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# *N*,*N*′-Bis[4-(trifluoromethyl)phenyl]-pyridine-2,6-dicarboxamide

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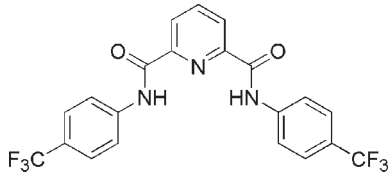
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.078; $wR$ factor = 0.181; data-to-parameter ratio = 10.4.

In the title molecule, $C_{21}H_{13}F_6N_3O_2$, the pyridine ring forms dihedral angles of 1.7 (1) and 5.2 (1)° with the two benzene rings. In the crystal structure, intermolecular N—H···O hydrogen bonds and $\pi\cdots\pi$ interactions [centroid–centroid distance of 3.628 (3) Å between aromatic rings] link molecules into stacks along the $c$ axis. The two trifluoromethyl groups are each rotationally disordered between two orientations, with occupancy ratios of 0.58 (1):0.42 (1) and 0.55 (1):0.45 (1).

## Related literature

For the synthesis and biological activity of acyl thiourea derivatives, see: Duan *et al.* (2003); Li *et al.* (2007).




## Experimental

### *Crystal data*

$C_{21}H_{13}F_6N_3O_2$
$M_r$ = 453.34
Monoclinic, $P2_1/c$
$a$ = 9.8308 (10) Å
$b$ = 23.787 (3) Å
$c$ = 8.9577 (10) Å
$\beta$ = 109.474 (2)°
$V$ = 1974.8 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.14 mm$^{-1}$
$T$ = 298 K
0.20 × 0.12 × 0.10 mm

### *Data collection*

Bruker SMART CCD area-detector diffractometer
12033 measured reflections
3653 independent reflections
3138 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.043

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.181
$S$ = 1.17
3653 reflections
351 parameters
96 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2$A$···O1$^{i}$ | 0.84 (3) | 2.58 (3) | 3.303 (4) | 144 (3) |
| N3—H3$A$···O2$^{ii}$ | 0.85 (4) | 2.36 (4) | 3.051 (3) | 139 (3) |

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2671).

## References

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Li, Y. J., Zhang, Z. G., Jin, K., Peng, Q. J., Ding, W. G. & Liu, J. (2007). *Acta Chim. Sin.* **65**, 834–840.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

*Acta Cryst.* (2010). E**66**, o405 [https://doi.org/10.1107/S1600536810001753]

## *N*,*N*′-Bis[4-(trifluoromethyl)phenyl]pyridine-2,6-dicarboxamide

**Le Chen, Hongwu He and Hao Peng**

### S1. Comment

Acyl thiourea derivatives have attracted considerable attention from chemists and biologists because of their wide range of biological activities and potential therapeutic value (Duan *et al.*, 2003; Li *et al.*, 2007). In our research work aimed at searching for novel agrochemicals, we attempted to synthesize the pyridine-2,6-dicarbonyl thiourea derivatives,and the title compound, (I), was obtained as byproduct. Here we report its crystal structure.

In (I) (Fig.1), the central pyridine ring makes the dihedral angles of 1.7 (1) and 5.2 (1)° with the two benzene rings, respectively. In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 1) and $\pi$–$\pi$ interactions proved by short distance of 3.628 (3) Å between the centroids of aromatic rings link molecules into stacks along axis *c*.

### S2. Experimental

Pyridine-2,6-dicarbonyl chloride (II) was prepared according to the literature procedures (Duan *et al.*, 2003). To a solution of (II) (5 mmol) in dichloromethane (30 ml) was added KSCN (15 mmol) and PEG-400 (0.2 g). The mixture was stirred at room temperature for 2 h, then 4-trifluoroaniline (10 mmol) was added and the suspension was stirred for 1 h. The mixture was filtered and the precipitate was washed with a little ethanol. The solvent was removed under reduced pressure and the residue was purified by recrystallization from DMF and water (20:1,*v*/*v*). Then recrystallization from actone over a period of one week gave colourless crystals of (I).

### S3. Refinement

C-bound H atoms were geometrically positioned (C—H 0.93 Å) and refined as riding, with $U_{iso}$(H) =1.2$U_{eq}$(C). H atoms bonded to N atoms were located at the difference map, and refined with bond restrains N—H = 0.84 (3) Å, and with constrains of $U_{iso}$(H) = 1.2$U_{eq}$(N). Two trifluoromethyl groups were treated as rotationally disordered between two orientations each with the ratios refined to 0.58 (1):0.42 (1) and 0.55 (1):0.45 (1), respectively.

