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# 1-[(6-Chloropyridin-3-yl)methyl]-4-[2,6-dinitro-4-(trifluoromethyl)phenyl]-piperazine

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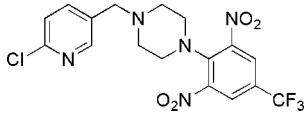
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.049; $wR$ factor = 0.153; data-to-parameter ratio = 11.2.

In the title compound, $C_{17}H_{15}ClF_3N_5O_4$, the piperazine ring has a chair conformation, with the benzene and pyridine rings attached to the piperazine in equatorial positions. In addition, C—H···N, C—H···O and C—H···F intermolecular hydrogen bonds link molecules into a two-dimensional network structure. The F atoms are disordered over two sites in an approximately 5:1 ratio.

## Related literature

For related literature, see: Barbaro *et al.* (2001); Grundt *et al.* (2005); Lopez-Rodriguez *et al.* (2002).




## Experimental

### Crystal data

$C_{17}H_{15}ClF_3N_5O_4$
$M_r$ = 445.79
Triclinic, $P\overline{1}$
$a$ = 8.131 (2) Å
$b$ = 10.171 (3) Å
$c$ = 13.159 (3) Å
$\alpha$ = 79.992 (5)°
$\beta$ = 73.465 (4)°
$\gamma$ = 68.099 (4)°
$V$ = 965.2 (4) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.26 mm$^{-1}$
$T$ = 298 (2) K
0.39 × 0.32 × 0.24 mm

### Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min}$ = 0.901, $T_{max}$ = 0.937
4840 measured reflections
3361 independent reflections
2387 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.049

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.153
$S$ = 1.09
3361 reflections
299 parameters
94 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C8—H8$A$···O2$^{i}$ | 0.97 | 2.52 | 3.387 (5) | 149 |
| C13—H13···N1$^{ii}$ | 0.93 | 2.51 | 3.331 (4) | 147 |
| C15—H15···O2$^{iii}$ | 0.93 | 2.54 | 3.466 (4) | 178 |
| C5—H5···O4$^{iv}$ | 0.93 | 2.68 | 3.593 (5) | 166 |
| C3—H3···F3$^{v}$ | 0.93 | 2.62 | 3.370 (16) | 138 |

Symmetry codes: (i) $-x+2, -y, -z+1$; (ii) $-x+2, -y+1, -z+1$; (iii) $x-1, y, z$; (iv) $x+1, y, z$; (v) $x, y, z+1$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2055).

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# supplementary materials

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# 1-[(6-Chloropyridin-3-yl)methyl]-4-[2,6-dinitro-4-(trifluoromethyl)phenyl]piperazine

**M.-Y. Cai, X.-Y. Xu, H.-J. Li, Z.-Y. Zou and L. Li**

## Comment

The derivatives of arylpiperazine are a core fragment of many bioactive compounds and have been widely investigated and found to show a variety of pharmacological effects, such as the ligands of the serotonin receptors (Barbaro *et al.*, 2001; Grundt *et al.*, 2005; Lopez-Rodriguez *et al.*, 2002, respectively). Introduction of an O-substituent in the aryl ring of the arylpiperazine derivative often greatly affects the affinity of the compound for binding the receptor, in part due to the influence of substituent on the compound conformation. As part of our investigation on the stereochemistry of the arylpiperazine derivatives, the crystal structure of the title compound $C_{17}H_{15}ClF_3N_5O_4$ (I), has been determined.

The molecular structure is shown in Fig. 1. Owing to the delocalization of the π-electrons in the phenyl ring, the bond length of C11—N3 is shorter than a single bond compared with C6—N2, C7—N2, C10—N2, C8—N3 and C9—N3 (Table 1). Additionally, the bond length of C1—N1 is shorter than that of C5—N1 due to the electron-withdrawing effect of Cl1. In the crystal structure (Fig. 2 and Table 2), a weak intermolecular hydrogen-bond contact exists between atoms C3 and F3, forming chains along the *c* axis. The solid state structure is also enhanced significantly by weak hydrogen-bond C15—H15···O2, C5—H5···O4, C8—H8A···O2 and C13—H13···N1.

