

Short Note

N-(2,3-Difluorophenyl)-2-fluorobenzamide

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Abstract: The title compound *N*-(2,3-difluorophenyl)-2-fluorobenzamide or (**Fo23**) was obtained at high yield (88%) from the condensation reaction of 2-fluorobenzoyl chloride with 2,3-difluoroaniline using standard synthetic procedures. The crystal structure of **Fo23** was determined at 294 (1) K using single crystal X-ray diffraction methods and represents the first regular tri-fluorinated benzamide with formula C₁₃H₈F₃NO compared to the difluorinated and tetra-fluorinated analogues. In the structure, both aromatic rings are effectively co-planar, with an interplanar angle of 0.5(2)°; however, the central amide group plane is oriented by 23.17(18)° and 23.44(17)° from the aromatic rings as influenced by 1D amide···amide hydrogen bonds along the *a*-axis direction. Longer C-H···F/O interactions and the arrangement of a R²₂(12) synthon involving two C-F, a N-H and two C-H, together with C-F···C ring···ring stacking contacts, complete the interactions in the **Fo23** crystal structure.

Keywords: amide; crystal structure; conformational analysis; fluorine; hydrogen bonding; molecular stacking

1. Introduction

Benzamides continue to attract considerable research attention (and especially in structural science) as a key area in the much wider field of amide research [1–5]. Consequently, benzamides have seen applications in organic synthesis and pharmaceuticals, and through biopolymers and biochemistry [1–5]. Therefore, in terms of amide-based structural chemistry, the exponential increase in organic (bio)materials and pharmaceuticals has been noted in the literature and as archived on databases such as the Cambridge Structural Database (CSD) [3]. Fluorine derivatives continue to attract extensive attention in medicinal chemistry and pharmaceuticals, with the on-going development of new drugs and therapies [6,7]. Fluorinated benzamides, carboxamides and their structures, both solid-state and gas-phase, have been investigated over the past three decades [3,8–13]. The advances in structural research are reflected in the huge increase in published and archived benzamide datasets [3,8–13]. Of further note is our experience with both halogenated benzamides and carbamates. The crystallization of benzamides tends to be easier and form better quality single crystals compared to the carbamate analogues. The additional -O- spacer in the central OCONH gives additional flexibility to the carbamates in solution, perhaps ultimately contributing to poorer crystal growth [12,14].

In this paper, we expand on the structural knowledge of halogenated benzamides and report the chemistry and crystal structure of *N*-(2,3-difluorophenyl)-2-fluorobenzamide or (**Fo23**). The **o** notation refers to *ortho*-substitution on the mono-substituted fluorobenzoyl ring, whereas the **23** refers to di-fluoro substitution on the *N*-substituted benzene ring. The crystal and molecular structure of **Fo23** forms part of an 18-molecule series comprising three sets of six **FpXY**, **FmXY** and **FoXY** isomers, where **p**, **m** and **o** refer to *para*-, *meta*- and *ortho*-substitution and **XY** represent the six disubstituted fluorinated aromatic isomers 2,3-; 2,4-; 2,5-; 2,6-; 3,4-; and 3,5- (**X** = 2 or 3 and **Y** = 3, 4, 5 or 6). In reporting the first compound in a series of papers of this type, we also highlight that a gap exists in the structural data for trisubstituted (fluorinated) benzamides. This contrasts with their di-fluorinated and tetra-fluorinated counterparts, which have been described well [3,8–10,13]. This is also



