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Reduction of Benzolactams to Isoindoles via an Alkoxide-Catalyzed Hydrosilylation

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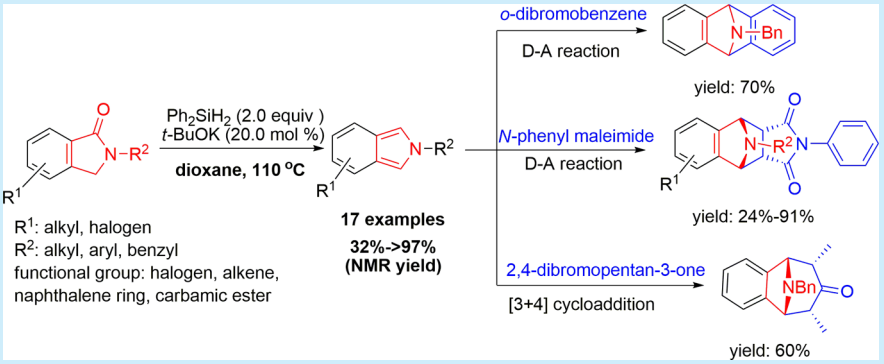
Supporting Information

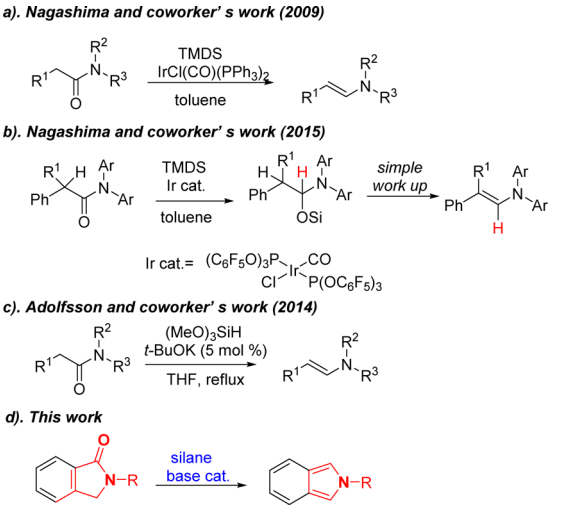


ABSTRACT: An alkoxide-catalyzed reduction of benzolactams to isoindoles with silanes was realized. With *t*-BuOK as the catalyst and Ph_2SiH_2 as the reductant, a series of benzolactams containing different functional groups were reduced to the corresponding isoindoles, which could be captured by *N*-phenyl maleimide to form Diels–Alder products in moderate to good yields. Deuterium labeling studies and the hydrosilylation of benzolactam in DMF indicated that the deprotonation of benzolactams took place at C3 potion during the reduction.

Hydrosilylation is an appealing method for the reduction of carbonyl compounds, which represents the most important and well-established transformation in organic chemistry.[1] Hydrosilanes are air- and moisture-stable hydride sources, and can be readily activated under mild conditions. Various catalysts had been explored for the chemo-, regio-, and/or stereoselective hydrosilylation of carbonyl compounds and carboxylic acid derivatives.[2] In particular, the hydrosilylation of amides showed high chemoselectivity, which preceded other methods.[3] Moreover, by tuning the catalysts and silanes, the hydrosilylation of amides enabled the selective formation of different products such as amines, imines, aldehydes, alcohols, nitriles or deacylated amines.[3,4]

Nagashima et al. first reported the hydrosilylation of aryl and alkyl substituted acetamide derivatives to form enamines efficiently, with $IrCl(CO)(PPh_3)_2$ as the catalyst and TMDS or PMHS as the hydride source (Scheme 1a).[5] The reaction may proceed through the addition of a Si–H bond to carbonyl group to form a silylhemiaminal as intermediate, followed by deprotonation and elimination of the siloxy moiety to give enamines.[5] Recently, the same group realized the stepwise synthesis of enamines by hydrosilylation of amides using iridium complex with electron-withdrawing phosphorus ligands as the catalyst (Scheme 1b).[6] Furthermore, Adolfsson's group reported *t*-BuOK catalyzed hydrosilylation of tertiary amides to enamines.[7] However, the hydrosilylation of benzolactams has not been reported.