## Experimental

A solution of 6-chloro-3-(chloromethyl)pyridine (2 mmol) in ethanol (10 ml) was added dropwise into a solution of piperazine (4 mmol) and triethylamine (0.2 ml)in ethanol (30 ml) at 323–328 K. Then the mixture was stirred for 5 h at 328 K. After cooling, the mixture was treated with water (50 ml) and extracted with $CH_2Cl_2$ (3*50 ml). The organic layer was washed with water, dried over anhydrous $Na_2SO_4$, and concentrated to yield 1-((6-chloropyridin-3-yl)methyl)piperazine. A mixture of 2-chloro-1,3-dinitro-5-(trifluoromethyl)benzene (1 mmol), 1-((6-chloropyridin-3-yl)methyl)piperazine (1 mmol), $K_2CO_3$ (1 mmol) and dimethylformamide (15 ml) was stirred at 313 K for 3 h. After cooling, the mixture was treated with water (50 ml), and extracted with $CH_2Cl_2$ (3*50 ml). The organic layer was washed with water, dried over anhydrous $Na_2SO_4$, and concentrated. The residue was chromatographed over a column of silica gel and eluted with petroleum ether-ethyl acetate (4:1 *v*/*v*) to give the desired product (51% yield). Single crystals suitable for X-ray analysis were obtained from methanol solution (m.p. 435.7–437.5 K.).

## Refinement

The CF3 disorder was modelled with the 3 Fatoms over 2 sites F1, F2 and F3 and F1', F2', F3' and their occupancies refined competitively to 0.836 (6) and 0.164 (6). The C—F distances were restrained to with 1.27±0.02 Å. All H atom were initially located in a difference Fourier map, placed in geometrically idealized position and constrained to ride on their parent atom with C—H distances in the range 0.86–0.96 Å and $U_{iso}$(H) = 1.2$_{eq}$(C).

## Figures

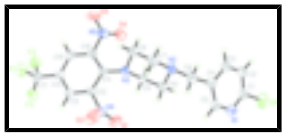

Fig. 1. The molecular structure of (I), with the atom numbering, showing displacement ellipsoids drawn at the 30% probability level. For clarity, the minor disorder component is omitted.

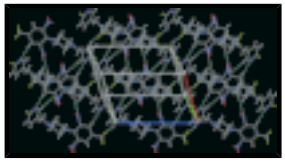

Fig. 2. The two-dimensional network structure of (I) formed by intermolecular hydrogen bonding interactions (shown as dashed lines). The minor disorder component is omitted for clarity.

## 1-[(6-Chloropyridin-3-yl)methyl]-4-[2,6-dinitro-4- (trifluoromethyl)phenyl]piperazine

### *Crystal data*

| | |
|---|---|
| $C_{17}H_{15}ClF_3N_5O_4$ | $Z = 2$ |
| $M_r = 445.79$ | $F_{000} = 456$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.534$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 8.131$ (2) Å | Cell parameters from 4932 reflections |
| $b = 10.171$ (3) Å | $\theta = 1.9$–$24.6$º |
| $c = 13.159$ (3) Å | $\mu = 0.26$ mm$^{-1}$ |
| $\alpha = 79.992$ (5)º | $T = 298$ (2) K |
| $\beta = 73.465$ (4)º | Block, colorless |
| $\gamma = 68.099$ (4)º | 0.39 × 0.32 × 0.24 mm |
| $V = 965.2$ (4) Å$^3$ | |

