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Rongalite-mediated sequential homologative fluorination of oxindoles en route to 3-(fluoromethyl)-3-methylindolin-2-ones

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A rongalite-mediated, transition-metal-free reductive homologative-fluorination strategy has been developed for the synthesis of 3-(fluoromethyl)-3-methylindolin-2-ones. This methodology involves the monofluoromethylation of indolin-2-one derivatives, employing rongalite as a C1-homologating reagent, and diethylaminosulfur trifluoride (DAST) as the fluorine source. The reaction proceeds under mild conditions, offering excellent yields, high selectivity, and broad functional group tolerance on oxindoles. Overall, this approach provides a practical and sustainable route for CH_2F incorporation, thereby expanding the scope of fluoroalkylation chemistry and opening new avenues for the synthesis of fluorinated bioactive molecules.

Introduction

Fluorinated organic compounds have increasingly permeated diverse scientific disciplines, especially pharmaceuticals, agrochemicals, and materials science.[1] Significantly, approximately 20% of marketed pharmaceuticals are fluorine-bearing, and in the agrochemical domain, the contribution of organofluorine compounds has exceeded 50% over recent decades.[2] The distinctive physicochemical characteristics of fluorine-particularly its high electronegativity and the robustness of the carbon–fluorine (C–F) bond exert a dominant influence on the behaviour of fluorine-bearing molecules.[3] These traits are strategically exploited in the design of molecular scaffolds of value, thereby attracting broad interest across pharmacology,[4] materials development,[5] and agricultural chemistry[6] due to their improved physicochemical properties relative to non-fluorinated analogues.

Incorporation of a fluorine atom often enhances the biological activity,[7] lipophilicity,[8] and binding selectivity[9] of lead molecules (Fig. 1). From a synthetic chemistry perspective, the construction of fluorinated molecular frameworks remains a significant challenge,[10] highlighting the importance of organofluorine chemistry as a vital, and interdisciplinary area of research.

The development of novel methods for incorporating fluorine atoms has attracted extensive interest. In particular, difluoromethyl- and trifluoromethylarenes have been widely explored by synthetic chemists,[11] resulting in a multitude of available synthetic approaches.

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Within this context, monofluoromethyl ($-CH_2F$) is of particular value by mimicking methyl (CH_3) or hydroxymethyl (CH_2OH) groups, which are frequently present in biologically active compounds.[12] These units enable retention of structural features while imparting the "fluorine effect". Their application spans pharmaceuticals,[13] agrochemicals,[14] and material science,[15] where they can contribute to enhanced molecular stability, bioactivity, and selectivity.

In contrast, despite the proven significance of the monofluoromethyl (CH_2F) group, synthetic methodologies for its incorporation remain limited. Most reported approaches rely on: (i) transition-metal-catalyzed reactions employing fluoro(-halo)methanes (*e.g.*, ICH_2F or $BrCH_2F$) as monofluoromethyl sources (Scheme 1a);[16] (ii) carbenoid-mediated nucleophilic fluoromethylation under strongly basic conditions (Scheme 1b);[17] and, (iii) fluorobis(phenylsulfonyl)methane (FBSM)-based monofluoromethylation strategies (Scheme 1c).[18]

Most established monofluoromethylation protocols utilize reagents such as fluoromethyllithium ($LiCH_2F$) or fluoroiodomethyllithium (LiCHIF), typically in the presence of metals or strong bases.

These transformations generally require prior preparation of fluoromethyl precursors, and involve multi-step homologation sequences. Although effective, they are often limited by harsh conditions, costly reagents, and operational complexity. Notably, a one-pot C1 homologation/fluorination protocol for the direct introduction of a monofluoromethyl group has not yet been reported.

On the other hand, oxindole skeleton represents a privileged heterocyclic framework that is widely distributed in natural products, pharmaceuticals, and bioactive compounds, attracting sustained interest in both synthetic, and medicinal

