

# **Oxidative Cycloaddition of Aldoximes with Maleimides using** Catalytic Hydroxy(aryl)iodonium Species

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Abstract: A mild catalytic procedure for the efficient oxidative cyclization of aldoximes with maleimides mediated by hypervalent iodine(III) active species has been developed. This catalytic cyclization affords the corresponding pyrrolo-isoxazole products in generally good yields. The catalytic cycle involves active hydroxy(aryl)iodonium species generated in situ from 2-iodobenzoic acid as precatalyst and *m*-chloroperoxybenzoic acid (*m*-CPBA) as terminal oxidant in the presence of trifluoromethanesulfonic acid. The presence of active hydroxy-(aryl)iodonium species in this reaction has been confirmed by ESI-mass spectrometry and <sup>1</sup>H NMR spectroscopy.

Keywords: cyclization; heterocycles; iodine; iodonium species; oxidation; pyrrolo-isoxazoles

The oxidative cyclization of aldoximes and alkenes or alkynes is an important chemical transformation leading to heterocyclic compounds with oxygen and nitrogen in the ring, which are commonly found in biologically active compounds, medicinal synthons and natural products.<sup>[1]</sup> Among the known procedures for the oxidative cyclization of aldoximes with alkenes or alkynes, particularly important are the procedures utilizing stoichiometric amounts of organohypervalent iodine(III) reagents,<sup>[2-7]</sup> such as (diacetoxyiodo)benzene,<sup>[3]</sup> iodosylbenzene,<sup>[4]</sup> [hydroxy(tosyloxy)]iodobenzene,<sup>[5]</sup> and [bis(trifluoroacetoxy)iodo]benzene.<sup>[6]</sup> Previously, our group has first found that catalytic hypervalent iodine(III) species, generated in situ from iodoarenes and Oxone, could promote the oxidative cyclization of aldoximes with alkenes or alkynes.<sup>[7a]</sup> In a later work, Yan and co-workers have reported the catalytic hypervalent iodine(III)-mediated cyclization of aldoximes with alkenes or alkynes under similar conditions.<sup>[7b,c]</sup> We have recently reported a novel hypervalent iodine reagent, IBA-OTf, which was prepared from 2-iodosylbenzoic acid and trifluoromethanesulfonic acid and could be used as a stoichiometric oxidant for the oxidative cyclization of aldoximes with nitriles.<sup>[8]</sup> However, to the best of our knowledge, the cyclization reaction of aldoximes with maleimides using hypervalent iodine(III) compounds has not been previously reported.

Herein, we report the first catalytic hypervalent iodine(III)-mediated oxidative cyclization reaction of aldoximes with maleimides using hydroxy(aryl)iodonium active species generated in situ from 2-iodobenzoic acid and *m*-CPBA in the presence of trifluoromethanesulfonic acid. The products of this reaction, bicyclic pyrrolo-isoxazoles, belong to an important structural class, which is commonly found in bioactive compounds and natural products.<sup>[1b,9]</sup> Several research groups have previously reported the synthesis of pyrrolo-isoxazoles from aldoximes and maleimides in two steps using various chlorinating reagents,<sup>[10]</sup> such as chloramine-T,<sup>[10a,b]</sup> N-chlorosuccinimide (NCS),<sup>[10c-e]</sup> N-tert-butyl-N-chlorocyanamide,<sup>[10f]</sup> and sodium hypochlorite (NaOCl).<sup>[10g-i]</sup> Minakata and co-workers have reported a procedure for the preparation of pyrroloisoxazoles from aldoximes with N-phenylmaleimide using the stoichiometric hypoiodite species generated in situ from the NaI-t-BuOCl combination.[11]

In the initial experiments, we investigated the oxidative cyclization of aldoximes with maleimides using stoichiometric amounts of a hypervalent iodine(III) reagent (IBA-TfOH 3). We have found that the reaction of benzaldoxime 1a (1 equiv.), maleimide 2a (1.5 to 5 equiv.), and reagent 3 (1.2 equiv.) in dichlorome-

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**Scheme 1.** Oxidative cyclization of benzaldoxime and maleimide using IBA-TfOH.

thane at room temperature afforded pyrrolo-isoxazole 4a in moderate to high yields (Scheme 1). Motivated by these results, we then attempted to develop a catalytic cyclization reaction of aldoximes with maleimides using *in situ* generated hypervalent iodine(III) species. In a search for optimized catalytic conditions, we investigated reactions of benzaldoxime 1a (1 equiv.) and maleimide 2 (5 equiv.) with m-CPBA (1.5 equiv.) as a terminal oxidant in the presence of trifluoromethanesulfonic acid (TfOH) using catalytic amounts of different aromatic iodides 5 as precatalysts in various solvents (Table 1; for additional details see Table S1 in the Supporting Information). In the absence of aryl iodide, the formation of pyrrolo-isoxazole 4a was not observed (Table 1, entry 1). Screening of various aryl iodides has indicated that 2-iodobenzoic acid 5a and 2-iodo-5-methylbenzoic acid 5c are the most effective precatalysts in this catalytic reaction (entries 2-8). Since 2-iodobenzoic acid **5a** is a readily available commercial product, we decided to use it as a precatalyst in our following studies. Out of several solvents tested, dichloromethane was found to be the best solvent in this reaction (entries 3, 9-13). Decreasing the amount of 2-iodobenzoic acid 5a from 10 to 5 mol% leads to decreased yields of the product 4a (entry 14). In the presence of catalytic amounts of TfOH or in the absence of TfOH, the product yield was also lower (entries 15 and 16). Warming the reaction mixture to 40°C and shortening the reaction time to 4 h did not improve the yields (entries 17 and 18).

