IUCrData

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5-Fluoro-1-[(1-{[(1*S*,2*R*,6*R*,8*S*,9*R*)-4,4,11,11-tetramethyl-3,5,7,10,12-pentaoxatricyclo[7.3.0.0^{2,6}]dodecan-8-yl]methyl}-1*H*-1,2,3-triazol-4-yl)methyl]-2,3-dihydro-1*H*-indole-2,3-dione

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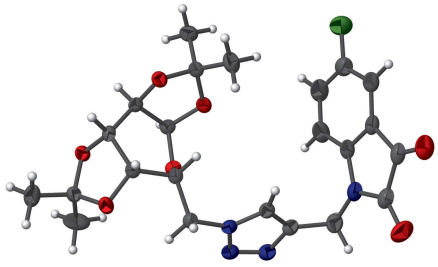
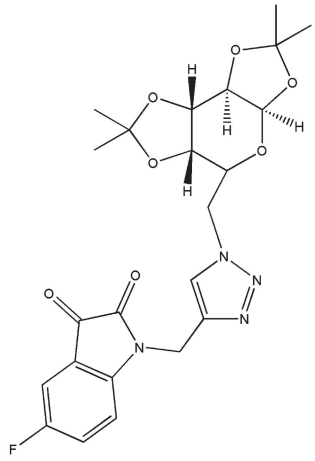
Structural data: full structural data are available from iucrdata.iucr.org

In the title molecule, $C_{23}H_{25}FN_4O_7$, the dihedral angle between the indole skeleton (r.m.s. deviation = 0.022 Å) and the triazole moiety is 74.67 (7)°. The molecules pack in a three-dimensional network in the crystal, being linked by C—H···O and N—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

Isatin and its derivatives have received attention in recent years due to their wide variety of biological activities, making them relevant to application as insecticides and fungicides and in a broad range of drug therapies, including as anticancer agents, antibiotics and anti-VIH activities (Malhotra *et al.*, 2011; Ramachandran, 2011; Pandeya *et al.*, 1999). In our work, we are interested in developing new isatin derivatives containing a 1,2,3-triazole nucleus by 1,3-dipolar cycloaddition reactions. As part of this study, we now describe the synthesis and structure of the title compound (Fig. 1).

The r.m.s. deviation of the indole skeleton from planarity is 0.022 Å and the dihedral angle between the mean plane and that of the triazole moiety is 74.67 (7)°. A puckering analysis of the tricyclododecane skeleton gave parameters Q_2 = 0.244 (2) Å and φ_2 = 266.5 (5)° for the C14/C15/C21/O4/O5 ring, Q_2 = 0.305 (2) Å and φ_2 = 172.7 (4)° for the C16–C18/O6/O7 ring, and Q = 0.647 (2) Å, θ = 99.1 (2)° and φ = 208.0 (2)° for the C13–C17/O3 ring.

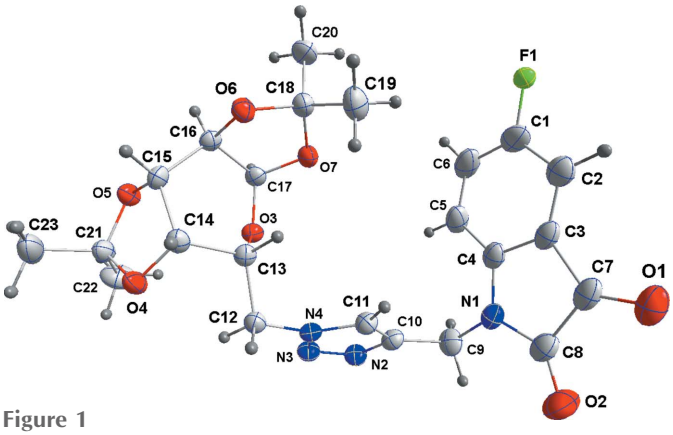


Figure 1
Perspective view of the molecule with 50% probability displacement ellipsoids.

In the crystal (Fig. 2), molecules are linked by weak C—H···O and C—H···N interactions (Table 1).

