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Iodocyclization of *N*-Arylpropynamides Mediated by Hypervalent Iodine Reagent: Divergent Synthesis of Iodinated Quinolin-2-ones and Spiro[4,5]trienones

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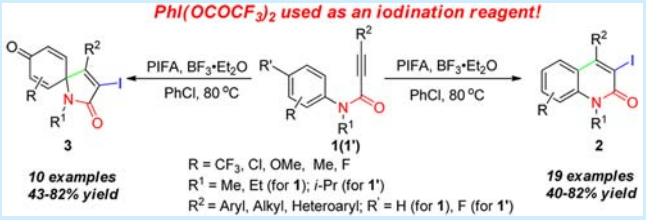
Supporting Information

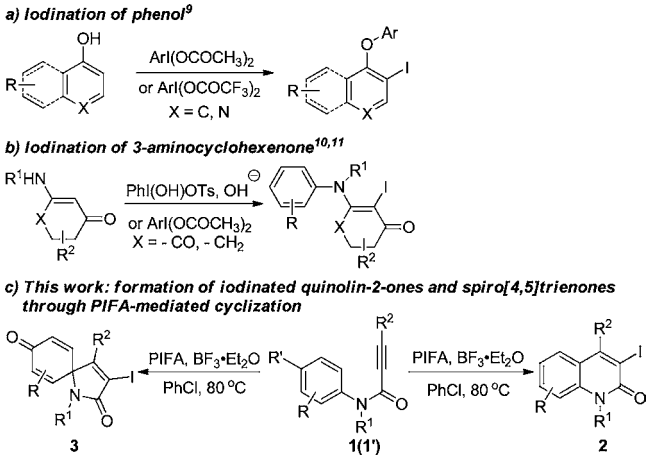
ABSTRACT: $PhI(OCOCF_3)_2$ acts as both a nonmetal oxidant and an iodination reagent to trigger iodocyclization of *N*-arylpropynamides while selectively affording iodinated quinolin-2-ones or the spiro[4,5]trienone skeleton, depending on the substituent pattern. In cases where the *N*-arylpropynamide bears a *para*-fluorine on the aniline ring, the spiro compound is formed via an exclusive defluorination process; otherwise, the product was quinolin-2-one.



Quinolines and spiro[4,5]trienones are essential parts of the skeleton of numerous natural compounds and pharmaceuticals[1] and are also valuable intermediates in various organic syntheses.[1,2] Among the known protocols, quinolines and isoquinolines are usually constructed via the electrophilic cyclization of arylalkynes.[3] Pioneering works have proven that iodine/cerium(IV) ammonium nitrate,[4] *N*-iodosuccinimide,[5a,c] iodine,[5b−d] and iodine monochloride[5c,d] can be employed as the iodine source for intramolecular electrophilic cyclization to afford iodinated quinolones. Similarly, iodinated spiro[4,5]-trienones could be synthesized through the intramolecular *ipso*-halocyclization,[6] using iodine monochloride,[6a,e] iodine,[6a,h] or *N*-iodosuccinimide[6b,c,g] as the iodine source. However, all of the transformations reported so far have been limited to using conventional electrophilic iodination reagents to act as both the oxidant and the iodine source. To the best of our knowledge, application of a hypervalent iodine reagent as an oxidant and an iodination reagent to access iodinated quinolin-2-ones and spiro[4,5]trienones has not yet been reported.