### *Data collection*

| | |
|---|---|
| Bruker APEX area-detector diffractometer | 3361 independent reflections |
| Radiation source: fine-focus sealed tube | 2387 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.049$ |
| $T = 298(2)$ K | $\theta_{max} = 25.1$º |
| φ and ω scans | $\theta_{min} = 1.6$º |
| Absorption correction: multi-scan (SADABS; Bruker, 2002) | $h = -9\rightarrow9$ |
| $T_{min} = 0.901$, $T_{max} = 0.937$ | $k = -12\rightarrow7$ |
| 4840 measured reflections | $l = -15\rightarrow14$ |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.049$ | H-atom parameters constrained |

| | |
|---|---|
| $wR(F^2) = 0.153$ | $w = 1/[\sigma^2(F_o^2) + (0.0852P)^2 + 0.0325P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.09$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 3361 reflections | $\Delta\rho_{max} = 0.41$ e Å$^{-3}$ |
| 299 parameters | $\Delta\rho_{min} = -0.28$ e Å$^{-3}$ |
| 94 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*Special details*

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on $F^2$, conventional *R*-factors *R* are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.7887 (5) | 0.7133 (4) | 0.9578 (3) | 0.0690 (9) | |
| C2 | 0.6835 (5) | 0.6354 (5) | 1.0161 (3) | 0.0755 (10) | |
| H2 | 0.5902 | 0.6706 | 1.0751 | 0.091* | |
| C3 | 0.7197 (5) | 0.5047 (4) | 0.9848 (3) | 0.0698 (10) | |
| H3 | 0.6512 | 0.4487 | 1.0230 | 0.084* | |
| C4 | 0.8582 (4) | 0.4550 (4) | 0.8963 (2) | 0.0618 (9) | |
| C5 | 0.9543 (5) | 0.5425 (4) | 0.8464 (3) | 0.0711 (10) | |
| H5 | 1.0500 | 0.5099 | 0.7878 | 0.085* | |
| C6 | 0.8963 (5) | 0.3145 (4) | 0.8573 (3) | 0.0716 (9) | |
| H6A | 1.0212 | 0.2810 | 0.8152 | 0.086* | |
| H6B | 0.8845 | 0.2461 | 0.9178 | 0.086* | |
| C7 | 0.7992 (5) | 0.1801 (4) | 0.7693 (3) | 0.0687 (9) | |
| H7A | 0.7762 | 0.1229 | 0.8353 | 0.082* | |
| H7B | 0.9250 | 0.1358 | 0.7314 | 0.082* | |
| C8 | 0.6745 (5) | 0.1841 (4) | 0.7032 (2) | 0.0632 (8) | |
| H8A | 0.6938 | 0.0885 | 0.6886 | 0.076* | |
| H8B | 0.5481 | 0.2266 | 0.7407 | 0.076* | |
| C9 | 0.6850 (4) | 0.4126 (3) | 0.6243 (2) | 0.0558 (8) | |
| H9A | 0.5578 | 0.4580 | 0.6591 | 0.067* | |
| H9B | 0.7129 | 0.4671 | 0.5576 | 0.067* | |
| C10 | 0.8044 (4) | 0.4098 (3) | 0.6939 (2) | 0.0574 (8) | |
| H10A | 0.9316 | 0.3722 | 0.6561 | 0.069* | |
| H10B | 0.7794 | 0.5060 | 0.7100 | 0.069* | |
| C11 | 0.6827 (4) | 0.2451 (3) | 0.5118 (2) | 0.0495 (7) | |

