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HFO-1234yf as a CF_3 Building Block: Synthesis of Trifluoromethyl Quinoline and Chromene Derivatives from Trifluoromethyl-ynones

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Trifluoromethyl ynones derived from the refrigerant gas HFO-1234yf (2,3,3,3-tetrafluoropropene) react with nucleophiles via a Michael-type addition process and the allenolate intermediate formed reacts intramolecularly with a carbonyl group to form heterocyclic derivatives. Thus, reaction of 2-amino and 2-hydroxyl benzaldehyde or ketone substrates with trifluoromethyl ynone derivatives gives trifluoromethyl quinoline and chromene products by a one-pot tandem ring closing sequence, forming pharmaceutically relevant structures from an inexpensive starting material without the need for transition metals or column chromatography. The 2-trifluoromethylquinoline products also show unusual fluxional behaviour, which was explored through 2-D NMR and X-ray crystallographic studies.

Introduction

Amongst the vast array of fluorinated compounds used in the pharmaceutical and agrochemical industries, nitrogen-containing heterocycles bearing a fluorine atom or trifluoromethyl group are commonplace. Amongst some of the earliest fluorinated therapeutic compounds introduced onto the market were the fluoroquinolone antibiotic derivatives, such as ciprofloxacin and moxifloxacin, which continue to be amongst the most prescribed medications to this day.[1] Quinoline systems bearing the trifluoromethyl group have come to the fore more recently, being the core scaffolds of anti-inflammatory drugs antrafenine and floctafenine[2] and the anti-malarial mefloquine (Figure 1a).[3] In recent years, various 2-(trifluoromethyl)quinolines have also been used as fluorescent probes in vivo and as having potential therapeutic value for the treatment of cancers and the Zika virus.[4]

Classical quinoline-forming annulation processes, such as the Skraup and Friedländer reactions, typically involve harsh reaction conditions.[5] Introduction of the trifluoromethyl group to quinoline rings generally relies on reactions of trifluoroacetic acid (TFA) derivatives at high temperatures or with strong acid catalysts.[6] For example, the work of Ma and co-workers (Figure 1b) where TFA at 140 °C was required to successfully install a trifluoromethyl group.[6c] Therefore, these types of trifluoromethylation strategies can be less than ideal for acid or

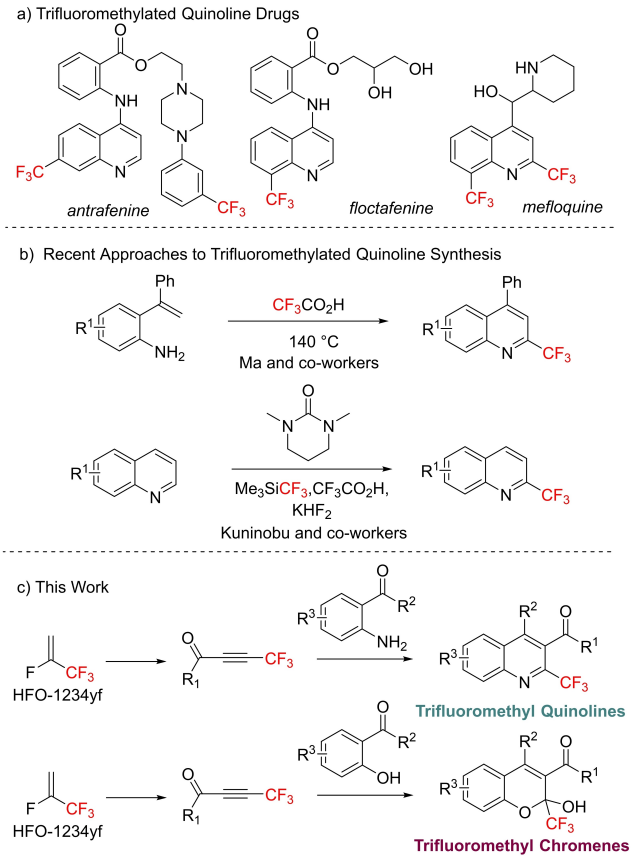

Figure 1. a) Examples of trifluoromethylated quinoline drug molecules b) Recent approaches to trifluormethyl quinolines c) This work, on accessing trifluoromethyl quinolines and chromenes from HFO-1234yf.

