

Photochemistry

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Fluorinated Sulfinates as Source of Alkyl Radicals in the Photo-Enantiocontrolled β -Functionalization of Enals

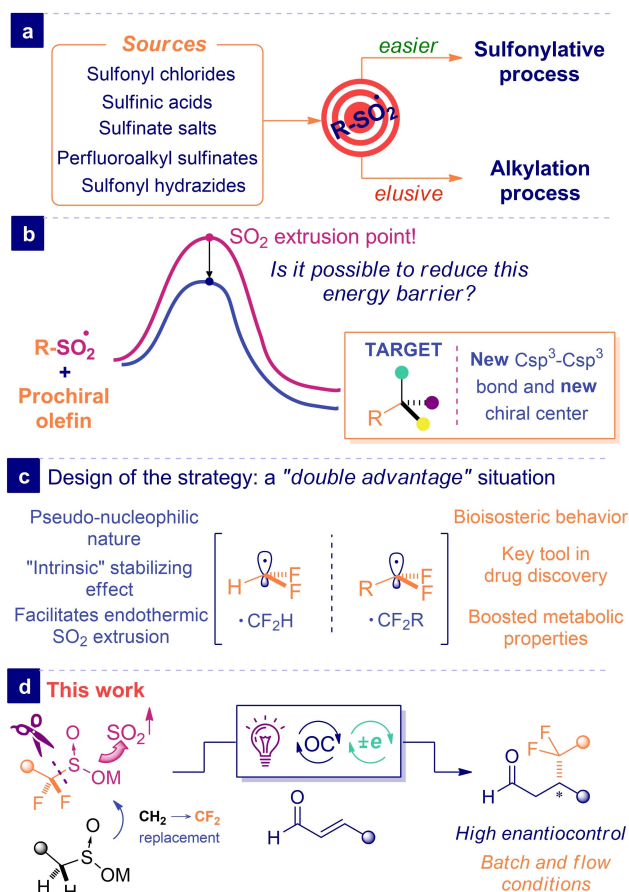
 Ricardo I. Rodríguez⁺, Marina Sicignano⁺, and José Alemán*

Abstract: The generation of sulfonyl radicals has long been known as a flexible strategy in a wide range of different sulfonylative transformations. Meanwhile their use in alkylation processes has been somehow limited due to their inherent difficulty in evolving to less-stable radicals after sulfur dioxide extrusion. Herein we report a convenient strategy that involves *gem*-difluorinated sulfinates as an “upgrading-mask”, allowing these precursors to decompose into their corresponding alkyl radicals. The electron-donor character of sulfinates in the formation of an electron donor-acceptor (EDA) complex with transient iminium ions is displayed, achieving the first example of a stereocontrolled light-driven insertion of *gem*-difluoro derivatives into unsaturated aldehydes. This methodology is compatible with flow conditions, maintaining identical levels of enantiocontrol.

Sulfonyl radicals have an illustrious history while speaking in the free-radicals context, since they work as a convergent point during the synthesis of a myriad of compounds, starting from diverse substrates.^[1] In this regard, the development of new chemical reactions has been accompanied by the refinement of classic protocols, resulting in more modern and minimalistic tools. Currently, visible light photocatalysis is part of the mighty artillery when aiming to generate sulfonyl radicals in a predictable manner, since the current pool of starting materials is vast.^[2] The latter widens the margin of design, giving the flexibility to invoke either a single-electron oxidation or reduction pathway.^[3] However, it is notorious when scrutinizing the literature that the introduction of the sulfonyl moiety is more frequently

encountered in comparison to the analogous adduct after loss of sulfur dioxide, despite the latent attractiveness that alkylation reactions represent (Scheme 1a). This might be addressed to what has been referred to, in seminal works as *α -scission of sulfonyl radicals: an unfavorable endothermic process*.^[1,4] Unsurprisingly, their application in the stereocontrolled crafting of C–C bonds is scarce, holding great promise in organic chemistry.

