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# 2-{2-[4-(4-Fluorophenyl)piperazin-1-yl]-2-oxoethyl}-6-(morpholin-4-yl)-4-phenylpyridazin-3(2*H*)-one

**Abdullah Aydın,[a]* Murat Şüküroğlu,[b] Mehmet Akkurt[c] and Orhan Büyükgüngör[d]**

[a]Department of Science Education, Faculty of Education, Kastamonu University, 37200 Kastamonu, Turkey, [b]Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Gazi University, 06330 Ankara, Turkey, [c]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, and [d]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey
Correspondence e-mail: aaydin@kastamonu.edu.tr

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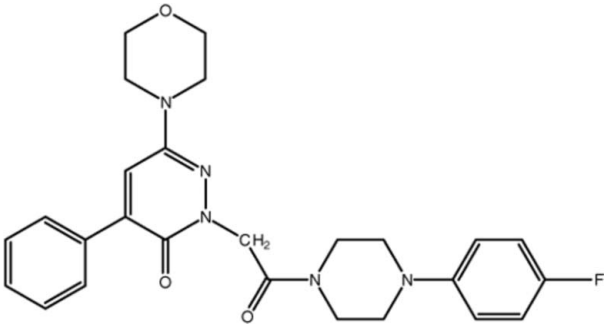
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.036; $wR$ factor = 0.091; data-to-parameter ratio = 15.4.

In the title compound, $C_{26}H_{28}FN_5O_3$, the morpholine ring adopts a chair conformation. The piperazine ring is puckered [$Q_T$ = 0.5437 (15) Å, $\theta$ = 8.89 (15) and $\varphi$ = 357.2 (11)°]. The 1,6-dihydropyridazine ring makes dihedral angles of 28.03 (7) and 77.46 (7)° with the phenyl and benzene rings, respectively. In the crystal, molecules are linked along the $c$ axis by C—H···O interactions and are flattened parallel to the $ac$ plane. C–H···$\pi$ interactions also contribute to the stability of the structure.

## Related literature

For the pharmacological effects, biological activity and synthesis of 3(2*H*)-pyridazinones, see: Şüküroğlu *et al.* 2006; Brogden 1986. For bond-length data, see: Allen *et al.* (1987). For ring conformational analysis, see: Cremer & Pople (1975). For the quantum mechanical *CNDO*/2 approximation, see: Pople & Beveridge (1970).



## Experimental

### Crystal data

$C_{26}H_{28}FN_5O_3$
$M_r$ = 477.53
Triclinic, $P\overline{1}$
$a$ = 8.9168 (5) Å
$b$ = 10.7106 (6) Å
$c$ = 13.5147 (8) Å
$\alpha$ = 73.489 (4)°
$\beta$ = 71.309 (4)°
$\gamma$ = 83.486 (4)°
$V$ = 1171.87 (12) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 296 K
0.60 × 0.49 × 0.20 mm

### Data collection

STOE IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min}$ = 0.945, $T_{max}$ = 0.981
13273 measured reflections
4861 independent reflections
3479 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.091
$S$ = 1.03
4861 reflections
316 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

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

*Cg*2, *Cg*4 and *Cg*5 are the centroids of the N1/N2/C7–C10, C1–C6 and C21–C26 rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C11—H11$B$···O3$^{i}$ | 0.97 | 2.41 | 3.3306 (18) | 159 |
| C5—H5···$Cg5^{ii}$ | 0.93 | 2.86 | 3.4941 (18) | 127 |
| C13—H13$B$···$Cg4^{i}$ | 0.97 | 2.92 | 3.7395 (19) | 143 |
| C18—H18$A$···$Cg2^{iii}$ | 0.97 | 2.73 | 3.5079 (16) | 138 |

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2227).

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# supporting information

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# 2-{2-[4-(4-Fluorophenyl)piperazin-1-yl]-2-oxoethyl}-6-(morpholin-4-yl)-4-phenylpyridazin-3(2*H*)-one

**Abdullah Aydın, Murat Şüküroğlu, Mehmet Akkurt and Orhan Büyükgüngör**

## S1. Comment

In recent years, the 3(2*H*)-pyridazinone system has aroused a great deal of attention due to its structural relationship to pyrazolone derivatives such as aminopyrine and dipyrone in view of the ring enlargement of pyrazolone to pyridazinone. These drugs possess analgesic and anti-inflammatory activities although they have limitations for their clinical use due to serious side effects such as blood dyscrasias (Şüküroğlu *et al.*, 2006; Brogden, 1986).

A series of 6-morpholino-4-aryl-3(2*H*)-pyridazinone alkanoic acids, their ester and amide derivatives were prepared and tested for their *in vivo* analgesic activity by using the *p*-benzoquinone-induced writhing test. The title compound, $C_{26}H_{28}FN_5O_3$, generally showed higher activity but caused gastric ulceration in the animals (Şüküroğlu *et al.*, 2006).

In the title molecule (I), Fig. 1, the morpholine ring (N3/O2/C11–C14) adopts a chair conformation. The piperazine ring (N4/N5/C17–C20) is puckered. The conformation of this ring is described by three puckering parameters: $Q_T$ = 0.5437 (15) Å, $\theta$ = 8.89 (15) ° and, $\varphi$ = 357.2 (11) ° (Cremer & Pople, 1975). The 1,6-dihydropyridazine ring (N1/N2/C7–C10) makes dihedral angles of 28.03 (7) and 77.46 (7) ° with the C1–C6 phenyl and C21–C26 benzene rings, respectively. The phenyl and benzene rings make a dihedral angle of 50.17 (8) ° with each other. In the crystal structure, molecules are linked along the *c*-axis direction and are flattened parallel to the plane containing the a and c axes. Furthermore, C–H···$\pi$ interactions contribute to the stability of the structure (Table 1). Fig. 2 shows the packing diagram of (I) down the *b* axis.