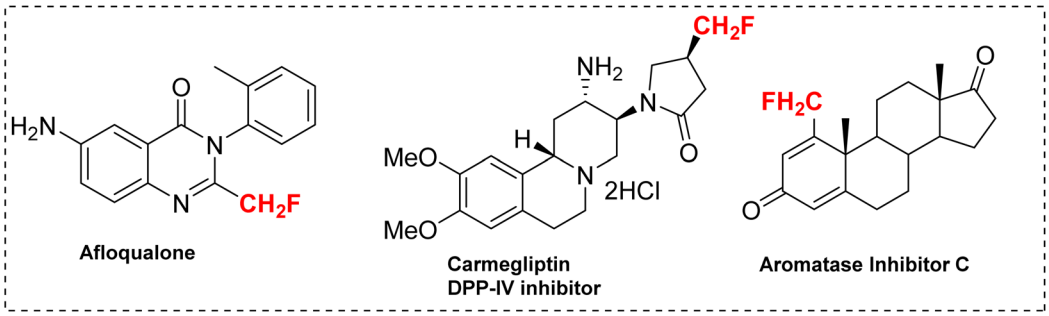
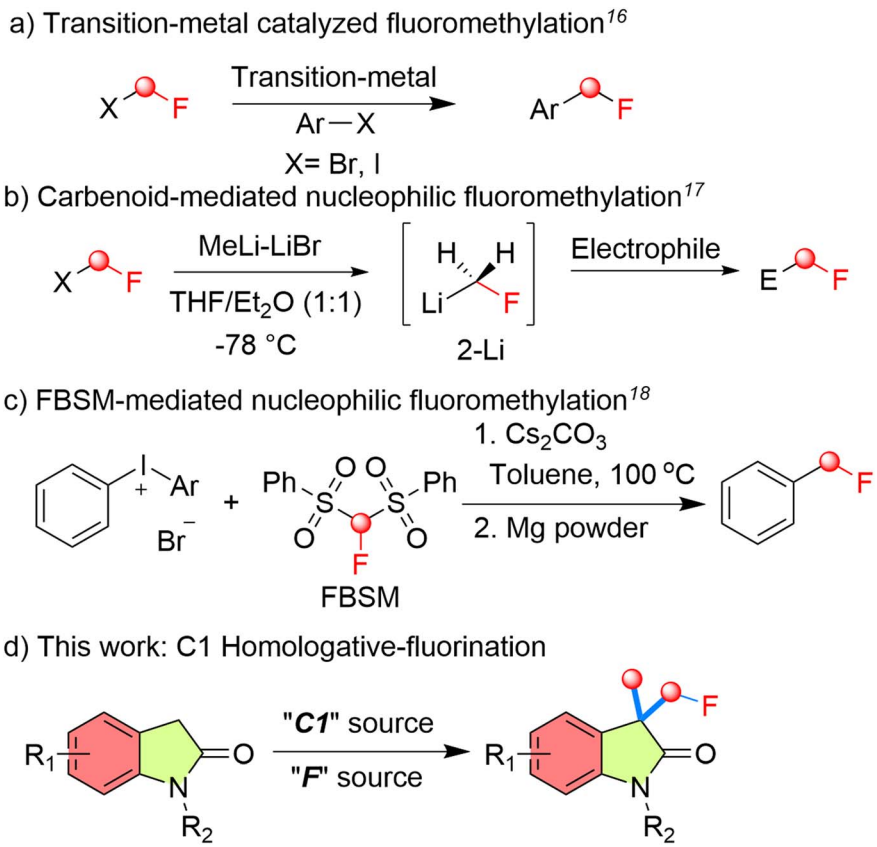


Fig. 1 Monofluoromethyl-containing bioactive scaffolds.



Scheme 1 Strategies for the synthesis of monofluoromethyl compounds.

chemistry.[19] Among these, 3,3-disubstituted oxindoles are of particular significance owing to their rigid structures, extended π-systems, and nitrogen-rich environments.[20] These features render them suitable pharmacophores exhibiting diverse biological activities such as spermicidal,[21] antimicrobial,[22] enzyme inhibitory,[23] and anticancer properties.[24] Further, 3-hydroxymethyl-3-methyl-2-oxindoles serve as key intermediates in the total synthesis of (–)-physostigmine and (–)-esermethole,[25] and their biological activity can be enhanced by fluorine incorporation.

Continuing our studies on the synthetic versatility of rongalite,[26] a multifaceted reagent functioning as a reducing agent,[27] a single-electron donor,[28] and a dual C1-unit donor,[29] we now report a straightforward rongalite-mediated monofluoromethylation of indolin-2-one. In this transformation, rongalite serves as the C1-unit donor and, in the presence of an external fluorine source, provides an efficient route to fluorinated oxindole derivatives (Scheme 1d).

