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Sterically Congested 2,6-Disubstituted Anilines from Direct C–N Bond Formation at an Iodine(III) Center

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Abstract: 2,6-Disubstituted anilines are readily prepared from the direct reaction between amides and diaryliodonium salts. As demonstrated for 24 different examples, the reaction is of unusually broad scope with respect to the sterically congested arene and the nitrogen source, occurs without the requirement for any additional promoter, and proceeds through a direct reductive elimination at the iodine(III) center. The efficiency of the coupling procedure is further demonstrated within the short synthesis of a chemerin binding inhibitor.

Carbon–nitrogen bond formation is a synthetic endeavor of paramount importance. Processes that enable direct amination reactions decidedly streamline organic synthesis, and can be of special significance in cases that have no biosynthetic precedence.^[1] A particularly prominent example of innovative C–N bond installment is the synthesis of anilines by transition-metal-catalyzed coupling between amine sources and aryl halides or related arene sources. In this area, palladium^[2] and copper^[3] have traditionally received major attention. A complementary approach consists of carbon–nitrogen bond formation through the application of activated diaryliodonium derivatives,^[4] which serve as suitable partners in transition-metal coupling processes.^[5] Alternatively, they may engage in direct C–N coupling reactions, even in the absence of a metal promoter, an aspect which has attracted considerable attention from biomedical sciences. Examples include aniline syntheses by the arylation of amides,^[6a,b] aromatic amines,^[6c,d] methoxyamines,^[6e] ammonia,^[6f] heterocycles,^[6g,h] and radical rearrangements.^[6i] Although aniline syntheses have thus far reached a significant level of general-

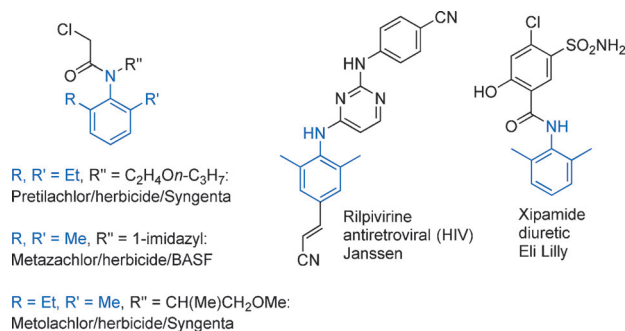


Figure 1. Examples of pharmaceuticals and herbicides containing a 2,6-dialkylaniline core.

ity,^[7] we observed that there is little synthetic prospect for C–N bond formation in the particular case of 2,6-disubstituted aniline derivatives.^[8] This scaffold is of significant interest, since it is incorporated in a series of relevant compounds of pharmaceutical and herbicidal interest. Selected examples are depicted in Figure 1.

We demonstrate here that iodine(III) compounds of the general formula $Ar_2I-NPhth$ ($HNPhth = phthalimide$), which can be accessed *in situ* by anion exchange of common diaryliodonium salts^[4b,c] with potassium phthalimide, efficiently promote a direct reductive $C(sp^2)-N$ bond formation, with a particularly broad scope for the coupling of sterically congested arene groups. In contrast to common methods in the area, no transition-metal promoters are required for this process.

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Table 1: Metal-free C–N bond formation from diaryliodonium salts: reaction optimization.

Entry	Amide (equiv)	Solvent	t [h]	T [°C]	Yield [%] ^[a]
1	phthalimide (5.0)	CH ₂ Cl ₂	44	40	9
2	phthalimide (4.0)	toluene	24	84	60
3	phthalimide (3.0)	toluene	24	84	54
4	phthalimide (3.0)	toluene	24	100	67
5	phthalimide (2.0)	toluene/PhCl	24	100	23
6	phthalimide (2.0)	toluene/PhCl	24	110	50
7	phthalimide (2.0)	toluene/PhCl	24	120	50
8	^{4F} phthalimide (3.0) ^[b]	toluene	24	100	75

[a] Yield of isolated product after purification. [b] ^{4F}Phthalimide = tetrafluorophthalimide. The product is *N*-phenyl tetrafluorophthalimide **3a**.

