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Synthesis of arylbenziodoxoles using pseudocyclic benziodoxole triflate and arenes

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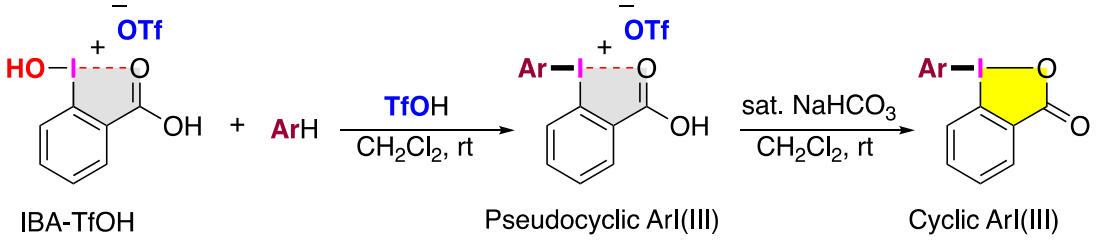
Abstract

An acid activated pseudocyclic hypervalent iodine reagent, 2-[hydroxy(trifluoromethanesufonyloxy)]-iodobenzoic acid, can easily react with various arenes in the presence of trifluoromethanesulfonic acid to produce pseudocyclic diaryliodonium triflate salts. This synthetic procedure proceeds under mild conditions to afford the respective iodonium salts in moderate to good yields. Several pseudocyclic diaryliodonium triflate salts structures have been confirmed by X-ray crystallography. Obtained products can be easily converted to cyclic hypervalent iodine(III) compounds, arylbenziodoxolones, in moderate to good yields under basic conditions.

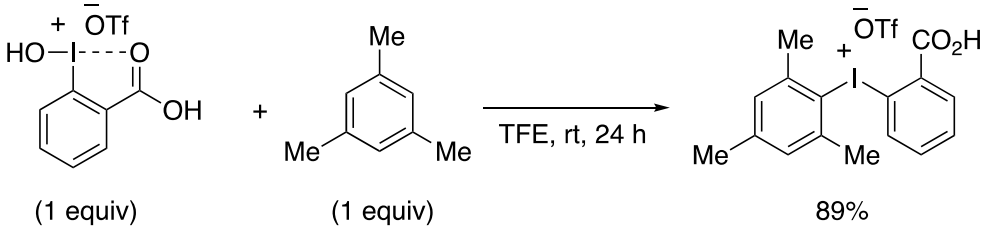


Keywords: Hypervalent iodine reagent, pseudocyclic diaryliodonium salts, arylbenziodoxolones, benziodoxole, cyclization

Introduction

Hypervalent iodine compounds have been used as efficient oxidizing reagents and employed in many conversion reactions in organic synthesis. Diaryliodonium salts are one of the most important classes of hypervalent iodine(III) reagents, and many attractive reactions using them have been reported.[1-11] In particular, most of these compounds are commonly used as the electrophilic aryl transfer reagents toward various organic substrates, resulting in the synthesis of various aryl derivatives.[12-17] Diaryliodonium salts are also used as benzyne precursor reagents, and many benzyne-mediated reactions utilizing these reagents have been reported.[18,19] While numerous reaction examples using diaryliodonium salts are well-known, a number of synthetic studies on them have also been reported.[12] A typical synthetic method of diaryiodonium salts is to combine common hypervalent iodine(III) reagents with aromatic compounds under appropriate conditions. As a developed synthetic example, the diaryliodonium compounds can be prepared from iodoarenes and aromatic compounds by treatment with suitable oxidants.

Recently, various pseudocyclic hypervalent iodine(III) compounds have been prepared and investigated.[20,21] Most of these compounds have higher stability and improved reactivity in comparison to their respective non-cyclic hypervalent iodine(III) reagents.[22-36] Previously, our group has reported the preparation, structure, and reactivity of 2-[hydroxy(trifluoromethanesulfonyloxy)]-iodobenzoic acid (IBA-TfOH), which is the pseudocylcic analogue compound of [hydroxy(trifluoromethanesulfonyloxy)iodo]benzene PhI(OH)OTf.[37-40] The novel IBA-TfOH reagent showed better reactivity as well as higher stability than PhI(OH)OTf. In our previous experiment, the reactivity of IBA-TfOH using mesitylene was investigated as an efficient approach to the pseudocyclic diaryliodonium triflate (Scheme 1).[37]



Scheme 1. Reaction of IBA-TfOH using mesitylene.

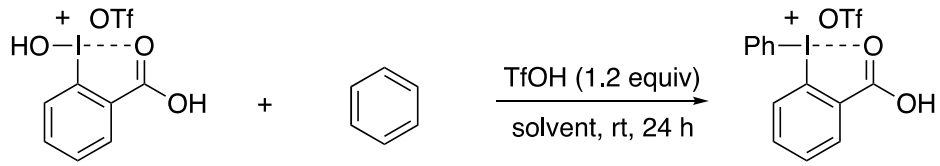
However, the reactivity towards other arenes using IBA-TfOH has not been reported. In the present study, we report the optimization and scope for the preparation of pseudocyclic diaryliodonium triflates from IBA-TfOH reagents with various arenes in the presence of trifluoromethanesulfonic acid. The obtained products can be further converted to the cyclic hypervalent iodine(III) compounds, arylbenziodoxolones, under mild basic conditions.[41,42]

Results and Discussion

We investigated the preparation of pseudocyclic diaryliodonium triflate **3a** using IBA-TfOH **1** and benzene **2a** in different solvents based on our previous results (Table 1). In the initial study, the reaction was performed in 2,2,2-trifluoroethanol (TFE) in the absence of TfOH under our previously reported condition resulting in no desired product **3a**, and the reagent **1** was recovered from the reaction mixture (entry 1). The reaction using

excess amount of benzene **2a** in TFE or dichloromethane also did not produce the desired product **3a** (entries 2 and 3). Addition of TfOH as an additive in dichloromethane dramatically improved the reaction to give the desired product **3a** in quantitative yield (entry 4). Screening of various solvents in the presence of TfOH has demonstrated that dichloromethane is the best solvent in this transformation reaction (entries 4-10). Decreasing the amount of benzene **2a** from 56.0 to 4.0 equivalents did not affect the yield of product **3a** (entries 4, 11-13). Further decreasing the amount of benzene **2a** led to a reduced yield of product **3a** (entries 14-16).

Table 1. Optimization of synthesis of pseudocyclic diaryliodonium triflate **3a**[a]



1 (1 equiv) **2a** **3a**

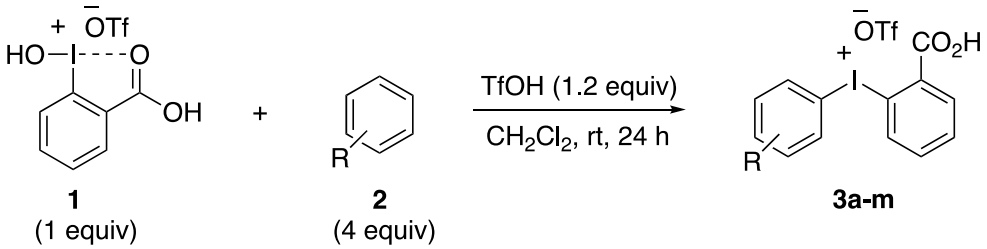
Entry	Benzene **2a** (equiv.)	Solvent	**3a** (%)[b]
1[c]	1	TFE	0[d]
2[c]	56	TFE	0[d]
3[c]	56	CH_2Cl_2	0[d]
4	56	CH_2Cl_2	>99
5	56	TFE	0[d]
6	56	MeCN	0[d]
7	56	AcOEt	0[d]
8	56	Heptane	0[d]
9	56	$ClCH_2CH_2Cl$	80
10	56	$CHCl_3$	82
11	10	CH_2Cl_2	>99
12	5	CH_2Cl_2	>99
13	4	CH_2Cl_2	>99
14	3	CH_2Cl_2	84
15	2	CH_2Cl_2	79
16	1	CH_2Cl_2	58

[a] Reaction conditions: IBA-TfOH **1** (1 equiv.), benzene **2a** (1–56 equiv.) and TfOH (0–1.2 equiv.) in various solvent (2.0 mL) at room temperature for 24 hours. [b] Yield of isolated product. [c] In the absence of TfOH. [d] IBA-TfOH **1** was recovered from the reaction mixture.

