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# Evolving utility of C($sp^3$)-substituted hypervalent iodine reagents: improved synthesis of aryl((arylsulfonyl)methyl)iodoniums

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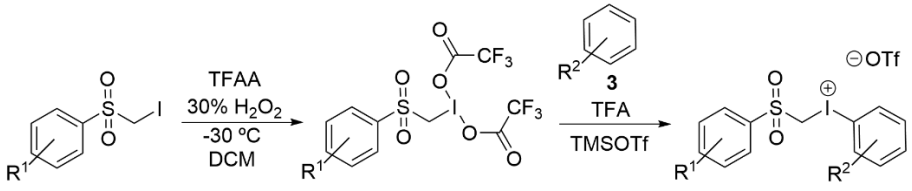
## Abstract

An improved synthesis of aryl((arylsulfonyl)methyl)iodonium triflates is reported. Aryl((arylsulfonyl)methyl) iodonium triflates are examples of a rare moiety: a $sp^3$ carbon bonded to an iodine(III) atom. The rarity of such a bond is related to the nature of the 3-center-4-electron bond of the hypervalent iodine. The improvement to the original synthetic procedure modifies a crucial step in which a bis(trifluoroacetate) iodine(III) intermediate forms, and it removes residual water by lyophilization. Multiple previously unreported aryl((arylsulfonyl)methyl)iodonium triflates are synthesized, and an updated reaction profile for their stability was investigated.

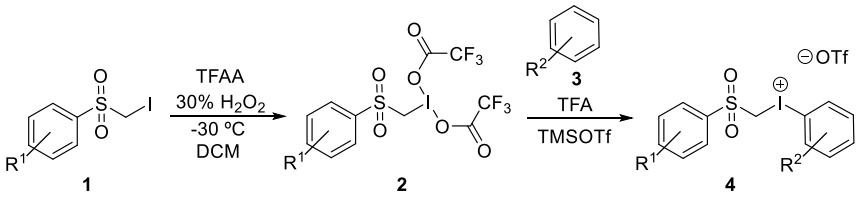



**Keywords:** Hypervalent iodine, iodine(III), sulfonyl, sulfone, 3-center-4-electron bond

## Introduction

Progress on methylsulfonyl-stabilized hypervalent iodine(III) (HVI) molecules as modular alternatives to aryl synthons has not been explored since the 1997 publication that first reported them.[1] The lack of examples of aryl((arylsulfonyl)methyl)iodonium triflates in chemical literature can be explained by the inherent instability of all non-fluorinated $sp^3$-hybridized carbons bound to hypervalent iodine atoms, and the difficulty in reproducing the previously reported procedure.[2] Herein, we demonstrate how modifications to the original procedure may lead to the emergence of HVI molecules as new tools in the organic chemist's toolbox (Scheme 1).




**Scheme 1**. Synthesis of aryl((arylsulfonyl)methyl)iodoniums.

Hypervalent iodine(III) compounds are stabilized by low electronegativity, high polarizability, and accessible valence orbitals. The appeal of HVI reagents arises from their ability to enable transformations without reliance on expensive, toxic transition metals and from the predominance of p-orbital-driven, main-group reactivity in their mechanistic manifold.[3–5] Iodine(III) complexes exhibit characteristics of a 3c-4e bonding pattern that holds them in a T-shaped structure that equilibrates based on trans-influence behavior. Alkylated-HVI molecules are unstable because the 3-center-4-electron (3c-4e) bond creates a highly polarized I-C dipole that classifies as a hypernucleofuge.[6] Only a few examples of stable alkylated-HVI species exist,[7–10] the most prevalent of which are Togni's reagents[11,12] and similarly (perfluoroalkyl)phenyliodonium triflates, or so-called FITS reagents. The stability of FITS reagents is a result of the 3c-4e bonding and the electron-withdrawing capabilities of the fluoroalkane.[2] The electron donation of an alkyl group from aryl((arylsulfonyl)methyl)iodoniums is opposite to that of the FITS reagents, and thus the major stability contribution is largely from sulfonyl group sterics, with only minor electronic effects.[13]

