Synthesis of arylbenziodoxoles using pseudocyclic benziodoxole triflate and arenes

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Received 08-05-2020  Accepted 09-12-2020  Published on line 09-24-2020

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

DOI: https://doi.org/10.24820/ark.5550190.p011.324

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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.20,21 A typical synthetic method of diaryliodonium 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 pseudocyclic 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.](image)

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

| Entry | Benzene 2a (equiv.) | Solvent     | 3a (%)\(^{b}\) |
|-------|---------------------|-------------|----------------|
| 1\(^{c}\) | 1                   | TFE         | 0\(^{d}\)      |
| 2\(^{c}\) | 56                  | TFE         | 0\(^{d}\)      |
| 3\(^{c}\) | 56                  | \(\text{CH}_2\text{Cl}_2\) | 0\(^{d}\)      |
| 4     | 56                  | \(\text{CH}_2\text{Cl}_2\) | >99           |
| 5     | 56                  | TFE         | 0\(^{d}\)      |
| 6     | 56                  | MeCN        | 0\(^{d}\)      |
| 7     | 56                  | AcOEt       | 0\(^{d}\)      |
| 8     | 56                  | Heptane     | 0\(^{d}\)      |
| 9     | 56                  | \(\text{ClCH}_2\text{CH}_2\text{Cl}\) | 80           |
| 10    | 56                  | \(\text{CHCl}_3\) | 82           |
| 11    | 10                  | \(\text{CH}_2\text{Cl}_2\) | >99          |
| 12    | 5                   | \(\text{CH}_2\text{Cl}_2\) | >99          |
| 13    | 4                   | \(\text{CH}_2\text{Cl}_2\) | >99          |
| 14    | 3                   | \(\text{CH}_2\text{Cl}_2\) | 84           |
| 15    | 2                   | \(\text{CH}_2\text{Cl}_2\) | 79           |
| 16    | 1                   | \(\text{CH}_2\text{Cl}_2\) | 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.\textsuperscript{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\textsuperscript{a}

|   |   |   |   |   |
|---|---|---|---|---|
|   |   |   |   |   |
| 1 | 2 | 3a-m |
|   |   |   |   |   |
|   |   |   |   |   |

\begin{align*}
\text{1} & \quad \text{1 equiv.} \\
\text{2} & \quad \text{(4 equiv.)} \\
& \quad \text{TfOH (1.2 equiv.)} \\
& \quad \text{CH}_2\text{Cl}_2, \text{rt, 24 h} \\
\end{align*}

\begin{align*}
3a & \quad \text{99\%} \\
3b & \quad \text{100\%} \\
3c & \quad \text{94\%} \\
3d & \quad \text{94\%} \\
3e & \quad \text{100\%} \\
3f & \quad \text{97\%} \\
3g & \quad \text{100\%} \\
3h & \quad \text{93\%} \\
3i & \quad \text{100\%} \\
3j & \quad \text{97\%} \\
3k & \quad \text{64\%} \\
3l & \quad \text{77\%} \\
\end{align*}

\textsuperscript{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-iodosylenzoic acid 4a with trifluoromethanesulfonic acid (Scheme 2). This reaction gave the pseudocyclic diaryliodonium triflate 3a in quantitative yield (eq. 1). 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. As expected, the reaction of substituted iodosylenzoic acids 4b,c under one-pot method resulted in the corresponding compounds 3m,n in good yields (eq. 1). 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).

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

Finally, we have demonstrated that the obtained pseudocyclic diaryliodonium triflates could be converted to arylbenziodoxolones. 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

| Reaction conditions: Pseudocyclic diaryliodonium triflates 3 in saturated NaHCO₃ aqueous –dichloromethane (1:2) at room temperature for 1 hour. | The isomeric ortho and para products were detected. |
|---|---|
| 6a 100% | 6b 89% | 6c 100% | 6d 82% |
| 6e 95% | 6f 69%⁵ | 6g 62% | 6h 66% |
| 6i 99% | 6j 86% | 6k 89% | 6l 28% |
| 6m 78% | 6n 55% |