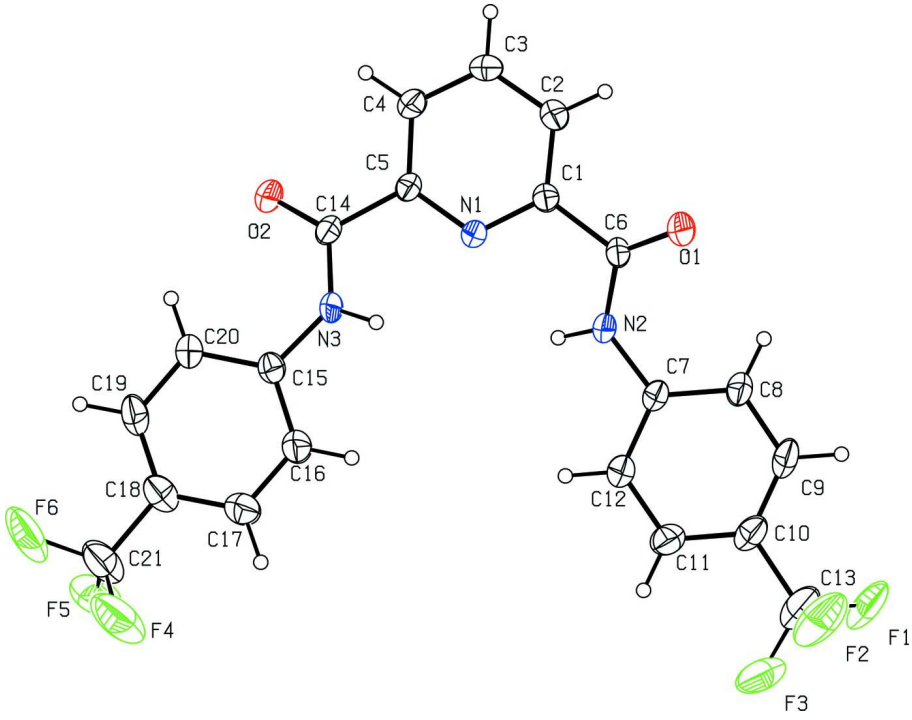


**Figure 1**

Molecular structure of (I), showing the labeling scheme with 50% probability displacement ellipsoids. Only major parts of disordered fluorine atoms are shown.