Theoretical calculations were carried out using the semiempirical quantum-mechanical *CNDO*/2 (**C**omplete **N**eglect of **D**ifferential **O**verlap) method (Pople and Beveridge, 1970). The spatial view of the single molecule calculated as closed-shell in a vacuum is shown in Fig. 3 with atomic labels. The calculated dipole moment of (I) is about 2.795 Debye. The *HOMO* and *LUMO* energy levels are -10.013 and 0.832 eV, respectively.

According to the theoretical *CNDO/2* and experimental X-ray structural results, the values of the geometric parameters of (I) are almost comparable within the experimental error interval (Allen *et al.*, 1987).

The 1,6-dihydropyridazine ring (N1/N2/C7–C10) forms dihedral angles of 2.24 and 60.48° with the C1–C6 phenyl and C21–C26 benzene rings, respectively. The dihedral angle between the phenyl and benzene rings is 62.62°. The orientations of the planes of the rings are however, slightly different in the *CNDO*/2 and X-ray results. That is, intermolecular interactions play an important role in determining the crystal state conformation of (I).

## S2. Experimental

A reaction mixture containing 2-[4-phenyl-6-(morpholin-4-yl)-3(2*H*)- pyridazinone-2-yl]acetic acid (0.01 mole) and tri-ethylamine (0.011 mole) in 20 ml dichloromethane at 273 K (ice-bath) was treated with ethyl chloroformate (0,01 mole). After stirring the reaction mixture at 273 K for 15 min, 0.011 mole of 4-(4-fluorophenyl)-piperazine derivative was added to this solution. The final mixture was stirred at room temperature for 24 h and evaporated to dryness and then acetone

was added. All undissolved salts were filtered off, the filtrate was evaporated to dryness and the residue was recrystallized from acetone-water (1:1) to yield 62%, [m.p.: 457 K].

$^1$H-NMR ($CDCl_3$), $\delta$ 7.75 (m, 2H, phenyl-H2, H6), 7.43 (m, 3H, phenyl-H3, H4, H5), 7.23 (s, 1H, pyridazinone-H5), 6.99 (m, 4H, 4-fluorophenyl-H2, H3, H5, H6), 4.98 (s, 2H, N—$CH_2$—CO), 3.82 (m, 6H, morpholine-H2, H6, piperazine-H2(6)), 3.71 (m, 2H, piperazine-H6(2)), 3.31 (t, 8H, morpholine-H3, H5), 3.15 (m, 4H, piperazine-H3, H5) p.p.m.. IR $\nu_{max}$ cm$^{-1}$ (KBr): 2845, 1661, 1643. Anal. C, H, N ($C_{26}H_{28}FN_5O_3$) (Şüküroğlu *et al.*, 2006). Elemental analysis: $C_{26}H_{28}FN_5O_3$, Calc.(%) / Found (%): C: 65.39/65.54, H: 5.91/5.49, N: 14.67/14.28.

## S3. Refinement

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 and 0.97 Å, and $U_{iso}$(H) = 1.2$U_{eq}$(C).

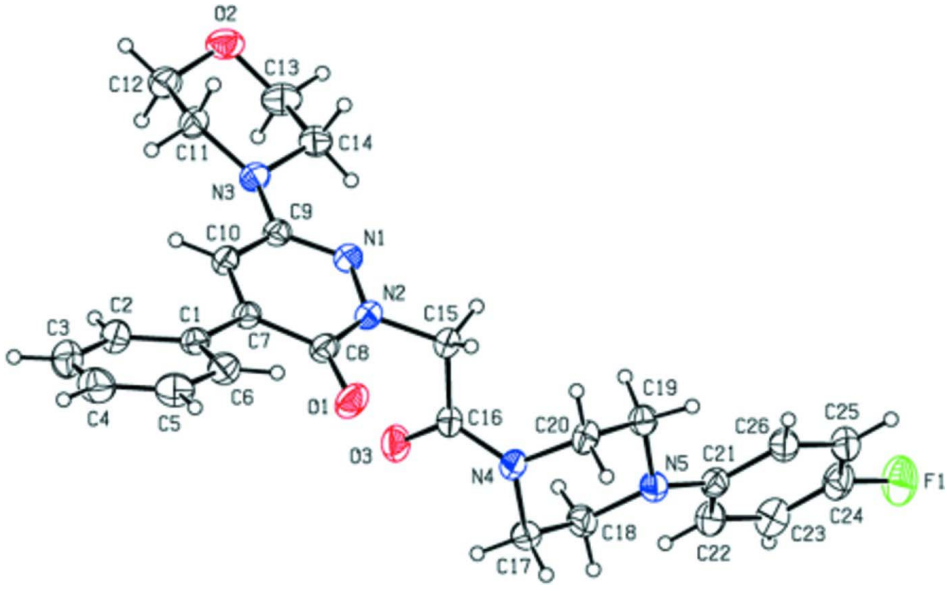


**Figure 1**

View of the title molecule with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

**Figure 2**

The packing and hydrogen bonding interactions of (I) down the *b* axis. H atoms not participating in hydrogen bonding have been omitted for clarity.

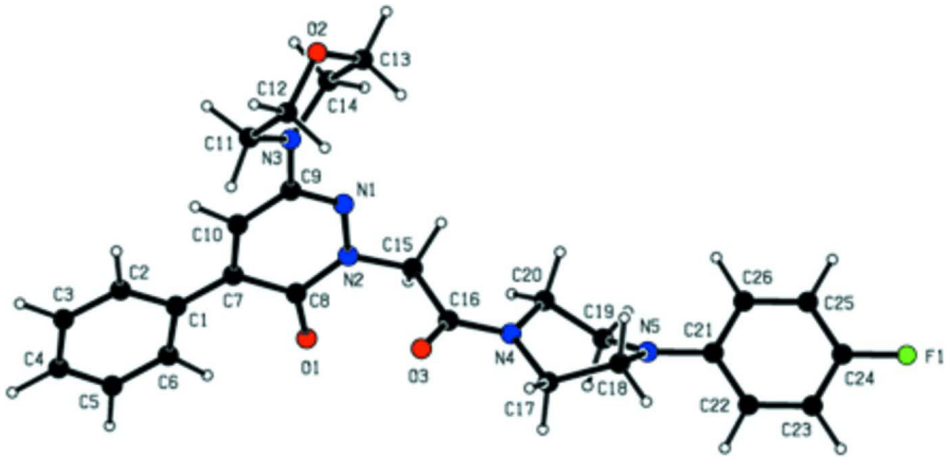


**Figure 3**

The spatial view of the title molecule (I), calculated by the *CNDO*/2 aproximation.