| | | | | | |
|---|---|---|---|---|---|
| C12 | 0.8179 (4) | 0.2309 (3) | 0.4177 (2) | 0.0512 (7) | |
| C13 | 0.8032 (4) | 0.2031 (3) | 0.3236 (2) | 0.0563 (8) | |
| H13 | 0.8972 | 0.1979 | 0.2627 | 0.068* | |
| C14 | 0.6456 (4) | 0.1826 (3) | 0.3203 (2) | 0.0531 (7) | |
| C15 | 0.5092 (4) | 0.1902 (3) | 0.4110 (2) | 0.0555 (8) | |
| H15 | 0.4051 | 0.1726 | 0.4104 | 0.067* | |
| C16 | 0.5276 (4) | 0.2240 (3) | 0.5028 (2) | 0.0516 (7) | |
| C17 | 0.6235 (5) | 0.1551 (4) | 0.2189 (3) | 0.0692 (9) | |
| Cl1 | 0.7482 (2) | 0.87989 (14) | 0.99492 (11) | 0.1126 (5) | |
| N1 | 0.9221 (4) | 0.6711 (4) | 0.8747 (2) | 0.0763 (9) | |
| N2 | 0.7724 (4) | 0.3222 (3) | 0.79287 (19) | 0.0588 (7) | |
| N3 | 0.7159 (3) | 0.2685 (2) | 0.60416 (18) | 0.0520 (6) | |
| N4 | 0.9940 (4) | 0.2365 (3) | 0.4205 (2) | 0.0644 (7) | |
| N5 | 0.3677 (3) | 0.2454 (4) | 0.5935 (2) | 0.0656 (7) | |
| O1 | 1.0076 (4) | 0.3452 (3) | 0.4285 (2) | 0.0932 (9) | |
| O2 | 1.1174 (4) | 0.1253 (4) | 0.4163 (4) | 0.1380 (14) | |
| O3 | 0.2940 (4) | 0.1571 (3) | 0.6140 (2) | 0.0954 (9) | |
| O4 | 0.3156 (3) | 0.3535 (3) | 0.6374 (2) | 0.0912 (9) | |
| F1 | 0.662 (3) | 0.0229 (16) | 0.2097 (13) | 0.094 (5) | 0.164 (6) |
| F2 | 0.456 (2) | 0.194 (2) | 0.2174 (14) | 0.097 (5) | 0.164 (6) |
| F3 | 0.706 (3) | 0.203 (2) | 0.1355 (11) | 0.087 (5) | 0.164 (6) |
| F1' | 0.5160 (7) | 0.0821 (5) | 0.2310 (3) | 0.1118 (14) | 0.836 (6) |
| F2' | 0.5492 (7) | 0.2745 (4) | 0.1667 (3) | 0.1255 (16) | 0.836 (6) |
| F3' | 0.7771 (5) | 0.0868 (6) | 0.1564 (3) | 0.1275 (17) | 0.836 (6) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.070 (2) | 0.085 (2) | 0.0592 (19) | −0.0259 (18) | −0.0246 (17) | −0.0084 (17) |
| C2 | 0.069 (2) | 0.106 (3) | 0.0493 (19) | −0.028 (2) | −0.0090 (16) | −0.0140 (19) |
| C3 | 0.067 (2) | 0.099 (3) | 0.0507 (19) | −0.041 (2) | −0.0125 (16) | 0.0024 (18) |
| C4 | 0.0554 (18) | 0.081 (2) | 0.0526 (18) | −0.0245 (17) | −0.0191 (15) | −0.0020 (16) |
| C5 | 0.0573 (19) | 0.095 (3) | 0.061 (2) | −0.032 (2) | −0.0004 (16) | −0.0135 (19) |
| C6 | 0.075 (2) | 0.076 (2) | 0.065 (2) | −0.0225 (19) | −0.0247 (18) | −0.0008 (17) |
| C7 | 0.087 (2) | 0.055 (2) | 0.0597 (19) | −0.0256 (18) | −0.0173 (18) | 0.0074 (15) |
| C8 | 0.076 (2) | 0.0559 (19) | 0.0567 (19) | −0.0302 (16) | −0.0053 (16) | −0.0038 (14) |
| C9 | 0.0631 (18) | 0.0478 (17) | 0.0546 (17) | −0.0200 (15) | −0.0098 (14) | −0.0043 (13) |
| C10 | 0.0621 (18) | 0.0559 (19) | 0.0558 (18) | −0.0249 (15) | −0.0112 (15) | −0.0030 (14) |
| C11 | 0.0445 (15) | 0.0467 (16) | 0.0552 (17) | −0.0161 (13) | −0.0057 (13) | −0.0078 (12) |
| C12 | 0.0405 (14) | 0.0585 (18) | 0.0568 (18) | −0.0206 (13) | −0.0067 (13) | −0.0099 (14) |
| C13 | 0.0496 (16) | 0.064 (2) | 0.0549 (18) | −0.0234 (15) | −0.0032 (14) | −0.0094 (14) |
| C14 | 0.0509 (16) | 0.0524 (17) | 0.0599 (17) | −0.0210 (13) | −0.0168 (14) | −0.0010 (13) |
| C15 | 0.0449 (16) | 0.0564 (19) | 0.070 (2) | −0.0232 (14) | −0.0165 (15) | 0.0016 (15) |
| C16 | 0.0377 (14) | 0.0492 (17) | 0.0610 (19) | −0.0127 (13) | −0.0037 (13) | −0.0050 (13) |