Using the optimized reaction conditions and 2-iodobenzoic acid **5a** as the precatalyst, we have investigated the conversion of various substituted aldoximes **1** to the respective pyrrolo-isoxazole derivatives (Table 2). In general, the reactions of substituted aromatic aldoximes **1** with electron-withdrawing or electron-donating substituents in the aromatic ring and maleimide **2a** gave the corresponding products **4a–j** in good yields. In the reaction of sterically hindered *ortho*-substituted aldoximes with maleimide, the respective products **4e** and **4f** were also obtained in good yields. However, the reaction of the tolyl deriva**Table 1.** Optimization of the catalytic cyclization of benzaldoxime 1a with maleimide 2.<sup>[a]</sup>



Entry	Solvent	Aryl iodide 5 (equiv.)	TfOH (equiv.)	<b>4a</b> [%] <sup>[b]</sup>
1	CH <sub>2</sub> Cl <sub>2</sub>	none	1.2	none <sup>[c]</sup>
2	CH <sub>2</sub> Cl <sub>2</sub>	<b>5a</b> (0.1)	1.2	100 (85)
3	CH <sub>2</sub> Cl <sub>2</sub>	<b>5b</b> (0.1)	1.2	75 <sup>[c]</sup>
4	CH <sub>2</sub> Cl <sub>2</sub>	<b>5c</b> $(0.1)$	1.2	100
5	CH <sub>2</sub> Cl <sub>2</sub>	5d (0.1)	1.2	78 <sup>[c]</sup>
6	CH <sub>2</sub> Cl <sub>2</sub>	<b>5e</b> (0.1)	1.2	85 <sup>[c]</sup>
7	$CH_2Cl_2$	<b>5f</b> $(0.1)$	1.2	95 <sup>[c]</sup>
8	$CH_2Cl_2$	<b>5g</b> (0.1)	1.2	95 <sup>[c]</sup>
9	$(CH_2CI)_2$	<b>5a</b> (0.1)	1.2	96 (82)
10	heptane	<b>5a</b> (0.1)	1.2	66 <sup>[c]</sup>
11	methanol	<b>5a</b> (0.1)	1.2	_[c,d]
12	CHCl <sub>3</sub>	<b>5a</b> (0.1)	1.2	95 <sup>[c]</sup>
13	EtOAc	<b>5a</b> (0.1)	1.2	95 <sup>[c]</sup>
14	CH <sub>2</sub> Cl <sub>2</sub>	<b>5a</b> (0.05)	1.2	86 <sup>[c]</sup>
15	CH <sub>2</sub> Cl <sub>2</sub>	<b>5a</b> (0.1)	0.6	93 <sup>[c]</sup>
16	CH <sub>2</sub> Cl <sub>2</sub>	<b>5a</b> (0.1)	none	72
17 <sup>[e]</sup>	CH <sub>2</sub> Cl <sub>2</sub>	<b>5a</b> (0.1)	1.2	90 <sup>[c]</sup>
18 <sup>[e,f]</sup>	$CH_2Cl_2$	<b>5a</b> (0.1)	1.2	71 <sup>[c]</sup>

 <sup>[a]</sup> Reaction conditions: benzaldoxime 1a (1 equiv.), maleimide 2a (5 equiv.), m-CPBA (1.5 equiv.), aryl iodide 5 (0-0.1 equiv.) and TfOH (0-1.2 equiv.) in various solvent at room temperature for 24 h.

<sup>[b]</sup> Yields of product **4a** determined from <sup>1</sup>H NMR spectra of reaction mixtures are shown (numbers in parentheses show isolated yields of **4a**).

- <sup>[c]</sup> Benzaldoxime **1a** was recovered from the reaction mixture.
- <sup>[d]</sup> Trace amount of product **4a** was detected, but the yield could not be calculated.
- <sup>[e]</sup> At 40 °C.
- <sup>[f]</sup> After 4 h.

tive gave product **4b** only in a moderate yield, probably, due to a partial oxidation of the methyl group, or the oxidation of the electron-rich aromatic ring during the reaction. The reaction of an aliphatic aldoxime with maleimide afforded the respective product **4k** only in a relatively low yield. Under similar condi-

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Table 2. Catalytic hypervalent iodine-mediated oxidative cyclization of aldoximes 1 and maleimides 2.<sup>[a]</sup>

[a] Reaction conditions: benzaldoxime 1 (1 equiv.), maleimide 2 (5 equiv.), m-CPBA (1.5 equiv.), 2-iodobenzoic acid 5a (0.1 equiv.) and TfOH (1.2 equiv.) in dichloromethane at room temperature for 24 h. Isolated yields are shown in parentheses.