Results and discussion

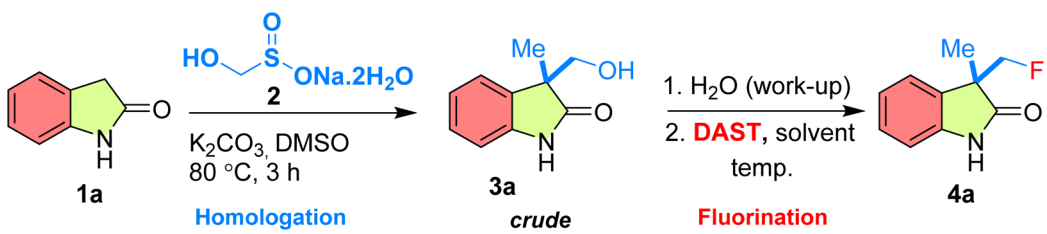
In our preliminary investigations, we aimed to develop a streamlined and efficient monofluoromethylation strategy for the synthesis of 3-(fluoromethyl)-3-methylindolin-2-one **4a** from indolin-2-one **1a**, Rongalite **2**, and DAST as model substrates. The study commenced with the C1-homologation of **1a** (1.0 mmol) using Rongalite **2** (3.0 mmol), and K_2CO_3 (2.5 mmol) in DMSO (2 mL) at 80 °C, affording the hydroxymethyl crude **3a**.

After completing the C1 homologation, the reaction mixture **3a** was washed with water and extracted with Et_2O, evaporated and the crude **3a** was directly subjected to fluorination in CH_2Cl_2 without purification, providing the desired product **4a** in 22% overall yield (Table 1, entry 1). Encouraged by this initial result, we proceeded to optimize the DAST stoichiometry and reaction temperature. Lowering the temperature to 0 °C improved the yield to 30% after 45 minutes (Table 1, entry 2). Further increasing the amount of DAST from 1.0 mmol to 2.0 mmol enhanced the yield to 46% in 30 minutes (Table 1, entry 3), while 3.0 mmol of DAST afforded a significantly higher yield of 60% within 20 minutes (Table 1, entry 4). Remarkably, using 4.0 mmol of DAST under the same conditions delivered **4a** in 85% yield within only 5 minutes (Table 1, entry 5), demonstrating that an excess of DAST effectively drives the fluorination to completion. To further understand the effect of temperature, reactions using 4.0 mmol DAST were carried out under varied conditions. At room temperature, **4a** was obtained in 60% yield in 15 minutes (Table 1 entry 6), whereas at −10 °C, the yield slightly decreased to 80% in 5 minutes (Table 1, entry 7). These observations revealed that 0 °C offered the optimal balance between reactivity and selectivity, providing the highest yield in the shortest time. Next, the solvent effect was examined (Table 1, entries 8–11). When CH_2Cl_2 was replaced by THF, $CHCl_3$, CH_3CN, and 1,2-dichloroethane (DCE), the yields decreased to 65%, 55%, 48%, and 64%, respectively. Notably, performing the fluorination in DMSO resulted in no product formation, even after 24 hours (Table 1, entry 12). It is worth mentioning that the other fluorinating agents such as PyFluor and AlkylFluor are failed to give desired product **4a** (see SI, Table S1). In summary, the optimization studies established that treating **3a** (1.0 mmol) with DAST (4.0 mmol) in CH_2Cl_2 at 0 °C constitutes the most effective set of conditions, affording 3-(fluoromethyl)-3-methylindolin-2-one **4a** in 85% yield within 5 minutes (Table 1, entry 5). These mild, and efficient reaction parameters were subsequently adopted for the substrate-scope investigations.

