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Synthesis of Ionic-Liquid-Supported Diaryliodonium Salts

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The synthesis of ionic-liquid-supported diaryliodonium salts is described. The synthesis is simple and practical, and the ionic liquid products require no chromatographic purification. The ionic-liquid-supported diaryliodonium salts are quite stable, and they did not show any sign of decomposition or loss of reactivity, even after being stored for one month at 5 °C. The reactivity of these salts was explored in the phenylation of substituted phenols and carboxylic acids, and the corresponding diaryl ethers and aryl esters, respectively, were synthesized in good to excellent yields and with high purities.

Introduction

Diaryliodonium salts[1] serve as efficient and powerful electrophilic arylating agents for carbon–carbon[2] and carbon–heteroatom[3] bond-forming reactions under both metal-free and metal-catalysed conditions, due to their high electron-deficiency and their ability to act as hyper leaving groups. Recently, diaryliodonium salts have been used in multicomponent cascade annulations[4] for the synthesis of diverse heterocyclic compounds. These salts have also been used as aryl sources in the enantioselective generation of "α-carbonyl benzylic sterocentres" in the presence of chiral catalysts.[5] Symmetrical diaryliodonium salts are generally preferred over unsymmetrical salts to avoid chemoselectivity problems, but electron-rich and electron-poor symmetrical diaryliodonium salts are not straightforward to synthesize. In addition, following this strategy, an expensive aryl iodide moiety is generated as waste, which renders this approach less attractive. In contrast, it is easier to synthesize unsymmetrical salts[1d,6] than the corresponding symmetrical salts, but controlling the chemoselectivity of the reactions of these unsymmetrical salts, and purification of the reaction products, are major concerns. Moreover, every now and then the possibility arises that the aryl iodide by-product interferes with the desired reaction products, which causes severe purification problems.[7] Furthermore, some aryl iodides are very hard to recover and reuse. An alternative approach using polymer-supported diaryliodonium reagents[8] also suffers from low loading capacity, long reaction times, multistep synthesis, the inability to monitor the reaction by various analytical techniques, and a paucity of literature reports.

Ionic-liquid-supported reagents are a unique class of supported reagents with distinctive chemical and physical properties. They offer several advantages for organic synthesis: they offer the same superior product isolation and purification as is seen with solid-phase chemistry, along with the benefits of traditional solution-phase synthesis.[9] Several ionic-liquid-supported reagents, including hypervalent iodine reagents,[10] have been synthesized and used for different organic transformations. These reagents showed comparable reactivity to the corresponding unsupported reagents, and also, the biphasic system offered the advantage of simple product isolation with high purity. To date, no report on ionic-liquid-supported diaryliodonium salts is available in the literature. Consequently, there is a need to develop ionic-liquid-supported diaryliodonium salts to deal with the purification, isolation, and loading problems associated with diaryliodonium salts. Our interest in ionic-liquid-supported reagents,[11] along with the synthetic utility of diaryliodonium salts, prompted us to pursue a project devoted to the synthesis of ionic-liquid-supported diaryliodonium salts. In this paper, we wish to report the synthesis of ionic-liquid-supported diaryliodonium salts. To the best of our knowledge, this is first attempt at the synthesis of ion-tagged diaryliodonium salts.

Results and Discussion

We envisioned that tagging electron-rich aryl groups to an ionic liquid would facilitate the synthesis of ionic-liquid-supported diaryliodonium salts. Thus, two electron-rich aryl-functionalized ionic liquids **4** and **5** were synthesized as shown in Scheme 1. Alkoxy tosylates **2** and **3** were pre-

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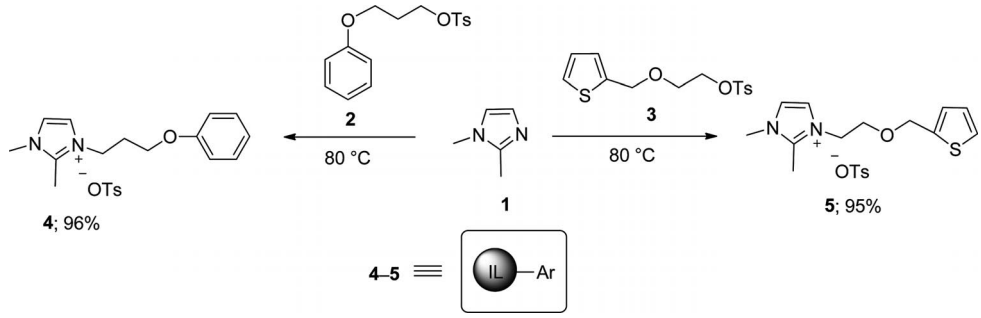


Scheme 1. Synthesis of various ionic-liquid-supported aryls.

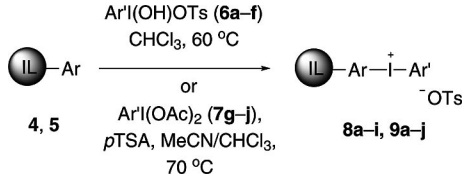
pared from phenol and thiophene-2-carbaldehyde (see Supporting Information). Reaction of **2** and **3** with 1,2-dimethylimidazole (**1**) at 80 °C gave the corresponding imidazolium-supported aryls (i.e., **4** and **5**) in excellent yields (95–96%). Simply washing the ionic-liquid layer with an ethyl acetate/hexane mixture (1:1 v/v) followed by drying under vacuum gave the pure products. The NMR spectroscopic data and MS data of the synthesized ionic liquids were consistent with the structures of **4** and **5**.