Scheme 1. Profiles for Hydrosilylation of Amides to Enamines



Due to benzolactams having acidic hydrogen atoms at their 3-position, we envision that the selective hydrosilylation of benzolactams may lead to the formation of isoindoles derivatives, which are very important 10π-electron aromatic hetero-

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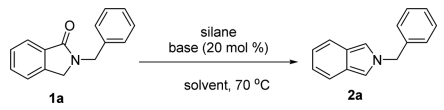
cycles,[8] and have often been employed as synthetic building blocks for highly conjugated systems in materials science and medicinal chemistry as antibiotic and antitumor agents and thrombin.[9] Moreover, isoindoles are highly reactive intermediates in cycloaddition reactions.[10] Several methods were developed to synthesize isoindoles, such as retro Diels–Alder reactions, 1,3-dipolar cycloadditions, oxidative processes, alkylation and isomerization reactions of isoindolinones, and condensation of *o*-phthalaldehyde or benzylic amines.[8,11] Among these methods, the reduction process is the most straightforward one. However, the noticeable over-reduction when reducing phthalimides/lactams using metal hydrides usually resulted in low yields of isoindoles, and instead, substituted pyrroles were often obtained.[12] Herein, we reported an efficient procedure of base-catalyzed reduction of benzolactams to isoindoles with hydrosilanes.

Considering the easy removability of the benzyl group, the hydrosilylation of 2-benzylisoindolin-1-one (**1a**) was chosen as the model reaction to explore the possibility of the reduction of benzolactams to isoindoles with hydrosilanes. Although the metal catalyzed hydrosilylation could enable the transformation of amides to enamines,[5,6] Lewis base-catalyzed hydrosilylation is no doubt more attractive in terms of environmental benignity and ready availability of the catalyst.[4c,13] KOH was initially selected as the catalyst in the model reaction, on the basis of our previous report on the hydrosilylation of imides.[14] When 20.0 mol % of KOH and Ph_2SiH_2 (2.0 equiv) were used, 85% of the benzolactam (**1a**) was converted to the desired product, *N*-benzyl isoindole (**2a**) (Table 1, entry 1). Then, other bases were examined in the hydrosilylation. When potassium methoxide or ethoxide was used as the catalyst, the conversion of the reduction increased slightly, but the yields of **2a** almost unchanged (Table 1, entries 3 and 5). Gratifyingly, *t*-BuOK displayed best catalytic activity to afford almost quantitative yield of *N*-benzyl isoindole (**2a**) (Table 1, entry 7). Comparing with potassium alkoxides, sodium alkoxides resulted in a decrease in conversions and yields of the hydrosilylation (Table 1, entries 2, 4, 6 and 8). In addition, potassium bis(trimethylsilyl) amide and lithium diisopropylamide were inferior for the reaction (Table 1, entries 9 and 10). No reactions occurred in the cases of using weaker bases (K_2CO_3 or K_3PO_4) as the catalyst. Omission of the base resulted in no desired reduction of the benzolactams (**1a**) (Table 1, entry 11). The yield of **2a** was decreased to 80% when the *t*-BuOK loading was lowered to 10.0 mol % (Table 1, entry 12).

We also screened some other silanes, solvents and reaction temperatures during the *t*-BuOK catalyzed hydrosilylation of the benzolactams. More reactive silane for example, $PhSiH_3$, improved the conversion of **1a**, but a lower selectivity and yield (85%) of isoindole (**2a**) was obtained (Table 1, entry 13). Using less active silanes led to the compromised conversions and yields (Table 1, entries 14–17). For example, TMDS (tetramethyldisiloxane) and PMHS (polymethylhydrosiloxane) gave **2a** in 70% and 13% yields, respectively. Changing the solvent from THF to DME (dimethoxyethane) decreased the yield of **2a** (Table 1, entry 18). A nearly quantitative yield of **2a** was obtained in dioxane (Table 1, entry 19). For the nonpolar solvent, toluene, the isoindole (**2a**) could also be obtained in 90% yield (Table 1, entry 20). However, only 30% of lactam (**1a**) was converted to isoindoles (**2a**) in CH_3CN (Table 1, entry 21), while no reaction occurred in DCE. When the reaction was carried out in refluxing dioxane, the reaction time was reduced to 10 h (Table 1, entry 22).

Table 1. Optimization of the Reaction Conditions of the Base-Catalyzed Reduction of 2-Benzylisoindolin-1-one (1a)[a]



entry	base	silane	solvent	conv (%)	yield (%)
1	KOH	Ph_2SiH_2	THF	85	85
2	NaOH	Ph_2SiH_2	THF	61	60
3	EtOK	Ph_2SiH_2	THF	90	80
4	EtONa	Ph_2SiH_2	THF	66	60
5	MeOK	Ph_2SiH_2	THF	92	81
6	MeONa	Ph_2SiH_2	THF	70	70
7	*t*-BuOK	Ph_2SiH_2	THF	>97	>97
8	*t*-BuONa	Ph_2SiH_2	THF	60	59
9	KHMDS	Ph_2SiH_2	THF	44	13
10	LDA	Ph_2SiH_2	THF	26	24
11	–	Ph_2SiH_2	THF	–	–
12[b]	*t*-BuOK	Ph_2SiH_2	THF	82	80
13[c]	*t*-BuOK	Ph_3SiH	THF	>97	85
14[d]	*t*-BuOK	TMDS	THF	52	51
15[e]	*t*-BuOK	$(EtO)_3SiH$	THF	54	53
16[e]	*t*-BuOK	$(MeO)_3SiH$	THF	14	13
17[e]	*t*-BuOK	PMHS	THF	52	51
18	*t*-BuOK	Ph_2SiH_2	DME	40	31
19	*t*-BuOK	Ph_2SiH_2	dioxane	>97	>97
20	*t*-BuOK	Ph_2SiH_2	toluene	>97	90
21	*t*-BuOK	Ph_2SiH_2	CH_3CN	30	30
22[f]	*t*-BuOK	Ph_2SiH_2	dioxane	>97	>97