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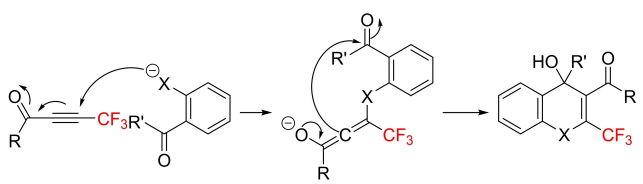
temperature sensitive substrates. Or a late-stage cross-coupling or C–H activation strategy can be employed,[7] such as that reported by Kuninobu and co-workers (Figure 1b).[7a] In this example it was found that a mixture of $TMSCF_3$, TFA, KHF_2 and DMPU were required for the efficient incorporation of trifluoromethyl groups into quinoline scaffolds. These late-stage approaches can often require complex trifluoromethylating reagents and transition metal catalysts, which can be prohibitively expensive on process scales.[7]

2,3,3,3-Tetrafluoropropene, HFO-1234yf, has emerged in recent years as a promising alternative trifluoromethylated building block to trifluoroacetic acid and trifluorotoluene derived systems because its use as a 4th generation low-global warming potential refrigerant makes it a relatively inexpensive and readily available feedstock.[8] As part of a larger scheme of work to probe the synthetic utility of HFO's we recently reported that HFO-1234yf can be converted via a two-step procedure to trifluoromethyl ynones, which are versatile building blocks for heterocycle formation via Michael addition reactions with appropriate difunctional nucleophiles.[9]

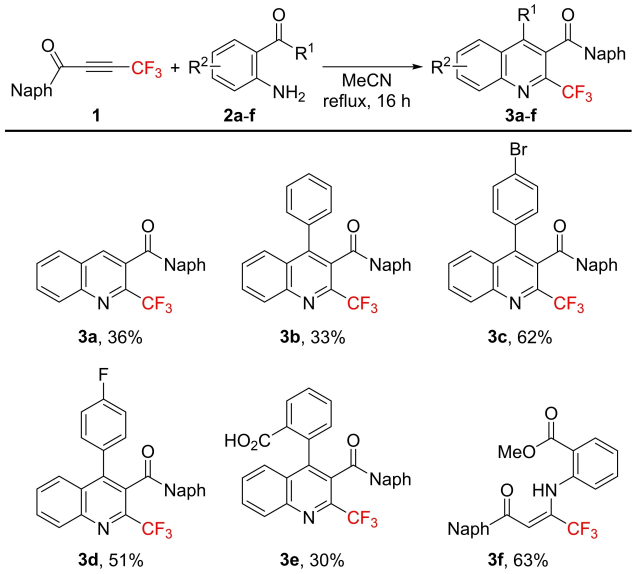
We envisioned that a tandem one-pot ring closure could give access to more complex heterocyclic scaffolds (Figure 1c) an approach which has been previously employed, all be it with the need for Lewis acid activation, to generate non-trifluoromethyl containing quinolines.[10] Rather than allenolate protonation after initial nucleophilic attack by a Michael addition reaction, followed by condensation, inclusion of an appropriately placed electrophilic moiety on the nucleophilic substrate, could enable ring-closure by attack of the intermediate allenolate at the electrophilic site (Scheme 1). For example, reaction of 2-amino and 2-hydroxyl benzaldehyde or ketone substrates with a trifluoromethyl ynone derivative could lead to heterocyclic derivatives by a tandem ring closure process. This reaction manifold would allow for multiple complex and biologically relevant classes of heterocycle to be prepared starting from HFO-1234yf under relatively mild conditions.