Synthesis and crystallization

To a solution of 5-fluoro-1-(prop-2-yn-1-yl)indoline-2,3-dione (0.2 g, 0.98 mmol) in ethanol (15 ml) was added (3a*R*,5*S*,5a*R*,8a*S*,8b*R*)-5-azido-2,2,7,7-tetramethyltetrahydro-3a*H*-di[1,3]dioxolo[4,5-*b*:4′,5′-*d*]pyran (0.42 g, 1.47 mmol). The mixture was stirred under reflux for 24 h. After completion of the reaction (monitored by TLC), the solution was concentrated and the residue was purified by column chromatography on silica gel by using a 3/1 (*v*/*v*) mixture of hexane and ethyl acetate. Crystals were obtained when the solvent was allowed to evaporate. The solid product was purified by recrystallization from ethanol solution to afford yellow blocks in 62% yield.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

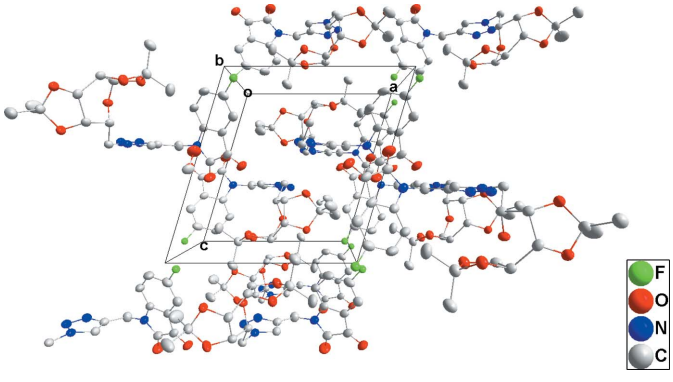


Figure 2
Packing viewed down the *b* axis. H atoms have been omitted for clarity.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O5^{i}	0.95	2.24	3.184 (3)	170
C15—H15···N2^{ii}	1.00	2.53	3.338 (3)	138

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{25}FN_4O_7$
M_r	488.47
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	150
a, b, c (Å)	11.1688 (2), 9.0576 (2), 11.9237 (2)
β (°)	106.770 (1)
V (Å^3)	1154.93 (4)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.94
Crystal size (mm)	0.22 × 0.14 × 0.12
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.85, 0.90
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8944, 4238, 4039
R_{int}	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.074, 1.07
No. of reflections	4238
No. of parameters	320
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.21
Absolute structure	Flack x determined using 1689 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.16 (7)

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170739 [https://doi.org/10.1107/S2414314617007398]

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5-Fluoro-1-[(1-{[(1*S*,2*R*,6*R*,8*S*,9*R*)-4,4,11,11-tetramethyl-3,5,7,10,12-pentaoxatricyclo[7.3.0.0^{2,6}]dodecan-8-yl]methyl}-1*H*-1,2,3-triazol-4-yl)methyl]-2,3-dihydro-1*H*-indole-2,3-dione

Crystal data

$C_{23}H_{25}FN_4O_7$
M_r = 488.47
Monoclinic, $P2_1$
a = 11.1688 (2) Å
b = 9.0576 (2) Å
c = 11.9237 (2) Å
β = 106.770 (1)°
V = 1154.93 (4) Å^3
Z = 2
$F(000)$ = 512
D_x = 1.405 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 7739 reflections
θ = 3.9–72.4°
μ = 0.94 mm^{-1}
T = 150 K
Block, yellow
0.22 × 0.14 × 0.12 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.85, T_{max} = 0.90
8944 measured reflections
4238 independent reflections
4039 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 72.4°, θ_{min} = 3.9°
h = −13→11
k = −10→10
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.074
S = 1.07
4238 reflections
320 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 0.124P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack x determined using 1689 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.16 (7)