| C17 | 0.072 (2) | 0.082 (3) | 0.065 (2) | −0.037 (2) | −0.0234 (18) | 0.0014 (19) |
| Cl1 | 0.1419 (11) | 0.0949 (9) | 0.1115 (10) | −0.0327 (8) | −0.0469 (8) | −0.0247 (7) |
| N1 | 0.0707 (19) | 0.096 (2) | 0.071 (2) | −0.0408 (18) | −0.0118 (16) | −0.0089 (17) |
| N2 | 0.0673 (16) | 0.0586 (16) | 0.0510 (15) | −0.0238 (13) | −0.0139 (13) | −0.0005 (12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N3 | 0.0568 (14) | 0.0505 (15) | 0.0481 (14) | −0.0226 (12) | −0.0048 (11) | −0.0057 (11) |
| N4 | 0.0459 (14) | 0.081 (2) | 0.0682 (17) | −0.0248 (15) | −0.0024 (12) | −0.0230 (14) |
| N5 | 0.0415 (14) | 0.076 (2) | 0.0690 (18) | −0.0172 (15) | −0.0021 (12) | −0.0052 (15) |
| O1 | 0.0840 (17) | 0.0972 (19) | 0.119 (2) | −0.0597 (16) | −0.0191 (15) | −0.0056 (16) |
| O2 | 0.0605 (17) | 0.134 (3) | 0.235 (4) | −0.0209 (18) | −0.037 (2) | −0.079 (3) |
| O3 | 0.0716 (17) | 0.102 (2) | 0.105 (2) | −0.0489 (17) | 0.0107 (15) | 0.0008 (16) |
| O4 | 0.0546 (14) | 0.102 (2) | 0.100 (2) | −0.0168 (14) | 0.0134 (13) | −0.0421 (17) |
| F1 | 0.111 (7) | 0.088 (6) | 0.088 (7) | −0.028 (5) | −0.034 (5) | −0.016 (4) |
| F2 | 0.090 (6) | 0.114 (8) | 0.096 (7) | −0.029 (5) | −0.040 (5) | −0.018 (5) |
| F3 | 0.097 (7) | 0.096 (7) | 0.070 (6) | −0.042 (5) | −0.014 (4) | −0.005 (4) |
| F1' | 0.140 (3) | 0.151 (3) | 0.097 (2) | −0.102 (3) | −0.041 (2) | −0.005 (2) |
| F2' | 0.181 (4) | 0.110 (3) | 0.098 (2) | −0.043 (2) | −0.080 (2) | 0.0219 (18) |
| F3' | 0.093 (2) | 0.196 (4) | 0.102 (2) | −0.027 (2) | −0.0241 (18) | −0.084 (2) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.303 (4) | C9—H9B | 0.9700 |
| C1—C2 | 1.367 (5) | C10—N2 | 1.461 (4) |
| C1—Cl1 | 1.732 (4) | C10—H10A | 0.9700 |
| C2—C3 | 1.361 (5) | C10—H10B | 0.9700 |
| C2—H2 | 0.9300 | C11—C12 | 1.391 (4) |
| C3—C4 | 1.383 (5) | C11—N3 | 1.393 (4) |
| C3—H3 | 0.9300 | C11—C16 | 1.395 (4) |
| C4—C5 | 1.363 (5) | C12—C13 | 1.364 (4) |
| C4—C6 | 1.495 (5) | C12—N4 | 1.465 (4) |
| C5—N1 | 1.333 (5) | C13—C14 | 1.386 (4) |
| C5—H5 | 0.9300 | C13—H13 | 0.9300 |
| C6—N2 | 1.465 (4) | C14—C15 | 1.370 (4) |
| C6—H6A | 0.9700 | C14—C17 | 1.482 (5) |
| C6—H6B | 0.9700 | C15—C16 | 1.373 (4) |
| C7—N2 | 1.455 (4) | C15—H15 | 0.9300 |
| C7—C8 | 1.498 (5) | C16—N5 | 1.469 (4) |
| C7—H7A | 0.9700 | C17—F3 | 1.240 (13) |
| C7—H7B | 0.9700 | C17—F2 | 1.275 (14) |
| C8—N3 | 1.458 (4) | C17—F1 | 1.281 (14) |
| C8—H8A | 0.9700 | N4—O1 | 1.177 (4) |
| C8—H8B | 0.9700 | N4—O2 | 1.197 (4) |
| C9—N3 | 1.451 (4) | N5—O4 | 1.207 (4) |
| C9—C10 | 1.503 (4) | N5—O3 | 1.210 (4) |
| C9—H9A | 0.9700 | | |
| N1—C1—C2 | 124.7 (4) | C9—C10—H10A | 109.4 |
| N1—C1—Cl1 | 115.9 (3) | N2—C10—H10B | 109.4 |
| C2—C1—Cl1 | 119.4 (3) | C9—C10—H10B | 109.4 |
| C3—C2—C1 | 117.8 (3) | H10A—C10—H10B | 108.0 |
| C3—C2—H2 | 121.1 | C12—C11—N3 | 119.6 (2) |
| C1—C2—H2 | 121.1 | C12—C11—C16 | 113.4 (3) |
| C2—C3—C4 | 120.1 (3) | N3—C11—C16 | 126.8 (3) |
| C2—C3—H3 | 119.9 | C13—C12—C11 | 124.7 (3) |
| C4—C3—H3 | 119.9 | C13—C12—N4 | 117.0 (2) |

