Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

# Synthesis and crystal structures of five fluorinated diphenidine derivatives

Ryan E. Mewis,[a]* Matthew C. Hulme,[a] Jack Marron,[a] Stuart K. Langley,[a] Oliver B. Sutcliffe[a]* and Sophie L. Benjamin[b]

[a]Department of Natural Sciences, Manchester Metropolitan University, John Dalton Building, Chester St., Manchester, M1 5GD, United Kingdom, and [b]School of Science and Technology, Nottingham Trent University, Nottingham NG11, 8NS, United Kingdom. *Correspondence e-mail: r.mewis@mmu.ac.uk, o.sutcliffe@mmu.ac.uk

Received 30 October 2024
Accepted 11 February 2025

Edited by F. F. Ferreira, Universidade Federal do ABC, Brazil

**Keywords:** crystal structure; diphenidine; novel psychoactive substances.

**CCDC references:** 2423059; 2423058; 2423057; 2423056; 2423055

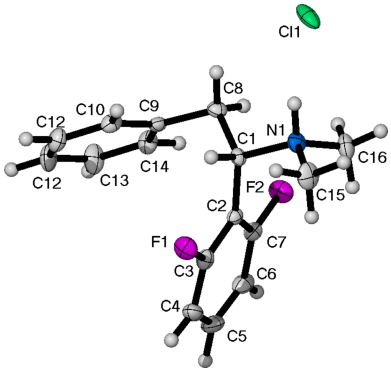
**Supporting information:** this article has supporting information at journals.iucr.org/e

Diphenidine (**1a**), a dissociative anaesthetic, was first reported in 2013. Since then, a number of derivatives *e.g.* 2-methoxphenidine (**1b**) have been produced by clandestine laboratories and sold as *research chemicals*. Fluorinated diphenidines, namely, [1-(2,6-difluorophenyl)-2-phenylethyl]dimethylazanium chloride, $C_{16}H_{18}F_2N^+\cdot Cl^-$, (**I**), [1-(2,6-difluorophenyl)-2-phenylethyl](ethyl)azanium chloride dichloromethane hemisolvate, $2C_{16}H_{18}F_2N^+\cdot 2Cl^-\cdot CH_2Cl_2$, (**II**), *tert*-butyl[1-(2,6-difluorophenyl)-2-phenylethyl]azanium chloride, $C_{18}H_{22}F_2N^+\cdot Cl^-$, (**III**), 1-[1-(2,6-difluorophenyl)-2-phenylethyl]pyrrolidin-1-ium chloride, $C_{18}H_{20}F_2N^+\cdot Cl^-$, (**IV**), and 1-[1-(2,3,4,5,6-pentafluorophenyl)-2-phenylethyl]-piperidin-1-ium chloride, $C_{19}H_{19}F_5N^+\cdot Cl^-$, (**V**), were synthesized and structurally characterized by $^1H$, $^{13}C$ and $^{19}F$ NMR spectroscopy, and single-crystal X-ray diffraction. All five structures exhibit hydrogen bonding between the quaternary amine hydrogen atoms and the chlorine. The N—H···Cl distances for (**II**) and (**III**) range from 2.21 to 2.31 Å, whereas (**I**), (**IV**) and (**V**) exhibit shorter N—H···Cl distances (2.07–2.20 Å). Compounds (**IV**) and (**V**) include pyrrolidine and piperidine rings, respectively; the pyrrolidine ring adopts an envelope conformation whereas the piperidine ring adopts a chair conformation. The crystal packing in compounds (**I**)–(**V**) is characterized by C—H···$\pi$ interactions; no $\pi$–$\pi$ interactions are observed.

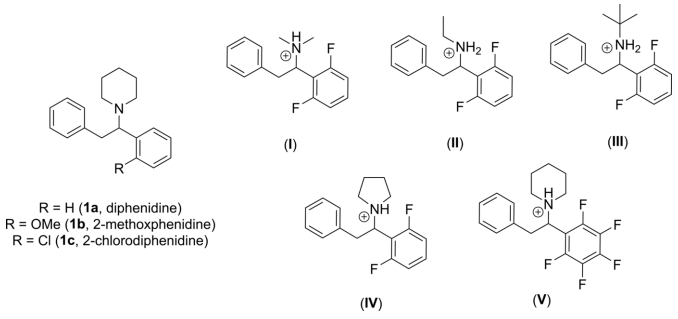
## 1. Chemical context

Over the past two decades, there has been a significant increase in the number of new psychoactive substances (NPS) seized by law enforcement agencies globally (King, 2013; UNODC, 2024). Current convention uses a functional 'effect group' categorization to define NPS within six broad overlapping groups: (i) synthetic cannabinoid receptor agonists; (ii) classic hallucinogens; (iii) stimulants; (iv) opioid receptor agonists; (v) sedatives/hypnotics and (vi) dissociatives (UNODC, 2024; Tettey *et al.*, 2018; Shafi *et al.*, 2020). NPS are assigned to a specific 'effect group' based on their chemical structure and psychopharmacological effects (UNODC, 2024; Tettey *et al.*, 2018). 1,2-Diarylethamines are dissociative, psychoactive substances, which distort perceptions, produce feelings of detachment, and induce a state of anaesthesia by antagonizing ionotropic *N*-methyl-D-aspartate receptors (NMDAR) in the central nervous system (UNODC, 2024; Morris & Wallach, 2014).

The first of these dissociative anaesthetics was 1-(1,2-diphenylethyl)piperidine (diphenidine, **1a**) (Wallach *et al.*, 2015) reported in 2013 (Morris & Wallach, 2014), followed by 1-[1-(2-methoxyphenyl)-2-phenylethyl]piperidine (2-methoxphenidine, **1b**) (McLaughlin *et al.*, 2016), which have both been marketed as '*research chemicals*' and encountered in tablet or



powder forms (UNODC, 2024; Wallach *et al.*, 2015; McLaughlin *et al.*, 2016; Odoardi *et al.*, 2016; Strano Rossi *et al.*, 2014) or in combination with synthetic cannabinoids such as AB-CHMINACA, 5F-AMB (Hasegawa *et al.*, 2015) and 5F-AB-PINACA (Wurita *et al.*, 2014). Though both the supply and production of **1a**, **1b** and the recently disclosed 1-[1-(2-chlorophenyl)-2-phenylethyl]piperidine (2-chlorodiphenidine, **1c**) (Wallach *et al.*, 2016; Sahai *et al.*, 2018), are now controlled in the United Kingdom by the 2016 Psychoactive Substances Act (Reuter & Pardo, 2017), the emergence of novel 1,2-diarylethylamine derivatives, such as the fluorinated compounds, (**I**)–(**V**), still raises considerable legal and analytical challenges in both the forensic identification and discrimination of these materials. This is due to the inference of diphenidine-based NPS in several fatalities in Europe (Morris & Wallach, 2014; Wallach *et al.*, 2015, 2016; McLaughlin *et al.*, 2016; Strano Rossi *et al.*, 2014; Hasegawa *et al.*, 2015; Wurita *et al.*, 2014; Sahai *et al.*, 2018; Reuter & Pardo, 2017; Elliott *et al.*, 2015; Helander *et al.*, 2015; Hofer *et al.*, 2014), Asia (Hasegawa *et al.*, 2015; Minakata *et al.*, 2016; Kudo *et al.*, 2015) and **1a** being placed under international control, within schedule II of the United Nations Convention on Psychotropic Substances (1971), on 14th April 2021 (UNODC, 2021).




## 2. Structural commentary

Compound (**I**) (Fig. 1) crystallizes in the monoclinic space group $P2_1/c$ with a single molecule in the asymmetric unit. The torsion angle between the two quaternary carbons of the phenyl rings and the bridging ethyl chain is 53.4 (2)°.

Compound (**II**) (Fig. 2) crystallizes in the *I*2/*a* space group. It consists of one molecule in the asymmetric unit, as well as half of a single molecule of dichloromethane (DCM). The terminal carbon of the ethyl group (C15, C15*A*) is disordered over two positions [0.707 (5):0.293 (5) occupancy]. The closest contact between one of the fluorine atoms of the 2,6-difluorophenyl ring and a hydrogen atom of DCM is 2.335 Å. The torsion angle for (**II**), as defined previously for (**I**), is −55.9 (2)°. The final non-cyclic aliphatic analogue, (**III**) (Fig. 3), crystallizes in the monoclinic space group $P2_1/c$ with a single formula unit in the asymmetric unit cell. The torsion angle is the largest of all the structures presented herein at 63.8 (2)°.

Compound (**IV**) (Fig. 4) crystallizes in the triclinic space group $P\overline{1}$ with two molecules in the asymmetric unit. Torsion

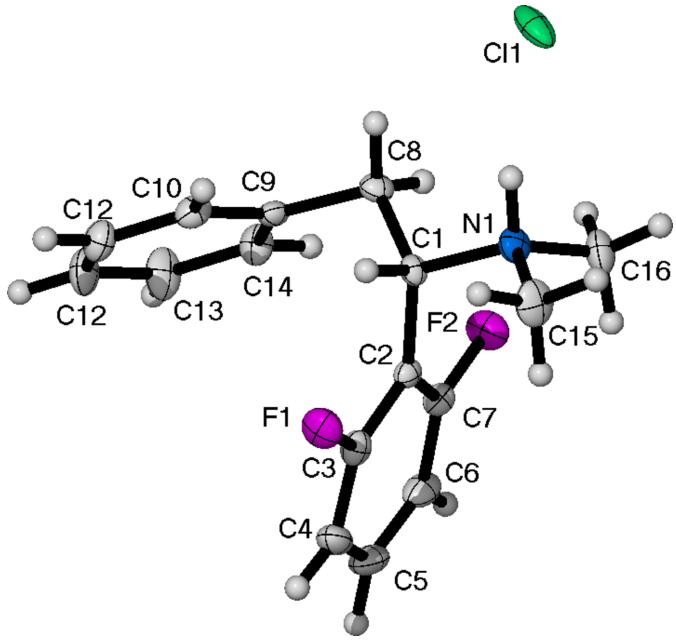



**Figure 1**
The molecular structure of (**I**), showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

angles of −54.6 (3) and 58.9 (3)° are very similar to (**I**) and (**III**). The pyrrolidine ring present in the structure adopts an envelope conformation.

Compound (**V**) (Fig. 5) crystallizes in the monoclinic space group $P2_1/c$ with a single molecule in the asymmetric unit. The torsion angle defined is the smallest of the crystal structures presented at 47.3 (2)°. The piperidine ring is in the chair conformation. All five structures exhibit hydrogen bonding between the quaternary amine and the chlorine (Tables 1–5). The five structures can be split in to two groups; (**II**) and (**III**) both have two *R* groups attached to the amine whereas the

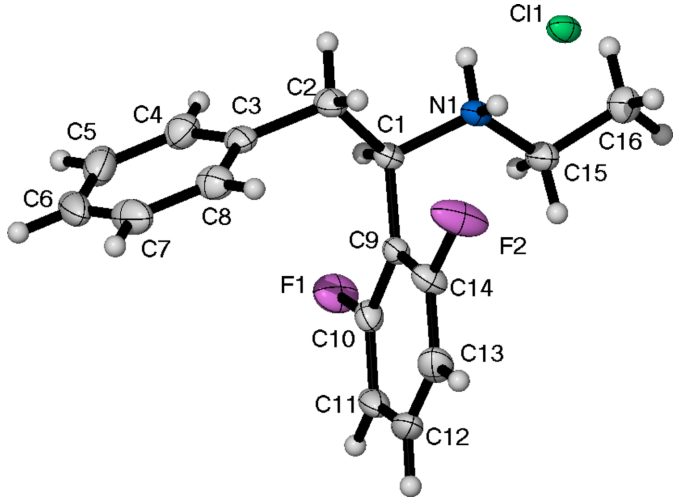



**Figure 2**
The molecular structure of (**II**), showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level. The half molecule of DCM present has been omitted.

**Table 1**
Hydrogen-bond geometry (Å, °) for (**I**).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*1···Cl1 | 0.93 (2) | 2.08 (2) | 3.0006 (17) | 167.3 (19) |

remainder all possess three. The N—H···Cl distance for the former grouping range from 2.21 to 2.31 Å, with N—H—Cl angles of 151–168° (Tables 2 and 3). Interestingly, in (**II**), a shorter N—H1*A*···Cl distance of 2.11 Å (compared to 2.30 Å for N—H1*B*···Cl) is observed to a symmetry-related [symmetry code: (i) $\frac{1}{2}-x, \frac{3}{2}-y, \frac{1}{2}-z$] Cl atom. The latter group, consisting of (**I**), (**IV**) and (**V**) exhibit shorter

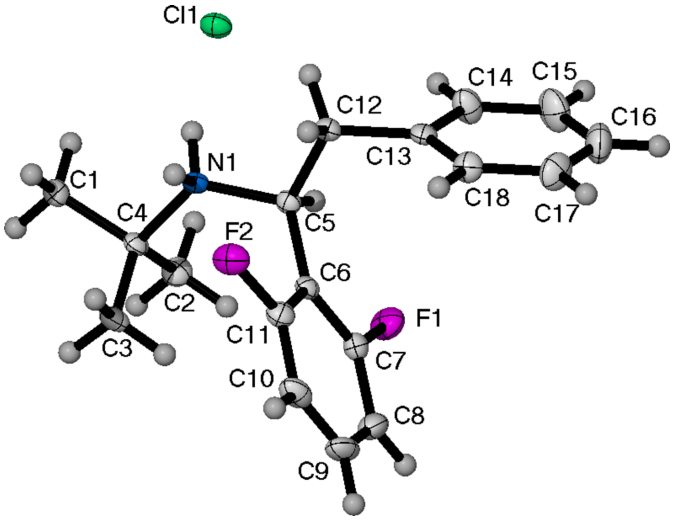



**Figure 3**
The molecular structure of (**III**), showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

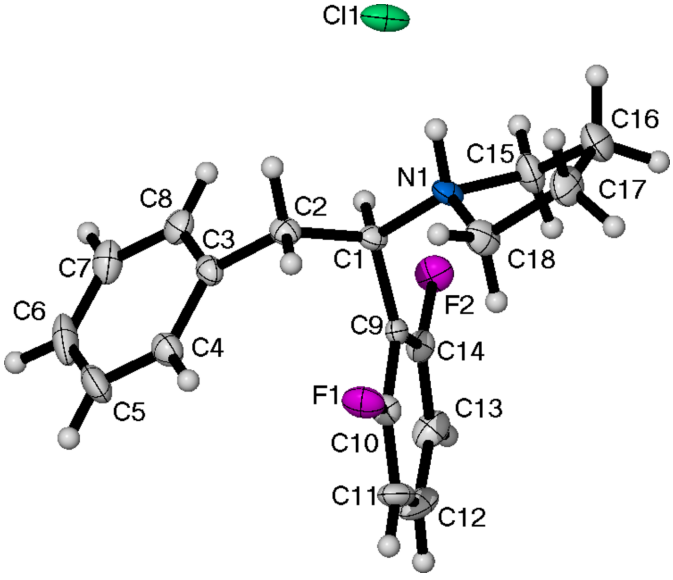



**Figure 4**
The molecular structure of (**IV**), showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level. Only one molecule present in the asymmetric unit is shown.

