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m-Iodosylbenzoic acid, a tagged hypervalent iodine reagent for the iodo-functionalization of alkenes and alkynes

Mekhman S. Yusubov [a,*], Roza Ya. Yusubova [a], Andreas Kirschning [b,*], Joo Yeon Park [c], Ki-Whan Chi [c,*]

[a] *The Siberian State Medical University, 2 Moskovsky Trakt, 634050 Tomsk, Russia and The Tomsk Polytechnic University, 30 Lenin Street, 634050 Tomsk, Russia*

[b] *Institut für Organische Chemie and Zentrum für Biomolekulare Wirkstoffe (BMWZ), Leibniz Universität Hannover, Schneiderberg 1B, D-30167 Hannover, Germany*

[c] *University of Ulsan, 680-749 Ulsan, Republic of Korea*

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Abstract

An efficient and facile method for the iodo-functionalization of alkenes **5** and alkynes **6** by using recyclable *m*-iodosylbenzoic acid (**2**) was developed. The final products can be easily isolated without any chromatographic purification by simple treatment of the crude mixture with an anionic exchange resin. Unreacted *m*-iodosylbenzoic acid and reduced *m*-iodobenzoic acid are effectively recovered from the resin by acidification with hydrochloric acid.
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Keywords: Alkenes; Alkynes; Hypervalent iodine; Iodination; *m*-Iodosobenzoic acid; Scavenger

Iodo-functionalization of alkenes and alkynes has been used as a pivotal transformation in organic synthesis.[1a,b] In particular, hypervalent iodine compounds have been successfully applied for iodination, iodo-functionalization, and oxidation reactions due to their mild and selective oxidizing characteristics.[2a–f] For example, Yusubov et al.[2g] recently demonstrated an effective iodo-functionalization of alkenes by using (dichloroiodo)benzene. Broader application of hypervalent iodine compounds, however, has been restricted due to the tedious purification and recovery process necessary following reactions. To circumvent this problem, several recyclable hypervalent iodine reagents have been developed in the form of polymer-supported[3a–y] or molecular species.[4a–i] Nevertheless, multi-step syntheses and subsequent separation are required for the preparation of these recyclable hypervalent reagents.

We have recently developed a straightforward approach to simplify the work-up of iodine(III)-mediated reactions by using 3-(dichloroiodo)benzoic acid[4c] (**1**) and *m*-iodosylbenzoic acid[5a,b] (**2**) as recyclable reagents (Fig. 1). A detailed spectroscopic investigation of *m*-iodosylbenzoic acid and its sodium salt by Katritzky et al. supported the polymeric structure of these compounds.[5a,c] Both hypervalent iodine reagents are prepared with ease and tagged with a carboxylic acid group so that reduced iodine by-products such as *m*-iodobenzoic acid (**3**) can be removed at the end of the reaction by simple treatment with an anion exchange resin (IRA 900; hydroxide form) or by the

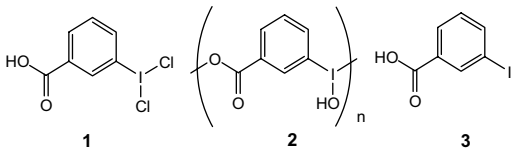

Fig. 1.

* Corresponding authors.
E-mail address: andreas.kirschning@oci.uni-hannover.de (A. Kirschning).

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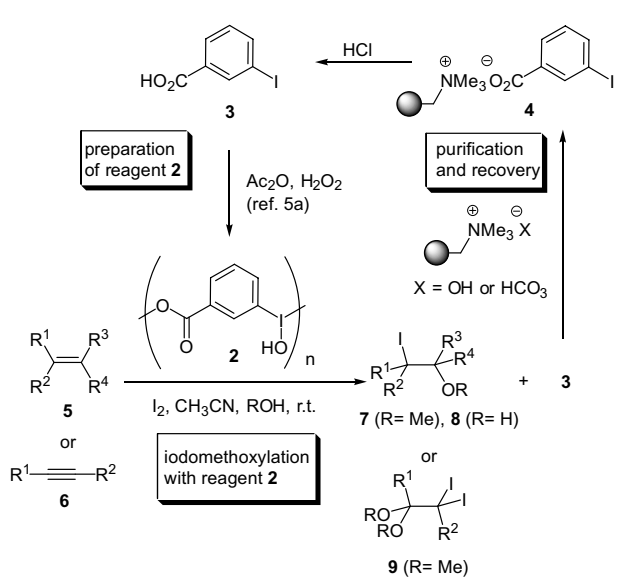
addition of $NaHCO_3$. Moreover, these scavenged iodine species could be easily recycled by simple acidification with HCl followed by reoxidation. Using these tagged reagents, we have shown that facile oxidation of primary and secondary alcohols and iodination of arenes are possible with a simplified purification step.[5a,b]