The fluorination behaviour of various 3-(hydroxymethyl)-3-methylindolin-2-one derivatives using DAST was systematically investigated (Table 2). The method displayed broad substrate tolerance: electron-donating substituents (methyl, dimethyl, methoxy) on the aromatic ring afforded fluoromethyl derivatives **4b–4d** in good yields (72–75%). These substituents likely enhance electron density on the indolinone ring, stabilizing the transition state in fluorination. Similarly, substrates bearing electron-withdrawing halogens (F, Cl, Br, I) at various positions tolerated the conditions well, giving products **4e–4l** in yields of 75–84%. Moreover, an $-OCF_3$ substituent was well accommodated, affording **4m** in 80% yield. These results demonstrate a versatile and efficient fluorination protocol for the preparation of fluoromethyl-substituted indolin-2-ones under mild conditions. Notably, *N*-alkylated, and *N*-aryl-substituted derivatives demonstrated remarkably enhanced reactivity compared to their N–H counterparts. Substrates with *N*-methyl, *N*-ethyl, *N*-butyl, and *N*-4-ethylphenyl groups afforded products **4n–4q** in excellent yields 91–93% (Table 2) within a very short reaction time. Likewise, *N*-substituted cyclopentyl, allyl, propargyl, and benzyl afforded the desired fluorinated products **4r–4u** in good to excellent yields 76–89% (Table 2).

Further, the substitutions on the benzene ring, and the nitrogen atom enhanced the formation of products **4v–4x** in

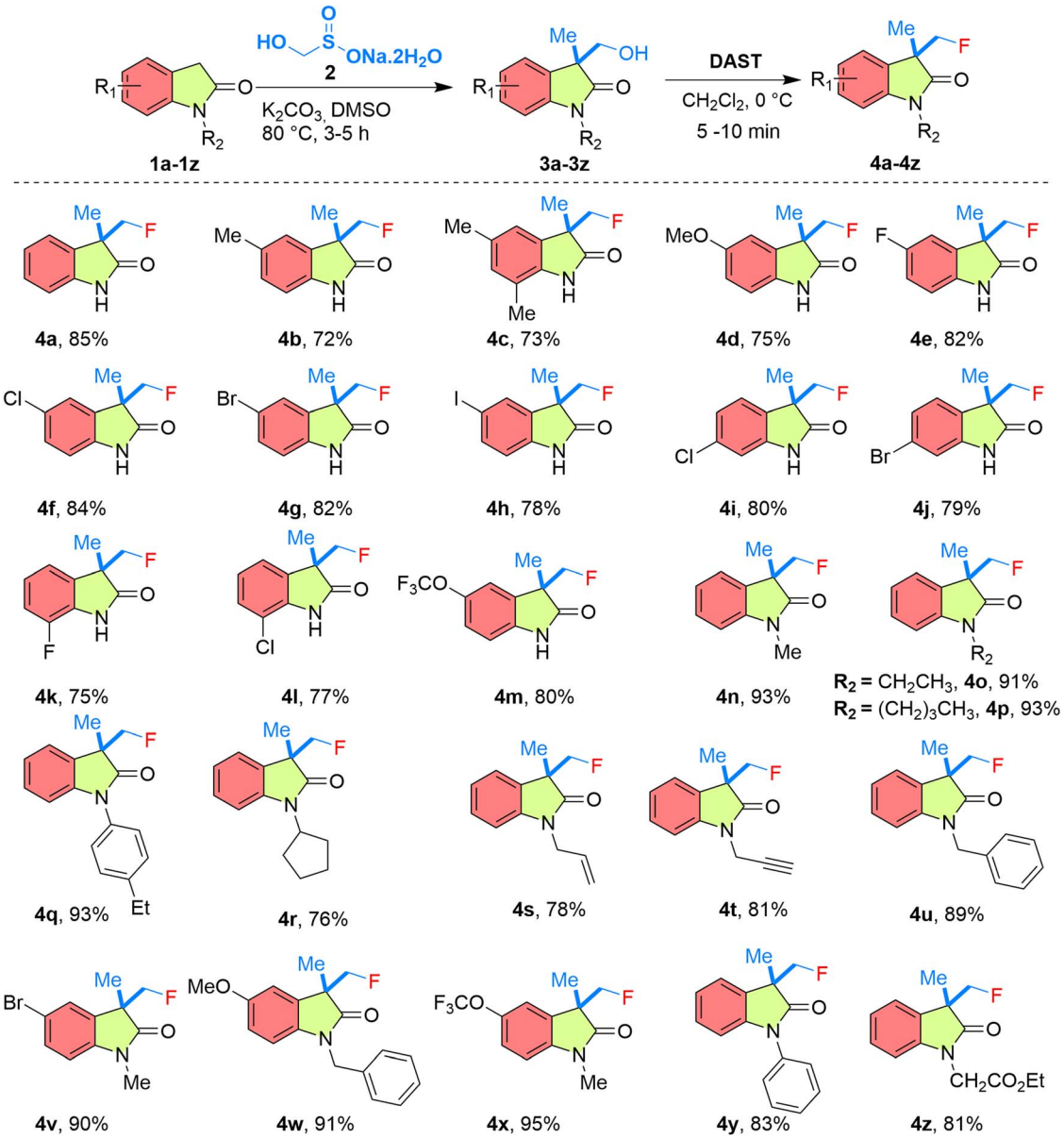
Table 1 Optimization of the reaction conditions[a]