**2-{2-[4-(4-Fluorophenyl)piperazin-1-yl]-2-oxoethyl}-6-(morpholin-4-yl)- 4-phenylpyridazin-3(2*H*)-one**

*Crystal data*

$C_{26}H_{28}FN_5O_3$
$M_r$ = 477.53
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a$ = 8.9168 (5) Å
$b$ = 10.7106 (6) Å
$c$ = 13.5147 (8) Å
$\alpha$ = 73.489 (4)°
$\beta$ = 71.309 (4)°
$\gamma$ = 83.486 (4)°
$V$ = 1171.87 (12) Å$^3$
$Z$ = 2
$F$(000) = 504
$D_x$ = 1.353 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 19046 reflections
$\theta$ = 1.7–28.2°
$\mu$ = 0.10 mm$^{-1}$
$T$ = 296 K
Prism, yellow
0.60 × 0.49 × 0.20 mm

*Data collection*

STOE IPDS 2 diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min}$ = 0.945, $T_{max}$ = 0.981
13273 measured reflections
4861 independent reflections
3479 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029
$\theta_{max}$ = 26.5°, $\theta_{min}$ = 1.7°
$h$ = −11→11
$k$ = −13→13
$l$ = −16→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.091
$S$ = 1.03
4861 reflections
316 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.0141P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| F1 | 1.31808 (14) | 0.32221 (10) | 1.12298 (8) | 0.0875 (4) |
| O1 | 0.65668 (12) | 0.66791 (10) | 0.43998 (8) | 0.0635 (4) |
| O2 | 0.02122 (12) | 1.05391 (10) | 0.84902 (8) | 0.0626 (3) |
| O3 | 0.84333 (12) | 0.78850 (9) | 0.56875 (9) | 0.0627 (3) |
| N1 | 0.44450 (12) | 0.78805 (9) | 0.66593 (8) | 0.0437 (3) |
| N2 | 0.54428 (12) | 0.72130 (10) | 0.59688 (8) | 0.0438 (3) |
| N3 | 0.25440 (13) | 0.94880 (10) | 0.69277 (8) | 0.0471 (3) |
| N4 | 0.93182 (13) | 0.58003 (10) | 0.61148 (9) | 0.0473 (4) |
| N5 | 1.09333 (13) | 0.46726 (10) | 0.76728 (8) | 0.0459 (3) |
| C1 | 0.43486 (15) | 0.84428 (12) | 0.33963 (10) | 0.0425 (4) |
| C2 | 0.38220 (17) | 0.96206 (13) | 0.28338 (11) | 0.0530 (5) |
| C3 | 0.3712 (2) | 0.97650 (17) | 0.18124 (13) | 0.0668 (6) |
| C4 | 0.4089 (2) | 0.87442 (18) | 0.13408 (12) | 0.0671 (6) |
| C5 | 0.46053 (19) | 0.75785 (16) | 0.18863 (12) | 0.0622 (6) |
| C6 | 0.47474 (17) | 0.74228 (13) | 0.29017 (11) | 0.0523 (5) |
| C7 | 0.44320 (14) | 0.82808 (11) | 0.45042 (10) | 0.0402 (4) |
| C8 | 0.55601 (15) | 0.73397 (12) | 0.49132 (10) | 0.0445 (4) |
| C9 | 0.35078 (14) | 0.87626 (11) | 0.62510 (10) | 0.0406 (4) |
| C10 | 0.34782 (15) | 0.89739 (11) | 0.51700 (10) | 0.0429 (4) |
| C11 | 0.10441 (17) | 1.00413 (13) | 0.67498 (11) | 0.0546 (5) |
| C12 | 0.04171 (18) | 1.10537 (13) | 0.73676 (12) | 0.0615 (5) |
| C13 | 0.17058 (19) | 1.00733 (16) | 0.86405 (13) | 0.0641 (6) |
| C14 | 0.24172 (18) | 0.90257 (14) | 0.80774 (11) | 0.0533 (5) |
| C15 | 0.64791 (15) | 0.62509 (12) | 0.64412 (11) | 0.0462 (4) |
| C16 | 0.81643 (16) | 0.67228 (12) | 0.60443 (10) | 0.0444 (4) |
| C17 | 1.09342 (16) | 0.61613 (15) | 0.59093 (11) | 0.0578 (5) |
| C18 | 1.12789 (17) | 0.59830 (13) | 0.69637 (12) | 0.0553 (5) |
| C19 | 0.93822 (16) | 0.42227 (12) | 0.78027 (11) | 0.0460 (4) |
| C20 | 0.90918 (17) | 0.44392 (12) | 0.67254 (11) | 0.0484 (4) |
| C21 | 1.14574 (15) | 0.43318 (12) | 0.85944 (10) | 0.0446 (4) |
| C22 | 1.24267 (18) | 0.51424 (13) | 0.87566 (12) | 0.0532 (5) |
| C23 | 1.2999 (2) | 0.47658 (15) | 0.96358 (13) | 0.0611 (6) |
| C24 | 1.26051 (19) | 0.35931 (15) | 1.03610 (12) | 0.0599 (5) |
| C25 | 1.16453 (19) | 0.27695 (15) | 1.02480 (12) | 0.0596 (5) |
| C26 | 1.10713 (17) | 0.31386 (13) | 0.93713 (11) | 0.0530 (5) |