The reaction was initially developed for the diphenyliodonium salt **1a** and phthalimide as the nucleophilic nitrogen source (Table 1).^[9] Treatment of **1a** with 5 equivalents of potassium phthalimide in dichloromethane at reflux led to a low yield of the desired product **2a** (entry 1). A change in the solvent allowed optimization of the reaction, and yields of 60% and 54% were obtained in toluene with 4 and 3 equivalents of phthalimide, respectively (entries 2 and 3). Alternative solvents such as dichloroethane, dioxane, or DMF resulted in little to no conversion.^[10] Increasing the reaction temperature to 100 °C led to the yield rising to 67% (entry 4), while the addition of chlorobenzene led to higher solubilization of potassium phthalimide, albeit at the expense of the yields (entries 5–7). Finally, tetrafluorophthalimide was tested (entry 8). Under the former best conditions for phthalimide, its tetrafluoro derivative led to formation of **3a** in an improved 75% yield of the isolated product.

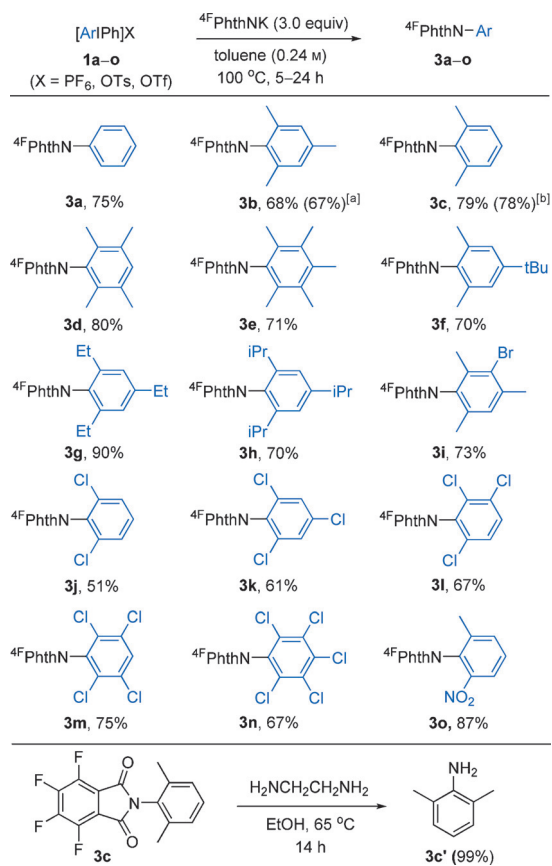
Replacement of the diphenyliodonium salt **1a** by the mixed reagent [MesIPh]OTs (**1b**) led to exclusive formation of a C–N bond involving the mesityl group, and *N*-mesityl tetrafluorophthalimide **3b** was obtained in 68% yield. A related preferential behavior for mesitylene transfer was reported for related studies on aniline syntheses with diaryliodonium reagents, including an amination with acetamide.^[6c,11] These processes are complemented by electrophilic amination of arenes initiated by hypervalent iodine,^[12]

although this approach has not yet been employed for the synthesis of 2,6-disubstituted aniline derivatives.

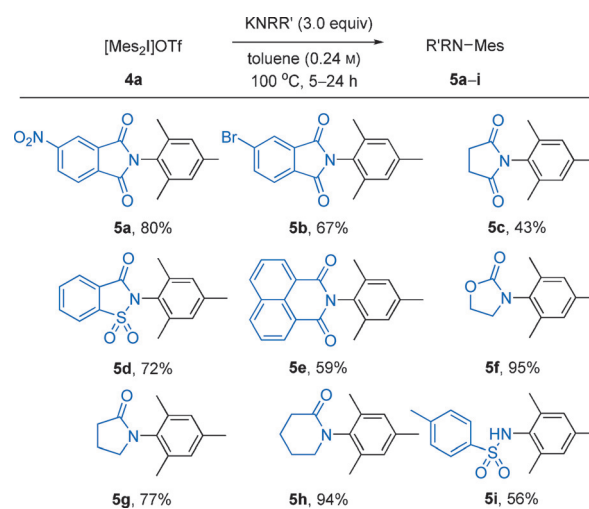
Based on this observation, the aryl amination reaction was investigated for additional sterically congested aryl substituents (Scheme 1). As a consequence of the successful formation of **3b**, preference was directly given to the challenging 2,6-disubstitution motif. For this particular class of compounds, the present transformation indeed provided excellent conditions. Investigation of [Mes₂I]OTf revealed an identical outcome for the synthesis of **3b**. As a result of their more economic synthesis,^[4] mixed aryl(phenyl)iodonium salts were employed in the following. In this way, purification is eased because, with PhI as the by-product, compounds **3** can usually be obtained by simple crystallization. In addition to the examples of **3a** and **3b**, several 2,6-dimethylated derivatives could be aminated cleanly to give the corresponding derivatives **3c–f** in yields of 70–80%. Aniline derivative **3c** was synthesized readily on a 4.6 mmol scale. Larger *ortho* substituents, such as ethyl or even 2-propyl, were likewise tolerated (**3g** and **3h**; 90 and 70% yield, respectively). Halogen substitution is readily compatible, as demonstrated for product **3i**, which would be difficult to synthesize through common transition-metal catalysis.^[2] The 2,6-dichloro motif and higher-substituted derivatives thereof were explored by using **3j–3n** (51–75% yields). Finally, the mixed 2-nitro-6-methyl derivative **3o** demonstrated that even the stereo-electronically demanding nitro substituent can be employed (87% yield). In all these reactions, exclusive transfer of the higher substituted arene was observed, and the alternative product **3a** was not detected in any of these cases. The attractiveness of tetrafluorophthalimide as the ammonia surrogate was demonstrated through the deprotection of **3c** by convenient aminolysis to provide 2,6-dimethylaniline **3c'** quantitatively.

