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Direct Preparation of 3-Iodochromenes from 3-Aryl- and 3-Alkyl-2-propyn-1-ols with Diaryliodonium Salts and NIS

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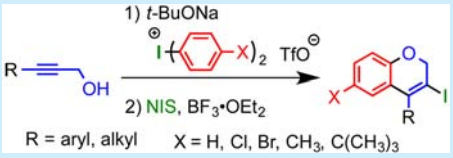
Supporting Information

ABSTRACT: On the basis of a study of the *O*-phenylation of 3-phenyl-2-propyn-1-ol with diphenyliodonium triflate and *t*-BuONa, a variety of 4-aryl-3-iodo-2*H*-benzopyrans were prepared in good to moderate yields in one pot from the reaction of 3-aryl-2-propyn-1-ols with diaryliodonium triflates and *t*-BuONa, followed by the treatment with *N*-iodosuccinimide and $BF_3{\cdot}OEt_2$, under transition-metal-free and mild conditions. The formed 4-phenyl-3-iodo-2*H*-benzopyran was converted into 4-phenyl-2*H*-benzopyran derivatives through C−C bond formations at the 3-position by Pd-catalyzed coupling reactions and into coumarin with oxidants.

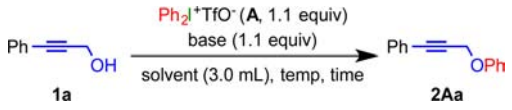


Chromene (2*H*-1-benzopyran) is an important building unit because it is a precursor or an intermediate of natural products, particularly for coumarins, and medicinals.[1] Chromene derivatives bearing anti-HIV,[2a] antitumor,[2b] and antibacterial activities[2c,d] are known. The coumarin unit is a key structure of natural products and biologically active molecules.[3] Extensive studies of the preparation of 2*H*-benzopyrans have been carried out.[4] Typically, 2*H*-benzopyrans are prepared by the cyclization of allyl *o*-vinylaryl ethers with Rh catalyst through an intramolecular ene−metathesis reaction (RCM),[4c,d] the cyclization of α-(*o*-hydroxyaryl)allyl alcohols with Au catalyst via *endo*-cyclization,[4e] and the cyclization of aryl propargyl ethers with Au, Ag, or Pt catalyst.[4f−i] An InI_3-catalyzed cyclization of aryl propargyl ethers[4j] and a AgOTf-catalyzed cycloisomerization of cyclopropyl(*o*-hydroxyphenyl)-carbinols[4k] into 2*H*-benzopyrans were also reported. As transition-metal-free conditions, an IPy_2BF_4 and HBF_4 system in dichloromethane[5] and an I_2, ICl, or PhSeBr system in nitromethane[6] were used for the electrophilic iodocyclization of aryl propargyl ethers to form 3-iodo-2*H*-benzopyrans.

It is well-known that diaryliodonium salts are efficient reagents for the *O*-arylation of phenols, alcohols, carboxylic acids, and oximes under transition-metal-free conditions.[7] We previously reported the *O*-arylation of various phenols using diaryliodonium salts.[7l] Here, as part of our study of diaryliodonium salts for organic synthesis, we report a one-pot preparation of 3-iodo-2*H*-benzopyrans through the reaction of 3-aryl-2-propyn-1-ols and 3-alkyl-2-propyn-1-ols with diaryliodoniums triflates and *t*-BuONa followed by the reaction with *N*-iodosuccinimide (NIS) and $BF_3{\cdot}OEt_2$ under transition-metal-free conditions.

First, the *O*-arylation of 3-phenyl-2-propyn-1-ol **1a** with diphenyliodonium triflate **A** in the presence of bases, such as NaH, *t*-BuONa, *t*-BuOK. *t*-BuOLi, K_2CO_3, and Cs_2CO_3 in DMF, THF, benzene, toluene, and acetonitrile, was carried out as shown in Table 1. Initial screening experiments revealed that

Table 1. *O*-Phenylation of 3-Phenyl-2-propyn-1-ol



1a 2Aa

entry	base	solvent	temp (°C)	time (h)	yield (%)
1	NaH	DMF	0 to 80	7	40
2	*t*-BuONa	DMF	0 to 60	5	70
3	*t*-BuONa	THF	0 to reflux	5	46
4	*t*-BuONa	benzene	0 to 60	25	79
5	*t*-BuONa	toluene	0 to 60	23	72
6	*t*-BuOK	benzene	0 to 60	2	78
7	*t*-BuOLi	benzene	0 to 60	7	39
8[a]	K_2CO_3	toluene	0 to 60	15	39
9	K_2CO_3	CH_3CN	0 to 60	5	3
10	Cs_2CO_3	benene	0 to 60	5	79
11	*t*-BuONa	benzene/DCE (1:1)	0 to 60	3	83
12[b]	*t*-BuONa	benzene/DCE (1:1)	0 to 60	3	89
13[c]	*t*-BuONa	benzene/DCE (1:1)	0 to 60	3	87