| | | | | |
|---|---|---|---|---|
| H2 | 0.35420 | 1.03140 | 0.31490 | 0.0640* |
| H3 | 0.33800 | 1.05620 | 0.14380 | 0.0800* |
| H4 | 0.39940 | 0.88440 | 0.06570 | 0.0810* |
| H5 | 0.48620 | 0.68860 | 0.15690 | 0.0750* |
| H6 | 0.51130 | 0.66300 | 0.32590 | 0.0630* |
| H10 | 0.27860 | 0.96040 | 0.49140 | 0.0510* |
| H11A | 0.02810 | 0.93570 | 0.69910 | 0.0660* |
| H11B | 0.12070 | 1.04350 | 0.59830 | 0.0660* |
| H12A | 0.11470 | 1.17670 | 0.70830 | 0.0740* |
| H12B | −0.05910 | 1.13980 | 0.72630 | 0.0740* |
| H13A | 0.15730 | 0.97320 | 0.94090 | 0.0770* |
| H13B | 0.24260 | 1.07930 | 0.83630 | 0.0770* |
| H14A | 0.34600 | 0.87750 | 0.81590 | 0.0640* |
| H14B | 0.17600 | 0.82650 | 0.84070 | 0.0640* |
| H15A | 0.64680 | 0.54450 | 0.62510 | 0.0550* |
| H15B | 0.60890 | 0.60740 | 0.72240 | 0.0550* |
| H17A | 1.10820 | 0.70640 | 0.54920 | 0.0690* |
| H17B | 1.16660 | 0.56240 | 0.54900 | 0.0690* |
| H18A | 1.23860 | 0.61530 | 0.68140 | 0.0660* |
| H18B | 1.06480 | 0.66110 | 0.73320 | 0.0660* |
| H19A | 0.85690 | 0.46850 | 0.82480 | 0.0550* |
| H19B | 0.93100 | 0.33020 | 0.81730 | 0.0550* |
| H20A | 0.98170 | 0.38910 | 0.63140 | 0.0580* |
| H20B | 0.80200 | 0.41990 | 0.68420 | 0.0580* |
| H22 | 1.26910 | 0.59500 | 0.82640 | 0.0640* |
| H23 | 1.36500 | 0.53130 | 0.97310 | 0.0730* |
| H25 | 1.13830 | 0.19710 | 1.07550 | 0.0720* |
| H26 | 1.04120 | 0.25820 | 0.92940 | 0.0630* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.1156 (9) | 0.0924 (7) | 0.0722 (6) | 0.0160 (6) | −0.0589 (6) | −0.0215 (5) |
| O1 | 0.0598 (7) | 0.0678 (6) | 0.0611 (6) | 0.0271 (5) | −0.0168 (5) | −0.0268 (5) |
| O2 | 0.0534 (6) | 0.0630 (6) | 0.0617 (6) | 0.0020 (5) | 0.0024 (5) | −0.0255 (5) |
| O3 | 0.0606 (6) | 0.0408 (5) | 0.0794 (7) | −0.0021 (4) | −0.0271 (5) | 0.0026 (5) |
| N1 | 0.0414 (6) | 0.0417 (5) | 0.0441 (6) | 0.0032 (4) | −0.0094 (5) | −0.0111 (5) |
| N2 | 0.0391 (6) | 0.0427 (5) | 0.0463 (6) | 0.0082 (4) | −0.0124 (5) | −0.0108 (5) |
| N3 | 0.0453 (6) | 0.0442 (6) | 0.0463 (6) | 0.0071 (5) | −0.0075 (5) | −0.0140 (5) |
| N4 | 0.0395 (6) | 0.0438 (6) | 0.0548 (7) | 0.0038 (5) | −0.0182 (5) | −0.0045 (5) |
| N5 | 0.0445 (6) | 0.0450 (6) | 0.0459 (6) | −0.0017 (5) | −0.0163 (5) | −0.0053 (5) |
| C1 | 0.0356 (7) | 0.0440 (7) | 0.0459 (7) | −0.0017 (5) | −0.0097 (5) | −0.0114 (5) |
| C2 | 0.0543 (9) | 0.0500 (8) | 0.0552 (8) | 0.0054 (6) | −0.0206 (7) | −0.0127 (6) |
| C3 | 0.0701 (11) | 0.0693 (10) | 0.0592 (9) | 0.0068 (8) | −0.0283 (8) | −0.0077 (8) |
| C4 | 0.0657 (11) | 0.0914 (12) | 0.0465 (8) | −0.0052 (9) | −0.0193 (7) | −0.0179 (8) |
| C5 | 0.0626 (10) | 0.0713 (10) | 0.0561 (9) | −0.0032 (8) | −0.0116 (7) | −0.0288 (8) |
| C6 | 0.0543 (9) | 0.0508 (8) | 0.0516 (8) | 0.0003 (6) | −0.0137 (6) | −0.0166 (6) |
| C7 | 0.0357 (7) | 0.0375 (6) | 0.0452 (7) | −0.0016 (5) | −0.0101 (5) | −0.0095 (5) |