**Table 2**
Hydrogen-bond geometry (Å, °) for (**II**).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*B*···Cl1 | 0.89 (2) | 2.30 (2) | 3.1417 (14) | 156.0 (16) |

**Table 3**
Hydrogen-bond geometry (Å, °) for (**III**).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···Cl1 | 0.92 (2) | 2.21 (3) | 3.115 (2) | 167.7 (19) |
| N1—H1*B*···Cl1$^{i}$ | 0.95 (2) | 2.31 (2) | 3.1684 (19) | 151 (2) |

Symmetry code: (i) $-x+1, -y+2, -z$.

**Table 4**
Hydrogen-bond geometry (Å, °) for (**IV**).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1′···Cl1 | 0.99 (1) | 2.07 (2) | 3.021 (5) | 163 (6) |
| N2—H2···Cl2 | 0.98 (1) | 2.08 (2) | 3.052 (5) | 169 (6) |

**Table 5**
Hydrogen-bond geometry (Å, °) for (**V**).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···Cl1 | 0.85 (3) | 2.20 (3) | 3.051 (3) | 178 (3) |

N—H···Cl distances (2.07–2.20 Å, Tables 1, 4 and 5) as well as N—H—Cl angles that are all greater than 163°.

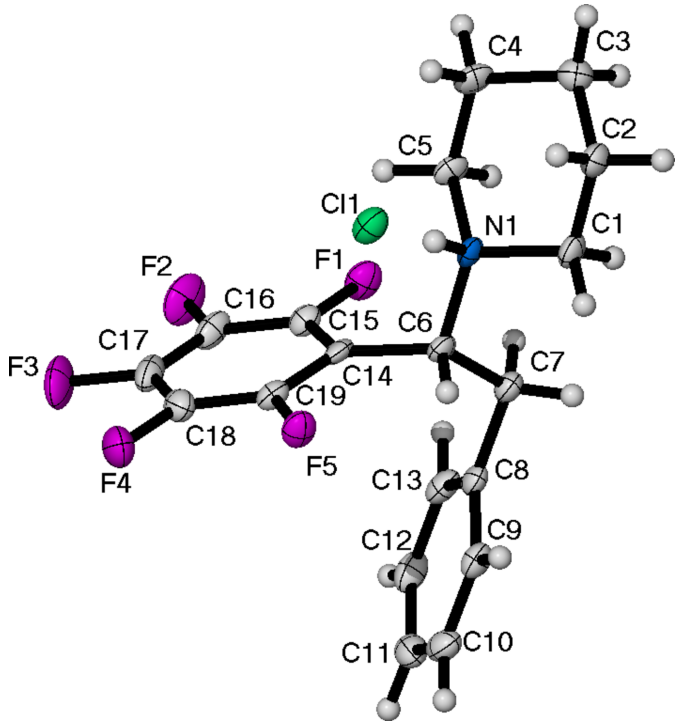



**Figure 5**
The molecular structure of (**V**), showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

## 3. Supramolecular features

Molecules of (**I**) exhibit no $\pi$–$\pi$ interactions, as despite the unsubstituted phenyl rings being aligned when viewed along the *c*-axis direction, the shortest centroid–centroid distance is 7.947 Å [symmetry operation $1 + x, y, z$]. Molecules are linked together by C—H$\cdots\pi$ interactions; the distance of the centroid of the unsubstituted phenyl ring to the nearest aromatic protons of a substituted aromatic ring are 3.274 and 3.951 Å [$Cg1\cdots H4^{i} = 3.274$ Å and $Cg1\cdots H5^{i} = 3.951$ Å; $Cg1$ is the centroid of C9-C14 ring; symmetry code: (i) $1 + x, y, z$]. Another C—H$\cdots\pi$ interaction exists between the centroid of the difluorinated ring and a phenyl ring proton of a neighbouring molecule [$Cg2\cdots H11^{ii} = 2.982$ Å; $Cg2$ is the centroid of the C2–C7 ring; symmetry code: (ii) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$].

Analysis of (**II**)–(**V**) reveals that these also exhibit no $\pi$–$\pi$ interactions. Similarly to (**I**), they do exhibit weak C—H$\cdots\pi$ interactions with distances of 3.244–3.425, 3.427–3.744 and 2.929–3.459 Å for (**II**), (**III**) and (**IV**), respectively. between the nearest ring hydrogen of the difluorinated ring and that of the centroid of the nearest neighbouring phenyl ring. (**II**) also exhibits a C—H$\cdots\pi$ interaction between the non-fluorinated phenyl rings of neighbouring molecules [$Cg3\cdots H4^{iii} = 2.969$ Å; $Cg3$ is the centroid of ring C3–C8; symmetry code: (iii) $-x, y - \frac{1}{2}, \frac{1}{2} - z$]. Similarly, (**III**) has the same interaction [$Cg4\cdots H17^{iv} = 3.785$ Å and $Cg4\cdots H18^{iv} = 4.105$ Å; $Cg4$ is the centroid of ring C13–C18; symmetry code: (iv) $-x, y - \frac{1}{2}, z - \frac{1}{2}$]. For (**IV**), the pyrrolidine ring exhibits two sets of C—H$\cdots\pi$ interactions to the phenyl [$Cg5\cdots H17A^{v} = 3.349$ Å and $Cg5\cdots H18A^{v} = 3.417$ Å; $Cg5$ is the centroid of ring C27–C32; symmetry code: (v) $1 - x, 2 - y, 1 - z$] and difluorinated rings [$Cg6\cdots H33A^{vi} = 4.179$ Å and $Cg6\cdots H33B^{vi} = 4.068$ Å; $Cg6$ is the centroid of ring C27–C32; symmetry code: (vi) $1 - x, 1 - y, 1 - z$. For **V**, there is a C—H$\cdots\pi$ interaction between a hydrogen atom of the piperidine ring and the pentafluorophenyl ring [$Cg7\cdots H4A^{vii} = 2.865$ Å; $Cg7$ is the centroid of ring C14–C19; symmetry code: (vii) (x) $-x, 1 - y, 1 - z$]. This C—H$\cdots\pi$ interaction is the shortest identified of the crystal structures presented. C—H$\cdots\pi$ interactions also exist between the two non-fluorinated phenyl rings of neighbouring molecules [$Cg8\cdots H12^{viii} = 3.550$ Å and $Cg8\cdots H11^{viii} = 3.748$ Å; $Cg8$ is the centroid of ring C8–C13; symmetry code: (viii) $-x, y, z$] and between piperidine ring hydrogen atoms and non-fluorinated phenyl rings [$Cg9\cdots H1B^{ix} = 3.220$ Å and $Cg9\cdots H3B^{ix} = 3.426$ Å; $Cg9$ is the centroid of ring C8–C13; symmetry code: (ix) $1 - x, 1 - y, -z$].

## 4. Database survey

A search of the Cambridge Structural Database (version 5.45, update in June 2024; Groom *et al.*; 2016) for phenidine derivatives resulted in four hits. All four hits are 2-methoxphenidine (**1b**) with a variety of solvates, some unknown (REBKOC; Jurásek *et al.*, 2022), and bromo- and chlorozincate ions (REBLOD and REBLIX; Jurásek *et al.*, 2022). Entry FIDHIN (Jurásek *et al.*, 2023) is the hydrochloride salt of the *R*-isomer of **1b** and as such is comparable to (**V**) due to the presence of a piperidine ring. Similar to (**V**), it has N—H$\cdots$Cl distances of 2.120 and 2.123 Å (two molecules in the asymmetric unit). The piperidine ring is the chair conformation, which is again directly comparable to (**V**). Entry REBKOC, mirrors that of FIDHIN except a chloroform solvent molecule is present in the asymmetric unit. It has an N—H—Cl distance of 2.209 Å and a $Cl_3C$—H$\cdots A$ distance of 2.387 Å; the presence of this solvent molecule has elongated the distance. The remaining two entries REBLOD and REBLIX (Jurásek *et al.*, 2022) both possess $ZnCl_2Br_4^{2-}$ and $ZnCl_2Br_4^{2-}$ ions in the asymmetric unit cell. Again, the piperidine ring is in the chair conformation for both REBLOD and REBLIX.

## 5. Synthesis and crystallization

### General method for diarylethylamine synthesis

All diphenidine derivatives and analogues were synthesized using an adaptation of the published method (Le Gall *et al.*, 2009). The following modifications were applied to the published method: To zinc dust (2.0 g, 30 mmol) suspended in acetonitrile (40 mL), was added benzyl bromide (0.4 mL, 3.4 mmol) and trifluoroacetic acid (0.2 mL). The resulting solution was stirred for 5 minutes and then benzyl bromide (3.0 mL, 25 mmol), the required amine (0.99 mL, 10 mmol) followed by the pre-requisite benzaldehyde (11 mmol), were introduced to the mixture, and the solution was stirred at room temperature for an additional 1 h. The resulting solution was poured into a saturated aqueous $NH_4Cl$ solution (150 mL) and extracted with dichloromethane (2 × 100 mL). The combined organic layers were dried ($MgSO_4$) and concentrated *in vacuo* to give a crude yellowish oil. The oil was then dissolved in diethyl ether (150 mL) and concentrated sulfuric acid (0.75 mL) was added dropwise to the vigorously stirred solution. After five minutes, the precipitated ammonium salt was filtered, washed with diethyl ether (2 × 50 mL) and air dried for 5–10 minutes. The ammonium salt was re-dissolved in aqueous sodium hydroxide (5% *w*/*v*, 150 mL) and then extracted with dichloromethane (2 × 100 mL). The combined organic fractions were again dried ($MgSO_4$) and concentrated *in vacuo* to give a yellow oil. The oil was dissolved in diethyl ether (200 mL), treated with hydrogen chloride (4 *M* in dioxane, 3.0 mL, 12 mmol) and left to stand for 5 minutes. The crystallized products were filtered and washed sequentially with the minimum amount of ice-cold acetone and if necessary an ice-cold mixture of ethyl acetate–diethyl ether (1:5) to afford the corresponding hydrochloride salts as colourless to off-white powders.

(**I**) afforded 0.40 g (15%) of a white powder. Colourless crystals suitable for X-ray diffraction were grown from EtOAc/diethyl ether. $^1$H NMR (400 MHz, $CD_2Cl_2$): $\delta$ 7.4–7.5 (*m*, 1 H), 7.1–7.2 (*m*, 5 H), 7.0 (*br. s*, 1 H), 6.9 (*br. s*, 1 H), 4.9 (*dd*, $J$ = 12.36, 2.75 Hz, 1 H), 4.0 (*dd*, $J$ = 12.82, 3.66 Hz, 1 H), 3.6–3.7 (*m*, 1 H), 2.8 (*br. s*, 3 H), 2.7 (*br. s*., 3 H). $^{13}$C{$^1$H} NMR (101 MHz, $CD_2Cl_2$): $\delta$ 162.5 (*dd*, $J$ = 251.12, 7.67 Hz, C-F), 135.8, 133.6, 133.5, 133.4, 129.3, 129.1, 127.7, 113.5, 112.7, 107.0, 61.8, 43.0, 38.4, 34.7. $^{19}$F NMR (56 MHz, $CD_2Cl_2$): $\delta$ −111.21

**Table 6**
Experimental details.

| | (**I**) | (**II**) | (**III**) | (**IV**) | (**v**) |
|---|---|---|---|---|---|
| Crystal data | | | | | |
| Chemical formula | $C_{16}H_{18}F_2N^+\cdot Cl^-$ | $2C_{16}H_{18}F_2N^+\cdot 2Cl^-\cdot CH_2Cl_2$ | $C_{18}H_{22}F_2N^+\cdot Cl^-$ | $C_{18}H_{20}F_2N^+\cdot Cl^-$ | $C_{19}H_{19}F_5N^+\cdot Cl^-$ |
| $M_r$ | 297.76 | 680.45 | 325.81 | 323.80 | 391.80 |
| Crystal system, space group | Monoclinic, $P2_1/c$ | Monoclinic, $I2/a$ | Monoclinic, $P2_1/c$ | Triclinic, $P\overline{1}$ | Monoclinic, $P2_1/c$ |
| Temperature (K) | 123 | 123 | 123 | 123 | 123 |
| $a$, $b$, $c$ (Å) | 7.9474 (3), 12.7652 (5), 15.3998 (7) | 22.9963 (14), 7.8729 (5), 19.033 (1) | 11.3115 (6), 10.5400 (5), 14.8039 (7) | 8.1365 (4), 12.7421 (10), 16.0451 (8) | 9.3155 (5), 22.2529 (13), 8.2699 (3) |
| $\alpha$, $\beta$, $\gamma$ (°) | 90, 99.368 (4), 90 | 90, 92.130 (5), 90 | 90, 105.044 (5), 90 | 88.059 (5), 82.349 (4), 86.140 (5) | 90, 90.165 (5), 90 |
| $V$ (Å$^3$) | 1541.48 (11) | 3443.5 (4) | 1704.48 (15) | 1644.42 (17) | 1714.32 (15) |
| $Z$ | 4 | 4 | 4 | 4 | 4 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ | Mo $K\alpha$ | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.26 | 0.39 | 0.24 | 0.25 | 0.28 |
| Crystal size (mm) | 0.5 × 0.4 × 0.2 | 0.3 × 0.2 × 0.1 | 0.4 × 0.2 × 0.1 | 0.5 × 0.4 × 0.3 | 0.2 × 0.1 × 0.05 |
| Data collection | | | | | |
| Diffractometer | Oxford Diffraction Xcalibur | Oxford Diffraction Xcalibur | Oxford Diffraction Xcalibur | Oxford Diffraction Xcalibur | Oxford Diffraction Xcalibur |
| Absorption correction | Analytical (*CrysAlis PRO*; Agilent 2014) | Analytical (*SADABS*; Krause *et al.*, 2015) | Analytical (*SADABS*; Krause *et al.*, 2015) | Analytical (*SADABS*; Krause *et al.*, 2015) | Analytical (*SADABS*; Krause *et al.*, 2015) |
| $T_{min}$, $T_{max}$ | 0.884, 0.950 | 0.911, 0.962 | 0.944, 0.976 | 0.888, 0.928 | 0.967, 0.986 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 6424, 2715, 2305 | 12738, 3034, 2813 | 7223, 3008, 2343 | 13277, 5775, 4892 | 5523, 2896, 2117 |
| $R_{int}$ | 0.027 | 0.021 | 0.036 | 0.032 | 0.049 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.595 | 0.595 | 0.595 | 0.595 | 0.595 |
| Refinement | | | | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.040, 0.088, 1.06 | 0.031, 0.077, 1.07 | 0.049, 0.095, 1.06 | 0.090, 0.206, 1.19 | 0.056, 0.123, 1.01 |
| No. of reflections | 2715 | 3034 | 3008 | 5775 | 2896 |
| No. of parameters | 186 | 224 | 210 | 403 | 239 |
| No. of restraints | 1 | 0 | 0 | 2 | 0 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.20, −0.26 | 0.25, −0.22 | 0.22, −0.25 | 0.59, −0.34 | 0.30, −0.25 |

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *X-SEED* (Barbour, 2020).