The successful synthesis of compounds **3b–o** significantly broadens the availability of 2,6-disubstituted anilines and higher-substituted derivatives thereof.

The present amination is not limited to phthalimide and tetrafluorophthalimide. By employing dimesityliodonium-



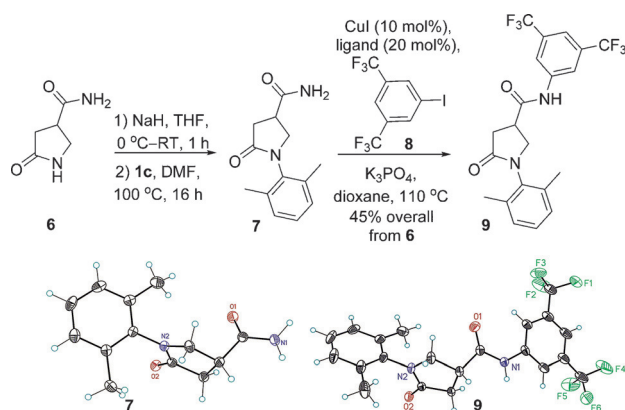
Scheme 1. Amination of 2,6-disubstituted arenes: scope. [a] Reaction with [Mes₂I]OTf (**4a**). [b] Reaction on a 4.6 mmol scale.



Scheme 2. Amination of [Mes₂I]OTf (**4a**) with different nitrogen sources: scope.

(III) triflate as the aryl component, other phthalimides such as 4-nitrophthalimide and 4-bromophthalimide provide similarly good results (Scheme 2, products **5a,b**). Additional successful nitrogen sources include succinimide (product **5c**), saccharin (product **5d**), and 1,8-naphthalimide (product **5e**), which led to products in 43–72% yield. Moreover, the pharmaceutically important class of oxazolidinones and lactams also undergo arylation, as demonstrated for the three products **5f–h** (77–95% yield). While common carboxamides display low reactivity, tosylimide underwent a clean arylation reaction to **5i** (56% yield).

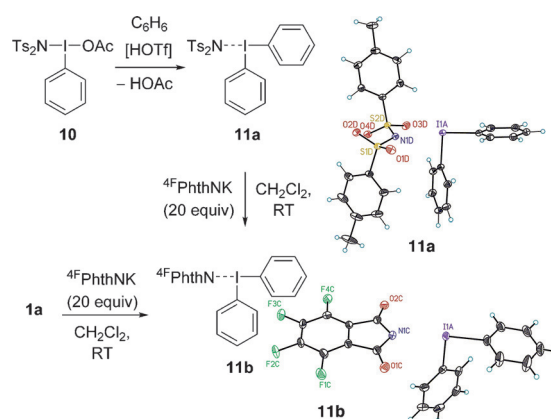
The synthetic utility of the present coupling was further demonstrated within a short synthesis of the N,N'-diarylated pyrrolidinone carboxamide **9** (Scheme 3). This compound is



Scheme 3. Synthesis of N,N'-diarylated pyrrolidinone carboxamide **9** and solid-state structures of **7** and **9** (ellipsoids at 50% probability).

representative of a family of binding inhibitors of the chemoattractant peptide chemerin to the G-protein coupled receptor ChemR23. Its reported preparation comprises a linear synthesis based on preformed anilines.^[13] By employing our new C–N coupling method as the key transformation, a convenient protecting-group-free two-step synthesis starts with selective N-arylation at the lactam of commercially available pyrrolidinone carboxamide **6**. The second N-arylation at the free amide group in **7** yields inhibitor **9**, which is obtained in an overall 45% yield from **6**.^[14,15] Depending on the chosen aryl groups, rapid structural diversification should be possible, thereby creating new pharmaceutical space through advanced C–N coupling.

