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Preparation, structure, and reactivity of *ortho*-hydroxy-substituted aryliodonium salts, precursors to *ortho*-iodo-substituted diaryl ethers

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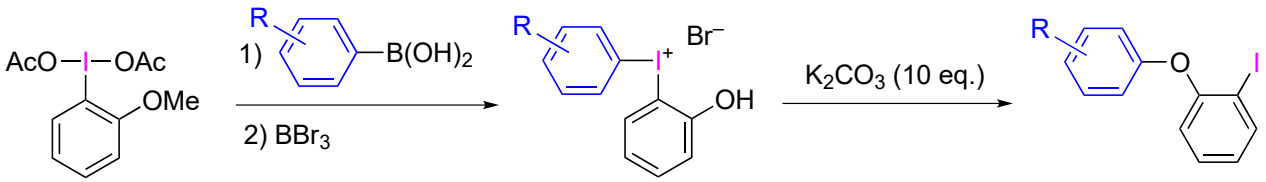
Abstract

ortho-Hydroxy-substituted aryliodonium salts were prepared by a two-step one-pot synthesis based on the reaction of 2-alkoxy-1-[(diacetoxy)iodo]arenes with arylboronic acids involving the initial formation of 2-alkoxyphenyl(aryl)iodonium salts and followed by deprotection of the intermediate product by boron tribromide. This procedure works well with halogen-, cyano-, 2-hydroxy-, 3-hydroxy-, 4-hydroxy-, methyl-, or trifluoromethyl-substituted arylboronic acids. Structures of three 2-hydroxyphenyl(aryl)iodonium bromides were confirmed by single crystal X-ray crystallography. The prepared 2-hydroxyphenyl(aryl)iodonium salts can be further converted to *ortho*-iodo-substituted diaryl ethers by treatment with a base via intermediate formation of the unstable aryliodonium betaines.



13 examples
X-ray for R = H, 2-Cl, and 4-OH

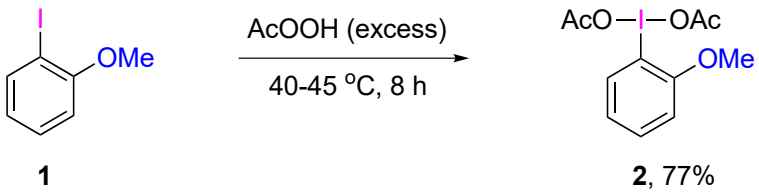
Keywords: Hypervalent iodine, iodonium salts, iodonium, phenols

Introduction

Hypervalent iodine compounds are widely used as reagents in organic synthesis due to their environmentally benign nature and their reactivity pattern being similar to that of heavy metals.[1-8] Iodonium salts represent a particularly important class of hypervalent iodine(III) compounds, finding numerous applications in industry, medicine, and organic synthesis.[9-18] They are widely used as photoinitiators for cationic photopolymerizations.[15-18] Furthermore, diaryliodonium salts and ylides are highly popular reagents for the efficient introduction of [18F]-fluoride (radiofluorination) via aromatic nucleophilic substitution, which has found widespread clinical application in medical diagnostics.[19-23] A summary of the biological properties of iodonium salts has been published in the 1996 review[24] and in the 2014 book.[4] In organic synthesis, symmetrical or unsymmetrical diaryliodonium salts are widely employed as effective electrophilic arylating reagents towards various organic nucleophilic substrates.[9-13,25] They also serve as efficient benzyne precursors under strongly basic conditions.[11,26] Recently, diaryliodonium compounds with silyl or boron-containing substituents have been synthesized and used as efficient precursors to aryne species.[27-30] In particular, our group has reported the preparation and structural investigation of novel benzyne precursors, pseudocyclic arylbenziodoxaborole triflates, which could react with various substrates in the presence of water to give the respective benzyne adducts in moderate to good yields.[28-31] Despite significant current interest in functionalized diaryliodonium compounds, the phenol-derived aryliodonium salts remain almost unexplored because of the general incompatibility of the phenolic moiety with iodonium groups, which possess strong oxidizing reactivity. The known phenolic iodonium compounds are primarily the *ortho*-hydroxy substituted diaryliodonium salts, which are important precursors to phenolic iodonium ylides.[31-35] Recently, we have reported a general synthetic approach to the *meta*-hydroxy substituted diaryliodonium[36] and *para*-hydroxy substituted diaryliodonium salts[37] and demonstrated that these compounds are potentially useful phenol transfer reagents in reactions with various anionic nucleophiles.[37] Herein, we report a new and convenient two-step one-pot synthesis and reactions of 2-hydroxyphenyl(aryl)iodonium salts.

Results and Discussion

Our synthetic approach to *ortho*-hydroxy-substituted aryliodonium salts is based on a one-pot two-step synthesis starting from 2-methoxy-1-[(diacetoxy)iodo]benzene (**2**). Diacetoxyiodoarene **2** was prepared according to slightly modified known procedures by oxidation of the commercially available 2-methoxy-1-iodobenzene (**1**) with peracetic acid (Scheme 1).[38]

