Review Article

Chemical Review and Letters
journal homepage: www.chemrevlett.com
ISSN (online): 2645-4947 (print) 2676-7279

Recent advances in iodofluorination of alkenes

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ARTICLE INFO

Article history:
Received 14 October 2024
Received in revised form 2 December 2024
Accepted 2 December 2024
Available online 20 August 2025

Keywords:
Organofluorine compounds, α-iodofluoroalkanes, alkenes, difunctionalization, iodofluorination.

ABSTRACT

This review gives an overview of recent findings and developments in research on direct iodofluorination of alkenes. The review is divided into two major sections according to iodofluorinating reagents. The first includes the iodofluorination of alkenes using bifunctional reagents, while the second contains the three-component reactions.

1. Introduction

Organofluorine compounds have found widespread applications in the field of pharmaceuticals, agrochemicals, and materials science owing to their outstanding physicochemical properties such as metabolic stability, lipophilicity, permeability, bioavailability, and excellent chemical and thermal stability [1-3]. Currently, about 25% of FDA-approved drugs and 35% (424 of 1,200) of commercial agrochemicals contain at least one fluorine atom in their structures (Scheme 1) [4]. Moreover, the radiochemical properties of fluorine-18 can be exploited in positron emission tomography (PET) radiopharmaceuticals [5]. The unique properties and wide ranging applications of fluorine-containing organic compounds in various scientific fields are providing a strong stimulus for the development of new strategic approaches to their synthesis, as well as new chemical methods [6, 7].

Fluorination-based 1,2-difunctionalization of double bonds in olefins is one of the most effective approaches for the synthesis of functionalized fluoroalkyl-containing organic compounds [8, 9]. By this powerful and viable strategy not only a fluorine (or fluorinated moiety) but also an additional functional group can be installed across the double bond within a single click [10-12] Subsequently, the second functionality can be employed as a handle for further manipulation of products. In this context, direct iodofluorination of inexpensive and widely available alkenes has attracted considerable attention as an efficient method for the simultaneous incorporation of both iodo and fluoro substituents to organic substrates (Figure 1). Although several interesting review articles have recently appeared related to the difunctionalization reactions, to our knowledge, no comprehensive review on iodofluorination of alkenes has yet been published. Our objective for this

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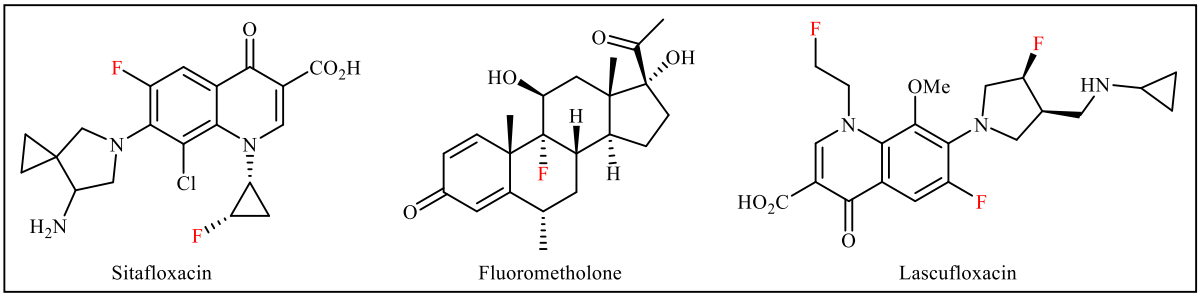
https://doi.org/10.22034/crl.2024.483453.1450



Scheme 1. Selected examples of drug molecules having fluorine atom(s).

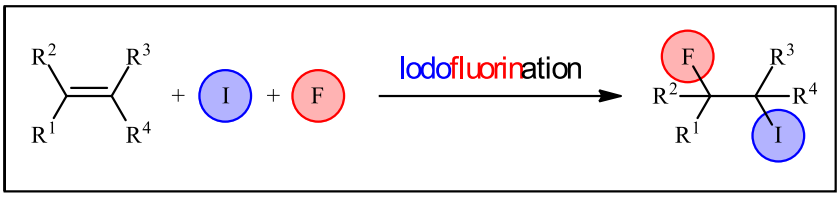


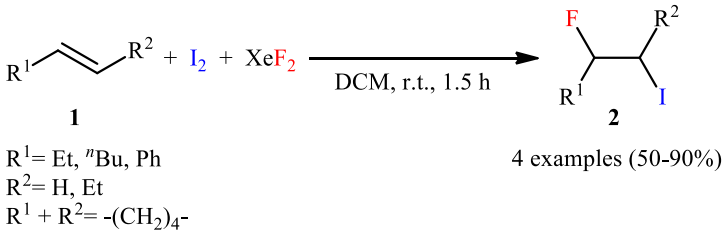
Fig. 1. Vicinal iodofluorination of alkenes.

paper is to summarize the available literature on 1,2-iodofluorination of alkenes published from 1966 to the end of end of July 2024