The aforementioned shortcomings took us to rationalize a plausible strategy to accomplish this task, the question thus becoming: how does one can favor sulfur dioxide extrusion? Letting the prerequisite for enantiocontrol aside, the idea of generating a long-lived alkyl radical and



Scheme 1. General considerations and blueprint (OC = Organocatalysts).

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introduce it into a suitable scaffold presented several challenges. For instance, it was crucial to decrease this endothermicity and a useful hint to our purpose was the generation of pseudo-stabilized radicals (Scheme 1b).

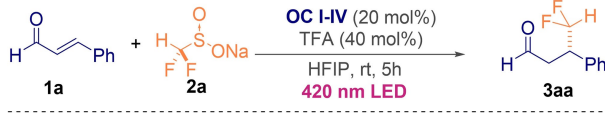
Conveniently, early investigations have shown that perfluoroalkyl radicals work under this logic.^[5] However, it was preferable tuning the entering scaffold in such mode that the radical would be stabilized enough but allowing us to modify it without being tightened to an imperative perfluorinated nature; since once installed, further functionalization is rare.^[6] We suspected then, that the *gem*-difluoro moiety was the perfect design line, not only because it might, in principle, facilitate the sulfur dioxide extrusion but because of its sharp attractiveness in the pharmaceutical arena as key tool in drug discovery (Scheme 1c).^[7] Scouting the current portfolio of sulfonyl radical precursors, we questioned the possibility of turning conventional alkyl sulfonates into their lightly fluorinated version, and under one electron oxidation conditions, serve as the right alkyl radical source. Herein, is described the completion of this goal with the β -difluoroalkylation of enals using photoredox organocatalysis as strategy (Scheme 1d).

Due to their easy-manipulation and high stability, sulfonates were idoneous in the design.^[8] Moreover, the fact that common difluoromethylations require ozone-depleting substances,^[9] propped up the plan. Pursuant to the idea of crafting attractive molecules, the preparation of upgraded carbonyl compounds came to us as principal motivation due to its inherent synthetic versatility. Hence, the studies started using *trans*-cinnamaldehyde **1a**, and sodium difluoromethane sulfinate **2a** as CF_2H radical source owing to its friendly manipulation conditions and commercial availability. After extensive testing an optimal scenario was found (Table 1, entry 1) (see Supporting Information for details).

Following these conditions, aldehyde **3aa** was isolated in 81 % yield and 86:14 enantiomeric ratio (e.r.), but several findings during this optimization process merit note. In the attempt to design the right catalytic system, conjugated addition of a preformed R-CF_2 radical to an olefin using external photocatalysts conducted to the recovery of starting materials. Therefore, the choice of activation mode was crucial, hinting on a key role of the organocatalyst (OC). In our case, its nature seemed not only to steer the space disposition of the reacting species (compare entries 1, 2, 3 and 4), but also suggesting photoredox properties. As previously described in the literature,^[10] well-tuned iminium ions might absorb the visible light and behave as photo-oxidants or due to its electron-poor character, trigger the formation of a photoactive EDA complex with a proper donor substrate. Replacing **2a** for DFMS-Zn **2a'** worked smoothly, without observing major changes (entry 5). Increasing or decreasing the reaction temperature did not conduct to any improvement (entries 6 and 7). Irradiation at 450 nm afforded the product in less than 10 % yield, and control experiments ensured that TFA, OC and light were necessary for a successful reaction (entries 9–11).

Still, it was uncertain if we would be able to extend this SO_2 extrusion to other fluorinated precursors. In this regard, we executed a comparative test in terms of radical reactivity

Table 1: Effect of reaction parameters.