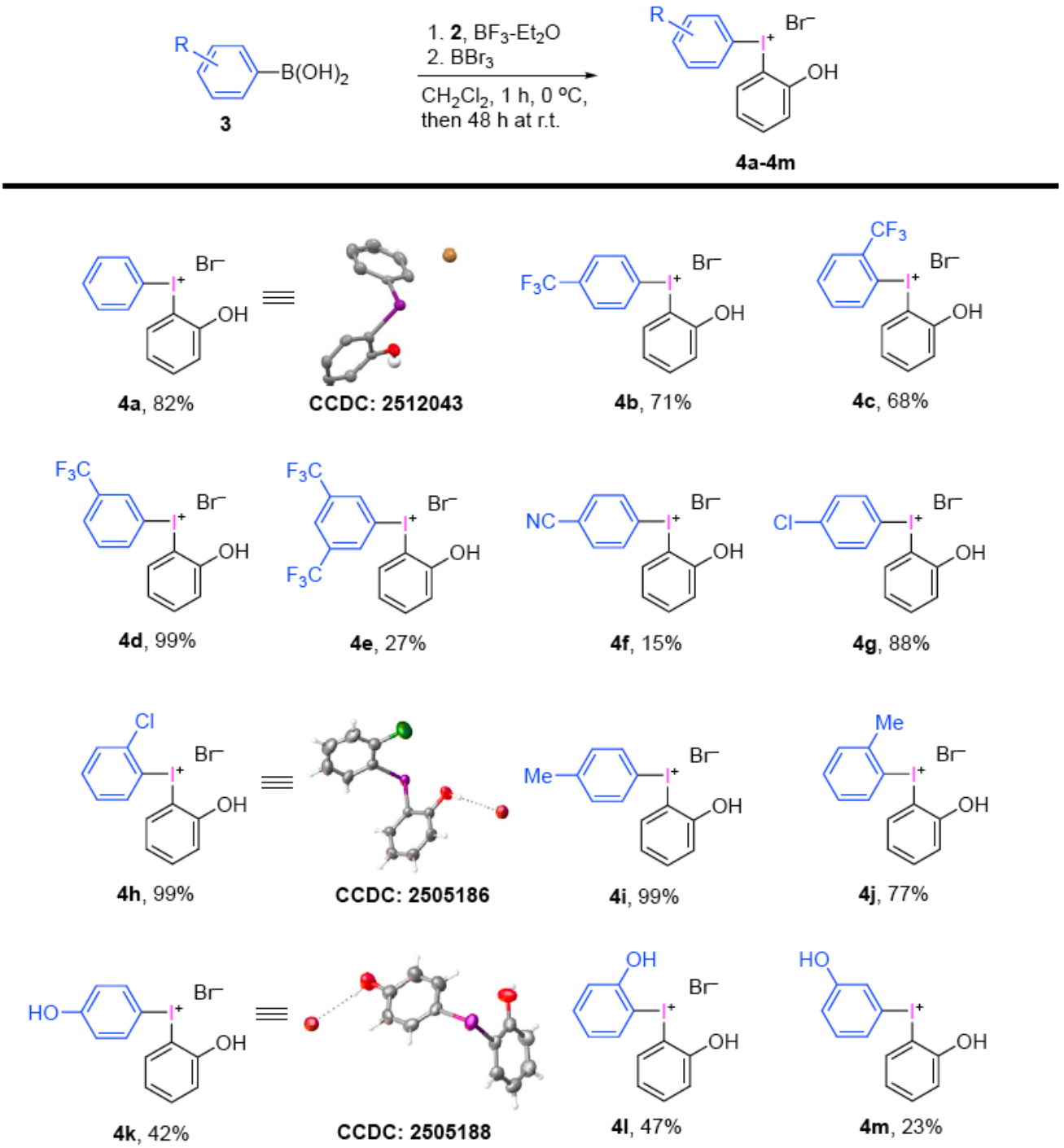


1 **2**, 77%

Scheme 1. Synthesis of 2-methoxy-1-[(diacetoxy)iodo]benzene **2**.

2-Methoxy-1-[(diacetoxy)iodo]benzene (**2**) was further reacted with arylboronic acids **3** followed by deprotection of the alkoxy group by adding solution of boron tribromide in dichloromethane (Scheme 2). The

final products, 2-hydroxyphenyl(aryl)iodonium salts **4**, were isolated in analytically pure form after removal of solvents and washing residual oil with ether and identified by NMR, high-resolution ESI, and X-ray crystallography.



Scheme 2. Reaction scope of *ortho*-phenolic iodonium salts **4** and X-ray crystal structures of **4a, 4h, 4k.** Ellipsoids were drawn to the 50% probability level. A solvent molecule and all non-oxygen hydrogen atoms in **4a** were removed for clarity.

Compounds **4a, 4h, 4k** were studied with X-ray crystallography (Figure 1 and Table 1). All three molecules have a pseudo-square planar geometry with two carbon atoms bonding to the iodine atom and two halogen-bonding bromide counter ions completing the coordination sphere with iodine as illustrated by X-ray crystal structure of **4a** in Figure 1. The second bromide ion [Br(1)*] was found in the unit cell and is related by an inversion center (Figure 1). Along with the bromide ions, the iodine atom formed a diamond-shaped dimer with a second iodine atom, also related by the same inversion center. The halogen-bonding dimers were also observed in a set of dibenzochlorolium and dibenzobromolium compounds.[39] Atomic distances and angles are reported in Table 1. The I–C bond distances ranged from 2.099(8) to 2.112(3) Å. Close contacts between the iodine atom and neighboring bromide counterions were also observed between 3.289(1) to 3.405(1) Å. Normalized contact values Nc (observed distance divided by Bondi distance) between the I(1)–Br(1) ranged from 0.86-0.89. The largest angles of the pseudo-square planar coordination sphere were observed between 163.9(2) and 178.5(2) ° for C(1)–I(1)– Br(1) and C(7)–I(1)– Br(1)*, respectively. Additionally, a mean planes (mpln) of the five atoms of the coordination sphere were calculated and the average distance of the atoms to the plane (I(1), C(1), C(7), Br(1) and Br(1)*) ranged from 0.032-0.201 Å. Considering the Nc values and the pseudo-square planar geometry around the iodine atom, sigma-donor halogen bonding is assigned.[40]

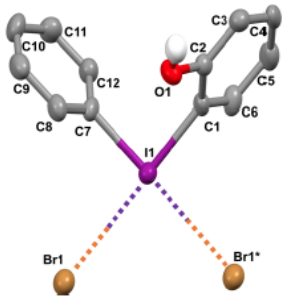
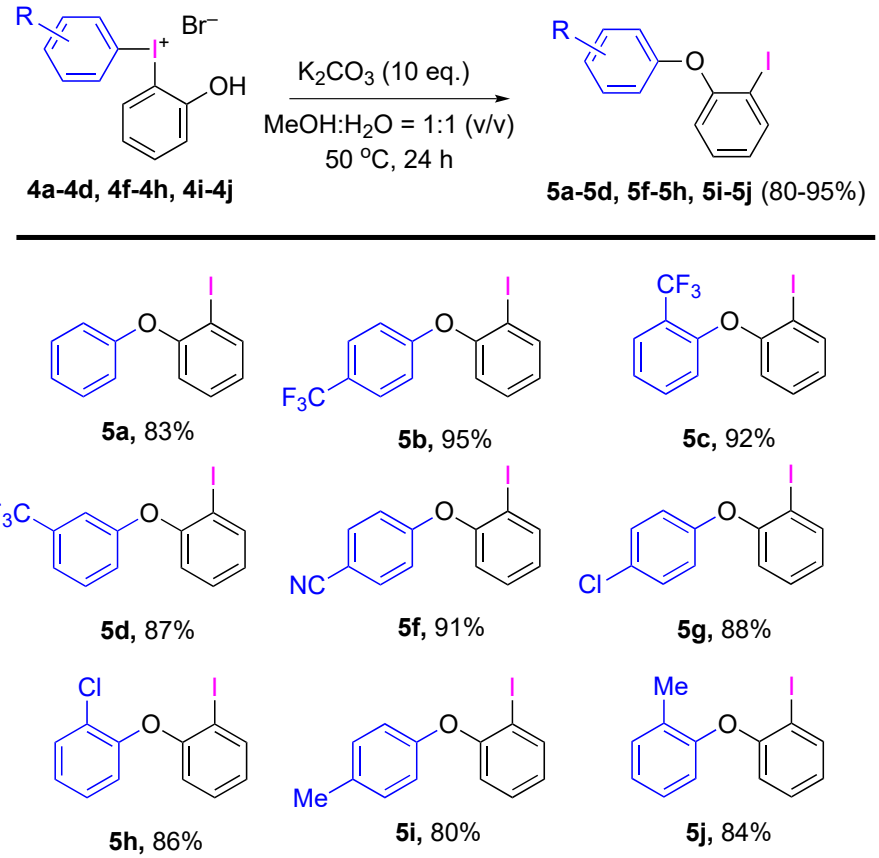


Figure 1. X-ray crystal structure of **4a**. Ellipsoids are drawn to the 50% probability level. Non-oxygen hydrogen atoms and a methanol solvent molecule were removed for clarity. A second bromide counter ion (Br1*) is related by inversion symmetry to Br1 and is displayed to illustrate the pseudo square planar geometry and halogen bonding interactions.