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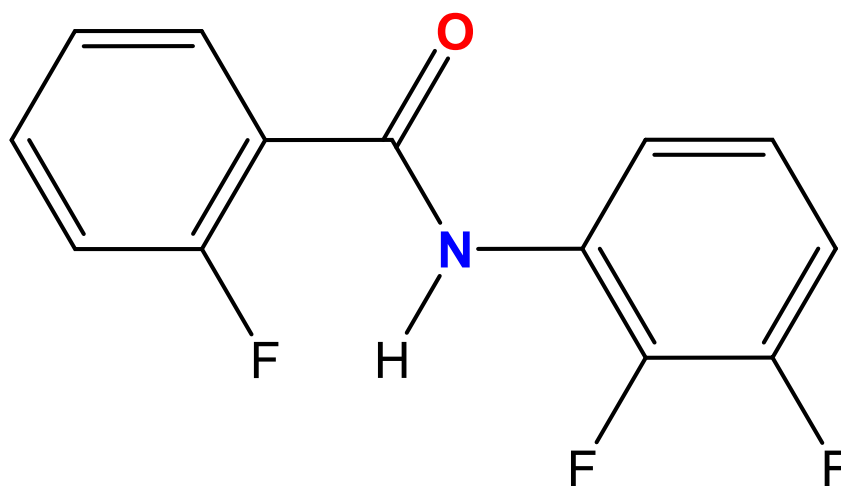
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highlighted in the ESI, where the relative numbers of halogenated (fluorinated) benzamides are compared.

2. Experimental Section

2.1. Materials and Characterisation

The chemicals and materials, spectroscopy, X-ray diffraction methods and analytical equipment are as described previously [12–14]. Chemicals utilised in the synthesis of **Fo23** (Scheme 1) were used without purification, as purchased from Sigma Aldrich (Ireland). The synthetic approaches are standard and have been used previously by us and other research groups [8–13]. The single crystal X-ray diffraction methods and data collection procedures for the **Fo23** crystal structure (Scheme 1) are routine for data collected at 294(2) K [14]. Data collection, reduction, structure solution and refinement used the SHELXS, SHELXL14 programs [15]. The molecular and hydrogen bonding diagrams (Figures 1–3) were generated using the Mercury graphics program [16] and with geometric analysis using both SHELXL14 [15] and PLATON [17]. CSD analyses were performed with version 5.42+4 updates on 21 July 2023 [3]. The CSD analyses and hydrogen bond/contact data (Table S1, Supplementary Materials) are included in the supplementary information.



Scheme 1. A schematic diagram of **Fo23**.

2.2. Reaction Procedure and Characterisation: Experimental and Spectroscopic Data

Synthetic yield (%) = 88%. Melting point range of 100–102 °C.

Experimental (Calculated) CHN Analysis (%): C = 62.5% (62.2); H = 3.0% (3.2); N = 6.0% (5.6). ¹H NMR data (CDCl₃): 6.88 (1H, qd, ³J = 8.88, ⁴J = 1.5), 7.05 (1H, ddd, ³J = 8.7, ⁴J = 2.1), 7.15 (1H, dd, ³J = 8.2, ⁴J = 1), 7.27 (1H, td, ³J = 7.6, ⁴J = 1), 7.50 (1H, m, ³J = 7.6), 8.12 (1H, td, ³J = 8.2, ⁴J = 1.7), 8.20 (1H, tt, ³J = 7.5, ⁴J = 1.5), 8.73 (1H, d, ³J = 16.4). ¹H NMR data (*d*⁶-DMSO): 7.30 (4H, m), 7.57 (1H, ddd, ³J = 6.9), 7.62 (1H, ddd, ³J = 6.5, ⁴J = 1.7), 7.73 (1H, td, ³J = 7.5, ⁴J = 1.65), 10.39 (1H, br. s). ¹⁹F NMR data (*d*⁶-DMSO) (ppm): −114, −139, −147. IR (ATR): 3370 (m), 2924 (w), 1661 (m), 1610 (m), 1546 (m), 1469 (s), 1289 (m), 1215 (m), 1134 (m), 820 (m). The ¹³C spectral data are presented in the supplementary information (ESI).

Fo23 crystal structure data were collected on an Oxford Diffraction Xcalibur Sapphire 3 (Gemini ultra) diffractometer.