| | | | |
|---|---|---|---|
| C5—C4—C3 | 116.1 (3) | C11—C12—N4 | 118.1 (3) |
| C5—C4—C6 | 122.5 (3) | C12—C13—C14 | 118.8 (3) |
| C3—C4—C6 | 121.4 (3) | C12—C13—H13 | 120.6 |
| N1—C5—C4 | 125.2 (3) | C14—C13—H13 | 120.6 |
| N1—C5—H5 | 117.4 | C15—C14—C13 | 119.6 (3) |
| C4—C5—H5 | 117.4 | C15—C14—C17 | 120.2 (3) |
| N2—C6—C4 | 112.3 (3) | C13—C14—C17 | 120.2 (3) |
| N2—C6—H6A | 109.1 | C14—C15—C16 | 119.3 (3) |
| C4—C6—H6A | 109.1 | C14—C15—H15 | 120.3 |
| N2—C6—H6B | 109.1 | C16—C15—H15 | 120.3 |
| C4—C6—H6B | 109.1 | C15—C16—C11 | 124.0 (3) |
| H6A—C6—H6B | 107.9 | C15—C16—N5 | 116.2 (3) |
| N2—C7—C8 | 111.3 (3) | C11—C16—N5 | 119.8 (3) |
| N2—C7—H7A | 109.4 | F3—C17—F2 | 108.7 (13) |
| C8—C7—H7A | 109.4 | F3—C17—F1 | 107.3 (12) |
| N2—C7—H7B | 109.4 | F2—C17—F1 | 96.7 (13) |
| C8—C7—H7B | 109.4 | F3—C17—C14 | 117.3 (8) |
| H7A—C7—H7B | 108.0 | F2—C17—C14 | 111.8 (8) |
| N3—C8—C7 | 107.6 (3) | F1—C17—C14 | 113.1 (7) |
| N3—C8—H8A | 110.2 | C1—N1—C5 | 116.0 (3) |
| C7—C8—H8A | 110.2 | C7—N2—C10 | 109.8 (2) |
| N3—C8—H8B | 110.2 | C7—N2—C6 | 109.9 (3) |
| C7—C8—H8B | 110.2 | C10—N2—C6 | 110.4 (3) |
| H8A—C8—H8B | 108.5 | C11—N3—C9 | 119.1 (2) |
| N3—C9—C10 | 109.6 (2) | C11—N3—C8 | 120.4 (2) |
| N3—C9—H9A | 109.8 | C9—N3—C8 | 111.0 (2) |
| C10—C9—H9A | 109.8 | O1—N4—O2 | 123.7 (3) |
| N3—C9—H9B | 109.8 | O1—N4—C12 | 120.3 (3) |
| C10—C9—H9B | 109.8 | O2—N4—C12 | 116.0 (3) |
| H9A—C9—H9B | 108.2 | O4—N5—O3 | 125.7 (3) |
| N2—C10—C9 | 111.0 (2) | O4—N5—C16 | 117.0 (3) |
| N2—C10—H10A | 109.4 | O3—N5—C16 | 117.2 (3) |
| N1—C1—C2—C3 | −0.4 (5) | C13—C14—C17—F1 | 98.4 (12) |
| Cl1—C1—C2—C3 | −179.8 (3) | C15—C14—C17—F3' | −149.5 (4) |
| C1—C2—C3—C4 | −0.5 (5) | C13—C14—C17—F3' | 31.5 (5) |
| C2—C3—C4—C5 | 1.4 (5) | C15—C14—C17—F1' | −27.9 (5) |
| C2—C3—C4—C6 | −177.4 (3) | C13—C14—C17—F1' | 153.0 (4) |
| C3—C4—C5—N1 | −1.6 (5) | C15—C14—C17—F2' | 90.2 (4) |
| C6—C4—C5—N1 | 177.2 (3) | C13—C14—C17—F2' | −88.8 (4) |
| C5—C4—C6—N2 | −97.5 (4) | C2—C1—N1—C5 | 0.2 (5) |
| C3—C4—C6—N2 | 81.2 (4) | Cl1—C1—N1—C5 | 179.6 (3) |
| N2—C7—C8—N3 | 60.1 (4) | C4—C5—N1—C1 | 0.8 (5) |
| N3—C9—C10—N2 | −56.4 (3) | C8—C7—N2—C10 | −58.3 (4) |
| N3—C11—C12—C13 | 177.7 (3) | C8—C7—N2—C6 | −179.9 (3) |
| C16—C11—C12—C13 | 1.7 (4) | C9—C10—N2—C7 | 55.7 (3) |
| N3—C11—C12—N4 | 2.1 (4) | C9—C10—N2—C6 | 177.0 (3) |
| C16—C11—C12—N4 | −173.9 (3) | C4—C6—N2—C7 | −172.4 (3) |
| C11—C12—C13—C14 | −2.1 (5) | C4—C6—N2—C10 | 66.4 (4) |
| N4—C12—C13—C14 | 173.6 (3) | C12—C11—N3—C9 | 85.1 (3) |