OC I-IV (20 mol%), TFA (40 mol%), HFIP, rt, 5h, 420 nm LED

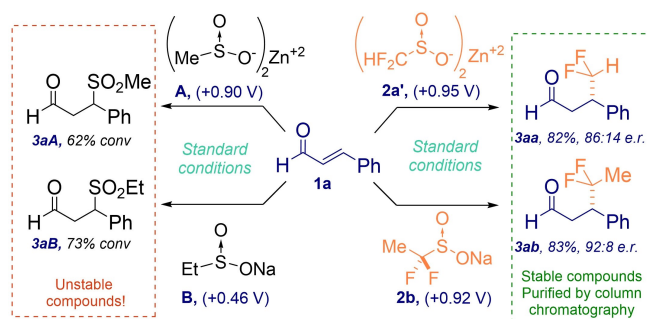
Ar = $\text{C}_6\text{H}_3(3,5-(\text{CF}_3)_2)$ Ar = $\text{C}_6\text{H}_3(3,5-(\text{CF}_3)_2)$ Ar = $\text{C}_6\text{H}_3(3,5-(\text{CF}_3)_2)$

Entry	Variation from "standard conditions"	Yield [%] ^[b] / [%] ^[c]	e.r. ^[d]
1 ^[a]	none	89/81	86:14
2	OC II instead of Cat I	92/80	77:23
3	OC III instead of Cat I	0	—
4	OC IV instead of Cat I	94/86	0
5 ^[e]	DFMS-Zn instead of 2a	91/82	86:14
6	0 °C instead of rt	71/62	85:15
7	45 °C instead of rt	81/69	75:25
8	450 nm instead of 420 nm	< 10	—
9	without TFA	0	—
10	without OC	0	—
11	dark conditions	0	—

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.1 mmol) in HFIP (500 μL). [b] Determined by ^1H NMR of unpurified reaction mixture using an I.S. [c] Isolated yield. [d] Determined by SFC analysis after derivatization with 2,4-dinitrophenylhydrazine. [e] DFMS-Zn was used in 0.05 mmol.

between DFMS **2a'**, sodium difluoroethane sulfinate (DFES) **2b** and their corresponding non-fluorinated analogues **A** and **B** (Scheme 2).

According to their oxidation potentials (**2a'**: +0.95 V; **2b**: +0.92 V; **A**: +0.90 V; **B**: +0.46 V vs SCE in DMF) measured by cyclic voltammetry (see Supporting Information), all of them could be easily oxidized by the excited iminium ion (+2.45 V vs. Ag/AgCl).^[10b] Thus, the four sulfonates were tested under the optimized conditions. As anticipated, **2a'** and **2b** conducted to the corresponding β -difluoroalkylated aldehydes in pleasing values of yield and e.r. Alternatively, the non-fluorinated **A** and **B** afforded the undesired sulfonyl adducts **3aA** and **3aB**.^[11] These findings allowed us to safely confirm that in our case, the fate of the sulfonyl radical was not dependent on the redox potentials

