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Continuous Flow Electrochemical Generator of Hypervalent Iodine Reagents: Synthetic Applications

Mohamed Elsherbini, Bethan Winterson, Haifa Alharbi, Ana A. Folgueiras-Amador, Célina Génot and Thomas Wirth*[a]

Abstract: An efficient and reliable electrochemical generator of hypervalent iodine reagents has been developed. In the anodic oxidation of iodoarenes under flow conditions to hypervalent iodine reagents the use of electricity replaces hazardous and costly chemical oxidants. Unstable hypervalent iodine reagents can be prepared easily and coupled with different substrates to achieve oxidative transformations in high yields. The unstable electrochemically generated reagents can also easily be transformed into classic bench stable hypervalent iodine reagents via ligand exchange. The combination of electrochemical and flow chemistry advantages largely improves the ecological footprint of the overall process compared to conventional approaches.

Hypervalent iodine compounds are efficient and versatile reagents with numerous applications in modern organic synthesis.[1] Their strong leaving group ability and electrophilic nature make them great candidates to perform selective oxidative transformations including oxidative rearrangements,[2] difunctionalization of alkenes and cyclopropanes,[3] oxidative C–C, C–heteroatom and heteroatom–heteroatom couplings and dearomatization of phenols.[4] Hypervalent iodine reagents are used under mild reaction conditions and present low toxicity, which makes them environmentally friendly reagents often used as an alternative to heavy metal oxidants. However, their synthesis from iodine(I) compounds usually requires stoichiometric amount or an excess of chemical oxidants, such as peroxides (hydrogen peroxide, *meta*-chloroperbenzoic acid, peracetic acid, etc.), Selectfluor® or Oxone®, making the process tedious and less convenient.[5]

The anodic oxidation of aryl iodides is an alternative to hazardous and sometimes costly chemical oxidants and has been successfully applied for the generation of hypervalent iodine compounds.[6] Organic electrosynthesis is known for a very long time and recently, a resurgence of this area has been observed. Electrons are used as a sustainable tool to perform oxidations and reductions in a clean manner, due to the replacement of chemical oxidizing and reducing reagents by inexpensive electricity.[7] Moreover, flow electroorganic synthesis has grown significantly in the last few years[8] and it is a useful tool to perform electroorganic transformations in a more efficient way, overcoming some of the limitations of batch electrolysis, such as the low conductivity of organic solvents, large current gradients and slow reaction rates.[9] Recent innovations in flow cells allow selective and successful syntheses[10] as the electrode surface-to-reactor

volume ratio is improved, translating into a higher mass transfer, and more importantly, a higher productivity.

Initially, common stable hypervalent iodine reagents such as Koser's reagent **2** and (diacetoxyiodo)benzene **3** were targeted in the electrochemical flow synthesis. All our trials to obtain Koser's reagent **2** through anodic oxidation of an iodobenzene solution in acetonitrile in the presence of *p*-toluene sulfonic acid (*p*-TsOH) in flow (Scheme 1) were unsuccessful due to blockage of the channels of the electrochemical flow cell, consequently leakage was observed after few minutes (see SI, Table S1). The blockage is attributed to the poor solubility of **2** under the applied flow conditions. The synthesis of the more soluble reagent (diacetoxyiodo)benzene **3** (Scheme 1) through electrolysis of a solution of iodobenzene in acetonitrile in the presence of 5 equivalents of acetic acid under various flow conditions resulted in poor conversion of the starting material in addition to the formation of 4-iodophenyl acetate **4** as a side product. Varying the amount of acetic acid had a great effect on the reaction outcome; a high conversion of the starting material (85%) and a high ratio of **3** : **4** (10:1) was achieved when 40 equivalents of acetic acid were used, but the isolated yield of (diacetoxyiodo)benzene was only 25% (see SI, Table S2).
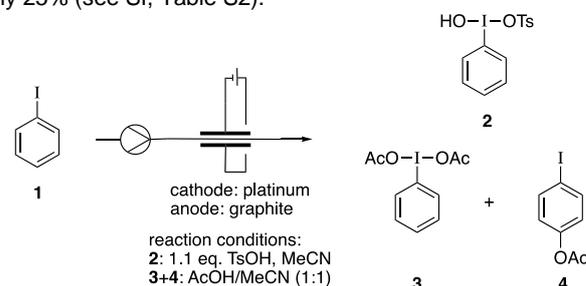
Scheme 1. Attempted direct synthesis of stable iodine(III) derivatives *via* anodic oxidation of iodobenzene in flow.