2. Two-component iodofluorination systems

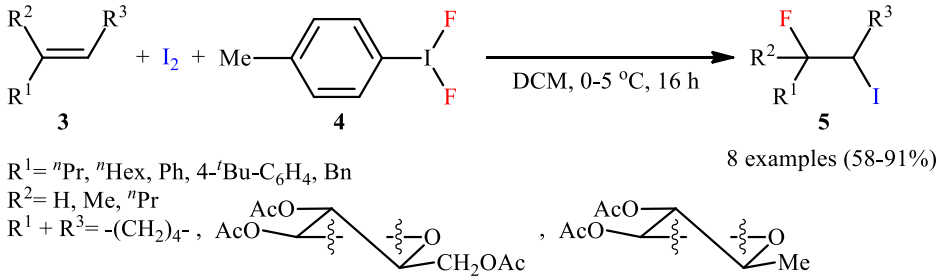
2.1. Iodofluorination systems based on I_2 and a fluorinating agent

One of the earliest reports on the direct iodofluorination of alkenes using molecular iodine (I_2)-based iodofluorinating systems was published by Shellhamer and co-workers in 1998 [13], who showed that the treatment of simple, unactivated alkenes **1** with I_2 and XeF_2 as the sources of iodine and fluorine, respectively, in the absence of any catalyst or additive in DCM, resulted in the formation of corresponding α-iodofluoroalkanes **2** in moderate to high yields as exclusively Markovnikov regioisomers with prevalent *anti*-stereoselectivity (Scheme 2). The same iodofluorination reaction was also investigated with an alkyne (*i.e.*, 3-hcayne). The respective 2-fluoro-1-iodo-1-alkene was obtained in moderate yield (50%). Notably, when arenes were subjected to this reaction, the corresponding mono-iodinated arenes were selectively obtained in synthetically useful yields.



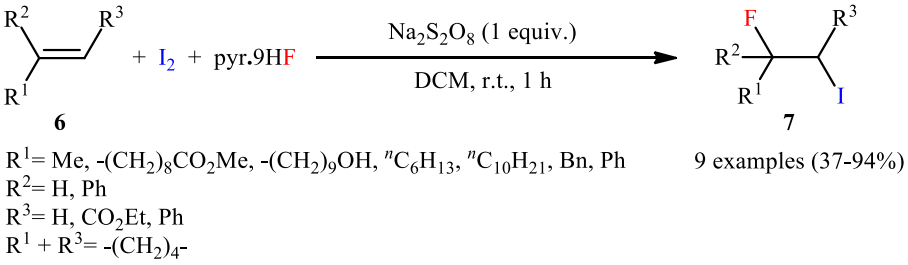
Scheme 2. Shellhamer's synthesis of α-iodofluoroalkenes **2**.

In 2006, the Tingoli group realized that 4-iodotoluene difluoride **4** could serve as good fluorine source in iodofluorination reactions of alkenes [14]. They showed that three-component reaction between alkenes **3**, 4-iodotoluene difluoride **4**, and I_2 under catalyst-/additive-free conditions formed a panel of eight corresponding α-iodofluoroalkenes **5** in good to excellent yields with outstanding stereo- and regio-selectivities (Scheme 3). The results showed that sterically less hindered alkenes gave higher yields as compared to bulkier ones. The relative reaction rates of alkenes in this difunctionalization reaction followed the order: terminal aromatic alkenes ≥ terminal aliphatic alkenes ≈ internal aliphatic alkenes > 1,1,2-trisubstituted alkenes. Under the identical conditions, three internal alkynes and one terminal alkyne were also successfully transformed into the respective iodofluoroalkenes.

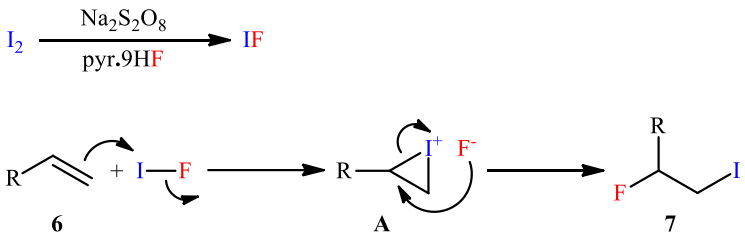


Scheme 3. Tingoli's synthesis of α-iodofluoroalkenes **5**.

Fifteen years later, in a related investigation, Kitamura and co-workers synthesized a library of α-iodofluoroalkene derivatives **7** in fair to excellent yields, ranging from 37% to 94%, via the reaction of corresponding alkenes **6** with I_2 and HF·pyridine complex (pyr·9HF) in the presence of $Na_2S_2O_8$ as an oxidant in DCM at room temperature (Scheme 4) [15]. It should be mentioned that the reaction could be easily scaled up to the gram-scale as exemplified by the synthesis of 2-fluoro-1-iodododecane on a 1.60 g scale (84%). Moreover, the obtained 2-fluoro-1-iodododecane was subjected to various substitution reactions with nitrogen, sulfur, and oxygen nucleophiles to show synthetic application of synthesized α-iodofluoroalkenes. Mechanistically, the reaction starts with the formation of IF through the reaction of I_2 with pyr·9HF in the presence of $Na_2S_2O_8$, which subsequently adds electrophilically to the alkene **6** to give the observed fluoroiodinated product **7** though an iodonium intermediate **A** (Scheme 5). Very recently, this innovative research group unraveled that treatment of alkene derivatives with TEA·5HF in the presence of a catalytic amount of I_2 and over-stoichiometric amounts of Selectfluor as the oxidant, afforded corresponding 1,2-difluoroalkanes as the sole products [16].