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-iodosylenbenzoic acid and trifluoromethanesulfonic acid, or 2-iodobenzoic acid and m-chlorperoxybenzoic 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 diaryliiodonium 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 CaH₂ 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 (¹H NMR and ¹³C 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-iodosylenbenzoic acids 4a-b, and 4c 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).** 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, 1444, 1292, 1163, 1023, 911, 745; ¹H NMR (300 MHz, CDCl₃): δ 8.40 (dd, J 7.5 Hz, 1.5 Hz, 1H), 8.17 (d, J 8.4 Hz, 2H), 7.92 (t, J 7.5 Hz, 1H), 7.84-7.66 (m, 4H), 7.07 (d, J 8.4 Hz, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 169.0, 138.1, 137.5, 134.1, 133.3, 132.7, 131.7, 129.7, 126.3, 120.9 (q, ¹³C CF = 318.2 Hz), 114.3, 109.2; ¹⁹F NMR (282 MHz, CDCl₃): δ -79.4; HRMS (APCI-positive ionization): calcd for C₁₃H₁₀I₂O₂ ([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 (l>2.0/σ(l)) = 0.0469, Rw (all) = 0.0760, CCDC 2021394.

**2-Carboxyphenyl(mesityl)iodonium triflate (3b).** 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; ¹H NMR (300 MHz, CDCl₃): δ 8.38 (d, J 7.5 Hz, 1H), 7.71 (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 C₁₆H₁₆I₂O₂ ([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; ¹H
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NMR (300 MHz, CD₃CN): 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); ¹³C NMR (100 MHz, CD₃CN): δ 169.5, 141.2, 141.1, 140.1, 138.1, 136.0, 134.0, 132.4, 129.6, 127.3, 121.5 (q, ¹JC = 318.3 Hz), 114.3, 113.5, 24.5, 20.2; ¹⁹F NMR (377 MHz, CD₃CN): δ -79.3; HRMS (APCI-positive ionization): calcd for C₁₅H₁₄O₂ ([M-OTf]⁺): 353.0038, found: 353.0056.

2-Carboxyphenyl(2,3,5,6-tetramethylyphenyl)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 172.1-175.0 ℃; IR (KBr) cm⁻¹ 3466, 3085, 2964, 2910, 1655, 1585, 1469, 1438, 1260, 1171, 1030, 755; ¹H NMR (500 MHz, CD₃CN): 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); ¹³C NMR (100 MHz, DMSO-d₆): δ 169.1, 156.9, 137.8, 136.5, 132.9, 131.5, 130.0, 129.8, 121.2, (q, ¹JC = 320.0 Hz), 116.34, 109.9, 35.6, 31.3; ¹⁹F NMR (377 MHz, DMSO-d₆): δ -77.7; HRMS (APCI-positive ionization): calcd for C₁₇H₁₈O₂ ([M-OTf]⁺): 381.0351, found: 381.0347.

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 202.1-203.6 ℃; (lit. ²⁴ mp 193-194 ℃); IR (KBr) cm⁻¹ 3469, 3089, 2990, 1670, 1587, 1472, 1440, 1255, 1166, 1029, 754; ¹H NMR (300 MHz, CD₃CN): 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 C₁₄H₁₃O₂ ([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 (l>2.0/σ(l)) = 0.0658, Rw (all) = 0.0844, CCDC 2021395.

2-Carboxyphenyl(2,4,6-trisopropylphenyl)iodonium triflate (3g). Reaction of 1,3,5-trisopropylbenzene 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 ℃; IR (KBr) cm⁻¹ 3430, 3078, 2964, 2931, 2876, 1674, 1588, 1465, 1443, 1304, 1170, 1024, 750; ¹H NMR (300 MHz, CD₃CN): 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); ¹³C NMR (100 MHz, CD₃CN): δ 169.4, 157.2, 154.3, 137.9, 134.0, 132.3, 129.4, 127.8, 125.8, 121.5 (q, ¹JC = 318.0 Hz), 117.0, 114.4, 39.4, 34.8, 23.5; ¹⁹F NMR (282 MHz, CD₃CN): δ -79.3; HRMS (APCI-positive ionization): calcd for C₂₂H₂₉ClO₂ ([M-OTf]⁺): 451.1134, found: 451.1136.

2-Carboxyphenyl(3,4-diethylyphenyl)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 ℃; IR (KBr) cm⁻¹ 3462, 2974, 2943, 2880, 1674, 1586, 1473, 1439, 1292, 1176, 1025, 746; ¹H NMR (500 MHz, CD₃CN): 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); ¹³C NMR (100 MHz, CD₃CN): 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, ¹JC = 318.0 Hz), 115.0, 106.2, 25.9, 25.6, 14.7, 14.6; ¹⁹F NMR
(377 MHz, CD$_3$CN): δ -79.3; HRMS (APCI-positive ionization): calcd for C$_{17}$H$_{18}$O$_2$ ([M-OTf]$^-_{\text{2}}$): 381.0351, found: 381.0348.