Table 1. X-ray crystal structure data of compounds **4a, 4h, 4k**. Atom Br(1)* is a second counterion related by an inversion center to Br(1). Selected bond, contact length [Å] and angles [°]; mpln is average distance from a mean plane by pseudo square planar coordination center: (I(1), C(1), C(7), Br(1), Br(1)*, [Å]. Nc, normalized contact distance: observed distance divided by sum of Bondi distances. The numbering scheme in Figure 1 (*ortho*-hydroxy is on the C2 carbon) is used for all compounds in Table 1. CCDC deposition numbers **4a** 2512043, **4h** 2505186, **4k** 2505188

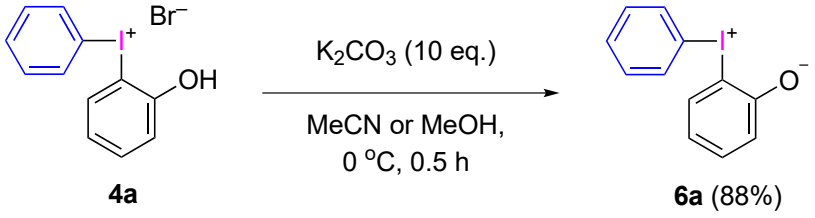
	I1–C1 (Å)	I1–C7 (Å)	C1–I1–C7 (°)	I-Br1 (Å)	I-Br1* (Å)	C1-I1-Br1 (°)	C1-I1-Br1* (°)	mpln (Å)
4a	2.117(6)	2.114(5)	94.8(2)	3.327(1) Nc 0.87	3.289(1) Nc 0.86	178.5(2)	177.0(2)	0.032
4h	2.107(3)	2.122(3)	92.9(1)	3.3183(4) Nc 0.87	3.3121(5) Nc 0.87	173.42(8)	167.10(9)	0.061
4k	2.099(8)	2.111(7)	96.2(3)	3.405(1) Nc 0.89	3.336(1) Nc 0.88	163.9(2)	173.1(2)	0.201

The general procedure for the preparation of phenolic iodonium salts **4** (Scheme 2) works well with trifluoromethyl-, halogen-, cyano-, methyl-, or hydroxy-substituted arylboronic acids **3**. 2-Hydroxyphenyl(aryl)iodonium salts **4** can be further converted to the corresponding ethers **5** by treatment with a potassium carbonate at 50 °C (Scheme 3).



Scheme 3. Reaction scope of diaryl ethers **5**.

It is known from the literature that this reaction (Scheme 3) proceeds via *ipso*-rearrangement in the initially formed and generally unstable betaines, which could be isolated only for the nitro-substituted derivatives.[33,41,42] In a recent publication of Han and coauthors,[35] the isolation of a methyl-substituted betaine was attempted. The authors of this paper were able to obtain such a betaine as a highly unstable precipitate and analyze it by ^{1}H NMR immediately after preparation.[35] We were able to confirm the formation of the betaine intermediate **6a** resulting from deprotonation of 2-hydroxyphenyl(phenyl)iodonium bromide **4a** with potassium carbonate in methanol (Scheme 4).



Scheme 4. Synthesis of betaine **6a**.

The treatment of iodonium salt **4a** with potassium carbonate afforded crude betaine **6a**, which was isolated in solid form after removal of solvents and washing residual oil with ether. Betaine **6a** has low thermal stability completely decomposing at 40-42 °C; however, we were able to confirm its structure by ^{1}H NMR spectroscopy (Figure 2). According to NMR betaine **6a** was observed after 0.5 h at 0°C in basic conditions, then it was completely transformed into ether **5a** after 24 h at 50 °C.

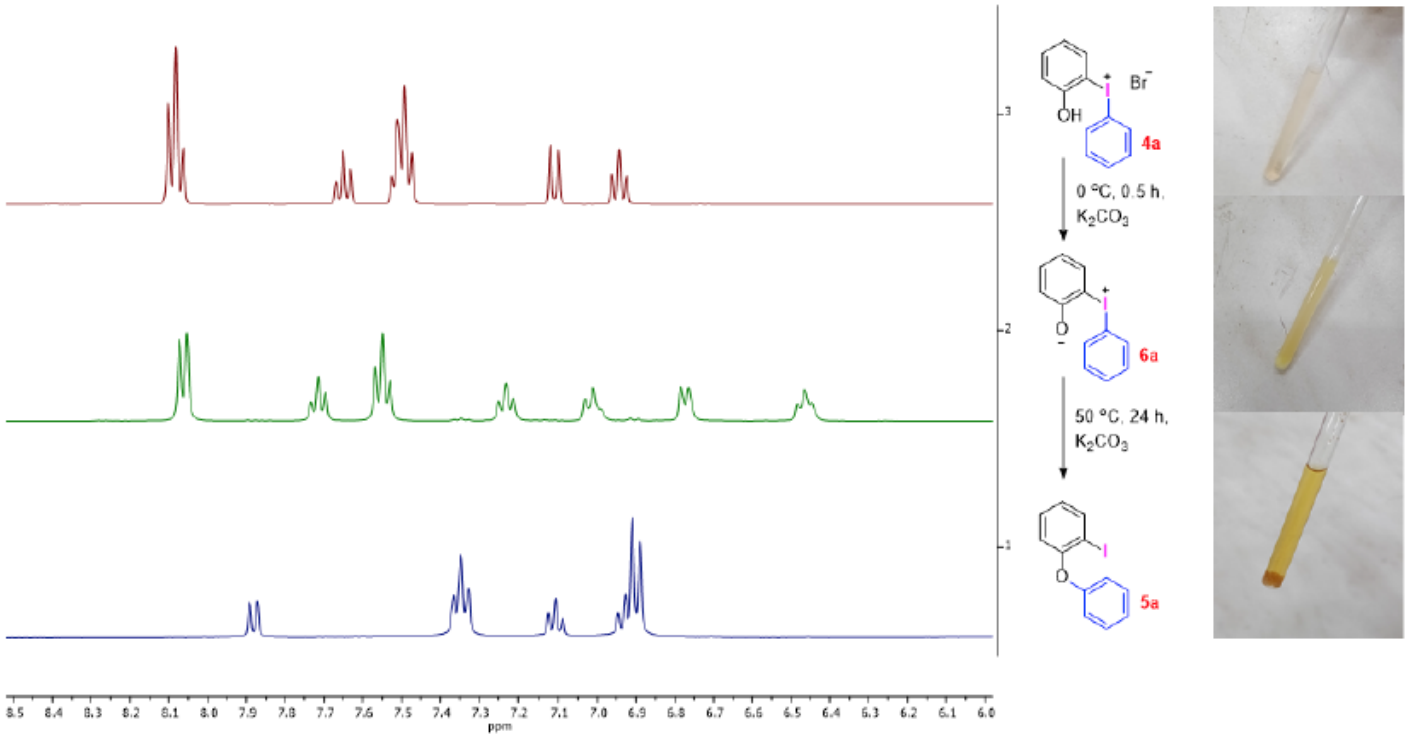


Figure 2. Transformations of iodonium salt **4a** into betaine **6a** then into diaryl ether **5a.**

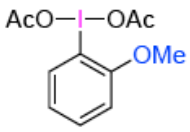
Conclusions

In summary, we have developed a general synthetic approach to important *ortho*-hydroxy-substituted aryliodonium salts by a two-step one-pot synthesis based on the reaction of 2-methoxy-1-[(diacetoxy)iodo]benzene with arylboronic acids involving the initial formation of 2-methoxyphenyl(aryl)iodonium salts and followed by deprotection of the intermediate product by boron tribromide. The procedure works well with halogen-, cyano-, 2-hydroxy-, 3-hydroxy-, 4-hydroxy-, methyl-, or trifluoromethyl-substituted arylboronic acids. The prepared 2-hydroxyphenyl(aryl)iodonium salts can be further converted to the unstable aryliodonium betaines, whose rearrangement affords the corresponding *ortho*-iodo-substituted diaryl ethers as final products.