Based on Pike's[6a] and Kita's[6b] work on the synthesis of unsymmetrical iodonium salts, we anticipated that the reaction of imidazolium-supported aryls with [hydroxy(tosyloxy)iodo]arenes (HTIA) or (diacetoxyiodo)arenes would lead to the corresponding ionic-liquid-supported diaryliodonium salts. Initial attempts to synthesize ionic-liquid-supported diaryliodonium salts were made by the reaction of **4** with HTIB (**6a**) in $CHCl_3$ at room temperature, but this did not result in the formation of the expected product (i.e., **8a**). However, when the same reaction mixture was heated to reflux temperature, the desired product (i.e., **8a**) was formed in good yield. Decanting the solvent $CHCl_3$, followed by washing with tetrahydrofuran gave the product in a quite pure form, without the need for any chromatographic purification. For unsupported diaryliodonium salts, in most cases, column chromatography is obligatory for purification.[12]

The structure of **8a** was confirmed by ^1H and ^{13}C NMR spectroscopy. The ^1H NMR spectrum of **8a** showed a characteristic multiplet at δ = 8.15 ppm due to the *ortho*-protons adjacent to the iodonium ion, and a singlet peak at δ = 3.71 ppm due to the *N*-methyl group, along with other protons. In the ^{13}C NMR spectrum, a peak at δ = 106 ppm due to the C–I carbon provides clear evidence for the structure of iodonium salt **8a**.

Having established the reaction conditions for the synthesis of such ionic-liquid-supported iodonium salts, we went on to examine the possibility of accessing unsymmetrical ionic-liquid-supported iodonium salts by the reaction of **4** and **5** with various functionalized HTIAs **6a**–**6f** (Table 1). Gratifyingly, both electron-releasing (**6b**) and electron-withdrawing (**6c**–**6f**) groups were tolerated quite well on the HTIAs to give the desired products (i.e., **8a**–**8f** and **9a**–**9f**) in good to excellent yields (Table 1).

Table 1. Preparation of ionic-liquid-supported diaryliodonium salts from **4** and **5**.

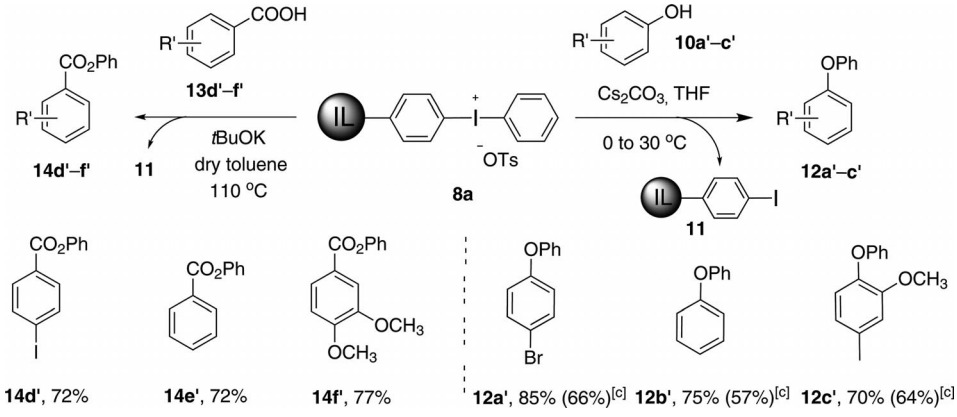


Entry	Ar	Ar′	Product	Yield [%]
1	C_6H_5	C_6H_5	**8a**	80[a]
2	C_6H_5	2,4,6-$(CH_3)_3C_6H_2$	**8b**	72[a]
3	C_6H_5	4-BrC_6H_4	**8c**	80[a]
4	C_6H_5	4-$(OCOPh)C_6H_4$	**8d**	85[a]
5	C_6H_5	4-$(CO_2CH_3)C_6H_4$	**8e**	76[a]
6	C_6H_5	4-$NO_2C_6H_4$	**8f**	72[a]
7	C_6H_5	4-$CH_3C_6H_4$	**8g**	87[b]
8	C_6H_5	2-$CH_3C_6H_4$	**8h**	86[b]
9	C_6H_5	4-$OCH_3C_6H_4$	**8i**	75[b]
10	2-C_4H_3S	C_6H_5	**9a**	72[a]
11	2-C_4H_3S	4-BrC_6H_4	**9c**	70[a]
12	2-C_4H_3S	4-$(OCOPh)C_6H_4$	**9d**	76[a]
13	2-C_4H_3S	4-$(CO_2CH_3)C_6H_4$	**9e**	70[a]
14	2-C_4H_3S	4-$NO_2C_6H_4$	**9f**	68[a]
15	2-C_4H_3S	4-$CH_3C_6H_4$	**9g**	80[b]
16	2-C_4H_3S	2-$CH_3C_6H_4$	**9h**	77[b]
17	2-C_4H_3S	4-$OCH_3C_6H_4$	**9i**	70[b]
18	2-C_4H_3S	2-$OCH_3C_6H_4$	**9j**	67[b]

[a] Reagents and reaction conditions; Method A: **4** or **5** (1.0 mmol), ArI(OH)(OTs) (1.0 mmol), $CHCl_3$ (10 mL), 62 °C, 10 h. [b] Method B: **4** or **5** (1.0 mmol), $ArI(OAc)_2$ (1.0 mmol), *p*TSA (1.0 mmol), $CH_3CN/CHCl_3$ (1:2, 10 mL), 70 °C, 12 h.

