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# 5-Fluorouracil–dimethylformamide (2/1)

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**Key indicators**

Single-crystal X-ray study
$T$ = 150 K
Mean $\sigma$(C–C) = 0.003 Å
$R$ factor = 0.049
$wR$ factor = 0.108
Data-to-parameter ratio = 14.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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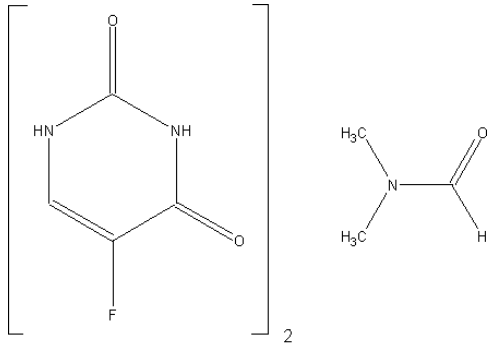
A solvate of 5-fluorouracil with dimethylformamide (DMF), $2C_4H_3FN_2O_2\cdot C_3H_7NO$, is reported. It crystallizes in the monoclinic space group $P2_1/n$, with two molecules of 5-fluorouracil and one molecule of DMF in the asymmetric unit. This solvate exhibits a sheet structure, with the DMF molecules present on both surfaces of the sheet and 5-fluorouracil molecules within the sheath of DMF molecules.

## Comment

In the course of a polymorph screen performed on 5-fluorouracil, three solvates were discovered. One of the solvate structures, (I), containing two independent molecules of 5-fluorouracil and one molecule of dimethyl formamide (DMF) in the asymmetric unit (Fig. 1), and crystallizing in the space group $P2_1/n$, is reported here.




(I)

The two 5-fluorouracil molecules in the asymmetric unit are linked to one another *via* N11—H11···O7 [N···O =

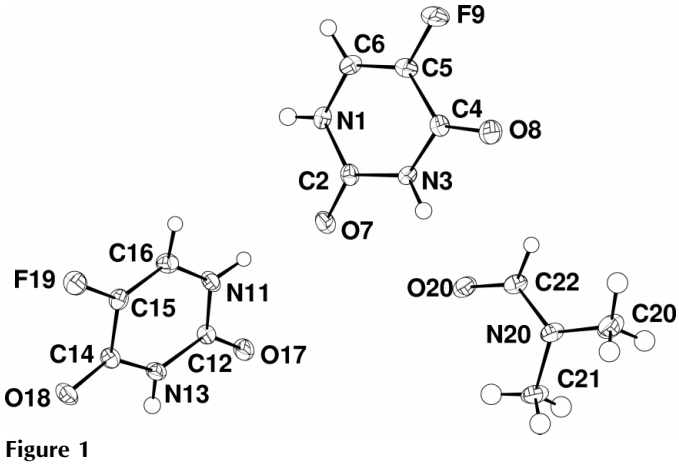



**Figure 1**
View (Watkin *et al.*, 1996) of the asymmetric unit of the title compound, with atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