Chemical formula: C₁₃H₈F₃ON; Mr 251.20; crystal system and space group, monoclinic *Pn* (No. 7); T = 294 (2) K; *a* = 4.9556 (2), *b* = 5.6718 (3), *c* = 19.6250 (15) Å, β = 96.618(6)°, V = 547.93 (6) Å³; radiation Mo-K_α; μ = 0.13 mm^{−1}; crystal size 0.14 × 0.06 × 0.04 mm; analytical absorption correction with T_{min,max} = 0.965, 0.988; number of measured, independent, observed [*I* > 2σ(*I*)] reflections and parameters, 4269, 1600, 1334 and 167 with 2 restraints; R_{int} = 0.021; R[*F*² > 2σ(*F*²)] = 0.038, wR(*F*²) = 0.079, Goodness of fit = 1.09; hydrogen atoms treated by a mixture of independent (N-H) and constrained (C-H) refinement;

$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (as $e \text{ \AA}^{-3}$) = 0.12, -0.09 ; the absolute structure = $-1.5(8)$; and the inverted structure = $2.5(8)$ (Flack).

3. Results and Discussion

The **Fo23** compound was synthesized at high yield using standard condensation procedures. The spectroscopic data were as expected, and an inspection of the ^1H NMR spectrum of **Fo23** attests to its overall purity; the ^{13}C and ^{19}F spectra were as predicted; for the ^{19}F NMR, the three peaks were located at -114 , -139 and -147 ppm and are typical of the fluorine substitution patterns expected on the two substituted aromatic rings.

The molecular structure of **Fo23** is largely planar with respect to the aromatic rings and these C_6 rings have an interplanar angle of $0.5(2)^\circ$ (Figure 1). The amide group ($\text{C}=\text{ONH}$) is oriented from the aromatic planes at angles of $23.17(18)^\circ$ and $23.44(17)^\circ$. This arises with the formation of 1D amide \cdots amide hydrogen bonds in the a -axis direction. There is intramolecular contact between N1 and F12, with $\text{N1} \cdots \text{F12} = 2.745(3) \text{ \AA}$. The amide H1 was refined with isotropic displacement parameters and this resulted in a $\text{H1} \cdots \text{F12}$ distance of $2.17(3) \text{ \AA}$ and with a $\text{N1-H1} \cdots \text{F12}$ angle = $126(3)^\circ$. This intramolecular distance is reasonably short [3], but not as short as the intramolecular $\text{H} \cdots \text{F}$ distances of ca. 1.95 \AA as reported on the CSD [3,18] and predicted by Leckta and co-workers in a series of naphthalenylbenzamide structures [19]. In the present structural study of **Fo23**, an auxiliary intramolecular $\text{C26-H26} \cdots \text{O1}$ interaction was also present, with a $\text{C26} \cdots \text{O1}$ distance of $2.853(5) \text{ \AA}$.