Results and Discussion

This methodology was initially trialed for reaction of model naphthyl ynone substrate **1** and difunctional system 2-aminobenzaldehyde (**2a**) bearing nucleophilic (NH_2) and electrophilic (C=O) sites. Reaction of **1** with **2a** in acetonitrile required heating at reflux temperature overnight to obtain full conversion to **3a**, which was isolated from the crude product mixture by recrystallisation (Scheme 2). Less reactive 2-amino



Scheme 1. Tandem ring closure strategy for the synthesis of trifluo-romethylated heterocyclic products.



Scheme 2. Synthesis of CF_3-quinolines.

benzophenone **2b** was also a suitable substrate, forming quinoline **3b** respectively. The reaction could be extended to variously substituted benzophenones **2c–e**, tolerating the presence of bromo (**3c**), fluoro (**3d**) and carboxylic acid (**3e**) functional groups. Reactions between an ynone and an aminobenzaldehyde or benzophenone derivative typically require transition metal catalysis and have not previously been carried out with any trifluoromethylated ynones.[11] However, attempts to expand the scope of this reaction to the 2-aminobenzoate ester **2f** substrate led to formation of enaminone **3f**, where the initial addition was successful but no subsequent intramolecular cyclisation occurred. Unfortunately, aminobenzaldehydes bearing bromo, chloro and nitro functional groups led to formation of complex mixtures from which no quinoline could be isolated, suggesting that electron-poor aminobenzaldehydes are not sufficiently nucleophilic to enable the initial addition. A similar intractable mixture resulted from the use of 2-aminoacetophenone, suggesting that aminoketone substrates with enolisable protons are also unsuitable for this approach.

4-Arylquinolines **3b–e** displayed unusual behaviour in their NMR spectra in that the ^{1}H NMR signals for the aryl ring are significantly broadened, and in some cases duplicated. This is typical of slow conformational exchange. To prove that this was the case, we acquired a ROESY of **3d** (Figure 2). ROESY experiments will show correlation peaks due to exchange as well as to spatial proximity, but they are distinguishable (this is not always possible using NOESY experiments); however, the experiment suffers from scalar interferences (TOCSY-[12] and COSY-types). The type of ROESY pulse sequence we used – the EASY-ROESY[13] – minimizes TOCSY-type correlations; the COSY-type signals are easy to identify (Figure 2). The results show that the aryl ring is re-orienting slowly (red peaks in Figure 2). The crystal structure of **3d**, Figure 3, provides further insight into the observed exchange behaviour. The crystal structure shows two molecules in the unit cell being disordered over two

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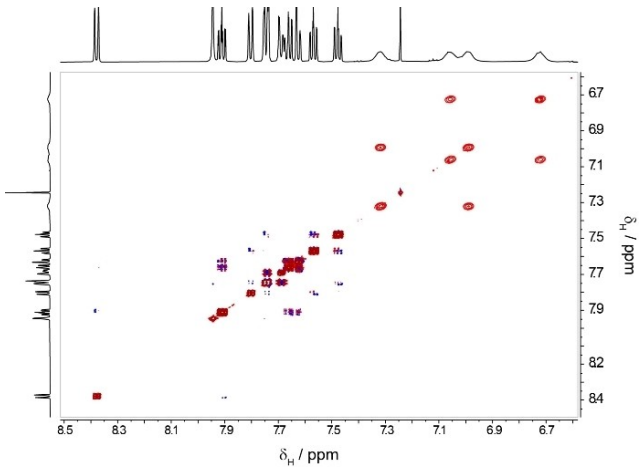


Figure 2. 600 MHz ^{1}H-^{1}H EASY-ROESY spectrum of **3 d** acquired in $CDCl_3$ at 25 °C. A 200 ms mixing time was used. Red peaks are correlations between conformationally exchanging hydrogens belonging to the aryl ring. These correlations show that the aryl ring re-orients slowly, causing signals to broaden. Blue peaks are correlations due to spatial proximity (rOe), while blue/red peaks are COSY-type correlations. No TOCSY-type signals are seen.