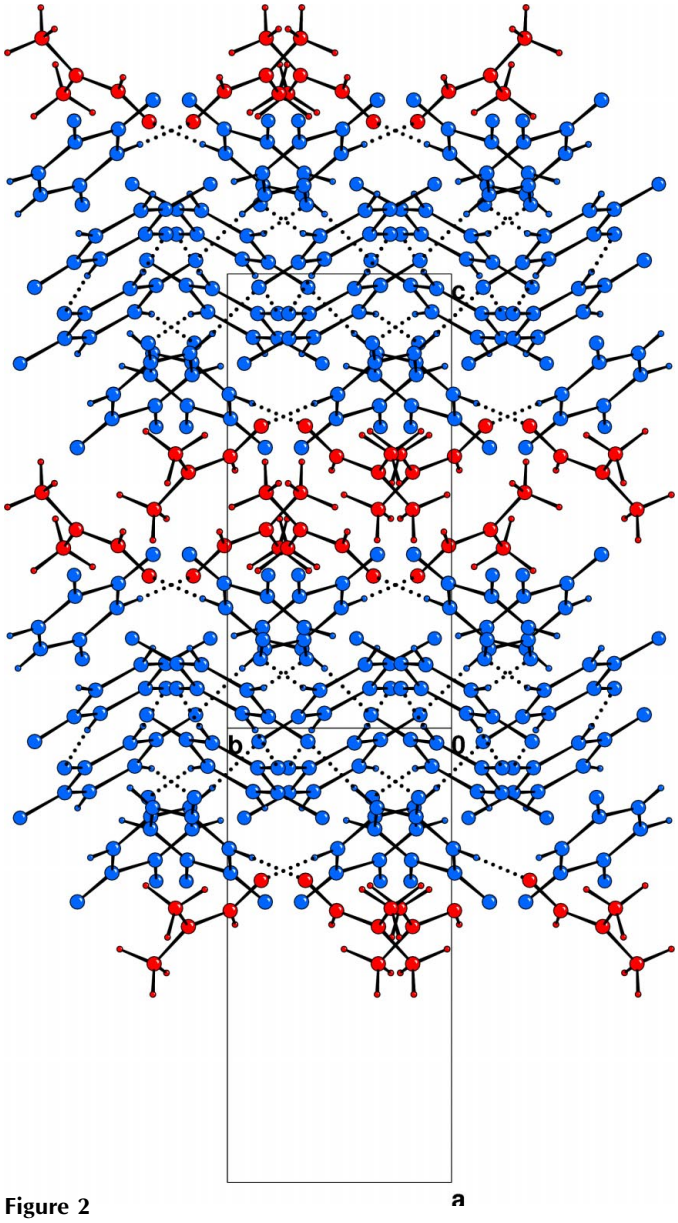



**Figure 2**
View parallel to the plane of two sheets of the structure. 5-Fluorouracil molecules are coloured blue, while the DMF molecules are red. Dashed lines indicate hydrogen bonds.

2.7962 (18) Å] hydrogen bonds. The DMF molecule forms a hydrogen bond to one of the 5-fluorouracil molecules in the asymmetric unit [N3—H3···O20, 2.7518 (19) Å]. A further two N—H···O hydrogen bonds link 5-fluorouracil molecules in the crystal structure; these bonds are N1—H1···O17$^{i}$ [N···O = 2.8205 (19) Å], and N13—H13···O18$^{ii}$ [N···O = 2.8203 (18) Å]. The N13—H13···O18$^{ii}$ hydrogen bonds link the 5-fluorouracil molecules into a centrosymmetric hydrogen-bonded dimer.

This solvate exhibits a sheet structure, in which the sheet has a discrete thickness of approximately 13.9 Å in the direction perpendicular to the plane of the sheet, and stacks parallel to the (10$\bar{1}$) planes. The DMF molecules are present on both surfaces of the sheet with 5-fluorouracil molecules within this sheath of DMF molecules. The parallel sheets