Hypervalent iodine(III) reagents, such as phenyliodine bis(trifluoroacetate) (PIFA), phenyliodine diacetate (PIDA), and iodosobenzene (PhIO),[7] are a class of efficient and environmentally friendly oxidants. They have been widely applied to the construction of various heterocyclic compounds.[8] However, in nearly all of the transformations, the iodo moiety in the hypervalent iodine(III) reagents was "wasted" as part of the byproduct phenyl iodine, mainly due to the unreactivity of the $C(sp^2)$−I bond under most circumstances. There have been only a few examples[9] that describe the application of hypervalent iodine(III) as an iodination reagent for the synthesis of iodinated organic compounds, in which the iodo moiety in the hypervalent iodine(III) reagent was incorporated into the final product. For example, PIFA- or PIDA-mediated oxidative cyclization of hydroxy-substituted (hetero)aromatic compounds was reported to lead to the synthesis of *ortho*-iododiaryl ethers (Scheme 1a).[9a−d] Similar applications were found in the synthesis of *N*-arylated α-iodoenaminone from 3-aminocyclohexenone through $PhI(OH)OTs$[9e,f] or PIDA-mediated oxidation,[9g] involving formation of an iodonium salt intermediate (Scheme 1b).

Scheme 1. Existing Reactions Using Hypervalent Iodine(III) as Iodination Reagents



However, there is known no report on the application of hypervalent iodine(III) for construction of iodinated heterocycles, in which the iodo moiety is from the hypervalent iodine reagent. Here, we report the divergent reaction pathways

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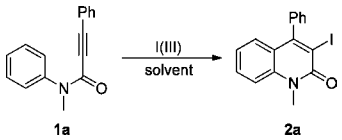
involving PIFA used as a nonmetal oxidant and an iodination reagent to trigger iodocyclization of *N*-arylpropynamides (Scheme 1c).

Based on the reactions we studied in our previous work, we initially postulated an intramolecular cyclization reaction of *N*-arylpropynamide (**1a**) in the presence of a hypervalent iodine(III) reagent to afford a quinolin-2-one with a trifluoroacetate or hydroxyl group at the 3-position (see Supporting Information (SI) for the proposed mechanism). Reaction of **1a** with PIFA in the presence of $BF_3{\cdot}Et_2O$ in DCE under stirring at 80 °C for 12 h was found to give an unexpected iodinated quinolin-2-one **2a** in fairly low yield (Table 1, entry 1). This result led us to discover that PIFA could be used as an iodination reagent to insert its iodo moiety into the final product. Based on the initial result, we used **1a** as the model substrate to probe the optimal conditions for this newly discovered transformation. After a series of experimental variables, including the type of the hypervalent iodine oxidant, solvent, temperature, and additive, had been systematically tested (see SI for details), we identified the best conditions to be 1 mmol of **1a** with 2.2 equiv of PIFA and 1.0 equiv of $BF_3{\cdot}Et_2O$ in PhCl at 80 °C (Table 1, entry 9).

Table 1. Optimization of Reaction Conditions[a]