[a]Reaction conditions: 2-benzylisoindolin-1-one (**1a**, 223.3 mg, 1.0 mmol), Ph_2SiH_2 (368.6 mg, 2.0 mmol), base (20.0 mol %), THF (2.0 mL), 70 °C, 36 h; conversion and yields were determined by ^{1}H NMR analysis (internal standard: 4,4′-di-*tert*-butyl-1,1′-biphenyl). [b]*t*-BuOK (10.0 mol %) was used. [c]$PhSiH_3$ (151.3 mg, 1.4 mmol) was used. [d]TMDS (268.6 mg, 2.0 mmol) was used. [e]Silane (4.0 mmol) was used. [f]The conversion was finished in 10 h at 110 °C.

Next, the scope of base-catalyzed hydrosilylation of benzolactams (**1**) to isoindoles (**2**) was probed under the optimized conditions. As shown in Table 2, the reduction reactions led to efficient formation of a number of isoindoles (**2**). Because of the instability of isoindoles, we listed the isolated yields of the compound **4**, which were Diels–Alder reaction products of isoindoles (**2**) and *N*-phenyl maleimide (**3**).[8,15] The hydrosilylation of *N*-benzyl, alkyl or aryl substituted benzolactams gave the corresponding isoindoles (**2**) in moderate to good yields. *N*-Phenyl benzolactam (**1c**) was reduced to the corresponding isoindole **2c** in 92% NMR yield and 84% isolated yield of **4c**. *N*-Ethyl isoindole (**2g**) was obtained in 87% yield. For benzolactams **1b**, **1d**, **1i** and **1j′**, bearing an alkylated benzene ring, the hydrosilylation proceeded smoothly to give the corresponding isoindoles (**2b**, **2d**, **2i** and **2j′**) in excellent yields. 2-(4-(*tert*-Butyl)phenyl)-2*H*-isoindole (**2d**) and 2-benzyl-5-methyl-2*H*-isoindole (**2i**) were generated in 97% and 96% yields, respectively. However, the steric hindrance of substitutions at the nitrogen atom or C4 position of benzolactams had obvious influence on the base-catalyzed hydrosilylation. The yield of corresponding isoindole **2h** was reduced to 60% in the case of bulkier *N*-isopropyl benzolactam (**1h**) as the substrate. The existence of propyl group at the 4-position of benzolactam **1j** inhibited the reaction and no corresponding product (**2j**) was obtained. While the isoindole **2j** could be generated in 87% yield when the

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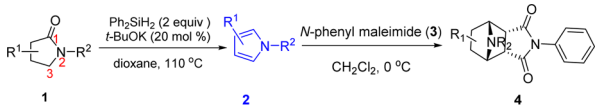
Table 2. *t*-BuOK -Catalyzed Reduction of Benzolactams (1) to Isoindoles (2)[a] with Hydrosilane



substrate	R^1	R^2	yield **2** (%)[b]	yield **4** (%)[c]
1a			91(86)[d]	88
1b			90	85
1c			92	84
1d			97	91
1e		MeO	53	49
1f[e]		F_3C	56	38
1g			87	81
1h			60	53
1i			96	85
1j		Me	trace	trace
1j'		Me	87	68
1k			75	55
1l[e]		Br	85	70
1m[e]	Cl		50	38
1n[e]			32	24
1o		Boc-N	80	78
1p			89	84

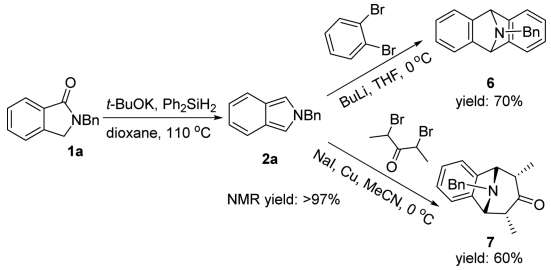
[a]Reaction conditions: benzolactam (**1**, 1.0 mmol), Ph_2SiH_2 (368.6 mg, 2.0 mmol), *t*-BuOK (22.4 mg, 20.0 mol %), dioxane (2.0 mL), 110 °C. [b]Yields determined by ^1H NMR analysis (internal standard: 4,4′-di-*tert*-butyl-1,1′-biphenyl). [c]Isolated yields. [d]Isolated yield of 2.0 g-scale of **1a**. [e]1.0 equiv of *t*-BuOK was used.