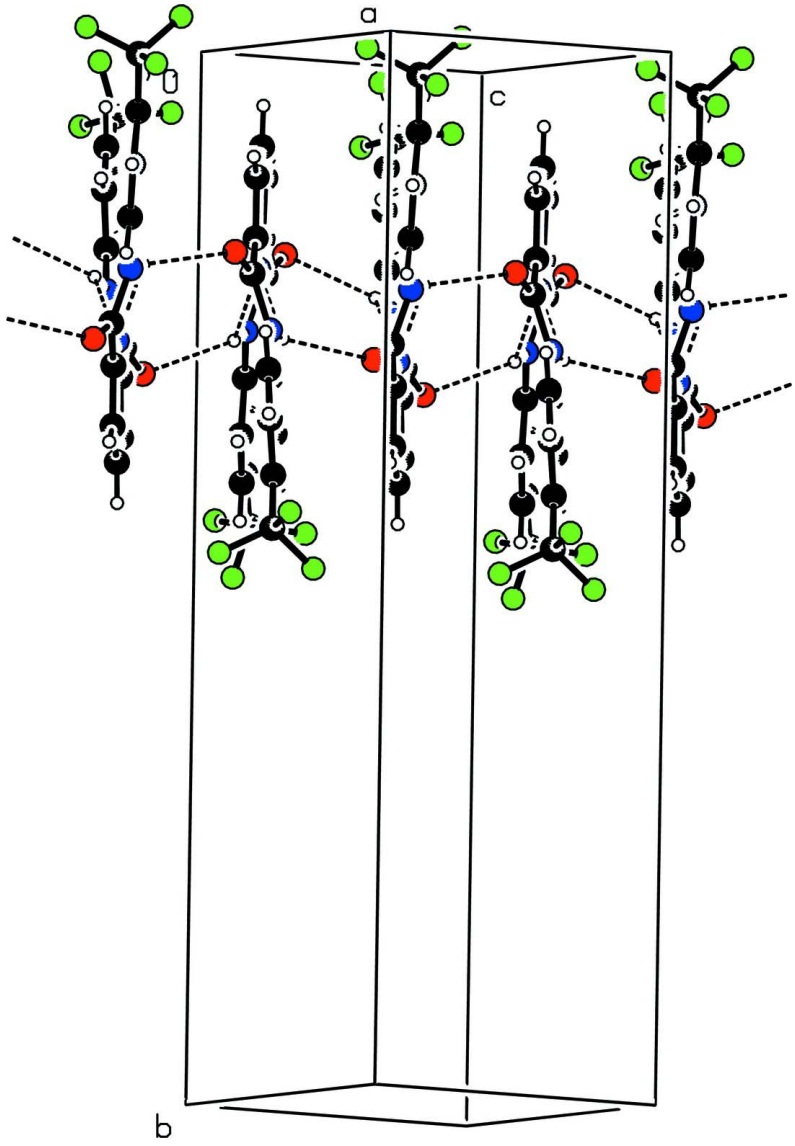


**Figure 2**

Part of the crystal packing, showing the intermolecular hydrogen bonds as dashed lines.

### *N*,*N*′-Bis[4-(trifluoromethyl)phenyl]pyridine-2,6-dicarboxamide

*Crystal data*

$C_{21}H_{13}F_6N_3O_2$
$M_r = 453.34$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.8308$ (10) Å
$b = 23.787$ (3) Å
$c = 8.9577$ (10) Å
$\beta = 109.474$ (2)°
$V = 1974.8$ (4) Å$^3$
$Z = 4$

$F(000) = 920$
$D_x = 1.525$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3607 reflections
$\theta = 2.4$–25.0°
$\mu = 0.14$ mm$^{-1}$
$T = 298$ K
Block, colourless
0.20 × 0.12 × 0.10 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and $\omega$ scans
12033 measured reflections
3653 independent reflections
3138 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.2°$
$h = -11 \rightarrow 11$
$k = -28 \rightarrow 22$
$l = -10 \rightarrow 10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.078$
$wR(F^2) = 0.181$
$S = 1.17$
3653 reflections
351 parameters
96 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 1.4024P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.25$ e Å$^{-3}$

*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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.1924 (3) | 0.17950 (12) | 0.0978 (3) | 0.0384 (7) | |
| C2 | 0.1982 (4) | 0.12169 (14) | 0.1004 (4) | 0.0491 (8) | |
| H2 | 0.1447 | 0.1009 | 0.1492 | 0.059* | |
| C3 | 0.2849 (4) | 0.09535 (14) | 0.0289 (4) | 0.0557 (9) | |
| H3 | 0.2902 | 0.0563 | 0.0282 | 0.067* | |
| C4 | 0.3637 (3) | 0.12687 (13) | −0.0413 (4) | 0.0461 (8) | |
| H4 | 0.4218 | 0.1097 | −0.0914 | 0.055* | |
| C5 | 0.3546 (3) | 0.18462 (12) | −0.0356 (3) | 0.0356 (6) | |
| C6 | 0.0990 (3) | 0.21071 (13) | 0.1728 (3) | 0.0400 (7) | |
| C7 | −0.0025 (3) | 0.30625 (13) | 0.1730 (3) | 0.0394 (7) | |
| C8 | −0.1031 (3) | 0.29432 (15) | 0.2475 (4) | 0.0498 (8) | |
| H8 | −0.1244 | 0.2573 | 0.2643 | 0.060* | |
| C9 | −0.1704 (3) | 0.33774 (17) | 0.2959 (4) | 0.0581 (9) | |
| H9 | −0.2373 | 0.3298 | 0.3458 | 0.070* | |
| C10 | −0.1409 (3) | 0.39243 (16) | 0.2721 (4) | 0.0565 (9) | |
| C11 | −0.0435 (4) | 0.40445 (15) | 0.1948 (5) | 0.0620 (10) | |

