Fluorinated organic compounds have attracted great attention in the field of medicinal chemistry owing to their unique physical and chemical properties, bioavailability, lipophilicity, and metabolic stability.1–4 This is especially true in the pharmaceuticals and agrochemicals, where fluorine is often considered a bioisostere of hydrogen. At present, about 30% of all agrochemicals (such as pyroxsulam and fluxapyroxad and so on) and 20% of all pharmaceuticals (such as 5-fluorouracil and norfloxacin and so on) incorporate at least one fluorine atom.5 As such, many efforts have been put into the development of fluorinated molecules based on the C–F bond formation in the past decade.6–10

Chromones and their derivatives are found in numerous natural products and pharmaceuticals11–13 that show many biological activities, such as monoamine oxidase inhibitors and antitubercular activity.14–18 They also exist widely in pigments, dyes, and essential nutrients for the human body.19–22 Consequently, the development of effective methods to construct these heterocyclic scaffolds, especially C3-functionalized analogs, has been studied extensively.23–31

Furthermore, enaminoles have been recognized as a class of available and powerful synthetic building blocks owing to their versatile reactivity in a variety of organic transformations,32–38 which were widely used in syntheses of many heterocyclic39–47 and fused heterocyclic compounds.48–56 Recently, many efforts have been devoted to develop new methods for the synthesis of 3-functionalized chromones from o-hydroxyarylenaminones via the electrophile-triggered cyclization reaction (Scheme 1(a)).29,30,57–66 As far as the reactions of enaminoles with fluorination reagents are concerned, a difluorination of enaminoles to access difluorinated carbonyl compounds by virtue of Selectfluor (1-chloromethyl-4-fluoro-1,4-diazoniabicyclo[2.2.2]octane bis(tetrafluoroborate), cheap and easily available) as the fluorine source was reported by Shreeve’s group in 2005.67 In 1996, Bolós’s group68 synthesized 3-fluorochromones from o-hydroxyarylenaminones by using 1-fluoro-2,4,6-trimethylpyridinium triflate (NFTP) as the fluorine source, but it still suffers from more expensive fluorination reagent and substrate scope. Therefore, the development of an efficient method using Selectfluor as a fluorine source would be valuable. In 2017, the groups of Zhao69 and Xu70 developed a Selectfluor-triggered tandem cyclization of o-hydroxyarylenaminones to obtain 3,3-difluorinated 2-amino-substituted

Selectfluor-triggered fluorination/cyclization of o-hydroxyarylenaminones: A facile access to 3-fluoro-chromones

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Abstract
A fast and efficient Selectfluor-triggered fluorination/cyclization reaction of o-hydroxyarylenaminones has been successfully developed. The reaction successfully provides an expedient method for the synthesis of 3-fluoro-chromones promoted by potassium carbonate, which shows readily available starting materials and is easy to operate. In addition, a plausible mechanism of this tandem cyclization reaction was proposed where 4H-chromen-4-one, 2-(dimethylamino)-3,3-difluorochroman-4-one, and 3,3-difluoro-2-hydroxychroman-4-one were not found to be the reactive intermediates. Moreover, these novel compounds have been obtained in moderate to good yields, and their structures have been confirmed by 1H NMR, 13C NMR, and high-resolution mass spectrometry.

Keywords
3-fluoro-chromones, Selectfluor, o-hydroxyarylenaminones, fluorination/cyclization

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In moderate yields (35%–60%). Furthermore, \( \alpha \)-hydroxyarylenaminones \( 1n-1r \) with multisubstituents were compatible with the transformation as well, leading to the corresponding products \( 3n-3r \) in 42–48 yields.

To gain insight into the mechanistic details of this fluorination/cyclization reaction, several control experiments were performed (Scheme 2). First, when the reaction of 4\( H \)-chromen-4-one \( 4 \) with Selectfluor \( 2 \) was performed under standard conditions, the expected product \( 3a \) was not observed at all (Scheme 2(a)).