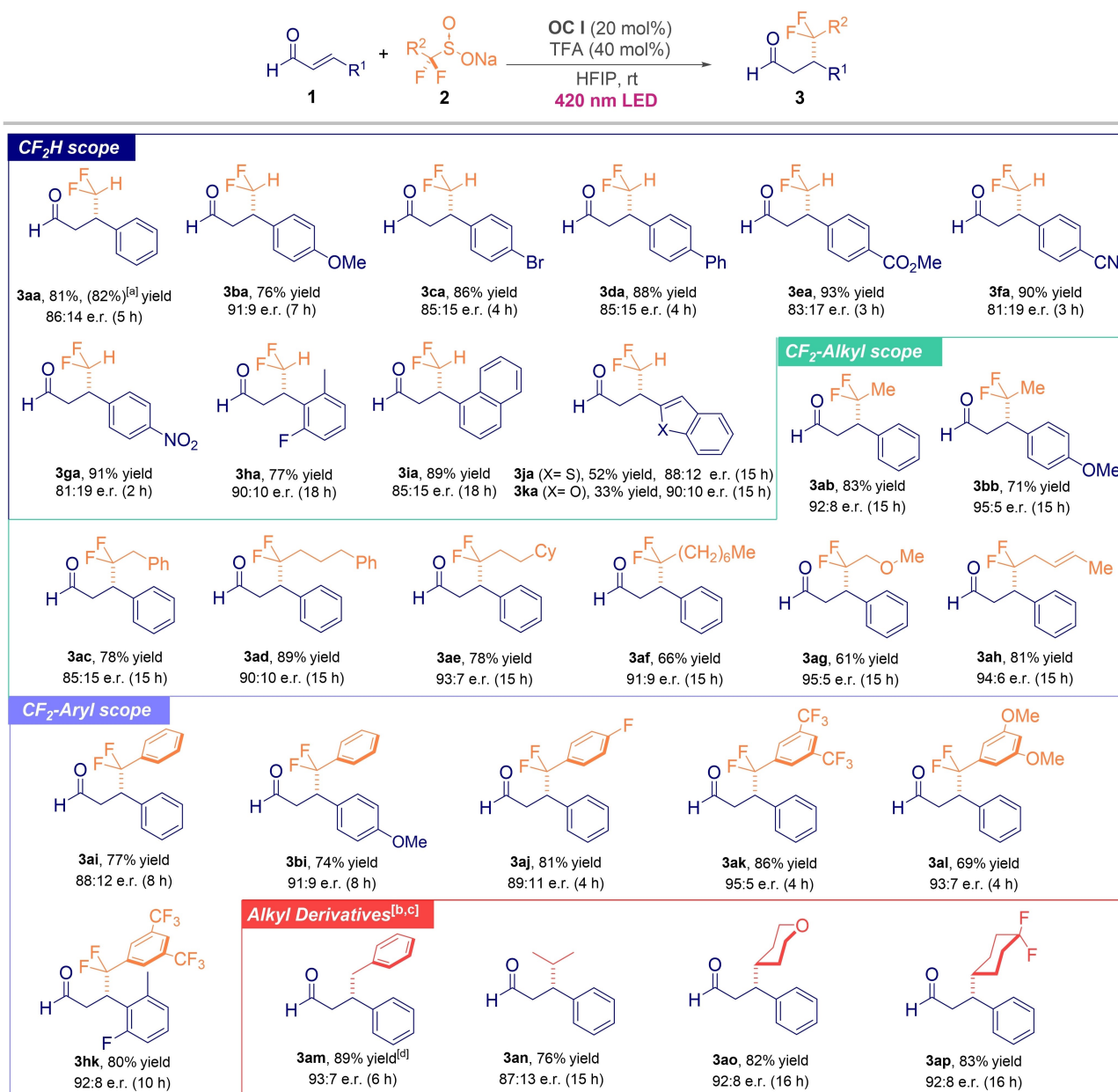


Scheme 2. Exploratory studies of sulfonates' nature.

but seemed like a consequence of isosterism by the $C(sp^3)-H^{\delta+} \rightarrow C(sp^3)-F^{\delta-}$ replacement.^[12]

With a clearer conceptual landscape, the generality of the protocol was tested (Scheme 3). *p*-Substituted aromatic rings proved well (**3ba–3ga**), pointing out that an electron-donating group (EDG) such as MeO- favored the enantiocontrol, whilst electron-withdrawing groups (EWGs) decreased it, compensated with high yields, however. Also, an *o,o'*-disubstituted ring conducted to the product (**3ha**) in good results, and testing a sterically demanding system such

as 1-naphtyl, led to the desired compound (**3ia**). Gladly, we found that benzo-fused heterocycles tolerated the reaction conditions as well (**3ja, 3ka**). Proceeding to the scope of CF_2 -alkyl motifs, **3ab** and **3bb** reinforced the previously observed influence of EDGs over the enantiocontrol. Linear chains adorned with a phenyl group (**3ac, 3ad**) and purely aliphatic ones (**3ae, 3af**), led to the corresponding products, allowing its isolation in good yield and enantioselectivity. Moreover, adducts bearing a heteroatom or a double bond (**3ag** and **3ah**) were suitable, highlighting that could work as



Scheme 3. Scope of the reaction. Reaction conditions: **1** (0.2 mmol), **2** (0.1 mmol), **OC I** (20 mol%), TFA (40 mol%), HFIP ($c=0.2$ M), rt unless otherwise noted. Isolated yields shown. Enantiomeric ratio was measured by SFC analysis after derivatization with 2,4-dinitrophenylhydrazine.