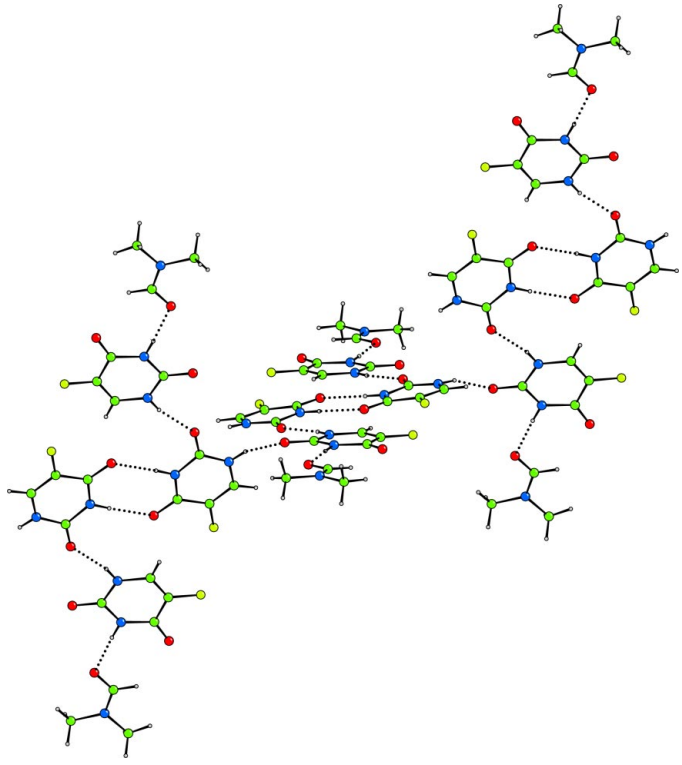


**Figure 3**
Three adjacent ribbons of a larger sheet, showing an alternating crossed orientation. Other ribbons stack parallel to each of the ribbons in the diagram. Dashed lines indicate hydrogen bonds.

approach each other closely, though there are no short intermolecular contacts between DMF molecules in adjacent sheets. Within the sheets the 5-fluorouracil molecules do not lie parallel to each other, but form a series of smaller blocks of parallel ribbons, as shown in Fig. 2. These ribbons are finite in length and are terminated by surface DMF molecules. Each of these ribbons is *ca* 26.8 Å long (Fig. 3).

## Experimental

5-Fluorouracil was obtained from the Aldrich Chemical Company Inc. The crystals were grown by vapour diffusion of diethyl ether into a saturated solution of 5-fluorouracil in dimethylformamide.

*Crystal data*

$2C_4H_3FN_2O_2\cdot C_3H_7NO$
$M_r$ = 333.26
Monoclinic, $P2_1/n$
$a$ = 14.7361 (18) Å
$b$ = 5.8693 (7) Å
$c$ = 16.397 (2) Å
$\beta$ = 100.524 (2)°
$V$ = 1394.3 (3) Å$^3$
$Z$ = 4
$D_x$ = 1.588 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 2752 reflections
$\theta$ = 2.5–27.7°
$\mu$ = 0.14 mm$^{-1}$
$T$ = 150 (2) K
Block, colourless
0.42 × 0.21 × 0.11 mm

*Data collection*

Bruker SMART APEX diffractometer
Narrow-frame $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.942, $T_{max}$ = 0.984
11 701 measured reflections
3331 independent reflections
2768 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
$\theta_{max}$ = 28.3°
$h = -19 \rightarrow 19$
$k = -7 \rightarrow 7$
$l = -21 \rightarrow 20$

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.5804P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.049$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $wR(F^2) = 0.108$ | $\Delta\rho_{max} = 0.29$ e Å$^{-3}$ |
| $S = 1.10$ | $\Delta\rho_{min} = -0.22$ e Å$^{-3}$ |
| 3331 reflections | |
| 238 parameters | |
| H atoms treated by a mixture of independent and constrained refinement | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O17$^{i}$ | 0.83 (2) | 2.01 (2) | 2.8205 (19) | 167 (2) |
| N3—H3···O20 | 0.85 (2) | 1.90 (2) | 2.7518 (19) | 176 (2) |
| N11—H11···O7 | 0.86 (2) | 1.97 (2) | 2.7962 (18) | 160 (2) |
| N13—H13···O18$^{ii}$ | 0.86 (2) | 1.96 (2) | 2.8203 (18) | 175 (2) |

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

The methyl H atoms were placed in geometrically idealized positions (C—H = 0.98 Å) and allowed to ride on their parent atoms with $U_{iso}(H) = 1.5U_{eq}(C)$. All other H atoms were located in a difference map and were refined isotropically; N—H and C—H distances were in the range 0.83 (2)–0.86 (2) and 0.94 (2)–0.97 (2) Å, respectively.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *SHELXL*97.