Mechanistically, the reaction should proceed by anion exchange at the iodine center, where the tetrafluorophthalimidato ligand is incorporated prior to aniline formation. To investigate this direct C–N bond formation from diaryliodonium compounds containing defined imidato groups, we synthesized two derivatives with different nitrogen entities (Scheme 4). Compound **11a** contains the bistosylimide moiety, which represents the standard nitrogen source in our recent iodine(III)-mediated amination chemistry.^[16,17] It was conveniently accessed from the known iodine(III) derivative **10**^[16a] by electrophilic activation of benzene. Compound **11b** contains the tetrafluorophthalimide anion and was generated through amide exchange with potassium



Scheme 4. Synthesis of diaryliodonium(III) amidato complexes **11a,b** and solid-state structures (ellipsoids at 50% probability).^[14] Selected bond lengths [Å] and angles [°]: **11a**: N1–I1 2.874(1); N1–I1–C1 170.09(5), N1–I1–C7 79.57(5). **11b**: N1–I1 2.758(2); N1–I1–C1 174.95(9), N1–I1–C7 86.29(8).

tetrafluorophthalimide from **11a** or **1a**, respectively. The latter synthesis successfully demonstrates the viability of common anion exchange for phthalimide in complexes **1a–o**. According to X-ray analysis, both species **11a,b** display the expected T-shape constitution at the iodine center, with only a small deviation of the N–I–C bond angles from linearity.^[14] The respective iodine–nitrogen bond lengths of 2.874(1) and 2.758(2) Å are comparable. They are longer than the N–I bond in a related phthalimidato iodine(III) derivative reported by Minakata and co-workers, which generates a nucleophilic phthalimide source under oxidation conditions.^[18] The present reactivity scenario corroborates an anionic character of the tetrafluorophthalimide in **11b**, which excludes involvement of electrophilic amination pathways commonly encountered in hypervalent iodine chemistry.^[12,19]

More instructively, **11a** and **11b** display significantly different chemical performances. As a consequence of its highly stabilized bistosylimide group as the nitrogen source, iodine(III) compound **11a** is stable against any observable reductive carbon–nitrogen bond formation.^[10] Even upon prolonged heating in toluene solution, only starting material was recovered. In contrast, isolated **11b** readily undergoes thermally induced quantitative formation of the C–N coupling product **3a** with a clear first order dependence and the expected temperature dependence.^[10] The reaction could thus be monitored by ¹⁹F NMR spectroscopy at different temperatures between 80 and 110 °C by employing a toluene/DMF solution to guarantee homogeneous conditions.^[20] An Arrhenius plot^[10] provides a value for the activation energy of 34.8 kcal mol^{−1} for C–N bond formation from **11b**, which is in agreement with the high reaction temperature required experimentally. The corresponding Eyring plot reveals an activation enthalpy $\Delta\Delta H^\ddagger$ of 34.1 kcal mol^{−1} and an entropy of $\Delta\Delta S^\ddagger = 105.5$ JK^{−1}. This scenario supports the assumption of an ordered transition state **A**, in which the original N–I bond has dissociated and in which product formation proceeds through a three-center-four-electron transition

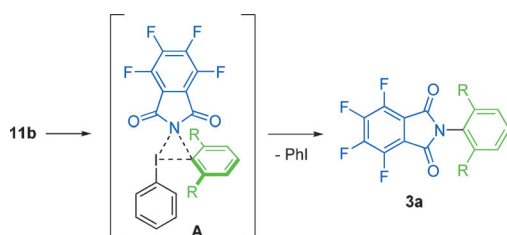


Figure 2. Mechanism of the C–N bond formation.

state (Figure 2).^[2,21,22] This pathway is reminiscent of related mechanisms in transition-metal chemistry. It is further aided by the 2,6-disubstitution motif, which adds steric bulk to transition state **A**, thus promoting reductive elimination within a completely predictable regioselective manner.^[23] Reductive elimination on diaryliodonium(III) derivatives through a transition state analogous to **A** was invoked previously by Ochiai and co-workers.^[24]

In summary, we have developed a new procedure for the rapid and productive synthesis of 2,6-disubstituted anilines and their higher-substituted derivatives. It proceeds through a direct reductive C–N bond formation, which constitutes a transition-metal-like performance of the iodine(III) reagent. This approach is applicable for a wide range of aryl groups and nitrogen sources. It significantly streamlines the synthetic path to this class of compounds and should enable a larger structural diversification of these aniline cores in the screening of molecular structures of pharmaceutical interest.

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