Based on this concept, we now report a simple and efficient method for the methoxy and hydroxy iodination of alkenes **5** and alkynes **6** by using *m*-iodosylbenzoic acid (**2**) (Scheme 1).

Thus, the treatment of **2** with alkenes **5a–j** in the presence of iodine effectively provides iodomethoxylation and iodohydroxylation products **7a–j** and **8a**,**c**,**e–j**, respectively, in 72–94% yields (Table 1). Reaction of cyclohexene (**5c**) with **2** in MeOH in the presence of iodine at room temperature gave *trans*-1-iodo-2-methoxycyclohexane (**7c**) in an 81% preparative yield. The addition proceeds in the expected *anti*-fashion, thereby exerting high *trans*-stereoselectivity. The stereoselective formation of *trans*-products **7** and **8** might be a result from the presence of bridged iodonium intermediates. And, the regioselectivity of our work is ascribed to the selective attack of a nucleophile (MeOH or H_2O) on the more substituted carbon of the cyclic iodonium ion.

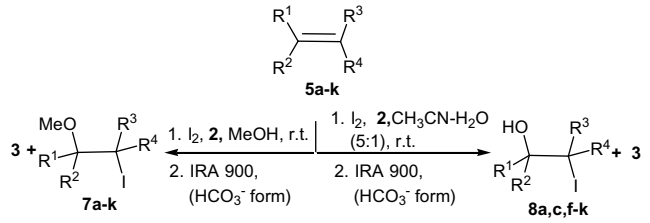
Treatment of the reaction mixture with Amberlite IRA 900[6] (hydrocarbonate or hydroxide form) and subsequent filtration followed by removal of the solvent under reduced pressure provided pure iodo-functionalized products. Formation of any other by-products was not encountered. Amberlite IRA 900 was used to remove the access of reagent **2**, iodine, and the by-product *m*-iodobenzoic acid from the crude mixtures.

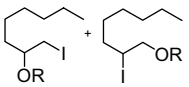
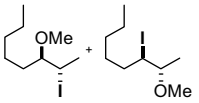
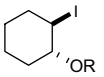
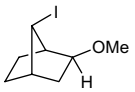
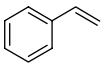
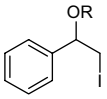
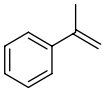
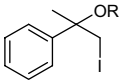
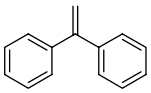
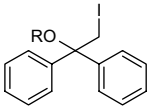
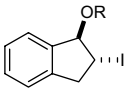
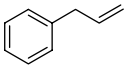
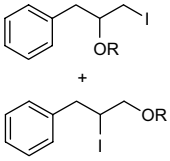
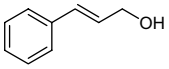
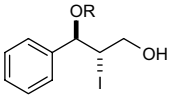
Reactions of *m*-iodosylbenzoic acid (**2**) with alkynes **6a–f** in the presence of iodine in MeOH lead to the diiododimethoxylation products **9a–f** in 67–90% yields (Table 2). To date, diiododimethoxylation of alkynes has been rarely described in the literature.[13a,b] If (dichloroiodo)benzene



Scheme 1.

Table 1
Iodohydroxylations and iodomethoxylations of alkenes **5**[14,15]



Alkene **5**	Product **7/8**	Yield[a,b] (%)
5a		**7a/7a′** R = Me 85[7] **8a/8a′** R = H 72[8]
5b		**7b/ 7b′**82[7]
5c		**7c** R = Me 81[2g] **8c** R = H 76[7,9]
5d		**7d** 85[10]
 **5e**		**7e** R = Me 83[2g] **8e** R = H 84[2g]
 **5f**		**7f** R = Me 91[2g] **8f** R = H 87[2g]
 **5g**		**7g** R = Me 94[2g] **8g** R = H 90[2g]
5h		**7h** R = Me 92[11] **8h** R = H 90[7]
 **5i**		**7i/7i′** R = Me 72[12] **8i/8i′** R = H 72[9]
 **5j**		**7j** R = Me 87 **8j** R = H 87

[a] Isolated yield of pure products.
[b] Products referenced.