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## References

Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.

# supporting information

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## 5-Fluorouracil–dimethylformamide (2/1)

**Ashley T. Hulme and Derek A. Tocher**

### 5-Fluorouracil Dimethylformamide (2/1)

*Crystal data*

$2C_4H_3FN_2O_2·C_3H_7NO$
$M_r = 333.26$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 14.7361$ (18) Å
$b = 5.8693$ (7) Å
$c = 16.397$ (2) Å
$\beta = 100.524$ (2)°
$V = 1394.3$ (3) Å$^3$
$Z = 4$
$F(000) = 688$
$D_x = 1.588$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2752 reflections
$\theta = 2.5$–27.7°
$\mu = 0.14$ mm$^{-1}$
$T = 150$ K
Block, colourless
0.42 × 0.21 × 0.11 mm

*Data collection*

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ rotation with narrow frames scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.942$, $T_{max} = 0.984$
11701 measured reflections
3331 independent reflections
2768 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.7°$
$h = -19 \rightarrow 19$
$k = -7 \rightarrow 7$
$l = -21 \rightarrow 20$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.108$
$S = 1.10$
3331 reflections
238 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: found from delta F, methyl hydrogens placed using rigid rotor model
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.5804P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å$^{-3}$
$\Delta\rho_{min} = -0.22$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| F9 | 0.74489 (8) | 0.3172 (2) | 0.40798 (8) | 0.0457 (3) |
| O7 | 0.39619 (8) | 0.3532 (2) | 0.24355 (8) | 0.0250 (3) |
| O8 | 0.68162 (9) | 0.6691 (2) | 0.29920 (10) | 0.0418 (4) |
| N1 | 0.50826 (10) | 0.1673 (3) | 0.33347 (9) | 0.0226 (3) |
| H1 | 0.4706 (15) | 0.067 (4) | 0.3390 (13) | 0.035 (6)* |
| N3 | 0.53929 (10) | 0.5079 (2) | 0.27412 (9) | 0.0217 (3) |
| H3 | 0.5228 (14) | 0.610 (4) | 0.2378 (13) | 0.032 (6)* |
| C2 | 0.47601 (11) | 0.3415 (3) | 0.28135 (10) | 0.0193 (3) |
| C4 | 0.63096 (12) | 0.5142 (3) | 0.31233 (11) | 0.0259 (4) |
| C5 | 0.65670 (12) | 0.3215 (3) | 0.36632 (11) | 0.0280 (4) |
| C6 | 0.59742 (13) | 0.1573 (3) | 0.37572 (11) | 0.0257 (4) |
| H6 | 0.6136 (13) | 0.031 (4) | 0.4103 (12) | 0.031 (5)* |
| F19 | 0.24134 (7) | −0.42399 (17) | 0.04386 (6) | 0.0274 (3) |
| O17 | 0.13892 (8) | 0.3590 (2) | 0.16837 (7) | 0.0242 (3) |
| O18 | 0.07207 (8) | −0.2263 (2) | −0.01531 (7) | 0.0260 (3) |
| N11 | 0.24840 (9) | 0.0874 (2) | 0.16475 (9) | 0.0209 (3) |
| H11 | 0.2885 (13) | 0.162 (3) | 0.1997 (12) | 0.024 (5)* |
| N13 | 0.10821 (9) | 0.0668 (2) | 0.07569 (9) | 0.0198 (3) |
| H13 | 0.0542 (15) | 0.124 (3) | 0.0582 (13) | 0.032 (6)* |
| C12 | 0.16439 (11) | 0.1830 (3) | 0.13902 (10) | 0.0193 (3) |
| C14 | 0.12779 (11) | −0.1352 (3) | 0.04047 (10) | 0.0196 (3) |
| C15 | 0.21743 (11) | −0.2249 (3) | 0.07521 (10) | 0.0196 (3) |
| C16 | 0.27456 (11) | −0.1152 (3) | 0.13482 (10) | 0.0222 (4) |
| H16 | 0.3353 (13) | −0.167 (3) | 0.1594 (12) | 0.026 (5)* |
| O20 | 0.49362 (8) | 0.8486 (2) | 0.15864 (8) | 0.0279 (3) |
| N20 | 0.54350 (10) | 1.1737 (2) | 0.10675 (9) | 0.0232 (3) |
| C20 | 0.61968 (13) | 1.3266 (3) | 0.10100 (13) | 0.0342 (4) |
| H20A | 0.6758 | 1.2713 | 0.1370 | 0.051* |
| H20B | 0.6051 | 1.4797 | 0.1186 | 0.051* |
| H20C | 0.6295 | 1.3318 | 0.0435 | 0.051* |
| C21 | 0.45396 (12) | 1.2308 (3) | 0.05755 (11) | 0.0304 (4) |
| H21A | 0.4120 | 1.1010 | 0.0569 | 0.046* |
| H21B | 0.4611 | 1.2666 | 0.0007 | 0.046* |
| H21C | 0.4285 | 1.3633 | 0.0819 | 0.046* |
| C22 | 0.55522 (12) | 0.9873 (3) | 0.15346 (10) | 0.0224 (4) |