[a]18-Crown-6 (10 mol %) was added. [b]$MgSO_4$ (0.2 equiv) was added. [c]$MgSO_4$ (0.5 equiv) was added.

use of *t*-BuONa as the base in DMF, THF, benzene, or toluene gave phenyl 3-phenyl-2-propynyl ether **2Aa** in moderate to good yields (Table 1, entries 1−10). Optimization of base in benzene was performed, and it was found that *t*-BuONa and Cs_2CO_3 in benzene were the best choices (Table 1, entries 4 and 10). Optimization of solvent revealed that the mixture of benzene and 1,2-dichloroethane (benzene/DCE = 1:1) was the most suitable (Table 1, entries 11−13), and the addition of $MgSO_4$ enhanced the reaction (Table 1, entries 12 and 13). *t*-BuONa is less expensive than deliquescent Cs_2CO_3. Finally, *t*-

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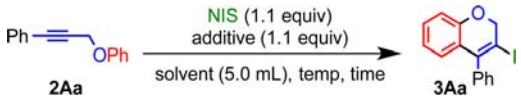
BuONa as a base and $MgSO_4$ were chosen for the present reaction. Next, the optimum conditions for the iodocyclization of phenyl 3-phenyl-2-propynyl ether **2Aa** with NIS were studied, as shown in Table 2. When phenyl 3-phenyl-2-propynyl ether **2Aa** was treated with NIS only in CH_3CN, 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** was not obtained at all (Table 2, entry 1). The addition of I_2 (1.0 equiv) induced iodocyclization to provide 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** in moderate yield (Table 2, entry 2). Use of 1,3-diiodo-5,5-dimethylhydantoin (DIH) instead of NIS did not enhance the reaction (Table 2, entry 3). Employing I_2 (3.0 equiv) and $NaHCO_3$ (3.0 equiv) in the absence of NIS gave 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** in 66% yield (Table 2, entry 4). Dichloromethane (DCM) was a better solvent than CH_3CN as a solvent (Table 2, entries 2 and 5), and treatment of phenyl 3-phenyl-2-propynyl ether **2Aa** with NIS (1.1 equiv) and I_2 (1.1 equiv) in CH_2Cl_2 at 0 °C gave 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** in 83% yield (Table 2, entry 6). The addition of $BF_3{\cdot}OEt_2$ instead of I_2 markedly improved the reactivity: treatment of phenyl 3-phenyl-2-propynyl ether **2Aa** with NIS (1.1 equiv) and $BF_3{\cdot}OEt_2$ (1.1 equiv) in DCM at 0 °C for 30 min gave 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** in 91% yield (Table 2, entry 7). On the other hand, the addition of excess NIS (2.0 equiv) and $BF_3{\cdot}OEt_2$ (2.0 equiv) reduced the yield of compound **3Aa** (Table 2, entry 8). On the basis of the results in Tables 1 and 2, a one-pot transformation of 3-phenyl-2-propyn-1-ol **1a** into 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** was carried out. 3-Phenyl-2-propyn-1-ol **1a** was treated with diphenyliodonium triflate **A** in the presence of *t*-BuONa and $MgSO_4$ in a mixture of benzene and DCE (1:1) at 60 °C for 3 h, and this was followed by the reaction with NIS (1.1 equiv) and $BF_3{\cdot}OEt_2$ (1.1 equiv) under various reaction conditions to give 3-iodo-4-phenyl-2*H*-benzopyran **3Aa**. It was found that evaporation prior to the second reaction step was not necessary for this reaction. Consequently, the addition of DCM (3 mL) prior to the second reaction step was the most effective. The use of 0.5 equiv of $BF_3{\cdot}OEt_2$ afforded low reactivity, and BCl_3, BBr_3, and $B(C_6F_5)_3$ also showed low reactivity, particularly BBr_3 (see the Supporting Information). Thus, it was clarified that the treatment of 3-phenyl-2-propyn-1-ol **1a** with *t*-BuONa (1.1 equiv) and diphenyliodonium triflate **A** (1.1 equiv) in the presence of $MgSO_4$ in a mixture of benzene and DCE (1:1) at 60 °C for 3 h, followed by the reaction with NIS (1.1 equiv) and $BF_3{\cdot}OEt_2$ (1.1 equiv) at 0 °C for 1 h, together with the addition of DCM (3.0 mL) was the best to give 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** in 80% yield, as shown in Scheme 1. A gram-scale preparation of 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** from 3-phenyl-2-propyn-1-ol **1a** (8 mmol) was also successfully carried out in 82% yield, as shown in Scheme 1. Using the optimum reaction conditions, various 3-aryl-2-propyn-1-ols **1b–k**, bearing *p*-fluorophenyl, *p*-chlorophenyl, *p*-methylphenyl, *m*-methylphenyl, *o*-methylphenyl, *p*-biphenyl, *p*-methoxyphenyl, *p*-benzyloxyphenyl, 1-naphthyl, and 2-naphthyl groups at the 3-position, were treated with diphenyliodonium triflate **A** in the presence of *t*-BuONa and $MgSO_4$ in a mixture of benzene and DCE (1:1) at 60 °C for 3 h, followed by the reaction with NIS and $BF_3{\cdot}OEt_2$ at 0 °C for 1 h, to give