(*br. s*, 2F). FT-IR (ATR, cm$^{-1}$) 2306[RM1], 1624 (C═O), 1457 (C═C). M.p. = 385–387 K.

(**II**) afforded 2.24 g (64%) of a white powder. Colourless crystals suitable for X-ray diffraction were grown from DCM/diethyl ether. $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 10.26 (*br. s*, 1H, NH), 9.18 (*br. s*, 1 H, NH), 7.02–7.20 (*m*, 7H, Ar-H), 7.50 (*dd*, 1H, $J$ = 11.2, 4.2 Hz, Ar-H), 4.75 (*m*, 1H, $NHCHCH_2$), 3.65 (*dd*, 1 H, $J$ = 12.8, 4.8 Hz, $NHCHCH_2$), 3.18 (*m*, 1 H, $NHCHCH_2$), 2.95 (*m*, 2 H, $NHCH_2CH_3$), 1.28 (*t*, 3 H, $J$ = 7.0 Hz, $NHCH_2CH_3$). $^{19}$F{$^1$H} NMR (400 MHz, DMSO-$d_6$) $\delta$ −113.68 (*s*, 2F); IR (ATR, cm$^{-1}$): 2944 (C—H), 2670 (C—H), 1475 (C═C), 1202 (C—F). M.p. = 478 K.

(**III**) afforded 1.93 g (67%) of a white powder. Colourless crystals suitable for X-ray diffraction were grown from $CHCl_3$/diethyl ether. $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 10.3 (*d*, $J$ = 9.16 Hz, 1 H), 8.9 (*dd*, $J$ = 11.91, 5.04 Hz, 1 H), 7.4–7.5 (*m*, 1 H), 7.1–7.3 (*m*, 4 H), 7.0 (*dd*, $J$ = 7.56, 2.06 Hz, 2 H), 6.9 (*s*, 1 H), 4.7 (*dd*, $J$ = 11.68, 4.35 Hz, 1 H), 3.7 (*dd*, $J$ = 12.82, 4.12 Hz, 1 H), 3.3–3.4 (*m*, 1 H), 1.4 (*s*, 9 H). $^{13}$C{$^1$H} NMR (101 MHz, $(CD_3)_2SO$) $\delta$ 135.4, 132.2, 132.1, 132.0, 128.7, 128.2, 126.9, 112.5, 112.3, 111.8, 111.6, 111.4, 58.8, 49.4, 38.1, 25.3. $^{19}$F NMR (56 MHz, $(CD_3)_2SO$) $\delta$ −109.94 (*br. s*, 1F), −116.79 (*br. s*, 1F). FT-IR (ATR, cm$^{-1}$) 2612, 1625, 1565, 1467; M.p. = 535–538 K.

(**IV**) afforded 2.22 g (77%) of a white powder. Colourless crystals suitable for X-ray diffraction were grown from DCM/diethyl ether. $^1$H NMR (400 MHz, $CD_2Cl_2$) $\delta$ 7.4 (*tt*, $J$ = 8.47, 6.41 Hz, 1 H), 7.1–7.2 (*m*, 5 H), 7.0 (*br. s*., 1 H), 6.8 (*br. s*., 1 H), 4.9 (*dd*, $J$ = 12.14, 3.89 Hz, 1 H), 3.9 (*dd*, $J$ = 13.28, 4.58 Hz, 2 H), 3.6 (*t*, $J$ = 12.59 Hz, 2 H), 2.9 (*br. s*., 1 H), 2.8 (*br. s*., 1 H), 2.2 (*br. s*., 5 H), 1.9 (*br. s*., 2 H). $^{13}$C{$^1$H} NMR (101 MHz, $CD_2Cl_2$) $\delta$ 161.6 (*dd*, $J$ = 250.16, 7.67 Hz) C—F, 136.0, 133.3, 133.2, 133.1, 129.2, 129.1, 127.6, 113.4, 112.5, 109.0, 108.8, 108.7, 59.9, 50.6, 35.9, 23.4, 23.2. $^{19}$F NMR (56 MHz, $CD_2Cl_2$) $\delta$ −108.10 (*br. s*, 1F), −114.18 (*br. s*, 1F). FT-IR (ATR, cm$^{-1}$) 2352, 1623, 1460. M.p. = 486–488 K.

(**V**) afforded 1.55 g (52%) of a white powder. Colourless crystals suitable for X-ray diffraction were grown from $CHCl_3$/

diethyl ether. $^1$H NMR (400 MHz, $CD_2Cl_2$) $\delta$ 7.11–7.28 (*m*, 5 H), 4.90 (*d*, *J* = 12.36 Hz, 1 H), 4.23 (*dd*, *J* = 12.82, 3.66 Hz, 1 H), 3.80 (*d*, *J* = 11.45 Hz, 1 H), 3.44–3.55 (*m*, 2 H), 2.57–2.70 (*m*, 1 H), 2.48 (*d*, *J* = 9.62 Hz, 1 H), 2.24–2.38 (*m*, 1 H), 1.91 (*t*, *J* = 14.88 Hz, 2 H), 1.80 (*d*, *J* = 13.74 Hz, 1 H), 1.22–1.37 (*m*, 1 H). $^{13}$C{$^1$H} NMR (101 MHz, $CD_2Cl_2$) $\delta$ 120.9, 115.0, 114.6, 113.5, 91.0, 48.2, 34.5, 19.2, 9.0, 8.9, 8.1. $^{19}$F NMR (56 MHz, $CD_2Cl_2$) $\delta$ −134.86, −139.28, −152.81 (*t*, *J* = 22.4 Hz), −162.69. FT-IR (ATR, cm$^{-1}$) 2309, 1503, 1459. M.p. = 502–504 K.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 6. Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included as riding contributions in idealized positions with isotropic displacement parameters $U_{iso}$(H) = 1.2$U_{eq}$(C) (1.5 for methyl groups). All structures were solved by direct methods. For (**II**) the terminal carbon of the ethyl group (C15, C15*A*), is disordered over two positions [0.707 (5):0.293 (5) occupancy]. All non H-atoms were refined anisotropically. The H atoms were placed in calculated positions, except for H1*N*1 (**I**), H1*A* and H1*B* (**II**), H1′ and H2 (**IV**) and H2 (**V**), which were all found. For (**V**), a DFIX instruction was applied to N1—H1′ and N2—H2 (fixed at 0.98 Å).

## Acknowledgements

We thank Manchester knowledge and drug exchange (MANDRAKE) for the preparation of the reference compounds, which were produced under Home Office licence (in accordance with Manchester Metropolitan University's Home Office license, Ref. No. 423023) requirements and agreed procedures.

## References

Agilent (2014). *CrysAlis PRO.*, Agilent Technologies, Yarnton, England.

Barbour, L. J. (2020). *J. Appl. Cryst.* **53**, 1141–1146.

Elliott, S. P., Brandt, S. D., Wallach, J., Morris, H. & Kavanagh, P. V. (2015). *J. Anal. Toxicol.* **39**, 287–293.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Hasegawa, K., Wurita, A., Minakata, K., Gonmori, K., Nozawa, H., Yamagishi, I., Watanabe, K. & Suzuki, O. (2015). *Forensic Toxicol.* **33**, 380-387, doi: 10.1007/s11419-015-0272-y.

Helander, A., Beck, O. & Bäckberg, M. (2015). *Clin. Toxicol.* **53**, 446–453.

Hofer, K. E., Degrandi, C., Müller, D. M., Zürrer-Härdi, U., Wahl, S., Rauber-Lüthy, C. & Ceschi, A. (2014). *Clin. Toxicol.* **52**, 1288–1291.

Jurásek, B., Fagan, P., Dolenský, B., Paškanová, N., Dobšíková, K., Raich, I., Jurok, R., Setnička, V., Kohout, M., Čejka, J. & Kuchař, M. (2023). *New J. Chem.* **47**, 4543–4551.

Jurásek, B., Rimpelová, S., Babor, M., Čejka, J., Bartůněk, V. & Kuchař, M. (2022). *Int. J. Mol. Sci.* **23**, 2083.

King, L. A. (2013). *Novel Psychoactive Substances*, edited by P. I. Dargan and D. M. Wood, pp. 3-27. Academic Press, Boston: Academic Press. doi: 10.1016/B978-0-12-415816-0.00001-8.

Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.

Kudo, K., Usumoto, Y., Kikura-Hanajiri, R., Sameshima, N., Tsuji, A. & Ikeda, N. (2015). *Leg. Med.* **17**, 421–426.

Le Gall, E., Haurena, C., Sengmany, S., Martens, T. & Troupel, M. (2009). *J. Org. Chem.* **74**, 7970–7973.

McLaughlin, G., Morris, N., Kavanagh, P. V., Power, J. D., O'Brien, J., Talbot, B., Elliott, S. P., Wallach, J., Hoang, K., Morris, H. & Brandt, S. D. (2016). *Drug Test. Anal.* **8**, 98–109.

Minakata, K., Yamagishi, I., Nozawa, H., Hasegawa, K., Gonmori, K., Suzuki, M., Wurita, A., Suzuki, O. & Watanabe, K. (2016). *Forensic Toxicol.* **34**, 151–157.

Morris, H. & Wallach, J. (2014). *Drug Test. Anal.* **6**, 614–632.

Odoardi, S., Romolo, F. S. & Strano-Rossi, S. (2016). *Forensic Sci. Int.* **265**, 116–120.

Reuter, P. & Pardo, B. (2017). *Addiction*, **112**, 25–31.

Sahai, M. A., Davidson, C. N., Dutta, C. & Opacka-Juffry, J. (2018). *Brain Sci.* pp. 8.

Shafi A., Berry, A. J., Sumnall, H., Wood, D. M. & Tracy, D. K. (2020). *Ther. Adv. Psychopharmacol*, **10**, 2045125320967197. https://doi.org/10.1177/2045125320967197.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Strano Rossi, S., Odoardi, S., Gregori, A., Peluso, G., Ripani, L., Ortar, G., Serpelloni, G. & Romolo, F. S. (2014). *Rapid Commun. Mass Spectrom.* **28**, 1904–1916.

Tettey, J. N. A., Crean, C., Ifeagwu, S. C. & Raithelhuber, M. (2018). *New Psychoactive Substances: Pharmacology, Clinical, Forensic and Analytical Toxicology*, edited by H. H. Maurer & S. D. Brandt. pp. 51–67. Cham: Springer International Publishing. https://doi.org/10.1007/164_2018_127

UNODC (2021). Resolution adopted by the Commission on 14th April 2021 – Inclusion of diphenidine in Schedule II of the Convention on Psychotropic Substances of 1971. Decision 64/6., 2021, https://www.unodcorg/documents/commissions/CND/Drug_Resolutions/2020-2029/2021/decision_64_6.pdf

UNODC (2024). The Challenge of New Psychoactive Substances – A Technical Update. A Report from the Global *SMART* programme (Laboratory and Scientific Section), https://www.unodc.org/unodc/en/scientists/the-challenge-of-new-psychoactive-substances.html

Wallach, J., Kang, H., Colestock, T., Morris, H., Bortolotto, Z. A., Collingridge, G. L., Lodge, D., Halberstadt, A. L., Brandt, S. D. & Adejare, A. (2016). *PLoS One*, **11**, e0157021.

Wallach, J., Kavanagh, P. V., McLaughlin, G., Morris, N., Power, J. D., Elliott, S. P., Mercier, M. S., Lodge, D., Morris, H., Dempster, N. M. & Brandt, S. D. (2015). *Drug Test. Anal.* **7**, 358–367.

Wurita, A., Hasegawa, K., Minakata, K., Watanabe, K. & Suzuki, O. (2014). *Forensic Toxicol.*. **32**, 331–337.

# supporting information

*Acta Cryst.* (2025). E**81** [https://doi.org/10.1107/S2056989025001288]

## Synthesis and crystal structures of five fluorinated diphenidine derivatives

**Ryan E. Mewis, Matthew C. Hulme, Jack Marron, Stuart K. Langley, Oliver B. Sutcliffe and Sophie L. Benjamin**

### Computing details

#### 1-[1-(2,3,4,5,6-Pentafluorophenyl)-2-phenylethyl]piperidin-1-ium chloride (V)

*Crystal data*

$C_{19}H_{19}F_5N^+\cdot Cl^-$
$M_r$ = 391.80
Monoclinic, $P2_1/c$
$a$ = 9.3155 (5) Å
$b$ = 22.2529 (13) Å
$c$ = 8.2699 (3) Å
$\beta$ = 90.165 (5)°
$V$ = 1714.32 (15) Å$^3$
$Z$ = 4
$F(000)$ = 808
$D_x$ = 1.518 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9843 reflections
$\theta$ = 3.0–26.5°
$\mu$ = 0.28 mm$^{-1}$
$T$ = 123 K
Block, colourless
0.2 × 0.1 × 0.05 mm