Experimental Section

Unless otherwise stated, all reactions were performed in open air with a stopper and oven-dried glassware. All commercial reagents were ACS grade reagents and used without further purification from freshly opened containers. All solvents were distilled in prior to use. The reactions were monitored by normal phase thin-layer chromatography (TLC) using Millipore Sigma glass-backed 60 Å plates (indicator F-254, 250 µM) using either hexanes/ethyl acetate or methanol/dichloromethane as the eluent system and were visualized using UV light at 254 nm followed by use of a permanganate stain or a p-anisaldehyde stain for visualization. Column chromatography was performed with silica gel (200-300 mesh ASTM). Melting points were determined in an open capillary tube with Buchi M-580 melting point apparatus. Infrared spectra were recorded as ATR on a P Agilent Cary 630 FT-IR spectrophotometer. NMR spectra were recorded on a Bruker BioSpin 400 MHz NMR spectrometer at 400 MHz (1H NMR), 101 MHz (13C NMR), 376 MHz (19F NMR). Chemical shifts are reported in parts per million (ppm). 1H and 13C chemical shifts are referenced relative to tetramethylsilane. High-resolution mass spectrometric measurements were performed using quadrupole-time-of-flight mass spectrometer Triple TOF 5600+ (AB Sciex, Canada) equipped with a Turbo Ion Spray electrospray ionization source and LC-30 "Nexera" (Shimadzu, Japan). X-ray crystal analysis of **4a** was performed by Rigaku RAPID II Image Plate or a Hybrid Pixel Array Detector system using graphite-monochromated MoKα radiation (λ = 0.71075 Å) at 173, 293 or 297 K. The crystals of **4h**, **4k** were measured using Mo Kα radiation on a Tongda TD-5000 diffractometer at 200K. Data reduction was performed via CrysAlisPro 1.171.42.59a software (Rigaku Oxford Diffraction, 2022). The structures were solved by direct methods and refined by the full-matrix least-squares method anisotropically for non-hydrogen atoms. H-atoms were refined in geometrically calculated positions. All calculations were performed using SHELX software packages[43,44] incorporated in the OLEX2 program package.[45] Please see cif files 2512043 (compound **4a)**, 2505186 (compounds **4h**), and 2505188 (compounds **4k**) for more detailed crystallography information. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre at http://www.ccdc.cam.ac.uk/structures.

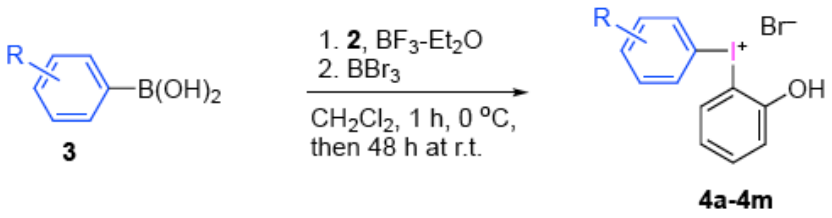
2-Methoxy(diacetoxyiodo)benzene 2b



2b

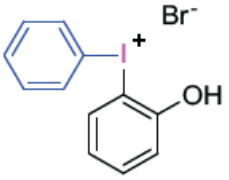
Commercial solution of peracetic acid in acetic acid (21.76 g, 40%) was added dropwise to 2-iodoanisole (2.34 g, 10 mmol) at 0 °C. The reaction mixture was stirred at 45 °C for 8 hours. After completion of the reaction, 100 mL of distilled H_2O was added and the resulting aqueous solution was extracted with DCM (3x50 mL). Organic layers were combined, dried over $MgSO_4$, concentrated under reduced pressure and residue was washed with diethyl ether (2x12.5 mL) to give 2.710 g (77%) of the titled compound **2b** as yellowish solid. NMR data corresponds to the literature.[38] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 7.9 Hz, 1H), 7.59 (t, *J* = 7.9 Hz, 1H), 7.15 (d, *J* = 8.4 Hz, 1H), 7.03 (t, *J* = 7.6 Hz, 1H), 3.98 (s, 3H), 1.97 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.8, 156.4, 137.9, 134.7, 122.9, 113.6, 112.2, 57.0, 20.5.

Preparation of 2-hydroxyphenyl(aryl)iodonium bromides 4, General procedure



Arylboronic acid **3** (0.5 mmol, 1 equiv.) and BF_3-Et_2O (85 mg, 74 µL, 0.6 mmol, 1.2 equiv.) in DCM (6.0 mL) was cooled down to 0 °C, then solution of **2b** (211 mg, 0.6 mmol, 1.2 equiv.) in DCM (6.0 mL) was added dropwise. The reaction mixture was stirred at 0 °C for 1 hour. After 1 hour solvent was removed under reduced pressure, and the residual 2-methoxyphenyl(aryl)iodonium salt was washed with diethyl ether (2x12.5 mL), dried in vacuum and dissolved in 6.0 mL DCM. After that 1.0 M solution of boron tribromide in DCM (2.5 mL, 2.5 mmol, 5 equiv.) was added to pre-cooled solution of 2-methoxyphenyl(aryl)iodonium salt in DCM at 0 °C. The reaction mixture was stirred for 48 hours allowing to achieve ambient temperature. After 48 hours the mixture was washed with distilled H_2O (2x12.5 mL), then dried in vacuum and washed with diethyl ether (2x12.5 mL) to give the desired 2-hydroxyphenyl(aryl)iodonium bromides **4a-4m**.

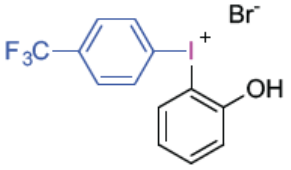
2-Hydroxyphenyl(phenyl)iodonium bromide 4a



Reaction of phenylboronic acid (61 mg, 0.5 mmol) according to general procedure afforded 155 mg (82%) of product **4a**, isolated as light-beige solid, mp 184-186 °C. IR (ATR, cm^{-1}): 3147, 3047, 1598, 1491, 1471, 1270, 1209, 1166, 643, 585, 454, 428. ^{1}H NMR (400 MHz, CD_3OD) δ 8.08 (d, *J* = 7.8 Hz, 3H), 7.65 (t, *J* = 7.5 Hz, 1H), 7.53-7.47 (m, 3H), 7.11 (d, *J* = 8.2 Hz, 1H), 6.94 (t, *J* = 7.7 Hz, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 157.6, 138.0, 136.4, 136.2, 133.2, 132.8, 123.4, 117.3, 115.6, 104.9. HRMS (ESI-positive mode): calcd for $C_{12}H_{10}IO^+$ ([M-Br])$^+$: 296.9771, found: 296.9774.

Single crystals of product **4a** suitable for X-ray crystallographic analysis were obtained by slow crystallization from MeOH-H_2O solution. Crystal data for **4a** $C_{13}H_{14}BrIO_2$, monoclinic, space group P2(1)/n, a = 9.984(3) Å, b = 6.282(2) Å, c = 22.663(7) Å, α = 90 °, β = 92.145(9) °, γ = 90 °, V = 1420.4(8) Å^3, Z = 4, 11805 reflections measured, 3114 unique reflections, 2465 I > 2/s(I), 159 parameters, 0 restraints; GooF = 1.017, final R = 0.0422, R (all) = 0.0599. Please refer to the cif for more detailed information: CCDC 2512043.