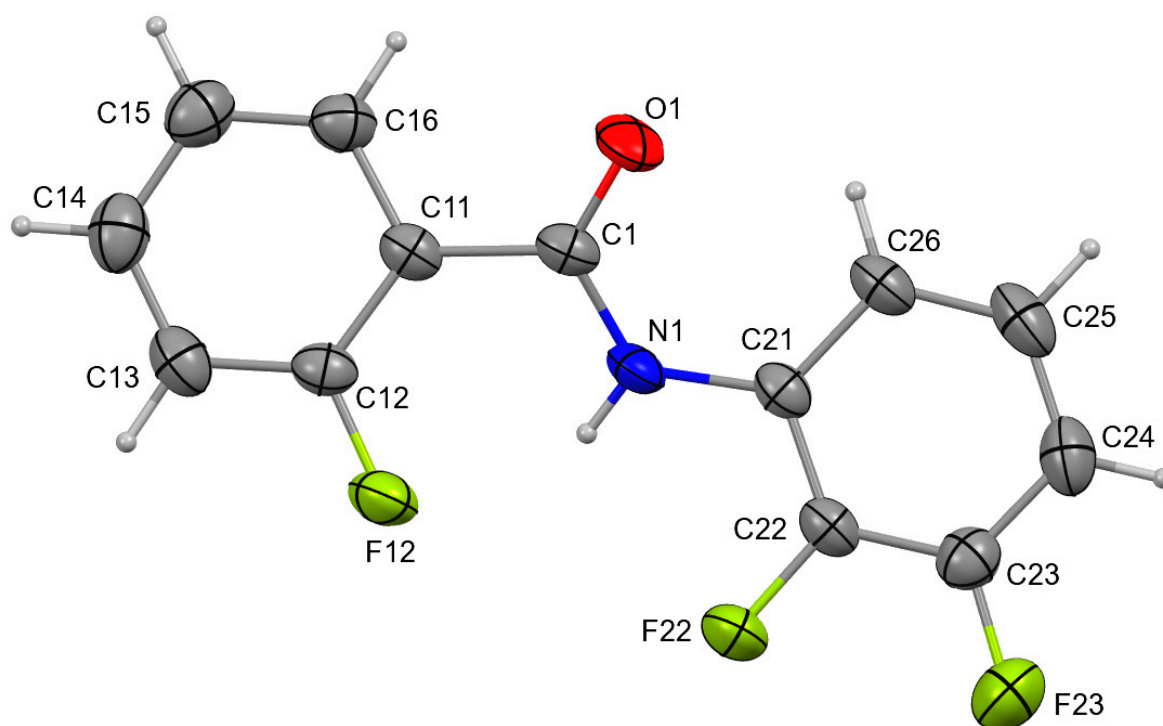


Figure 1. An ORTEP diagram of the **Fo23** structure with displacement ellipsoids at the 30% probability level.

The primary intermolecular hydrogen bonding interaction is the $\text{N1} \cdots \text{O1}^i = 3.054(4) \text{ \AA}$ as an amide \cdots amide interaction linking **Fo23** molecules into 1D chains along the a -axis direction (symmetry code: $i = 1+x,y,z$) (Figure 2). In addition there is a notable synthon involving longer intermolecular $\text{C-H} \cdots \text{F}^{ii}$ contacts, such that two C-Hs (H25, H26) form a cyclic hydrogen bonded $R^2_2(12)$ motif with fluorine atoms F12 and F22 on an adjacent **Fo23** molecule (symmetry code: $ii = x-1,1+y,z$). The $\text{H} \cdots \text{F}^{ii}$ distances are 2.51 and 2.56 \AA . This involves the N-H group as positioned *syn*- to the two fluorine atoms

(Figure 3). The non-hydrogen atoms in this cyclic arrangement are co-planar and effectively parallel to the (112) plane. There are C-F \cdots C ring \cdots ring stacking contacts involving C12-F12 \cdots C26 with F12 \cdots C26ⁱⁱⁱ = 3.151(4) Å and F12 \cdots Cg1ⁱⁱⁱ = 3.399(2) Å (symmetry code: *iii* = *x*,*y*-1,*z*; Cg1 is the C₆ ring centroid; Table S1, ESI). The cyclic type of composite interaction in **Fo23** as noted in Figure 3 is also observed in **PIHQUT** [3,5], where the H \cdots F distances are 2.54 Å and **YAZBOT** [10] (H \cdots F distances of 2.42 and 2.49 Å). The **YAZBIN** structure (or *N*-(2,4-difluorophenyl)-2,4-difluorobenzamide) with X-ray data collected at 100 K is effectively isomorphous with **Fo23** and the molecular conformations show a good overlap [17]. Differences between the interactions in the **Fo23** and **YAZBIN** structures can be attributed to the different fluorine substitution pattern, data collection temperatures and disorder in one of the benzene rings in **YAZBIN** [10]. Of further note is that the four related crystal structures [**RUXZOB** in *P*₂₁₂₁₂₁, **RUXZUH** in *P*₂₁/*n* [9]; **YAZBAF** in *P*₂₁/*c*, **YAZBEJ** in *Pna*₂₁ [10] differ from **Fo23** by changing a C-H for C-F (a H for F swap). The four crystal structures are quite different to **Fo23** and use different packing arrangements in three different space groups. Only **YAZBEJ** of the four related structures [9,10] displays a similar but more asymmetric *R*²₂(12) synthon, but with the C=O positioned *syn*- to the two C-F groups. This highlights once again the differences that can arise in closely related structures and why a thorough and up to date analysis is needed, with additional crystal structures archived to fill the known gaps in the CSD [3]. Rationalizing why similarities and differences in series of molecules (isomers) require complete series of crystal structures, preferably at different temperatures, and their polymorphs allowed us to gain a thorough insight into crystallization behaviour.