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|---|---|---|---|---|---|---|
| C8 | 0.0389 (7) | 0.0434 (7) | 0.0497 (7) | 0.0048 (5) | −0.0112 (6) | −0.0145 (6) |
| C9 | 0.0371 (7) | 0.0360 (6) | 0.0450 (7) | −0.0004 (5) | −0.0082 (5) | −0.0100 (5) |
| C10 | 0.0388 (7) | 0.0386 (6) | 0.0492 (7) | 0.0048 (5) | −0.0136 (5) | −0.0102 (5) |
| C11 | 0.0529 (9) | 0.0497 (7) | 0.0521 (8) | 0.0148 (6) | −0.0100 (6) | −0.0122 (6) |
| C12 | 0.0565 (9) | 0.0448 (7) | 0.0671 (10) | 0.0079 (6) | −0.0004 (7) | −0.0142 (7) |
| C13 | 0.0578 (10) | 0.0736 (10) | 0.0607 (9) | −0.0014 (8) | −0.0060 (7) | −0.0310 (8) |
| C14 | 0.0518 (8) | 0.0563 (8) | 0.0478 (8) | 0.0018 (6) | −0.0088 (6) | −0.0158 (6) |
| C15 | 0.0420 (7) | 0.0415 (6) | 0.0503 (7) | 0.0058 (5) | −0.0156 (6) | −0.0057 (6) |
| C16 | 0.0471 (8) | 0.0411 (7) | 0.0432 (7) | 0.0035 (5) | −0.0178 (6) | −0.0053 (5) |
| C17 | 0.0400 (8) | 0.0661 (9) | 0.0545 (8) | −0.0036 (6) | −0.0137 (6) | 0.0039 (7) |
| C18 | 0.0478 (8) | 0.0533 (8) | 0.0591 (9) | −0.0096 (6) | −0.0200 (7) | 0.0015 (7) |
| C19 | 0.0476 (8) | 0.0372 (6) | 0.0512 (7) | 0.0004 (5) | −0.0142 (6) | −0.0100 (5) |
| C20 | 0.0513 (8) | 0.0384 (6) | 0.0588 (8) | 0.0083 (5) | −0.0240 (6) | −0.0131 (6) |
| C21 | 0.0442 (7) | 0.0434 (7) | 0.0448 (7) | 0.0072 (5) | −0.0134 (6) | −0.0126 (5) |
| C22 | 0.0596 (9) | 0.0455 (7) | 0.0574 (8) | 0.0032 (6) | −0.0224 (7) | −0.0144 (6) |
| C23 | 0.0685 (10) | 0.0594 (9) | 0.0690 (10) | 0.0083 (7) | −0.0328 (8) | −0.0282 (8) |
| C24 | 0.0682 (10) | 0.0666 (9) | 0.0525 (8) | 0.0179 (8) | −0.0302 (7) | −0.0213 (7) |
| C25 | 0.0634 (10) | 0.0571 (8) | 0.0518 (8) | 0.0062 (7) | −0.0197 (7) | −0.0045 (7) |
| C26 | 0.0534 (9) | 0.0490 (7) | 0.0551 (8) | 0.0000 (6) | −0.0203 (7) | −0.0077 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C24 | 1.369 (2) | C21—C26 | 1.3999 (19) |
| O1—C8 | 1.2343 (17) | C22—C23 | 1.380 (2) |
| O2—C12 | 1.4173 (18) | C23—C24 | 1.357 (2) |
| O2—C13 | 1.424 (2) | C24—C25 | 1.366 (2) |
| O3—C16 | 1.2215 (17) | C25—C26 | 1.378 (2) |
| N1—N2 | 1.3698 (15) | C2—H2 | 0.9300 |
| N1—C9 | 1.3098 (17) | C3—H3 | 0.9300 |
| N2—C8 | 1.3646 (16) | C4—H4 | 0.9300 |
| N2—C15 | 1.4531 (18) | C5—H5 | 0.9300 |
| N3—C9 | 1.3883 (16) | C6—H6 | 0.9300 |
| N3—C11 | 1.464 (2) | C10—H10 | 0.9300 |
| N3—C14 | 1.4612 (17) | C11—H11A | 0.9700 |
| N4—C16 | 1.3501 (18) | C11—H11B | 0.9700 |
| N4—C17 | 1.4532 (19) | C12—H12A | 0.9700 |
| N4—C20 | 1.4571 (17) | C12—H12B | 0.9700 |
| N5—C18 | 1.4586 (18) | C13—H13A | 0.9700 |
| N5—C19 | 1.4572 (19) | C13—H13B | 0.9700 |
| N5—C21 | 1.4067 (17) | C14—H14A | 0.9700 |
| C1—C2 | 1.393 (2) | C14—H14B | 0.9700 |
| C1—C6 | 1.3933 (19) | C15—H15A | 0.9700 |
| C1—C7 | 1.4827 (18) | C15—H15B | 0.9700 |
| C2—C3 | 1.379 (2) | C17—H17A | 0.9700 |
| C3—C4 | 1.375 (3) | C17—H17B | 0.9700 |
| C4—C5 | 1.371 (3) | C18—H18A | 0.9700 |
| C5—C6 | 1.380 (2) | C18—H18B | 0.9700 |
| C7—C8 | 1.4639 (19) | C19—H19A | 0.9700 |

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|---|---|---|---|
| C7—C10 | 1.3528 (18) | C19—H19B | 0.9700 |
| C9—C10 | 1.4219 (18) | C20—H20A | 0.9700 |
| C11—C12 | 1.504 (2) | C20—H20B | 0.9700 |
| C13—C14 | 1.502 (2) | C22—H22 | 0.9300 |
| C15—C16 | 1.518 (2) | C23—H23 | 0.9300 |
| C17—C18 | 1.508 (2) | C25—H25 | 0.9300 |
| C19—C20 | 1.509 (2) | C26—H26 | 0.9300 |
| C21—C22 | 1.396 (2) | | |