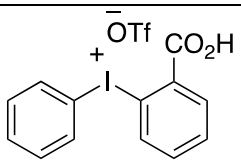
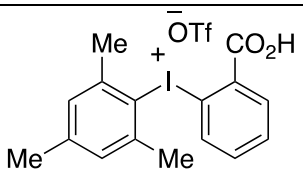
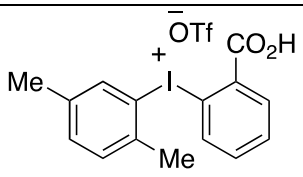
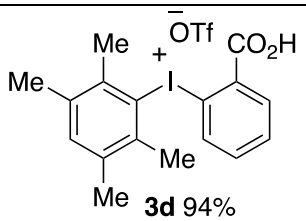
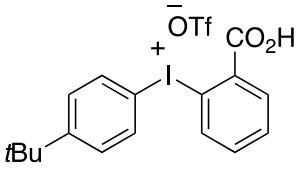
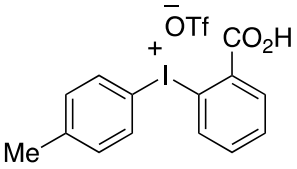
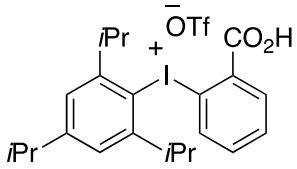
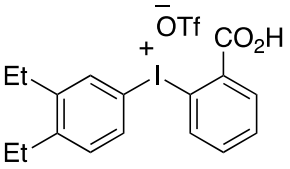
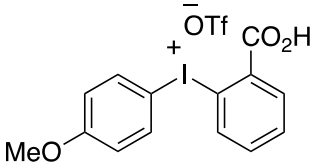
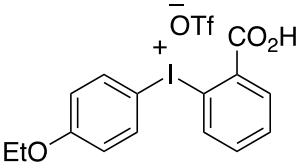
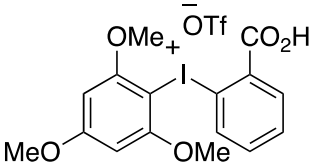
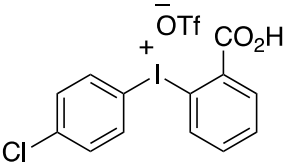
By using the optimal conditions, we have investigated the conversion of various substituted arenes **2** to the respective pseudocyclic diaryliodonium triflates **3**. In general, the reaction of arenes with either electron-donating or electron-withdrawing substituents gave the corresponding desired products **3a-l** in moderate to good yields. The reaction with sterically bulky *ortho*-substituted arenes also gave the pseudocyclic diaryliodonium triflates **3** in moderate to good yields (Table 2). Structures of pseudocyclic diaryliodonium triflates **3a**, **3f** and **3k** were established by X-ray crystallography (Figure 2). According to the X-ray data, the

pseudocyclic diaryliodonium structures with strong intramolecular interaction between iodine and oxygen atoms were observed.[20,30,31,37,43] The triflate oxygen atom was also involved in a weak intermolecular interaction with iodine atom resulting in a pseudo-square planar coordination of hypervalent iodine center.

Table 2. Preparation of pseudocyclic diaryliodonium triflates **3** using IBA-TfOH **1** with various arene **2**[a]



1 (1 equiv) + **2** (4 equiv) → **3a-m**

 **3a** 99%	 **3b** 100%	 **3c** 94%	 **3d** 94%
 **3e** 100%	 **3f** 97%	 **3g** 100%	 **3h** 93%
 **3i** 100%	 **3j** 97%	 **3k** 64%	 **3l** 77%

[a] Reaction conditions: IBA-TfOH **1** (1 equiv.), arene **2** (4 equiv.) and TfOH (1.2 equiv.) in dichloromethane at room temperature for 24 hours.

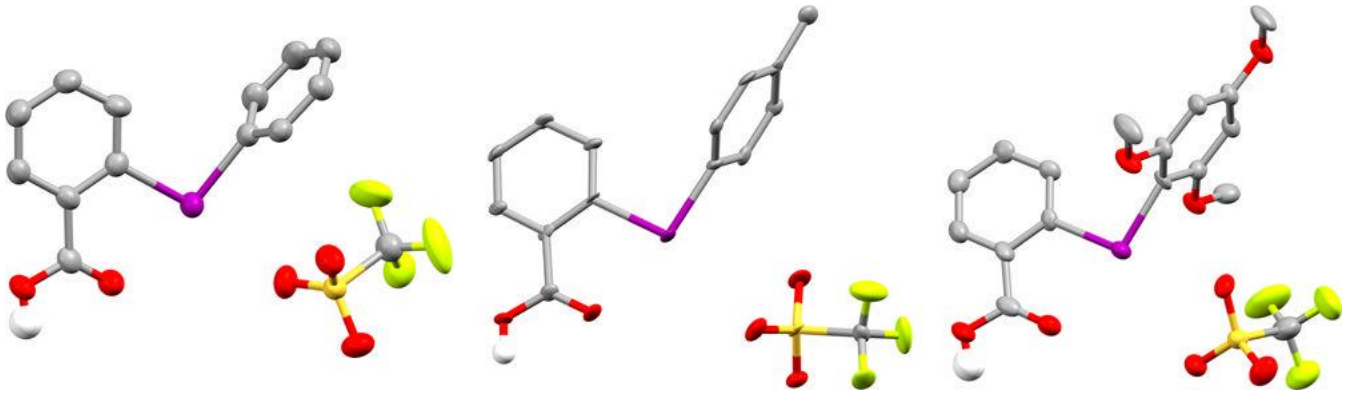
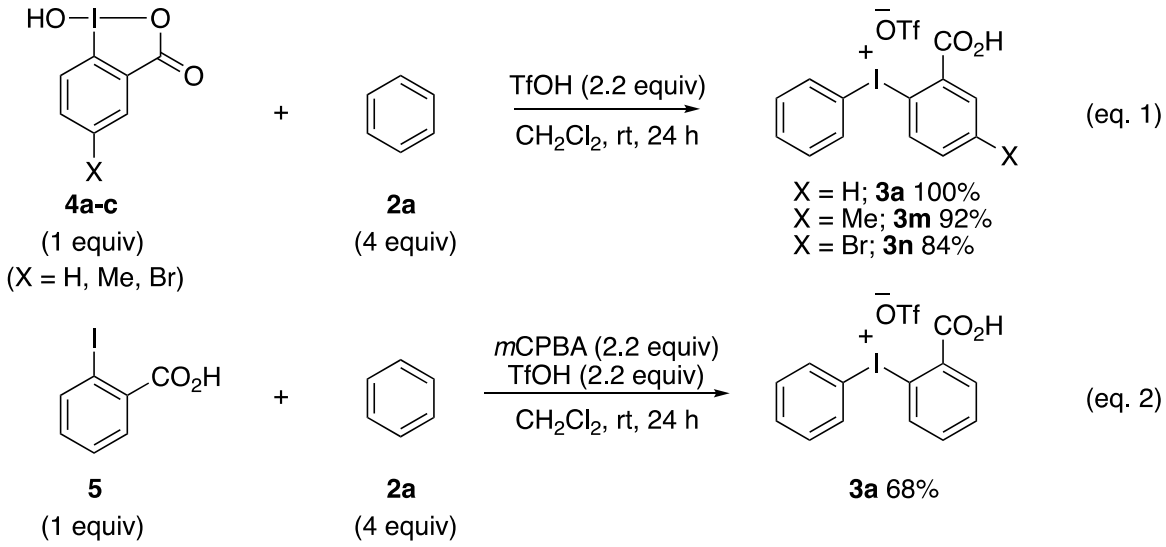


