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Oxidation of sulfides using recyclable pseudocyclic benziodoxole triflate

Akira Yoshimura* and Viktor V. Zhdankin*

Department of Chemistry and Biochemistry, University of Minnesota Duluth, Duluth, Minnesota, 55812, USA
Emails: ayoshimu@d.umn.edu, vzhdanki@d.umn.edu

Dedicated to Prof. Oleg A. Rakitin on the occasion of his 65th birthday

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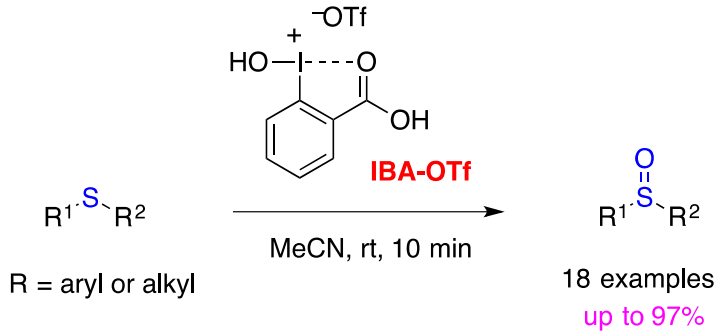
Abstract

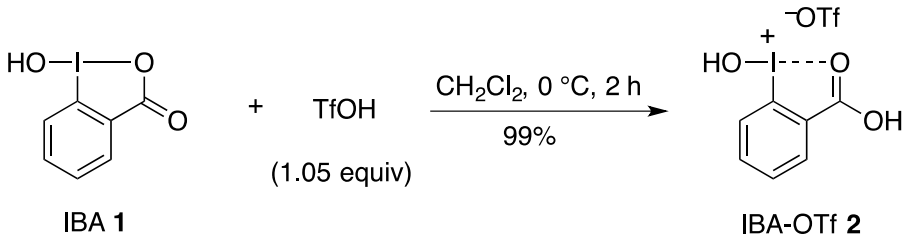
A new pseudocyclic hypervalent iodine reagent, benziodoxole triflate (IBA-OTf, a complex of 2-iodosylbenzoic acid with trifluoromethanesulfonic acid), can be used as an efficient oxidant for selective oxidation of various organic sulfides to sulfoxides. This oxidation proceeds under mild condition to afford the corresponding sulfoxides in moderate to good yields without overoxidation. The reduced form of the hypervalent iodine reagent, 2-iodobenzoic acid, can be easily recovered from the reaction mixture in good yields by a simple acid-base liquid-liquid biphasic protocol.



Keywords: Hypervalent iodine, benziodoxole, oxidation, sulfides, recyclable reagents

Introduction

Hypervalent iodine compounds have found wide synthetic application as environmentally sustainable reagents for various synthetically important chemical transformations.[1-10] An important class of powerful oxidants and electrophiles is represented by the triflic acid-activated hypervalent iodine reagents. In particular, [hydroxy(trifluoromethanesulfonyloxy)iodo]benzene PhI(OH)OTf can be generated in situ from iodosylbenzene and trifluoromethanesulfonic acid and used without isolation in the various oxidative transformations.[11-18] However, despite its useful oxidative reactivity, PhI(OH)OTf has serious drawbacks such as low thermal stability and sensitivity to moisture.[19] Recently, we have reported the synthesis and structural characterization of a new triflic acid-activated hypervalent iodine(III) reagent, benziodoxole triflate (IBA-OTf **2**), which was prepared by treatment of 2-iodosylbenzoic acid (IBA **1**) with trifluoromethanesulfonic acid (Scheme 1).[20]