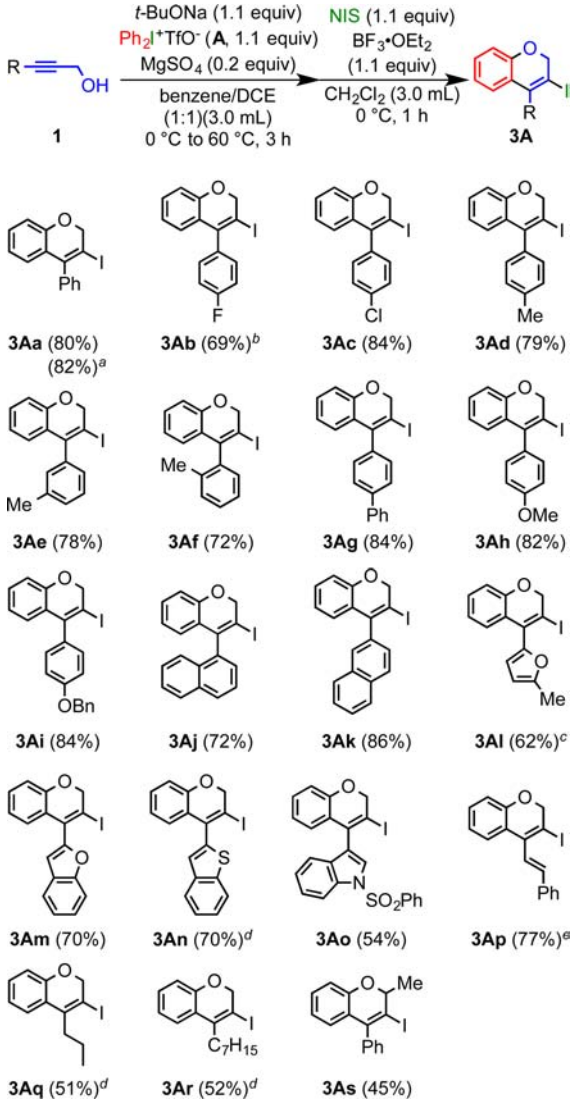
Table 2. Iodocyclization of Phenyl 3-Phenyl-2-propynyl Ether to 3-Iodo-4-phenyl-2*H*-benzopyran 3Aa



entry	additive	solvent	temp (°C)	time (h)	yield (%)
1		CH_3CN	0 to rt	7	0
2[a,b]	I_2	CH_3CN	0 to rt	3	55
3[b,c]	I_2	CH_3CN	0 to rt	3	35
4[d]	I_2	CH_3CN	rt	16	66
5[a,b]	I_2	DCM	0 to rt	3	63
6	I_2	DCM	0 to rt	3	83
7	$BF_3{\cdot}OEt_2$	DCM	0	0.5	91
8[a,e]	$BF_3{\cdot}OEt_2$	DCM	0	0.5	72

[a]NIS (2.0 equiv) was used. [b]Additive (1.0 equiv) was used. [c]DIH (1.0 equiv) was used instead of NIS. [d]I_2 (3.0 equiv) and $NaHCO_3$ (3.0 equiv) were used, without NIS. [e]Additive (2.0 equiv) was used.