Based on the above observations and previous reports, a plausible mechanism for the synthesis of 3-fluoro-chromones 3 was shown in Scheme 3. Initially, \( \alpha \)-hydroxyarylenaminone formed species 7 via imine–enamine tautomerization, which rapidly quenched with Selectfluor to deliver species 8. Then, the intermediate 9 could be generated through keto–enol tautomerization, which sequentially produced the cyclic species 10 and released a molecule of HBF₄. The subsequent deamination ultimately generated the desired 3-fluoro-chromone 3a.
Conclusion

In conclusion, we successfully developed a facile and general approach accessing a range of 3-fluoro-chromones from \( o \)-hydroxyarylenaminones. Importantly, several species, such as 4\( H \)-chromen-4-ones; 2-(dimethylamino)-3,3-difluorochroman-4-ones; and 3,3-difluoro-2-hydroxychroman-4-ones, are proved not to be the reactive intermediates during the reaction process. This transformation involves the Selectfluor-triggered fluorination of the enaminone moiety, followed by subsequent intramolecular cyclization and deamination.

Experimental

All compounds were fully characterized by spectroscopic data. The nuclear magnetic resonance (NMR) spectra were recorded on a DRX600 spectrometer (1H: 600MHz, 13C: 150MHz), chemical shifts (\( \delta \)) are expressed in ppm, and J values are given in Hz, and deuterated CDCl\(_3\) was used as solvent. The reactions were monitored by thin-layer chromatography (TLC) using silica gel GF254. The melting points were determined on XT-4A melting point apparatus and are uncorrected. High-resolution mass spectrometry (HRMS) was performed on an Agilent liquid chromatography–mass spectrometry (LC/MS) time-of-flight (TOF) instrument.

Compounds 1a–1r were prepared according to the literature. Selectfluor was purchased from Adams-beta and Aldrich Corporation Limited.

General procedure for the synthesis of 3-fluoro-chromones 3a–3r

0.5-mmol-substituted \( o \)-hydroxyarylenaminones 1, 1.1-mmol Selectfluor 2 (389.4mg), 0.5-mmol K\(_2\)CO\(_3\) (69mg), and 2.0-mL MeCN were charged into a 10-mL Ace Glass pressure tube, and the mixture was stirred at 100 °C for 0.5 h until 1 was completely consumed. The mixture was

| Entry | \( R \) | Yield (%) | Entry | \( R \) | Yield (%) |
|-------|--------|-----------|-------|--------|-----------|
| 1     | 6-H (1a) | 60        | 10    | 7-F (1j) | 49        |
| 2     | 6-OMe (1b) | 53        | 11    | 7-Cl (1k) | 49        |
| 3     | 6-Me (1c) | 57        | 12    | 7-Br (1I) | 38        |
| 4     | 6-F (1d) | 40        | 13    | 8-F (1m) | 35        |
| 5     | 6-Cl (1e) | 45        | 14    | 6-Me, 8-Me (1n) | 48        |
| 6     | 6-Br (1f) | 42        | 15    | 6-Cl, 8-Me (1o) | 46        |
| 7     | 6-NO\(_2\) (1g) | 45        | 16    | 6-Me, 8-NO\(_2\) (1p) | 44        |
| 8     | 7-OMe (1h) | 51        | 17    | 6-Cl, 8-Cl (1q) | 45        |
| 9     | 7-Me (1i) | 53        | 18    | 6-Cl, 8-Br (1r) | 42        |

\( ^{a}\)Reaction conditions: 1 (0.5 mmol), 2 (1.1 mmol), and K\(_2\)CO\(_3\) (1.0 equiv.) in 2 mL of MeCN, stirred at 100 °C for 0.5 h.

\( ^{b}\)Isolated yields.
cooled to room temperature. Quenched with water (15 mL), and then EtOAc (15 mL x 3) was added. The organic phase was washed with water (10 mL), dried over Na₂SO₄, concentrated, and purified by flash column chromatography to afford 3-fluoro-chromenes 3.

3-Fluoro-4H-chromen-4-one (3a): Yield: 60%; white solid; m.p. 163–165°C; 1H NMR (600 MHz, CDCl₃); δ 8.32 (dd, J = 8.0, 1.3 Hz, 1H, C=CH), 8.18 (d, J = 3.4 Hz, 1H, ArH), 7.75–7.73 (m, 1H, ArH), 7.54 (d, J = 8.5 Hz, 1H, ArH), 7.47 (t, J = 7.6 Hz, 1H, ArH); 13C NMR (150 MHz, CDCl₃); δ 170.6 (d, J = 15.5 Hz, C=O), 154.9 (C9), 149.4 (d, J = 249.2 Hz, C2), 142.9 (d, J = 40.0 Hz, C1), 134.1 (C7), 126.0 (d, J = 3.4 Hz, C5), 125.3 (C6), 124.8 (d, J = 7.7 Hz, C4), 118.4 (C8); HRMS (TOF ES⁺): m/z calcd for C₁₁H₉FO₂⁺ [(M + H)⁺], 165.0346; found, 165.0348.