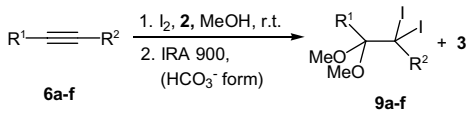
was applied instead of **2**, only the ketal **9e** of α,α-diiodoacetophenone was obtained from phenylethyne (**6e**). In other reactions with (dichloroiodo)benzene, the tentative products of ketals **9a**–**d**,**f** decomposed once flash chromatography on silica gel was attempted. These results clearly demonstrate the usefulness and preference of tagged *m*-iodosylbenzoic acid (**2**) over other iodine(III) reagents.[2g]

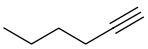
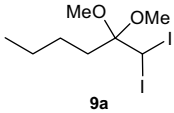
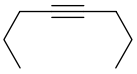
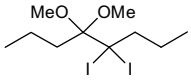
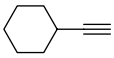
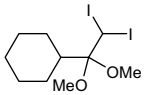
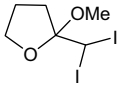
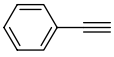
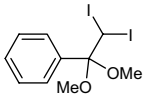
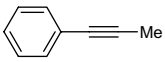
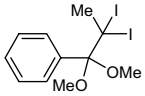
Reaction of pent-4-yn-1-ol (**6d**) with iodine and **2** in MeOH provided 2-(diiodomethyl)-2-methoxytetrahydro furan (**9d**). It is plausible to suggest that the iodocyclization of **6d** firstly yields cyclized intermediate **10** followed by iodomethoxylation of the exocyclic double bond to produce the final product **9d** (Scheme 2).

Based on the results[5b] of iodine(III)-mediated iodination of arenes, we suggest that the methoxy or hydrated form of **2** oxidizes iodine to MeOI (in MeOH) or HOI (in H_2O) which serves as a reactive electrophilic intermediate (Scheme 3).

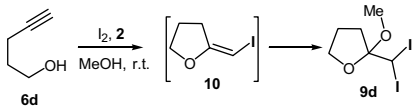
In conclusion, the inexpensive and easy-to-handle hypervalent iodine reagent *m*-iodosylbenzoic acid (**2**) was successfully applied for the iodomethoxylation and iodohydroxylation of alkenes and alkynes under mild conditions. This tagged reagent and the aryliodide by-product are easily removed from the reaction mixture by simple treatment with an anion exchange resin. This methodology allows easier preparation and simplified purification of organoiodo compounds.

Table 2
Iodomethoxylation of alkynes **6** with *m*-iodosylbenzoic acid **2**[14,15]

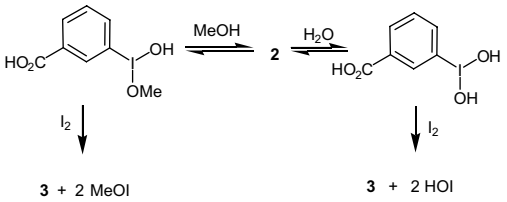


Alkyne **6**	Product **9**	Yield[a] (%)
 **6a**	 **9a**	83
 **6b**	 **9b**	99
 **6c**	 **9c**	78
6d	 **9d**	76
 **6e**	 **9e**	90
 **6f**	 **9f**	84

[a] Isolated yield of pure products.



Scheme 2.



Scheme 3.