Ionic-liquid-supported diaryliodonium salts **8** and **9** could also be synthesized by the reaction of **4** and **5** with (diacetoxyiodo)arenes **7g**–**7j** and *p*-toluenesulfonic acid (*p*TSA) by generation of the corresponding HTIAs in situ. Generally, this approach is more useful for electron-rich iodoaryls, for which the synthesis of the corresponding HTIAs is difficult due to their instability. Thus, (diacetoxyiodo)arenes **7g**–**7j** were treated with **4** and **5** to give the corresponding products **8g**–**8i** and **9g**–**9j** in good to excellent yields (Table 1, entries 7–9, 15–18).

Different unsymmetrical ionic-liquid-supported diaryliodonium salts bearing functional groups such as bromo, acetoxy, nitro, methyl, and methoxy were synthesized in



Scheme 2. Phenylation of substituted phenols and carboxylic acids using ionic-liquid-supported diaryl iodonium salts **8a** and **9a**. *Reagents and reaction conditions:* (a) **10a′**–**10c′** (1 mmol), Cs_2CO_3 (1.5 mmol), THF (5 mL), **8a** (1.1 mmol), 0–30 °C, 4 h; (b) **13d′**–**13f′** (1 mmol), *t*BuOK (1.5 mmol), **8a** (1.1 mmol), dry toluene (5 mL), 110 °C, 3 h. (c) Yields of **12a′**–**12c′** when **9a** was used as arylating agent.

overall good to excellent yields (67–87%). In all cases, the products were purified by simply washing with THF (no chromatographic purification was required). The synthesized ionic-liquid-supported diaryliodonium salts were characterized by NMR spectroscopy and mass spectrometry (see Supporting Information). The yields of the diaryliodonium salts were better starting from **4** than from **5**, the latter of which uses thiophene as the aryl source. The stability of these salts is particularly noteworthy. The synthesized ionic-liquid-supported diaryliodonium salts (i.e., **8** and **9**) did not show any sign of decomposition or loss of reactivity, even after storage for one month at 5 °C.

The direct electrophilic arylation of various nucleophiles is a synthetically useful reaction of diaryliodonium salts. To demonstrate the utility of the ionic-liquid-supported diaryliodonium salts, we examined the *O*-arylation of substituted phenols and acids under metal-free conditions (Scheme 2). Initial attempts to optimize the reaction conditions for the *O*-arylation of phenols using **8a** were made using 4-bromophenol (**10a′**) as a model substrate. When **10a′** was treated with potassium *tert*-butoxide at 0 °C, followed by the addition of **8a** at room temperature, 1-bromo-4-phenoxybenzene (**12a′**) was formed in 70% yield. After further optimization of the reaction conditions for this transformation by varying solvents and bases, we found that the best yields for this and other phenol starting materials were obtained by using THF as solvent and Cs_2CO_3 as base. For the similar arylation of carboxylic acid starting materials, the best yields were obtained by using toluene as solvent and *t*BuOK as base.

Having established standard reaction conditions, we set out to survey a range of phenols **10a′**–**10c′** and carboxylic acids **13d′**–**13f′** towards *O*-arylation (Scheme 2). Interestingly, both phenols and carboxylic acids reacted smoothly with **8a** to give the corresponding diaryl ethers (i.e., **12**) and aryl esters (i.e., **14**), respectively, in good to excellent yields (70–85%). A significant advantage of this protocol is that the products are obtained in excellent purity without the need for chromatographic purification. After completion of the reaction, the solvent is evaporated, and the product can be extracted with a hexane/ethyl acetate mixture. This leaves behind the by-product supported on the ionic liquid. *O*-Arylation of phenols using **9a** gave the desired products (i.e., **12a′**–**12c′**) in yields slightly lower than those obtained with **8a**. The recovered ionic-liquid-supported iodophenyl (i.e., **11**; Scheme 2) can potentially be reused as a supported reagent in many other organic transformations,[13] and can also serve as a starting material to synthesize ionic-liquid-supported hypervalent iodine reagents.[10]

Conclusions

In summary, we have designed and synthesized ionic-liquid-supported diaryliodonium salts. The ionic-liquid-supported diaryliodonium salts were prepared by two approaches: i) using HTIAs; and ii) using (diacetoxyiodo)-arenes and *p*-toluenesulfonic acid, where the HTIAs were generated in situ. One of the salient features of this protocol is that chromatographic purification is avoided. The electrophilic phenylation of phenols and carboxylic acids by ionic-liquid-supported diaryliodonium salt **8a** was demonstrated, and the products were purified by simple extraction with a hexane/ethyl acetate mixture. The by-product of the reaction, ionic-liquid-supported iodophenyl **11**, was easily isolated, and can be further reused as a reagent in different organic transformations.[13]

Experimental Section

General Remarks: NMR spectra were recorded with 300 and 400 MHz spectrometers using $CDCl_3$ and [D_6]DMSO as solvents. Chemical shifts are expressed in ppm. The purity of the products was determined by TLC, carried out on silica-coated aluminum plates. Melting points were determined using open capillary tubes and an automated melting-point apparatus. 1,2-Dimethylimidazole, aryl iodides, and other reagents and solvents were purchased from commercial suppliers, and were used without further purification unless otherwise specified.