2-Carboxyphenyl(4-methoxyphenyl)iodonium triflate (3i). 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.); 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$_3$CN): 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$_3$CN): δ 169.7, 164.8, 140.8, 137.9, 133.8, 132.2, 130.0, 127.0, 121.6 (q, $^1$JC = 319.5 Hz), 115.5, 97.8, 56.4; HRMS (ESI-positive ionization): calcd for C$_{14}$H$_{12}$O$_3$ ([M-OTf]$^-_{\text{2}}$): 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$_3$CN): 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, $^1$JC = 320.0 Hz), 118.8, 116.9, 101.5, 64.4, 14.5; $^{19}$F NMR (377 MHz, CD$_3$CN): δ -79.4; HRMS (ESI-positive ionization): calcd for C$_{15}$H$_{12}$O$_3$ ([M-OTf]$^-_{\text{2}}$): 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$_3$CN): 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$_3$CN): δ 169.8, 169.3, 162.4, 137.9, 133.9, 132.0, 128.7, 127.3, 118.4 (q, $^1$JC = 317.3 Hz), 113.7, 92.9, 79.7, 57.7, 56.9; $^{19}$F NMR (377 MHz, CD$_3$CN): δ -79.4; HRMS (ESI-positive ionization): calcd for C$_{16}$H$_{16}$O$_5$ ([M-OTf]$^-_{\text{2}}$): 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>2σ(I)) = 0.0438, Rw (all) = 0.0582, CCDC 2021393.

2-Carboxyphenyl(4-chlorophenyl)iodonium triflate (3l). 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.); 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$_3$CN): δ -77.7; HRMS (ESI-positive ionization): calcd for C$_{13}$H$_9$ClO$_2$ ([M-OTf]$^-_{\text{2}}$): 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-iodosylybenzoic 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-iodosylybenzoic 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; ¹H NMR (400 MHz, CD₃OD): 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); ¹³C NMR (125 MHz, CD₃OD): δ 169.8, 142.5, 137.7, 137.2, 133.4, 133.4, 132.2, 129.1, 127.1, 120.4 (q, J₁CF = 317 Hz), 19.21; ¹⁹F NMR (377 MHz, CD₃OD): δ -80.1; HRMS (ESI-positive ionization): calcd for C₁₄H₁₂O₂ ([M+OTf]+): 338.9882, found: 338.9900.

4-Bromo-2-carboxyphenyl(phenyl)iodonium triflate (3n). Reaction of 4-bromo-2-iodosylybenzoic 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; ¹H NMR (400 MHz, CD₃CN): 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); ¹³C NMR (100 MHz, CD₃CN): δ 168.5, 140.4, 138.7, 136.3, 134.8, 133.4, 131.9, 128.8, 126.3, 121.6 (q, J₁CF = 317 Hz), 113.5, 109.8; ¹⁹F NMR (377 MHz, CD₃CN): δ -79.4; HRMS (ESI-positive ionization): calcd for C₁₃H₁⁹BrI₂O₂ ([M-OTf]+): 402.8831, found: 338.9885.

General procedure for preparation of pseudoaromatic diaryliodonium triflates 3a using 2-iodosylybenzoic acid 5 and benzene 2a using TfOH with MCPBA. Benzene 2a (62 mg, 0.80 mmol), trifluoromethanesulfonic acid (66 mg, 0.44 mmol), and MCPBA (76 mg, 0.44 mmol) was added to a solution of 2-iodosylybenzoic 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.

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MHZ, CD$_3$OD): $\delta$ 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$_{14}$O$_2$ ([M+H]$^+$): 353.0038, found: 353.0032.

1-(2,3,5,6-Tetramethylphenyl)-1$\lambda^3$-benzo[d][1,2]iodaoxol-3(1H)-one (6d). 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.); 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$_3$): $\delta$ 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$_{18}$O$_2$ ([M+H]$^+$): 381.0356, found: 381.0366.

1-(4-(tert-butyl)phenyl)-1$\lambda^3$-benzo[d][1,2]iodaoxol-3(1H)-one (6e). 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.); mp 233.5-234 °C; IR (KBr) cm$^{-1}$ 3059, 2959, 2869, 1612, 1556, 1482, 1347, 1058, 748; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 8.48 (d, $J$ 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$_{21}$H$_{22}$O$_2$ ([M+H]$^+$): 381.0366.