| | | | |
|---|---|---|---|
| C12—C13—C14—C15 | −0.2 (4) | C16—C11—N3—C9 | −99.5 (3) |
| C12—C13—C14—C17 | 178.8 (3) | C12—C11—N3—C8 | −131.6 (3) |
| C13—C14—C15—C16 | 2.8 (4) | C16—C11—N3—C8 | 43.8 (4) |
| C17—C14—C15—C16 | −176.2 (3) | C10—C9—N3—C11 | −153.9 (2) |
| C14—C15—C16—C11 | −3.3 (5) | C10—C9—N3—C8 | 59.6 (3) |
| C14—C15—C16—N5 | 173.5 (3) | C7—C8—N3—C11 | 153.2 (3) |
| C12—C11—C16—C15 | 1.0 (4) | C7—C8—N3—C9 | −60.8 (3) |
| N3—C11—C16—C15 | −174.6 (3) | C13—C12—N4—O1 | 110.7 (4) |
| C12—C11—C16—N5 | −175.7 (3) | C11—C12—N4—O1 | −73.3 (4) |
| N3—C11—C16—N5 | 8.7 (5) | C13—C12—N4—O2 | −70.7 (4) |
| C15—C14—C17—F3 | 151.7 (12) | C11—C12—N4—O2 | 105.3 (4) |
| C13—C14—C17—F3 | −27.3 (13) | C15—C16—N5—O4 | −128.9 (3) |
| C15—C14—C17—F2 | 25.2 (13) | C11—C16—N5—O4 | 48.0 (4) |
| C13—C14—C17—F2 | −153.8 (12) | C15—C16—N5—O3 | 47.2 (4) |
| C15—C14—C17—F1 | −82.6 (12) | C11—C16—N5—O3 | −135.9 (3) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C8—H8A···O2$^{i}$ | 0.97 | 2.52 | 3.387 (5) | 149 |
| C13—H13···N1$^{ii}$ | 0.93 | 2.51 | 3.331 (4) | 147 |
| C15—H15···O2$^{iii}$ | 0.93 | 2.54 | 3.466 (4) | 178 |
| C5—H5···O4$^{iv}$ | 0.93 | 2.68 | 3.593 (5) | 166 |
| C3—H3···F3$^{v}$ | 0.93 | 2.62 | 3.370 (16) | 138 |