Scheme 4. Kitamura's synthesis of α-iodofluoroalkenes **7**.

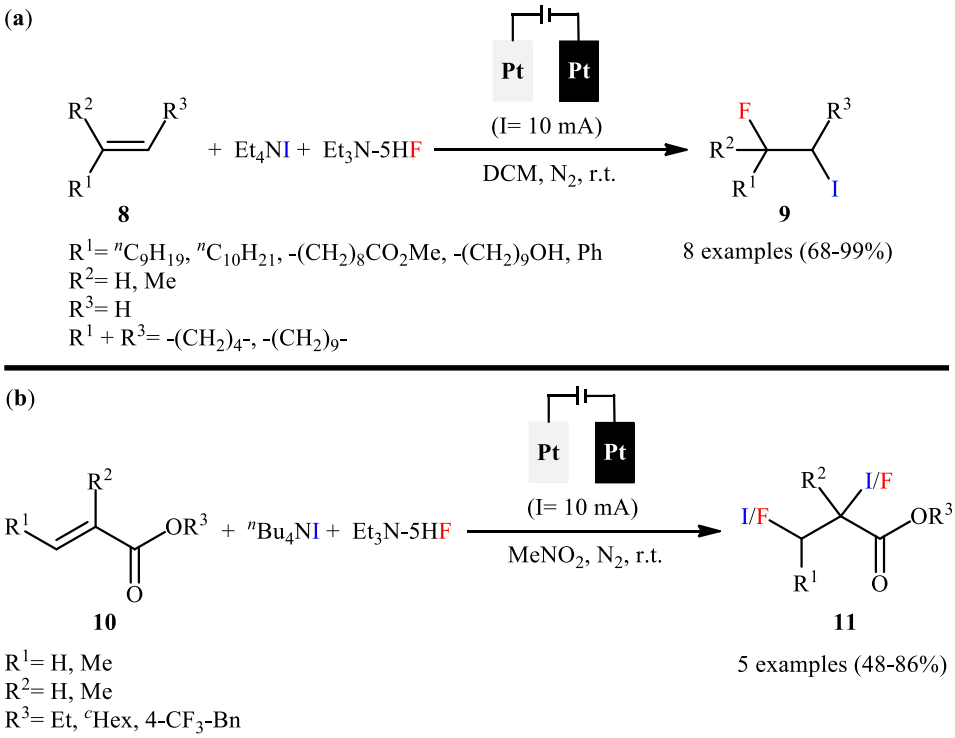


Scheme 5. Proposed mechanism for the formation of α-iodofluoroalkenes **7**.

2.2. Iodofluorination systems based on an ammonium iodide salt and a fluorinating agent

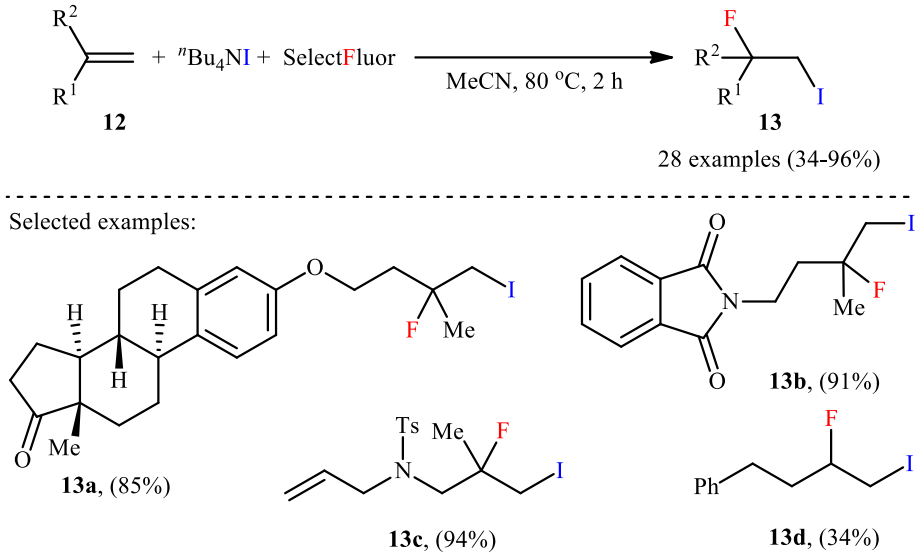
In 2001, Hara and co-workers reported the first example of the direct iodofluorination of olefinic double bonds using the combination of an ammonium iodide salt and a fluorinating agent as the iodine fluoride precursor [17]. They demonstrated regioselective iodofluorination of a series of terminal and internal alkenes **8** with Et_4NI and Et_3N-5HF in a divided cell assembled with two smooth Pt sheets under constant current conditions (10 mA). The reaction was conducted in DCM at room temperature and proceeded smoothly without the addition of any transition-metal catalyst or additive, which provides an efficient strategy for the synthesis of α-iodofluoroalkanes **9** (Scheme 6a). Notably, Et_3N-5HF played a dual role in this transformation; the fluorine source and the electrolyte. The

