observed in the related rearrangements such as Pinacol rearrangement.[1] These results argued against either a free radical mechanism or a nitrene mechanism.[13b]

Scheme 2. Control Experiments

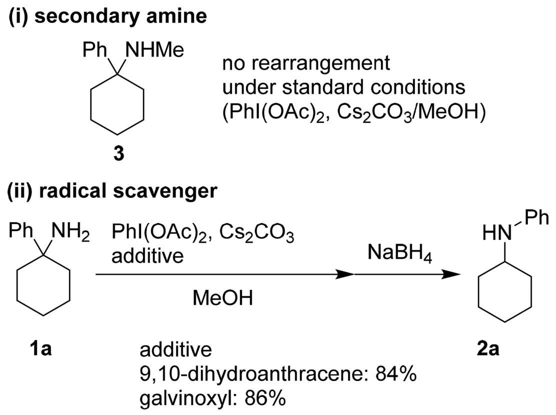
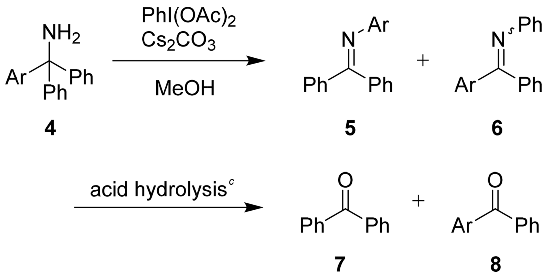


Table 3. Study on Migratory Aptitude of Substituted Phenyl Groups Using Monosubstituted Tritylamines[a]



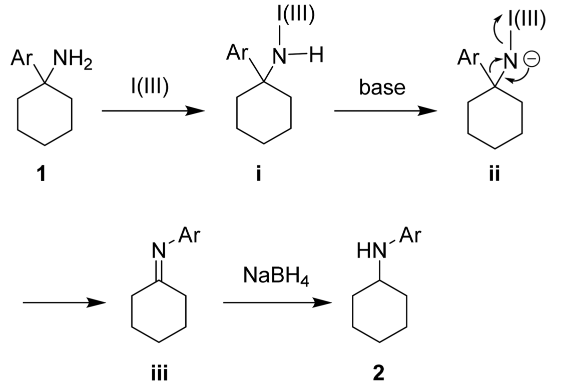
entry	Ar	combined yield (%) (**7** + **8**)	M.A.[b]
1	Ph (**4a**)	82	1
2	4-$MeOC_6H_4$ (**4b**)	80	>100
3	4-MeC_6H_4 (**4c**)	91	8.2
4	4-FC_6H_4 (**4d**)	96	1.36
5	4-ClC_6H_4 (**4e**)	92	0.72

[a] Reaction conditions: reagent (1.3 equiv) in solvent (0.05 M) at 0 °C to rt then $NaCNBH_3$ (5.0 equiv) at 0 °C to rt. [b] M.A. (migratory aptitude) = 2(% benzophenone (**7**))/% substituted benzophenone **8** [c] Conditions for hydrolysis: HCl aq., DMSO.

On the basis of these observations, a possible pathway for the rearrangement is proposed in Scheme 3. Initially, the reaction of amine **1** with $PhI(OAc)_2$ gave intermediate **i**. Under basic reaction conditions using Cs_2CO_3, the N–H of **i** was deprotonated to form anion **ii**. Since the generation of imino iodanes (PhI = NR) from sulfonamides under basic conditions is known,[21] an analogous imino iodane might be possible as an alternative intermediate for the current structure of intermediate **ii**. The intermediate **ii** then underwent rearrangement in a concerted manner to give imine **iii**. Finally, reduction of **iii** by $NaBH_4$ afforded amine **2**. In other words, we can say that the hypervalent iodine(III) reagent-mediated rearrangement is an analogue of the reaction using *N*-haloamines and strong bases,[2] but allows the use of primary amines directly without prefunctionalization and proceeds under mild conditions near room temperature.

In summary, we have presented the hypervalent iodine-mediated oxidative rearrangement reaction of primary amines.

Scheme 3. Possible Reaction Mechanism



Different from the reaction of secondary amines, the combined usage of $PhI(OAc)_2$ and Cs_2CO_3 is revealed to be the key for the reaction of primary amines. The method can be applied to the preparation of both acyclic and cyclic amines including an eight-membered ring compound. Several mechanistic investigations show that the reaction likely proceeded via a concerted mechanism rather than a radical or a nitrene mechanism. This method is practical and can be easily carried out using commercially available hypervalent iodine reagents. Further investigation of these transformations is underway in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b00559.

Full experimental details and characterization data (PDF)

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: murai@phs.osaka-u.ac.jp.

ORCID

Mitsuhiro Arisawa: 0000-0002-7937-670X
Kenichi Murai: 0000-0002-1689-6492

Notes

The authors declare no competing financial interest.

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(15) The reactions of **1a** using Koser's reagents or iodosobenzen in the presence of Cs_2CO_3 were also examined. However, decrease of the yield were observed: with Koser's reagents, 58%; with iodosobenzen, 31% (details see, SI).

(16) A substrate having 2-thiophene as an aryl group gave complex mixtures.

(17) We tried the reactions of trialkyl methyl amines, such as 1,1-dibenzylethylamine and 1-adamantylamine, but the method was not applicable for these substrates.

(18) The remaining of starting material was observed with **1l** and **1m**.

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