Figure 1. X-ray crystal structure of **3a** (CCDC 2021394), **3f** (CCDC 2021395) and **3k** (CCDC 2021393). Thermal Ellipsoids were drawn at the 50% probability level. Hydrogen atoms bonded to carbon atoms and water molecules in **3a** and **3f** were removed for clarity. Iodine-oxygen close contact distances in Å: **3a** I···O(BA) = 2.619(3), I···O(OTf) = 2.950(4); **3f** I···O(BA) = 2.623(7), I···O(OTf) = 3.150(7); **3k** I···O(BA) = 2.635(6), I···O(OTf) = 2.838(6).

Next, we investigated a one-pot preparation of pseudocyclic diaryliodonium triflate **3a** from benzene **2a** and IBA-TfOH **1** generated *in situ* from 2-iodosylbenzoic acid **4a** with trifluoromethanesulfonic acid (Scheme 2). This reaction gave the pseudocyclic diaryliodonium triflate **3a** in quantitative yield (eq. 1).[37] Compared to the previously reported method for preparation of pseudocyclic diaryliodonium triflates, our one-pot procedure was able to afford these products in comparable yields.[44] As expected, the reaction of substituted iodosylbenzoic acids **4b,c** under one-pot method resulted in the corresponding compounds **3m,n** in good yields (eq. 1).[45] Furthermore, we have found that **3a** could be prepared in moderate yield from 2-iodobenzoic acid and benzene using *m*-chloroperoxybenzoic acid in the presence of trifluoromethanesulfonic acid (eq. 2).[41]

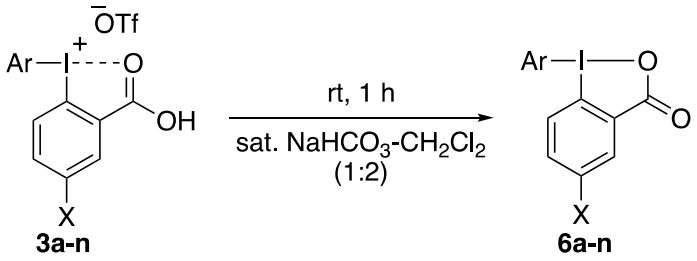


Scheme 2. One-pot synthesis of pseudocyclic diaryliodonium triflates.

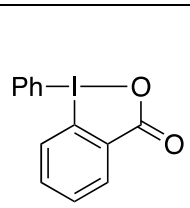
Finally, we have demonstrated that the obtained pseudocyclic diaryliodonium triflates could be converted to arylbenziodoxolones.[41,42,46,47] In particular, the treatment of **3a** with sodium bicarbonate could lead to phenylbenziodoxolone **6a** in quantitative yield. Following this strategy, we performed the reaction of

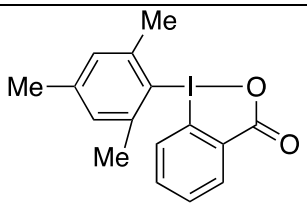
substituted pseudocyclic diaryliodonium triflates **3a-n** resulting in the desired arylbenziodoxolones **6a-n** in moderate to good yields (Table 3).

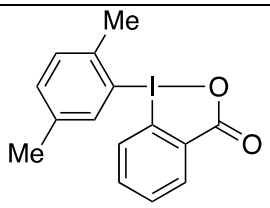
Table 3. Preparation of arylbenziodoxolones **6** from pseudocyclic diaryliodonium triflates **3**[a]

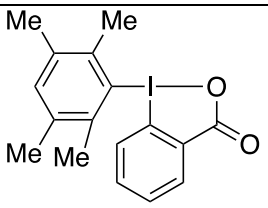


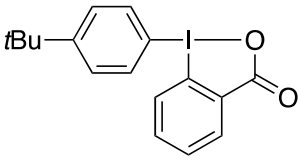
3a-n **6a-n**

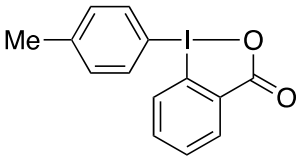

6a 100%

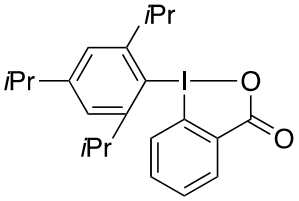

6b 89%

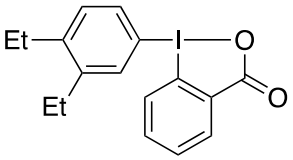

6c 100%

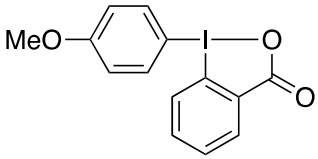

6d 82%

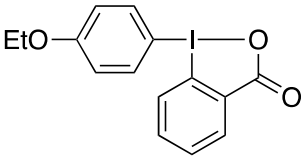

6e 95%

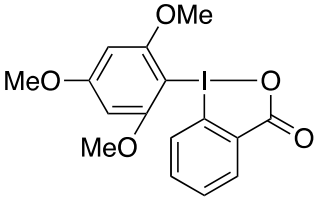

6f 69%[b]

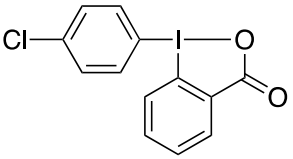

6g 62%

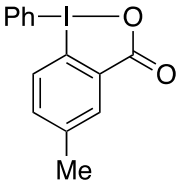

6h 66%

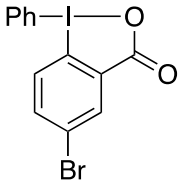

6i 99%


6j 86%


6k 89%


6l 28%


6m 78%


6n 55%

[a] Reaction conditions: Pseudocyclic diaryliodonium triflates **3** in saturated $NaHCO_3$ aqueous –dichloromethane (1:2) at room temperature for 1 hour. [b] The isomeric *ortho* and *para* products were detected.

Conclusions

In summary, we have prepared the pseudocyclic diaryliodonium triflates from IBA-TfOH and arenes in the presence of TfOH. The structure of several products **3** was confirmed by X-ray crystallography. The

combination of 2-iodosylbenzoic acid and trifluoromethanesulfonic acid, or 2-iodobenzoic acid and *m*-chloroperoxybenzoic acid in the presence of trifluoromethanesulfonic acid generates IBA-TfOH in-situ, which can be used for the preparation of pseudocyclic diaryliodonium triflates. Furthermore, the produced pseudocyclic diaryliodonium triflates can be easily converted to the respective arylbenziodoxolones under mild basic condition.