tions, the reaction of cinnamaldehyde oxime produced product 41 in 70% yield. The reaction of benzaldoxime 1a with N-methylmaleimide 2b under these catalytic conditions also gave the corresponding N-substituted pyrrolo-isoxazole 4m in good yield. Compared to the previously reported procedure of oxidative cycloaddition of aldoximes and N-phenyl-substituted maleimides using stoichiometric t-BuOI generated from the NaI-t-BuOCl combination, our catalytic procedure gives comparable or higher yields of pyrroloisoxazoles.<sup>[11]</sup> Moreover, our procedure allows us to synthesize the parent N-unsubstituted products (4a-41), which cannot be obtained by using stochiometric t-BuOI as the oxidant.<sup>[11]</sup> The structure of one of these products, pyrrolo-isoxazole 4c, was characterized by X-ray crystallography (see the Supporting Information for details).

In order to clarify the mechanism of this catalytic reaction, we have performed several control experiments (see the Supporting Information for details). Most likely, this reaction involves the hydroxy(aryl)iodonium species as active species generated from 2-iodobenzoic acid **5a** and *m*-CPBA in the presence of TfOH. We were able to confirm the presence of hydroxy(aryl)iodonium species in the reaction mixture by ESI-mass spectrometry and <sup>1</sup>H NMR spectroscopy (see the spectra in Sections 4 and 5 of the Supporting

Information). The presence of stoichiometric TfOH is essential in this reaction; the special blank experiments have demonstrated that in the absence of TfOH 2-iodobenzoic acid 5a reacts with m-CPBA to produce 2-iodosylbenzoic acid (IBA), which is then converted to IBA-OTf 3 by subsequent reaction with TfOH. Furthermore, the reaction of benzaldoxime 1a and maleimide 2a with stoichiometric hydroxy(aryl)iodonium triflate (IBA-OTf<sup>[8]</sup>) 3 afforded product 4a in high yield (Scheme 1). As expected, the reaction of maleimide 2a with the protected benzaldoxime, Omethylbenzaldoxime, under optimized conditions failed to produce product 4a. This result indicates that the ligand exchange between the aldoxime and the hydroxyl group of the activated hypervalent iodine(III) species is required in this catalytic reaction. In the reaction of acetophenone oxime with maleimide 2a under the same conditions, only a trace amount of product 4 was detected, and the starting material, acetophenone oxime, could be recovered from the reaction mixture. This result suggests that the oxidation step of aldoxime to nitrile oxide using activated iodine(III) species is necessary in this reaction.[3-7]

From these control experiments and based on the previously reported reactions of aldoximes using stoichiometric iodine(III) reagents, we propose a catalytic

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**Scheme 2.** Proposed mechanism for the oxidative cyclization of aldoxime and maleimide.

reaction mechanism shown in Scheme 2. The active iodine(III) species, hydroxy(aryl)iodonium salt **6**, which is generated from 2-iodobenzoic acid **5a** and *m*-CPBA in the presence of TfOH, oxidizes the aldoxime **1** via ligand exchange to give the corresponding nitrile oxide **7** and the reduced 2-iodobenzoic acid **5a**. The generated nitrile oxide reacts with maleimide **2** to afford pyrrolo-isoxazole **4**. Finally, the 2-iodobenzoic acid **5a** is reoxidized to continue the next catalytic cycle.

In conclusion, we have reported the first catalytic reaction for the synthesis of pyrrolo-isoxazolines in one step under mild conditions. In comparison to the previously reported procedure of oxidative cycloaddition of aldoximes and *N*-phenyl-substituted maleimides using stochiometric *t*-BuOI *in situ*, our catalytic procedure gives generally higher yields and allows us to synthesize the parent *N*-unsubstituted products. The reaction mechanism involves the hydroxy(aryl)iodonium species generated *in situ* from 2-iodobenzoic acid and *m*-CPBA in the presence of TfOH.

### **Experimental Section**

#### **Typical Procedure**

2-Iodobenzoic acid **5a** (6.2 mg, 0.025 mmol), maleimide **2a** (121 mg, 1.25 mmol), *m*CPBA (65 mg, 0.375 mmol), and trifluoromethanesulfonic acid (45 mg, 0.300 mmol) were added to a solution of aldoxime **1** (0.250 mmol) in dichloromethane (2 mL). The reaction mixture was stirred at room temperature for 24 h. After completion of the reaction, 5%

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aqueous  $Na_2S_2O_3$  (5 mL) and then saturated  $NaHCO_3$  (5 mL) were added, and the mixture was extracted with ethyl acetate. The organic phase was dried over anhydrous  $Na_2SO_4$  and concentrated under reduced pressure. Purification by column chromatography (hexane-ethyl acetate = 3:1 to 1:1) afforded analytically pure pyrrolo-isoxazole **4**.

CCDC 1470155 contains the supplementary crystallographic data for this paper (compound **4c**). These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data\_request/ cif.

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### UPDATES

6 Oxidative Cycloaddition of Aldoximes with Maleimides using Catalytic Hydroxy(aryl)iodonium Species

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