authors also examined the other iodinating agents, such as Ph_4PI, I_2, and LiI, using dodecene as the model substrate. However, they were found to be unsuitable for this reaction, and passivation on the anode took place before the complete consumption of dodecene. In this study, the authors also used dodec-1-yne as the substrate for this electro-iodofluorination system. The desired 2-fluoro-1-iodo-1-alkene product was obtained in good yield and outstanding stereo- and regioselectively. However, this iodofluorination reaction appears to be limited to simple alkenes and cannot be applied to electron-deficient alkenes. Eight years later, the group of Fuchigami investigated electrochemical iodo-fluorination of electron-deficient alkenes using Hara's iodofluorination systems [18]. Thus, in an undivided cell with two platinum electrodes a small library of α,β-unsaturated estersrs **10** treated with Et_4NI/Et_3N-5HF/$MeNO_2$ to give the respective α-iodofluoroalkanes **11** in moderate to high yields (Scheme 6b). However, the regioselectivity of products was modest at best. Moreover, in some cases, such as *p*-methoxybenzyl crotonate, the expected iodo-fluorinated product was not formed at all. Shortly afterwards, the same research group applied the similar strategy for iodofluorination of other electron-deficient alkenes such as α,β-unsaturated amides and phosphonates [19]. However, in most cases poor yields and/or regioselectivity were observed.

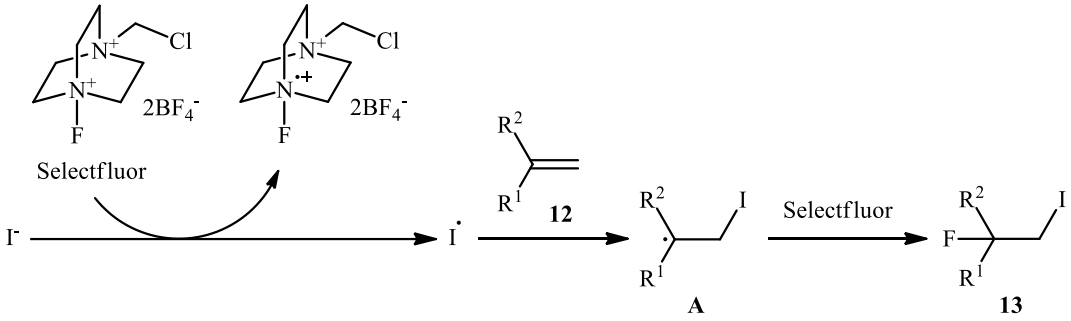


Scheme 6. (a) Hara's synthesis of α-iodofluoroalkanes **9**; (b) Fuchigami's synthesis of α-iodofluoroalkanes **11**.

Drawing inspiration from these works, recently, Qian *et al* have described a three-component α-iodofluoroalkanes **13** synthesis by reaction between terminal alkenes **12**, $^nBu_4NI$, and Selectfluor in near refluxing MeCN in the absence of any catalyst or additive [20]. This procedure efficiently provided the desired vicinal iodofluorinated products in modest to excellent yields and complete anti-Markovnikov-selectivity within 2 h (Scheme 7). In addition, a tolerance for cyclooctene (a cyclic alkene) was also demonstrated. The reaction was also successfully applied to the "late-stage iodofluorination" of medicines and natural products, such as probenecid, estrone and tyrosine. The large-scale synthesis of this transformation was carried out in 8 mmol scales using 2-(3-methylbut-3-en-1-yl)isoindoline-1,3-dione as the model substrate, and an excellent yield was obtained (86%, 2.48 g). Concerning the substrate scope, the reaction works better with electron-rich and –neutral than with electron-poor substrates (64% yield for 4-methyl-styrene compared to 48% for the 4-chloro derivative). After a series of mechanistic investigations and radical trapping experiments using 2,6-di-tert-butyl-4-methylphenol (BHT) and hydroquinone as radical scavengers, it was confirmed that this iodofluorination reaction most likely proceeds *via* a radical pathway as depicted in Scheme 8.



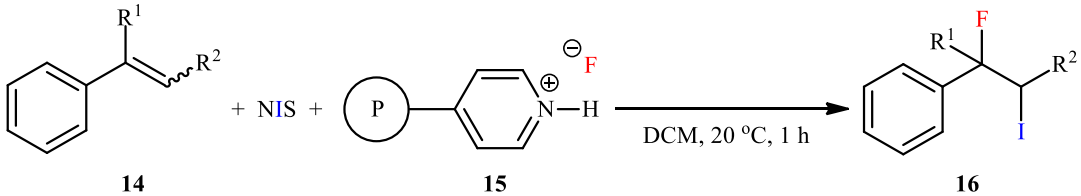
Scheme 7. Iodofluorination of alkenes **12** developed by Qian *et al*.



Scheme 8. Proposed mechanistic pathways for the formation of α-iodofluoroalkanes **13**.