| | | | | | |
|---|---|---|---|---|---|
| H11 | −0.0236 | 0.4416 | 0.1772 | 0.074* | |
| C12 | 0.0234 (4) | 0.36158 (14) | 0.1444 (4) | 0.0535 (9) | |
| H12 | 0.0869 | 0.3698 | 0.0904 | 0.064* | |
| C13 | −0.2060 (4) | 0.4394 (2) | 0.3348 (5) | 0.0853 (14) | |
| C14 | 0.4430 (3) | 0.22150 (13) | −0.1048 (3) | 0.0377 (7) | |
| C15 | 0.5242 (3) | 0.32122 (13) | −0.0892 (3) | 0.0400 (7) | |
| C16 | 0.4878 (4) | 0.37330 (15) | −0.0475 (4) | 0.0564 (9) | |
| H16 | 0.4215 | 0.3760 | 0.0057 | 0.068* | |
| C17 | 0.5477 (4) | 0.42116 (16) | −0.0833 (5) | 0.0661 (10) | |
| H17 | 0.5200 | 0.4561 | −0.0567 | 0.079* | |
| C18 | 0.6488 (4) | 0.41772 (16) | −0.1585 (4) | 0.0599 (10) | |
| C19 | 0.6873 (4) | 0.36594 (17) | −0.1989 (4) | 0.0620 (10) | |
| H19 | 0.7560 | 0.3635 | −0.2492 | 0.074* | |
| C20 | 0.6258 (3) | 0.31734 (15) | −0.1662 (4) | 0.0491 (8) | |
| H20 | 0.6518 | 0.2825 | −0.1952 | 0.059* | |
| C21 | 0.7100 (6) | 0.4701 (2) | −0.2000 (5) | 0.0930 (16) | |
| N1 | 0.2702 (2) | 0.21094 (10) | 0.0320 (3) | 0.0357 (6) | |
| N2 | 0.0727 (3) | 0.26423 (11) | 0.1232 (3) | 0.0430 (6) | |
| H2A | 0.110 (4) | 0.2753 (14) | 0.057 (4) | 0.052* | |
| N3 | 0.4573 (3) | 0.27437 (11) | −0.0487 (3) | 0.0442 (7) | |
| H3A | 0.420 (4) | 0.2803 (14) | 0.022 (4) | 0.053* | |
| O1 | 0.0553 (3) | 0.18852 (10) | 0.2706 (3) | 0.0663 (7) | |
| O2 | 0.4942 (3) | 0.20366 (10) | −0.2017 (3) | 0.0566 (7) | |
| F1 | −0.3471 (7) | 0.4321 (5) | 0.3055 (10) | 0.104 (3) | 0.55 (1) |
| F2 | −0.1415 (10) | 0.4449 (5) | 0.4902 (8) | 0.130 (4) | 0.55 (1) |
| F3 | −0.1944 (10) | 0.4899 (3) | 0.2690 (12) | 0.127 (4) | 0.55 (1) |
| F4 | 0.7508 (11) | 0.5071 (4) | −0.0789 (9) | 0.143 (4) | 0.58 (1) |
| F5 | 0.6273 (14) | 0.4946 (5) | −0.3320 (9) | 0.122 (4) | 0.58 (1) |
| F6 | 0.8385 (8) | 0.4579 (5) | −0.2243 (14) | 0.133 (4) | 0.58 (1) |
| F4′ | 0.8527 (8) | 0.4711 (6) | −0.1667 (18) | 0.119 (5) | 0.45 (1) |
| F1′ | −0.3145 (10) | 0.4220 (6) | 0.3872 (14) | 0.130 (5) | 0.45 (1) |
| F5′ | 0.6697 (11) | 0.5178 (4) | −0.1386 (12) | 0.109 (3) | 0.45 (1) |
| F3′ | −0.1123 (11) | 0.4662 (5) | 0.4586 (11) | 0.088 (3) | 0.42 (1) |
| F2′ | −0.2616 (11) | 0.4801 (5) | 0.2255 (12) | 0.110 (4) | 0.42 (1) |
| F6′ | 0.6506 (17) | 0.4783 (6) | −0.3602 (9) | 0.102 (4) | 0.42 (1) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0394 (16) | 0.0387 (17) | 0.0408 (16) | −0.0035 (12) | 0.0182 (13) | −0.0019 (13) |
| C2 | 0.0565 (19) | 0.0434 (19) | 0.0562 (19) | −0.0060 (15) | 0.0305 (16) | −0.0004 (15) |
| C3 | 0.073 (2) | 0.0319 (17) | 0.072 (2) | 0.0011 (15) | 0.038 (2) | −0.0021 (16) |
| C4 | 0.0534 (18) | 0.0430 (18) | 0.0489 (18) | 0.0050 (14) | 0.0265 (15) | −0.0026 (14) |
| C5 | 0.0329 (14) | 0.0392 (16) | 0.0366 (15) | 0.0050 (12) | 0.0141 (12) | 0.0006 (12) |
| C6 | 0.0395 (16) | 0.0422 (18) | 0.0456 (17) | −0.0055 (13) | 0.0240 (14) | −0.0025 (13) |
| C7 | 0.0328 (15) | 0.0467 (18) | 0.0411 (16) | 0.0030 (13) | 0.0156 (13) | −0.0040 (13) |
| C8 | 0.0390 (17) | 0.055 (2) | 0.063 (2) | 0.0000 (15) | 0.0275 (16) | −0.0046 (16) |
| C9 | 0.0402 (18) | 0.079 (3) | 0.065 (2) | 0.0021 (17) | 0.0302 (17) | −0.0110 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.0404 (18) | 0.064 (2) | 0.066 (2) | 0.0063 (16) | 0.0181 (16) | −0.0184 (18) |