General Procedure for the Synthesis of Ionic-Liquid-Supported Diaryliodonium Salts 8a–8f and 9a–9f from Various HTIA (Method

A): HTIA (1.0 mmol) was added to the ionic-liquid-supported arene **4** or **5** (1.0 mmol) in $CHCl_3$ (10 mL), and the resulting reaction mixture was heated at reflux for 10 h. The progress of the reaction was monitored by TLC. After the reaction was complete, the $CHCl_3$ was decanted, and the crude product was washed with THF (3 × 5 mL) to remove unreacted starting materials. The resulting compound was dried under reduced pressure to give the desired product. The purity of the compounds was adequate, and further purification by column chromatography was not required.

8a: White solid (648 mg, 80%), m.p. 149–157 °C. ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.28–8.09 (m, 4 H), 7.65 (d, J = 2.1 Hz, 1 H), 7.63 (d, J = 7.4 Hz, 1 H), 7.60 (d, J = 2.0 Hz, 1 H), 7.51 (d, J = 8.0 Hz, 2 H), 7.48 (d, J = 8.1 Hz, 4 H), 7.10 (d, J = 7.9 Hz, 4 H), 7.03 (d, J = 9.1 Hz, 2 H), 4.25 (t, J = 6.9 Hz, 2 H), 4.03 (t, J = 5.9 Hz, 2 H), 3.71 (s, 3 H), 2.52 (s, 3 H), 2.28 (s, 6 H), 2.23–2.11 (m, 2 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 161.3, 146.1, 144.9, 138.1, 137.7, 135.3, 132.3, 132.1, 128.5, 125.9, 122.8, 121.4, 118.3, 117.5, 106.2, 65.3, 45.1, 35.1, 28.9, 21.2, 9.6 ppm.

8b: Off-white viscous liquid (588 mg, 72%). ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 7.90 (d, J = 8.9 Hz, 2 H), 7.65 (d, J = 1.8 Hz, 1 H), 7.60 (d, J = 1.7 Hz, 1 H), 7.46 (d, J = 7.9 Hz, 4 H), 7.18 (s, 2 H), 7.10 (d, J = 7.8 Hz, 4 H), 7.00 (d, J = 8.9 Hz, 2 H), 4.25 (t, J = 6.8 Hz, 2 H), 4.02 (t, J = 5.8 Hz, 2 H), 3.71 (s, 3 H), 2.59 (s, 6 H), 2.53 (s, 3 H), 2.28 (s, 9 H), 2.22–2.09 (m, 2 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 161.0, 146.1, 144.9, 143.3, 141.8, 138.1, 137.0, 130.1, 128.5, 125.9, 123.6, 122.8, 121.4, 118.3, 104.1, 65.3, 55.4, 45.1, 35.1, 28.9, 26.7, 21.2, 20.9, 9.6 ppm.

8c: White solid (683 mg, 80%), m.p. 152–159 °C. ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.18–8.15 (m, 4 H), 7.72 (d, J = 8.6 Hz, 2 H), 7.65 (d, J = 1.9 Hz, 1 H), 7.60 (d, J = 1.9 Hz, 1 H), 7.47 (d, J = 8.0 Hz, 4 H), 7.10 (d, J = 7.9 Hz, 4 H), 7.04 (d, J = 9.0 Hz, 2 H), 4.26 (t, J = 7.0 Hz, 2 H), 4.04 (t, J = 5.9 Hz, 2 H), 3.72 (s, 3 H), 2.53 (s, 3 H), 2.28 (s, 6 H), 2.12–2.10 (m, 2 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 161.3, 146.1, 144.9, 138.1, 137.7, 137.3, 134.9, 128.5, 126.41, 125.9, 122.8, 121.4, 118.3, 116.1, 106.5, 65.3, 45.1, 35.1, 28.9, 21.2, 9.6 ppm.

8d: White solid (761 mg, 85%), m.p. 155–162 °C. ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.32 (d, J = 7.1 Hz, 2 H), 8.21 (d, J = 6.9 Hz, 2 H), 8.11 (d, J = 5.5 Hz, 2 H), 7.82–7.72 (m, 1 H), 7.63 (d, J = 11.5 Hz, 4 H), 7.47 (d, J = 5.6 Hz, 6 H), 7.16–6.99 (m, 6 H), 4.33–4.19 (m, 2 H), 4.11–4.01 (m, 2 H), 3.71 (s, 3 H), 2.50 (s, 3 H), 2.27 (s, 6 H), 2.22–2.12 (m, 2 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 164.7, 161.3, 153.6, 146.1, 144.9, 138.1, 137.7, 137.1, 134.9, 130.4, 129.5, 128.8, 128.5, 126.1, 125.9, 122.84, 121.4, 118.3, 113.8, 106.6, 65.3, 45.1, 35.1, 28.9, 21.2, 9.6 ppm.