These preliminary results indicate that an electrochemical oxidation of iodobenzene is possible, but either insolubility of the product **2** or oxidation of the aromatic moiety as seen in **4** are problematic. We then investigated the synthesis of hypervalent iodine reagents with ligands derived from fluorinated alcohols. Although such reagents are not bench stable and cannot be isolated, they are stable in solution as their synthesis has been reported in batch-type electrolysis cells.[11] The anodic oxidation of iodobenzene in fluorinated alcohols under flow conditions was investigated in detail, varying the electrode materials, concentrations of starting material iodobenzene and supporting electrolyte, flow rate, current, and the alcohol used as solvent and as source of the ligands.

Initially, 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) was investigated in the reaction. As the voltage was high in the initial experiments, tetrabutylammonium tetrafluoroborate (TBATFB) was added as a supporting electrolyte in low concentration (5 mM, 0.1 eq.) which is much lower compared to required amount (0.1 – 1.0 M, 2 – 20 eq.) of supporting electrolyte in batch electrolysis. This led to product formation in 90% yield (Table 1, entry 4).

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Hence, the reaction parameters were studied in the presence of Bu₄NBF₄. Screening various electrode materials showed that the reaction yield was good to high in the presence of supporting electrolyte (Table 1, entries 4-6), but no electrode pair gave a better result than the Pt-graphite pair (see the Supporting Information, Table S4). Increasing the current from 24 mA (3 F) to 28 mA (3.5 F) did not lead to an improvement (entry 7), while reducing the current to 20 mA (2.5 F) lead to a decrease of the yield to 79% (entry 8). A slight improvement was attained by lowering the flow rate to half (0.05 mL/min) and doubling the iodobenzene concentration (0.1 M), while maintaining the same current (24 mA, 3F, entry 9, 93%). Then, the reaction was studied using different concentration / flow rate combinations while keeping the same amount of charge (3 F), but the reaction yield was lower in all cases compared to entry 9 (see the Supporting Information, Table S5). As the fluorinated alcohols cause some erosion to graphite, a glassy carbon (GC) anode was used instead[12] leading to the formation of the product **5a** in a quantitative yield (entry 10). Changing the alcohol from HFIP to 2,2,2-trifluoroethanol (TFE) resulted in the corresponding hypervalent iodine reagent in 70% yield (entry 11). Increasing the current to 28 mA (3.5 F) led to an improvement of the yield to 80% (entry 12), further increase of the current to 32 mA (4 F, entry 13) did not lead to further improvement of the yield (see the Supporting Information, Table S6).

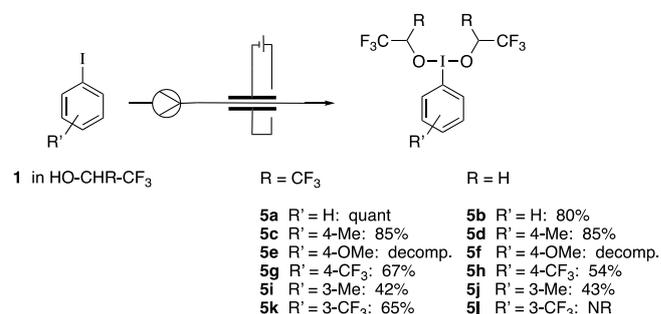**Table 1.** Optimization of the anodic oxidation of iodobenzene **1** under flow conditions