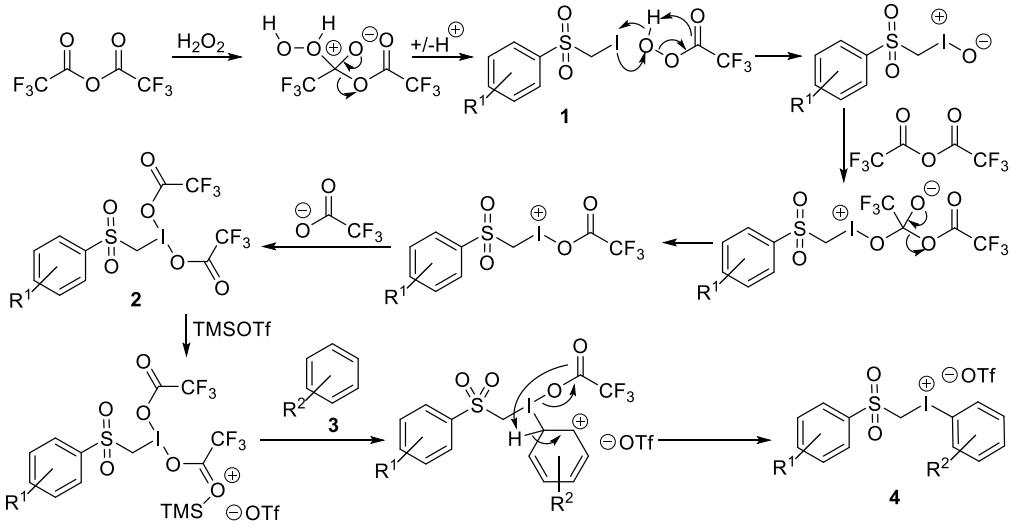
In the original procedure[1] by Zhdankin *et al.*, iodomethyl sulfones (**1**) act as precursors to ((arylsulfonyl)methyl)(phenyl)iodonium triflates (**4**) and are easily synthesized from commercially available starting materials.[14–16] The iodomethyl sulfone (**1**) is added to a hydrogen peroxide, trifluoroacetic acid, and trifluoroacetic anhydride solution (*in situ* generation of trifluoroperacetic acid) to form an iodine(III) bis-trifluoroacetate intermediate (**2**). Keeping the reaction mixture cool is critical to prevent thermal decomposition of **2**. TMSOTf is added to the reaction mixture, and the products (**4**) were isolated through precipitation.

The major changes between the original procedure and our revised one are that a vacuum line is attached to the reaction flask and that excess TMSOTf is used to overpower any residual water, thus ensuring a full equivalent of Lewis acid is added. In the improved procedure, the reaction mixture is placed under a vacuum of approximately 2 torr until the solution becomes a paste. Once the vacuum is removed, the arene and TMSOTf are added to the flask. A final evaporation of solvents and subsequent precipitation of the crude mixture with diethyl ether or a combination of ether, hexane, and dichloromethane afforded the desired aryl((arylsulfonyl)methyl)iodonium. Other revisions to the procedure include using 30% hydrogen peroxide instead of 80% hydrogen peroxide (Zhdankin *et al.* actually used a 50% solution, as confirmed by direct communication).

## Results and Discussion

The synthesis of the aryl-substituted λ$^3$-iodanes (**4**) proceeds through three distinct mechanistic steps (Scheme 2): oxidation of trifluoroacetic anhydride to trifluoroperacetic acid, *in situ* formation of a bis-trifluoroacetate iodo-λ$^3$ intermediate (**2**), and an electrophilic addition of an arene (**3**). The production and use of trifluoroperacetic acid is critical for the formation of the active iodine(III) species. Attempts to use commercially available peracetic acid failed to yield **4**; only the starting material was obtained. Other oxidants that failed to produce **4** included mCPBA, Selectfluor, Oxone, and potassium persulfate, even when solvents and additives (NaOTf, NaBF4) were varied. Crude NMRs indicated iodo-transfer from the iodomethylsulfone (**1**) to the arene (**3**) (i.e., iodobenzene produced when arene (**3**) was benzene). The transfer suggests that the formation of **4** did occur, but it was not stable in the reaction mixture. Other strategies, activating phenyliodine diacetate with TMSOTf in the presence of trimethylsilylmethylsulfones, failed to yield the desired aryl((arylsulfonyl)methyl)iodonium (**4**) and instead led to diaryliodonium triflates via dimerization.