8e: Brown viscous liquid (633 mg, 76%). ^{1}H NMR (300 MHz, [D$_6$]-DMSO): δ = 8.32 (d, J = 7.8 Hz, 2 H), 8.20 (d, J = 8.0 Hz, 2 H), 7.99 (d, J = 7.5 Hz, 2 H), 7.65 (d, J = 2.0 Hz, 1 H), 7.60 (d, J = 2.0 Hz, 1 H), 7.48 (d, J = 7.1 Hz, 4 H), 7.10 (d, J = 7.2 Hz, 4 H), 7.05 (d, J = 8.3 Hz, 2 H), 4.26 (t, J = 7.0 Hz, 2 H), 4.04 (t, J = 5.9 Hz, 2 H), 3.86 (s, 3 H), 3.71 (s, 3 H), 2.53 (s, 3 H), 2.28 (s, 6 H), 2.23–2.11 (m, 2 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]-DMSO): δ = 165.6, 161.4, 146.1, 144.9, 138.1, 137.9, 135.7, 132.8, 132.2, 128.5, 125.9, 122.8, 122.3, 121.4, 118.4, 106.2, 65.3, 53.2, 45.0, 35.1, 28.9, 21.2, 9.6 ppm.

8f: Pale brown viscous liquid (590 mg, 72%). ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.44 (d, J = 9.0 Hz, 2 H), 8.29–8.20 (m, 4 H), 7.65 (d, J = 2.0 Hz, 1 H), 7.60 (d, J = 2.0 Hz, 1 H), 7.47 (d, J = 8.0 Hz, 4 H), 7.10 (d, J = 7.9 Hz, 4 H), 7.06 (d, J = 9.1 Hz, 2 H), 4.26 (t, J = 7.0 Hz, 2 H), 4.05 (t, J = 5.9 Hz, 2 H), 3.72 (s, 3 H), 2.53 (s, 3 H), 2.28 (s, 6 H), 2.22–2.25 (m, 2 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 161.5, 149.7, 146.1, 144.9, 138.1, 138.1, 136.6, 128.5, 126.5, 125.9, 123.7, 122.8, 121.4, 118.4, 106.5, 65.3, 45.1, 35.1, 28.9, 21.2, 9.6 ppm.

9a: Yellow viscous liquid (562 mg, 72%). ^{1}H NMR (300 MHz, [D$_6$]-DMSO): δ = 8.24 (d, J = 7.7 Hz, 2 H), 7.94 (d, J = 3.7 Hz, 1 H), 7.70–7.51 (m, 5 H), 7.48 (d, J = 8.0 Hz, 4 H), 7.11 (d, J = 7.8 Hz, 4 H), 7.04 (d, J = 3.6 Hz, 1 H), 4.74 (s, 2 H), 4.33 (t, J = 4.6 Hz, 2 H), 3.79–3.65 (m, 5 H), 2.53 (s, 3 H), 2.28 (s, 6 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 152.2, 146.0, 145.2, 140.5, 138.2, 135.1, 132.5, 132.1, 128.6, 128.4, 125.9, 122.7, 121.7, 119.9, 100.7, 68.5, 66.8, 47.8, 35.2, 21.2, 9.8 ppm.

9c: Brown viscous liquid (602 mg, 70%). ^{1}H NMR (300 MHz, [D$_6$]-DMSO): δ = 8.18 (d, J = 8.6 Hz, 2 H), 7.95 (d, J = 3.8 Hz, 1 H), 7.74 (d, J = 8.6 Hz, 2 H), 7.60 (d, J = 1.9 Hz, 1 H), 7.57 (d, J = 2.1 Hz, 1 H), 7.48 (d, J = 8.0 Hz, 4 H), 7.11 (d, J = 7.9 Hz, 4 H), 7.06 (d, J = 3.8 Hz, 1 H), 4.75 (s, 2 H), 4.34 (t, J = 4.8 Hz, 2 H), 3.79–3.70 (m, 5 H), 2.53 (s, 3 H), 2.28 (s, 6 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 152.4, 146.0, 145.2, 140.7, 138.2, 137.1, 135.0, 128.5, 128.4, 126.6, 125.9, 122.7, 121.7, 118.5, 101.0, 68.5, 66.8, 47.8, 35.2, 21.2, 9.8 ppm.

9d: Brown viscous liquid (685 mg, 76% yield). ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.36 (d, J = 7.9 Hz, 2 H), 8.11 (d, J = 7.4 Hz, 2 H), 7.98 (s, 1 H), 7.77 (t, J = 6.9 Hz, 1 H), 7.60 (dd, J = 15.5, 6.4 Hz, 4 H), 7.48 (d, J = 6.1 Hz, 6 H), 7.11 (d, J = 7.4 Hz, 4 H), 7.06 (d, J = 3.7 Hz, 1 H), 4.76 (s, 2 H), 4.31–4.37 (m, 2 H), 3.69–3.79 (m, 5 H), 2.53 (s, 3 H), 2.27 (s, 6 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 164.7, 153.7, 152.4, 145.9, 145.2, 140.7, 138.3, 136.9, 134.9, 130.4, 129.5, 128.8, 128.6, 128.4, 126.0, 125.9, 122.7, 121.7, 116.1, 101.0, 68.5, 66.9, 47.8, 35.2, 21.2, 9.8 ppm.