3-Fluoro-6-methoxy-4H-chromen-4-one (3b): Yield: 53%; white solid; m.p. 174–176°C; 1H NMR (600 MHz, CDCl₃); δ 8.03 (d, J = 2.6 Hz, 1H, C=CH), 7.60 (t, J = 8.4 Hz, 1H, ArH), 7.06 (dd, J = 8.5, 0.7 Hz, 1H, ArH), 6.84 (d, J = 8.3 Hz, 1H, ArH), 4.00 (s, 3H, ArOCH₃); 13C NMR (150 MHz, CDCl₃); δ 170.3 (d, J = 16.5 Hz, C=O), 160.4 (d, J = 2.8 Hz, C6), 157.9 (C9), 149.6 (d, J = 245.7 Hz, C2), 140.7 (d, J = 40.5 Hz, C1), 138.5 (C4), 134.3 (C5), 110.3 (C7), 106.2 (C8), 56.5 (OCH₃). HRMS (TOF ES⁺): m/z calcd for C₁₃H₁₂FO₂⁺ [(M + H)⁺], 195.0452; found, 195.0452.

3-Fluoro-6-nitro-4H-chromen-4-one (3c): Yield: 42%; yellow solid; m.p. 179–181°C; 1H NMR (600 MHz, CDCl₃); δ 8.45 (d, J = 2.4 Hz, 1H, ArH), 8.19 (d, J = 3.3 Hz, 1H, C=CH), 7.82 (dd, J = 8.9, 2.5 Hz, 1H, ArH), 7.44 (d, J = 8.9 Hz, 1H, ArH); 13C NMR (150 MHz, CDCl₃); δ 169.3 (d, J = 16.0 Hz, C=O), 154.9 (C9), 149.3 (d, J = 250.5 Hz, C2), 143.2 (d, J = 40.1 Hz, C1), 137.2 (C7), 128.6 (d, J = 3.4 Hz, C5), 126.2 (d, J = 8.1 Hz, C4), 120.3 (C8), 119.0 (C6). HRMS (TOF ES⁺): m/z calcd for C₁₃H₁₀BrF₂O⁺ [(M + H)⁺], 242.9451; found, 242.9451.

3-Fluoro-6-nitro-4H-chromen-4-one (3d): Yield: 53%; white solid; m.p. 138–140°C; 1H NMR (600 MHz, CDCl₃); δ 8.20 (d, J = 9.0 Hz, 1H, ArH), 8.10 (d, J = 3.3 Hz, 1H, C=CH), 7.03 (dd, J = 9.0, 2.3 Hz, 1H, ArH), 6.88 (d, J = 2.3 Hz, 1H, ArH), 3.93 (s, 3H, ArOCH₃); 13C NMR (150 MHz, CDCl₃); δ 170.0 (d, J = 15.9 Hz, C=O), 164.4 (C7), 157.7 (C9), 149.4 (d, J = 248.7 Hz, C2), 142.4 (d, J = 40.5 Hz, C1), 127.3 (d, J = 3.5 Hz, C5), 118.6 (d, J = 7.4 Hz, C4), 115.0 (C6), 100.3 (C8), 56.0 (OCH₃). HRMS (TOF ES⁺): m/z calcd for C₁₃H₁₀FNO₂⁺ [(M + H)⁺], 210.0197; found, 210.0196.

3-Fluoro-6-nitro-4H-chromen-4-one (3e): Yield: 51%; white solid; m.p. 137–139°C; 1H NMR (600 MHz, CDCl₃); δ 8.19 (d, J = 8.2 Hz, 1H, ArH), 8.13 (d, J = 3.4 Hz, 1H, C=CH), 7.31 (s, 1H, ArH), 7.28 (d, J = 4.0 Hz, 1H, ArH), 2.52 (s, 3H, ArOCH₃); 13C NMR (150 MHz, CDCl₃); δ 170.5 (d, J = 15.6 Hz, C=O), 156.0 (C9), 149.3 (d, J = 248.7 Hz, C2), 145.7 (C7), 142.6 (d, J = 40.0 Hz, C8), 126.9 (C1), 125.7 (d, J = 3.3 Hz, C6), 122.6 (d, J = 7.5 Hz, C5), 118.0 (C3), 21.8 (C13). HRMS (TOF ES⁺): m/z calcd for C₁₃H₁₂F₂O₂⁺ [(M + H)⁺], 179.0503; found, 179.0503.