Experimental Section

General. All reactions were performed under dry argon atmosphere with flame-dried glassware. Dichloromethane was distilled from CaH_2 immediately prior to use. All commercial reagents were ACS reagent grade and used without further purification. Melting points were determined in an open capillary tube with a Mel-temp II melting point apparatus. Infrared spectra were recorded on a PerkinElmer 1600 series FT-IR spectrophotometer. NMR spectra were recorded on a Varian Inova 500, 300 MHz and Bruker 400 MHz NMR spectrometer (^{1}H NMR and ^{13}C NMR). X-ray crystal analysis was performed by Rigaku RAPID II XRD Image Plate using graphite-monochromated Cu or Mo Kα radiation ((λ = 1.54187 or 0.71073 Å) at 125 or 173 K. Please see the cif file for more detailed crystallography information. Hypervalent iodine reagents, IBA-TfOH **1**,[39], 2-Iodosylbenzoic acids **4a-b**[48], and **4c**[49] were prepared according to the reported procedure.

General procedure for preparation of pseudocyclic diaryliodonium triflates 3 using IBA-TfOH 1 and arenes 2. Arenes **2** (0.80 mmol), and trifluoromethanesulfonic acid (30 mg, 0.20 mmol) were added to a solution of IBA-TfOH **1** (83 mg, 0.20 mmol) in dichloromethane (1 mL). The reaction was stirred at room temperature for 24 hours. After completion of the reaction, the solvent was removed under reduced pressure and the solid product was washed with diethyl ether several times then dried in vacuum to give the pure compound **3**.

2-Carboxyphenyl(phenyl)iodonium triflate (3a).[44] Reaction of benzene **2a** (62 mg, 0.80 mmol) according to general procedure afforded 98 mg (99%) of product **3a** isolated as a white solid: mp 230.5-231.4 °C (lit.[44]; mp 198-220 °C); IR (KBr) cm^{-1} 3479, 3075, 3052, 1674, 1586, 1472, 1444, 1292, 1163, 1023, 911, 745; ^{1}H NMR (300 MHz, CD_3CN): δ 8.40 (dd, *J* 7.5 Hz, 1.5 Hz, 1H), 8.17 (d, *J* 8.4 Hz, 2H), 7.96 (t, *J* 7.5 Hz, 1H), 7.84-7.66 (m, 4H), 7.07 (d, *J* 8.4 Hz, 1H); ^{13}C NMR (75 MHz, CD_3CN): δ 169.0, 138.1, 137.5, 134.1, 133.3, 132.7, 131.7, 129.7, 126.3, 120.9 (q, $^1J_{CF}$ = 318.2 Hz), 114.3, 109.2; ^{19}F NMR (282 MHz, CD_3CN): δ -79.4; HRMS (APCI-positive ionization): calcd for $C_{13}H_{10}IO_2$ ([M-OTf]$^+$): 324.9725, found: 324.9741.

Single crystals of product **3a** suitable for X-ray crystallographic analysis were obtained by slow evaporation of acetonitrile solution. For details on crystal structure of compound **3a** see the CIF file in Supporting Information. Selected crystallographic data for **3a**: Monoclinic, P21n, a = 11.5802(10) Å, b = 9.0047(10)) Å, c = 16.6435(18), β= 95.433(7) Å, V = 1727.7(3) Å3, Z = 4, R (I>2.0/σ(I)) = 0.0469, Rw (all) = 0.0760, CCDC 2021394.

2-Carboxyphenyl(mesityl)iodonium triflate (3b).[37] Reaction of mesitylene **2b** (96 mg, 0.80 mmol) according to general procedure afforded 103 mg (100%) of product **3b** isolated as a white solid: mp 187.6-190.5 °C (lit.[37]; mp 185.3-186.6 °C); IR (KBr) cm^{-1} 3456, 3073, 3026, 1684, 1585, 1465, 1440, 1289, 1169, 1034, 990, 750; ^{1}H NMR (300 MHz, CD_3CN): δ 8.38 (d, *J* 7.5 Hz, 1H), 7.77 (t, *J* 7.5 Hz, 1H), 7.70-7.62 (m, 1H), 7.84-7.66 (m, 4H), 7.34 (s, 2H), 6.93 (t, *J* 8.1 Hz, 1H), 2.50 (s, 6H), 2.44 (s, 3H); HRMS (ESI-positive ionization): calcd for $C_{16}H_{16}IO_2$ ([M-OTf]$^+$): 367.0195, found: 367.0205.

2-Carboxyphenyl(2,5-dimethylphenyl)iodonium triflate (3c). Reaction of 1,4-dimethylbenzene **2c** (75 mg, 0.80 mmol) according to general procedure afforded 94 mg (94%) of product **3c** isolated as a white solid: mp 175.5-176.7 °C; IR (KBr) cm^{-1} 3504, 3078, 3052, 2928, 1673, 1586, 1492, 1470, 1442, 1280, 1162, 1029, 754; ^{1}H

NMR (300 MHz, CD_3CN): δ 8.38 (dd, *J* 7.5 Hz, 1.8 Hz, 1H), 7.96 (s, 1H), 7.77 (t, *J* 7.5 Hz, 1H), 7.72-7.59 (m, 3H), 6.98 (d, *J* 8.4 Hz, 1H), 2.48 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, CD_3CN): δ 169.5, 141.2, 141.1, 140.1, 138.1, 136.0, 134.0, 132.4, 132.3, 129.6, 127.3, 121.5 (q, $^1J_{CF}$ = 318.3 Hz), 114.3, 113.5, 24.5, 20.2; ^{19}F NMR (377 MHz, CD_3CN): δ -79.3; HRMS (APCI-positive ionization): calcd for $C_{15}H_{14}IO_2$ ([M-OTf]$^+$): 353.0038, found: 353.0056.

2-Carboxyphenyl(2,3,5,6-tetramethylphenyl)iodonium triflate (3d). Reaction of 1,2,4,5-tetramethylbenzene **2d** (107 mg, 0.80 mmol) according to general procedure afforded 100 mg (94%) of product **3d** isolated as a white solid: mp 165.4-166.2 °C; IR (KBr) cm^{-1} 3431, 3050, 2985, 2927, 1676, 1588, 1472, 1443, 1287, 1161, 1024, 743; ^{1}H NMR (300 MHz, CD_3CN): δ 8.38 (dd, *J* 7.5 Hz, 1.8 Hz, 1H), 7.80-7.77 (m, 1H), 7.64 (td, *J* 7.5 Hz, 1.5 Hz, 1H), 7.44 (s, 1H), 6.93 (d, *J* 8.1 Hz, 1H), 2.48 (s, 6H), 2.40 (s, 6H); ^{13}C NMR (75 MHz, CD_3CN): δ 168.7, 139.9, 137.5, 137.4, 133.6, 131.7, 128.7, 122.1, 120.1 (q, $^1J_{CF}$ = 321.1 Hz), 112.1, 110.0, 23.6, 20.6; ^{19}F NMR (282 MHz, CD_3CN): δ -79.3; HRMS (APCI-positive ionization): calcd for $C_{17}H_{18}IO_2$ ([M-OTf]$^+$): 381.0351, found: 381.0355.