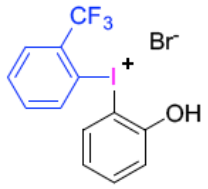
2-Hydroxyphenyl(4-trifluoromethylphenyl)iodonium bromide 4b



Reaction of 4-trifluoromethylphenylboronic acid (95 mg, 0.5 mmol) according to general procedure afforded 158 mg (71%) of product **4b**, isolated as white or light-beige solid, mp > 400 °C. IR (ATR, cm^{-1}): 3433, 3136,

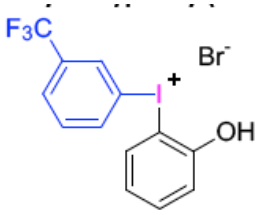
1599, 1572, 1484, 1304, 1271, 1208, 1186, 1160, 1116, 644, 538, 476, 436. ^{1}H NMR (400 MHz, CD_3OD) δ 8.28 (d, *J* = 8.3 Hz, 2H), 8.14 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.78 (d, *J* = 8.3 Hz, 2H), 7.52 (ddd, *J* = 8.5, 7.3, 1.5 Hz, 1H), 7.12 (dd, *J* = 8.2, 1.4 Hz, 1H), 6.97 (td, *J* = 7.7, 1.4 Hz, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 157.6, 138.3, 137.0, 136.5, 134.7 (q, $^{1}J_{CF}$ = 32.98 Hz), 129.3 (q, $^{2}J_{CF}$ = 3.76 Hz), 126.2, 123.5, 120.1, 117.4, 105.7. ^{19}F NMR (376 MHz, CD_3OD) δ -64.74. HRMS (ESI-positive mode): calcd for $C_{13}H_9F_3IO^+$ ([M-Br])$^+$: 364.9645, found: 364.9653.

2-Hydroxyphenyl(2-trifluoromethylphenyl)iodonium bromide 4c



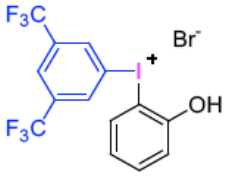
Reaction of 2-trifluoromethylphenylboronic acid (95 mg, 0.5 mmol) according to general procedure afforded 151 mg (68%) of product **4c**, isolated as white solid, mp 209-210 °C. IR (ATR, cm^{-1}): 3197, 1598, 1588, 1566, 1491, 1300, 1298, 1265, 1188, 1140, 1113, 641, 633, 595, 568, 548, 451, 426; ^{1}H NMR (400 MHz, CD_3OD) δ 8.63 (d, *J* = 8.0 Hz, 1H), 8.02-7.98 (m, 2H), 7.87 (t, *J* = 7.7 Hz, 1H), 7.77-7.72 (m, 1H), 7.50 (ddd, *J* = 8.6, 7.4, 1.5 Hz, 1H), 7.10 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.95 (ddd, *J* = 8.6, 7.3, 1.5 Hz, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 157.9, 141.9, 137.8, 136.4, 136.3, 134.3, 129.8 (q, $^{2}J_{CF}$ = 5.49 Hz), 123.4, 117.4, 111.7, 105.6, quartet from $^{1}J_{CF}$ is unobserved. ^{19}F NMR (376 MHz, CD_3OD) δ -60.39. HRMS (ESI-positive mode): calcd for $C_{13}H_9F_3IO^+$ ([M-Br])$^+$: 364.9645, found: 364.9653.

2-Hydroxyphenyl(3-trifluoromethylphenyl)iodonium bromide 4d



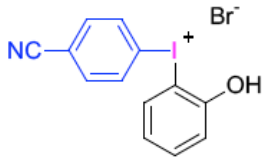
Reaction of 3-trifluoromethylphenylboronic acid (95 mg, 0.5 mmol) according to general procedure afforded 220 mg (99%) of product **4d**, isolated as white solid, mp 375-379 °C. IR (ATR, cm^{-1}): 3171, 1597, 1567, 1490, 1305, 1276, 1266, 1213, 1176, 1165, 1123, 640, 597, 546, 500, 476, 441, 423. ^{1}H NMR (400 MHz, CD_3OD) δ 8.46 (s, 1H), 8.35 (d, *J* = 8.2 Hz, 1H), 8.15 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.69 (t, *J* = 8.0 Hz, 1H), 7.53 (ddd, *J* = 8.3, 7.3, 1.5 Hz, 1H), 7.12 (dd, *J* = 8.2, 1.4 Hz, 1H), 6.97 (ddd, *J* = 8.3, 7.4, 1.4 Hz, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 157.6, 140.0, 138.2, 136.6, 134.1 (q, $^{1}J_{CF}$ = 33.64 Hz), 133.4, 133.0 (q, $^{2}J_{CF}$ = 3.89 Hz), 129.9 (q, $^{2}J_{CF}$ = 3.42 Hz), 123.5, 117.4, 116.0, 105.5. ^{19}F NMR (376 MHz, CD_3OD) δ -64.38. HRMS (ESI-positive mode): calcd for $C_{13}H_9F_3IO^+$ ([M-Br])$^+$: 364.9645, found: 364.9652.

2-Hydroxyphenyl(*bis*-3,5-trifluoromethylphenyl)iodonium bromide 4e



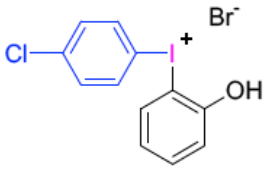
Reaction of *bis*-3,5-trifluoromethylphenylboronic acid (129 mg, 0.5 mmol) according to general procedure afforded 69 mg (27%) of product **4e**, isolated as gray amorphous solid, mp 166-167 °C. IR (ATR, cm^{-1}): 3188, 2520, 2262, 1700, 1653, 1413, 1193, 632, 543, 503, 473, 458, 420. ^{1}H NMR (400 MHz, CD_3OD) δ 8.73 (s, 2H), 8.25 (s, 1H), 8.20 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.52 (ddd, *J* = 8.6, 7.3, 1.6 Hz, 1H), 7.12 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.98 (ddd, *J* = 8.5, 7.4, 1.4). ^{13}C NMR (101 MHz, CD_3OD) δ 157.5, 138.3, 136.8, 136.7, 134.8 (q, $^{1}J_{CF}$ = 35.9 Hz), 126.8 (q, $^{2}J_{CF}$ = 3.7 Hz), 125.0, 123.6, 122.4, 117.4, 106.6. ^{19}F NMR (376 MHz, CD_3OD) δ -64.36. HRMS (ESI-positive mode): calcd for $C_{14}H_8F_6IO^+$ ($[M-Br]$)$^+$: 432.9519, found: 432.9525.

2-Hydroxyphenyl(4-cyanophenyl)iodonium bromide 4f



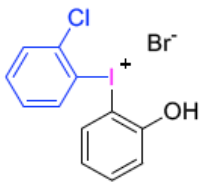
Reaction of 4-cyanophenylboronic acid (74 mg, 0.5 mmol) according to general procedure afforded 30 mg (15%) of product **4f**, isolated as light-beige or light-brown solid, mp 142-143 °C. IR (ATR, cm^{-1}): 3188, 2519, 2260, 1506, 1472, 1279, 1226, 1192, 1125, 634, 543, 443, 420. ^{1}H NMR (400 MHz, CD_3OD) δ 8.28-8.24 (m, 2H), 8.23 (dd, *J* = 8.0, 1.5, 1H), 7.86-7.82 (m, 2H), 7.70 (ddd, *J* = 8.8, 7.5, 1.5 Hz, 1H), 7.31 (dd, *J* = 8.4, 1.1 Hz, 1H), 7.12 (td, *J* = 8.0, 1.3 Hz, 1H). HRMS (ESI-positive mode): calcd for $C_{13}H_9NIO^+$ ($[M-Br]$)$^+$: 321.9723, found: 321.9733.