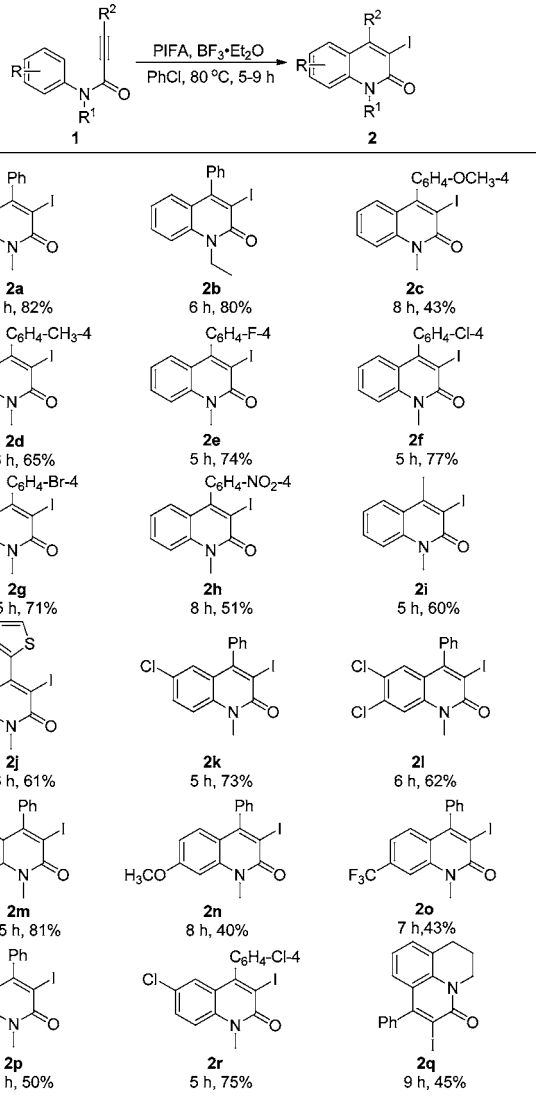


entry	oxidant	additive	solvent	temp (°C)	time (h)	yield (%)[b]
1[c,d]	PIFA	$BF_3{\cdot}Et_2O$	DCE	80	12	56
2[d]	PIFA	$BF_3{\cdot}Et_2O$	DCE	80	12	72
3	PIFA	none	DCE	80	24	NR
4[d]	PIFA	TMSOTf	DCE	80	7	70
5	PIFA	$BF_3{\cdot}Et_2O$	DCE	80	12	80
6	PIFA	$BF_3{\cdot}Et_2O$	toluene	80	5	43
7	PIFA	$BF_3{\cdot}Et_2O$	TFE	80	24	NR
8	PIFA	$BF_3{\cdot}Et_2O$	xylene	80	5	49
9	PIFA	$BF_3{\cdot}Et_2O$	PhCl	80	5	82
10	PIFA	$BF_3{\cdot}Et_2O$	PhCl	60	9	75
11	PIFA	$BF_3{\cdot}Et_2O$	PhCl	110	3	53
12	PIDA	$BF_3{\cdot}Et_2O$	DCE	80	24	trace
13	PhIO	$BF_3{\cdot}Et_2O$	DCE	80	24	NR

[a]Reaction conditions: **1a** (1.0 mmol), oxidant (2.2 mmol), additive (1.0 mmol) in solvent (20 mL) unless otherwise stated. [b]Isolated yields. [c]1.5 equiv of PIFA was used. [d]Reaction conducted using 0.5 mmol of additive.

Under these conditions, the scope and limitation of this iodocyclization method is investigated and depicted in Scheme 2. As shown in Scheme 2, the amide with a *N*-methyl group was replaced by an ethyl group and underwent iodocyclization to afford **2b**. Unfortunately, the *N*-H-substituted substrate failed to afford the desired product (not shown). These results suggest that R^1 is limited to alkyl groups for this method. For aryl R^2 groups, both electron-donating and electron-deficient substituents on the phenyl ring could be tolerated (Scheme 2, **2c**–**h**), with the lowest yields coming from substrates carrying the most electron-donating (**2c**, 43%) and the most electron-withdrawing group (**2h**, 51%). With the R^2 group, both alkyl and heteroaryl groups were allowed (**2i**,**j**). For the R group, various substitution patterns, including mono- or disubstituted Cl and Me and strongly electron-donating (MeO) or electron-withdrawing

Scheme 2. PIFA-Mediated Synthesis of Iodinated Quinolin-2-ones[a]



[a]All reactions carried out at 80 °C with **1** (1.0 mmol), PIFA (2.2 mmol), $BF_3{\cdot}Et_2O$ (1.0 mmol) in PhCl (20 mL); isolated yields are given.

groups (CF_3), were tolerated for this method (**2k**–**p**). The yield for **2r** is similar to that of **2f**,**k**. We found that this iodocyclization protocol could be utilized in the conversion of dihydroquinolinamide substrate **1q** into the more complicated iodinated **2q**.

As reported previously,[6c,d,10] reaction of **1a** bearing a *para*-methoxy on the aniline ring gave iodinated spiro[4,5]trienones **3a** under similar reaction conditions; in our study, we found that a substrate bearing a *para*-fluoro group on the aniline ring (**1a**′) could be converted to the same **3a**, with no detection of quinolin-2-one. No mechanistic study has been carried out on this unusual defluorination process since the discovery of these transformations in previous works.[10e,f,11] Our further investigation showed that this defluorination phenomenon was not excluded to **1a**′. Various derivatives of **1a**′ were found to go through the same conversion to form the corresponding substituted spiro[4,5]trienones.