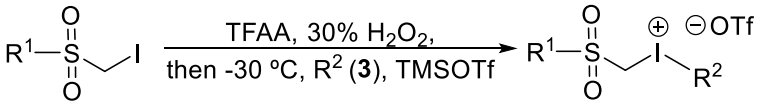



**Scheme 2**. Mechanism to aryl((arylsulfonyl)methyl)iodonium triflates.

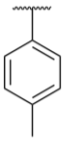
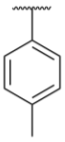
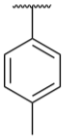
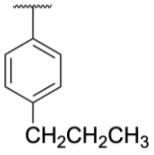
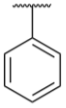
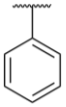
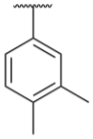
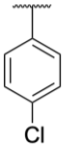
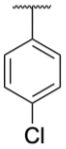
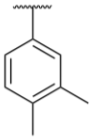
The aryl((arylsulfonyl)methyl)iodoniums (**4**) were examined with a range of arenes bearing both electron-donating and electron-withdrawing substituents (Table 1, see Supplementary Material for all trials). The sulfonyl-substituted arenes (**1**) work best with σ-electron-donating groups but can tolerate deactivating groups like chlorine. The iodine-bound arenes (**3**) bearing π- or σ-withdrawing groups consistently failed to yield isolable products. From crude NMR spectra, compound **4** was identified in reactions in which **3** contained π-electron-donating groups such as anisole, but isolation by ether precipitation failed, resulting in decomposition.

**Table 1.** Substrate scope and yields




**1** **4**

| Entry | $R^1$ | $R^2$ | Yield |
|---|---|---|---|
| 1 |  |  | 36% |
| 2 |  |  | 48% |
| 3 |  |  | 58% |
| 4 |  |  | 53% |
| 5 |  |  | 28% |
| 6 |  |  | 24% |

When attempting to obtain spectroscopic characterization, the aryl((arylsulfonyl)methyl)iodonium triflates were found to react with DMSO-$d_6$, $CD_3OD$, $D_2O$, $d_6$-acetone, and $CD_3CN$, with $CD_3CN$ showing slow enough kinetics to allow for $^{13}C$ NMR spectra (see Supplementary Material). Solubility in $CDCl_3$ was too low to obtain a usable NMR spectrum. The stability of the synthesized aryl((arylsulfonyl)methyl)iodonium triflate derivatives was highly dependent on the choice of deuterated NMR solvent. In most solvents, degradation was observed in under 4 hours, except for acetonitrile-$d_3$, which extended stability to roughly 8 hours. Distinct degradation pathways were apparent across the solvent set. Deuterated dimethyl sulfoxide causes the aryl((arylsulfonyl)methyl)iodonium triflate to undergo a Kornblum-type oxidation that forms a sulfonyl aldehyde along with the iodomethylsulfone. Deuterated methanol appears to attack the methylene or possibly promote the formation of a diiodo-complex, as suggested by the emergence of a new peak at approximately 4.9 ppm, yielding unreacted starting material and 4-iodotoluene as the primary degradation products. Acetonitrile-$d_3$

produces a mixture containing the iodonium triflate salt, unreacted starting material, and 4-iodotoluene. In acetone-$d_6$, degradation proceeds via a less-defined route, yielding a complex mixture that includes the starting material, 4-iodotoluene, and a potential secondary hypervalent iodine species.

## Conclusions

Methylsulfonyl-substituted $\lambda^3$-iodanes were synthesized through an improved procedure. Previously unreported reactivity with common deuterated solvents was investigated, with degradation pathways vary by solvent. Degradation pathways included Kornblum-type oxidation in DMSO-$d_6$, suspected methylene attack or possible diiodo-complex formation in $CD_3OD$, and progressive decomposition in both $d_6$-acetone and $CD_3CN$. The electron-withdrawing and steric-hindrance effects of the sulfonyl group allow for the stabilization of a $sp^3$ hybridized carbon to be bound to a hypervalent iodine atom. The revised procedure incorporated a minimal peroxide concentration, excess triflate to account for moisture, and concentrated the mixture between steps. Evidence of solvent-induced decomposition is presented in the **Supplementary Material**. Our future work endeavors to evolve alkylated hypervalent iodine species from obscurity to utility for the synthetic chemistry community.