| C11 | 0.053 (2) | 0.048 (2) | 0.089 (3) | 0.0048 (16) | 0.029 (2) | −0.0085 (19) |
| C12 | 0.0492 (19) | 0.050 (2) | 0.071 (2) | 0.0047 (15) | 0.0340 (17) | 0.0023 (17) |
| C13 | 0.063 (3) | 0.097 (4) | 0.100 (4) | 0.010 (3) | 0.033 (3) | −0.036 (3) |
| C14 | 0.0383 (15) | 0.0421 (17) | 0.0366 (15) | 0.0093 (12) | 0.0176 (13) | 0.0027 (13) |
| C15 | 0.0410 (16) | 0.0450 (18) | 0.0356 (15) | −0.0033 (13) | 0.0148 (13) | 0.0016 (13) |
| C16 | 0.065 (2) | 0.052 (2) | 0.064 (2) | −0.0091 (17) | 0.0378 (19) | −0.0066 (17) |
| C17 | 0.084 (3) | 0.047 (2) | 0.076 (3) | −0.0112 (19) | 0.039 (2) | −0.0073 (18) |
| C18 | 0.069 (2) | 0.060 (2) | 0.051 (2) | −0.0187 (18) | 0.0202 (18) | 0.0026 (17) |
| C19 | 0.062 (2) | 0.077 (3) | 0.059 (2) | −0.0150 (19) | 0.0363 (19) | 0.0027 (19) |
| C20 | 0.0469 (18) | 0.054 (2) | 0.0533 (19) | −0.0017 (15) | 0.0264 (16) | 0.0008 (16) |
| C21 | 0.127 (5) | 0.078 (4) | 0.087 (4) | −0.039 (3) | 0.053 (4) | 0.003 (3) |
| N1 | 0.0353 (12) | 0.0357 (13) | 0.0395 (13) | 0.0021 (10) | 0.0172 (10) | 0.0003 (10) |
| N2 | 0.0471 (15) | 0.0428 (16) | 0.0516 (15) | 0.0024 (11) | 0.0330 (13) | 0.0022 (12) |
| N3 | 0.0548 (16) | 0.0436 (15) | 0.0489 (15) | −0.0040 (12) | 0.0371 (13) | −0.0027 (12) |
| O1 | 0.0879 (18) | 0.0535 (15) | 0.0839 (18) | 0.0034 (13) | 0.0640 (16) | 0.0105 (13) |
| O2 | 0.0733 (16) | 0.0520 (14) | 0.0635 (15) | 0.0044 (12) | 0.0482 (13) | −0.0026 (11) |
| F1 | 0.063 (4) | 0.126 (6) | 0.131 (6) | 0.029 (4) | 0.044 (4) | −0.042 (5) |
| F2 | 0.124 (7) | 0.159 (10) | 0.107 (5) | 0.023 (5) | 0.040 (5) | −0.073 (5) |
| F3 | 0.128 (8) | 0.071 (4) | 0.203 (11) | 0.015 (5) | 0.084 (7) | −0.041 (6) |
| F4 | 0.209 (10) | 0.102 (6) | 0.134 (6) | −0.089 (6) | 0.078 (7) | −0.024 (5) |
| F5 | 0.175 (7) | 0.070 (7) | 0.137 (7) | −0.004 (4) | 0.072 (6) | 0.036 (5) |
| F6 | 0.147 (7) | 0.129 (8) | 0.147 (8) | −0.095 (5) | 0.081 (5) | −0.024 (5) |
| F4′ | 0.115 (7) | 0.088 (7) | 0.157 (11) | −0.060 (5) | 0.049 (6) | −0.004 (7) |
| F1′ | 0.091 (7) | 0.134 (9) | 0.197 (12) | −0.011 (6) | 0.092 (7) | −0.084 (9) |
| F5′ | 0.168 (9) | 0.055 (4) | 0.127 (7) | −0.043 (5) | 0.082 (7) | −0.019 (5) |
| F3′ | 0.071 (4) | 0.078 (6) | 0.111 (7) | 0.003 (4) | 0.024 (5) | −0.050 (4) |
| F2′ | 0.097 (8) | 0.094 (8) | 0.118 (6) | 0.052 (6) | 0.007 (6) | −0.025 (6) |
| F6′ | 0.171 (10) | 0.067 (8) | 0.090 (6) | −0.008 (5) | 0.075 (6) | 0.022 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.339 (4) | C13—F3′ | 1.342 (8) |
| C1—C2 | 1.376 (4) | C13—F2′ | 1.355 (8) |
| C1—C6 | 1.502 (4) | C13—F3 | 1.358 (7) |
| C2—C3 | 1.376 (4) | C13—F1′ | 1.365 (8) |
| C2—H2 | 0.9300 | C14—O2 | 1.216 (3) |
| C3—C4 | 1.372 (4) | C14—N3 | 1.344 (4) |
| C3—H3 | 0.9300 | C15—C16 | 1.375 (5) |
| C4—C5 | 1.379 (4) | C15—C20 | 1.393 (4) |
| C4—H4 | 0.9300 | C15—N3 | 1.402 (4) |
| C5—N1 | 1.335 (3) | C16—C17 | 1.368 (5) |
| C5—C14 | 1.506 (4) | C16—H16 | 0.9300 |
| C6—O1 | 1.217 (3) | C17—C18 | 1.377 (5) |
| C6—N2 | 1.345 (4) | C17—H17 | 0.9300 |
| C7—C12 | 1.381 (5) | C18—C19 | 1.372 (5) |
| C7—C8 | 1.394 (4) | C18—C21 | 1.484 (5) |
| C7—N2 | 1.402 (4) | C19—C20 | 1.380 (5) |