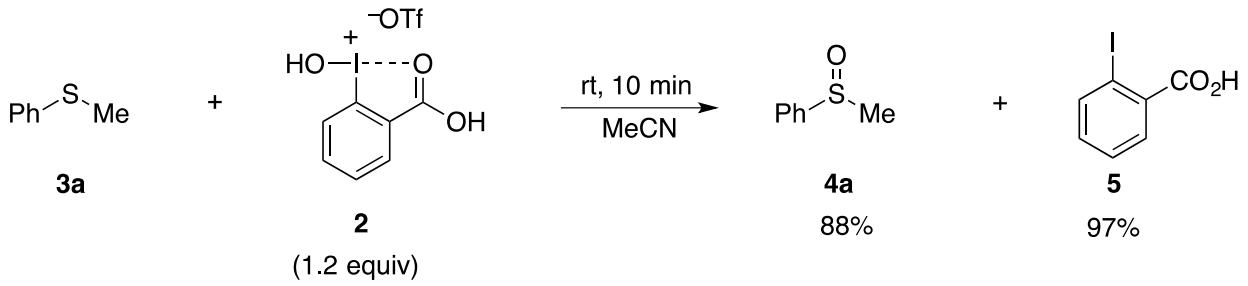


Scheme 1. Preparation of benziodoxole triflate **2**.

IBA-OTf **2** is stable at room temperature in the presence of air and can be used as an effective electrophilic oxidizing reagent toward various organic substrates.[21,22] The X-ray crystallographic study of IBA-OTf revealed a pseudocyclic structure with strong intramolecular secondary bonding between the hypervalent iodine center and the oxygen atom of *ortho*-carboxylic group. The presence of this intramolecular coordination leads to the stabilization of the usually unstable hydroxy(aryl)iodonium structure. It has been documented in the literature that hypervalent iodine compounds with intramolecular secondary bonding demonstrate higher stability and improved reactivity compared to the common, noncyclic hypervalent iodine reagents.[23-28] Previously, we have published preliminary results on the reactivity of IBA-OTf with various organic substrates including sulfides.[20] In the present paper, we report the scope and limitations for the oxidation of organic sulfides using IBA-OTf under mild conditions. This procedure is applicable toward oxidation of alkyl- or arylsulfides bearing other sensitive functionalities and gives the corresponding sulfoxides without overoxidation.