Scheme 3. Proposed mechanism.
3,7-Difluoro-4H-chromen-4-one (3j): Yield: 49%; white solid; m.p. 113–115°C; 1H NMR (600 MHz, CDCl3): δ 8.35–8.33 (m, 1H, ArH), 8.17 (d, J = 3.2 Hz, 1H, C=CH), 7.23–7.20 (m, 2H, ArH), 3.17 (s, 3H, ArCH3); 13C NMR (150 MHz, CDCl3): δ 169.7 (d, J = 16.1 Hz, C=O), 165.8 (d, J = 256.6 Hz, C7), 156.8 (d, J = 13.5 Hz, C9), 149.4 (d, J = 250.4 Hz, C2), 143.1 (dd, J = 40.5, 1.4 Hz, C1), 128.6 (dd, J = 10.7, 3.5 Hz, C5), 121.7 (d, J = 7.8 Hz, C4), 114.5 (d, J = 23.1 Hz, C6), 105.1 (d, J = 25.6 Hz, C8). HRMS (TOF ES+): m/z calcd for C10H10FO4+ ([M + H]+), 213.0325; found, 213.0320.

7-Chloro-3-fluoro-4H-chromen-4-one (3k): Yield: 49%; yellow solid; m.p. 136–138°C; 1H NMR (600 MHz, CDCl3): δ 8.25 (d, J = 8.6 Hz, 1H, ArH), 8.16 (d, J = 3.3 Hz, 1H, C=CH), 7.56 (d, J = 1.8 Hz, 1H, ArH), 7.44 (dd, J = 8.6, 1.7 Hz, 1H, ArH); 13C NMR (150 MHz, CDCl3): δ 169.8 (d, J = 16.0 Hz, C=O), 155.8 (C9), 149.4 (d, J = 250.6 Hz, C2), 143.0 (d, J = 40.3 Hz, C1), 140.5 (C7), 127.4 (d, J = 3.4 Hz, C5), 126.3 (C6), 123.4 (d, J = 8.0 Hz, C4), 118.4 (C8). HRMS (TOF ES+): m/z calcd for C11H9ClFO3+ ([M + H]+), 232.9567; found, 232.9571.

3,8-Difluoro-4H-chromen-4-one (3l): Yield: 38%; white solid; m.p. 168–170°C; 1H NMR (600 MHz, CDCl3): δ 8.16 (m, 1H, ArH, and C=CH), 7.74 (d, J = 1.0 Hz, 1H, ArH), 7.59 (d, J = 8.6 Hz, 1H, ArH); 13C NMR (150 MHz, CDCl3): δ 169.9 (d, J = 16.0 Hz, C=O), 155.8 (C9), 149.4 (d, J = 250.8 Hz, C2), 143.0 (d, J = 40.3 Hz, C1), 129.1 (C6), 128.6 (C7), 127.4 (d, J = 3.4 Hz, C5), 123.7 (d, J = 8.0 Hz, C4), 121.5 (C8). HRMS (TOF ES+): m/z calcd for C10H8F2O3+ ([M + H]+), 242.9451; found, 242.9452.

8-Bromo-3-fluoro-4H-chromen-4-one (3m): Yield: 35%; white solid; m.p. 156–158°C; 1H NMR (600 MHz, CDCl3): δ 8.24 (d, J = 3.2 Hz, 1H, C=CH), 8.09–8.07 (m, 1H, ArH), 7.73–7.79 (m, 1H, ArH), 7.43–7.39 (m, 1H, ArH); 13C NMR (150 MHz, CDCl3): δ 169.7 (d, J = 16.0, 2.6 Hz, C=O), 151.3 (C9), 149.5 (d, J = 250.9 Hz, C2), 144.7 (d, J = 10.9 Hz, C9), 142.8 (d, J = 40.8 Hz, C1), 126.8 (d, J = 7.8 Hz, C4), 125.3 (d, J = 6.6 Hz, C5), 121.1 (t, J = 4.0 Hz, C6), 119.8 (s, J = 16.5 Hz, C17). HRMS (TOF ES+): m/z calcd for C11H9BrFO2+ ([M + H]+), 242.9451; found, 242.9452.

Declaration of conflicting interests

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