1 in HO-CHR-CF₃

5a R = CF₃
5b R = H

Entry	Solvent	Cathode	Anode	Concentration of **1** (mol/L)	Flow rate (mL/min)	Current (mA)	Yield **5** [%][a]
1	HFIP	Pt	graphite	0.05	0.1	24 (3 F)	27[b]
2	HFIP	graphite	Pt	0.05	0.1	24 (3 F)	15[b]
3	HFIP	Pt	Pt	0.05	0.1	24 (3 F)	43[b]
4	HFIP	Pt	graphite	0.05	0.1	24 (3 F)	90
5	HFIP	Pt	Pt	0.05	0.1	24 (3 F)	70
6	HFIP	Pt	BDD	0.05	0.1	24 (3 F)	60
7	HFIP	Pt	graphite	0.05	0.1	28 (3.5 F)	86
8	HFIP	Pt	graphite	0.05	0.1	20 (2.5 F)	79
9	HFIP	Pt	graphite	0.1	0.05	24 (3 F)	93
10	HFIP	Pt	GC	0.1	0.05	24 (3 F)	quant
11	TFE	Pt	GC	0.1	0.05	24 (3 F)	70
12	TFE	Pt	GC	0.1	0.05	28 (3.5 F)	80
13	TFE	Pt	GC	0.1	0.05	32 (4 F)	80
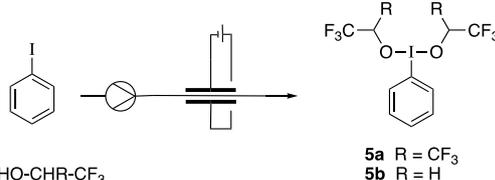
Supporting electrolyte (TBATFB) = 0.005 M; [a] Yield determined by ¹H NMR with PhNO₂ as an internal standard; [b] Experiment performed without TBATFB. BDD: boron-doped diamond; GC: glassy carbon.

To evaluate the possibility of coupling the generation of the electrochemically generated hypervalent iodine reagents with a second step in flow, the stability of the electrolysis step was investigated. Three different experiments applying the conditions

of Table 1, entries 9, 10 and 12 were run and analysed every 30 minutes for 4 hours. With HFIP as solvent the yield slowly decreases after the first 90 minutes, the formation of an insoluble black solid was observed in case of using graphite as anode or a yellow precipitate on the glassy carbon anode. Although the reaction yield with TFE is lower at the beginning (80%), it remained unchanged over time and a stream of a stable concentration of the product was achieved without electrode passivation even after 6 hours. The yield is still higher than reported for similar transformations using batch-type electrolysis (67%).[11c] In addition, the productivity (space-time yield) of this process is about 2.5 fold higher than the same reaction under batch electrolysis conditions.[13] Hence TFE was the solvent of choice for further investigations applying the reaction conditions of entry 12 in Table 1.

As shown in Scheme 2, a range of iodoarenes can be oxidized to the corresponding hypervalent iodine reagents under flow conditions in good to excellent yield except for 4-iodoanisole, which decomposed under the reaction conditions in both HFIP and TFE. Decomposition of 4-iodoanisole in TFE under electrolysis conditions was also previously reported in batch-type cells.[14] Although 3-iodobenzotrifluoride was oxidised to the corresponding hypervalent iodine reagent **5f** in 65% yield, it failed to react in TFE, and the starting material was recovered. All these hypervalent iodine reagents are only stable in solution and decompose immediately upon removal of the solvent hence the yields were determined by ¹H NMR with nitrobenzene or 1,4-dinitrobenzene as internal standard.
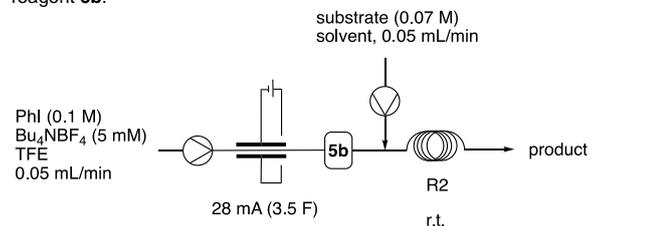

1 in HO-CHR-CF₃   R = CF₃   R = H

5a R' = H: quant   **5b** R' = H: 80%
5c R' = 4-Me: 85%   **5d** R' = 4-Me: 85%
5e R' = 4-OMe: decomp.   **5f** R' = 4-OMe: decomp.
5g R' = 4-CF₃: 67%   **5h** R' = 4-CF₃: 54%
5i R' = 3-Me: 42%   **5j** R' = 3-Me: 43%
5k R' = 3-CF₃: 65%   **5l** R' = 3-CF₃: NR

Scheme 2. Anodic oxidation of iodoarenes under flow conditions. Yields determined by crude ¹H NMR with with nitrobenzene or 1,4-dinitrobenzene as internal standard.