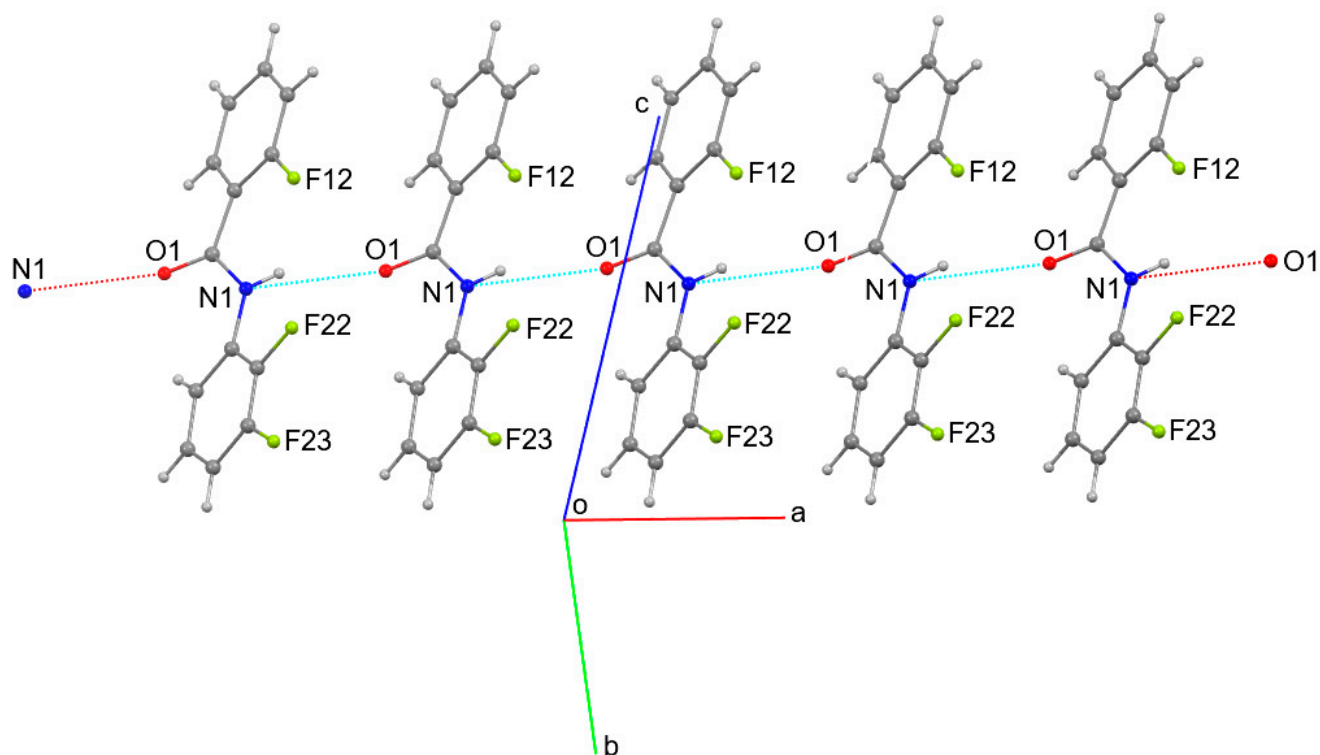


Figure 2. A view of the amide \cdots amide interaction along the *a*-axis in **Fo23**.