2-Carboxyphenyl(4-*tert*-butylphenyl)iodonium triflate (3e). Reaction of *tert*-butylbenzene **2e** (107 mg, 0.80 mmol) according to general procedure afforded 106 mg (100%) of product **3e** isolated as a white solid: mp 172.1-175.0 °C; IR (KBr) cm^{-1} 3466, 3085, 2964, 2910, 1655, 1585, 1469, 1438, 1260, 1171, 1030, 755; ^{1}H NMR (500 MHz, CD_3CN): δ 8.37 (d, *J* 8.0 Hz, 1H), 8.02 (d, *J* 8.0 Hz, 1H), 7.78-7.71 (m, 3H), 7.02 (d, *J* 8.5 Hz, 1H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 169.1, 156.9, 137.8, 136.5, 132.9, 131.5, 130.0, 129.8, 121.2, (q, $^1J_{CF}$ = 320.0 Hz), 116.34, 109.9, 35.6, 31.3; ^{19}F NMR (377 MHz, DMSO-d_6): δ -77.7; HRMS (APCI-positive ionization): calcd for $C_{17}H_{18}IO_2$ ([M-OTf]$^+$): 381.0351, found: 381.0347.

2-Carboxyphenyl(4-methylphenyl)iodonium triflate (3f).[44] Reaction of toluene **2f** (74 mg, 0.80 mmol) according to general procedure afforded 95 mg (97%) of product **3f** isolated as a white solid: mp 202.1-203.6 °C; (lit.[44]; mp 193-194 °C); IR (KBr) cm^{-1} 3469, 3089, 2990, 1670, 1587, 1472, 1440, 1255, 1166, 1029, 754; ^{1}H NMR (300 MHz, CD_3CN): δ 8.37 (d, *J* 6.9 Hz, 1H), 7.98 (d, *J* 8.1 Hz, 2H), 7.82-7.64 (m, 2H), 7.53 (d, *J* 8.1 Hz, 2H), 7.05 (d, *J* 7.5 Hz, 1H), 2.53 (s, 3H); HRMS (APCI-positive ionization): calcd for $C_{14}H_{12}IO_2$ ([M-OTf]$^+$): 338.9882, found: 338.9885.

Single crystals of product **3f** suitable for X-ray crystallographic analysis were obtained by slow evaporation of acetonitrile solution. For details on crystal structure of compound **3f** see the CIF file in Supporting Information. Selected crystallographic data for **3f**: Monoclinic, P21n, a =11.2493(2) Å, b = 10.31970(10) Å, c = 15.9332(11) Å, β= 104.159(7) V = 1793.48(12) Å3, Z = 4, R (I>2.0/σ(I)) = 0.0658, Rw (all) = 0.0844, CCDC 2021395.

2-Carboxyphenyl(2,4,6-triisopropylphenyl)iodonium triflate (3g). Reaction of 1,3,5-triisopropylbenzene **2g** (163 mg, 0.80 mmol) according to general procedure afforded 120 mg (100%) of product **3g** isolated as a light brown solid: mp 174.0-175.1 °C; IR (KBr) cm^{-1} 3430, 3078, 2964, 2931, 2876, 1674, 1588, 1465, 1443, 1304, 1170, 1024, 750; ^{1}H NMR (300 MHz, CD_3CN): δ 8.38 (dd, *J* 7.5 Hz, 1.8 Hz, 1H), 8.20 (brs, 1H), 7.80-7.63 (m, 2H), 7.45 (s, 2H), 6.94 (d, *J* 7.8 Hz, 1H), 3.24-3.20 (m, 1H), 3.11-3.04 (m, 2H), 1.33 (d, *J* 6.9 Hz, 6H), 1.28-1.16 (m, 12H); ^{13}C NMR (100 MHz, CD_3CN): δ 169.4, 157.2, 154.3, 137.9, 134.0, 132.3, 129.4, 127.8, 125.8, 121.5 (q, $^1J_{CF}$ = 318.0 Hz), 117.0, 114.4, 39.4, 34.8, 23.5; ^{19}F NMR (282 MHz, CD_3CN): δ -79.3; HRMS (APCI-positive ionization): calcd for $C_{22}H_{28}IO_2$ ([M-OTf]$^+$): 451.1134, found: 451.1136.

2-Carboxyphenyl(3,4-diethylphenyl)iodonium triflate (3h). Reaction of 1,2-diethylbenzene **2h** (107 mg, 0.80 mmol) according to general procedure afforded 99 mg (93%) of product **3h** isolated as a white solid: mp 193.8-195.6 °C; IR (KBr) cm^{-1} 3462, 2974, 2943, 2880, 1674, 1586, 1473, 1439, 1292, 1176, 1025, 746; ^{1}H NMR (500 MHz, CD_3CN): δ 8.37 (dd, *J* 8.0 Hz, 1.8 Hz, 1H), 7.91 (d, *J* 2.3 Hz, 1H), 7.87 (dd, *J* 8.0 Hz, 2.3 Hz, 1H), 7.76 (t, *J* 8.0 Hz, 1H), 7.69 (t, *J* 8.0 Hz, 1H), 7.51 (d, *J* 8.0 Hz, 1H), 7.06 (d, *J* 8.0 Hz, 1H), 2.83 (q, *J* 7.5 Hz, 2H), 2.78 (q, *J* 7.5 Hz, 2H), 1.28 (t, *J* 7.5 Hz, 3H), 1.25 (t, *J* 7.5 Hz, 3H); ^{13}C NMR (100 MHz, CD_3CN): δ 169.5, 149.8, 148.0, 138.0, 137.9, 136.1, 133.8, 133.1, 132.2, 130.1, 126.9, 121.5 (q, $^1J_{CF}$ = 318.0 Hz), 115.0, 106.2, 25.9, 25.6, 14.7, 14.6; ^{19}F NMR

(377 MHz, CD_3CN): δ -79.3; HRMS (APCI-positive ionization): calcd for $C_{17}H_{18}IO_2$ ([M-OTf]$^+$): 381.0351, found: 381.0348.

2-Carboxyphenyl(4-methoxyphenyl)iodonium triflate (3i).[44] Reaction of methoxybenzene **2i** (87 mg, 0.80 mmol) according to general procedure afforded 101 mg (100%) of product **3i** isolated as a gray solid: mp 213.6-214.9 °C (lit.[44]; mp 195-206 °C); IR (KBr) cm^{-1} 3486, 3093, 2984, 2950, 2851, 1668, 1582, 1492, 1464, 1308, 1258, 1164, 1026, 756; ^{1}H NMR (400 MHz, CD_3CN): δ 8.36 (d, *J* 8.0 Hz, 1H), 8.01 (d, *J* 8.0 Hz, 1H), 7.76 (t, *J* 8.0 Hz, 1H), 7.70 (t, *J* 8.0 Hz, 1H), 7.22 (d, *J* 8.0 Hz, 1H), 7.06 (d, *J* 8.0 Hz, 1H), 3.94 (s, 3H); ^{13}C NMR (100 MHz, CD_3CN): δ 169.7, 164.8, 140.8, 137.9, 133.8, 132.2, 130.0, 127.0, 121.6 (q, $^1J_{CF}$ = 319.5 Hz), 115.5, 97.8, 56.4; HRMS (ESI-positive ionization): calcd for $C_{14}H_{12}IO_3$ ([M-OTf]$^+$): 354.9831, found: 354.9833.