2.3. Iodofluorination systems based on NIS and a fluorinating agent

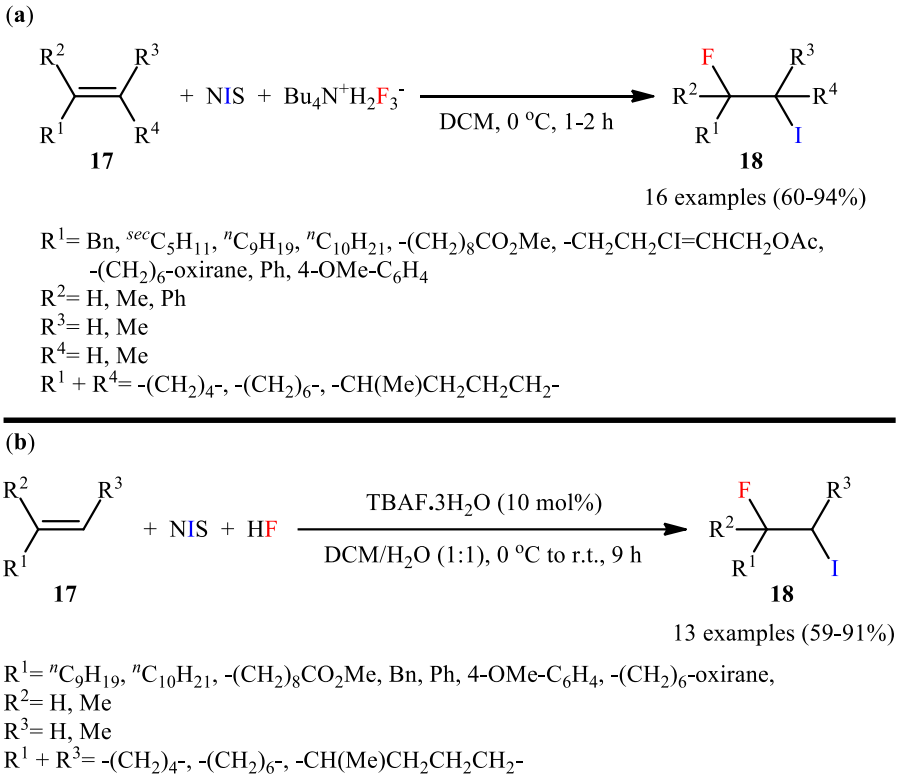
Drawing inspiration from the preliminary work by Fisher and co-workers on direct iodofluorination of tri-*O*-acetyl-D-glucal using *N*-iodosuccinimide (NIS)/HF system [21], in 1987, Zupan's research group disclosed an efficient stereospecific vicinal iodofluorination of styrene derivatives **14** employing NIS as a source of electrophilic iodine and an insoluble poly(4-vinylpyridine-co-styrene) supported hydrogen fluoride **15** as the source of fluorine [22]. The reaction was carried out in DCM at 20 °C under additive-free conditions, proceeded with Markovnikov regioselectivity, and generally afforded the desired iodofluorination products **16** in poor to high yields and excellent *anti*-stereospecificity (Scheme 9).



Scheme 9. Zupan's synthesis of (1-fluoro-2-iodoethyl)benzenes **16**.

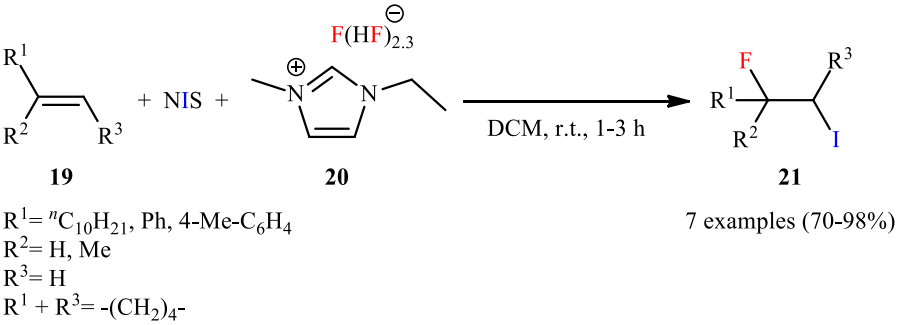
Following these works, Hiyama and co-workers reacted various terminal and internal alkenes **17** with NIS and tetrabutylammonium bifluoride (TBABF) in DCM at 0 °C to selectively provide α-iodofluoroalkanes **18** in good to

excellent yields (60–94%) and outstanding regio- and stereo-selectivity (Scheme 10a) [23]. In their subsequent studies, they reinvestigated the same reaction by replacing TBABF with HF and using a catalytic amount of TBAF·3H_2O in binary solvent DCM/H_2O with ratio 1:1 at room temperature (Scheme 10b) [24]. No obvious difference in yield of products by both methods was observed. Later, the authors successfully extended this chemistry to iodofluorinationo of bicyclo[2.2.1]hept-2-ene and cyclopentene derivatives [25].



Scheme 10. Hiyama's synthesis of α-iodofluoroalkanes **18**.