9e: White solid (587 mg, 70%), m.p. 170–173 °C. ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.36 (d, J = 8.4 Hz, 2 H), 8.03 (d, J = 8.4 Hz, 2 H), 7.98 (d, J = 3.7 Hz, 1 H), 7.60 (d, J = 1.7 Hz, 1 H), 7.56 (d, J = 1.6 Hz, 1 H), 7.47 (d, J = 7.9 Hz, 4 H), 7.11 (d, J = 7.8 Hz, 4 H), 7.07 (d, J = 4.0 Hz, 1 H), 4.76 (s, 2 H), 4.34 (t, J = 4.6 Hz, 2 H), 3.87 (s, 3 H), 3.75–3.70 (m, 5 H), 2.54 (s, 3 H), 2.28 (s, 6 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 165.6, 160.0, 158.1, 152.6, 146.1, 145.2, 141.0, 138.1, 135.4, 133.0, 132.3, 128.5, 125.9, 124.5, 122.7, 121.7, 68.5, 66.9, 53.2, 47.8, 35.2, 21.2, 9.8 ppm.

9f: Pale brown viscous liquid (562 mg, 68% yield). ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.49 (d, J = 8.7 Hz, 2 H), 8.28 (d, J = 8.7 Hz, 2 H), 8.02 (d, J = 3.6 Hz, 1 H), 7.63–7.60 (m, 1 H), 7.59–7.55 (m, 1 H), 7.47 (d, J = 7.8 Hz, 4 H), 7.11 (d, J = 7.8 Hz, 4 H), 7.08–7.06 (m, 1 H), 4.76 (s, 2 H), 4.38–4.30 (m, 2 H), 3.75–3.70 (m, 5 H), 2.54 (s, 3 H), 2.28 (s, 6 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]-DMSO): δ = 152.9, 149.7, 146.0, 145.2, 141.3, 138.2, 136.4, 128.6, 128.5, 126.6, 125.9, 122.7, 121.7, 101.0, 68.6, 66.9, 47.8, 35.2, 21.2, 9.8 ppm.

General Procedure for the Synthesis of Ionic-Liquid-Supported Diaryliodonium Salts (8g–8i and 9g–9j) from Various (Diacetoxyiodo)-arenes (Method B): *para*-Toluenesulfonic acid (1.0 mmol) was added to a suspension of (diacetoxyiodo)arene (1.0 mmol) in MeCN (4.0 mL) to give an intensely yellow solution, which was further diluted with $CHCl_3$. Ionic-liquid-supported arene **4** or **5** (1.0 mmol) was added to the reaction mixture, and the resulting pale yellow solution was heated at reflux for 12 h. After TLC indicated that the reaction was complete, the solvent was evaporated. The residue was washed with diethyl ether (2 × 5 mL) and THF (3 × 5 mL), and then dried under reduced pressure to give the pure ionic-liquid-supported diaryliodonium salt.

8g: White solid (687 mg, 87%), m.p. 160–166 °C. ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.14–8.06 (m, 4 H), 7.65 (d, J = 2.2 Hz, 1 H), 7.60 (d, J = 2.1 Hz, 1 H), 7.48 (d, J = 8.1 Hz, 4 H), 7.31 (d, J = 8.2 Hz, 2 H), 7.10 (d, J = 7.8 Hz, 4 H), 7.02 (d, J = 9.1 Hz, 2 H), 4.25 (t, J = 6.9 Hz, 2 H), 4.03 (t, J = 6.0 Hz, 2 H), 3.71 (s, 3 H), 2.52 (s, 3 H), 2.33 (s, 3 H), 2.28 (s, 6 H), 2.24–2.11 (m, 2 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 161.2, 146.2, 144.9, 142.7, 138.1, 137.6, 135.3, 132.7, 128.5, 125.9, 122.8, 121.4, 118.21, 113.9, 106.4, 65.3, 45.1, 35.1, 28.9, 21.3, 21.2, 9.6 ppm.

8h: Colourless viscous liquid (679 mg, 86%). ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.36 (d, J = 7.9 Hz, 1 H), 8.12 (d, J = 8.9 Hz, 2 H), 7.65 (d, J = 2.0 Hz, 1 H), 7.60 (d, J = 2.0 Hz, 1 H), 7.54 (d, J = 6.5 Hz, 2 H), 7.48 (d, J = 8.0 Hz, 4 H), 7.32–7.23 (m, 1 H), 7.10 (d, J = 7.9 Hz, 4 H), 7.00 (d, J = 9.0 Hz, 2 H), 4.25 (t, J = 6.8 Hz, 2 H), 4.02 (t, J = 5.9 Hz, 2 H), 3.70 (s, 3 H), 2.60 (s, 3 H), 2.51 (s, 3 H), 2.27 (s, 6 H), 2.22–2.09 (m, 2 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 161.2, 146.0, 144.9, 140.8, 138.2, 137.57, 137.4, 133.1, 131.7, 129.6, 128. 6, 125.9, 122.8, 122.5, 121.4, 118.3, 105.6, 65.3, 45.0, 35.1, 28.9, 25.4, 21.2, 9.5 ppm.