Acknowledgments

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14. *Typical procedure for iodomethoxylation*. Alkenes **5a–c,e** (0.3 mmol) were added to a mixture of iodine (25.4 mg, 0.10 mmol) and *m*-iodosylbenzoic acid (**2**) (29 mg, 0.11 mmol) in MeOH (0.5 mL) and the reaction mixture was stirred at room temperature for 1 h (the reactions were monitored by TLC). Then, dichloromethane (1.0 mL) was added and the resulting solution was cooled to 5 °C. IRA 900 (350–400 mg, hydrocarbonate form) was added and the mixture was stirred for 5 min. The resin was removed by filtration and the solution was concentrated under reduced pressure to afford pure methoxyiodides **7a–c,e** as judged by NMR-spectroscopy. *m*-Iodobenzoic acid (**3**) can be easily regenerated from the IRA 900 resin **4** by treatment with aqueous HCl and reoxidized to reagent **2** without additional purification as described in Ref. 5a. For the iodomethoxylation of alkenes **5d,g–k**, a molar ratio of alkene:**2**:iodine = 0.2:0.11:0.2 (mmol) was employed, instead. The iodohydroxylation was carried out accordingly accept that MeCN–H_2O (5:1, 0.5 mL) was employed as solvent instead of MeOH.
Typical procedure for diiododimethoxylation. To a solution of iodine (28 mg, 0.11 mmol) and *m*-iodosylbenzoic acid (**2**) (29 mg, 0.11 mmol) in MeOH (0.5 mL) were added alkynes **6a–f** (0.15 mmol) in MeOH (0.5 mL), and the reaction mixture was stirred at room temperature for 1 h. Then, dichloromethane (1.0 mL) was added and the resulting solution was cooled to 5 °C. IRA 900 (350–400 mg, hydroxide form) was added and the mixture was stirred for 5 min in the dark. The resin was removed by filtration and the solution was concentrated under reduced pressure to afford pure diiodoketals **9a–f** as judged by NMR-spectroscopy.
15. Spectral data for **7k**, **8k** and new compounds. *2-Iodo-3-methoxy-3-phenylpropan-1-ol* (**7k**): Oil: ^{1}H NMR: δ 2.89 (1H, OH, s), 3.27 (3H, OCH_3, s), 3.81 ($1H_a$, CH_2, m), 3.95 ($1H_b$, CH_2, m), 4.34 (1H, CH–OMe, m), 4.52 (1H, CH–I, m); IR (neat) ν 3356 (OH), 1123 and 1085 (C–OH and C–O–C), 762 and 690 (C–I) cm^{-1}.
2-Iodo-1-phenylpropane-1,3-diol (**8k**): Colorless solid: mp 84–86 °C; ^{1}H NMR: δ 2.72 (1H, OH, s), 3.14 (1H, OH, s), 3.82 ($1H_a$, CH_2, m), 3.96 ($1H_b$, CH_2, m), 4.44 (1H, CH–OMe, d), 5.04 (1H, CH–I, m); IR (neat) ν 3380 (OH), 1074 (C–O), 780 and 684 (C–I) cm^{-1}.
3-Iodo-6-methoxycyclohex-1-ene (**7e**): Oil: ^{1}H NMR: δ 2.00–2.08 (4H, CH_2, m), 3.34 (3H, OCH_3, s), 3.99 (1H, CH–OMe, m), 4.43 (1H, CH–I, m), 5.72 (1H, =CH, m), 5.93 (1H, =CH, m); ^{13}C NMR: δ 25.4 (CI), 30.0 (CH_2), 30.1 (CH_2), 57.0 (OCH_3), 80.6 (COMe), 124.1 (=*C*–COMe), 130.9 (=*C*–CI); IR (neat) ν 1125 (C–O–C), 926 (C=C), 644 (C–I) cm^{-1}. Anal. Calcd for $C_7H_{11}IO$: C, 35.32; H, 4.66. Found: C, 35.17; H, 4.73.