[a] Carried out with DFMS-Zn. [b] Performed using the corresponding non *gem*-difluorinated alkyl sulfonates. [c] Executed at 50 °C and $c=0.1$ M.

[d] Carried out using the zinc sulfinate.

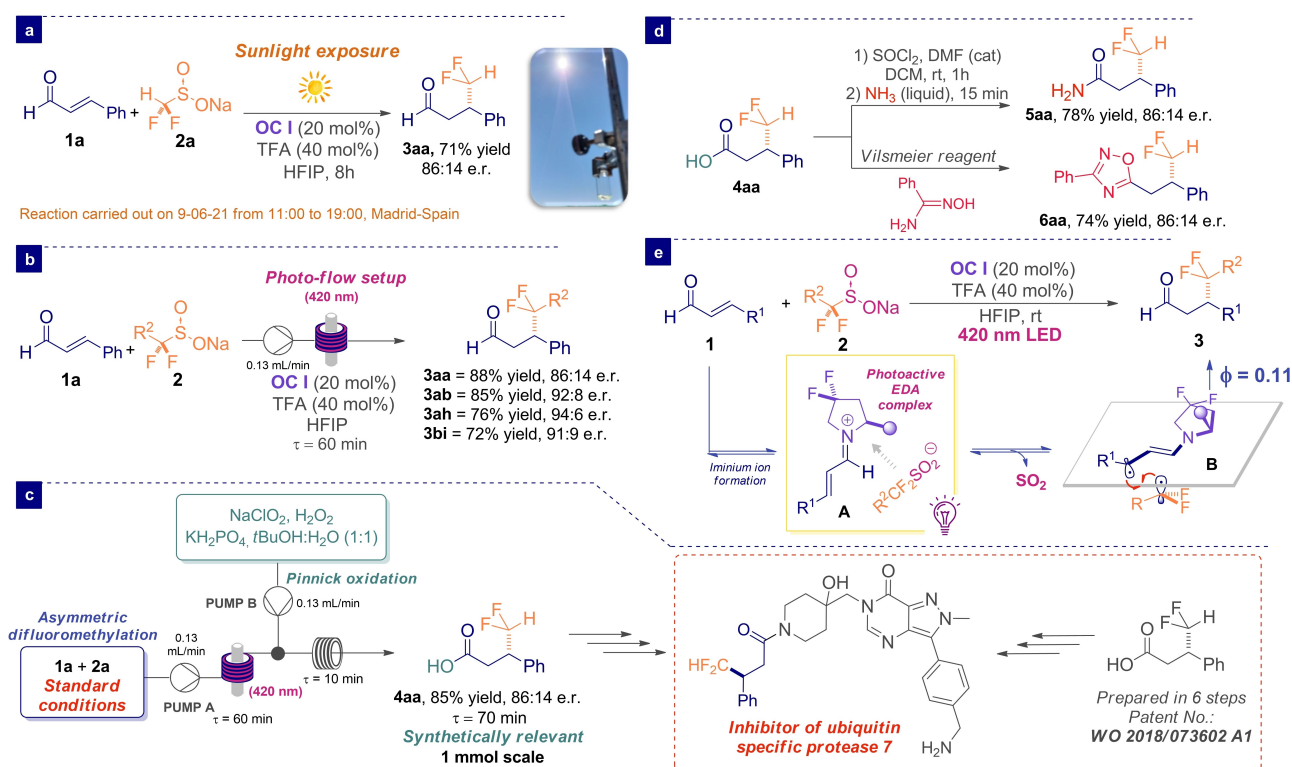
flexible candidates for further synthetic modification. Later on, a series of *gem*-difluoro benzyl substituents were examined. Once again, the PMP group conducted to a pleasing higher e.r. (**3ai** vs **3bi**). Sulfinates bearing *para*- and *meta*-substituted aromatic rings worked properly, regardless its electronic nature (**3aj**, **3ak** and **3al**). Furthermore, mixing an *o,o'*-disubstituted aldehyde with a *m,m'*-disubstituted sulfinate gleaned the interesting product **3hk**.