*Data collection*

Oxford Diffraction Xcalibur diffractometer
Radiation source: fine-focus sealed tube
scans in phi and $\omega$
Absorption correction: analytical (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.967, $T_{max}$ = 0.986
5523 measured reflections
2896 independent reflections
2117 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.049
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 3.1°
$h$ = −10→11
$k$ = −18→26
$l$ = −8→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.123
$S$ = 1.01
2896 reflections
239 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.3121P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.24269 (9) | 0.51019 (4) | 0.68922 (9) | 0.0315 (2) |
| F1 | 0.07331 (19) | 0.42667 (8) | 0.0555 (2) | 0.0327 (5) |
| F5 | 0.31205 (19) | 0.36912 (8) | 0.53597 (19) | 0.0307 (5) |
| F4 | 0.1190 (2) | 0.28212 (8) | 0.5801 (2) | 0.0404 (5) |
| F2 | −0.1166 (2) | 0.33964 (10) | 0.1010 (2) | 0.0474 (6) |
| F3 | −0.0977 (2) | 0.26613 (9) | 0.3625 (2) | 0.0492 (6) |
| C14 | 0.1989 (3) | 0.40279 (14) | 0.2969 (3) | 0.0218 (7) |
| C18 | 0.1081 (4) | 0.31807 (14) | 0.4497 (4) | 0.0281 (8) |
| C6 | 0.3167 (3) | 0.44932 (13) | 0.2723 (3) | 0.0215 (7) |
| H6 | 0.395538 | 0.438117 | 0.348882 | 0.026* |
| C5 | 0.1334 (3) | 0.53469 (14) | 0.2445 (4) | 0.0256 (8) |
| H5A | 0.055024 | 0.505280 | 0.260780 | 0.031* |
| H5B | 0.148162 | 0.539641 | 0.126760 | 0.031* |
| C4 | 0.0922 (3) | 0.59416 (15) | 0.3182 (4) | 0.0308 (8) |
| H4A | 0.070518 | 0.588238 | 0.434277 | 0.037* |
| H4B | 0.003969 | 0.609188 | 0.264765 | 0.037* |
| C8 | 0.4205 (3) | 0.38395 (14) | 0.0529 (3) | 0.0246 (8) |
| C1 | 0.3883 (3) | 0.55697 (14) | 0.3002 (4) | 0.0267 (8) |
| H1A | 0.477032 | 0.541589 | 0.351631 | 0.032* |
| H1B | 0.407412 | 0.562601 | 0.183476 | 0.032* |
| C19 | 0.2066 (3) | 0.36364 (14) | 0.4273 (3) | 0.0229 (7) |
| C10 | 0.5654 (4) | 0.29493 (15) | 0.0849 (4) | 0.0320 (8) |
| H10 | 0.641115 | 0.274303 | 0.138504 | 0.038* |
| C16 | −0.0118 (4) | 0.34743 (16) | 0.2098 (4) | 0.0310 (8) |
| C2 | 0.3486 (3) | 0.61639 (15) | 0.3748 (4) | 0.0286 (8) |
| H2A | 0.427250 | 0.645549 | 0.357103 | 0.034* |
| H2B | 0.336184 | 0.611273 | 0.492819 | 0.034* |
| N1 | 0.2689 (3) | 0.51174 (11) | 0.3216 (3) | 0.0208 (6) |
| C7 | 0.3833 (3) | 0.44745 (14) | 0.1024 (3) | 0.0258 (8) |
| H7A | 0.314639 | 0.464662 | 0.023408 | 0.031* |
| H7B | 0.471239 | 0.472407 | 0.101079 | 0.031* |
| C15 | 0.0873 (3) | 0.39271 (14) | 0.1886 (3) | 0.0242 (8) |
| C17 | −0.0007 (4) | 0.30990 (15) | 0.3412 (4) | 0.0323 (9) |
| C12 | 0.3791 (4) | 0.29713 (16) | −0.1148 (4) | 0.0327 (9) |
| H12 | 0.327115 | 0.277949 | −0.199197 | 0.039* |
| C3 | 0.2103 (4) | 0.64091 (15) | 0.3016 (4) | 0.0317 (8) |
| H3A | 0.182086 | 0.678299 | 0.358033 | 0.038* |
| H3B | 0.225342 | 0.650474 | 0.185948 | 0.038* |
| C11 | 0.4883 (4) | 0.26676 (15) | −0.0364 (4) | 0.0315 (9) |
| H11 | 0.510387 | 0.226548 | −0.065800 | 0.038* |
| C9 | 0.5318 (3) | 0.35331 (15) | 0.1277 (4) | 0.0279 (8) |
| H9 | 0.586002 | 0.372763 | 0.209846 | 0.033* |
| C13 | 0.3452 (4) | 0.35548 (15) | −0.0706 (4) | 0.0303 (8) |
| H13 | 0.270025 | 0.376141 | −0.125034 | 0.036* |
| H1 | 0.261 (4) | 0.5105 (14) | 0.425 (4) | 0.038 (10)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0238 (5) | 0.0421 (5) | 0.0286 (4) | 0.0016 (4) | 0.0007 (4) | 0.0024 (4) |
| F1 | 0.0234 (11) | 0.0457 (12) | 0.0289 (9) | −0.0008 (10) | −0.0060 (8) | 0.0025 (8) |
| F5 | 0.0222 (11) | 0.0387 (12) | 0.0313 (9) | −0.0010 (9) | −0.0049 (8) | 0.0063 (8) |
| F4 | 0.0404 (13) | 0.0362 (12) | 0.0446 (11) | −0.0041 (10) | 0.0081 (10) | 0.0109 (9) |
| F2 | 0.0290 (12) | 0.0668 (15) | 0.0464 (11) | −0.0172 (11) | −0.0084 (10) | −0.0126 (10) |
| F3 | 0.0418 (14) | 0.0461 (13) | 0.0596 (13) | −0.0280 (12) | 0.0088 (11) | −0.0072 (10) |
| C14 | 0.0128 (17) | 0.0257 (17) | 0.0271 (16) | 0.0031 (15) | 0.0054 (14) | −0.0046 (13) |
| C18 | 0.030 (2) | 0.0277 (19) | 0.0271 (17) | 0.0013 (17) | 0.0061 (16) | 0.0021 (14) |
| C6 | 0.0123 (17) | 0.0248 (18) | 0.0273 (16) | 0.0005 (15) | 0.0013 (14) | −0.0005 (13) |
| C5 | 0.0143 (18) | 0.034 (2) | 0.0285 (16) | 0.0029 (16) | −0.0038 (14) | −0.0015 (14) |
| C4 | 0.0173 (19) | 0.039 (2) | 0.0362 (18) | 0.0073 (17) | −0.0046 (16) | −0.0018 (15) |
| C8 | 0.0188 (18) | 0.0282 (18) | 0.0268 (16) | −0.0001 (16) | 0.0079 (15) | 0.0034 (14) |
| C1 | 0.0140 (18) | 0.033 (2) | 0.0329 (18) | −0.0044 (16) | 0.0020 (15) | 0.0030 (14) |
| C19 | 0.0171 (18) | 0.0295 (19) | 0.0222 (16) | 0.0020 (16) | 0.0003 (14) | −0.0027 (14) |
| C10 | 0.027 (2) | 0.037 (2) | 0.0325 (18) | 0.0074 (18) | 0.0072 (16) | 0.0058 (16) |
| C16 | 0.0186 (19) | 0.044 (2) | 0.0302 (18) | −0.0056 (18) | 0.0010 (16) | −0.0122 (16) |
| C2 | 0.0187 (19) | 0.0299 (19) | 0.0373 (18) | −0.0037 (16) | 0.0022 (15) | 0.0005 (15) |
| N1 | 0.0128 (15) | 0.0278 (15) | 0.0220 (14) | −0.0014 (13) | −0.0002 (12) | 0.0023 (12) |
| C7 | 0.0196 (19) | 0.0291 (19) | 0.0288 (17) | 0.0010 (16) | 0.0056 (15) | 0.0031 (14) |
| C15 | 0.0213 (19) | 0.0306 (19) | 0.0207 (16) | 0.0004 (16) | 0.0025 (14) | −0.0021 (14) |
| C17 | 0.025 (2) | 0.0299 (19) | 0.042 (2) | −0.0084 (17) | 0.0106 (17) | −0.0100 (16) |
| C12 | 0.025 (2) | 0.043 (2) | 0.0299 (18) | −0.0082 (19) | 0.0031 (16) | −0.0075 (16) |
| C3 | 0.030 (2) | 0.0292 (19) | 0.0355 (18) | 0.0026 (17) | 0.0013 (17) | 0.0020 (15) |
| C11 | 0.035 (2) | 0.0279 (19) | 0.0321 (19) | 0.0015 (18) | 0.0110 (17) | −0.0003 (15) |
| C9 | 0.0194 (19) | 0.035 (2) | 0.0289 (17) | −0.0028 (16) | 0.0026 (15) | −0.0007 (14) |
| C13 | 0.0197 (19) | 0.043 (2) | 0.0284 (17) | 0.0030 (17) | 0.0048 (15) | 0.0011 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C15 | 1.342 (3) | C1—C2 | 1.506 (4) |
| F5—C19 | 1.335 (3) | C1—N1 | 1.511 (4) |
| F4—C18 | 1.347 (3) | C1—H1A | 0.9900 |
| F2—C16 | 1.338 (4) | C1—H1B | 0.9900 |
| F3—C17 | 1.340 (4) | C10—C9 | 1.383 (5) |
| C14—C15 | 1.388 (4) | C10—C11 | 1.382 (5) |
| C14—C19 | 1.388 (4) | C10—H10 | 0.9500 |
| C14—C6 | 1.523 (4) | C16—C17 | 1.374 (5) |
| C18—C17 | 1.364 (5) | C16—C15 | 1.378 (4) |
| C18—C19 | 1.380 (4) | C2—C3 | 1.523 (4) |
| C6—N1 | 1.515 (4) | C2—H2A | 0.9900 |
| C6—C7 | 1.538 (4) | C2—H2B | 0.9900 |
| C6—H6 | 1.0000 | N1—H1 | 0.85 (3) |
| C5—N1 | 1.502 (4) | C7—H7A | 0.9900 |
| C5—C4 | 1.507 (4) | C7—H7B | 0.9900 |
| C5—H5A | 0.9900 | C12—C11 | 1.381 (5) |

| | | | |
|---|---|---|---|
| C5—H5B | 0.9900 | C12—C13 | 1.386 (5) |
| C4—C3 | 1.521 (4) | C12—H12 | 0.9500 |
| C4—H4A | 0.9900 | C3—H3A | 0.9900 |
| C4—H4B | 0.9900 | C3—H3B | 0.9900 |
| C8—C9 | 1.385 (4) | C11—H11 | 0.9500 |
| C8—C13 | 1.390 (4) | C9—H9 | 0.9500 |
| C8—C7 | 1.512 (4) | C13—H13 | 0.9500 |

| | | | |
|---|---|---|---|
| C15—C14—C19 | 115.9 (3) | C1—C2—C3 | 111.1 (3) |
| C15—C14—C6 | 124.2 (3) | C1—C2—H2A | 109.4 |
| C19—C14—C6 | 119.6 (3) | C3—C2—H2A | 109.4 |
| F4—C18—C17 | 120.1 (3) | C1—C2—H2B | 109.4 |
| F4—C18—C19 | 119.7 (3) | C3—C2—H2B | 109.4 |
| C17—C18—C19 | 120.2 (3) | H2A—C2—H2B | 108.0 |
| N1—C6—C14 | 112.0 (2) | C5—N1—C1 | 110.0 (2) |
| N1—C6—C7 | 113.0 (2) | C5—N1—C6 | 116.4 (2) |
| C14—C6—C7 | 113.3 (3) | C1—N1—C6 | 111.2 (2) |
| N1—C6—H6 | 105.9 | C5—N1—H1 | 111 (2) |
| C14—C6—H6 | 105.9 | C1—N1—H1 | 102 (2) |
| C7—C6—H6 | 105.9 | C6—N1—H1 | 105 (2) |
| N1—C5—C4 | 110.0 (3) | C8—C7—C6 | 111.5 (2) |
| N1—C5—H5A | 109.7 | C8—C7—H7A | 109.3 |
| C4—C5—H5A | 109.7 | C6—C7—H7A | 109.3 |
| N1—C5—H5B | 109.7 | C8—C7—H7B | 109.3 |
| C4—C5—H5B | 109.7 | C6—C7—H7B | 109.3 |
| H5A—C5—H5B | 108.2 | H7A—C7—H7B | 108.0 |
| C5—C4—C3 | 112.2 (3) | F1—C15—C16 | 116.9 (3) |
| C5—C4—H4A | 109.2 | F1—C15—C14 | 120.6 (3) |
| C3—C4—H4A | 109.2 | C16—C15—C14 | 122.5 (3) |
| C5—C4—H4B | 109.2 | F3—C17—C18 | 120.7 (3) |
| C3—C4—H4B | 109.2 | F3—C17—C16 | 119.8 (3) |
| H4A—C4—H4B | 107.9 | C18—C17—C16 | 119.5 (3) |
| C9—C8—C13 | 118.6 (3) | C11—C12—C13 | 120.2 (3) |
| C9—C8—C7 | 120.7 (3) | C11—C12—H12 | 119.9 |
| C13—C8—C7 | 120.6 (3) | C13—C12—H12 | 119.9 |
| C2—C1—N1 | 110.8 (2) | C4—C3—C2 | 109.3 (3) |
| C2—C1—H1A | 109.5 | C4—C3—H3A | 109.8 |
| N1—C1—H1A | 109.5 | C2—C3—H3A | 109.8 |
| C2—C1—H1B | 109.5 | C4—C3—H3B | 109.8 |
| N1—C1—H1B | 109.5 | C2—C3—H3B | 109.8 |
| H1A—C1—H1B | 108.1 | H3A—C3—H3B | 108.3 |
| F5—C19—C18 | 117.7 (3) | C12—C11—C10 | 119.9 (3) |
| F5—C19—C14 | 120.2 (3) | C12—C11—H11 | 120.0 |
| C18—C19—C14 | 122.1 (3) | C10—C11—H11 | 120.0 |
| C9—C10—C11 | 119.6 (3) | C10—C9—C8 | 121.2 (3) |
| C9—C10—H10 | 120.2 | C10—C9—H9 | 119.4 |
| C11—C10—H10 | 120.2 | C8—C9—H9 | 119.4 |
| F2—C16—C17 | 120.4 (3) | C12—C13—C8 | 120.4 (3) |

| | | | |
|---|---|---|---|
| F2—C16—C15 | 119.8 (3) | C12—C13—H13 | 119.8 |
| C17—C16—C15 | 119.7 (3) | C8—C13—H13 | 119.8 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···Cl1 | 0.85 (3) | 2.20 (3) | 3.051 (3) | 178 (3) |

**[1-(2,6-Difluorophenyl)-2-phenylethyl]dimethylazanium chloride (I)**

*Crystal data*

$C_{16}H_{18}F_2N^+\cdot Cl^-$
$M_r$ = 297.76
Monoclinic, $P2_1/c$
$a$ = 7.9474 (3) Å
$b$ = 12.7652 (5) Å
$c$ = 15.3998 (7) Å
$\beta$ = 99.368 (4)°
$V$ = 1541.48 (11) Å$^3$
$Z$ = 4
$F(000)$ = 624
$D_x$ = 1.283 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 8632 reflections
$\theta$ = 3.0–26.6°
$\mu$ = 0.26 mm$^{-1}$
$T$ = 123 K
Block, colourless
0.5 × 0.4 × 0.2 mm