2-Hydroxyphenyl(4-chlorophenyl)iodonium bromide 4g



Reaction of 4-chlorophenylboronic acid (78 mg, 0.5 mmol) according to general procedure afforded 181 mg (88%) of product **4g**, isolated as light-beige or light-gray solid, mp 151-155 °C. IR (ATR, cm^{-1}): 3185, 2262, 1595, 1568, 1486, 1274, 1191, 1110, 668, 543, 476, 435. ^{1}H NMR (400 MHz, CD_3OD) δ 8.10 (dd, *J* = 8.2, 1.5 Hz, 1H), 8.08-8.04 (m, 2H), 7.54-7.48 (m, 3H), 7.11 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.95 (ddd, *J* = 8.5, 7.4, 1.4 Hz, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 157.6, 139.8, 138.1, 137.9, 136.3, 132.9, 123.4, 117.3, 113.3, 105.5. HRMS (ESI-positive mode): calcd for $C_{12}H_9Cl^{35}IO^+$ ($[M-Br]$)$^+$: 330.9381, found: 330.9374 and calcd for $C_{12}H_9Cl^{37}IO^+$ ($[M-Br]$)$^+$: 332.9337, found: 332.9352.

2-Hydroxyphenyl(2-chlorophenyl)iodonium bromide 4h

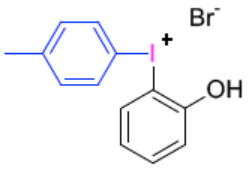


Reaction of 2-chlorophenylboronic acid (78 mg, 0.5 mmol) according to general procedure afforded 203 mg (99%) of product **4h**, isolated as white solid: mp 177-181 °C; IR (ATR, cm^{-1}): 3195, 1595, 1565, 1488, 1297, 1267, 1200, 1163, 1112, 668, 638, 546, 442, 423; ^{1}H NMR (400 MHz, CD_3OD) δ 8.28 (dd, *J* = 8.1, 1.5 Hz, 1H),

8.10 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.74 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.64 (td, *J* = 7.8, 1.5 Hz, 1H), 7.50 (ddd, *J* = 8.3, 7.3, 1.6 Hz, 1H), 7.42-7.37 (m, 1H), 7.09 (dd, *J* = 8.2, 1.4 Hz, 1H), 6.95 (ddd, *J* = 8.1, 7.4, 1.4 Hz, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 157.8, 140.1, 138.3, 138.3, 136.3, 135.4, 131.5, 130.7, 123.3, 118.4, 117.3, 105.3. HRMS (ESI-positive mode): calcd for $C_{12}H_9Cl^{35}IO^+$ ([M-Br])$^+$: 330.9381, found: 330.9371 and calcd for $C_{12}H_9Cl^{37}IO^+$ ([M-Br])$^+$: 332.9352, found: 332.9335.

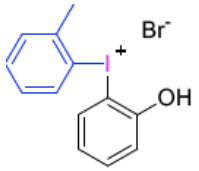
Single crystals of product **4h** suitable for X-ray crystallographic analysis were obtained by slow crystallization from MeOH solution. Crystal Data for $C_{12}H_9BrClIO$ monoclinic, space group $P2_1/c$ (no. 14), a = 10.4440(7) Å, b = 8.7604(7) Å, c = 14.3274(8) Å, β = 91.595(5)°, V = 1310.36(16) Å^3, Z = 4, T = 297.15 K, μ(Mo Kα) = 5.673 mm^{-1}, *Dcalc* = 2.086 g/cm^3, 10188 reflections measured (5.452° ≤ 2Θ ≤ 78.014°), 5047 unique (R_{int} = 0.0242, R_{sigma} = 0.0479) which were used in all calculations. The final R_1 was 0.0413 (I > 2σ(I)) and wR_2 was 0.0871 (all data). CCDC: 2505186.

2-Hydroxyphenyl(4-tolyl)iodonium bromide 4i



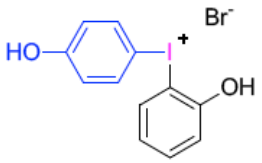
Reaction of 4-tolylboronic acid (68 mg, 0.5 mmol) according to general procedure afforded 194 mg (99%) of product **4i**, isolated as white solid, mp 190-192 °C. IR (ATR, cm^{-1}): 3104, 1594, 1568, 1486, 1297, 1268, 1171, 1123, 1103, 1066, 1048, 646, 588, 544, 492, 476, 434. ^{1}H NMR (400 MHz, CD_3OD) δ 8.04 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.96 (d, *J* = 8.4 Hz, 2H), 7.49 (ddd, *J* = 8.4, 7.3, 1.5 Hz, 1H), 7.30 (d, *J* = 8.1 Hz, 2H), 7.10 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.95-6.90 (m, 1H), 2.39 (s, 3H). ^{13}C NMR (101 MHz, CD_3OD) δ 157.5, 144.6, 137.8, 136.4, 136.1, 133.5, 123.4, 117.3, 111.9, 105.1, 21.3. HRMS (ESI-positive mode): calcd for $C_{13}H_{12}IO^+$ ([M-Br])$^+$: 310.9927, found: 310.9921.

2-Hydroxyphenyl(2-tolyl)iodonium bromide 4j



Reaction of 2-tolylboronic acid (68 mg, 0.5 mmol) according to general procedure afforded 151 mg (77%) of product **4j**, isolated as light-gray solid, mp 213-218 °C. IR (ATR, cm^{-1}): 3171, 1596, 1566, 1488, 1469, 1297, 1268, 1204, 1163, 1112, 1037, 640, 581, 539, 471, 443, 424. ^{1}H NMR (400 MHz, CD_3OD) δ 8.20 (dd, *J* = 7.8 Hz, 1H), 8.04 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.58-7.51 (m, 2H), 7.48 (ddd, *J* = 8.2, 7.3, 1.5 Hz, 1H), 7.25 (td, *J* = 7.3, 6.2, 2.7, 1H), 7.08 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.93 (ddd, *J* = 8.1, 7.5, 1.4 Hz, 1H), 2.71 (s, 3H). ^{13}C NMR (101 MHz, CD_3OD) δ 157.6, 142.7, 138.7, 137.7, 136.0, 134.1, 132.6, 130.2, 123.4, 120.5, 117.3, 104.4, 25.7. HRMS (ESI-positive mode): calcd for $C_{13}H_{12}IO^+$ ([M-Br])$^+$: 310.9927, found: 310.9926.