Lastly, we selected some alkyl sulfinates that are frequently used in late-stage-functionalization but requiring harsh conditions.^[13] Conveniently, products were obtained in satisfactory results and noteworthy, the unique primary substrate that succeeded was the one which SO₂ extrusion could be leveraged by generating a more stabilized radical (**3am**). Besides, secondary substrates (**3an**, **3ao**, **3ap**) worked smoothly under the reaction scenario (see Supporting Information for details and limitations). These latter compounds (**3am**–**3ao**) helped us to reveal the configuration of the new chiral center by optical activity correlation with the same products reported in the literature (see Supporting Information). Since the conditions for the preparation of the whole series implied the same OC and mechanism is assumed to be the same, the configuration was extended for the rest of the products.

To demonstrate the operational simplicity of the protocol, the reaction was successfully performed under natural sunlight irradiation within 8 h (Scheme 4a). Additionally, the synthetic method proved compatible using a commercial

flow-photoreactor (Scheme 4b, see Supporting Information). Applying the optimized configuration, four representative aldehydes were synthesized with comparable efficiency to the batch conditions (72–88 % yield, 91:9 to 96:4 e.r.) in a clear shortened timeframe. This latter provides a reaction throughput up to ≈ 30 times higher than conventional batch setup. Encouraged by this, we sought to point out the synthetic potential of the protocol towards the preparation of bioactive ingredients. In this sense, the carboxylic acid **4aa** was obtained in 85 % yield at 1 mmol scale under continuous flow conditions, requiring the use of an additional pump (Scheme 4c). The importance of this achievement lies in two aspects: 1) molecule's key role during the synthesis of a wide family of assets tested to selectively inhibit USP7^[14] and 2) a reduction from six to one step for its preparation. The synthetic flexibility of **4aa** was showcased by preparing amide **5aa** and 1,2,4-oxadiazole **6aa** (Scheme 4d), an attractive heterocycle because of its demand in drug discovery.^[15]

Quantum yield of the reaction was determined ($\phi = 0.11$) which is in accordance with a closed catalytic cycle (see Supporting Information for details). In the light of the experimental data obtained herein and drawing knowledge from previous reports,^[10a–c] a plausible mechanism for the witnessed asymmetric transformation is suggested (Scheme 4e). Upon acidic condensation of aldehyde **1** with OC **I**, a transient iminium ion **A** is produced, which could bear the sulfinate as counteranion in an intimate ion-pair system. A



Scheme 4. Flexibility of the procedure, derivatization, and plausible catalytic cycle.

series of optical absorption spectra of this colored solution were recorded (see Supporting Information) and the appearance of a red-shifted new absorption band suggested a photoactive EDA complex between the sulfinate (donor)^[16] and the iminium ion (acceptor). The latter intermediate reaches its excited state by light irradiation unleashing a SET process, which simultaneously gives rise to the corresponding sulfonyl radical and β -enaminy radical **B**. Thereafter, the sulfonyl radical evolves to the corresponding *gem*-difluoro radical by means of SO₂ release, and through a radical recombination occurring at the less hindered face of the system, the new C–C bond is formed. Closing the catalytic cycle, compound **3** is rendered along with the recovery of the OC

In summary, the first visible-light driven enantioselective *gem*-difluoroalkylation of α,β -unsaturated aldehydes has been described. The performed studies suggest a double advantage of the CF₂ group, since it facilitates a typical unfavorable process and upgrades the properties of the final products. Additionally, the preparation of attractive bioactive ingredients was showcase, as well as the flexibility of flow photochemistry, a currently sought task by the pharmaceutical sector.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: Asymmetric Catalysis • Fluorination • Photocatalysis • Visible Light

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