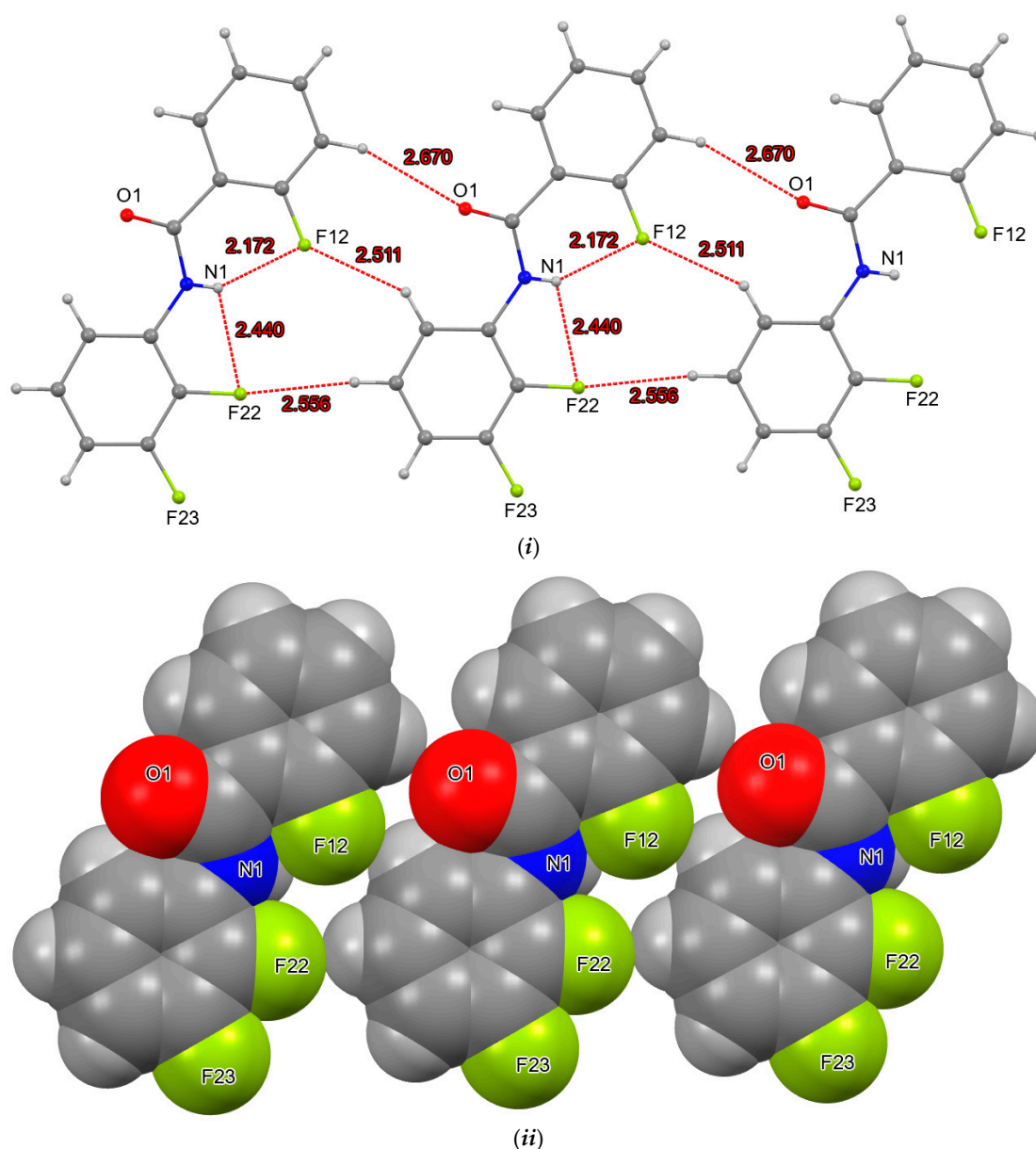


Figure 3. (i) A view of the C-H...O/F interactions in **Fo23** involving O1, F12 and F22 (with the interaction distances in Å) and (ii) a similar view with atoms depicted as their van der Waals spheres.

From the CSD [3], it is noted that **JOFHAO** is 2-chloro-*N*-(2,3-dichlorophenyl)benzamide [20] and is the trichloro-equivalent crystal structure of **Fo23**. Indeed, **JOFHAO** crystallizes in space group *Pc* (No. 7), but unlike **Fo23** (in space group *Pn*) has $Z' = 2$. The two molecules adopt different conformations, with one molecular conformation similar to that of **Fo23** and the second with the *ortho*-chlorobenzene ring rotated by ca. 180° degrees and positioned *syn* to the amide C=O group. The beauty of the **JOFHAO** structure is that it shows that both conformations are easily accessible and crystallize in the structure in equal numbers without disorder [20].