| | | | | |
|---|---|---|---|---|
| H22 | 0.6169 (13) | 0.966 (3) | 0.1847 (12) | 0.028 (5)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F9 | 0.0267 (6) | 0.0442 (7) | 0.0568 (8) | −0.0046 (5) | −0.0171 (5) | 0.0188 (6) |
| O7 | 0.0196 (6) | 0.0247 (6) | 0.0289 (6) | −0.0033 (5) | −0.0005 (5) | −0.0026 (5) |
| O8 | 0.0291 (7) | 0.0347 (8) | 0.0558 (9) | −0.0127 (6) | −0.0073 (6) | 0.0173 (7) |
| N1 | 0.0245 (7) | 0.0172 (7) | 0.0256 (8) | −0.0045 (6) | 0.0033 (6) | 0.0015 (6) |
| N3 | 0.0222 (7) | 0.0170 (7) | 0.0240 (7) | −0.0026 (6) | −0.0010 (6) | 0.0058 (6) |
| C2 | 0.0220 (8) | 0.0180 (8) | 0.0182 (8) | −0.0018 (6) | 0.0045 (6) | −0.0038 (6) |
| C4 | 0.0227 (9) | 0.0225 (9) | 0.0302 (9) | −0.0047 (7) | −0.0011 (7) | 0.0025 (7) |
| C5 | 0.0225 (9) | 0.0279 (9) | 0.0298 (9) | −0.0010 (7) | −0.0050 (7) | 0.0054 (8) |
| C6 | 0.0305 (9) | 0.0201 (9) | 0.0246 (9) | 0.0019 (7) | 0.0003 (7) | 0.0047 (7) |
| F19 | 0.0264 (5) | 0.0233 (5) | 0.0315 (6) | 0.0062 (4) | 0.0028 (4) | −0.0078 (4) |
| O17 | 0.0244 (6) | 0.0196 (6) | 0.0265 (6) | 0.0022 (5) | −0.0005 (5) | −0.0063 (5) |
| O18 | 0.0218 (6) | 0.0260 (7) | 0.0266 (6) | 0.0025 (5) | −0.0048 (5) | −0.0097 (5) |
| N11 | 0.0173 (7) | 0.0211 (7) | 0.0223 (7) | −0.0017 (6) | −0.0015 (6) | −0.0051 (6) |
| N13 | 0.0164 (7) | 0.0201 (7) | 0.0210 (7) | 0.0032 (5) | −0.0015 (5) | −0.0022 (6) |
| C12 | 0.0198 (8) | 0.0183 (8) | 0.0187 (8) | −0.0017 (6) | 0.0008 (6) | −0.0005 (6) |