| | | | |
|---|---|---|---|
| C8—C9 | 1.373 (5) | C19—H19 | 0.9300 |
| C8—H8 | 0.9300 | C20—H20 | 0.9300 |
| C9—C10 | 1.365 (5) | C21—F5 | 1.326 (8) |
| C9—H9 | 0.9300 | C21—F4′ | 1.334 (8) |
| C10—C11 | 1.386 (5) | C21—F4 | 1.350 (7) |
| C10—C13 | 1.488 (5) | C21—F6′ | 1.371 (8) |
| C11—C12 | 1.369 (5) | C21—F5′ | 1.375 (8) |
| C11—H11 | 0.9300 | C21—F6 | 1.383 (8) |
| C12—H12 | 0.9300 | N2—H2A | 0.84 (3) |
| C13—F2 | 1.330 (7) | N3—H3A | 0.85 (4) |
| C13—F1 | 1.335 (7) | | |

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| N1—C1—C2 | 122.6 (3) | F3′—C13—C10 | 113.9 (6) |
| N1—C1—C6 | 116.4 (2) | F2′—C13—C10 | 112.7 (7) |
| C2—C1—C6 | 120.9 (3) | F3—C13—C10 | 113.1 (6) |
| C1—C2—C3 | 118.4 (3) | F1′—C13—C10 | 112.7 (7) |
| C1—C2—H2 | 120.8 | O2—C14—N3 | 125.2 (3) |
| C3—C2—H2 | 120.8 | O2—C14—C5 | 121.6 (3) |
| C4—C3—C2 | 119.8 (3) | N3—C14—C5 | 113.2 (2) |
| C4—C3—H3 | 120.1 | C16—C15—C20 | 119.2 (3) |
| C2—C3—H3 | 120.1 | C16—C15—N3 | 117.3 (3) |
| C3—C4—C5 | 118.3 (3) | C20—C15—N3 | 123.5 (3) |
| C3—C4—H4 | 120.8 | C17—C16—C15 | 121.0 (3) |
| C5—C4—H4 | 120.8 | C17—C16—H16 | 119.5 |
| N1—C5—C4 | 122.8 (3) | C15—C16—H16 | 119.5 |
| N1—C5—C14 | 116.4 (2) | C16—C17—C18 | 120.2 (4) |
| C4—C5—C14 | 120.8 (3) | C16—C17—H17 | 119.9 |
| O1—C6—N2 | 125.0 (3) | C18—C17—H17 | 119.9 |
| O1—C6—C1 | 121.5 (3) | C19—C18—C17 | 119.3 (3) |
| N2—C6—C1 | 113.5 (2) | C19—C18—C21 | 121.2 (4) |
| C12—C7—C8 | 119.1 (3) | C17—C18—C21 | 119.4 (4) |
| C12—C7—N2 | 118.1 (3) | C18—C19—C20 | 121.1 (3) |
| C8—C7—N2 | 122.7 (3) | C18—C19—H19 | 119.4 |
| C9—C8—C7 | 119.5 (3) | C20—C19—H19 | 119.4 |
| C9—C8—H8 | 120.3 | C19—C20—C15 | 119.1 (3) |
| C7—C8—H8 | 120.3 | C19—C20—H20 | 120.4 |
| C10—C9—C8 | 121.2 (3) | C15—C20—H20 | 120.4 |
| C10—C9—H9 | 119.4 | F5—C21—F4′ | 118.5 (12) |
| C8—C9—H9 | 119.4 | F5—C21—F4 | 111.1 (7) |
| C9—C10—C11 | 119.6 (3) | F4′—C21—F4 | 78.5 (7) |
| C9—C10—C13 | 121.1 (3) | F5—C21—F6′ | 23.6 (8) |
| C11—C10—C13 | 119.3 (4) | F4′—C21—F6′ | 106.3 (8) |
| C12—C11—C10 | 119.9 (3) | F4—C21—F6′ | 131.1 (9) |
| C12—C11—H11 | 120.0 | F5—C21—F5′ | 79.7 (7) |
| C10—C11—H11 | 120.0 | F4′—C21—F5′ | 108.5 (7) |
| C11—C12—C7 | 120.7 (3) | F4—C21—F5′ | 35.8 (6) |
| C11—C12—H12 | 119.7 | F6′—C21—F5′ | 102.9 (7) |
| C7—C12—H12 | 119.7 | F5—C21—F6 | 105.8 (7) |