*Data collection*

Oxford Diffraction Xcalibur diffractometer
Radiation source: fine-focus sealed tube
scans in phi and $\omega$
Absorption correction: analytical (CrysAlisPro; Agilent 2014)
$T_{min}$ = 0.884, $T_{max}$ = 0.950
6424 measured reflections
2715 independent reflections
2305 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 3.1°
$h$ = −9→9
$k$ = −14→15
$l$ = −18→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.088
$S$ = 1.06
2715 reflections
186 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0281P)^2 + 0.7459P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C8 | 0.8942 (2) | 0.41635 (15) | 0.29692 (12) | 0.0230 (4) |
| H8A | 0.997147 | 0.441707 | 0.336303 | 0.028* |
| H8B | 0.918077 | 0.345200 | 0.276392 | 0.028* |

| | | | | |
|---|---|---|---|---|
| C3 | 0.4368 (2) | 0.45234 (14) | 0.28669 (13) | 0.0235 (4) |
| C7 | 0.5501 (2) | 0.30531 (14) | 0.22760 (13) | 0.0234 (4) |
| C2 | 0.5752 (2) | 0.38564 (13) | 0.28907 (12) | 0.0188 (4) |
| C14 | 0.8167 (2) | 0.45121 (15) | 0.13360 (13) | 0.0286 (5) |
| H14 | 0.825573 | 0.378177 | 0.123370 | 0.034* |
| C6 | 0.4032 (2) | 0.29047 (16) | 0.16854 (13) | 0.0295 (5) |
| H6 | 0.392555 | 0.233545 | 0.128282 | 0.035* |
| C10 | 0.8393 (2) | 0.59657 (15) | 0.23204 (13) | 0.0251 (4) |
| H10 | 0.863656 | 0.624451 | 0.289921 | 0.030* |
| C5 | 0.2715 (2) | 0.36051 (17) | 0.16928 (14) | 0.0346 (5) |
| H5 | 0.168621 | 0.351866 | 0.128876 | 0.042* |
| C12 | 0.7556 (3) | 0.62399 (16) | 0.07667 (14) | 0.0357 (5) |
| H12 | 0.722285 | 0.669788 | 0.028305 | 0.043* |
| C11 | 0.7913 (3) | 0.66314 (15) | 0.16121 (14) | 0.0303 (5) |
| H11 | 0.782997 | 0.736312 | 0.170946 | 0.036* |
| C4 | 0.2871 (2) | 0.44303 (17) | 0.22793 (14) | 0.0318 (5) |
| H4 | 0.197122 | 0.492047 | 0.227789 | 0.038* |
| C13 | 0.7688 (3) | 0.51778 (16) | 0.06310 (14) | 0.0384 (5) |
| H13 | 0.744851 | 0.490217 | 0.005099 | 0.046* |
| F1 | 0.45166 (14) | 0.53325 (8) | 0.34416 (8) | 0.0334 (3) |
| F2 | 0.68097 (14) | 0.23779 (8) | 0.22353 (8) | 0.0329 (3) |
| C1 | 0.7428 (2) | 0.41269 (14) | 0.34703 (11) | 0.0186 (4) |
| H1 | 0.729073 | 0.485786 | 0.368148 | 0.022* |
| C9 | 0.8521 (2) | 0.48917 (14) | 0.21896 (12) | 0.0204 (4) |
| Cl1 | 1.14563 (7) | 0.36912 (4) | 0.51711 (4) | 0.03725 (17) |
| N1 | 0.7819 (2) | 0.34594 (12) | 0.42918 (10) | 0.0248 (4) |
| C15 | 0.6669 (3) | 0.37192 (17) | 0.49322 (14) | 0.0363 (5) |
| H15A | 0.703925 | 0.333884 | 0.548299 | 0.055* |
| H15B | 0.549887 | 0.351557 | 0.468782 | 0.055* |
| H15C | 0.671182 | 0.447444 | 0.504801 | 0.055* |
| C16 | 0.7872 (3) | 0.23087 (15) | 0.41449 (14) | 0.0336 (5) |
| H16A | 0.672229 | 0.205541 | 0.391314 | 0.050* |
| H16B | 0.829292 | 0.195665 | 0.470357 | 0.050* |
| H16C | 0.863511 | 0.215610 | 0.372127 | 0.050* |
| H1N1 | 0.893 (3) | 0.3638 (16) | 0.4538 (15) | 0.039 (6)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C8 | 0.0196 (9) | 0.0288 (10) | 0.0196 (10) | −0.0001 (8) | −0.0003 (8) | −0.0001 (8) |
| C3 | 0.0274 (10) | 0.0239 (10) | 0.0210 (10) | −0.0010 (8) | 0.0090 (8) | 0.0012 (8) |
| C7 | 0.0241 (10) | 0.0230 (9) | 0.0234 (10) | −0.0028 (8) | 0.0048 (8) | −0.0003 (8) |
| C2 | 0.0198 (9) | 0.0210 (9) | 0.0156 (9) | −0.0032 (7) | 0.0033 (7) | 0.0019 (8) |
| C14 | 0.0388 (12) | 0.0233 (10) | 0.0241 (11) | −0.0015 (9) | 0.0064 (9) | −0.0025 (9) |
| C6 | 0.0299 (11) | 0.0352 (11) | 0.0219 (11) | −0.0117 (9) | −0.0004 (9) | −0.0028 (9) |
| C10 | 0.0257 (10) | 0.0296 (10) | 0.0205 (11) | −0.0053 (8) | 0.0049 (8) | −0.0032 (9) |
| C5 | 0.0228 (10) | 0.0516 (14) | 0.0274 (12) | −0.0124 (10) | −0.0020 (9) | 0.0082 (11) |
| C12 | 0.0502 (13) | 0.0307 (11) | 0.0262 (12) | −0.0007 (10) | 0.0061 (10) | 0.0109 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C11 | 0.0386 (12) | 0.0222 (10) | 0.0317 (12) | −0.0025 (9) | 0.0105 (9) | −0.0004 (9) |
| C4 | 0.0192 (10) | 0.0413 (12) | 0.0353 (13) | 0.0027 (9) | 0.0052 (9) | 0.0120 (10) |
| C13 | 0.0638 (15) | 0.0343 (12) | 0.0167 (11) | −0.0026 (11) | 0.0051 (10) | 0.0008 (9) |
| F1 | 0.0348 (6) | 0.0300 (6) | 0.0367 (7) | 0.0077 (5) | 0.0094 (5) | −0.0057 (5) |
| F2 | 0.0351 (6) | 0.0283 (6) | 0.0339 (7) | 0.0039 (5) | 0.0018 (5) | −0.0116 (5) |
| C1 | 0.0219 (9) | 0.0192 (9) | 0.0141 (9) | 0.0008 (7) | 0.0010 (7) | 0.0006 (8) |
| C9 | 0.0145 (9) | 0.0260 (10) | 0.0210 (10) | −0.0017 (7) | 0.0042 (7) | 0.0026 (8) |
| Cl1 | 0.0431 (3) | 0.0269 (3) | 0.0332 (3) | 0.0104 (2) | −0.0191 (2) | −0.0082 (2) |
| N1 | 0.0298 (9) | 0.0277 (9) | 0.0159 (8) | −0.0003 (7) | 0.0008 (7) | 0.0024 (7) |
| C15 | 0.0497 (13) | 0.0419 (12) | 0.0202 (11) | 0.0030 (10) | 0.0141 (10) | 0.0049 (10) |
| C16 | 0.0461 (13) | 0.0251 (10) | 0.0282 (12) | 0.0029 (9) | 0.0019 (10) | 0.0076 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C8—C9 | 1.512 (3) | C5—C4 | 1.380 (3) |
| C8—C1 | 1.532 (3) | C5—H5 | 0.9500 |
| C8—H8A | 0.9900 | C12—C13 | 1.378 (3) |
| C8—H8B | 0.9900 | C12—C11 | 1.380 (3) |
| C3—F1 | 1.353 (2) | C12—H12 | 0.9500 |
| C3—C4 | 1.378 (3) | C11—H11 | 0.9500 |
| C3—C2 | 1.386 (3) | C4—H4 | 0.9500 |
| C7—F2 | 1.360 (2) | C13—H13 | 0.9500 |
| C7—C6 | 1.371 (3) | C1—N1 | 1.515 (2) |
| C7—C2 | 1.388 (3) | C1—H1 | 1.0000 |
| C2—C1 | 1.518 (2) | N1—C15 | 1.487 (3) |
| C14—C13 | 1.382 (3) | N1—C16 | 1.488 (2) |
| C14—C9 | 1.386 (3) | N1—H1N1 | 0.93 (2) |
| C14—H14 | 0.9500 | C15—H15A | 0.9800 |
| C6—C5 | 1.378 (3) | C15—H15B | 0.9800 |
| C6—H6 | 0.9500 | C15—H15C | 0.9800 |
| C10—C11 | 1.386 (3) | C16—H16A | 0.9800 |
| C10—C9 | 1.392 (3) | C16—H16B | 0.9800 |
| C10—H10 | 0.9500 | C16—H16C | 0.9800 |
| | | | |
| C9—C8—C1 | 109.13 (14) | C3—C4—C5 | 118.14 (19) |
| C9—C8—H8A | 109.9 | C3—C4—H4 | 120.9 |
| C1—C8—H8A | 109.9 | C5—C4—H4 | 120.9 |
| C9—C8—H8B | 109.9 | C12—C13—C14 | 120.2 (2) |
| C1—C8—H8B | 109.9 | C12—C13—H13 | 119.9 |
| H8A—C8—H8B | 108.3 | C14—C13—H13 | 119.9 |
| F1—C3—C4 | 118.01 (17) | N1—C1—C2 | 113.84 (14) |
| F1—C3—C2 | 117.86 (16) | N1—C1—C8 | 111.51 (14) |
| C4—C3—C2 | 124.11 (18) | C2—C1—C8 | 113.39 (15) |
| F2—C7—C6 | 117.17 (17) | N1—C1—H1 | 105.8 |
| F2—C7—C2 | 118.28 (16) | C2—C1—H1 | 105.8 |
| C6—C7—C2 | 124.53 (18) | C8—C1—H1 | 105.8 |
| C3—C2—C7 | 114.24 (16) | C14—C9—C10 | 118.25 (17) |
| C3—C2—C1 | 119.49 (16) | C14—C9—C8 | 121.50 (17) |

| | | | |
|---|---|---|---|
| C7—C2—C1 | 125.73 (16) | C10—C9—C8 | 120.17 (17) |
| C13—C14—C9 | 121.15 (18) | C15—N1—C16 | 110.90 (16) |
| C13—C14—H14 | 119.4 | C15—N1—C1 | 111.34 (14) |
| C9—C14—H14 | 119.4 | C16—N1—C1 | 115.81 (15) |
| C7—C6—C5 | 118.07 (19) | C15—N1—H1N1 | 108.8 (14) |
| C7—C6—H6 | 121.0 | C16—N1—H1N1 | 104.7 (13) |
| C5—C6—H6 | 121.0 | C1—N1—H1N1 | 104.7 (14) |
| C11—C10—C9 | 120.47 (18) | N1—C15—H15A | 109.5 |
| C11—C10—H10 | 119.8 | N1—C15—H15B | 109.5 |
| C9—C10—H10 | 119.8 | H15A—C15—H15B | 109.5 |
| C6—C5—C4 | 120.89 (18) | N1—C15—H15C | 109.5 |
| C6—C5—H5 | 119.6 | H15A—C15—H15C | 109.5 |
| C4—C5—H5 | 119.6 | H15B—C15—H15C | 109.5 |
| C13—C12—C11 | 119.35 (19) | N1—C16—H16A | 109.5 |
| C13—C12—H12 | 120.3 | N1—C16—H16B | 109.5 |
| C11—C12—H12 | 120.3 | H16A—C16—H16B | 109.5 |
| C12—C11—C10 | 120.54 (18) | N1—C16—H16C | 109.5 |
| C12—C11—H11 | 119.7 | H16A—C16—H16C | 109.5 |
| C10—C11—H11 | 119.7 | H16B—C16—H16C | 109.5 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*1···Cl1 | 0.93 (2) | 2.08 (2) | 3.0006 (17) | 167.3 (19) |

**1-[1-(2,6-Difluorophenyl)-2-phenylethyl]pyrrolidin-1-ium chloride (IV)**

*Crystal data*

$C_{18}H_{20}F_2N^+\cdot Cl^-$
$M_r$ = 323.80
Triclinic, $P\overline{1}$
$a$ = 8.1365 (4) Å
$b$ = 12.7421 (10) Å
$c$ = 16.0451 (8) Å
$\alpha$ = 88.059 (5)°
$\beta$ = 82.349 (4)°
$\gamma$ = 86.140 (5)°
$V$ = 1644.42 (17) Å$^3$
$Z$ = 4
$F(000)$ = 680
$D_x$ = 1.308 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9872 reflections
$\theta$ = 3.0–25.4°
$\mu$ = 0.25 mm$^{-1}$
$T$ = 123 K
Block, colourless
0.5 × 0.4 × 0.3 mm

*Data collection*

Oxford Diffraction Xcalibur diffractometer
Radiation source: fine-focus sealed tube
scans in phi and $\omega$
Absorption correction: analytical (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.888, $T_{max}$ = 0.928
13277 measured reflections
5775 independent reflections
4892 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 3.0°
$h$ = −9→8
$k$ = −15→15
$l$ = −19→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.090$
$wR(F^2) = 0.206$
$S = 1.19$
5775 reflections
403 parameters
2 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0143P)^2 + 10.4102P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.59$ e Å$^{-3}$
$\Delta\rho_{min} = -0.34$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl2 | 1.11424 (18) | 0.86611 (11) | 0.52129 (8) | 0.0292 (3) |
| Cl1 | 0.6097 (2) | 0.63420 (11) | 1.00579 (9) | 0.0366 (4) |
| F1 | 0.3393 (4) | 0.7014 (3) | 0.6933 (2) | 0.0340 (8) |
| F2 | 0.0752 (4) | 0.4329 (3) | 0.8568 (2) | 0.0335 (8) |
| F3 | 0.9182 (4) | 0.7720 (3) | 0.2168 (2) | 0.0339 (8) |
| F4 | 0.5878 (4) | 1.0740 (3) | 0.3087 (2) | 0.0396 (9) |
| N2 | 0.8306 (6) | 0.8731 (4) | 0.4140 (3) | 0.0242 (10) |
| N1 | 0.3190 (6) | 0.6384 (4) | 0.9063 (3) | 0.0244 (10) |
| C27 | 0.7599 (6) | 0.9234 (4) | 0.2683 (3) | 0.0201 (11) |
| C9 | 0.2164 (6) | 0.5657 (4) | 0.7790 (3) | 0.0205 (11) |
| C28 | 0.6271 (7) | 0.9934 (4) | 0.2546 (3) | 0.0251 (12) |
| C1 | 0.3471 (6) | 0.5569 (4) | 0.8386 (3) | 0.0212 (11) |
| H1 | 0.335348 | 0.487106 | 0.868486 | 0.025* |
| C21 | 1.0944 (6) | 1.0169 (4) | 0.2351 (3) | 0.0227 (11) |
| C19 | 0.8667 (6) | 0.9430 (4) | 0.3359 (3) | 0.0218 (11) |
| H19 | 0.836336 | 1.016817 | 0.354039 | 0.026* |
| C3 | 0.5585 (6) | 0.4696 (4) | 0.7320 (3) | 0.0244 (12) |
| C20 | 1.0529 (7) | 0.9374 (4) | 0.3056 (3) | 0.0252 (12) |
| H20A | 1.113949 | 0.951212 | 0.353117 | 0.030* |
| H20B | 1.088997 | 0.865768 | 0.285632 | 0.030* |
| C14 | 0.0907 (7) | 0.4965 (4) | 0.7874 (3) | 0.0252 (12) |
| C32 | 0.7907 (7) | 0.8440 (4) | 0.2094 (3) | 0.0258 (12) |
| C11 | 0.1145 (8) | 0.6272 (5) | 0.6475 (4) | 0.0325 (14) |
| H11 | 0.122464 | 0.673672 | 0.599757 | 0.039* |
| C22 | 1.1007 (7) | 1.1225 (5) | 0.2533 (4) | 0.0309 (13) |
| H22 | 1.075832 | 1.144702 | 0.309803 | 0.037* |
| C2 | 0.5263 (7) | 0.5557 (4) | 0.7956 (3) | 0.0242 (12) |
| H2A | 0.546795 | 0.624627 | 0.767039 | 0.029* |
| H2B | 0.603436 | 0.544026 | 0.838268 | 0.029* |
| C25 | 1.1662 (7) | 1.0602 (5) | 0.0882 (4) | 0.0327 (14) |