2-Carboxyphenyl(4-ethoxyphenyl)iodonium triflate (3j). Reaction of ethoxybenzene **2j** (98 mg, 0.80 mmol) according to general procedure afforded 100 mg (97%) of product **3j** isolated as a gray solid: mp 214.8-216.0 °C; IR (KBr) cm^{-1} 3463, 3089, 2984, 2897, 1672, 1586, 1493, 1471, 1261, 1179, 1027, 748; ^{1}H NMR (400 MHz, CD_3CN): δ 8.36 (d, *J* 8.0 Hz, 1H), 7.99 (d, *J* 8.0 Hz, 2H), 7.76 (t, *J* 8.0 Hz, 1H), 7.70 (t, *J* 8.0 Hz, 1H), 7.19 (d, *J* 8.0 Hz, 2H), 7.07 (d, *J* 8.0 Hz, 1H), 4.20 (q, *J* 6.5 Hz, 2H), 1.44 (t, *J* 6.5 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 169.3, 162.7, 140.1, 136.7, 133.0, 131.5, 129.7, 129.3, 121.2 (q, $^1J_{CF}$ = 320.0 Hz), 118.8, 116.9, 101.5, 64.4, 14.5; ^{19}F NMR (377 MHz, CD_3CN): δ -79.4; HRMS (ESI-positive ionization): calcd for $C_{15}H_{14}IO_3$ ([M-OTf]$^+$): 368.9988, found: 368.9990.

2-Carboxyphenyl(2,4,6-trimethoxyphenyl)iodonium triflate (3k). Reaction of 1,3,5-trimethoxybenzene **2k** (135 mg, 0.80 mmol) according to general procedure afforded 72 mg (64%) of product **3k** isolated as a purple solid: mp 167.7 °C (decomp.); IR (KBr) cm^{-1} 3445, 3100, 2954, 2913, 1672, 1585, 1470, 1418, 1291, 1164, 1028, 750; ^{1}H NMR (400 MHz, CD_3CN): δ 8.28 (dd, *J* 7.6 Hz, 1.4 Hz, 1H), 7.69 (dd, *J* 7.6 Hz, 1.4 Hz, 1H), 7.66-7.59 (m, 1H), 7.04 (dd, *J* 8.4 Hz, 1.2 Hz, 1H), 6.43 (s, 2H), 3.91 (s, 3H), 3.81 (s, 6H); ^{13}C NMR (100 MHz, CD_3CN): δ 169.8, 169.3, 162.4, 137.9, 133.9, 132.0, 128.7, 127.3, 118.4 (q, $^1J_{CF}$ = 317.3 Hz), 113.7, 92.9, 79.7, 57.7, 56.9; ^{19}F NMR (377 MHz, CD_3CN): δ -79.4; HRMS (ESI-positive ionization): calcd for $C_{16}H_{16}IO_5$ ([M-OTf]$^+$): 415.0042, found: 415.0026.

Single crystals of product **3k** suitable for X-ray crystallographic analysis were obtained by slow evaporation of methylene chloride-ether solution. For details on crystal structure of compound **3k** see the CIF file in Supporting Information. Selected crystallographic data for **3k**: Triclinic, P-1, a = 8.1177(2) Å, b = 8.7075(2) Å, c = 14.9429(11) Å, α= 73.353(5), β= 89.458(6), γ= 83.352(6), V = 1004.85(9) Å^3, Z = 2, R (I>2.0/σ(I)) = 0.0438, Rw (all) = 0.0582, CCDC 2021393.

2-Carboxyphenyl(4-chlorophenyl)iodonium triflate (3l).[44] Reaction of chlorobenzene **2l** (90 mg, 0.80 mmol) according to general procedure afforded 78 mg (77%) of product **3l** isolated as a white solid: mp 136.7-138.4 °C (lit.[44]; mp 218-220 °C); IR (KBr) cm^{-1} 3511, 3084, 2880, 1661, 1613, 1472, 1442, 1257, 1171, 1093, 1030, 743; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.03 (d, *J* 8.0 Hz, 2H), 8.00-7.93 (m, 2H), 7.88 (d, *J* 8.0 Hz, 2H), 7.74-7.68 (m, 2H); ^{19}F NMR (377 MHz, CD_3CN): δ -77.7; HRMS (ESI-positive ionization): calcd for $C_{13}H_9{}^{35}ClIO_2$ ([M-OTf]$^+$): 358.9336, found: 358.9312.

One-pot preparation of pseudocyclic diaryliodonium triflates 3 using 2-iodosylbenzoic acid 4 and benzene 2a using TfOH. Benzene **2a** (62 mg, 0.80 mmol) and trifluoromethanesulfonic acid (66 mg, 0.44 mmol) was added to a solution of 2-iodosylbenzoic acids **4** (0.20 mmol) in dichloromethane (1 mL). The reaction was stirred at room temperature for 24 hours. After completion of the reaction, the solvent was removed under reduced pressure and the solid product was washed with diethyl ether several times then dried in vacuum to give the pure compound **3a, n, o**.

2-Carboxyphenyl(phenyl)iodonium triflate (3a). Reaction of 2-iodosylbenzoic acid **4a** (53 mg, 0.20 mmol) according to general procedure afforded 95 mg (100%) of product **3a** isolated as a white solid identical to the same from previous experiment.

4-Methyl-2-carboxyphenyl(phenyl)iodonium triflate (3m). Reaction of 4-methyl-2-iodosylbenzoic acid **4b** (56 mg, 0.20 mmol) according to general procedure afforded 96 mg (92%) of product **3m** isolated as a white solid: mp 149.3-152.6 °C; IR (KBr) cm^{-1} 3410, 3087, 3061, 2928, 1660, 1574, 1448, 1257, 1167, 1033, 744; ^{1}H NMR (400 MHz, CD_3OD): δ 8.27-8.20 (m, 3H), 7.92 (t, *J* 8.0 Hz, 1H), 7.73 (t, *J* 8.0 Hz, 1H), 7.73 (t, *J* 8.0 Hz, 2H), 7.75-7.49 (m, 1H), 6.88 (d, *J* 8.0 Hz, 1H), 2.46 (s, 3H); ^{13}C NMR (125 MHz, CD_3OD): δ 169.8, 142.5, 137.7, 137.2, 133.4, 133.4, 132.2, 129.1, 127.1, 120.4 (q, $^1J_{CF}$ = 317 Hz), 19.21; ^{19}F NMR (377 MHz, CD_3OD): δ -80.1; HRMS (ESI-positive ionization): calcd for $C_{14}H_{12}IO_2$ ([M-OTf]$^+$): 338.9882, found: 338.9900.

4-Bromo-2-carboxyphenyl(phenyl)iodonium triflate (3n). Reaction of 4-bromo-2-iodosylbenzoic acid **4c** (69 mg, 0.2 mmol) according to general procedure afforded 93 mg (84%) of product **3n** isolated as a white solid: mp 186.1-187.5 °C; IR (KBr) cm^{-1} 3463, 3080, 1663, 1620, 1553, 1445, 1256, 1172, 1024, 738; ^{1}H NMR (400 MHz, CD_3CN): δ 8.48 (d, *J* 2.2 Hz, 1H), 8.12 (d, *J* 8.4 Hz, 2H), 7.99-7.89 (m, 1H), 7.80 (dd, *J* 9.0 Hz, 2.2 Hz, 1H), 7.77-7.65 (m, 2H), 6.90 (d, *J* 9.0 Hz, 1H); ^{13}C NMR (100 MHz, CD_3CN): δ 168.5, 140.4, 138.7, 136.3, 134.8, 133.4, 131.9, 128.8, 126.3, 121.6 (q, $^1J_{CF}$ = 317 Hz), 113.5, 109.8; ^{19}F NMR (377 MHz, CD_3CN): δ -79.4; HRMS (ESI-positive ionization): calcd for $C_{13}H_9{}^{79}BrIO_2$ ([M-OTf]$^+$): 402.8831, found: 338.9885.