In 2004, Matsubara and co-workers reported the related vicinal iodofluorination of alkenes **19** with NIS and ionic liquid, 3-ethyl-1-methyl-imidazorium oligo hydrogen fluoride **20** which did not require any catalyst or additive [26, 27]. The reactions were carried out in DCM under ambient conditions, tolerated various terminal, internal (cyclic and acyclic), 1,1-disubstituted, and 1,1,2-trisubstituted alkenes, and generally delivered the expected Markovnikov regiochemistry α-iodofluoroalkane *anti*-products **21** in good to almost quantitative yields, ranging from 70% to 98% (Scheme 11). In this study, the authors highlighted the usefulness of their methodology by investigation of synthetic application of prepared compounds using the known dehydroiodination reactions. Bromofluorination of the same set of alkenes were also occurred under the standard conditions using *N*-bromosuccinimide (NBS) instead of NIS.

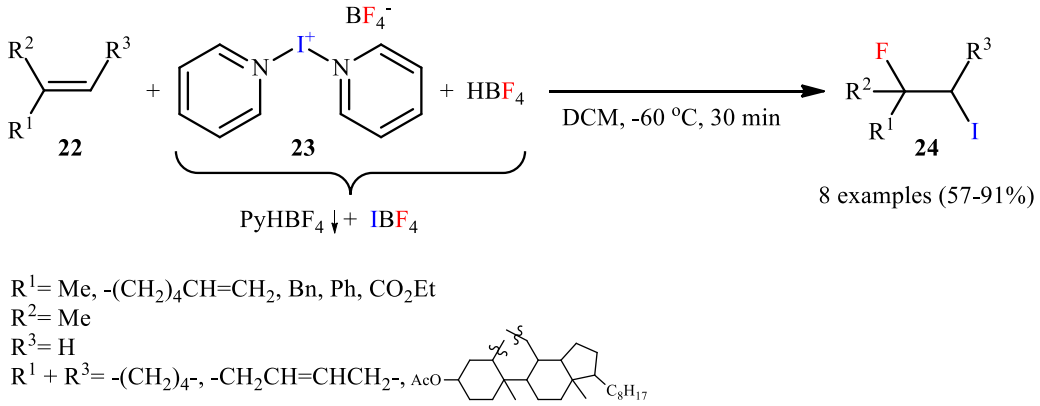


Scheme 11. Matsubara's synthesis of α-iodofluoroalkanes **21**.

Later, Walkowiak's research group reported that 1,1,3,3,3-pentafluoropropene-diethylamine complex (PFPDEA) could also be used as a fluoride source in iodofluorination reactions of alkenes using NIS as a source of electrophilic iodine [28]. However, in this study only 5 examples were provided. Moreover, requirement for over-stoichiometric amounts of toxic hexamethylphosphoric triamide (HMPA) and the production of a mixture of Markovnikov (major) and anti-Markovnikov (minor) addition products can be considered as the main drawbacks of this synthetic strategy. Along this line, very recently, Kiss and co-workers were able to synthesize a small library of cyclic α-iodofluoroalkanes (two examples) through the iodofluorination of corresponding cyclic alkenes under the action of NIS/Deoxo-Fluor system [29]. It should be mentioned that similar to iodofluorination of alkenes, the combination of NIS with various nucleophilic fluorine transfer reagents has also been successfully applied in the iodofluorination of allene and alkyne substrates [30-32].

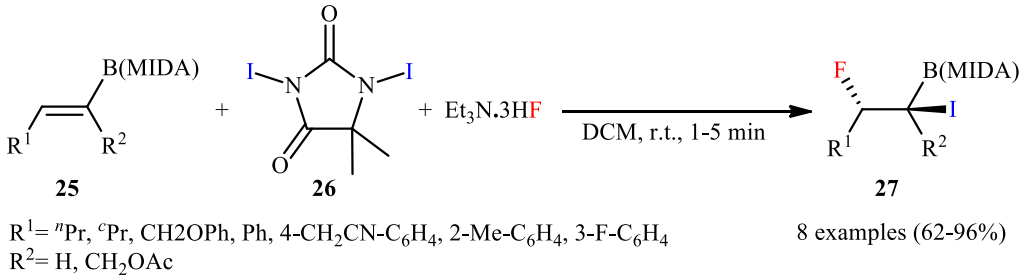
2.4. Related reactions

In 1991, Barluenga and co-workers found that a combination of bis(pyridine)iodonium tetrafluoroborate (IPy_2BF_4; **23**) and HBF_4 is a suitable reagent for direct iodofluorination of alkenes **22** under catalyst- and additive-free conditions [33]. As shown in Scheme 12, various alkenes **22** including 1-substituted, 1,2-disubstituted, and 1,1-dialkyl substituted alkenes were compatible by this reaction. However, the procedure was unsuccessful for 1,1-diaryl alkenes, and instead of the expected iodofluorinated products, corresponding (2-iodoethene-1,1-diyl)diarenes were obtained as the sole products.



Scheme 12. Barluenga's synthesis of α-iodofluoroalkanes **24**.