As shown in Scheme 3, the R groups can be Cl or F (**3c** and **3d**); R^1 groups can be an alkyl group other than methyl (i.e.,

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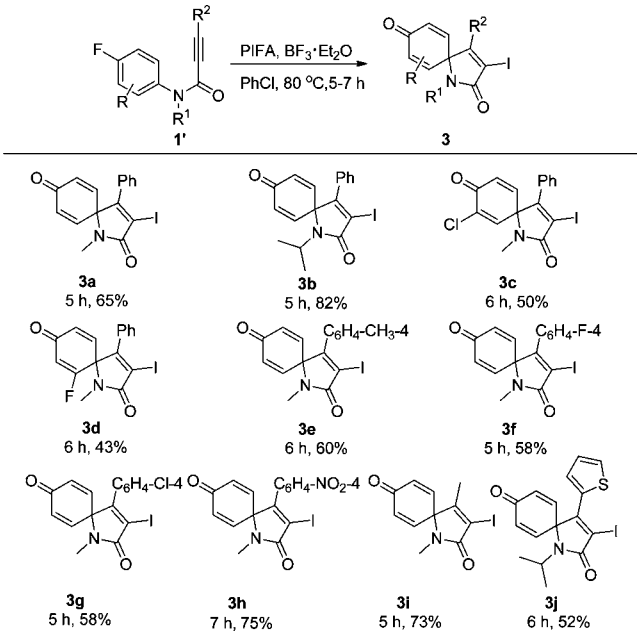
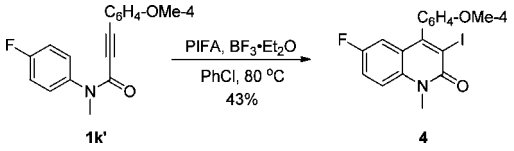
Scheme 3. PIFA-Mediated Synthesis of Iodinated Spiro[4,5]trienones[a]



[a]All reactions carried out at 80 °C with **1′** (1.0 mmol), PIFA (2.2 mmol), BF_3·Et_2O (1.0 mmol) in PhCl (20 mL); yield values refer to isolated yields.

isopropyl group, **3b,j**). A broad range of R^2 groups was found to be tolerated, including aryl groups substituted with an electron-donating (**3e**) or electron-withdrawing group (**3f**–**h**), alkyl groups (**3i**), and heteroaryl groups such as a thienyl substituent (**3j**). Note that the reaction of **1k′**, a derivative of **1a′** bearing a *para*-methoxy group in aryl R^2, gave iodinated quinolin-2-one product **4** in 43% yield with no anticipated product detected at all (Scheme 4).

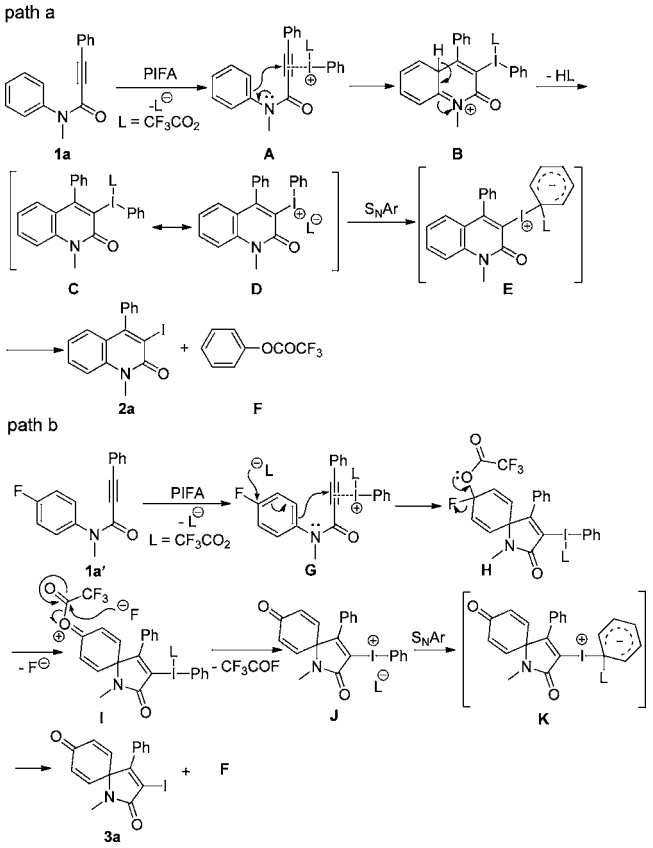
Scheme 4. Exception for the Defluorination Reaction