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| F2—C13—F1 | 108.7 (6) | F4′—C21—F6 | 24.6 (7) |
| F2—C13—F3′ | 29.8 (7) | F4—C21—F6 | 102.8 (6) |
| F1—C13—F3′ | 127.3 (8) | F6′—C21—F6 | 87.6 (10) |
| F2—C13—F2′ | 128.5 (8) | F5′—C21—F6 | 129.5 (7) |
| F1—C13—F2′ | 79.0 (7) | F5—C21—C18 | 114.4 (8) |
| F3′—C13—F2′ | 105.0 (7) | F4′—C21—C18 | 116.3 (8) |
| F2—C13—F3 | 106.7 (6) | F4—C21—C18 | 112.5 (6) |
| F1—C13—F3 | 105.2 (6) | F6′—C21—C18 | 108.3 (8) |
| F3′—C13—F3 | 78.8 (7) | F5′—C21—C18 | 113.5 (6) |
| F2′—C13—F3 | 29.3 (5) | F6—C21—C18 | 109.3 (6) |
| F2—C13—F1′ | 79.9 (7) | C5—N1—C1 | 118.1 (2) |
| F1—C13—F1′ | 31.5 (6) | C6—N2—C7 | 129.3 (3) |
| F3′—C13—F1′ | 105.0 (7) | C6—N2—H2A | 117 (2) |
| F2′—C13—F1′ | 106.9 (7) | C7—N2—H2A | 114 (2) |
| F3—C13—F1′ | 127.2 (8) | C14—N3—C15 | 130.2 (3) |
| F2—C13—C10 | 110.7 (6) | C14—N3—H3A | 115 (2) |
| F1—C13—C10 | 112.1 (6) | C15—N3—H3A | 115 (2) |