Special details

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

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. H-atoms were placed in calculated positions (C—H = 0.95 - 0.98 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached carbon atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	−0.04911 (18)	0.4129 (2)	0.95449 (14)	0.0612 (5)
O1	−0.20772 (16)	0.2171 (2)	0.49881 (15)	0.0453 (4)
O2	−0.0128 (2)	0.0471 (2)	0.43373 (18)	0.0567 (5)
O3	0.52752 (13)	0.55460 (16)	0.80812 (11)	0.0251 (3)
O4	0.62417 (15)	0.7676 (2)	0.65424 (13)	0.0366 (4)
O5	0.68733 (13)	0.83262 (18)	0.84705 (12)	0.0284 (3)
O6	0.37527 (13)	0.82992 (17)	0.87562 (13)	0.0285 (3)
O7	0.38313 (14)	0.58384 (17)	0.91212 (14)	0.0318 (3)
N1	0.10525 (19)	0.1240 (2)	0.61668 (17)	0.0342 (4)
N2	0.44173 (19)	0.1415 (2)	0.63368 (17)	0.0328 (4)
N3	0.50589 (18)	0.2619 (2)	0.62900 (16)	0.0308 (4)
N4	0.42566 (16)	0.3758 (2)	0.61216 (15)	0.0256 (4)
C1	−0.0085 (3)	0.3410 (3)	0.8720 (2)	0.0439 (6)
C2	−0.0915 (2)	0.3165 (3)	0.7638 (2)	0.0381 (5)
H2	−0.1764	0.3469	0.7454	0.046*
C3	−0.0444 (2)	0.2450 (2)	0.68326 (19)	0.0314 (5)
C4	0.0803 (2)	0.1984 (3)	0.71184 (19)	0.0327 (5)
C5	0.1617 (2)	0.2254 (3)	0.8207 (2)	0.0440 (6)
H5	0.2466	0.1949	0.8400	0.053*
C6	0.1150 (3)	0.2991 (4)	0.9015 (2)	0.0507 (7)
H6	0.1687	0.3206	0.9774	0.061*
C7	−0.1034 (2)	0.1984 (3)	0.5624 (2)	0.0354 (5)
C8	0.0000 (2)	0.1122 (3)	0.5249 (2)	0.0374 (5)
C9	0.2257 (2)	0.0618 (3)	0.6191 (2)	0.0393 (5)
H9A	0.2157	−0.0017	0.5494	0.047*
H9B	0.2562	−0.0008	0.6896	0.047*
C10	0.3211 (2)	0.1795 (2)	0.62036 (18)	0.0295 (5)
C11	0.3094 (2)	0.3294 (2)	0.60663 (18)	0.0280 (4)
H11	0.2361	0.3875	0.5957	0.034*
C12	0.4715 (2)	0.5241 (2)	0.60179 (18)	0.0268 (4)
H12A	0.4234	0.5671	0.5258	0.032*
H12B	0.5604	0.5187	0.6028	0.032*
C13	0.45991 (19)	0.6235 (2)	0.70029 (17)	0.0237 (4)
H13	0.3698	0.6325	0.6973	0.028*

C14	0.51263 (19)	0.7761 (2)	0.69012 (17)	0.0254 (4)
H14	0.4486	0.8372	0.6330	0.030*
C15	0.55554 (19)	0.8558 (2)	0.80891 (18)	0.0244 (4)
H15	0.5362	0.9636	0.7989	0.029*
C16	0.50427 (19)	0.7914 (2)	0.90313 (17)	0.0247 (4)
H16	0.5510	0.8316	0.9816	0.030*
C17	0.5044 (2)	0.6209 (2)	0.90607 (17)	0.0259 (4)
H17	0.5680	0.5860	0.9788	0.031*
C18	0.3164 (2)	0.7158 (3)	0.92364 (19)	0.0303 (5)
C19	0.1825 (2)	0.7038 (3)	0.8498 (2)	0.0454 (6)
H19A	0.1800	0.6834	0.7684	0.068*
H19B	0.1391	0.7968	0.8535	0.068*
H19C	0.1414	0.6233	0.8794	0.068*
C20	0.3298 (3)	0.7443 (3)	1.0523 (2)	0.0410 (6)
H20A	0.2941	0.6614	1.0846	0.061*
H20B	0.2855	0.8354	1.0601	0.061*
H20C	0.4186	0.7546	1.0952	0.061*
C21	0.7299 (2)	0.8122 (3)	0.74624 (18)	0.0293 (4)
C22	0.8243 (3)	0.6889 (4)	0.7723 (3)	0.0576 (8)
H22A	0.7871	0.6008	0.7962	0.086*
H22B	0.8978	0.7190	0.8357	0.086*
H22C	0.8496	0.6667	0.7019	0.086*
C23	0.7782 (3)	0.9559 (3)	0.7127 (2)	0.0521 (7)
H23A	0.8053	0.9413	0.6423	0.078*
H23B	0.8493	0.9897	0.7771	0.078*
H23C	0.7116	1.0301	0.6970	0.078*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0725 (12)	0.0748 (12)	0.0426 (9)	−0.0128 (10)	0.0268 (8)	−0.0144 (8)
O1	0.0361 (10)	0.0514 (11)	0.0391 (9)	−0.0054 (8)	−0.0041 (8)	0.0050 (8)
O2	0.0608 (13)	0.0598 (13)	0.0431 (11)	−0.0057 (10)	0.0046 (9)	−0.0193 (10)
O3	0.0290 (8)	0.0241 (7)	0.0210 (7)	0.0040 (6)	0.0053 (6)	0.0022 (5)
O4	0.0339 (9)	0.0505 (9)	0.0282 (8)	−0.0157 (7)	0.0135 (7)	−0.0091 (7)
O5	0.0225 (7)	0.0365 (8)	0.0251 (7)	−0.0017 (6)	0.0051 (5)	−0.0014 (6)
O6	0.0260 (7)	0.0271 (7)	0.0334 (8)	0.0013 (6)	0.0103 (6)	0.0016 (6)
O7	0.0352 (8)	0.0272 (8)	0.0373 (8)	−0.0035 (6)	0.0170 (7)	−0.0001 (6)
N1	0.0331 (10)	0.0325 (10)	0.0337 (10)	−0.0068 (8)	0.0045 (8)	−0.0002 (8)
N2	0.0359 (11)	0.0280 (10)	0.0345 (10)	0.0043 (8)	0.0099 (8)	0.0009 (7)
N3	0.0335 (10)	0.0292 (9)	0.0299 (9)	0.0051 (7)	0.0092 (7)	−0.0004 (7)
N4	0.0284 (9)	0.0257 (9)	0.0223 (8)	0.0017 (7)	0.0070 (7)	−0.0024 (7)
C1	0.0537 (16)	0.0478 (15)	0.0340 (12)	−0.0140 (12)	0.0185 (11)	−0.0038 (11)
C2	0.0396 (13)	0.0344 (11)	0.0401 (12)	−0.0070 (10)	0.0113 (10)	0.0026 (10)
C3	0.0329 (12)	0.0283 (11)	0.0291 (11)	−0.0082 (9)	0.0025 (9)	0.0029 (9)
C4	0.0328 (12)	0.0334 (11)	0.0293 (10)	−0.0080 (9)	0.0050 (9)	0.0049 (9)
C5	0.0339 (13)	0.0637 (18)	0.0298 (11)	−0.0094 (11)	0.0020 (10)	0.0028 (11)
C6	0.0490 (16)	0.071 (2)	0.0270 (12)	−0.0193 (14)	0.0031 (10)	−0.0022 (12)