1,1-Diiodo-2,2-dimethoxyhexane (**9a**): Oil: ^{1}H NMR: δ 0.94 (3H, CH_3, t), 1.34–1.43 (4H, CH_2, m), 2.02 (2H, CH_2, t), 3.33 (6H, CH_3, s) 5.39 (1H, CI_2H, s); ^{13}C NMR: δ −15.10 (CI_2), 12.2 (CH_3), 21.8 (CH_2), 26.4 (CH_2), 33.7 (CH_2), 50.4 (OCH_3), 98.8 ($C(OMe)_2$); IR (neat) ν 1075 and 1042 (C–O–C), 682 and 640 (C–I) cm^{-1}. Anal. Calcd for $C_8H_{16}I_2O_2$: C, 24.14; H, 4.05. Found: C, 23.89; H, 4.18.
4,4-Diiodo-5,5-dimethoxyoctane (**9b**): Oil: ^{1}H NMR: δ 0.97 (3H, CH_3, t), 1.05 (3H, CH_3, t), 1.54–1.62 (4H, CH_2, m), 2.03 (2H, CH_2–$C(OMe)_2$, t), 2.23 (2H, CH_2–CI_2, t), 3.56 (6H, CH_3, s); IR (neat) ν 1095 and 1056 (C–O–C), 678 and 632 (C–I) cm^{-1}. Elemental analysis and ^{13}C NMR could not be obtained for **9c** due to slow decomposition of the sample on storage.
(2,2-Diiodo-1,1-dimethoxyethyl)cyclohexane or1,1-Diiodo-2,2-dimethoxyethyl-2-cyclohexylethane (**9c**): Colorless solid: mp 34–35 °C (decomp.); ^{1}H NMR: δ 1.12–1.26 (6H, CH_2, m), 1.66 (2H, CH_3, m), 1.77 ($1H_a$, CH_2, m), 1.89 ($1H_b$, CH_2, m), 2.28 (H, CH, m), 3.45 (6H, CH_3, s), 5.47 (1H, CI_2, s); ^{13}C NMR: δ −18.5 (CHI_2), 26.3 (CH_2), 27.1 (CH_2), 29.2 (CH_2), 43.9 (CH), 50.6 (OCH_3), 51.8 (OCH_3), 97.1 ($C(OMe)_2$). Anal. Calcd for $C_{10}H_{18}I_2O_2$: C, 28.32; H, 4.28. Found: C, 28.70; H, 4.65.
2-Diiodomethyl-2-methoxytetrahydrofuran (**9d**): Oil: ^{1}H NMR: δ 2.05 (2H, CH_2, m), 2.35 (1H, CH_2, m), 2.44 (H, CH_2, m), 3.22 (6H, CH_3, s), 4.11 (2H, CH_2, m), 5.52 (1H, CI_2H, s); ^{13}C NMR: δ −20.1 (CI_2), 25.8 (CH_2), 38.1 (CH_2), 48.2 (OCH_3), 71.5 (CH_2–O), 108.0 (O–C–OMe); IR (neat) ν 1050 and 1022 (C–O–C), 690 and 647 (C–I) cm^{-1}. Anal. Calcd for $C_6H_{10}I_2O_2$: C, 19.59; H, 2.74. Found: C, 19.44; H, 2.81.
1,1-Diiodo-2,2-dimethoxyethyl-2-phenylethane (**9e**): Colorless solid: mp 84–86 °C (decomp.); ^{1}H NMR: δ 3.32 (6H, OCH_3, s), 5.56 (1H, CI_2H, s), 7.30–7.35 ($3H_{arom.}$, m) 7.62–7.67 ($2H_{arom.}$, m); ^{13}C NMR: δ −19.4 (CI_2), 49.0 (OCH_3), 97.4 ($C(OMe)_2$), 125.8, 127.3, 128.0, 133.7 ($C_{arom.}$); IR (KBr) ν 1089 and 1045 (C–O–C), 679 and 635 (C–I) cm^{-1}. Anal. Calcd for $C_{10}H_{12}I_2O_2$: C, 28.73; H, 2.89. Found: C, 28.86; H, 2.85.
2,2-Diiodo-1,1-dimethoxyethyl-1-phenylpropane (**9f**): Colorless solid: mp 65–66 °C (decomp.); ^{1}H NMR: δ 2.86 (3H, CH_3, s), 3.59 (6H, OCH_3, s), 7.37–7.40 ($3H_{arom.}$, m) 7.64–7.66 ($2H_{arom.}$, m); ^{13}C NMR: δ 15.1 (CH_3), 45.3 (CI_2), 53.89 (ICH_3), 102.3 ($C(OMe)_2$), 126.2, 128.0, 131.2, 132.5 ($C_{arom.}$); IR (KBr) ν 1068 and 1015 (C–O–C), 694 and 665 (C–I) cm^{-1}. Anal. Calcd for $C_{11}H_{14}I_2O_2$: C, 30.58; H, 3.27. Found: C, 30.71; H, 3.59.