Scheme 1. One-Pot Transformation of 3-Aryl-2-propyn-1-ols and 3-Alkyl-2-propyn-1-ols into 3-Iodo-2*H*-Benzopyrans 2 with Diphenyliodonium Triflate A



[a]Starting material (8.0 mmol) was used. [b]The second reaction step was carried out for 2 h. [c]The second reaction step was conducted at −20 °C for 30 min. [d]The second reaction step was conducted at −10 °C for 20 h. [e]The second reaction step was carried out for 30 min.

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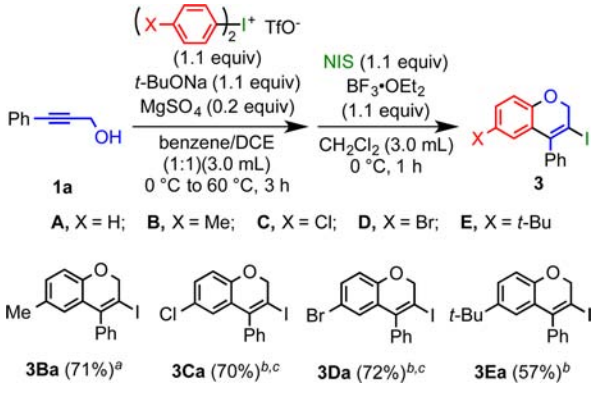
the corresponding 3-iodo-4-aryl-2*H*-benzopyrans **3Ab−Ak** in good yields, as shown in Scheme 1. Thus, 3-aryl-2-propyn-1-ols **1** bearing an electron-withdrawing group or an electron-donating group on the aromatic ring gave the corresponding 3-iodo-4-aryl-2*H*-benzopyrans **3** in good yields. The same treatment of 3-aryl-2-propyn-1-ols **1l−o** bearing heteroaromatic groups, such as 5-methyl-2-furyl, 2-benzofuryl, 2-benzothienyl, and 1-(benzenesulfonyl)-3-indolyl groups at the 3-position of the 3-aryl-2-propyn-1-ols, also gave the corresponding 3-iodo-4-aryl-2*H*-benzopyrans **3Al−Ao** in good to moderate yields, respectively. In addition, the same treatment of 3-styryl-2-propyn-1-ol **1p** and 3-alkyl-2-propyn-1-ols, such as 2-hexyn-1-ol **1q** and 2-decyn-1-ol **1r**, also gave 4-styryl- and 4-alkyl-3-iodo-2*H*-benzopyrans **3Ap−Ar** in good to moderate yields, respectively. In total, the yields of compounds **3** with 3-alkyl-2-propyn-1-ols were lower than those with 3-aryl-2-propyn-1-ols. We propose two reasons for this: one is that the *O*-phenylation of 3-alkyl-2-propyn-1-ols **1** with diphenyliodonium triflate **A** and *t*-BuONa does not proceed as smoothly as that of 3-aryl-2-propyn-1-ols **1**, and the other is that 4-alkyl-3-iodo-2*H*-benzopyrans **3Aq** and **3Ar** are not as stable as 4-aryl-3-iodo-2*H*-benzopyrans **3Aa−Ao** at room temperature. The same treatment of 4-phenyl-3-butyn-2-ol **1s**, a secondary propargyl alcohol, gave 3-iodo-2-methyl-4-phenyl-2*H*-benzopyran **3As** in moderate yield.

Then, the substituent effect on the *O*-arylation of 3-phenyl-2-propyn-1-ol **1a** with diaryliodonium salts, such as di(*p*-methylphenyl)iodonium triflate **B**, di(*p*-chlorophenyl)-iodonium triflate **C**, di(*p*-bromophenyl)iodonium triflate **D**, and di(*p*-*tert*-butylphenyl)iodonium triflate **E**, in the presence of *t*-BuONa and $MgSO_4$ in a mixture of benzene and DCE (1:1) at 60 °C for 3 h followed by the reaction with NIS and $BF_3{\cdot}OEt_2$ at 0 °C was studied. 6-Methyl-3-iodo-4-phenyl-2*H*-benzopyran **3Ba**, 6-chloro-3-iodo-4-phenyl-2*H*-benzopyran **3Ca**, and 6-bromo-3-iodo-4-phenyl-2*H*-benzopyran **3Da** were produced in good yields, as shown in Scheme 2, whereas the yield of 6-*tert*-butyl-3-iodo-4-phenyl-2*H*-benzopyran **3Ea** was moderate.

The structure of 6-bromo-3-iodo-4-phenyl-2*H*-benzopyran **3Da** was determined by X-ray crystallographic analysis (see the Supporting Information).