| C14 | 0.0200 (8) | 0.0204 (8) | 0.0183 (8) | −0.0010 (6) | 0.0031 (6) | −0.0023 (6) |
| C15 | 0.0216 (8) | 0.0165 (8) | 0.0212 (8) | 0.0015 (6) | 0.0052 (6) | −0.0020 (6) |
| C16 | 0.0178 (8) | 0.0244 (9) | 0.0239 (8) | 0.0027 (6) | 0.0025 (7) | 0.0012 (7) |
| O20 | 0.0271 (6) | 0.0235 (6) | 0.0321 (7) | 0.0006 (5) | 0.0025 (5) | 0.0098 (5) |
| N20 | 0.0231 (7) | 0.0218 (7) | 0.0247 (7) | 0.0044 (6) | 0.0046 (6) | 0.0064 (6) |
| C20 | 0.0280 (10) | 0.0291 (10) | 0.0459 (12) | 0.0015 (8) | 0.0080 (8) | 0.0141 (9) |
| C21 | 0.0285 (9) | 0.0316 (10) | 0.0291 (10) | 0.0062 (8) | 0.0002 (8) | 0.0117 (8) |
| C22 | 0.0236 (9) | 0.0214 (9) | 0.0224 (8) | 0.0057 (7) | 0.0048 (7) | 0.0029 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F9—C5 | 1.353 (2) | N13—C14 | 1.372 (2) |
| O7—C2 | 1.227 (2) | N13—C12 | 1.384 (2) |
| O8—C4 | 1.220 (2) | N13—H13 | 0.86 (2) |
| N1—C2 | 1.361 (2) | C14—C15 | 1.439 (2) |
| N1—C6 | 1.370 (2) | C15—C16 | 1.333 (2) |
| N1—H1 | 0.83 (2) | C16—H16 | 0.963 (19) |
| N3—C2 | 1.370 (2) | O20—C22 | 1.234 (2) |
| N3—C4 | 1.382 (2) | N20—C22 | 1.328 (2) |
| N3—H3 | 0.85 (2) | N20—C20 | 1.454 (2) |
| C4—C5 | 1.444 (2) | N20—C21 | 1.454 (2) |
| C5—C6 | 1.329 (3) | C20—H20A | 0.98 |
| C6—H6 | 0.94 (2) | C20—H20B | 0.98 |
| F19—C15 | 1.3491 (19) | C20—H20C | 0.98 |
| O17—C12 | 1.227 (2) | C21—H21A | 0.98 |
| O18—C14 | 1.2340 (19) | C21—H21B | 0.98 |
| N11—C12 | 1.354 (2) | C21—H21C | 0.98 |
| N11—C16 | 1.369 (2) | C22—H22 | 0.967 (19) |