8i: Pale yellow viscous liquid (604 mg, 75%). ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.12 (d, J = 8.7 Hz, 4 H), 7.64 (s, 1 H), 7.59 (s, 1 H), 7.47 (d, J = 7.2 Hz, 4 H), 7.10 (d, J = 7.4 Hz, 4 H), 7.03 (t, J = 7.9 Hz, 4 H), 4.25 (t, J = 6.5 Hz, 2 H), 4.09–3.94 (m, 2 H), 3.78 (s, 3 H), 3.71 (s, 3 H), 2.52 (s, 3 H), 2.27 (s, 6 H), 2.22–2.11 (m, 2 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 162.3, 161.1, 146.1, 144.9, 138.1, 137.4, 137.4, 128.5, 125.9, 122.8, 121.4, 118.2, 117.8, 106.8, 106.5, 65.2, 56.2, 45.1, 35.1, 25.59, 21.2, 9.6 ppm.

9g: Pale brown viscous liquid (636 mg, 80%). ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.11 (d, J = 7.2 Hz, 2 H), 7.90 (s, 1 H), 7.57 (d, J = 9.3 Hz, 2 H), 7.48 (d, J = 7.2 Hz, 4 H), 7.32 (d, J = 7.6 Hz, 2 H), 7.11 (d, J = 7.3 Hz, 4 H), 7.03 (s, 1 H), 4.73 (s, 2 H), 4.27–4.38 (m, 2 H), 3.81–3.67 (m, 5 H), 2.52 (s, 3 H), 2.33 (s, 3 H), 2.28 (s, 6 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 152.0, 145.9, 145.2, 143.0, 140.3, 138.2, 135.1, 132.7, 128.6, 128.4, 125.9, 122.7, 121.7, 116.3, 100.9, 68.5, 66.8, 47.8, 35.2, 21.32, 21.2, 9.8 ppm.

9h: Pale brown viscous liquid (612 mg, 77% yield). ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.41 (d, J = 7.9 Hz, 1 H), 7.91 (d, J = 3.8 Hz, 1 H), 7.60 (t, J = 2.5 Hz, 2 H), 7.57 (t, J = 2.5 Hz, 2 H), 7.47 (d, J = 8.0 Hz, 4 H), 7.35–7.28 (m, 1 H), 7.11 (d, J = 7.9 Hz, 4 H), 7.03 (d, J = 3.8 Hz, 1 H), 4.73 (s, 2 H), 4.33 (t, J = 4.9 Hz, 2 H), 3.75–3.72 (m, 5 H), 2.63 (s, 3 H), 2.53 (s, 3 H), 2.28 (s, 6 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 151.9, 146.1, 145.2, 140.4, 140.2, 138.1, 137.2, 133.4, 131.9, 129.8, 128.5, 128.4, 125.9, 124.9, 122.7, 121.7, 100.1, 68.5, 66.8, 47.8, 35.2, 25.4, 21.2, 9.8 ppm.

9i: Pale brown viscous liquid (568 mg, 70%). ^{1}H NMR (300 MHz, [D$_6$]DMSO): δ = 8.17 (d, J = 9.1 Hz, 2 H), 7.89 (d, J = 3.8 Hz, 1 H), 7.60 (d, J = 2.2 Hz, 1 H), 7.56 (d, J = 2.1 Hz, 1 H), 7.47 (d, J = 8.1 Hz, 4 H), 7.14–7.06 (m, 5 H), 7.07–7.01 (m, 2 H), 4.74 (s, 2 H), 4.34 (t, J = 4.9 Hz, 2 H), 3.80 (s, 3 H), 3.77–3.72 (m, 5 H), 2.54 (s, 3 H), 2.28 (s, 6 H) ppm. ^{13}C NMR (75 MHz, [D$_6$]DMSO): δ = 162.4, 151.8, 146.2, 145.2, 140.0, 138.1, 137.3, 128.5, 128.3, 126.0, 122.7, 121.7, 117.8, 108.9, 101.4, 68.5, 66.8, 56.2, 47.8, 35.2, 21.2, 9.8 ppm.

9j: Brown viscous liquid (543 mg, 67%). ^{1}H NMR (300 MHz, [D$_6$]-DMSO): δ = 8.11 (d, J = 7.2 Hz, 2 H), 7.90 (s, 1 H), 7.57 (d, J = 9.3 Hz, 2 H), 7.48 (d, J = 7.2 Hz, 4 H), 7.32 (d, J = 7.6 Hz, 2 H), 7.11 (d, J = 7.3 Hz, 4 H), 7.03 (s, 1 H), 4.73 (s, 2 H), 4.33 (s, 2 H), 3.97 (s, 3 H), 3.75–3.74 (m, 5 H), 2.52 (s, 3 H), 2.28 (s, 6 H) ppm. ^{13}C NMR (75 MHz, DMSO): δ = 156.3, 151.6, 145.9, 145.2, 140.2, 138.3, 137.3, 135.5, 128.6, 128.2, 125.9, 123.9, 122.7, 121.7, 113.4, 109.9, 100.0, 68.5, 66.8, 57.5, 47.8, 35.2, 21.2, 9.8 ppm.

General Procedure for *O*-Phenylation of Substituted Phenols: Substituted phenol **10** (1.0 mmol) was dissolved in THF (5.0 mL), and Cs_2CO_3 (1.5 mmol) was added slowly at 0 °C. The mixture was stirred for 20 min, then it was treated with ionic-liquid-supported iodonium salt **8a** or **9a** (1.1 mmol). The resulting solution was stirred at room temperature for 4 h. After the reaction was complete, the THF was evaporated, and the product was extracted with hexane/ethyl acetate (1:1 v/v). The organic phase was washed with water, dried with anhydrous sodium sulfate, and concentrated to give the desired product.