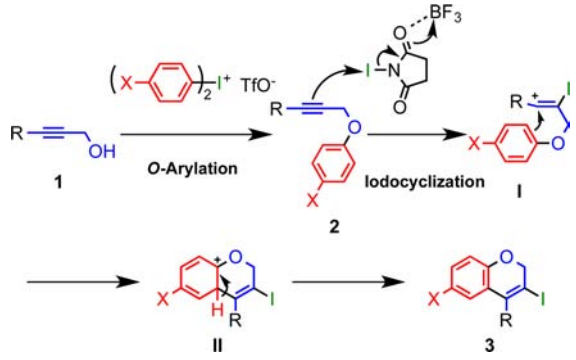
Scheme 2. One-Pot Transformation of 3-Phenyl-2-propyn-1-ol 1a into 3-Iodo-4-Phenyl-2*H*-Benzopyrans 3 with Diaryliodonium Triflates B−E



[a]The second reaction step was carried out for 2 h. [b]NIS (1.3 equiv) and $BF_3{\cdot}OEt_2$ (1.3 equiv) were used. [c]NIS (0.3 equiv) and $BF_3{\cdot}OEt_2$ (0.3 equiv) were added after the second reaction step.

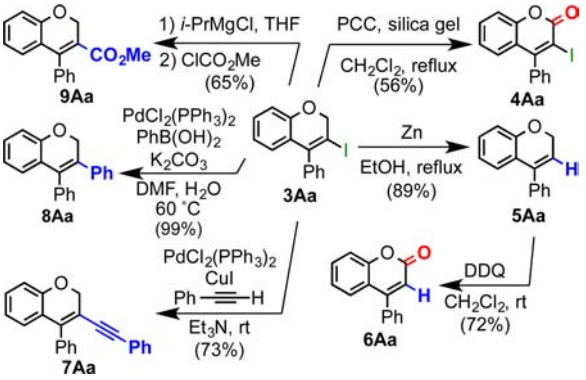
The plausible reaction mechanism for the present iodocyclization is shown in Scheme 3. Thus, BF_3 promotes NIS-iodocyclization of aryl 3-aryl(alkyl)-2-propynyl ether **2** to 3-iodo-4-aryl(alkyl)-2*H*-benzopyran **3** through the intermediates **I** and **II**.

Scheme 3. Plausible Reaction Mechanism for Iodocyclization



Finally, the functional group transformation of 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** was carried out, as shown in Scheme 4, as chromenes and coumarins are important units for pharmaceuticals and biologically active compounds.[3]

Scheme 4. Derivatization of 3-Iodo-4-Phenyl-2*H*-1-Benzopyran



Oxidation of 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** into 3-iodo-4-phenyl-2*H*-1-benzopyran-2-one **4Aa** was carried out in 56% yield by the reaction with PCC in DCM. Reduction of 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** into 4-phenyl-2*H*-benzopyran **5Aa** was carried out in 89% yield by treatment with Zn in ethanol. Oxidation of 4-phenyl-2*H*-benzopyran **5Aa** by DDQ gave γ-phenylcoumarin **6Aa** in 72% yield. Then, the Sonogashira coupling reaction of 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** with phenylacetylene and the Suzuki−Miyaura coupling reaction of 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** with phenylboronic acid and $PdCl_2(PPh_3)_2$ provided the corresponding coupling products **7Aa** and **8Aa** in 73% and 99% yields, respectively. Moreover, treatment of 3-iodo-4-phenyl-2*H*-benzopyran **3Aa** with *i*-PrMgCl at −78 °C followed by the reaction with $ClCO_2Me$ provided 3-(methoxycarbonyl)-4-phenyl-2*H*-benzopyran **9Aa** in 65% yield.

In conclusion, 3-aryl-2-propyn-1-ols and 3-alkyl-2-propyn-1-ols were treated with diaryliodonium triflates in the presence of *t*-BuONa followed by a reaction with NIS and $BF_3{\cdot}OEt_2$ to give

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3-iodo-4-aryl(alkyl)-2*H*-benzopyrans in good to moderate yields in one pot under mild and transition-metal-free conditions, and the products can be functionalized by oxidation or C−C bond-coupling reactions. The present one-pot method would be useful for the preparation of various 3-iodo-4-aryl-2*H*-benzopyrans and their derivatives.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b03651.

Experimental details, characterization data by IR, H NMR, and C NMR of all products **3** (PDF)
X-ray analysis of **3Da** (CIF)

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Notes

The authors declare no competing financial interest.

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