Symmetry codes: (i) −*x*+2, −*y*, −*z*+1; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) *x*+1, *y*, *z*; (v) *x*, *y*, *z*+1.

**Fig. 1**

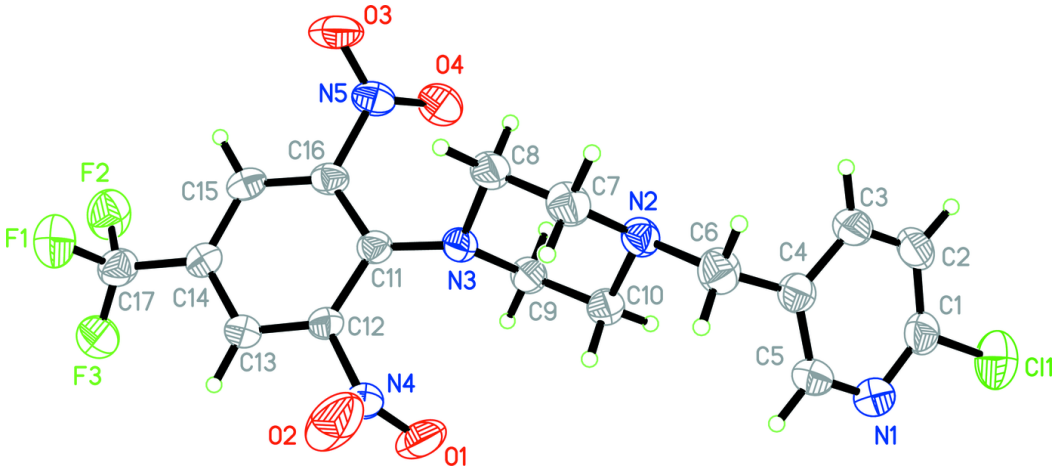
O3
O4
N5
C16
C8
C7
N2
C6
C3
C4
C2
C1
Cl1
C5
N1
F2
F1
C15
C17
C14
C11
N3
C9
C10
C12
C13
N4
O2
O1
F3

**Fig. 2**