## Experimental Section

**General.** Unless stated otherwise, all reactions were carried out under a nitrogen atmosphere. All commercially available precursor reagents were used without additional purification. Spectra were acquired on a 500 MHz NMR spectrometer with nuclei $^1H$, $^{19}F$, and $^{13}C$ using $CDCl_3$ and $CD_3CN$ as solvents.

**Experimental procedure of iodomethylsulfonyl precursors.**[14–16] Sodium arylsulfinate (1.00 equiv) and diiodomethane (1.65-1.70 equiv) were added to DMSO (20-60 mL), and the mixture was stirred at 50 °C for 48 h. The reaction was cooled to room temperature and quenched with deionized water. The solution is either extracted with DCM or filtered directly. If extracted, the organic layer was dried and concentrated to afford the crystallized iodomethylsulfonyl precursor.

**Experimental procedure of products.**[1] Trifluoroacetic anhydride (17.1 mmol) and 30% hydrogen peroxide (11.2 mmol) were stirred for 10 minutes at -30 °C in a sealed flask. The solution was allowed to warm to 25 °C and stirred for another 10 minutes, and then cooled to -30 °C. A solution of the iodomethylsulfonyl derivative (1.5 mmol) and dichloromethane (2.0 mL) was added slowly to the cold flask, then stirred for 30 minutes to 3.5 hours at -30 °C. A vacuum was then applied to the solution, and it was lyophilized at -30 °C for 30 minutes until a paste formed. Trifluoroacetic Acid (26.1 mmol), arene (3.16 mmol), trimethylsilyl trifluoromethanesulfonate (3.3 mmol), and dichloromethane (2.0 mL) were added to the reaction mixture, and the mixture was stirred for 1 to 24 hours at 25 °C. The reaction mixture was evaporated at 2 torr for 30 minutes and then concentrated under vacuum for 24 hours. The product was precipitated in diethyl ether (5 mL) and stirred for 15 minutes. The precipitate was filtered to afford the product. Residual trifluoroacetic acid and other solvents can impede precipitation, causing the crude product to “oil out” instead of forming a solid.

**Phenyl(tosylmethyl)iodonium triflate (4a).** $^1H$ NMR (500 MHz, $CD_3CN$): δ ppm 2.47 (s, 3 *H*) 5.51 (s, 2 *H*) 7.46 (d, *J* = 8.2 Hz, 2 *H*) 7.54 (t, *J* = 7.7 Hz, 2 *H*) 7.77 (t, *J* = 7.6 Hz, 1 *H*) 7.81 (d, *J* = 8.2 Hz, 2 *H*) 8.04 (d, *J* = 7.6, 2 *H*). $^{13}C$ NMR (126 MHz, $CD_3CN$): δ ppm 21.90 (s, 1 *C*), 52.96 (s, 1 *C*), 110.51 (s, 1 *C*) 130.20 (s, 2 *C*) 131.63 (s, 2 *C*) 133.20

(s, 2 *C*) 133.29 (s, 1 *C*) 134.69 (s, 1 *C*) 138.20 (s, 2 *C*) 148.59 (s, 1 *C*). $^{19}$F NMR (470 MHz, $CD_3CN$): δ ppm -79.34 (s, 3 *F*).

***p*-Tolyl(tosylmethyl)iodonium triflate (4b).** $^{1}$H NMR (500 MHz, $CD_3CN$): δ ppm 2.46 (s, 3 *H*) 2.48 (s, 3 *H*) 5.48 (s, 2 *H*) 7.35 (d, *J* = 8.2 Hz, 2 *H*) 7.46 (d, *J* = 8.6 Hz, 2 *H*) 7.80 (d, *J* = 8.2 Hz, 2 *H*) 7.89 (d, *J* = 8.5 Hz, 2 *H*). $^{13}$C NMR (126 MHz, $CD_3CN$): δ ppm 21.63 (s, 1 *C*) 21.89 (s, 1 *C*) 52.55 (s, 1 *C*) 106.61 (s, 1 *C*) 130.08 (s, 2 *C*) 131.56 (s, 2 *C*) 133.50 (s, 1 *C*) 133.77 (s, 2 *C*) 138.10 (s, 2 *C*) 146.08 (s, 1 *C*) 148.61 (s, 1 *C*). $^{19}$F NMR (470 MHz, $CD_3CN$): δ ppm -79.22 (s, 3 *F*).