One-pot preparation of pseudocyclic diaryliodonium triflates 3a using 2-iodobenzoic acid 5 and benzene 2a using TfOH with *m*CPBA. Benzene **2a** (62 mg, 0.80 mmol), trifluoromethanesulfonic acid (66 mg, 0.44 mmol), and *m*CPBA (76 mg, 0.44 mmol) was added to a solution of 2-iodobenzoic acid **5** (50 mg, 0.20 mmol) in dichloromethane (1 mL). The reaction was stirred at room temperature for 24 hours. After completion of the reaction, the solvent was removed under reduced pressure and the solid product was washed with diethyl ether several times then dried in vacuum to give the pure compound **3a**; 64 mg (68%) isolated as a brown solid identical to the same from previous experiment.

General procedure for preparation of arylbenziodoxolones 6 from pseudocyclic diaryliodonium triflates 3. **Pseudocyclic diaryliodonium triflates 3** was added to dichloromethane (2 mL) and saturated $NaHCO_3$ (1 mL). The reaction was stirred at room temperature for 1 hour. After completion of the reaction, the solvent was removed under reduced pressure and the solid product was washed with diethyl ether several times then dried in vacuum to give the pure compound **6**.

1-Phenyl-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6a).[42] Reaction of **3a** (95 mg, 0.20 mmol) according to general procedure afforded 64 mg (100%) of product **6a** isolated as a white solid: mp 210.1-211.0 °C (lit.[42]; mp 221-222 °C); IR (KBr) cm^{-1} 3042, 1609, 1557, 1475, 1340, 738; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.40 (dd, *J* 7.5 Hz, 1.2 Hz, 1H), 8.02 (d, *J* 7.8 Hz, 2H), 7.77 (t, *J* 7.5 Hz, 1H), 7.65-7.50 (m, 3H), 7.43-7.32 (m, 1H), 6.74 (d, *J* 8.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.6, 137.2, 133.5, 133.4, 132.7, 132.6, 131.8, 130.7, 126.1, 115.6, 115.3; HRMS (ESI-positive ionization): calcd for $C_{13}H_{10}IO_2$ ([M+H]$^+$): 324.9725, found: 324.9736.

1-Mesityl-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6b).[42] Reaction of **3b** (103 mg, 0.20 mmol) according to general procedure afforded 65 mg (89%) of product **6b** isolated as a white solid: mp 222.4-223.1 °C (lit.[42]; mp 223-223.5 °C); IR (KBr) cm^{-1} 3058, 2974, 2948, 2920, 1616, 1558, 1441, 1034, 758; ^{1}H NMR (300 MHz, CD_3OD): δ 8.31 (d, *J* 7.8 Hz, 1H), 7.72-7.61 (m, 1H), 7.55-7.44 (m, 1H), 7.29 (s, 2H), 6.79 (d, *J* 8.1 Hz, 1H), 2.53 (s, 6H), 2.43 (s, 3H); HRMS (ESI-positive ionization): calcd for $C_{16}H_{16}IO_2$ ([M+H]$^+$): 367.0195, found: 367.0210.

1-(2,5-Dimethylphenyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6c).[45] Reaction of **3c** (100 mg, 0.20 mmol) according to general procedure afforded 70 mg (100%) of product **6c** isolated as a light yellow solid: mp 110.7-111.8 °C (lit.[45]; mp 214-214.5 °C); IR (KBr) cm^{-1} 3068, 2970, 2927, 1608, 1489, 1368, 1031, 750; ^{1}H NMR (500

MHz, CD_3OD): δ 8.46 (d, *J* 7.5 Hz, 1H), 7.72-7.65 (m, 1H), 7.56-7.45 (m, 1H), 7.43-7.38 (m, 2H), 7.37-7.29 (m, 1H), 6.63 (d, *J* 8.5 Hz, 1H), 2.40 (s, 3H), 2.43 (s, 3H); HRMS (APCI-positive ionization): calcd for $C_{15}H_{14}IO_2$ ($[M+H]^+$): 353.0038, found: 353.0032.

1-(2,3,5,6-Tetramethylphenyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6d).[42] Reaction of **3d** (96 mg, 0.18 mmol) according to general procedure afforded 56 mg (82%) of product **6d** isolated as a white solid: mp 218.0-220.4 °C (lit.[42]; mp 221.5-222.5 °C); IR (KBr) cm^{-1} 3060, 3008, 2963, 2941, 2920, 2850, 1598, 1554, 1466, 1346, 1006, 760; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.51 (dd, *J* 8.0 Hz, 1.5 Hz, 1H), 7.64-7.58 (m, 1H), 7.43-7.37 (m, 1H), 7.28-7.24 (m, 1H), 7.43-7.38 (m, 2H), 7.37-7.29 (m, 1H), 6.63 (d, *J* 8.5 Hz, 1H), 2.40 (s, 3H), 2.43 (s, 3H); HRMS (ESI-positive ionization): calcd for $C_{17}H_{18}IO_2$ ($[M+H]^+$): 381.0356, found: 381.0366.

1-(4-(*tert*-butyl)phenyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6e).[42] Reaction of **3e** (106 mg, 0.20 mmol) according to general procedure afforded 72 mg (95%) of product **6e** isolated as a white solid: mp 230.9-231.4 °C (lit.[42]; mp 233.5-234 °C); IR (KBr) cm^{-1} 3059, 2959, 2869, 1612, 1556, 1482, 1347, 1058, 748; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.48 (dd, *J* 7.8 Hz, 1.8 Hz, 1H), 7.88 (d, *J* 9.0 Hz, 2H), 7.64-7.57 (m, 3H), 7.47-7.41 (m, 1H), 6.82 (d, *J* 8.0 Hz, 1H), 1.40 (s, 9H); HRMS (ESI-positive ionization): calcd for $C_{17}H_{18}IO_2$ ($[M+H]^+$): 381.0356, found: 381.0366.

1-(*p*-Tolyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6f).[42] Reaction of **3f** (88 mg, 0.18 mmol) according to general procedure afforded 42 mg (69%) of product **6f** isolated as a white solid: mp 195.9-197.7 °C (lit.[42]; mp 217-218 °C); IR (KBr) cm^{-1} 3074, 2977, 2923, 1610, 1557, 1488, 1329, 747; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (d, *J* 8.0 Hz, 1H), 7.81 (d, *J* 8.0 Hz, 2H), 7.65-7.56 (m, 1H), 7.47-7.37 (m, 3H), 6.78 (d, *J* 8.0 Hz, 2H), 2.52 (s, 3H); HRMS (APCI-positive ionization): calcd for $C_{14}H_{12}IO_2$ ($[M+H]^+$): 338.9882, found: 338.9882.