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|---|---|---|---|
| F1···H3$^{i}$ | 2.7700 | H2···H10 | 2.1900 |
| F1···H15B$^{ii}$ | 2.6900 | H2···O3$^{iii}$ | 2.9100 |
| F1···H19A$^{ii}$ | 2.7100 | H2···N1$^{iii}$ | 2.9000 |
| O1···C6 | 2.8742 (19) | H3···F1$^{xii}$ | 2.7700 |
| O1···C16 | 3.0123 (18) | H3···C25$^{xii}$ | 3.0300 |
| O2···N3 | 2.8383 (15) | H3···H25$^{xii}$ | 2.4400 |
| O3···N2 | 2.7209 (16) | H5···C22$^{iv}$ | 3.0700 |
| O3···C11$^{iii}$ | 3.3306 (18) | H5···C23$^{iv}$ | 2.8800 |
| O3···C8 | 3.2257 (18) | H5···C24$^{iv}$ | 2.9800 |
| O1···H12A$^{iii}$ | 2.6800 | H6···O1 | 2.3200 |
| O1···H15A | 2.4500 | H6···C8 | 2.7100 |
| O1···H6 | 2.3200 | H6···H15A$^{xi}$ | 2.5800 |
| O1···H17B$^{iv}$ | 2.7700 | H10···C2 | 2.6600 |
| O2···H13A$^{v}$ | 2.7200 | H10···C11 | 2.6100 |
| O2···H26$^{vi}$ | 2.7500 | H10···H2 | 2.1900 |
| O3···H17A | 2.3800 | H10···H11B | 2.0000 |
| O3···H2$^{iii}$ | 2.9100 | H10···O3$^{iii}$ | 2.7800 |
| O3···H10$^{iii}$ | 2.7800 | H11B···C10 | 2.5700 |
| O3···H11B$^{iii}$ | 2.4100 | H11B···H10 | 2.0000 |
| N2···O3 | 2.7209 (16) | H11B···O3$^{iii}$ | 2.4100 |
| N3···O2 | 2.8383 (15) | H12A···H13B | 2.3100 |
| N4···N5 | 2.8407 (16) | H12A···H20A$^{vi}$ | 2.5500 |
| N5···N4 | 2.8407 (16) | H12A···O1$^{iii}$ | 2.6800 |
| N1···H22$^{vii}$ | 2.7300 | H12B···C3$^{x}$ | 2.9200 |
| N1···H2$^{iii}$ | 2.9000 | H12B···C4$^{x}$ | 3.0800 |
| N1···H18A$^{vii}$ | 2.6700 | H13A···O2$^{v}$ | 2.7200 |
| N1···H14A | 2.3600 | H13B···H12A | 2.3100 |
| N1···H14B | 2.8600 | H13B···C6$^{iii}$ | 3.0700 |
| N2···H18A$^{vii}$ | 2.8300 | H14A···N1 | 2.3600 |
| C2···C14$^{iii}$ | 3.508 (2) | H14A···C2$^{iii}$ | 2.8600 |
| C6···O1 | 2.8742 (19) | H14B···N1 | 2.8600 |
| C7···C9$^{iii}$ | 3.5673 (18) | H14B···H22$^{vii}$ | 2.5600 |
| C8···O3 | 3.2257 (18) | H15A···O1 | 2.4500 |
| C9···C7$^{iii}$ | 3.5673 (18) | H15A···C20 | 2.6500 |
| C9···C18$^{vii}$ | 3.497 (2) | H15A···H20B | 2.0000 |
| C9···C10$^{iii}$ | 3.5089 (18) | H15A···H6$^{xi}$ | 2.5800 |
| C10···C9$^{iii}$ | 3.5089 (18) | H15B···C20 | 3.0200 |
| C10···C10$^{iii}$ | 3.5197 (19) | H15B···H20B | 2.5500 |
| C11···O3$^{iii}$ | 3.3306 (18) | H15B···F1$^{ii}$ | 2.6900 |

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|---|---|---|---|
| C12···C19$^{vi}$ | 3.577 (2) | H17A···O3 | 2.3800 |
| C14···C2$^{iii}$ | 3.508 (2) | H17A···C10$^{viii}$ | 2.9700 |
| C16···O1 | 3.0123 (18) | H17B···H20A | 2.4000 |
| C18···C9$^{viii}$ | 3.497 (2) | H17B···O1$^{iv}$ | 2.7700 |
| C19···C12$^{ix}$ | 3.577 (2) | H18A···N1$^{viii}$ | 2.6700 |
| C2···H14A$^{iii}$ | 2.8600 | H18A···N2$^{viii}$ | 2.8300 |
| C2···H10 | 2.6600 | H18A···C9$^{viii}$ | 2.8900 |
| C3···H12B$^{x}$ | 2.9200 | H18A···C22 | 2.5500 |
| C4···H12B$^{x}$ | 3.0800 | H18A···H22 | 2.0100 |
| C5···H20B$^{xi}$ | 2.9400 | H18B···C22 | 2.8900 |
| C6···H20B$^{xi}$ | 3.0500 | H18B···H22 | 2.4700 |
| C6···H13B$^{iii}$ | 3.0700 | H19A···F1$^{ii}$ | 2.7100 |
| C8···H6 | 2.7100 | H19A···C23$^{ii}$ | 2.9500 |
| C9···H18A$^{vii}$ | 2.8900 | H19A···C24$^{ii}$ | 2.8800 |
| C10···H17A$^{vii}$ | 2.9700 | H19B···C12$^{ix}$ | 2.8700 |
| C10···H2 | 2.6900 | H19B···C26 | 2.5500 |
| C10···H11B | 2.5700 | H19B···H26 | 1.9900 |
| C11···H10 | 2.6100 | H20A···C12$^{ix}$ | 3.0300 |
| C12···H20A$^{vi}$ | 3.0300 | H20A···H12A$^{ix}$ | 2.5500 |
| C12···H19B$^{vi}$ | 2.8700 | H20A···H17B | 2.4000 |
| C13···H26$^{vi}$ | 3.0500 | H20B···C15 | 2.4800 |
| C15···H20B | 2.4800 | H20B···H15A | 2.0000 |
| C18···H22 | 2.4600 | H20B···H15B | 2.5500 |
| C19···H26 | 2.6100 | H20B···C5$^{xi}$ | 2.9400 |
| C20···H15A | 2.6500 | H20B···C6$^{xi}$ | 3.0500 |
| C20···H15B | 3.0200 | H22···N1$^{viii}$ | 2.7300 |
| C22···H18A | 2.5500 | H22···C18 | 2.4600 |
| C22···H18B | 2.8900 | H22···H14B$^{viii}$ | 2.5600 |
| C22···H5$^{iv}$ | 3.0700 | H22···H18A | 2.0100 |
| C23···H5$^{iv}$ | 2.8800 | H22···H18B | 2.4700 |
| C23···H19A$^{ii}$ | 2.9500 | H25···H3$^{i}$ | 2.4400 |
| C24···H5$^{iv}$ | 2.9800 | H26···O2$^{ix}$ | 2.7500 |
| C24···H19A$^{ii}$ | 2.8800 | H26···C13$^{ix}$ | 3.0500 |
| C25···H3$^{i}$ | 3.0300 | H26···C19 | 2.6100 |
| C26···H19B | 2.5500 | H26···H19B | 1.9900 |
| H2···C10 | 2.6900 | | |