**((Phenylsulfonyl)methyl)(*p*-tolyl)iodonium triflate (4c).** $^{1}$H NMR (500 MHz, $CD_3CN$): δ ppm 2.44 (s, 3 *H*) 5.51 (s, 2 *H*) 7.34 (d, *J* = 8.3 Hz, 2 *H*) 7.65 (t, *J* = 8.2 Hz, 2 *H*) 7.81 (tt, *J* = 7.5, 1.1 Hz, 1 *H*) 7.92 – 7.94 (m, 4 *H*). $^{13}$C NMR (126 MHz, $CD_3CN$): δ ppm 21.79 (s, 1 *C*) 52.58 (s, 1 *C*) 106.84 (s, 1 *C*) 130.24 (s, 2 *C*) 131.24 (s, 2 *C*) 134.00 (s, 2 *C*) 136.66 (s, 1 *C*) 137.05 (s, 1 *C*) 138.36 (s, 2 *C*) 146.35 (s, 1 *C*). $^{19}$F NMR (470 MHz, $CD_3CN$): δ ppm -79.24 (s, 3 *F*).

**(((4-Chlorophenyl)sulfonyl)methyl)(3,4-dimethylphenyl)iodonium triflate (4d).** $^{1}$H NMR (500 MHz, $CD_3CN$): δ ppm 2.28 (s, 3 *H*) 2.35 (s, 3 *H*) 5.55 (s, 2 *H*) 7.26 (d, *J* = 8.8 Hz, 1 *H*) 7.60 (d, *J* = 8.8 Hz, 2 *H*) 7.73 – 7.74 (m, 2 *H*) 7.86 (d, *J* = 8.6 Hz, 2 *H*). $^{13}$C NMR (126 MHz, $CD_3CN$): δ ppm 19.91 (s, 1 *C*) 20.04 (s, 1 *C*) 51.45 (s, 1 *C*) 106.51 (s, 1 *C*) 131.16 (s, 2 *C*) 131.79 (s, 2 *C*) 133.95 (s, 1 *C*) 135.37 (s, 1 *C*) 135.41 (s, 1 *C*) 138.09 (s, 1 *C*) 142.78 (s, 1 *C*) 142.91 (s, 1 *C*) 144.87 (s, 1 *C*). $^{19}$F NMR (470 MHz, $CD_3CN$): δ ppm -79.22 (s, 3 *F*).

**(4-Propylphenyl)(tosylmethyl)iodonium triflate (4e).** $^{1}$H NMR (500 MHz, $CD_3CN$): δ ppm 0.94 (t, *J* = 7.3 Hz, 3 *H*) 1.64 (sxt, *J* = 7.1 Hz, 2 *H*) 2.46 (s, 3 *H*) 2.68 (t, *J* = 7.8 Hz, 2 *H*) 5.51 (s, 2 *H*) 7.33 (d, *J* = 8.5 Hz, 2 *H*) 7.44 (d, *J* = 8.5 Hz, 2 *H*) 7.79 (d, *J* = 8.5, 2 *H*) 7.91 (d, *J* = 8.5 Hz, 2 *H*). $^{13}$C NMR (126 MHz, $CD_3CN$): δ ppm 13.84 (s, 1 *C*) 21.75 (s, 1 *C*) 24.90 (s, 1 *C*) 38.04 (s, 1 *C*) 52.43 (s, 1 *C*) 106.72 (s, 1 *C*) 129.92 (s, 2 *C*) 131.40 (s, 2 *C*) 133.00 (s, 2 *C*) 133.35 (s, 1 *C*) 137.93 (s, 2 *C*) 148.42 (s, 1 *C*) 150.32 (s, 1 *C*). $^{19}$F NMR (470 MHz, $CD_3CN$): δ ppm -79.20 (s, 3 *F*).