benzolactam **1j′** with propyl group at 7-position was used as substrate. The electron-withdrawing groups decreased the reactivity of benzolactams. The reduction of 2-(4-Bromophenyl)isoindolin-1-one (**1l**) gave the corresponding isoindole **2l** in 47% yield when 20.0 mol % ^{t}BuOK was used. Increasing the loading of *t*-BuOK to 1.0 equiv resulted that the yield was increased to 85% yield. 2-(4-(Trifluoro-methyl)-phenyl)-2*H*-isoindole (**2f**) was produced in only 56% yield. The hydrosilylation of the benzolactam with an amide group (**1n**) gave the product in only 32% of yield even 1.0 equiv of *t*-BuOK was used. Moreover, the reduction system showed good tolerance of functional groups, such as halogens (**1l** and **1m**), alkene (**1p**), naphthalene ring (**1k**), and carbamic ester (**1o**).

The reduction of benzolactam with a pendant alkene group (**1p**) and a Boc-protected amine (**1o**) proceeded smoothly with good chemoselectivity to afford the corresponding isoindoles (**2p** and **2o**) in excellent yields.

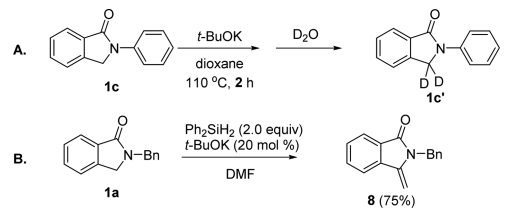
Unlike the classical reduction process employing modified $LiAlH_4$ or Red-Al which result in only moderate yield and over-reduction contaminants, the hydrosilylation of *N*-benzyl benzolactam (**1a**) could be run effectively on a 2.0 g scale wherein quantitative NMR yield and 86% isolated yield of **2a** was observed (Scheme S1). Isoindoles are useful building blocks in the synthesis of functional materials and medicinal molecules.[8,9,9d,16] For example, **2a** was reacted with benzyne to form 1-benzyl-9,10-dihydro-9,10-epimino-anthracene (**6**) in 70% yield which may serve as a useful precursor of luminescent materials.[17] Furthermore, [4 + 3] cycloaddition of the isoindole (**1a**) afford seven-membered compound (**7**) in 60% yield, which occurred in many classes of natural products (Scheme 2).[18]

Scheme 2. Applications of Isoindoles in Materials Science and Synthetic Chemistry



The plausible mechanism of the reduction of benzolactams **1** to isoindoles **2** via the alkoxide catalyzed hydrosilylation may be similar to the reaction path for the hydrosilylation of tertiary amides to enamines that was proposed by Adolfsson et al.[7] The base activates the silane via formation of a pentacoordinated intermediate, which facilitates the transfer of the hydride to the amide carbonyl to generate the silylhemiaminal intermediate. Then the C3-deprotonation followed by elimination of the siloxy moiety to generate isoindoles **2**. The possibility of deprotonation of benzolactam with *t*-BuOK as base was explored. Benzolactam was reacted with *t*-BuOK in dioxane, and then treated with D_2O to give the C3-deuterated benzolactam in 90% ratio (Scheme 3, A). In contrast, the

Scheme 3. Experiments on the Deprotonation of Benzolactam (1)



deuteration did not occur without *t*-BuOK. Moreover, when DMF was used as solvent, the hydrosilylation of benzolactam (**1a**) introduced a methylene group at C3 position instead of the formation of the isoindole (**2a**). In addition, on the basis of our experimental results in Table 2, the steric hindrance of substitution at the nitrogen atom and C4 position of

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benzolactam influenced obviously on the base-catalyzed reduction leading to isoindoles, we speculated that the deprotonation may be the rate-determining step in the transformation of benzolactam to isoindole.

In summary, we have developed an alkoxide-catalyzed hydrosilylation of benzolactams to isoindoles. With *t*-BuOK (20 mol %) as the catalyst and Ph_2SiH_2 as the reductant, various benzolactams were effectively reduced to the corresponding isoindoles in moderate to excellent yields. This catalytic protocol showed good functional tolerance of halogens, alkenes, naphthalene ring, and carbamic esters. Moreover, the products in the hydrosilylation were synthetic skeletons useful in luminescent materials and natural products. The deprotonation of benzolactams occurred during the reduction based on the deuteration of benzolactam (**1c**) and the hydrosilylation of benzolactam (**1c**) in DMF.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b02739.

Comparison data with the traditional reduction process, experimental procedures, and product characterization data (PDF)

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Notes

The authors declare no competing financial interest.

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