1-(2,4,6-Triisopropylphenyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6g). Reaction of **3g** (120 mg, 0.20 mmol) according to general procedure afforded 56 mg (62%) of product **6g** isolated as a white solid: mp 197.9-198.8 °C; IR (KBr) cm^{-1} 3057, 2962, 2870, 1602, 1552, 1357, 745; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.51 (d, *J* 7.5 Hz, 1H), 7.61 (t, *J* 7.5 Hz, 1H), 7.46-7.38 (m, 1H), 7.25 (s, 2H), 6.76 (d, *J* 8.1 Hz, 1H), 3.19 (sept, *J* 6.9 Hz, 2H), 3.02 (sept, *J* 6.9 Hz, 1H), 1.31 (d, *J* 6.9 Hz, 12H), 1.16 (d, *J* 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.7, 155.0, 153.6, 134.2, 133.5, 133.1, 130.9, 125.4, 123.9, 119.3, 115.2, 37.9, 34.4, 25.1, 23.8; HRMS (APCI-positive ionization): calcd for $C_{22}H_{28}IO_2$ ($[M+H]^+$): 451.1134, found: 451.1112.

1-(3,4-Diethylphenyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6h). Reaction of **3h** (99 mg, 0.19 mmol) according to general procedure afforded 47 mg (66%) of product **6h** isolated as a white solid: mp 193.8-195.6 °C; IR (KBr) cm^{-1} 3064, 2966, 2936, 2880, 1611, 1558, 1331, 740; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.42 (d, *J* 7.5 Hz, 1H), 7.78-7.73 (m, 2H), 7.55 (t, *J* 7.0 Hz, 1H), 7.43-7.34 (m, 2H), 6.79 (d, *J* 7.0 Hz, 1H), 2.83-2.71 (m, 4H), 1.31 (t, *J* 7.5 Hz, 3H), 1.28 (t, *J* 7.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.3, 147.5, 146.3, 136.5, 134.6, 133.5, 132.9, 131.7, 130.8, 125.8, 115.5, 111.5, 25.6, 25.5, 14.9, 14.8; HRMS (APCI-positive ionization): calcd for $C_{17}H_{18}IO_2$ ($[M+H]^+$): 381.0351, found: 381.0352.

1-(4-Methoxyphenyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6i).[47] Reaction of **3i** (105 mg, 0.20 mmol) according to general procedure afforded 70 mg (99%) of product **6i** isolated as a white solid: mp 159.5-160.1 °C (lit.[47]; mp 212-214 °C); IR (KBr) cm^{-1} 3065, 2941, 2840, 1599, 1489, 1359, 1257, 748; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49-8.39 (m, 1H), 7.83 (d, *J* 8.0 Hz, 2H), 7.62-7.51 (m, 1H), 7.45-7.34 (m, 1H), 6.81-6.69 (m, 1H), 7.06 (d, *J* 8.0 Hz, 2H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.1, 139.0, 133.5, 132.9, 130.8, 125.7, 117.8, 115.9, 104.0, 55.7; HRMS (ESI-positive ionization): calcd for $C_{14}H_{12}IO_3$ ($[M+H]^+$): 354.9831, found: 354.9847.

1-(4-Ethoxyphenyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6j). Reaction of **3j** (100 mg, 0.19 mmol) according to general procedure afforded 60 mg (86%) of product **6j** isolated as a light brown solid: mp 218.9-219.8 °C; IR (KBr) cm^{-1} 3086, 2977, 2928, 1603, 1488, 1340, 1252, 750; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.48 (d, *J* 8.0 Hz, 1H), 7.83 (d, *J* 10.0 Hz, 1H), 7.59 (t, *J* 8.0 Hz, 1H), 7.42 (t, *J* 8.0 Hz, 1H), 7.05 (t, *J* 8.0 Hz, 1H), 6.78 (d, *J* 8.0 Hz, 2H),

4.15 (7.42 (q, *J* 7.0 Hz, 2H), 1.50 (t, *J* 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5, 162.5, 139.0, 133.5, 132.9, 120.8, 125.6, 118.1, 116.0, 103.7, 64.2, 14.6; HRMS (ESI-positive ionization): calcd for $C_{15}H_{14}IO_3$ ($[M+H]^+$): 368.9988, found: 368.9999.

1-(2,4,6-Trimethoxyphenyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6k). Reaction of **3k** (66 mg, 0.12 mmol) according to general procedure afforded 44 mg (89%) of product **6k** isolated as a white solid: mp 249.2-250.3 °C; IR (KBr) cm^{-1} 3100, 3025, 2900, 1615, 1460, 1333, 1238, 752; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (d, *J* 8.0 Hz, 1H), 7.42-7.32 (m, 1H), 6.81 (d, *J* 8.0 Hz, 1H), 6.26 (s, 2H), 3.94 (s, 3H), 3.83 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.9, 162.1, 134.0, 133.02, 132.7, 130.3, 124.7, 115.0, 91.0, 88.3, 56.6, 55.9, 30.9; HRMS (ESI-positive ionization): calcd for $C_{16}H_{16}IO_5$ ($[M+H]^+$): 415.0042, found: 415.0047.

1-(4-Chlorophenyl)-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6l).[42] Reaction of **3l** (52 mg, 0.10 mmol) according to general procedure afforded 10 mg (28%) of product **6l** isolated as a white solid: mp 207.7-208.3 °C (lit.[42]; mp 226-226.5 °C); IR (KBr) cm^{-1} 3045, 2930, 1611, 1472, 1345, 1087, 738; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (d, *J* 8.0 Hz, 1H), 7.90 (d, *J* 8.0 Hz, 2H), 7.69-7.61 (m, 1H), 7.58 (d, *J* 8.0 Hz, 2H), 7.51-7.42 (m, 1H), 6.77 (d, *J* 8.0 Hz, 1H); HRMS (ESI-positive ionization): calcd for $C_{13}H_9{}^{35}ClIO_2$ ($[M+H]^+$): 358.9336, found: 358.9835.

5-Methyl-1-phenyl-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6m).[50] Reaction of **3m** (88 mg, 0.18 mmol) according to general procedure afforded 47 mg (78%) of product **6m** isolated as a white solid: mp 235.6-236.8 °C (lit.[50]; mp 222-223 °C); IR (KBr) cm^{-1} 3049, 2966, 1618, 1454, 1338, 742; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (s, 1H), 8.03 (d, *J* 8.0 Hz, 2H), 7.78 (t, *J* 8.0 Hz, 2H), 7.60 (t, *J* 8.0 Hz, 2H), 7.29 (d, *J* 8.0 Hz, 1H), 6.67 (d, *J* 8.0 Hz, 1H), 2.41 (s, 3H); HRMS (ESI-positive ionization): calcd for $C_{14}H_{12}IO_2$ ($[M+H]^+$): 338.9882, found: 338.9865.

5-Bromo-1-phenyl-1λ^3-benzo[*d*][1,2]iodaoxol-3(1*H*)-one (6n).[47] Reaction of **3n** (30 mg, 0.054 mmol) according to general procedure afforded 12 mg (55%) of product **6n** isolated as a white solid: mp 243.8-244.4 °C (lit.[47]; mp 234-236 °C); IR (KBr) cm^{-1} 3045, 2955, 1621, 1443, 1382, 739; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.61 (d, *J* 3.0 Hz, 1H), 7.97 (d, *J* 8.4 Hz, 2H), 7.80 (t, *J* 7.6 Hz, 1H), 7.68-7.57 (m, 2H), 7.52 (dd, *J* 8.6 Hz, 3.0 Hz, 1H), 6.58 (d, *J* 8.6 Hz, 1H); HRMS (ESI-positive ionization): calcd for $C_{13}H_9{}^{79}BrIO_2$ ($[M+H]^+$): 402.8831, found: 402.8805.

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Supplementary Material

NMR spectra (1H, 13C and 19F) of products can be found in the supplementary material file.

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