To further demonstrate the efficiency of the developed electrochemical generator of hypervalent iodine reagents, several typical hypervalent iodine mediated transformations were investigated in a coupled manner. For all reactions, the solution of the electrochemically generated hypervalent iodine reagent **5b** was mixed with the substrate by pumping it together through reactor R2, and the results are summarized in Table 2.

The oxidation of thioanisoles[15] (Table 2, entries 1+2) proceeded smoothly to the corresponding sulfoxides in excellent yields. The oxidative dearomatization[16] of hydroquinone to benzoquinone proceeded quantitatively (entry 3) while the cyclization of 3-(4-hydroxyphenyl)propanoic acid[14] afford the spirolactone in 93% yield (entry 4). Under the same conditions, benzoxazoles[11d] were obtained in high yields from the corresponding *ortho*-hydroxyimines (entries 5-7). Furthermore, the α-acetoxylation[17] and tosyloxylation[15] of acetophenone and propiophenone was

easily achieved in high yields using the same reaction set-up, but by increasing the residence time to 20-30 minutes (entries 8-12). Treatment of an amide with **5b** in batch needed 48 hours to give the carbazole derivative in 60% yield.[18] Performing this transformation using our protocol (10 min at r.t.) led to the for-

Table 2. Synthetic applications of the electrogenerated hypervalent iodine reagent **5b**.
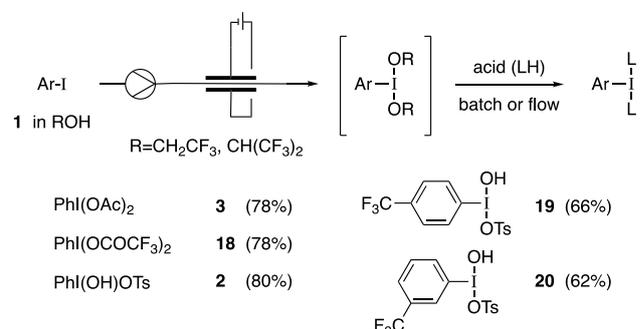
Entry	Substrate	Product	R2 residence time [min]	Yield [%]
1	PhSMe	PhS(O)Me **6**	10	84[a]
2	4-Cl-C$_6$H$_4$SMe	4-Cl-C$_6$H$_4$S(O)Me **7**	20	98[a]
3	HO—C$_6$H$_4$—OH	O=C$_6$H$_4$=O **8**	10	quant. [a]
4	HO$_2$C / HO	**9**	10	93[b]
5	N=Ph / OH	N—Ph **10**	10	quant. [a]
6	N=/Ph / OH	N—/Ph **11**	10	68[a]
7	N / O —OMe	N / O —OMe **12**	10	62[a]
8	Ph—C(O)—CH$_3$	Ph—C(O)—CH$_2$OAc **13**	30	77[c]
9	Ph—C(O)—CH$_2$CH$_3$	Ph—C(O)—CH(OAc)CH$_3$ **14**	30	82[c]
10	Ph—C(O)—CH$_3$	Ph—C(O)—CH$_2$OTs **15**	20	52[d]
11	Ph—C(O)—CH$_2$CH$_3$	Ph—C(O)—CH(OTs)CH$_3$ **16**	20	62[d]
12			30	83[d]
13	biphenyl-NHAc	carbazole-NAc **17**	60	48[b,e]

[a] Solvent for substrate: MeCN; [b] Solvent for substrate: TFE; [c] Solvent for substrate: AcOH/MeCN 1:4, 30 mol% BF$_3$•OEt$_2$; [d] Solvent for substrate: MeCN, 1.2 eq. TsOH; [e] Reactor temperature (R2): 50 ºC.

mation of the carbazole in trace amounts. Increasing the residence time to 60 min at 50 ºC led to the isolation of the carbazole in 48% yield (entry 13). The above results show the efficiency and reliability of the electrochemical hypervalent iodine reagents generation. The yields of the final products are generally high and can be easily improved by controlling the residence time and temperature of the second reaction. All reactions shown in Table 2 have been performed on an approx. 0.4 – 1.0 mmol scale leading to product amounts between 44 and 134 mg.