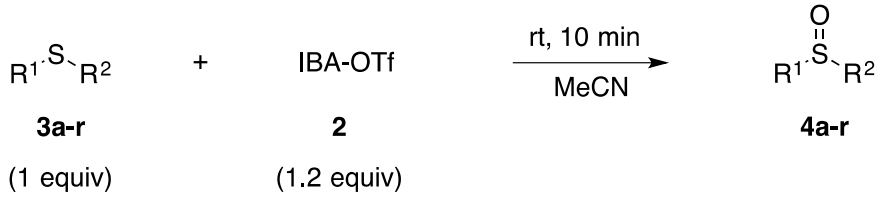
Results and Discussion

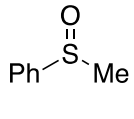
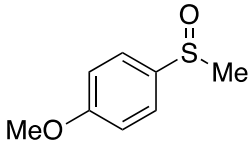
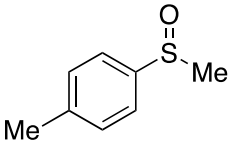
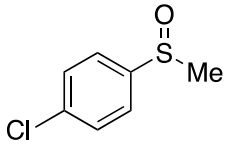
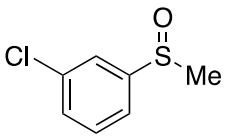
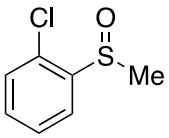
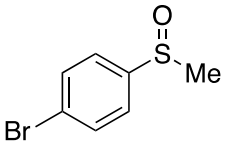
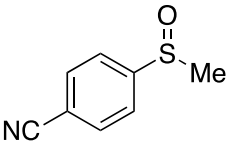
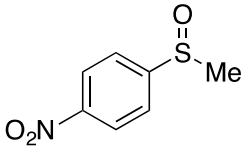
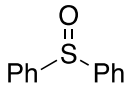
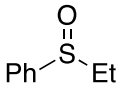
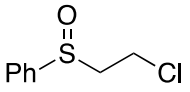
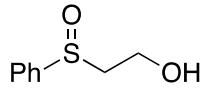
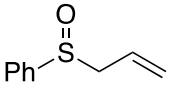
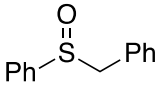
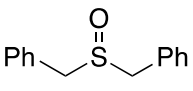
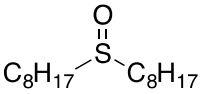
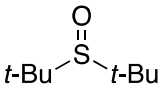
In the initial experiment, we have found that the oxidation of thioanisole **3a** using IBA-OTf **2** in acetonitrile solution at room temperature is complete in 10 min affording the respective sulfoxide **4a** in 88% yield without any overoxidation to sulfone (Scheme 2). The reduced form of IBA-OTf, 2-iodobenzoic acid **5**, can be easily recovered from reaction mixture in 97% by a simple acid-base liquid-liquid biphasic protocol. The recovered 2-iodobenzoic acid **5** can be easily converted to 2-iodosylbenzoic acid **1** by a standard procedure.[29]



Scheme 2. Oxidation of thioanisole **3a** using IBA-OTf **2**.

Table 1. Oxidation of sulfides **3** using IBA-OTf **2**[a]

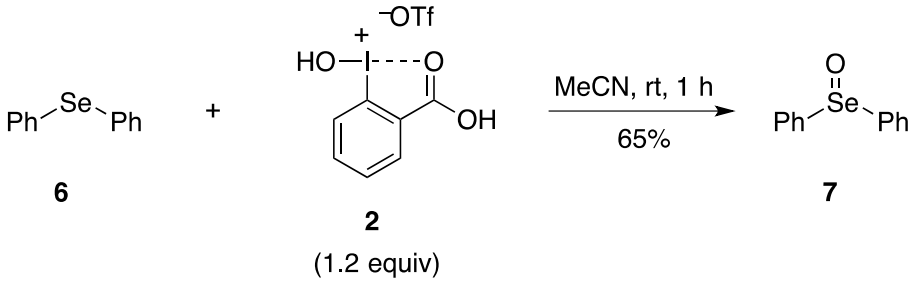


 **4a** 88%	 **4b** 90%	 **4c** 84%	 **4d** 91%
 **4e** 82%	 **4f** 59%	 **4g** 97%	 **4h** 81%
 **4i** 96%	 **4j** 56%[b]	 **4k** 84%	 **4l** 79%
 **4m** 24%	 **4n** 86%	 **4o** 89%	 **4p** 83%
	 **4q** 100%	 **4r** 0%[c]	

[a] Reaction conditions: IBA-OTf **2** (62 mg, 0.15 mmol) and sulfides **3** (0.125 mmol) in acetonitrile (1 mL) at room temperature for 10 min. [b] Reaction time was 1 h. [c] Reaction time was 24 h.