In this context, in 2020, Wang's research team described an interesting vicinal iodofluorination of alkenyl MIDA (*N*-methyliminodiacetyl) boronates **25** using 1,3-diiodo-5,5,-dimethylhydantoin **26** an electrophilic iodo source and $Et_3N{\cdot}3HF$ as a nucleophilic fluorine source in DCM at room temperature [34]. Various functionalized alkenyl MIDA (*N*-methyliminodiacetyl) boronates **25** were well tolerated under the standard conditions and delivered the respective *trans* iodofluorination products **27** in good to excellent yields (Scheme 13). In addition to the high stereoselectivity, an exclusive regioselectivity was observed, thanks to the directing effect of MIDA boron, in which iodo atom predominantly attached α to the C–B bond.

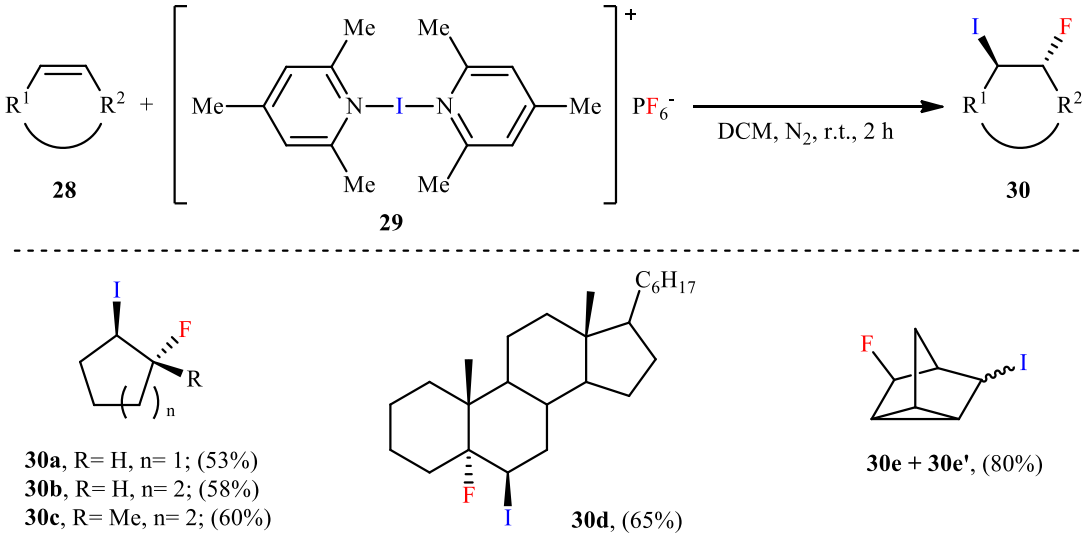


Scheme 13. Wang's synthesis of α-iodofluoroalkanes **27**.

3. Bifunctional iodofluorination reagents

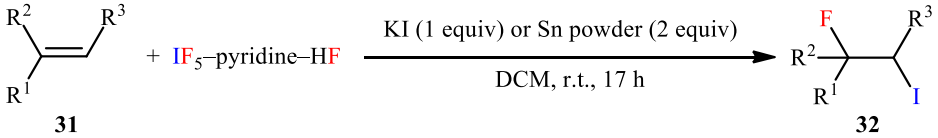
Methyliodine(III) difluoride (CH_3IF_2) is one of the earliest bifunctional iodofluorinating reagent which was first reported in the mid-1970s by Zupan and co-workers to iodofluorination of alkenes [35-38]. However, this reagent never gained popularity and was soon forgotten.

In 1987, Evans and Schauble demonstrated that the iodofluorination of a range of cyclic alkenes **28** can be achieved using bis(2,4,6-trimethylpyridine)iodine(I) hexafluorophosphate **29** in DCM at room temperature to give the corresponding α-iodofluoroalkanes **30** in moderate to good yields within 2 h (Scheme 14) [39]. Notably, that the identical condition was also successfully used for iodofluorination of a series of 2,4-diacycloxy-3,4-dihydro-2*H*-pyrans (glycal esters). However, reaction of bicycle[2.2.1]heptene with bis(2,4,6-trimethylpyridine)iodine(I) hexafluorophosphate under similar conditions gave 3-iodotricyclo[2.2.1.0]heptane as the only isolable product.



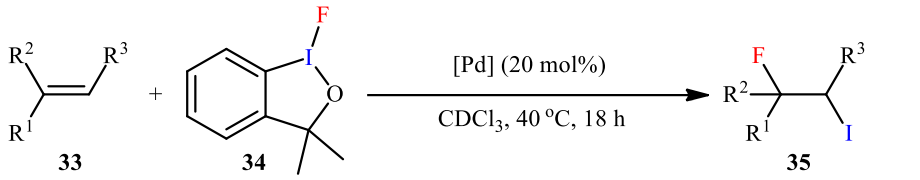
Scheme 14. Evans-Schauble's synthesis of α-iodofluoroalkanes **30**.