Finally, the electrochemically generated bench unstable hypervalent iodine reagents of type **5** were used for the facile synthesis of bench stable classic hypervalent iodine reagents such as (diacetoxyiodo)benzene **3**, [(bistrifluoroacetoxy)iodo]benzene **18**, and Koser's reagent **2** via quantitative ligand exchange with the appropriate acids. In addition, this protocol (electrochemical oxidation / ligand exchange) enabled an easy synthesis of electrondeficient reagents **19** and **20** that are difficult to prepare by oxidation of trifluoroiodotoluene derivatives using chemical oxidants. Both compounds have been analyzed by X-ray structural analysis.[19] The ligand exchange was achieved by coupling the flow of the electrochemically generated hypervalent iodine reagent with a second flow of the appropriate acid / acid solution or by collecting the electrochemically generated reagents in a flask containing the appropriate acid (Scheme 3). The given yields relate to the iodoarene starting materials **1**. Compound **20** was also prepared on a slightly larger scale and 214 mg were isolated after a 2.5 h reaction. In contrast to electrolysis in HFIP, doubling the flow rate and current while using TFE enabled a larger scale preparation of (diacetoxyiodo)benzene **3** (1.2 g, 75%) within 8 hours without significant decrease of the yield. The fluorinated alcohol (ROH) employed in the process can be distilled and reused as previously reported, making the whole process more sustainable.[20]

PhI(OAc)$_2$ **3** (78%) F$_3$C—C$_6$H$_4$—I(OH)OTs **19** (66%)
PhI(OCOCF$_3$)$_2$ **18** (78%)
PhI(OH)OTs **2** (80%) F$_3$C—C$_6$H$_4$—I(OH)OTs **20** (62%)

Scheme 3. Synthesis of bench stable hypervalent iodine reagents via ligand exchange.

In summary, an efficient and reliable electrochemical generator of hypervalent iodine reagents has been developed, replacing hazardous chemical oxidants by electricity. The anodic oxidation of iodoarenes under flow conditions proceeds smoothly in fluorinated alcohols using a glassy carbon anode and a platinum cathode. As these reagents are not bench stable and decompose immediately upon removal of solvent, their generation and immediate use in flow is therefore highly advantageous. Various hypervalent iodine mediated oxidative transformations were easily achieved in high yields by coupling the flow of the in situ electrochemically generated hypervalent iodine reagents with a second flow of various substrates using a simple reaction setup. The unstable electrochemically generated hypervalent iodine reagents can also be easily transformed into bench stable hypervalent iodine reagents via ligand exchange.

Experimental Section

The electrolysis was performed in an undivided cell using a Vapourtec Ion Electrochemical Flow Reactor (reactor volume = 0.6 mL, spacer 0.5 mm) using a glassy carbon electrode (immersed surface area: A = 12 cm^2) as the anode and a platinum electrode as the cathode. A solution of iodobenzene (0.1 M) in HFIP or TFE, containing Bu$_4$NBF$_4$ (0.005 M), was pumped into the electrochemical reactor with a syringe pump (0.05 mL/min) applying constant current conditions (24 mA, 3 F in case of HFIP or 28 mA, 3.5 F in case of TFE). After reaching a steady state (24 min, 2 reactor vol.), the solution was collected for 3 x 10 min and 5.5 μL of nitrobenzene was added to each collection as an internal standard. Due to the instability of the electrogenerated compound an aliquot was diluted with CDCl$_3$ and the yield was determined by integration of the aromatic peaks in the ^{1}H NMR spectrum obtained.

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COMMUNICATION

A fast and efficient generation of unstable iodine(III) reagents is reported. They can be either used directly in oxidative transformations or be converted into stable hypervalent iodine reagents.
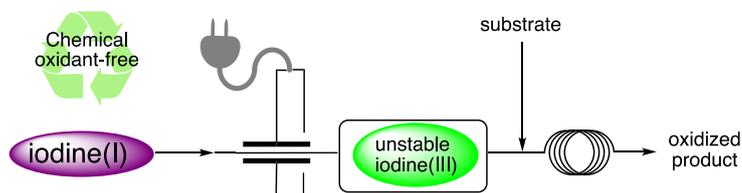
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