| | | | |
|---|---|---|---|
| N11—H11 | 0.86 (2) | | |
| | | | |
| C2—N1—C6 | 122.85 (15) | O18—C14—N13 | 121.60 (15) |
| C2—N1—H1 | 116.2 (15) | O18—C14—C15 | 124.96 (15) |
| C6—N1—H1 | 120.9 (15) | N13—C14—C15 | 113.45 (14) |
| C2—N3—C4 | 127.17 (15) | C16—C15—F19 | 121.59 (15) |
| C2—N3—H3 | 116.8 (14) | C16—C15—C14 | 121.62 (15) |
| C4—N3—H3 | 115.5 (14) | F19—C15—C14 | 116.79 (14) |
| O7—C2—N1 | 123.49 (15) | C15—C16—N11 | 120.07 (15) |
| O7—C2—N3 | 121.39 (15) | C15—C16—H16 | 124.6 (11) |
| N1—C2—N3 | 115.12 (14) | N11—C16—H16 | 115.3 (11) |
| O8—C4—N3 | 121.27 (16) | C22—N20—C20 | 121.68 (15) |
| O8—C4—C5 | 126.24 (16) | C22—N20—C21 | 121.25 (15) |
| N3—C4—C5 | 112.49 (15) | C20—N20—C21 | 117.06 (14) |
| C6—C5—F9 | 121.25 (16) | N20—C20—H20A | 109.5 |
| C6—C5—C4 | 122.34 (16) | N20—C20—H20B | 109.5 |
| F9—C5—C4 | 116.41 (15) | H20A—C20—H20B | 109.5 |
| C5—C6—N1 | 120.00 (16) | N20—C20—H20C | 109.5 |
| C5—C6—H6 | 123.0 (12) | H20A—C20—H20C | 109.5 |
| N1—C6—H6 | 117.0 (12) | H20B—C20—H20C | 109.5 |
| C12—N11—C16 | 123.32 (14) | N20—C21—H21A | 109.5 |
| C12—N11—H11 | 118.2 (13) | N20—C21—H21B | 109.5 |
| C16—N11—H11 | 118.4 (13) | H21A—C21—H21B | 109.5 |
| C14—N13—C12 | 126.76 (14) | N20—C21—H21C | 109.5 |
| C14—N13—H13 | 116.6 (14) | H21A—C21—H21C | 109.5 |
| C12—N13—H13 | 116.6 (14) | H21B—C21—H21C | 109.5 |
| O17—C12—N11 | 123.64 (15) | O20—C22—N20 | 124.28 (16) |
| O17—C12—N13 | 121.68 (15) | O20—C22—H22 | 120.6 (12) |
| N11—C12—N13 | 114.68 (14) | N20—C22—H22 | 115.1 (12) |
| | | | |
| C6—N1—C2—O7 | −179.53 (16) | C16—N11—C12—N13 | −3.9 (2) |
| C6—N1—C2—N3 | −0.2 (2) | C14—N13—C12—O17 | −177.25 (16) |
| C4—N3—C2—O7 | −179.27 (16) | C14—N13—C12—N11 | 3.1 (2) |
| C4—N3—C2—N1 | 1.3 (3) | C12—N13—C14—O18 | 179.08 (16) |
| C2—N3—C4—O8 | 177.84 (18) | C12—N13—C14—C15 | −0.7 (2) |
| C2—N3—C4—C5 | −1.7 (3) | O18—C14—C15—C16 | 179.13 (17) |
| O8—C4—C5—C6 | −178.5 (2) | N13—C14—C15—C16 | −1.1 (2) |
| N3—C4—C5—C6 | 1.1 (3) | O18—C14—C15—F19 | −0.1 (3) |
| O8—C4—C5—F9 | 2.1 (3) | N13—C14—C15—F19 | 179.71 (13) |
| N3—C4—C5—F9 | −178.35 (16) | F19—C15—C16—N11 | 179.46 (14) |
| F9—C5—C6—N1 | 179.27 (17) | C14—C15—C16—N11 | 0.3 (3) |
| C4—C5—C6—N1 | −0.1 (3) | C12—N11—C16—C15 | 2.4 (3) |
| C2—N1—C6—C5 | −0.4 (3) | C20—N20—C22—O20 | −177.80 (17) |
| C16—N11—C12—O17 | 176.43 (16) | C21—N20—C22—O20 | 0.8 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O17$^{i}$ | 0.83 (2) | 2.01 (2) | 2.8205 (19) | 167 (2) |
| N3—H3···O20 | 0.85 (2) | 1.90 (2) | 2.7518 (19) | 176 (2) |
| N11—H11···O7 | 0.86 (2) | 1.97 (2) | 2.7962 (18) | 160 (2) |
| N13—H13···O18$^{ii}$ | 0.86 (2) | 1.96 (2) | 2.8203 (18) | 175 (2) |

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