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| N1—C1—C2—C3 | −1.6 (5) | C4—C5—C14—N3 | −160.5 (3) |
| C6—C1—C2—C3 | 179.6 (3) | C20—C15—C16—C17 | 1.3 (5) |
| C1—C2—C3—C4 | 0.5 (5) | N3—C15—C16—C17 | −179.7 (3) |
| C2—C3—C4—C5 | 0.9 (5) | C15—C16—C17—C18 | −1.7 (6) |
| C3—C4—C5—N1 | −1.5 (5) | C16—C17—C18—C19 | 0.9 (6) |
| C3—C4—C5—C14 | 177.6 (3) | C16—C17—C18—C21 | 178.5 (4) |
| N1—C1—C6—O1 | −159.9 (3) | C17—C18—C19—C20 | 0.3 (6) |
| C2—C1—C6—O1 | 19.0 (5) | C21—C18—C19—C20 | −177.2 (4) |
| N1—C1—C6—N2 | 18.9 (4) | C18—C19—C20—C15 | −0.7 (5) |
| C2—C1—C6—N2 | −162.2 (3) | C16—C15—C20—C19 | 0.0 (5) |
| C12—C7—C8—C9 | −2.3 (5) | N3—C15—C20—C19 | −179.0 (3) |
| N2—C7—C8—C9 | 178.7 (3) | C19—C18—C21—F5 | 96.4 (7) |
| C7—C8—C9—C10 | 0.1 (5) | C17—C18—C21—F5 | −81.2 (7) |
| C8—C9—C10—C11 | 1.3 (5) | C19—C18—C21—F4′ | −47.7 (8) |
| C8—C9—C10—C13 | −175.6 (3) | C17—C18—C21—F4′ | 134.7 (8) |
| C9—C10—C11—C12 | −0.6 (5) | C19—C18—C21—F4 | −135.6 (6) |
| C13—C10—C11—C12 | 176.3 (4) | C17—C18—C21—F4 | 46.8 (7) |
| C10—C11—C12—C7 | −1.5 (5) | C19—C18—C21—F6′ | 71.8 (8) |
| C8—C7—C12—C11 | 3.0 (5) | C17—C18—C21—F6′ | −105.7 (7) |
| N2—C7—C12—C11 | −178.0 (3) | C19—C18—C21—F5′ | −174.6 (6) |
| C9—C10—C13—F2 | 75.5 (6) | C17—C18—C21—F5′ | 7.8 (7) |
| C11—C10—C13—F2 | −101.4 (6) | C19—C18—C21—F6 | −22.1 (7) |
| C9—C10—C13—F1 | −46.1 (7) | C17—C18—C21—F6 | 160.3 (6) |
| C11—C10—C13—F1 | 137.0 (6) | C4—C5—N1—C1 | 0.6 (4) |
| C9—C10—C13—F3′ | 107.6 (7) | C14—C5—N1—C1 | −178.6 (2) |
| C11—C10—C13—F3′ | −69.4 (7) | C2—C1—N1—C5 | 1.0 (4) |
| C9—C10—C13—F2′ | −133.0 (6) | C6—C1—N1—C5 | 179.9 (2) |
| C11—C10—C13—F2′ | 50.1 (7) | O1—C6—N2—C7 | 2.6 (5) |
| C9—C10—C13—F3 | −164.8 (6) | C1—C6—N2—C7 | −176.2 (3) |
| C11—C10—C13—F3 | 18.2 (7) | C12—C7—N2—C6 | 159.3 (3) |

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| C9—C10—C13—F1′ | −11.9 (7) | C8—C7—N2—C6 | −21.7 (5) |
| C11—C10—C13—F1′ | 171.2 (6) | O2—C14—N3—C15 | 3.4 (5) |
| N1—C5—C14—O2 | −161.2 (3) | C5—C14—N3—C15 | −176.4 (3) |
| C4—C5—C14—O2 | 19.7 (4) | C16—C15—N3—C14 | 161.7 (3) |
| N1—C5—C14—N3 | 18.6 (4) | C20—C15—N3—C14 | −19.3 (5) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*A*···O1$^{i}$ | 0.84 (3) | 2.58 (3) | 3.303 (4) | 144 (3) |
| N3—H3*A*···O2$^{ii}$ | 0.85 (4) | 2.36 (4) | 3.051 (3) | 139 (3) |

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