Entry	DAST (mmol)	Solvent	Temp. (°C)	Time (min)	Yield[b] (%)
1	1.0	CH_2Cl_2	rt	1 h	22
2	1.0	CH_2Cl_2	0	45	30
3	2.0	CH_2Cl_2	0	30	46
4	3.0	CH_2Cl_2	0	20	60
5	**4.0**	**CH_2Cl_2**	**0**	**5**	**85**
6	4.0	CH_2Cl_2	rt	15	60
7	4.0	CH_2Cl_2	−10	5	80
8	4.0	THF	0	30	65
9	4.0	$CHCl_3$	0	20	55
10	4.0	CH_3CN	0	30	48
11	4.0	$C_2H_4Cl_2$	0	15	64
12	4.0	DMSO	0	24 h	n.r[c]

[a] Reaction conditions: indolin-2-one **1a** (1 mmol), rongalite **2** (3 mmol) and K_2CO_3 (2.5 mmol) in 2 mL of DMSO at 80 °C, after completing the C1 homologation, the reaction mixture **3a** was washed with water and extracted with Et_2O, evaporated, re-dissolved in CH_2Cl_2 (2 mL), and added DAST. [b] Isolated yield from homologation-fluorination sequence. [c] n.r. = no reaction, rt = room temperature.

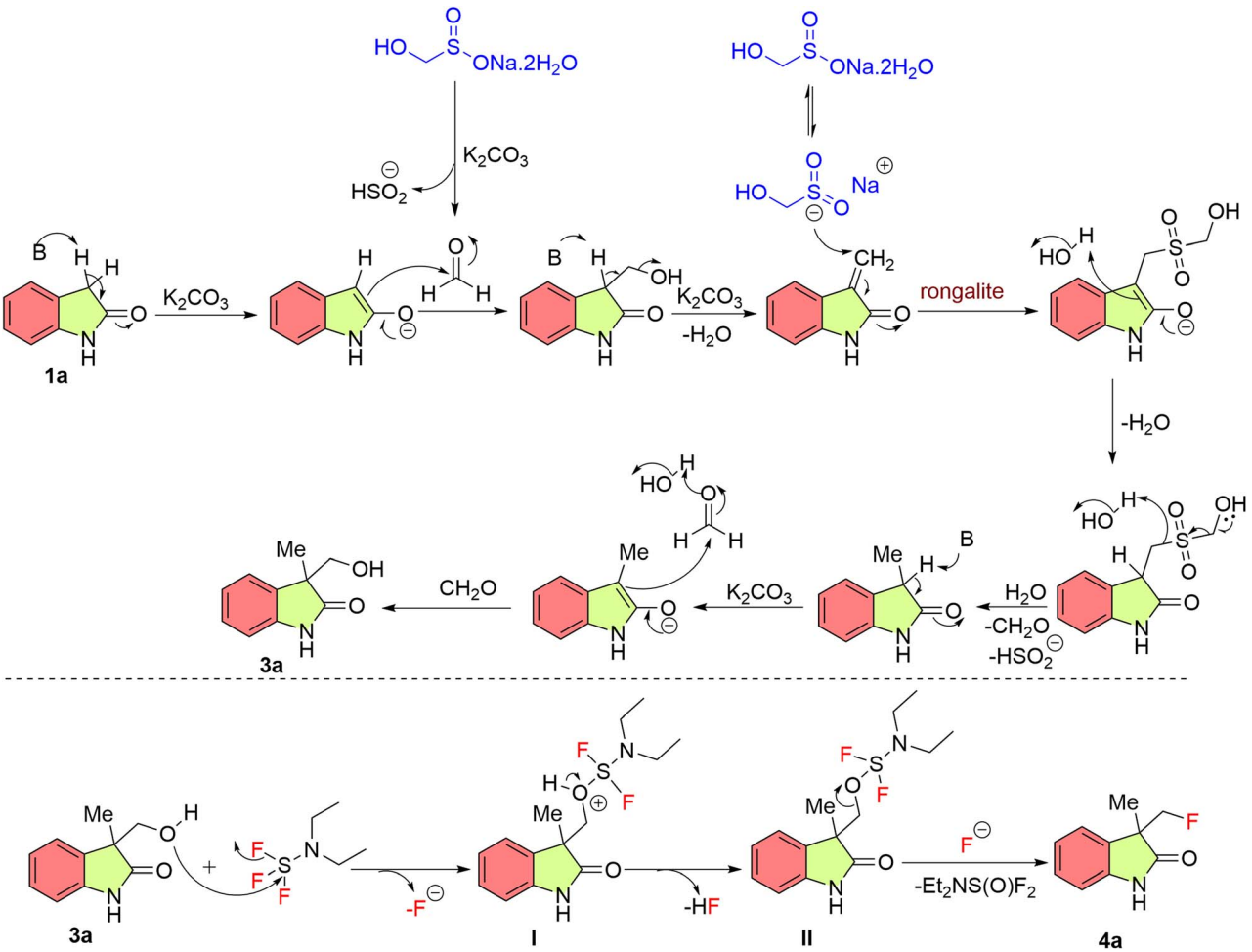
Table 2 Substrate scope[a,b]