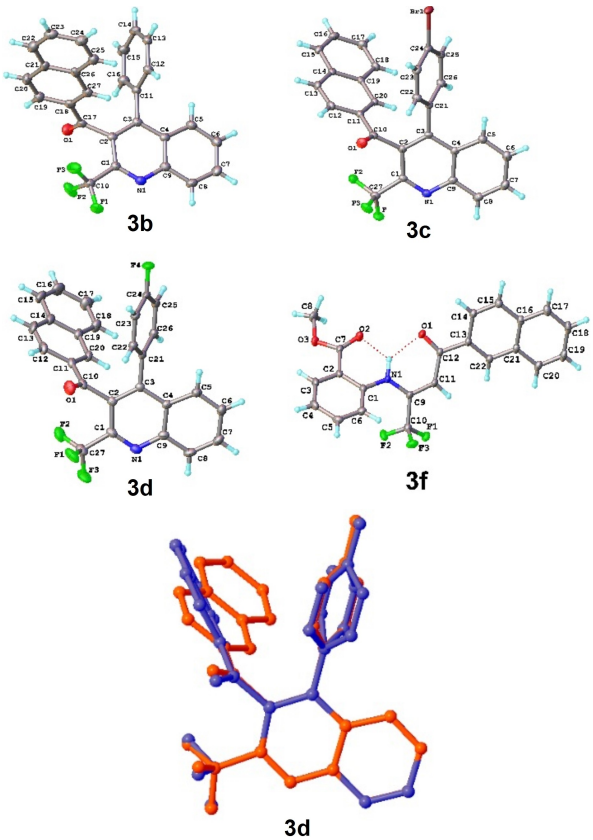


Figure 3. Structures of CF_3-quinolines **3 b**–**f** as confirmed by X-ray crystallography and disorder observed in the crystal of compound **3 d**.

positions with equal occupancy while the phenyl group sits orthogonal to the quinoline ring system and adopts two equally populated rotameric geometries, each stabilised by π-π edge-face stacking interactions to the naphthyl ring. Consequently, in solution, these rotamers interconvert at a rate similar to the NMR timescale, resulting in line-broadening and signal duplication.

Next we wished to expand the scope of the developed reaction protocol therefore we targeted the use of salicylaldeydes to generate oxygen containing heterocycles. To begin with reaction of **1** with salicylaldehyde (**4 a**) under the same conditions which produced quinolines **3 a–e** was attempted but no reaction was observed refluxing overnight. However, by adding a catalytic amount of potassium *tert*-butoxide, complete conversion was observed at room temperature in under five minutes to what was initially believed to be a 4-hydroxychromene. However, X-ray crystallographic studies demonstrated that the methyl derivative **5 b** (Figure 4) was in fact a 2-hydroxychromene, analogous to the reduced form of a coumarin, and the NMR spectra of **5 a** was consistent with the same structure (Scheme 3). Various substituted salicylaldehydes