4. Overall Structural Results and Related Literature

There are no tri-fluorinated benzamide structures available of the type C_6CONHC_6 with chemical formula $C_{13}H_8F_3ON$ for direct comparisons with **Fo23** [3]. This is regardless of whether the structure is trisubstituted fluorine on one C_6 aromatic ring or in any combination of *o*-/*m*-/*p*-monosubstituted F and disubstituted F_2 as [2,3-; 2,4-; 2,5-; 2,6-; 3,4- and 3,5- F_2] on the second aromatic ring. It is noted there are many di- and tetra-fluorinated

analogues present on the CSD for comparisons [3,8–10,13]. In expanding the general CSD search and reviewing all combinations of trihalides ($X = F, Cl, Br, I$), the majority of structures are chloro-derived and have 10 structural ‘hits’ and 10 structures available on the CSD [3].

For the difluorinated benzamide analogues (formula = $C_{13}H_9F_2ON$), there are 30 crystal structure ‘hits’ with 19 individual molecules. The discrepancy arises whereby some crystal structures have datasets collected at different temperatures, datasets, polymorphs, etc. The expanded halide series (from using only fluorine) has 89 structural ‘hits’ and 74 individual structures. For the tetra-fluorinated benzamides there are 29 structural ‘hits’ and 27 individual structures using $C_{13}H_7F_4ON$ [3,9,10]. Interestingly, thus far, there are no other Cl, Br, I combinations available [3]. In addition, the mono-fluorinated *N*-phenylbenzamides (as $C_{13}H_{10}FON$) reveals a total of eight structural ‘hits’ and six crystal structures, although **UXEZIH** is a mixed 1:1 molecular system with a methyl/F 50:50 present (site occupancies of 0.5) [21]. Expanding the CSD search to include all halides X ($X = F, Cl, Br, I$) provides 24 structural ‘hits’ and 16 individual crystal structures. In the analysis, all searches were conducted using the latest CSD version [3]. Therefore, the crystal structure of **Fo23** is unique (thus far) in terms of it being the first structural report of a regular tri-fluorinated benzamide of chemical formula $C_{13}H_8F_3ON$. However, it can be stated that the tri-fluorinated benzamide group of unpublished crystal structures will soon be reported and available for larger analyses using both CSD and AI approaches.

5. Conclusions and Future Work

The sheer number of simple benzamide structures that are now available on structural databases such as the Cambridge Structural Database [3] means that both structural and physicochemical research has a rich base from which to develop and expand. Future work is aimed at developing larger $n \times m$ isomer grids with a view to correlating melting points, spectroscopic and structural data based on halogenated benzamides [3,10,13]. However, as noted in the crystal structure of **Fo23**, there are a number of gaps in the currently available structural data, especially for tri-fluorinated benzamide analogues and mixed halogenated benzamides [3]. This could develop into a rich vein of structural research, as halogenated benzamide and their mixed halide analogue datasets are reported and archived [3]. However, often a reason for the lack of reporting on some crystal structures is the prevalence of group and molecular disorder, as seen in many benzamide structures [3,13]. Work is on-going by our group to increase the number of benzamides communicated to augment the data already available and to assist in the future endeavours of crystal structure analysis for structural systematics [3,12,13,21]. Our future research will complement that of on-going fluorine chemical research and developments in benzamide chemistry and related compounds [22–26].

Supplementary Materials: The following are available online: the 1H , ^{13}C and ^{19}F NMR spectral data, four supplementary diagrams, CSD analyses, the hydrogen bonding table and **Fo23** CheckCIF report.

Author Contributions: N.H. synthesized, characterised and crystallized **Fo23**; J.F.G. collected and analysed the crystallographic data; J.F.G. wrote and submitted the **Fo23** manuscript. All authors have read and agreed to the published version of the manuscript.

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