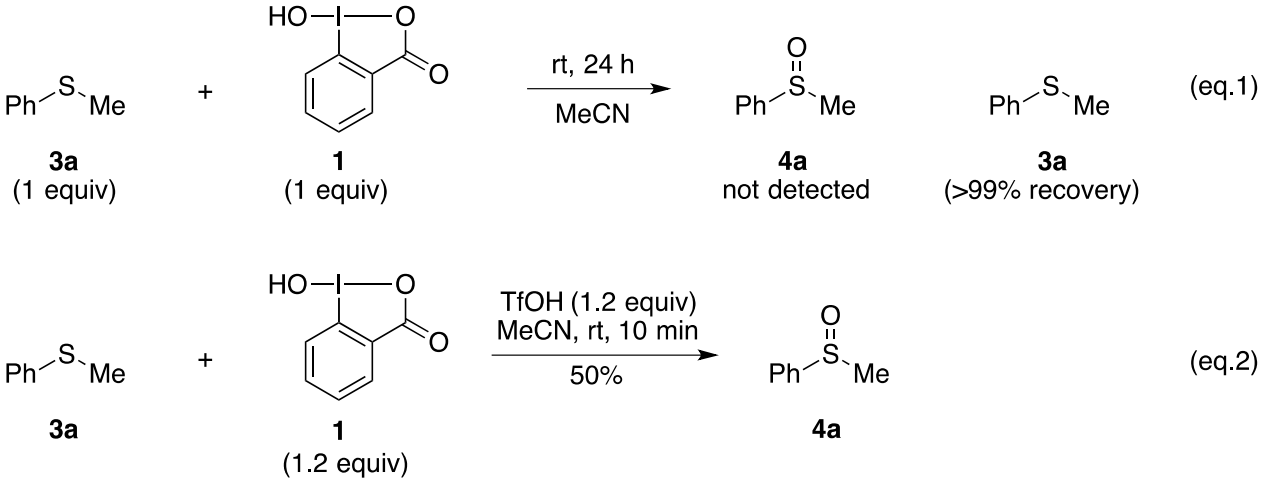
In order to determine the scope and limitation of this reaction, we investigated the oxidation of various sulfides using IBA-OTf **2** under similar conditions (Table 1). In general, the reactions of various substituted thioanisoles **3a-i** with either electron-donating or electron-withdrawing substituents in acetonitrile solution afforded the corresponding substituted sulfoxides **4a-i** in moderate to good yields. The reactions of phenyl alkyl sulfides **3k-o** under these conditions also gave the respective sulfoxides **4k-o** in low to good yields. As expected, the reactions of dialkyl sulfides **3p-q** also afforded sulfoxides **4p-q** in good yields. In the reaction of sulfides **3f** and **3j** with sterically hindered substituents, the corresponding sulfoxides **4f** and **4j** were obtained in relatively low yields. The most steric bulky sulfide, di-*tert*-butyl sulfide **3r**, did not react with IBA-OTf **2** even after 24 h at room temperature.

It is noteworthy that the reaction of diphenylselenide **6** using IBA-OTf **2** under same condition gave the corresponding selenoxide **7** in 65% isolated yield without overoxidation (Scheme 3). Compared to the analogous oxidation of diphenylsulfide **3j**, the reaction of selenide afforded the corresponding product **7** in better yield.



Scheme 3. Oxidation of diphenylselenide **6** using IBA-OTf **2**.

Finally, we have investigated a one-pot oxidation of thioanisole **3a** using reagent **2** generated in situ from 2-iodosylbenzoic acid **1** with trifluoromethanesulfonic acid (Scheme 4). A control experiment has demonstrated that the reaction of thioanisole with IBA **1** in the absence of trifluoromethanesulfonic acid does not produce compound **4a** and 99% of unreacted thioanisole **3a** can be recovered from the reaction mixture (eq.1). However, when thioanisole **3a** was treated with IBA **1** and trifluoromethanesulfonic acid in acetonitrile solution, product **4a** was isolated in 50% yield (eq.2), which is a much lower yield compared to the reaction with pure reagent **2**.



Scheme 4. One-pot oxidation of thioanisole **3a** using IBA **1**.

Conclusions

We have found that IBA-OTf is an effective oxidant for the oxidation of sulfides to sulfoxides. This oxidation proceeds as a selective reaction without overoxidation to sulfones. Moreover, the reduced form of IBA-OTf, 2-iodobenzoic acid, can be easily recovered from reaction mixture by a simple quenching method. This reagent can also be used for the oxidation of diphenylselenide to the corresponding selenoxide in moderate yield. Furthermore, IBA-OTf can be generated in situ from 2-iodosylbenzoic acid and trifluoromethanesulfonic acid and used for the oxidation of thioanisole.