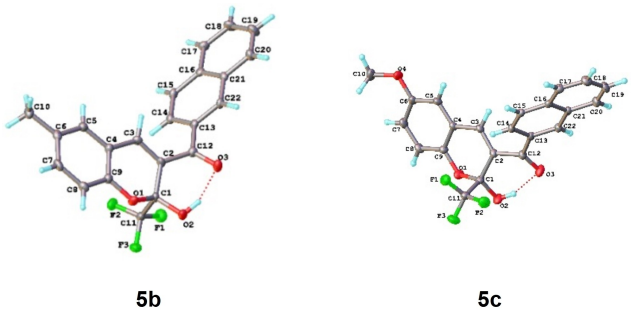
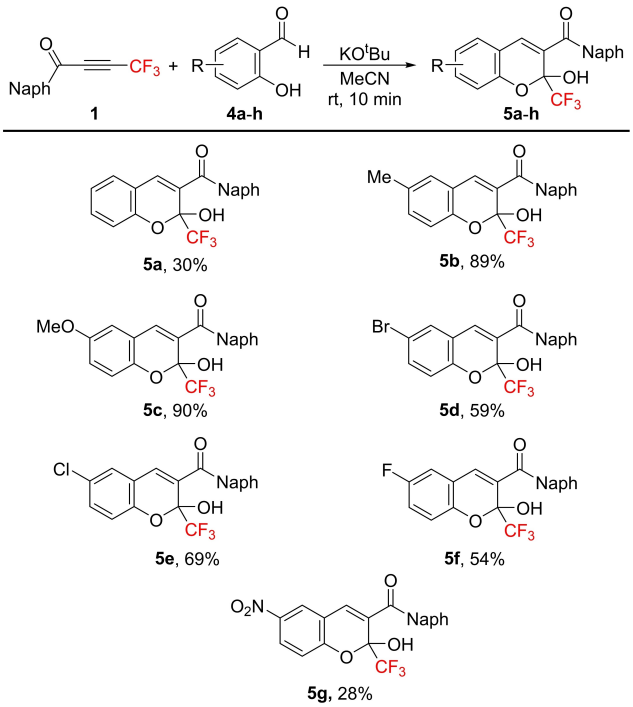


Figure 4. Structures of CF_3-chromenes **5 b** (left) and **5 c** (right) as confirmed by X-ray crystallography.



Scheme 3. Synthesis of CF_3-chromenes **5 a**–**f**.

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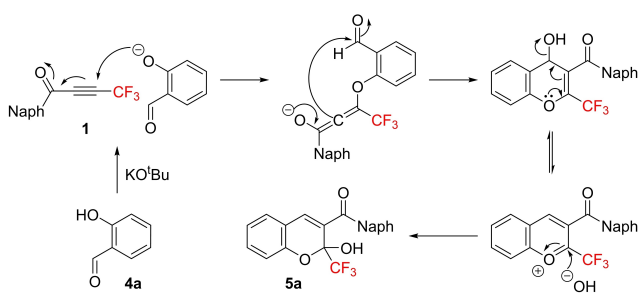
(**4a**–**g**) also reacted in a similar fashion, showing tolerance for methoxy (**5c**), bromo (**5d**), chloro (**5e**), and fluoro (**5f**) functional groups, this chromene synthesis was not affected to the same extent by electron-withdrawing groups as the quinoline-forming reactions above. This was attributed to the increased nucleophilicity of the oxygen anion.

However, the use of an iodine-substituted salicylaldehyde was unsuccessful and gave an intractable mixture of products, likely due to side reactions enabled by cleavage of the C–I bond. Similarly, no product could be recovered from reaction of **1** with 2-hydroxyacetophenone, side reactions in this case occurring due to the presence of enolisable protons. Syntheses of 2-hydroxy-2-trifluoromethyl chromene derivatives have been accomplished previously by the reaction of salicylaldehyde substrates with CF_3-ynoates,[14] CF_3-alkenyl triflates,[15] and CF_3-alkynyl phosphonates[16] but all required amine catalysts at reflux temperatures and/or long reaction times. This is in stark contrast to the relatively mild and rapid conditions utilised here.

From a mechanistic standpoint the of formation of chromenes **5a**–**h** is believed to initially follow the same path as for quinolines **3a**–**e**, involving Michael addition of the nucleophile to the ynone to generate an allenolate intermediate that cyclises via the aldehyde moiety *ortho* to the initial nucleophilic group (Scheme 4). This follows other proposals of the mechanisms of cyclisation of ynones occurring through attack of a nucleophile on the alkyne followed by the formation of an allenolate intermediate.[17] It is believed that the resulting 4-hydroxychromene then reacts with water at the 2-position at the work-up stage, to generate the final hydroxylated product.