On the basis of the experimental results and previous reports,[4,5,6b,c] a plausible mechanism was proposed for this PIFA-mediated iodocyclization process (Scheme 5). In path a, an electrophilic activation of the C–C triple bond in **1a**, using BF_3·Et_2O-activated PIFA, afforded iminium salt **A**.[12] An intramolecular electrophilic *ortho*-cyclization occurred to give intermediate **B**, which can be converted to intermediate **C** via subsequent proton elimination. Intermediate **D**, a resonance structure of **C**, underwent an S_NAr process[9a–c] to form the final iodinated quinolin-2-ones **2a**, accompanied by loss of a molecule of phenyl-2,2,2-trifluoroacetate from intermediate **E**.[13]

The mechanism for defluorination reactions for the **1′** series is illustrated in Scheme 5 path b. After formation of intermediate **G** in a similar manner as shown in path a, the trifluoroacetate anion, released in the previous step, nucleophilically attacked the electron-positive aromatic sp^2 carbon due to the neighboring electron-withdrawing F atom, initiating an electrophilic *ipso*-cyclization to form **H**. **H** was converted to **I** by removal of one

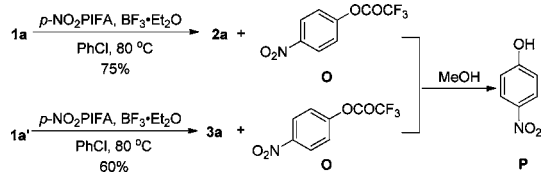
Scheme 5. Proposed Mechanistic Pathways



molecule of the fluoride anion. Nucleophilic attack by the released fluoride anion on the electron-positive carbonyl carbon in **I** resulted in the formation of **J**.[14] A similar S_NAr reaction as in path a occurred to form **K**, which further afforded final product **3a** after the loss of a phenyl-2,2,2-trifluoroacetate molecule. With **1k′**, the reaction is proposed to proceed through an alternative pathway (see SI).

To verify the proposed mechanisms, we attempted to capture the side product **F**. Unfortunately, we did not detect **F** or phenol (possible hydrolysis product of **F**) during the entire reaction process. Using *p*- NO_2PIFA[15] instead of PIFA, we captured 4-nitrophenol via gas chromatography in 10 and 5% yields from reactions involving **1a** and **1a′**, respectively (Scheme 6). Detection of **P** provided cogent support for the proposed reaction pathway.

Scheme 6. Experimental Evidence of the Mechanism



In conclusion, we have reported an alternative metal-free oxidative protocol for the synthesis of iodinated quinolin-2-one and spiro[4,5]trienone skeletons from a series of readily available arylacetylene derivatives. Both processes feature an oxidative C–C coupling reaction and introduction of an iodo moiety with PIFA as the oxidant and iodination reagent. An exclusive

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defluorination process was discovered for a series of *N*-arylpropynamides bearing *para*-fluorine on the aniline ring.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b03455.

Experimental procedure, compound characterization, and NMR spectra (PDF)

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Notes

The authors declare no competing financial interest.

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(14) Although it could be isolated and characterized, intermediate **J** could be confirmed to be formed in the reaction mixture by HRMS; see SI for details.

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