Experimental Section

General. All reactions were performed under dry argon atmosphere with flame-dried glassware. Dichloromethane was distilled from CaH_2 immediately prior to use. All commercial reagents were ACS reagent grade and used without further purification. Melting points were determined in an open capillary tube with a Mel-temp II melting point apparatus. NMR spectra were recorded on a Varian Inova 500 MHz (^{1}H NMR). Chemical shifts are reported in parts per million (ppm) and referenced relative to the tetramethylsilane.

1,3-Dihydroxy-1*H*-1λ^3-benzo[*d*][1,2]iodoxol-1-yl trifluoromethanesulfonate (IBA-OTf) (2).[20] Trifluoromethanesulfonic acid (0.612 g, 4.08 mmol) was added dropwise at 0 °C to a stirred mixture of 2-iodosylbenzoic acid **1** (1.026 g, 3.89 mmol) with CH_2Cl_2 (5 mL). The reaction was stirred at 0 °C for 2 h. After completion of the reaction, the solvent was removed under reduced pressure and the solid product was washed with hexane and diethyl ether several times then dried in vacuum to give 1.593 g (99%) of compound **2** as a white solid. Recrystallization of product **4** from acetonitrile at 0 °C afforded colorless prisms; mp 154.6-155.1 °C; ^{1}H NMR (500 MHz, CD_3CN): δ 8.38 (d, *J* 7.8 Hz 1H), 8.25-8.19 (m, 1H), 8.02 (d, *J* 8.5 Hz, 1H), 7.94-7.89 (m, 1H).

General procedure for oxidation of sulfides to sulfoxides using IBA-OTf 2. Sulfide **3** (0.125 mmol) was added to a solution of **2** (62 mg, 0.15 mmol) in acetonitrile (1 mL). The reaction was stirred at room temperature for 10 min to 1 h (reaction completion was controlled by TLC). After completion of the reaction, 5% aqueous $Na_2S_2O_3$ (5 mL) and saturated $NaHCO_3$ (5 mL) were added, and the mixture was extracted with dichloromethane. The organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. Purification using short chromatographic column (hexane-ethyl acetate = 3 : 1) afforded analytically pure sulfoxide **4.**

*Recovery of 2-iodobenzoic acid **5**:* The aqueous layer was acidified with 12M HCl to pH about 1-2 and then extracted with dichloromethane. The organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give 36 mg (97%) of 2-iodobenzoic acid **5**.

Methyl phenyl sulfoxide (4a).[20] Reaction of thioanisole **3a** (16 mg, 0.125 mmol) according to general procedure afforded 15 mg (83%) of product **4a** as a yellow oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.68-7.63 (m, 2H), 7.57-7.48 (m, 3H), 2.73 (s, 3H).

***p*-Methoxy phenyl methyl sulfoxide (4b).**[30] Reaction of *p*-methoxyphenyl methyl sulfide **3b** (19 mg, 0.125 mmol) according to general procedure afforded 19 mg (90%) of product **4b** as a light yellow oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.58 (d, *J* 8.8, Hz, 2H), 7.02 (d, *J* 8.8 Hz, 2H), 3.84 (s, 3H), 2.68 (s, 3H).

Methyl *p*-tolyl sulfoxide (4c).[31] Reaction of methyl *p*-tolyl sulfide **3c** (17 mg, 0.125 mmol) according to general procedure afforded 16 mg (84%) of product **4c** as a yellow oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52 (d, *J* 7.5 Hz, 1H), 7.31 (d, *J* 7.5 Hz, 1H), 2.69 (s, 3H), 2.40 (s, 3H).

***p*-Chlorophenyl methyl sulfoxide (4d).**[30] Reaction of *p*-chlorophenyl methyl sulfide **3d** (20 mg, 0.125 mmol) according to general procedure afforded 20 mg (91%) of product **4d** as a yellow oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.60 (d, *J* 8.0 Hz, 2H), 7.52 (d, *J* 8.0 Hz, 2H), 2.72 (s, 3H).