[a] Reaction conditions: indolin-2-one **1** (1 mmol), rongalite **2** (3 mmol) and K_2CO_3 (2.5 mmol) in 2 mL of DMSO at 80 °C, after completing the C1 homologation, the reaction mixture **3a** was washed with water, and extracted with Et_2O, evaporated, re-dissolved in CH_2Cl_2 (2 mL), and added DAST (4 mmol). [b] Isolated yield from homologation-fluorination sequence.

excellent yields 90–95% (Table 2), confirming the high efficiency of the reaction. Further, *N*-phenyl 3-(hydroxymethyl)-3-methylindolin-2-one also actively participated in the reaction giving the product **4y** in excellent yield 83% (Table 2). The *N*-substituted ethyl acetate also participated in the reaction forming the product **4z** in good yield 81% (Table 2). Overall, both electron-donating, and electron-withdrawing groups on the indolinone ring were well tolerated, but *N*-alkylation, and *N*-arylation significantly improved the reaction efficiency, leading to excellent yields within a short time frame.

Based on the literature reports[27,30] a plausible mechanism is outlined in Scheme 2. Initially, indolin-2-one **1a** reacts with *in situ* generated formaldehyde from rongalite **2** to form 3-(hydroxymethyl)-3-methylindolin-2-one **3a** *via* aldol condensation reaction followed by a reductive aldol reaction.[27] Which further undergoes nucleophilic attack of oxygen atom from alcohol **3a** to the electrophilic sulfur atom of diethylaminosulfur trifluoride would form an alkoxy-sulfur intermediate **I**, subsequent deprotonation facilitates the generation of a neutral intermediate **II**, which then undergoes an intramolecular displacement by the liberated fluoride ion on intermediate, yielding 3-(fluoromethyl)-3-methylindolin-2-one **4a** with loss of the sulfur-containing leaving group.



Scheme 2 Plausible reaction mechanism.

Conclusions

We have developed an efficient monofluoromethylation by sequential hydroxymethylation, and monofluorination strategy, which involves a classical aldol condensation reaction followed by a reductive aldol reaction using rongalite. In this method, rongalite is an industrial product with low cost (1 g, 0.03$), which plays a vital role as a hydride-free reducing agent, and double C1 unit donor. This transition metal, and hydride-free reductive aldol protocol allows rapid access to 3-(hydroxymethyl)-3-methylindolin-2-ones, and DAST (diethylaminosulfur trifluoride) is commercially available, easy to handle, and highly selective for hydroxyl groups, making it ideal for deoxyfluorination of alcohols. It can also work under mild conditions, preserving sensitive functional groups in the molecule. 3-(fluoromethyl)-3-methylindolin-2-one is an organofluorine compound that belongs to the class of fluorinated indolinones, a group of molecules with significant importance in medicinal, and pharmaceutical chemistry.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: experimental procedures, characterization data, and copies of the ^{1}H, $^{13}C\{^{1}H\}$, ^{19}F NMR and HRMS spectra of all compounds are included. See DOI: https://doi.org/10.1039/d5ra08987e.

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