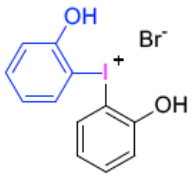
2-Hydroxyphenyl(4-hydroxyphenyl)iodonium bromide 4k



Reaction of 4-methoxyphenylboronic acid (76 mg, 0.5 mmol) with an additional excess (2.5 mmol, 2.5 mL) of BBr_3 solution in DCM (1.0 M) according to general procedure afforded 83 mg (42%) of product **4k**, isolated as light-gray solid, mp 186-188 °C. IR (ATR, cm^{-1}): 3193, 1591, 1572, 1489, 1481, 1296, 1278, 1263, 1205, 1173, 1154, 1110, 645, 595, 509, 474, 434. ^{1}H NMR (400 MHz, CD_3OD) δ 7.98 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.94-7.89 (m, 2H), 7.48 (ddd, *J* = 8.3, 7.3, 1.5 Hz, 1H), 7.09 (dd, *J* = 8.3, 1.4 Hz, 1H), 6.92 (ddd, *J* = 8.0, 7.4, 1.4 Hz, 1H), 6.88-6.83 (m, 2H). ^{13}C NMR (101 MHz, CD_3OD) δ 162.7, 157.4, 138.8, 137.4, 135.9, 123.3, 119.9, 117.2, 105.3, 101.9. HRMS (ESI-positive mode): calcd for $C_{12}H_9IO_2^+$ ($[M-Br]^+$ 312.9720, found: 312.9717.

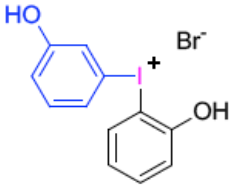
Single crystals of product **4k** suitable for X-ray crystallographic analysis were obtained by slow crystallization from MeOH solution. for $C_{12}H_{10}BrIO_2$ orthorhombic, space group Pbca (no. 61), *a* = 15.9002(17) Å, *b* = 9.9835(7) Å, *c* = 16.0963(17) Å, *V* = 2555.1(4) $Å^3$, *Z* = 8, *T* = 293(2) K, μ(Mo Kα) = 5.617 mm^{-1}, *Dcalc* = 2.043 g/cm^3, 7529 reflections measured (6.99° ≤ 2Θ ≤ 51.99°), 2426 unique (R_{int} = 0.0661, R_{sigma} = 0.1044) which were used in all calculations. The final R_1 was 0.0605 (I > 2σ(I)) and wR_2 was 0.1362 (all data). CCDC: 2505188.

***Bis*-(2-hydroxyphenyl)iodonium bromide 4l**



Reaction of 2-methoxyphenylboronic acid (76 mg, 0.5 mmol) with an additional excess (2.5 mmol, 2.5 mL) of BBr_3 solution in DCM (1.0 M) according to general procedure afforded 92 mg (47%) of product **4l**, isolated as light-brown solid, mp 165-167 °C. IR (ATR, cm^{-1}): 3133, 1594, 1566, 1482, 1294, 1265, 1192, 1181, 1107, 1035, 641, 594, 541, 471, 441, 423. ^{1}H NMR (400 MHz, CD_3OD) δ 7.93 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.47 (ddd, *J* = 8.2, 7.3, 1.6 Hz, 2H), 7.08 (dd, *J* = 8.3, 1.4 Hz, 2H), 6.91 (ddd, *J* = 8.1, 7.3, 1.4 Hz, 2H). ^{13}C NMR (101 MHz, CD_3OD) δ 157.7, 137.9, 135.7, 123.1, 117.2, 103.0. HRMS (ESI-positive mode): calcd for $C_{12}H_9IO_2^+$ ($[M-Br]^+$ 312.9720, found: 312.9728.

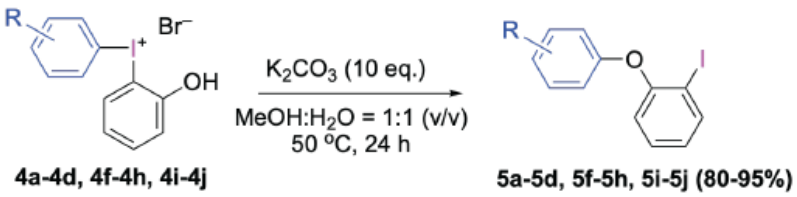
2-Hydroxyphenyl(3-hydroxyphenyl)iodonium bromide 4m



Reaction of 3-methoxyphenylboronic acid (76 mg, 0.5 mmol) with an additional excess (2.5 mmol, 2.5 mL) of BBr_3 solution in DCM (1.0 M) according to general procedure afforded 45 mg (23%) of product **4m**, isolated as

dark-brown solid, mp 140-143 °C. IR (ATR, cm^{-1}): 3178, 1594, 1473, 1299, 1198, 1156643, 548, 503, 438, 429; ^{1}H NMR (400 MHz, CD_3OD) δ 8.04 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.54-7.46 (m, 3H), 7.29 (t, *J* = 8.1 Hz, 1H), 7.12 (ddd, *J* = 8.2, 1.3 Hz, 1H), 7.02 (ddd, *J* = 8.3, 2.2, 0.9 Hz, 1H), 6.97-6.92 (m, 1H). HRMS (ESI-positive mode): calcd for $C_{12}H_9IO_2^+$ ([M-Br])$^+$ 312.9720, found: 312.9730.

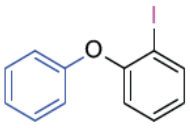
Preparation of diaryl ethers 5



4a-4d, 4f-4h, 4i-4j **5a-5d, 5f-5h, 5i-5j (80-95%)**

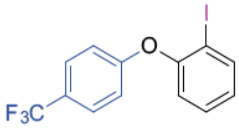
An excess of over-dried K_2CO_3 (0.69 g, 5 mmol, 10 equiv.) was added to the solution of iodonium salt **4a-4m** (0.5 mmol) in methanol-water mixture (v/v = 1:1, 2.0 mL), then the reaction mixture was stirred at ambient temperature for 24 hours. The process of reaction was monitored with TLC (Hexane : Ethyl Acetate = 1:1, $R_{f(ether)}$ = 0.6-0.7). After 24 hours absence of iodonium salt was confirmed and solvent was removed under reduced pressure. The resulting products were purified using flash column chromatography on silica gel with TLC system, separating the product, 2-iodophenol and corresponding arylphenols. Further solvent was removed under reduced pressure to give the desired diaryl ethers **5a-5m**.

2-Iodophenyl(phenyl) ether 5a



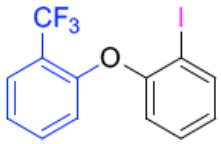
Reaction of **4a** (188 mg, 0.5 mmol) according to general procedure afforded 123 mg (83%) of product **5a**, isolated as light-brown solid: mp 53-56 °C. NMR data corresponds to the literature.[35,46] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.38-7.26 (m, 3H), 7.15-7.09 (m, 1H), 7.00-6.95 (m, 2H), 6.92-6.84 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.0, 156.6, 140.0, 129.9, 129.8, 125.5, 123.0, 119.6, 118.6, 89.1.

2-Iodophenyl(4-trifluoromethylphenyl) ether 5b



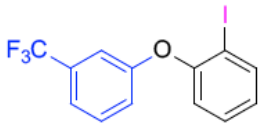
Reaction of **4b** (222 mg, 0.5 mmol) according to general procedure afforded 173 mg (95%) of product **5b**, isolated as brown solid: NMR data corresponds to the literature.[35,47] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 7.9 Hz, 1.4 Hz, 1H), 7.60 (d, *J* = 8.6 Hz, 2H), 7.39-7.35 (m, 1H), 7.03-6.97 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.0, 155.2, 140.4, 130.1, 127.4 (q, J_{CF} = 3.8 Hz), 126.8, 121.2, 117.4, 89.9; quartet from CF_3 and the quaternary carbon were not observed. ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.75.