***m*-Chlorophenyl methyl sulfoxide (4e)**.[32] Reaction of *m*-chlorophenyl methyl sulfide **3e** (20 mg, 0.125 mmol) according to general procedure afforded 18 mg (82%) of product **4e** as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.66 (s, 1H), 7.52-7.44 (m, 3H), 2.74 (s, 3H).

***o*-Chlorophenyl methyl sulfoxide (4f)**.[33] Reaction of *o*-chlorophenyl methyl sulfide **3f** (20 mg, 0.125 mmol) according to general procedure afforded 13 mg (59%) of product **4f** as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.97 (dd, *J* 8.0 Hz, 1.5 Hz, 1H), 7.57-7.52 (m, 1H), 7.45 (dt, *J* 7.8 Hz, 1.5 Hz, 1H), 7.40 (d, *J* 7.5 Hz, 1H), 2.83 (s, 3H).

***p*-Bromophenyl methyl sulfoxide (4g)**.[30] Reaction of *p*-bromophenyl methyl sulfide **3g** (25 mg, 0.125 mmol) according to general procedure afforded 26 mg (97%) of product **4g** as a white solid: mp 84.3-84.6 °C (lit.[30]; mp 82.4-83.3 °C): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.66 (d, *J* 8.3 Hz, 2H), 7.51 (d, *J* 8.3 Hz, 2H), 2.71 (s, 3H).

***p*-Cyanophenyl methyl sulfoxide (4h)**.[34] Reaction of *p*-cyanophenyl methyl sulfide **3h** (19 mg, 0.125 mmol) according to general procedure afforded 17 mg (81%) of product **4h** as a white solid: mp 91.2-91.5 °C (lit.[34]; mp 87-88 °C): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82 (d, *J* 8.3 Hz, 2H), 7.76 (d, *J* 8.3 Hz, 2H), 2.76 (s, 3H).

***p*-Nitrophenyl methyl sulfoxide (4i)**.[35] Reaction of *p*-nitrophenyl methyl sulfide **3i** (21 mg, 0.125 mmol) according to general procedure afforded 22 mg (96%) of product **4i** as a white solid: mp 150.5-150.8 °C (lit.[35]; mp 153.0-155.0 °C): ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.38 (d, *J* 9.0 Hz, 2H), 7.83 (d, *J* 9.0 Hz, 2H), 3.79 (s, 3H).

Diphenyl sulfoxide (4j).[35] Reaction of diphenyl sulfide **3i** (23 mg, 0.125 mmol) according to general procedure afforded 14 mg (56%) of product **4i** as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.78-7.63 (m, 4H), 7.50-7.41 (m, 6H).

Phenyl ethyl sulfoxide (4k).[30] Reaction of phenyl ethyl sulfide **3k** (17 mg, 0.125 mmol) according to general procedure afforded 16 mg (84%) of product **4k** as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.63-7.59 (m, 2H), 7.55-7.47 (m,3H), 2.95-2.86 (m, 1H), 2.81-2.72 (m, 1H), 1.20 (t, *J* 7.5 Hz, 3H).

2-Chloroethyl phenyl sulfoxide (4l).[30] Reaction of 2-chloroethyl phenyl sulfide **3l** (22 mg, 0.125 mmol) according to general procedure afforded 19 mg (79%) of product **4l** as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.64 (d, *J* 7.5 Hz, 2H), 7.58-7.50 (m, 3H), 4.00-3.93 (m, 1H), 3.69-3.62 (m, 1H), 3.22-3.11 (m, 2H).

2-Phenyl sulfoxy ethanol (4m).[35] Reaction of 2-(phenylthio)ethanol **3m** (19 mg, 0.125 mmol) according to general procedure afforded 5 mg (24%) of product **4m** as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.66 (dd, *J* 8.3 Hz, 1.3 Hz, 2H), 7.60-7.51 (m, 3H), 4.21-4.12 (m, 1H), 4.08-4.02 (m, 1H), 3.26-3.19 (m, 1H), 2.90-2.83 (m, 1H).