Conclusions

In conclusion, we have shown that trifluoromethyl ynones readily derived from a refrigerant gas feedstock (HFO-1234yf) can be converted to a range of substituted CF_3-quinolines through reactions with 2-aminobenzophenone and 2-aminobenzaldehyde substrates. The 2-trifluoromethyl-4-arylquinoline products display interesting fluxional rotameric exchange behaviour, as confirmed by EASY-ROESY NMR experiments, and X-ray crystallography. Analogous tandem reactions of ynones with a range of salicylaldehyde derivatives form corresponding 2-hydroxy-2-trifluoromethylchromene product. These two tandem reactions further demonstrate the potential utility of using HFO-1234yf for the preparation of pharmaceutically relevant CF_3-substituted heterocyclic systems.



Scheme 4. Proposed mechanism for formation of CF_3-chromene **5a**.

Experimental Section

General procedure: quinoline synthesis

Ynone **1** was prepared from 2,3,3,3-tetrafluoropropene (HFO-1234yf) using the method we have previously described.[9] The 2-aminocarbonyl derivative **2a**–**f** (1 equiv.) was added to a stirred solution of **1** (1 equiv.) in acetonitrile (MeCN, 20 mL) and the reaction mixture stirred at reflux for 16 h. On reaction completion, solvent was removed *in vacuo*, the crude residue diluted with ethyl acetate, then sequentially washed with dilute HCl (3×10 mL), sodium bicarbonate (3×10 mL), and brine (3×10 mL). The combined organic extracts were dried ($MgSO_4$) and solvent removed *in vacuo*. The product was precipitated from hexane and purified by recrystallisation (ethyl acetate: hexane) to give the quinoline product (**3a**–**f**) with no further purification unless otherwise stated.

1-(Napthalen-2-yl)-1-(2-(trifluoromethyl)quinol-3-yl)methanone (3a): Following the stated general procedure, 2-aminobenzaldehyde (0.18 g, 1.5 mmol) and **1** (0.37 g, 1.5 mmol) were combined and, on removal of solvent and dilution in ethyl acetate, the crude mixture was additionally washed with sodium bisulfite (3×10 mL) to give 1-(naphthalen-2-yl)-1-(2-(trifluoromethyl)quinol-3-yl)methanone (**3a**) (0.19 g, 36%) as a pale yellow solid, m.p. 48–50 °C; δ_H (700 MHz, $CDCl_3$): 7.52–7.56 (1H, m, H16), 7.62–7.66 (1H, m, H17), 7.75–7.79 (1H, m, H4), 7.84 (1H, d, $^3J_{HH}$ = 8.2, H15), 7.90–7.98 (3H, m, H3, H5, H18), 7.97 (1H, d, $^3J_{HH}$ = 8.6, H20), 8.07 (1H, dd, $^3J_{HH}$ = 8.6, $^4J_{HH}$ = 1.8, H21), 8.14 (1H, s, H13), 8.33–8.52 ppm (2H, m, H2, H17); δ_F (376 MHz, $CDCl_3$): −62.70 (s); δ_C (176 MHz, $CDCl_3$): 121.9 (q, $^1J_{CF}$ = 276.3, C10), 124.7 (s, C21), 127.2 (s, C16), 127.5 (s, C6), 128.0 (C3 or C5 or C18), 129.0 (s, C20), 129.4 (s, C17), 129.8 (s, C4), 129.9 (s, C15), 130.3 (s, C2 or C7), 130.9 (s, C8), 132.1 (s, C3 or C5 or C18), 132.4 (s, C14), 133.5 (s, C13), 134.0 (s, C19), 136.2 (s, C12), 137.6 (s, C2 or C7), 145.0 (q, $^2J_{CF}$ = 34.7, C9), 147.0 (s, C1), 193.7 ppm (s, C11); $\tilde{\nu}_{max}$ = 1663 (C=O), 1625 (Ar, C=C), 1309, 1179, 1129, 1042 cm^{-1}; LC–MS (ESI+): 352 ($[M+H]^+$, 100%), 332 (51), 198 (7), 155 ($[C11H7O]^+$, 13); HRMS (AI+) *m/z* calc. for $C_{21}H_{13}NOF_3$ ($[M^+]$) 352.0949; found 352.0934.