1-(p-Toly)-1$\lambda^3$-benzo[d][1,2]iodaoxol-3(1H)-one (6f). 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.); mp 217-218 °C; IR (KBr) cm$^{-1}$ 3074, 2977, 2923, 1610, 1557, 1488, 1329, 747; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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$_{16}$H$_{12}$O$_2$ ([M+H]$^+$): 338.9882, found: 338.9882.

1-(2,4,6-Trisopropylphenyl)-1$\lambda^3$-benzo[d][1,2]iodaoxol-3(1H)-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$_3$): $\delta$ 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), 6.67 (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$_3$): $\delta$ 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$_{28}$O$_2$ ([M+H]$^+$): 451.1134, found: 451.1112.

1-(3,4-Diethylphenyl)-1$\lambda^3$-benzo[d][1,2]iodaoxol-3(1H)-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$_3$): $\delta$ 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$_3$): $\delta$ 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$_{17}$H$_{18}$O$_3$ ([M+H]$^+$): 354.0351, found: 354.0352.

1-(4-Methoxyphenyl)-1$\lambda^3$-benzo[d][1,2]iodaoxol-3(1H)-one (6i). 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.); mp 212-214 °C; IR (KBr) cm$^{-1}$ 3065, 2941, 2840, 1599, 1489, 1359, 1257, 748; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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$_3$): $\delta$ 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$_{17}$H$_{18}$O$_3$ ([M+H]$^+$): 354.0381, found: 354.0387.
4.15 (7.42 (q, J 7.0 Hz, 2H), 1.50 (t, J 7.0 Hz, 3H); 13C NMR (100 MHz, CDCl3): 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 C15H14IO3 ([M+H]+): 368.9988, found: 368.9999.

1-(2,4,6-Trimethoxyphenyl)-1λ3-beno[d][1,2]iodaoxol-3(1H)-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, 1615, 1460, 1333, 1238, 752; 1H NMR (400 MHz, CDCl3): δ 8.45 (d, J 8.0 Hz, 1H), 7.42-7.32 (m, 1H), 6.31 (d, J 8.0 Hz, 1H), 6.26 (s, 2H), 3.94 (s, 3H), 3.83 (s, 6H); 13C NMR (100 MHz, CDCl3): δ 166.9, 162.1, 134.0, 133.02, 132.7, 130.3, 129.0 (2,4,6-Trimethoxyphenyl) ; IR (KBr) cm⁻¹ 3045, 2955, 1621, 1443, 1382, 739 (1H); HRMS (ESI-positive ionization): calcd for C16H16IO3 ([M+H]+): 415.0042, found: 415.0047.

1-(4-Chlorophenyl)-1λ3-beno[d][1,2]iodaoxol-3(1H)-one (6l). 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; IR (KBr) cm⁻¹ 226-226.5 °C; IR (KBr) cm⁻¹ 3045, 2930, 1611, 1472, 1345, 1087, 738; 1H NMR (400 MHz, CDCl3): δ 8.49 (d, J 8.0 Hz, 1H), 7.90 (d, J 8.0 Hz, 1H), 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 C13H935ClIO2 ([M+H]+): 358.9336, found: 358.9835.

5-Methyl-1-phenyl-1λ3-beno[d][1,2]iodaoxol-3(1H)-one (6m). 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; IR (KBr) cm⁻¹ 3049, 2966, 1618, 1454, 1338, 742; 1H NMR (400 MHz, CDCl3): 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 C14H12IO2 ([M+H]+): 338.9882, found: 338.9865.

5-Bromo-1-phenyl-1λ3-beno[d][1,2]iodaoxol-3(1H)-one (6n). 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; IR (KBr) cm⁻¹ 3045, 2955, 1621, 1443, 1382, 739; 1H NMR (400 MHz, CDCl3): 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 C13H979BrIO2 ([M+H]+): 402.8831, found: 402.8805.

Acknowledgements

This work was supported by a research grant from the Russian Science Foundation (RSF-16-13-10081-P) and National Science Foundation (CHE-1759798). A.S. is thankful to JSPS Fund for the Promotion of Joint International Research (Grant No 16KK0199) and JST CREST (No. JRMJCR19R2). Some research was carried out using the core facilities of TPU’s “Physical and chemical methods of analysis

Supplementary Material

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

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