Allyl phenyl sulfoxide (4n).[35] Reaction of allyl phenyl sulfide **3n** (19 mg, 0.125 mmol) according to general procedure afforded 18 mg (86%) of product **4n** as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.62-7.57 (m, 2H), 7.54-7.46 (m, 3H), 5.70-5.58 (m, 1H), 5.33 (d, *J* 11.0 Hz, 1H), 5.19 (d, *J* 17.0 Hz, 1H), 3.61-3.46 (m, 2H).

Benzyl phenyl sulfoxide (4o).[30,36] Reaction of benzyl phenyl sulfide **3o** (25 mg, 0.125 mmol) according to general procedure afforded 24 mg (89%) of product **4o** as a white solid: mp 123.2-123.4 °C (lit.[30]; mp 122.4-123.6 °C): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.49-7.36 (m, 5H), 7.32-7.22 (m, 3H), 6.99 (d, *J* 7.0 Hz, 2H), 4.10 (d, *J* 7.8 Hz, 1H), 4.00 (d, *J* 7.8 Hz, 1H).

Dibenzylsulfoxide (4p).[37] Reaction of dibenzylsulfide **3p** (27 mg, 0.125 mmol) according to general procedure afforded 24 mg (83%) of product **4p** as a white solid: mp 128.1-128.3 °C (lit.[36]; mp 129.0-131.0 °C): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41-7.27 (m, 10H), 3.93 (d, *J* 8.0 Hz, 2H), 3.87 (d, *J* 8.0 Hz, 2H).

Dioctylsulfoxide (4q).[30] Reaction of dioctylsulfide **3q** (32 mg, 0.125 mmol) according to general procedure afforded 34 mg (100%) of product **4q** as a white solid: mp 72.2-72.7 °C (lit.[30], mp 75.0-76.0 °C): ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.72-2.57 (m, 4H), 1.82-1.70 (m, 4H), 1.53-1.20 (m, 20H), 0.88 (t, *J* 7.0 Hz, 6H).

Oxidation of diphenylselenide 6 to diphenylselenoxide 7 using IBA-OTf 2. Diphenylselenide **6** (29 mg, 0.125 mmol) was added to a solution of **2** (62 mg, 0.15 mmol) in acetonitrile (1 mL). The reaction was stirred at room temperature for 1h (reaction completion was controlled by TLC). After completion of the reaction, 5% aqueous $Na_2S_2O_3$ (5 mL) and saturated $NaHCO_3$ (5 mL) were added, and the mixture was extracted with dichloromethane. The organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. Purification by short column chromatography (hexane-ethyl acetate 3 : 1) afforded analytically pure diphenylselenoxide **7**; 20 mg (65%) isolated as a brown solid: mp 110.0-111.6 °C (lit.[38], mp 100.0-101.0 °C): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.72-7.67 (m, 4H), 7.49-7.42 (m, 6H).

One-pot oxidation of thioanisole 3a using IBA-OTf 1 and trifluoromethanesulfonic acid. Thioanisole **3a** (16 mg, 0.125 mmol), and trifluoromethanesulfonic acid (23 mg, 0.150 mmol) were added to a solution of **1** (40 mg, 0.150 mmol) in acetonitrile (1 mL). The reaction was stirred at room temperature for 1 h (reaction completion was controlled by TLC). After completion of the reaction, 5% aqueous $Na_2S_2O_3$ (5 mL) and saturated $NaHCO_3$ (5 mL) were added, and the mixture was extracted with dichloromethane. The organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. Purification by short column chromatography (hexane-ethyl acetate = 3 : 1) afforded analytically pure product **4a**; 9 mg (50%) isolated as a yellow oil identical to the sample from previous experiment.

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