| | | | | |
|---|---|---|---|---|
| H25 | 1.187292 | 1.039035 | 0.031390 | 0.039* |
| C10 | 0.2208 (7) | 0.6305 (4) | 0.7066 (3) | 0.0239 (12) |
| C8 | 0.5647 (7) | 0.3644 (5) | 0.7585 (4) | 0.0321 (13) |
| H8 | 0.546327 | 0.346785 | 0.816886 | 0.038* |
| C36 | 0.6658 (7) | 0.8977 (5) | 0.4644 (4) | 0.0348 (14) |
| H36A | 0.575698 | 0.900454 | 0.428380 | 0.042* |
| H36B | 0.663620 | 0.965988 | 0.492346 | 0.042* |
| C15 | 0.1649 (8) | 0.6286 (5) | 0.9678 (4) | 0.0351 (14) |
| H15A | 0.174375 | 0.564876 | 1.004224 | 0.042* |
| H15B | 0.065945 | 0.625638 | 0.938275 | 0.042* |
| C33 | 0.8366 (8) | 0.7563 (5) | 0.4037 (4) | 0.0346 (14) |
| H33A | 0.952397 | 0.726924 | 0.389866 | 0.042* |
| H33B | 0.771135 | 0.738088 | 0.359065 | 0.042* |
| C26 | 1.1262 (7) | 0.9866 (4) | 0.1518 (3) | 0.0256 (12) |
| H26 | 1.120489 | 0.914988 | 0.138423 | 0.031* |
| C31 | 0.6991 (8) | 0.8323 (5) | 0.1447 (4) | 0.0346 (14) |
| H31 | 0.722886 | 0.774732 | 0.107981 | 0.042* |
| C13 | −0.0197 (7) | 0.4882 (5) | 0.7300 (4) | 0.0338 (14) |
| H13 | −0.103175 | 0.438879 | 0.738846 | 0.041* |
| C5 | 0.6205 (8) | 0.4144 (6) | 0.5880 (4) | 0.0428 (16) |
| H5 | 0.638539 | 0.431937 | 0.529606 | 0.051* |
| C29 | 0.5330 (8) | 0.9887 (5) | 0.1896 (4) | 0.0359 (15) |
| H29 | 0.445057 | 1.039810 | 0.182690 | 0.043* |
| C17 | 0.2484 (8) | 0.8097 (5) | 0.9603 (4) | 0.0394 (15) |
| H17A | 0.172625 | 0.871072 | 0.948940 | 0.047* |
| H17B | 0.342017 | 0.834403 | 0.986302 | 0.047* |
| C30 | 0.5719 (8) | 0.9062 (6) | 0.1348 (4) | 0.0392 (16) |
| H30 | 0.509337 | 0.900738 | 0.089399 | 0.047* |
| C7 | 0.5979 (7) | 0.2849 (5) | 0.7000 (4) | 0.0380 (15) |
| H7 | 0.599135 | 0.213208 | 0.718453 | 0.046* |
| C24 | 1.1755 (8) | 1.1644 (5) | 0.1071 (4) | 0.0386 (15) |
| H24 | 1.204076 | 1.214593 | 0.063287 | 0.046* |
| C23 | 1.1434 (8) | 1.1956 (5) | 0.1892 (4) | 0.0361 (14) |
| H23 | 1.150355 | 1.267240 | 0.202027 | 0.043* |
| C18 | 0.3116 (8) | 0.7526 (4) | 0.8797 (4) | 0.0329 (14) |
| H18A | 0.234606 | 0.766623 | 0.837207 | 0.040* |
| H18B | 0.423028 | 0.774539 | 0.855950 | 0.040* |
| C12 | −0.0056 (8) | 0.5533 (5) | 0.6597 (4) | 0.0390 (15) |
| H12 | −0.078813 | 0.547876 | 0.618826 | 0.047* |
| C4 | 0.5858 (7) | 0.4928 (5) | 0.6459 (4) | 0.0312 (13) |
| H4 | 0.580421 | 0.564153 | 0.626864 | 0.037* |
| C6 | 0.6290 (8) | 0.3101 (6) | 0.6148 (5) | 0.0487 (19) |
| H6 | 0.656059 | 0.256025 | 0.575020 | 0.058* |
| C35 | 0.6475 (9) | 0.8081 (6) | 0.5286 (4) | 0.0473 (18) |
| H35A | 0.530490 | 0.788934 | 0.539572 | 0.057* |
| H35B | 0.683247 | 0.827842 | 0.582205 | 0.057* |
| C16 | 0.1547 (9) | 0.7276 (5) | 1.0185 (4) | 0.0420 (16) |
| H16A | 0.207772 | 0.713990 | 1.070222 | 0.050* |

| | | | | |
|---|---|---|---|---|
| H16B | 0.037391 | 0.752837 | 1.034725 | 0.050* |
| C34 | 0.7597 (9) | 0.7161 (6) | 0.4895 (4) | 0.0449 (17) |
| H34A | 0.847256 | 0.694674 | 0.525112 | 0.054* |
| H34B | 0.693919 | 0.654893 | 0.483513 | 0.054* |
| H1′ | 0.397 (7) | 0.632 (5) | 0.948 (3) | 0.054* |
| H2 | 0.911 (6) | 0.875 (5) | 0.454 (3) | 0.054* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl2 | 0.0390 (8) | 0.0279 (7) | 0.0220 (7) | 0.0035 (6) | −0.0099 (6) | −0.0062 (5) |
| Cl1 | 0.0553 (10) | 0.0307 (8) | 0.0290 (8) | −0.0163 (7) | −0.0203 (7) | 0.0075 (6) |
| F1 | 0.041 (2) | 0.0338 (19) | 0.0285 (18) | −0.0134 (15) | −0.0076 (15) | 0.0094 (14) |
| F2 | 0.0333 (19) | 0.0309 (18) | 0.0360 (19) | −0.0100 (14) | −0.0017 (15) | 0.0077 (15) |
| F3 | 0.038 (2) | 0.0304 (18) | 0.0321 (19) | 0.0034 (15) | −0.0013 (15) | −0.0111 (14) |
| F4 | 0.034 (2) | 0.0281 (18) | 0.057 (2) | 0.0064 (15) | −0.0083 (17) | −0.0129 (16) |
| N2 | 0.028 (3) | 0.027 (2) | 0.016 (2) | −0.003 (2) | −0.0015 (19) | 0.0012 (19) |
| N1 | 0.031 (3) | 0.027 (2) | 0.016 (2) | −0.003 (2) | −0.0052 (19) | −0.0038 (19) |
| C27 | 0.023 (3) | 0.021 (3) | 0.017 (3) | −0.004 (2) | −0.002 (2) | 0.000 (2) |
| C9 | 0.022 (3) | 0.021 (3) | 0.018 (3) | 0.005 (2) | −0.002 (2) | −0.005 (2) |
| C28 | 0.023 (3) | 0.025 (3) | 0.027 (3) | −0.006 (2) | −0.002 (2) | −0.001 (2) |
| C1 | 0.026 (3) | 0.020 (3) | 0.018 (3) | −0.001 (2) | −0.006 (2) | −0.002 (2) |
| C21 | 0.015 (3) | 0.029 (3) | 0.024 (3) | −0.001 (2) | −0.001 (2) | −0.001 (2) |
| C19 | 0.026 (3) | 0.021 (3) | 0.018 (3) | −0.005 (2) | −0.003 (2) | 0.002 (2) |
| C3 | 0.020 (3) | 0.031 (3) | 0.023 (3) | −0.003 (2) | −0.001 (2) | −0.005 (2) |
| C20 | 0.026 (3) | 0.029 (3) | 0.022 (3) | −0.004 (2) | −0.006 (2) | −0.001 (2) |
| C14 | 0.023 (3) | 0.023 (3) | 0.030 (3) | 0.000 (2) | −0.003 (2) | −0.002 (2) |
| C32 | 0.027 (3) | 0.027 (3) | 0.022 (3) | −0.006 (2) | 0.002 (2) | −0.001 (2) |
| C11 | 0.043 (4) | 0.033 (3) | 0.024 (3) | 0.005 (3) | −0.015 (3) | 0.001 (2) |
| C22 | 0.031 (3) | 0.034 (3) | 0.028 (3) | −0.006 (3) | −0.002 (3) | −0.006 (3) |
| C2 | 0.023 (3) | 0.028 (3) | 0.022 (3) | −0.006 (2) | −0.005 (2) | −0.003 (2) |
| C25 | 0.032 (3) | 0.044 (4) | 0.021 (3) | −0.005 (3) | 0.002 (2) | 0.001 (3) |
| C10 | 0.024 (3) | 0.023 (3) | 0.025 (3) | −0.004 (2) | −0.002 (2) | −0.001 (2) |
| C8 | 0.020 (3) | 0.039 (3) | 0.037 (3) | 0.002 (2) | −0.003 (2) | −0.002 (3) |
| C36 | 0.027 (3) | 0.053 (4) | 0.024 (3) | −0.008 (3) | 0.004 (2) | −0.004 (3) |
| C15 | 0.038 (4) | 0.035 (3) | 0.029 (3) | 0.001 (3) | 0.008 (3) | −0.004 (3) |
| C33 | 0.045 (4) | 0.032 (3) | 0.028 (3) | −0.007 (3) | −0.007 (3) | 0.005 (3) |
| C26 | 0.023 (3) | 0.031 (3) | 0.024 (3) | −0.005 (2) | −0.003 (2) | −0.001 (2) |
| C31 | 0.036 (3) | 0.044 (4) | 0.025 (3) | −0.020 (3) | 0.003 (3) | −0.011 (3) |
| C13 | 0.025 (3) | 0.029 (3) | 0.049 (4) | −0.007 (2) | −0.010 (3) | 0.000 (3) |
| C5 | 0.038 (4) | 0.063 (5) | 0.026 (3) | 0.002 (3) | 0.004 (3) | −0.015 (3) |
| C29 | 0.030 (3) | 0.038 (4) | 0.042 (4) | −0.008 (3) | −0.016 (3) | 0.011 (3) |
| C17 | 0.044 (4) | 0.032 (3) | 0.043 (4) | 0.005 (3) | −0.007 (3) | −0.016 (3) |
| C30 | 0.035 (4) | 0.062 (4) | 0.026 (3) | −0.022 (3) | −0.013 (3) | 0.003 (3) |
| C7 | 0.027 (3) | 0.035 (3) | 0.050 (4) | 0.000 (3) | 0.002 (3) | −0.011 (3) |
| C24 | 0.037 (4) | 0.047 (4) | 0.031 (3) | −0.007 (3) | −0.002 (3) | 0.015 (3) |
| C23 | 0.034 (3) | 0.026 (3) | 0.048 (4) | −0.003 (3) | −0.002 (3) | −0.001 (3) |
| C18 | 0.046 (4) | 0.022 (3) | 0.031 (3) | −0.001 (3) | −0.005 (3) | −0.005 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C12 | 0.033 (3) | 0.041 (4) | 0.049 (4) | 0.000 (3) | −0.024 (3) | −0.005 (3) |
| C4 | 0.029 (3) | 0.038 (3) | 0.026 (3) | −0.004 (3) | 0.000 (2) | −0.003 (3) |
| C6 | 0.031 (4) | 0.057 (5) | 0.056 (5) | 0.000 (3) | 0.008 (3) | −0.034 (4) |
| C35 | 0.045 (4) | 0.075 (5) | 0.023 (3) | −0.020 (4) | 0.001 (3) | 0.003 (3) |
| C16 | 0.046 (4) | 0.047 (4) | 0.031 (3) | 0.004 (3) | 0.001 (3) | −0.012 (3) |
| C34 | 0.047 (4) | 0.056 (4) | 0.034 (4) | −0.015 (3) | −0.009 (3) | 0.018 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C10 | 1.357 (6) | C3—C4 | 1.393 (8) |
| F2—C14 | 1.352 (6) | C3—C2 | 1.510 (7) |
| F3—C32 | 1.353 (6) | C14—C13 | 1.381 (8) |
| F4—C28 | 1.361 (6) | C32—C31 | 1.373 (8) |
| N2—C36 | 1.491 (7) | C11—C10 | 1.369 (8) |
| N2—C33 | 1.500 (7) | C11—C12 | 1.393 (9) |
| N2—C19 | 1.520 (6) | C22—C23 | 1.389 (8) |
| N1—C15 | 1.498 (7) | C25—C24 | 1.381 (9) |
| N1—C18 | 1.502 (7) | C25—C26 | 1.383 (8) |
| N1—C1 | 1.513 (6) | C8—C7 | 1.394 (8) |
| C27—C28 | 1.390 (7) | C36—C35 | 1.513 (9) |
| C27—C32 | 1.396 (7) | C15—C16 | 1.515 (8) |
| C27—C19 | 1.513 (7) | C33—C34 | 1.521 (8) |
| C9—C14 | 1.385 (7) | C31—C30 | 1.373 (9) |
| C9—C10 | 1.400 (7) | C13—C12 | 1.374 (9) |
| C9—C1 | 1.518 (7) | C5—C4 | 1.378 (9) |
| C28—C29 | 1.378 (8) | C5—C6 | 1.382 (10) |
| C1—C2 | 1.528 (7) | C29—C30 | 1.386 (9) |
| C21—C26 | 1.391 (7) | C17—C18 | 1.516 (8) |
| C21—C22 | 1.392 (8) | C17—C16 | 1.550 (9) |
| C21—C20 | 1.513 (7) | C7—C6 | 1.388 (10) |
| C19—C20 | 1.526 (7) | C24—C23 | 1.375 (9) |
| C3—C8 | 1.392 (8) | C35—C34 | 1.534 (10) |
| | | | |
| C36—N2—C33 | 103.9 (4) | F3—C32—C31 | 117.2 (5) |
| C36—N2—C19 | 114.4 (4) | F3—C32—C27 | 118.1 (5) |
| C33—N2—C19 | 118.5 (4) | C31—C32—C27 | 124.8 (5) |
| C15—N1—C18 | 103.7 (4) | C10—C11—C12 | 117.7 (5) |
| C15—N1—C1 | 115.1 (4) | C23—C22—C21 | 120.2 (5) |
| C18—N1—C1 | 118.3 (4) | C3—C2—C1 | 110.2 (4) |
| C28—C27—C32 | 113.3 (5) | C24—C25—C26 | 120.2 (6) |
| C28—C27—C19 | 120.6 (5) | F1—C10—C11 | 117.3 (5) |
| C32—C27—C19 | 125.7 (5) | F1—C10—C9 | 118.0 (5) |
| C14—C9—C10 | 113.8 (5) | C11—C10—C9 | 124.7 (5) |
| C14—C9—C1 | 120.1 (5) | C3—C8—C7 | 120.4 (6) |
| C10—C9—C1 | 125.5 (5) | N2—C36—C35 | 104.2 (5) |
| F4—C28—C29 | 117.1 (5) | N1—C15—C16 | 103.7 (5) |
| F4—C28—C27 | 117.7 (5) | N2—C33—C34 | 103.2 (5) |
| C29—C28—C27 | 125.2 (5) | C25—C26—C21 | 120.3 (5) |