12a′: Colourless liquid (211 mg, 85%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.43 (d, J = 2.2 Hz, 1 H), 7.40 (d, J = 2.2 Hz, 1 H), 7.37–7.30 (m, 2 H), 7.12 (dt, J = 7.0, 1.08 Hz, 1 H), 7.01 (q, J = 1.8 Hz, 1 H), 6.99–6.97 (m, 1 H), 6.89 (d, J = 2.2 Hz, 1 H), 6.86 (d, J = 2.2 Hz, 1 H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 156.7, 156.6, 132.7, 129.9, 123.7, 120.4, 119.0, 115.6 ppm.

12b′: Colourless liquid (121 mg, 75%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.33 (t, J = 2.2 Hz, 1 H), 7.32–7.29 (m, 2 H), 7.29–7.26 (m, 1 H), 7.10–7.04 (m, 2 H), 7.01 (t, J = 1.6 Hz, 2 H), 6.99–6.97 (m, 2 H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 157.3, 129.8, 123.3, 119.0 ppm.

12c′: White solid (149 mg, 70%), m.p. 70–72 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.30–7.22 (m, 2 H), 7.04–6.96 (m, 1 H), 6.93 (t, J = 1.6 Hz, 1 H), 6.90 (dd, J = 3.1, 2.1 Hz, 1 H), 6.86 (s, 1 H), 6.81 (d, J = 1.6 Hz, 1 H), 6.72 (dd, J = 8.0, 1.2 Hz, 1 H), 3.79 (s, 3 H), 2.35 (s, 3 H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 158.4, 151.3, 142.4, 134.9, 129.4, 122.1, 121.5, 121.3, 116.7, 113.8, 55.9, 21.3 ppm.

General Procedure for *O*-Phenylation of Substituted Benzoic Acids: A mixture of substituted benzoic acid **13** (1 mmol), *t*BuOK (1.5 mmol), and ionic-liquid-supported diphenyliodonium salt **8a** (1.1 mmol) in dry toluene (5 mL) was heated at reflux under a nitrogen atmosphere. The progress of the reaction was monitored by TLC. After the reaction was complete, the toluene was evaporated, and the product was extracted using a hexane/ethyl acetate mixture (1:1 v/v). The combined organic layers were washed with saturated sodium hydrogen carbonate solution, dried with anhydrous sodium sulfate, and evaporated under reduced pressure to give the pure *O*-phenylated product.

14d′: Pale brown solid; m.p. 119–124 °C (233 mg, 72%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.89 (s, 4 H), 7.42 (d, J = 6.7 Hz, 2 H), 7.35–7.14 (m, 3 H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 164.7, 150.8, 138.0, 131.5, 129.5, 129.1, 126.1, 121.6, 101.6 ppm.

14e′: White solid, m.p. 58–60 °C; (142 mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.22 (d, J = 7.5 Hz, 2 H), 7.64 (t, J = 7.4 Hz, 1 H), 7.51 (t, J = 7.7 Hz, 2 H), 7.44 (t, J = 7.8 Hz, 2 H), 7.27 (dd, J = 12.7, 5.1 Hz, 1 H), 7.22 (d, J = 8.0 Hz, 2 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 165.3, 151.1, 133.7, 130.3, 129.7, 129.6, 128.7, 126.0, 121.8 ppm.

14f′: Colourless solid; m.p. 129–132 °C; (198 mg, 72%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.87 (dd, J = 8.4, 2.0 Hz, 1 H), 7.68 (d, J = 1.9 Hz, 1 H), 7.43 (t, J = 7.8 Hz, 2 H), 7.30–7.17 (m, 3 H), 6.95 (d, J = 8.5 Hz, 1 H), 3.97 (s, 3 H), 3.96 (s, 3 H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 165.0, 153.6, 151.1, 148.8, 129.5, 125.8, 124.4, 122.0, 121.8, 112.4, 110.4, 56.1, 56.1 ppm.

11: Off-white solid; m.p. 71–75 °C (369 mg, 70%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.63 (d, J = 8.1 Hz, 2 H), 7.44 (d, J = 8.9 Hz, 2 H), 7.35 (s, 2 H), 7.06 (d, J = 8.0 Hz, 2 H), 6.54 (d, J = 8.9 Hz, 2 H), 4.20 (t, J = 6.9 Hz, 2 H), 3.78 (t, J = 5.6 Hz, 2 H), 3.69 (s, 3 H), 2.47 (s, 3 H), 2.27 (s, 3 H), 2.18–2.05 (m, 2 H) ppm.

^{13}C NMR (75 MHz, $CDCl_3$): δ = 158.1, 144.0, 143.6, 139.4, 138.3, 128.7, 125.8, 122.9, 121.2, 116.9, 83.1, 64.1, 45.3, 35.3, 28.9, 21.3, 9.6 ppm.

Supporting Information (see footnote on the first page of this article): Experimental details, spectroscopic data, and copies of ^1H and ^{13}C NMR spectra for compounds **2**–**5**, **8**, **9**, **11**, **12**, and **14**.

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