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|---|---|---|---|
| C12—O2—C13 | 108.95 (12) | C4—C5—H5 | 120.00 |
| N2—N1—C9 | 116.01 (10) | C6—C5—H5 | 120.00 |
| N1—N2—C8 | 127.91 (11) | C1—C6—H6 | 120.00 |
| N1—N2—C15 | 114.75 (10) | C5—C6—H6 | 120.00 |
| C8—N2—C15 | 117.34 (11) | C7—C10—H10 | 119.00 |
| C9—N3—C11 | 119.14 (11) | C9—C10—H10 | 119.00 |
| C9—N3—C14 | 117.13 (11) | N3—C11—H11A | 110.00 |
| C11—N3—C14 | 112.25 (11) | N3—C11—H11B | 110.00 |
| C16—N4—C17 | 120.60 (12) | C12—C11—H11A | 110.00 |
| C16—N4—C20 | 126.08 (12) | C12—C11—H11B | 110.00 |
| C17—N4—C20 | 110.06 (12) | H11A—C11—H11B | 108.00 |

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|---|---|---|---|
| C18—N5—C19 | 114.00 (11) | O2—C12—H12A | 109.00 |
| C18—N5—C21 | 116.78 (11) | O2—C12—H12B | 109.00 |
| C19—N5—C21 | 117.13 (10) | C11—C12—H12A | 109.00 |
| C2—C1—C6 | 118.22 (12) | C11—C12—H12B | 109.00 |
| C2—C1—C7 | 120.29 (12) | H12A—C12—H12B | 108.00 |
| C6—C1—C7 | 121.46 (12) | O2—C13—H13A | 109.00 |
| C1—C2—C3 | 120.40 (14) | O2—C13—H13B | 109.00 |
| C2—C3—C4 | 120.71 (16) | C14—C13—H13A | 109.00 |
| C3—C4—C5 | 119.50 (15) | C14—C13—H13B | 109.00 |
| C4—C5—C6 | 120.59 (15) | H13A—C13—H13B | 108.00 |
| C1—C6—C5 | 120.57 (14) | N3—C14—H14A | 110.00 |
| C1—C7—C8 | 120.11 (11) | N3—C14—H14B | 110.00 |
| C1—C7—C10 | 122.01 (12) | C13—C14—H14A | 110.00 |
| C8—C7—C10 | 117.88 (11) | C13—C14—H14B | 110.00 |
| O1—C8—N2 | 119.12 (12) | H14A—C14—H14B | 108.00 |
| O1—C8—C7 | 126.40 (12) | N2—C15—H15A | 109.00 |
| N2—C8—C7 | 114.49 (11) | N2—C15—H15B | 109.00 |
| N1—C9—N3 | 116.54 (11) | C16—C15—H15A | 109.00 |
| N1—C9—C10 | 121.96 (11) | C16—C15—H15B | 109.00 |
| N3—C9—C10 | 121.49 (11) | H15A—C15—H15B | 108.00 |
| C7—C10—C9 | 121.60 (12) | N4—C17—H17A | 110.00 |
| N3—C11—C12 | 109.61 (12) | N4—C17—H17B | 110.00 |
| O2—C12—C11 | 112.03 (12) | C18—C17—H17A | 110.00 |
| O2—C13—C14 | 112.09 (14) | C18—C17—H17B | 110.00 |
| N3—C14—C13 | 110.40 (12) | H17A—C17—H17B | 108.00 |
| N2—C15—C16 | 111.27 (11) | N5—C18—H18A | 109.00 |
| O3—C16—N4 | 122.80 (14) | N5—C18—H18B | 109.00 |
| O3—C16—C15 | 120.54 (13) | C17—C18—H18A | 109.00 |
| N4—C16—C15 | 116.67 (11) | C17—C18—H18B | 109.00 |
| N4—C17—C18 | 110.23 (12) | H18A—C18—H18B | 108.00 |
| N5—C18—C17 | 112.17 (12) | N5—C19—H19A | 109.00 |
| N5—C19—C20 | 111.62 (11) | N5—C19—H19B | 109.00 |
| N4—C20—C19 | 110.40 (11) | C20—C19—H19A | 109.00 |
| N5—C21—C22 | 121.73 (12) | C20—C19—H19B | 109.00 |
| N5—C21—C26 | 121.14 (12) | H19A—C19—H19B | 108.00 |
| C22—C21—C26 | 117.10 (13) | N4—C20—H20A | 110.00 |
| C21—C22—C23 | 121.10 (14) | N4—C20—H20B | 110.00 |
| C22—C23—C24 | 119.61 (16) | C19—C20—H20A | 110.00 |
| F1—C24—C23 | 119.38 (15) | C19—C20—H20B | 110.00 |
| F1—C24—C25 | 118.95 (14) | H20A—C20—H20B | 108.00 |
| C23—C24—C25 | 121.67 (15) | C21—C22—H22 | 119.00 |
| C24—C25—C26 | 119.02 (14) | C23—C22—H22 | 119.00 |
| C21—C26—C25 | 121.49 (14) | C22—C23—H23 | 120.00 |
| C1—C2—H2 | 120.00 | C24—C23—H23 | 120.00 |
| C3—C2—H2 | 120.00 | C24—C25—H25 | 121.00 |
| C2—C3—H3 | 120.00 | C26—C25—H25 | 120.00 |
| C4—C3—H3 | 120.00 | C21—C26—H26 | 119.00 |
| C3—C4—H4 | 120.00 | C25—C26—H26 | 119.00 |