In 2015, Yano and Hara have shown that IF5–pyridine–HF can be used in the presence of a reductant such as potassium iodide (KI) or tin (Sn) powder to effect the iodofluorination of alkenes **31** in moderate to good yields (Scheme 15) [40]. Employing this protocol, the addition of IF to the double bond proceeded with stereo- and regioselectivity, in which *trans*-addition products were obtained selectively. As the reaction was conducted under very mild conditions, functional groups such as hydroxyl, ketone, and ester functionalities were well tolerated Furthermore, it was possible to distinguish between two double bonds of different reactivities, in which the iodofluorination selectively took place at the more reactive double bond. Shortly afterwards, the same authors extended the substrate scope of this chemistry to alkynes [41]. Thus, several iodofluoroalkenes were selectively synthesized in good yields (60-81%, 11 examples) *via* iodofluorination of the corresponding alkynes.



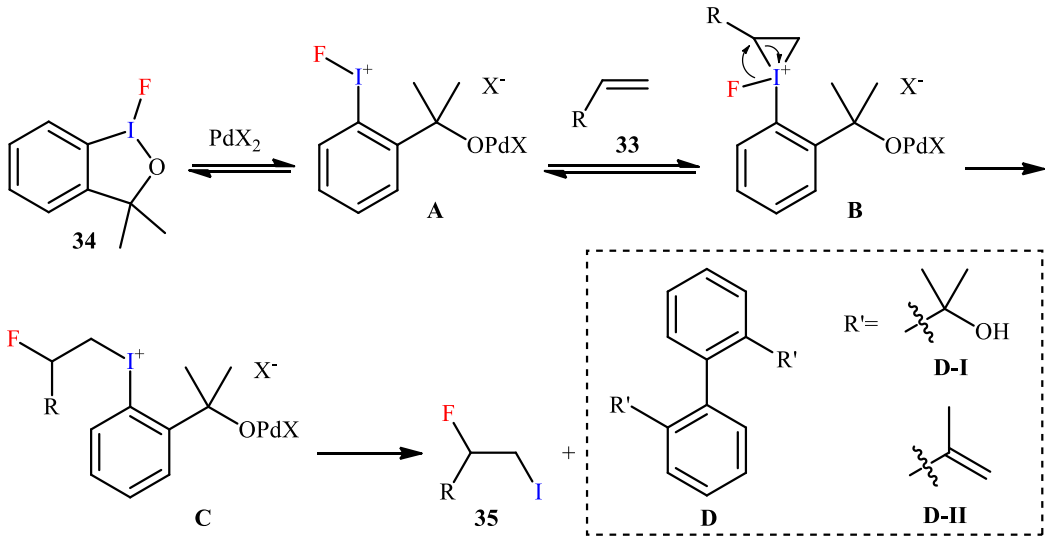
Scheme 15. Yano-Hara's synthesis of α-iodofluoroalkanes **32**.

In a significant contribution in this field, Szabo and co-workers developed an interesting Pd-catalyzed iodofluorination of alkenes using 1-fluoro-3,3-dimethyl-1,3-dihydro-1λ^3-benzo[*d*][1,2]iodaoxole as an air- and moisture-stable fluoroiodane reagent [42]. Using 20 mol% of certain palladium catalysts [*i.e.*, $Pd(BF_4)_2(MeCN)_4$, $Pd(TFA)_2$, $PdCl_2(MeCN)_2$], a library of alkenes **33** underwent regioselective iodofluorination with hypervalent iodine **34** to afford the corresponding α-iodofluoroalkanes **35** in modest to high yields (Scheme 16). The results indicated that iodofluorination of electron-rich alkenes proceeded faster and cleaner than for electron-deficient ones. No conversion of alkene starting material was observed in the absence of catalyst. According to the authors proposed mechanism (Scheme 17), palladium catalyst is necessary for the activation of hypervalent iodine reagent.



[Pd] = $Pd(BF_4)_2(MeCN)_4$, $Pd(TFA)_2$, $PdCl_2(MeCN)_2$
R^1= Ph, 4-Br-C_6H_4, Bn, CH_2Bn, 4-Me-Bn, 4-OMe-Bn, 4-F-Bn, 4-CF_3-Bn, 2-Me-Bn, 2-OMe-Bn, 2-Br-Bn, 3,4-$(OMe)_2$-Bn, 2-naphthyl
R^2= H, Me
R^3= H, Me
$R^1 + R^3$= -$(CH_2)_4$-, -$(CH_2)_5$-

Scheme 16. Szabo's synthesis of α-iodofluoroalkanes **35**.



Scheme 17. Proposed mechanistic pathways for the formation of α-iodofluoroalkanes **35**.

4. Conclusion

The direct vicinal difunctionalization of alkenes is a simple and efficient method for rapid enrichment of the structural complexity by concomitant introduction of two functional groups across the carbon–carbon double bonds. In this context, the vicinal fluorofunctionalization of alkenes has received continuous interests as it provides a strong synthetic route to convert feedstock alkenes into valuable fluorinated molecules. Among them, the vicinal iodofluorination has attracted considerable attention from researchers as the iodo functionality can be employed as a handle for further manipulation of products to build up more complex molecules. As illustrated, various iodofluorinating systems were developed and applied in this chemistry. Importantly, majority of reported iodofluorination reactions were performed under mild and catalyst-/additive-free conditions. We conclude this review by hoping that it will inspire researchers to make further progress in this chemistry and investigate the synthetic applicability of these reactions in the fabrication of natural products and biologically important compounds.

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