General procedure: chromene synthesis

Salicylaldehyde derivative **4a**–**h** (1 equiv.) and **1** (1 equiv.) were dissolved in MeCN, to which tBuOK (10 mol%) was added. The reaction mixture was stirred at room temperature (~20 °C) for 10 min. On completion, solvent was removed in vacuo, the crude residue diluted in ethyl acetate, and sequentially washed with aq. sodium bisulfite (3×10 mL) and brine (3×10 mL). The combined organic extracts were dried ($MgSO_4$), solvent removed in vacuo, and product precipitated from hexane and recrystallised (hexane: ethyl acetate), yielding 2-hydroxychromene product (**1**–**h**) with no further purification unless otherwise stated.

1-(Naphthalen-2-yl)-1-(2-hydroxy-2-trifluoromethyl-4*H*-chromen-3-yl)methanone (5a): Following the stated general procedure, salicylaldehyde (0.12 g, 1 mmol), **1** (0.25 g, 1 mmol), and tBuOK (11 mg, 10 mol%) were combined to yield 1-(naphthalen-2-yl)-1-(2-hydroxy-2-trifluoromethyl-4H-chromen-3-yl)methanone (**5a**) (0.11 g, 30%) as a white solid, m.p. 104–106 °C; δ_H (599 MHz, $CDCl_3$): 7.03 (1H, dt, $^3J_{HH}$ = 7.5, $^4J_{HH}$ = 1.1, H4), 7.11 (1H, d, $^3J_{HH}$ = 8.3, H2), 7.18 (1H, dd, $^3J_{HH}$ = 7.6, $^4J_{HH}$ = 1.6, H5), 7.34 (1H, s, H7), 7.41–7.45 (1H, m, H3), 7.63 (1H, ddd, $^3J_{HH}$ = 8.1, $^3J_{HH}$ = 6.8 $^4J_{HH}$ = 1.2, H17), 7.69 (1H,

ddd, $^{3}J_{HH}=8.2$, $^{3}J_{HH}=6.9$ $^{4}J_{HH}=1.3$, H16), 7.91 (1H, dd, $^{3}J_{HH}=8.5$, $^{4}J_{HH}=1.8$, H21), 7.95 (1H, d, $^{3}J_{HH}=8.0$, H15), 7.98–8.03 (3H, m, H18, H20, H22), 8.35 ppm (1H, s, H13); δ_F (376 MHz, $CDCl_3$): −86.60 (s); δ_C (151 MHz, $CDCl_3$): 96.1 (q, $^{2}J_{CF}=34.8$, C9), 116.4 (s, C2), 116.8 (s, C6), 121.9 (s, C12), 122.8 (s, C4), 122.8 (q, $^{1}J_{CF}$ 290.8, C10), 125.1 (s, C21), 127.5 (s, C17), 128.1 (s, C15), 129.2 (s, C18), 129.4 (s, C16), 129.7 (s, C5), 129.9 (s, C20), 132.3 (s, C19), 132.4 (s, C13), 133.2 (s, C8), 134.3 (s, C3), 136.1 (s, C14), 140.1 (s, C7), 152.2 (s, C1), 197.8 ppm (s, C11); $\tilde{\nu}_{max}=3053$ (OH br), 1601 (C=O), 1238, 1171 cm^{-1}; ASAP-MS (ESI+): 371 ($[M+H]^+$, 2%), 353 ($[M-OH]^+$, 100), 301 (39); HRMS (AI+) *m/z* calc. for $C_{21}H_{14}O_3F_3$ ($[M+H]^+$) 371.0895; found 371.0888.

Characterisation data for all other compounds are given in the accompanying Supporting Information.

Crystallographic data

Deposition Number(s) 2085484 (**3b**), 2085485 (**3c**), 2085486 (**3d**), 2085487 (**3e**), 2085488 (**3f**), 2085489 (**5b**), 2085490 (**5c**) contain(s) the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service.

Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

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