**(((4-Chlorophenyl)sulfonyl)methyl)(*p*-tolyl)iodonium triflate (4f).** $^{1}$H NMR (500 MHz, $CD_3CN$): δ ppm 2.45 (s, 3 *H*) 5.54 (s, 2 *H*) 7.34 (d, *J* = 8.2 Hz, 2 *H*) 7.63 (d, *J* = 8.8 Hz, 2 *H*) 7.88 (d, *J* = 8.8 Hz, 2 *H*) 7.92 (d, *J* = 8.5 Hz, 2 *H*). $^{13}$C NMR (126 MHz, $CD_3CN$): δ ppm 21.65 (s, 1 *C*) 51.90 (s, 1 *C*) 106.68 (s, 1 *C*) 131.23 (s, 2 *C*) 131.85 (s, 2 *C*) 133.83 (s, 2 *C*) 135.25 (s, 1 *C*) 138.07 (s, 2 *C*) 143.01 (s, 1 *C*) 146.18 (s, 1 *C*). $^{19}$F NMR (470 MHz, $CD_3CN$): δ ppm -79.21 (s, 3 *F*).

**(3,4-Dimethylphenyl)((phenylsulfonyl)methyl)iodonium triflate (4g).** $^{1}$H NMR (500 MHz, $CD_3CN$): δ ppm 2.27 (s, 3 *H*) 2.34 (s, 3 *H*) 5.52 (s, 2 *H*) 7.27 (d, *J* = 7.9 Hz, 1 *H*) 7.64 (t, *J* = 7.9 Hz, 2 *H*) 7.75 – 7.77 (m, 2 *H*) 7.80 (t, *J* = 7.5, 1 *H*) 7.91 (dd, *J* = 8.0, 1.3 Hz, 2 *H*). $^{13}$C NMR (126 MHz, $CD_3CN$): δ ppm 20.12 (s, 1 *C*) 20.19 (s, 1 *C*) 52.29 (s, 1 *C*) 106.74 (s, 1 *C*) 130.20 (s, 2 *C*) 131.19 (s, 2 *C*) 134.11 (s, 1 *C*) 135.70 (s, 1 *C*) 136.74 (s, 1 *C*) 136.99 (s, 1 *C*) 138.42 (s, 1 *C*) 142.92 (s, 1 *C*) 145.07 (s, 1 *C*). $^{19}$F NMR (470 MHz, $CD_3CN$): δ ppm -79.22 (s, 3 *F*).

**(4-Chlorophenyl)(tosylmethyl)iodonium triflate (4h).** $^{1}$H NMR (500 MHz, $CD_3CN$): δ ppm 2.47 (s, 3 *H*) 5.53 (s, 2 *H*) 7.44 (d, *J* = 8.2 Hz, 2 *H*) 7.52 (d, *J* = 8.8 Hz, 2 *H*) 7.79 (d, *J* = 8.2 Hz, 2 *H*) 8.01 (d, *J* = 9.2 Hz, 2 *H*). $^{13}$C NMR (126 MHz, $CD_3CN$): 21.74 (s, 1 *C*) 52.84 (s, 1 *C*) 107.64 (s, 1 *C*) 129.97 (s, 2 *C*) 131.43 (s, 2 *C*) 132.90 (s, 2 *C*) 133.24 (s, 1 *C*) 139.61 (s, 2 *C*) 140.73 (s, 1 *C*) 148.57 (s, 1 *C*). $^{19}$F NMR (470 MHz, $CD_3CN$): -79.23 (s, 3 *F*).

## Acknowledgements

I. F. Dempsey Hyatt thanks Viktor Zhdankin and Akira Yoshimura for their assistance with providing advice and valuable NMR spectra of aryl((arylsulfonyl)methyl)iodonium triflates from the original synthetic method publication. The Hyatt Research Group thanks Adelphi University's Department of Chemistry and Biochemistry for supporting our research.

## Supplementary Material

Copies of $^{1}H$ and $^{13}C$ NMR spectra of all synthesized compounds are available in the supplementary material associated to this manuscript. The NMR spectra demonstrating the solvent stability is also included in the supplementary material.

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