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|---|---|---|---|
| C5—C4—H4 | 120.00 | | |
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| C13—O2—C12—C11 | 60.78 (16) | C6—C1—C7—C10 | −151.12 (14) |
| C12—O2—C13—C14 | −59.74 (16) | C6—C1—C7—C8 | 28.3 (2) |
| C9—N1—N2—C8 | −1.23 (19) | C6—C1—C2—C3 | −0.4 (2) |
| N2—N1—C9—C10 | 2.71 (18) | C7—C1—C2—C3 | −178.64 (14) |
| C9—N1—N2—C15 | 179.39 (11) | C2—C1—C7—C8 | −153.52 (13) |
| N2—N1—C9—N3 | −177.06 (11) | C2—C1—C6—C5 | −0.7 (2) |
| C15—N2—C8—O1 | −2.74 (19) | C7—C1—C6—C5 | 177.56 (14) |
| N1—N2—C8—C7 | −2.13 (19) | C2—C1—C7—C10 | 27.1 (2) |
| C15—N2—C8—C7 | 177.24 (11) | C1—C2—C3—C4 | 1.3 (3) |
| N1—N2—C15—C16 | −105.23 (12) | C2—C3—C4—C5 | −1.1 (3) |
| C8—N2—C15—C16 | 75.32 (14) | C3—C4—C5—C6 | 0.1 (3) |
| N1—N2—C8—O1 | 177.89 (12) | C4—C5—C6—C1 | 0.9 (3) |
| C14—N3—C11—C12 | 52.25 (15) | C1—C7—C10—C9 | 176.59 (12) |
| C14—N3—C9—N1 | −12.56 (18) | C8—C7—C10—C9 | −2.82 (19) |
| C14—N3—C9—C10 | 167.66 (13) | C1—C7—C8—O1 | 4.6 (2) |
| C9—N3—C14—C13 | 165.09 (13) | C10—C7—C8—N2 | 3.99 (18) |
| C11—N3—C14—C13 | −51.70 (16) | C1—C7—C8—N2 | −175.43 (12) |
| C9—N3—C11—C12 | −165.36 (11) | C10—C7—C8—O1 | −176.03 (14) |
| C11—N3—C9—N1 | −153.17 (12) | N1—C9—C10—C7 | −0.7 (2) |
| C11—N3—C9—C10 | 27.06 (18) | N3—C9—C10—C7 | 179.07 (12) |
| C16—N4—C20—C19 | −98.68 (16) | N3—C11—C12—O2 | −57.26 (16) |
| C17—N4—C16—O3 | 8.9 (2) | O2—C13—C14—N3 | 55.52 (17) |
| C17—N4—C16—C15 | −170.73 (11) | N2—C15—C16—N4 | −158.32 (11) |
| C20—N4—C16—C15 | −13.24 (19) | N2—C15—C16—O3 | 22.07 (17) |
| C20—N4—C17—C18 | −60.28 (15) | N4—C17—C18—N5 | 53.86 (16) |
| C16—N4—C17—C18 | 100.50 (15) | N5—C19—C20—N4 | −54.43 (15) |
| C20—N4—C16—O3 | 166.37 (13) | N5—C21—C22—C23 | −176.61 (14) |
| C17—N4—C20—C19 | 60.79 (15) | C26—C21—C22—C23 | 1.2 (2) |
| C19—N5—C18—C17 | −48.75 (16) | N5—C21—C26—C25 | 176.70 (14) |
| C21—N5—C19—C20 | −169.67 (11) | C22—C21—C26—C25 | −1.1 (2) |
| C19—N5—C21—C26 | 34.26 (18) | C21—C22—C23—C24 | −0.5 (2) |
| C18—N5—C19—C20 | 48.87 (15) | C22—C23—C24—F1 | 179.76 (15) |
| C18—N5—C21—C26 | 174.65 (13) | C22—C23—C24—C25 | −0.3 (3) |
| C18—N5—C21—C22 | −7.63 (19) | F1—C24—C25—C26 | −179.68 (14) |
| C21—N5—C18—C17 | 169.65 (12) | C23—C24—C25—C26 | 0.4 (3) |
| C19—N5—C21—C22 | −148.02 (13) | C24—C25—C26—C21 | 0.4 (2) |

Symmetry codes: (i) *x*+1, *y*−1, *z*+1; (ii) −*x*+2, −*y*+1, −*z*+2; (iii) −*x*+1, −*y*+2, −*z*+1; (iv) −*x*+2, −*y*+1, −*z*+1; (v) −*x*, −*y*+2, −*z*+2; (vi) *x*−1, *y*+1, *z*; (vii) *x*−1, *y*, *z*; (viii) *x*+1, *y*, *z*; (ix) *x*+1, *y*−1, *z*; (x) −*x*, −*y*+2, −*z*+1; (xi) −*x*+1, −*y*+1, −*z*+1; (xii) *x*−1, *y*+1, *z*−1.

*Hydrogen-bond geometry (Å, º)*

Cg2, Cg4 and Cg5 are the centroids of the N1/N2/C7–C10, C1–C6 and C21–C26 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C6—H6···O1 | 0.93 | 2.32 | 2.8742 (19) | 117 |
| C11—H11*B*···O3$^{iii}$ | 0.97 | 2.41 | 3.3306 (18) | 159 |
| C17—H17*A*···O3 | 0.97 | 2.38 | 2.7660 (19) | 103 |

| | | | | |
|---|---|---|---|---|
| C5—H5···*Cg*5$^{iv}$ | 0.93 | 2.86 | 3.4941 (18) | 127 |
| C13—H13*B*···*Cg*4$^{iii}$ | 0.97 | 2.92 | 3.7395 (19) | 143 |
| C18—H18*A*···*Cg*2$^{viii}$ | 0.97 | 2.73 | 3.5079 (16) | 138 |

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