| | | | |
|---|---|---|---|
| N1—C1—C9 | 113.3 (4) | C30—C31—C32 | 118.0 (6) |
| N1—C1—C2 | 110.0 (4) | C12—C13—C14 | 118.2 (5) |
| C9—C1—C2 | 114.7 (4) | C4—C5—C6 | 120.0 (6) |
| C26—C21—C22 | 119.0 (5) | C28—C29—C30 | 117.2 (6) |
| C26—C21—C20 | 121.1 (5) | C18—C17—C16 | 105.3 (5) |
| C22—C21—C20 | 119.9 (5) | C31—C30—C29 | 121.4 (5) |
| C27—C19—N2 | 113.2 (4) | C6—C7—C8 | 120.1 (6) |
| C27—C19—C20 | 114.3 (4) | C23—C24—C25 | 120.0 (6) |
| N2—C19—C20 | 110.0 (4) | C24—C23—C22 | 120.2 (6) |
| C8—C3—C4 | 118.4 (5) | N1—C18—C17 | 104.2 (5) |
| C8—C3—C2 | 120.3 (5) | C13—C12—C11 | 121.0 (5) |
| C4—C3—C2 | 121.3 (5) | C5—C4—C3 | 121.4 (6) |
| C21—C20—C19 | 111.0 (4) | C5—C6—C7 | 119.7 (6) |
| F2—C14—C13 | 117.9 (5) | C36—C35—C34 | 105.5 (5) |
| F2—C14—C9 | 117.6 (5) | C15—C16—C17 | 105.7 (5) |
| C13—C14—C9 | 124.6 (5) | C33—C34—C35 | 105.9 (5) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1′···Cl1 | 0.99 (1) | 2.07 (2) | 3.021 (5) | 163 (6) |
| N2—H2···Cl2 | 0.98 (1) | 2.08 (2) | 3.052 (5) | 169 (6) |

**[1-(2,6-Difluorophenyl)-2-phenylethyl](ethyl)azanium chloride dichloromethane hemisolvate (II)**

*Crystal data*

$2C_{16}H_{18}F_2N^+\cdot 2Cl^-\cdot CH_2Cl_2$
$M_r$ = 680.45
Monoclinic, *I*2/*a*
*a* = 22.9963 (14) Å
*b* = 7.8729 (5) Å
*c* = 19.033 (1) Å
*β* = 92.130 (5)°
*V* = 3443.5 (4) Å$^3$
*Z* = 4
*F*(000) = 1416
$D_x$ = 1.313 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 8967 reflections
*θ* = 3.0–26.4°
*μ* = 0.39 mm$^{-1}$
*T* = 123 K
Block, colourless
0.3 × 0.2 × 0.1 mm

*Data collection*

Oxford Diffraction Xcalibur diffractometer
Radiation source: fine-focus sealed tube
scans in phi and *ω*
Absorption correction: analytical (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.911, $T_{max}$ = 0.962
12738 measured reflections
3034 independent reflections
2813 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.021
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 3.3°
*h* = −27→26
*k* = −9→9
*l* = −21→22

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.077
*S* = 1.07
3034 reflections
224 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 2.8401P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.22$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cl1 | 0.28641 (2) | 0.60990 (5) | 0.16223 (2) | 0.02972 (12) | |
| Cl1S | 0.31326 (2) | 0.28821 (7) | 0.00859 (3) | 0.05486 (16) | |
| F1 | 0.05520 (5) | 1.14129 (13) | 0.11535 (6) | 0.0466 (3) | |
| F2 | 0.17656 (5) | 0.69870 (15) | 0.04908 (5) | 0.0570 (3) | |
| N1 | 0.19574 (5) | 0.90437 (17) | 0.17878 (7) | 0.0248 (3) | |
| C1 | 0.13169 (6) | 0.88167 (19) | 0.16264 (7) | 0.0251 (3) | |
| H1 | 0.110554 | 0.967062 | 0.191048 | 0.030* | |
| C9 | 0.11629 (6) | 0.91562 (18) | 0.08602 (7) | 0.0242 (3) | |
| C3 | 0.05036 (7) | 0.6689 (2) | 0.17558 (8) | 0.0308 (3) | |
| C13 | 0.12414 (8) | 0.8489 (2) | −0.03848 (9) | 0.0391 (4) | |
| H13 | 0.140233 | 0.779839 | −0.073793 | 0.047* | |
| C12 | 0.08611 (7) | 0.9784 (2) | −0.05562 (9) | 0.0392 (4) | |
| H12 | 0.075593 | 0.999429 | −0.103567 | 0.047* | |
| C11 | 0.06315 (7) | 1.0777 (2) | −0.00420 (9) | 0.0369 (4) | |
| H11 | 0.037235 | 1.168104 | −0.016069 | 0.044* | |
| C14 | 0.13806 (7) | 0.8227 (2) | 0.03156 (8) | 0.0326 (4) | |
| C10 | 0.07854 (6) | 1.04341 (19) | 0.06485 (8) | 0.0288 (3) | |
| C4 | 0.00903 (8) | 0.7455 (2) | 0.21652 (9) | 0.0399 (4) | |
| H4 | 0.021169 | 0.820557 | 0.253303 | 0.048* | |
| C15 | 0.2196 (4) | 1.0751 (12) | 0.1558 (4) | 0.0316 (19) | 0.707 (5) |
| H15A | 0.195508 | 1.167814 | 0.174557 | 0.038* | 0.707 (5) |
| H15B | 0.217609 | 1.082569 | 0.103855 | 0.038* | 0.707 (5) |
| C8 | 0.03174 (8) | 0.5597 (2) | 0.12252 (9) | 0.0388 (4) | |
| H8 | 0.059436 | 0.506416 | 0.093985 | 0.047* | |
| C2 | 0.11439 (7) | 0.7045 (2) | 0.18834 (9) | 0.0326 (4) | |
| H2A | 0.137251 | 0.617767 | 0.163763 | 0.039* | |
| H2B | 0.124134 | 0.695173 | 0.239296 | 0.039* | |
| C7 | −0.02704 (9) | 0.5276 (3) | 0.11066 (10) | 0.0501 (5) | |
| H7 | −0.039328 | 0.451887 | 0.074176 | 0.060* | |
| C5 | −0.04964 (8) | 0.7136 (3) | 0.20423 (11) | 0.0490 (5) | |
| H5 | −0.077544 | 0.767277 | 0.232379 | 0.059* | |
| C6 | −0.06771 (8) | 0.6042 (3) | 0.15123 (11) | 0.0524 (5) | |
| H6 | −0.107970 | 0.581969 | 0.142816 | 0.063* | |
| C16 | 0.28347 (10) | 1.0963 (3) | 0.18310 (13) | 0.0360 (7) | 0.707 (5) |

| | | | | | |
|---|---|---|---|---|---|
| H16A | 0.285642 | 1.081935 | 0.234280 | 0.054* | 0.707 (5) |
| H16B | 0.297386 | 1.209892 | 0.171032 | 0.054* | 0.707 (5) |
| H16C | 0.307818 | 1.010445 | 0.161259 | 0.054* | 0.707 (5) |
| C1S | 0.250000 | 0.4136 (3) | 0.000000 | 0.0302 (5) | |
| H1S1 | 0.252617 | 0.487565 | −0.041826 | 0.036* | 0.5 |
| H1S2 | 0.247383 | 0.487565 | 0.041827 | 0.036* | 0.5 |
| C16A | 0.1932 (3) | 1.2115 (7) | 0.1843 (3) | 0.0383 (18) | 0.293 (5) |
| H16D | 0.155523 | 1.220984 | 0.158750 | 0.057* | 0.293 (5) |
| H16E | 0.216492 | 1.313058 | 0.175641 | 0.057* | 0.293 (5) |
| H16F | 0.186879 | 1.201409 | 0.234797 | 0.057* | 0.293 (5) |
| H1B | 0.2162 (8) | 0.819 (2) | 0.1612 (9) | 0.036 (5)* | |
| H1A | 0.2010 (7) | 0.893 (2) | 0.2273 (10) | 0.034 (5)* | |
| C15A | 0.2248 (10) | 1.057 (3) | 0.1594 (10) | 0.037 (6) | 0.293 (5) |
| H15C | 0.227354 | 1.060956 | 0.107617 | 0.044* | 0.293 (5) |
| H15D | 0.264950 | 1.056010 | 0.180153 | 0.044* | 0.293 (5) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0298 (2) | 0.0380 (2) | 0.02123 (18) | 0.00784 (16) | 0.00001 (14) | −0.00054 (15) |
| Cl1S | 0.0491 (3) | 0.0558 (3) | 0.0581 (3) | 0.0262 (2) | −0.0191 (2) | −0.0251 (2) |
| F1 | 0.0486 (6) | 0.0405 (6) | 0.0507 (6) | 0.0214 (5) | 0.0023 (5) | −0.0013 (5) |
| F2 | 0.0669 (7) | 0.0629 (7) | 0.0402 (6) | 0.0416 (6) | −0.0113 (5) | −0.0158 (5) |
| N1 | 0.0261 (7) | 0.0268 (7) | 0.0211 (7) | 0.0025 (6) | −0.0028 (5) | 0.0011 (6) |
| C1 | 0.0246 (7) | 0.0264 (8) | 0.0239 (7) | 0.0019 (6) | −0.0022 (6) | 0.0004 (6) |
| C9 | 0.0217 (7) | 0.0253 (8) | 0.0254 (7) | −0.0011 (6) | −0.0027 (6) | 0.0023 (6) |
| C3 | 0.0340 (8) | 0.0268 (8) | 0.0313 (8) | −0.0038 (7) | −0.0044 (7) | 0.0103 (7) |
| C13 | 0.0366 (9) | 0.0524 (11) | 0.0280 (8) | −0.0036 (8) | −0.0014 (7) | −0.0061 (8) |
| C12 | 0.0325 (9) | 0.0556 (11) | 0.0287 (8) | −0.0121 (8) | −0.0080 (7) | 0.0119 (8) |
| C11 | 0.0271 (8) | 0.0381 (9) | 0.0448 (10) | −0.0028 (7) | −0.0082 (7) | 0.0179 (8) |
| C14 | 0.0301 (8) | 0.0358 (9) | 0.0314 (8) | 0.0074 (7) | −0.0057 (6) | −0.0028 (7) |
| C10 | 0.0242 (7) | 0.0266 (8) | 0.0356 (8) | 0.0002 (6) | 0.0009 (6) | 0.0031 (7) |
| C4 | 0.0433 (10) | 0.0393 (10) | 0.0372 (9) | −0.0060 (8) | 0.0029 (8) | 0.0050 (8) |
| C15 | 0.023 (2) | 0.030 (3) | 0.041 (3) | −0.011 (3) | −0.0100 (19) | 0.014 (2) |
| C8 | 0.0451 (10) | 0.0303 (9) | 0.0405 (10) | −0.0053 (8) | −0.0048 (8) | 0.0039 (7) |
| C2 | 0.0331 (9) | 0.0294 (8) | 0.0349 (9) | −0.0014 (7) | −0.0056 (7) | 0.0078 (7) |
| C7 | 0.0563 (12) | 0.0426 (11) | 0.0499 (11) | −0.0201 (9) | −0.0178 (9) | 0.0104 (9) |
| C5 | 0.0373 (10) | 0.0504 (11) | 0.0599 (12) | 0.0000 (9) | 0.0101 (9) | 0.0196 (10) |
| C6 | 0.0349 (10) | 0.0546 (12) | 0.0665 (13) | −0.0143 (9) | −0.0130 (9) | 0.0315 (11) |
| C16 | 0.0326 (14) | 0.0355 (14) | 0.0397 (14) | −0.0085 (10) | −0.0007 (10) | 0.0018 (10) |
| C1S | 0.0286 (11) | 0.0273 (11) | 0.0340 (12) | 0.000 | −0.0070 (9) | 0.000 |
| C16A | 0.040 (3) | 0.031 (3) | 0.043 (3) | −0.010 (3) | −0.010 (3) | −0.002 (3) |
| C15A | 0.053 (9) | 0.042 (8) | 0.015 (5) | 0.024 (5) | −0.009 (4) | 0.006 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1S—C1S | 1.7606 (13) | C3—C2 | 1.510 (2) |
| F1—C10 | 1.3580 (18) | C13—C12 | 1.375 (3) |

| | | | |
|---|---|---|---|
| F2—C14 | 1.3516 (18) | C13—C14 | 1.375 (2) |
| N1—C15A | 1.43 (2) | C12—C11 | 1.374 (3) |
| N1—C1 | 1.5040 (19) | C11—C10 | 1.375 (2) |
| N1—C15 | 1.522 (8) | C4—C5 | 1.384 (3) |
| C1—C9 | 1.512 (2) | C15—C16 | 1.550 (8) |
| C1—C2 | 1.535 (2) | C8—C7 | 1.385 (3) |
| C9—C14 | 1.378 (2) | C7—C6 | 1.374 (3) |
| C9—C10 | 1.379 (2) | C5—C6 | 1.379 (3) |
| C3—C8 | 1.382 (2) | C16A—C15A | 1.51 (2) |
| C3—C4 | 1.389 (2) | | |

| | | | |
|---|---|---|---|
| C15A—N1—C1 | 120.8 (11) | F2—C14—C9 | 116.80 (14) |
| C1—N1—C15 | 114.0 (4) | C13—C14—C9 | 124.89 (15) |
| N1—C1—C9 | 111.62 (12) | F1—C10—C11 | 118.07 (14) |
| N1—C1—C2 | 107.84 (12) | F1—C10—C9 | 117.92 (14) |
| C9—C1—C2 | 114.45 (13) | C11—C10—C9 | 124.01 (15) |
| C14—C9—C10 | 114.19 (14) | C5—C4—C3 | 120.64 (17) |
| C14—C9—C1 | 123.60 (13) | N1—C15—C16 | 110.2 (7) |
| C10—C9—C1 | 122.21 (13) | C3—C8—C7 | 120.44 (18) |
| C8—C3—C4 | 118.70 (16) | C3—C2—C1 | 112.35 (13) |
| C8—C3—C2 | 120.46 (15) | C6—C7—C8 | 120.56 (18) |
| C4—C3—C2 | 120.84 (15) | C6—C5—C4 | 120.15 (19) |
| C12—C13—C14 | 117.66 (16) | C7—C6—C5 | 119.51 (18) |
| C11—C12—C13 | 120.75 (15) | Cl1S—C1S—Cl1S$^{i}$ | 111.79 (12) |
| C12—C11—C10 | 118.49 (15) | N1—C15A—C16A | 111.2 (13) |
| F2—C14—C13 | 118.30 (14) | | |