2-Iodophenyl(2-trifluoromethylphenyl) ether 5c



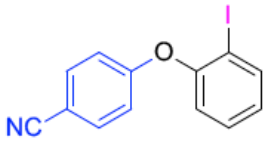
Reaction of **4c** (222 mg, 0.5 mmol) according to general procedure afforded 167 mg (92%) of product **5c**, isolated as white solid: NMR data corresponds to the literature.[48] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.70 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.44 (td, *J* = 7.2, 1.6, 1H), 7.33 (td, *J* = 7.7, 1.6 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 6.99-6.90 (m, 2H), 6.76 (d, *J* = 8.3 Hz, 1H). ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.77.

2-Iodophenyl(3-trifluoromethylphenyl) ether 5d



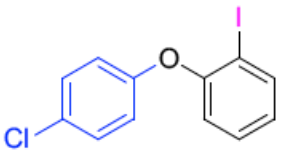
Reaction of **4d** (222 mg, 0.5 mmol) according to general procedure afforded 158 mg (87%) of product **5d**, isolated as grey solid: NMR data corresponds to the literature.[35] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.44 (t, *J* = 8.0 Hz, 1H), 7.35 (td, *J* = 7.7, 1.6 Hz, 2H), 7.21 (s, 1H), 7.11 (dd, *J* = 8.2, 2.5 Hz, 1H), 6.98-6.92 (m, 2H). ^{13}C NMR (101 MHz, CDCl3) δ 157.4, 155.6, 140.3, 132.4 (q, J_{CF} = 32.7 Hz), 130.5, 130.1, 126.5, 123.8 (q, J_{CF} = 273.5 Hz), 121.1, 120.4, 120.0 (q, J_{CF} = 3.8 Hz), 115.0 (q, J_{CF} = 3.8 Hz), 89.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.67.

2-Iodophenyl(4-cyanophenyl) ether 5f



Reaction of **4f** (201 mg, 0.5 mmol) according to general procedure afforded 146 mg (91%) of product **5f**, isolated as white solid: NMR data corresponds to the literature.[35] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (dd, J = 7.8, 1.5 Hz, 1H), 7.68-7.55 (m, 2H), 7.41 (td, J = 8.2, 1.4 Hz, 1H), 7.05 (dd, J = 8.1, 1.3 Hz, 1H), 7.01 (td, J = 7.7, 1.4 Hz, 1H), 6.97-6.88 (m, 2H). ^{13}C NMR (101 MHz, CDCl3) δ 160.9, 154.4, 140.6, 134.4, 130.4, 127.5, 121.8, 119.0, 117.6, 106.3, 90.3.

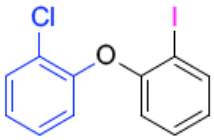
2-Iodophenyl(4-chlorophenyl) ether 5g



Reaction of **4g** (205 mg, 0.5 mmol) according to general procedure afforded 145 mg (88%) of product **5g**, isolated as grey solid: NMR data corresponds to the literature.[35] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 8.2,

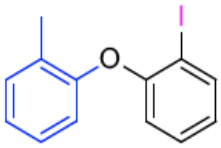
1.6 Hz, 1H), 7.34-7.27 (m, 3H), 6.92-6.87 (m, 4H). ^{13}C NMR (101 MHz, CDCl3) δ 156.2, 155.7, 140.2, 129.9, 128.6, 126.0, 119.9, 119.6, 89.2.

2-Iodophenyl(2-chlorophenyl) ether 5h



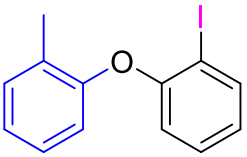
Reaction of **4h** (205 mg, 0.5 mmol) according to general procedure afforded 142 mg (86%) of product **5h**, isolated as grey solid: NMR data corresponds to the literature.[49] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.48 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.30-7.26 (m, 1H), 7.22 (td, *J* = 8.3, 1.4 Hz, 1H), 7.11 (td, *J* = 7.9, 1.2 Hz, 1H), 6.91-6.84 (m, 2H), 6.75 (dd, *J* = 8.2, 0.9 Hz, 1H).

2-Iodophenyl(4-tolyl) ether 5i



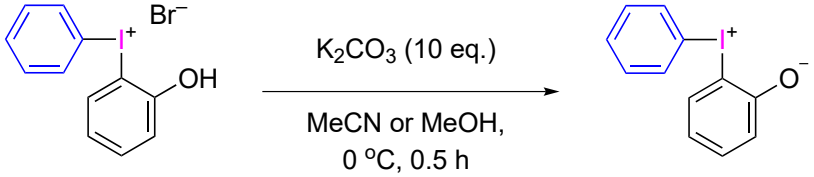
Reaction of **4i** (195 mg, 0.5 mmol) according to general procedure afforded 124 mg (80%) of product **5i**, isolated as beige solid: NMR data corresponds to the literature.[35] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.33-7.25 (m, 1H), 7.22 (t, *J* = 7.8 Hz, 1H), 6.93 (d, *J* = 7.5 Hz, 1H), 6.91-6.83 (m, 2H), 6.81 (s, 1H), 6.76 (dd, *J* = 8.1, 2.3 Hz, 1H), 2.33 (s, 3H). 13C NMR (101 MHz, $CDCl_3$) δ 156.9, 156.8, 140.2, 139.9, 129.7, 129.6, 125.3, 124.5, 119.5, 119.3, 115.6, 89.0, 21.6.

2-Iodophenyl(2-tolyl) ether 5j



Reaction of **4j** (195 mg, 0.5 mmol) according to general procedure afforded 130 mg (84%) of product **5j**, isolated as beige solid: NMR data corresponds to the literature.[35] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.28-7.20 (m, 2H), 7.17 (t, *J* = 7.3 Hz, 1H), 7.08 (t, *J* = 7.3 Hz, 1H), 6.85-6.79 (m, 2H), 6.67 (dd, *J* = 8.2, 0.9 Hz, 1H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.9, 154.3, 139.9, 131.7, 129.8, 129.6, 127.3, 124.5, 124.4, 119.3, 117.2, 87.6, 16.5.

Procedure for the synthesis of 2-hydroxyphenyl(phenyl)iodonium betaine 6a



4a **6a** (88%)

An excess of K_2CO_3 (1.38 g, 10 mmol, 10 equiv.) was added to the solution of **4a** (0.376 g, 1 mmol) in methanol or acetonitrile (2.0 mL), then the reaction mixture was stirred at 0 °C for 0.5 hour. After 0.5 hours solvent was removed under reduced pressure at r.t. The resulting residue was washed with pre-cooled (0 °C) diethyl ether (2x25 mL), then dried under reduced pressure (0 °C) to give 0.260 g (88%) of the betaine **6a** as yellowish solid, decomposes at 40-42 °C. ^{1}H NMR (400 MHz, CD_3OD) δ 8.06 (d, *J* = 7.9 Hz, 2H), 7.72 (t, *J* = 7.5 Hz, 1H), 7.6 (t, *J* = 7.7 Hz, 2H), 7.02 (t, *J* = 8.2, 1H), 6.77 (d, *J* = 7.8 Hz, 1H), 6.47 (t, *J* = 7.6 Hz, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 165.6, 137.3, 134.4, 133.2, 132.9, 131.2, 119.8, 117.5, 112.3, 111.0.

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Supplementary Material

NMR spectra of compounds are provided in the supplementary material file associated with this manuscript.

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