Symmetry code: (i) −*x*+1/2, *y*, −*z*.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*B*···Cl1 | 0.89 (2) | 2.30 (2) | 3.1417 (14) | 156.0 (16) |

## *tert*-Butyl[1-(2,6-difluorophenyl)-2-phenylethyl]azanium chloride (III)

*Crystal data*

$C_{18}H_{22}F_2N^+\cdot Cl^-$
$M_r$ = 325.81
Monoclinic, $P2_1/c$
$a$ = 11.3115 (6) Å
$b$ = 10.5400 (5) Å
$c$ = 14.8039 (7) Å
$\beta$ = 105.044 (5)°
$V$ = 1704.48 (15) Å$^3$
$Z$ = 4
$F(000)$ = 688
$D_x$ = 1.270 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 8922 reflections
$\theta$ = 3.0–26.2°
$\mu$ = 0.24 mm$^{-1}$
$T$ = 123 K
Block, colourless
0.4 × 0.2 × 0.1 mm

*Data collection*

Oxford Diffraction Xcalibur diffractometer
Radiation source: fine-focus sealed tube
scans in phi and $\omega$
Absorption correction: analytical (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.944, $T_{max}$ = 0.976
7223 measured reflections

3008 independent reflections
2343 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 3.3°
$h$ = −13→13
$k$ = −8→12
$l$ = −17→12

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.095
$S$ = 1.06
3008 reflections
210 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0303P)^2 + 0.5824P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.39037 (5) | 1.13981 (5) | 0.05585 (4) | 0.02430 (17) |
| F1 | 0.09857 (12) | 0.74034 (14) | 0.10106 (9) | 0.0339 (4) |
| F2 | 0.39994 (13) | 0.60187 (13) | −0.04167 (9) | 0.0323 (4) |
| N1 | 0.41820 (17) | 0.84575 (19) | 0.06664 (12) | 0.0173 (4) |
| C1 | 0.5951 (2) | 0.8889 (2) | 0.19595 (15) | 0.0253 (6) |
| H1C | 0.648652 | 0.862937 | 0.156776 | 0.038* |
| H1D | 0.636607 | 0.873592 | 0.261856 | 0.038* |
| H1E | 0.576024 | 0.979356 | 0.186458 | 0.038* |
| C2 | 0.3904 (2) | 0.8518 (2) | 0.22761 (15) | 0.0266 (6) |
| H2A | 0.367671 | 0.941068 | 0.215264 | 0.040* |
| H2B | 0.431267 | 0.840815 | 0.294127 | 0.040* |
| H2C | 0.316581 | 0.799009 | 0.211062 | 0.040* |
| C3 | 0.5069 (2) | 0.6716 (2) | 0.18050 (15) | 0.0264 (6) |
| H3A | 0.430789 | 0.622529 | 0.168216 | 0.040* |
| H3B | 0.555718 | 0.655092 | 0.244475 | 0.040* |
| H3C | 0.553596 | 0.646429 | 0.136139 | 0.040* |
| C4 | 0.4769 (2) | 0.8122 (2) | 0.16915 (14) | 0.0193 (5) |
| C5 | 0.2864 (2) | 0.8140 (2) | 0.02029 (14) | 0.0186 (5) |
| H5 | 0.234618 | 0.867019 | 0.050999 | 0.022* |
| C6 | 0.2527 (2) | 0.6777 (2) | 0.03035 (14) | 0.0185 (5) |
| C7 | 0.1596 (2) | 0.6448 (2) | 0.07147 (15) | 0.0233 (5) |
| C8 | 0.1273 (2) | 0.5219 (3) | 0.08481 (17) | 0.0325 (6) |

| | | | | |
|---|---|---|---|---|
| H8 | 0.064723 | 0.504267 | 0.115178 | 0.039* |
| C9 | 0.1874 (2) | 0.4250 (3) | 0.05328 (17) | 0.0352 (7) |
| H9 | 0.166274 | 0.339447 | 0.061977 | 0.042* |
| C10 | 0.2784 (2) | 0.4506 (2) | 0.00904 (17) | 0.0313 (6) |
| H10 | 0.319903 | 0.384076 | −0.013306 | 0.038* |
| C11 | 0.3068 (2) | 0.5751 (2) | −0.00151 (15) | 0.0234 (6) |
| C12 | 0.2600 (2) | 0.8573 (2) | −0.08193 (14) | 0.0213 (5) |
| H12A | 0.290438 | 0.944996 | −0.084222 | 0.026* |
| H12B | 0.304422 | 0.801691 | −0.115937 | 0.026* |
| C13 | 0.1247 (2) | 0.8529 (2) | −0.12948 (14) | 0.0207 (5) |
| C14 | 0.0489 (2) | 0.9493 (3) | −0.11500 (17) | 0.0332 (6) |
| H14 | 0.082380 | 1.018435 | −0.075390 | 0.040* |
| C15 | −0.0752 (3) | 0.9463 (3) | −0.15751 (19) | 0.0444 (8) |
| H15 | −0.126360 | 1.013158 | −0.147073 | 0.053* |
| C16 | −0.1250 (2) | 0.8465 (3) | −0.21498 (19) | 0.0415 (7) |
| H16 | −0.210343 | 0.844521 | −0.244199 | 0.050* |
| C17 | −0.0509 (2) | 0.7503 (3) | −0.22978 (17) | 0.0368 (7) |
| H17 | −0.084782 | 0.681460 | −0.269479 | 0.044* |
| C18 | 0.0738 (2) | 0.7533 (2) | −0.18684 (15) | 0.0274 (6) |
| H18 | 0.124631 | 0.685936 | −0.197099 | 0.033* |
| H1A | 0.423 (2) | 0.933 (2) | 0.0648 (15) | 0.020 (6)* |
| H1B | 0.471 (2) | 0.818 (2) | 0.0302 (16) | 0.033 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0305 (3) | 0.0186 (3) | 0.0261 (3) | 0.0042 (3) | 0.0115 (2) | 0.0024 (3) |
| F1 | 0.0296 (8) | 0.0364 (9) | 0.0419 (8) | 0.0001 (7) | 0.0202 (7) | 0.0009 (7) |
| F2 | 0.0383 (9) | 0.0276 (8) | 0.0363 (8) | 0.0065 (7) | 0.0190 (7) | 0.0004 (7) |
| N1 | 0.0222 (11) | 0.0132 (11) | 0.0172 (10) | 0.0009 (10) | 0.0063 (8) | 0.0011 (9) |
| C1 | 0.0248 (14) | 0.0286 (15) | 0.0205 (12) | −0.0010 (12) | 0.0021 (10) | 0.0037 (11) |
| C2 | 0.0302 (14) | 0.0320 (15) | 0.0186 (12) | −0.0025 (13) | 0.0081 (10) | −0.0008 (11) |
| C3 | 0.0329 (15) | 0.0232 (14) | 0.0205 (12) | 0.0024 (12) | 0.0021 (10) | 0.0061 (11) |
| C4 | 0.0213 (13) | 0.0210 (12) | 0.0142 (11) | 0.0003 (11) | 0.0021 (9) | 0.0035 (10) |
| C5 | 0.0198 (13) | 0.0168 (12) | 0.0196 (12) | 0.0025 (11) | 0.0056 (9) | 0.0006 (10) |
| C6 | 0.0197 (13) | 0.0191 (12) | 0.0151 (11) | 0.0001 (11) | 0.0019 (9) | 0.0020 (10) |
| C7 | 0.0207 (13) | 0.0252 (14) | 0.0225 (12) | 0.0007 (12) | 0.0029 (10) | 0.0010 (11) |
| C8 | 0.0302 (15) | 0.0340 (16) | 0.0317 (14) | −0.0100 (13) | 0.0051 (11) | 0.0075 (13) |
| C9 | 0.0429 (17) | 0.0215 (15) | 0.0338 (15) | −0.0113 (14) | −0.0031 (12) | 0.0069 (12) |
| C10 | 0.0389 (16) | 0.0182 (13) | 0.0315 (14) | 0.0024 (13) | −0.0003 (12) | −0.0005 (12) |
| C11 | 0.0264 (14) | 0.0242 (14) | 0.0198 (12) | 0.0009 (12) | 0.0061 (10) | 0.0019 (11) |
| C12 | 0.0228 (13) | 0.0209 (13) | 0.0197 (11) | 0.0004 (11) | 0.0047 (9) | 0.0041 (11) |
| C13 | 0.0241 (13) | 0.0213 (13) | 0.0171 (11) | −0.0003 (12) | 0.0059 (9) | 0.0054 (11) |
| C14 | 0.0326 (16) | 0.0272 (15) | 0.0347 (14) | 0.0043 (13) | −0.0005 (12) | −0.0044 (12) |
| C15 | 0.0326 (17) | 0.0459 (19) | 0.0513 (18) | 0.0166 (15) | 0.0047 (13) | −0.0013 (16) |
| C16 | 0.0226 (15) | 0.052 (2) | 0.0457 (16) | 0.0001 (15) | 0.0015 (12) | 0.0001 (16) |
| C17 | 0.0356 (17) | 0.0408 (17) | 0.0305 (14) | −0.0099 (15) | 0.0024 (12) | −0.0106 (13) |
| C18 | 0.0272 (15) | 0.0280 (15) | 0.0278 (13) | 0.0015 (12) | 0.0086 (11) | −0.0018 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C7 | 1.356 (3) | C6—C7 | 1.390 (3) |
| F2—C11 | 1.367 (2) | C7—C8 | 1.374 (3) |
| N1—C5 | 1.508 (3) | C8—C9 | 1.374 (4) |
| N1—C4 | 1.532 (3) | C8—H8 | 0.9500 |
| N1—H1A | 0.92 (2) | C9—C10 | 1.382 (4) |
| N1—H1B | 0.95 (2) | C9—H9 | 0.9500 |
| C1—C4 | 1.524 (3) | C10—C11 | 1.369 (3) |
| C1—H1C | 0.9800 | C10—H10 | 0.9500 |
| C1—H1D | 0.9800 | C12—C13 | 1.510 (3) |
| C1—H1E | 0.9800 | C12—H12A | 0.9900 |
| C2—C4 | 1.523 (3) | C12—H12B | 0.9900 |
| C2—H2A | 0.9800 | C13—C18 | 1.379 (3) |
| C2—H2B | 0.9800 | C13—C14 | 1.382 (3) |
| C2—H2C | 0.9800 | C14—C15 | 1.381 (4) |
| C3—C4 | 1.520 (3) | C14—H14 | 0.9500 |
| C3—H3A | 0.9800 | C15—C16 | 1.377 (4) |
| C3—H3B | 0.9800 | C15—H15 | 0.9500 |
| C3—H3C | 0.9800 | C16—C17 | 1.369 (4) |
| C5—C6 | 1.504 (3) | C16—H16 | 0.9500 |
| C5—C12 | 1.534 (3) | C17—C18 | 1.389 (3) |
| C5—H5 | 1.0000 | C17—H17 | 0.9500 |
| C6—C11 | 1.384 (3) | C18—H18 | 0.9500 |
| | | | |
| C5—N1—C4 | 121.50 (16) | F1—C7—C8 | 118.5 (2) |
| C5—N1—H1A | 105.3 (14) | F1—C7—C6 | 117.6 (2) |
| C4—N1—H1A | 104.3 (14) | C8—C7—C6 | 123.9 (2) |
| C5—N1—H1B | 111.4 (14) | C9—C8—C7 | 118.6 (2) |
| C4—N1—H1B | 108.5 (15) | C9—C8—H8 | 120.7 |
| H1A—N1—H1B | 104.2 (19) | C7—C8—H8 | 120.7 |
| C4—C1—H1C | 109.5 | C8—C9—C10 | 120.7 (2) |
| C4—C1—H1D | 109.5 | C8—C9—H9 | 119.6 |
| H1C—C1—H1D | 109.5 | C10—C9—H9 | 119.6 |
| C4—C1—H1E | 109.5 | C11—C10—C9 | 117.8 (2) |
| H1C—C1—H1E | 109.5 | C11—C10—H10 | 121.1 |
| H1D—C1—H1E | 109.5 | C9—C10—H10 | 121.1 |
| C4—C2—H2A | 109.5 | F2—C11—C10 | 118.5 (2) |
| C4—C2—H2B | 109.5 | F2—C11—C6 | 116.5 (2) |
| H2A—C2—H2B | 109.5 | C10—C11—C6 | 124.9 (2) |
| C4—C2—H2C | 109.5 | C13—C12—C5 | 111.39 (17) |
| H2A—C2—H2C | 109.5 | C13—C12—H12A | 109.4 |
| H2B—C2—H2C | 109.5 | C5—C12—H12A | 109.4 |
| C4—C3—H3A | 109.5 | C13—C12—H12B | 109.4 |
| C4—C3—H3B | 109.5 | C5—C12—H12B | 109.4 |
| H3A—C3—H3B | 109.5 | H12A—C12—H12B | 108.0 |
| C4—C3—H3C | 109.5 | C18—C13—C14 | 118.6 (2) |
| H3A—C3—H3C | 109.5 | C18—C13—C12 | 121.4 (2) |

| | | | |
|---|---|---|---|
| H3B—C3—H3C | 109.5 | C14—C13—C12 | 120.0 (2) |
| C3—C4—C2 | 111.26 (19) | C15—C14—C13 | 120.7 (3) |
| C3—C4—C1 | 109.42 (19) | C15—C14—H14 | 119.7 |
| C2—C4—C1 | 110.85 (19) | C13—C14—H14 | 119.7 |
| C3—C4—N1 | 111.22 (18) | C16—C15—C14 | 120.2 (3) |
| C2—C4—N1 | 108.81 (18) | C16—C15—H15 | 119.9 |
| C1—C4—N1 | 105.11 (17) | C14—C15—H15 | 119.9 |
| C6—C5—N1 | 114.40 (18) | C17—C16—C15 | 119.6 (3) |
| C6—C5—C12 | 113.12 (18) | C17—C16—H16 | 120.2 |
| N1—C5—C12 | 107.39 (16) | C15—C16—H16 | 120.2 |
| C6—C5—H5 | 107.2 | C16—C17—C18 | 120.1 (3) |
| N1—C5—H5 | 107.2 | C16—C17—H17 | 119.9 |
| C12—C5—H5 | 107.2 | C18—C17—H17 | 119.9 |
| C11—C6—C7 | 114.0 (2) | C13—C18—C17 | 120.7 (2) |
| C11—C6—C5 | 124.53 (19) | C13—C18—H18 | 119.6 |
| C7—C6—C5 | 121.5 (2) | C17—C18—H18 | 119.6 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···Cl1 | 0.92 (2) | 2.21 (3) | 3.115 (2) | 167.7 (19) |
| N1—H1*B*···Cl1$^{i}$ | 0.95 (2) | 2.31 (2) | 3.1684 (19) | 151 (2) |

Symmetry code: (i) −*x*+1, −*y*+2, −*z*.