C7	0.0348 (13)	0.0327 (11)	0.0329 (11)	−0.0094 (9)	0.0005 (10)	0.0045 (10)
C8	0.0416 (14)	0.0323 (12)	0.0341 (12)	−0.0082 (9)	0.0042 (10)	−0.0004 (10)
C9	0.0401 (13)	0.0284 (12)	0.0490 (14)	−0.0012 (10)	0.0125 (11)	0.0033 (10)
C10	0.0330 (12)	0.0275 (11)	0.0261 (10)	0.0004 (8)	0.0057 (9)	−0.0005 (8)
C11	0.0284 (11)	0.0265 (10)	0.0287 (10)	0.0007 (8)	0.0074 (8)	−0.0002 (9)
C12	0.0288 (11)	0.0283 (11)	0.0230 (9)	−0.0028 (8)	0.0072 (8)	0.0006 (8)
C13	0.0229 (10)	0.0255 (10)	0.0205 (9)	0.0010 (7)	0.0029 (7)	0.0026 (8)
C14	0.0256 (11)	0.0269 (10)	0.0221 (9)	−0.0007 (8)	0.0044 (8)	0.0030 (8)
C15	0.0229 (10)	0.0217 (10)	0.0263 (10)	−0.0016 (7)	0.0036 (7)	−0.0004 (8)
C16	0.0237 (10)	0.0262 (11)	0.0223 (9)	0.0000 (7)	0.0039 (8)	−0.0019 (8)
C17	0.0308 (11)	0.0255 (10)	0.0209 (9)	0.0009 (8)	0.0067 (8)	0.0024 (8)
C18	0.0306 (12)	0.0286 (11)	0.0339 (11)	−0.0027 (8)	0.0132 (9)	−0.0015 (9)
C19	0.0295 (13)	0.0516 (15)	0.0547 (15)	−0.0071 (11)	0.0116 (11)	−0.0023 (13)
C20	0.0485 (15)	0.0424 (14)	0.0384 (12)	−0.0040 (11)	0.0228 (11)	−0.0029 (10)
C21	0.0272 (11)	0.0346 (11)	0.0267 (10)	−0.0047 (9)	0.0086 (8)	−0.0027 (9)
C22	0.0545 (18)	0.070 (2)	0.0521 (16)	0.0269 (15)	0.0212 (13)	0.0013 (15)
C23	0.0678 (19)	0.0529 (17)	0.0401 (14)	−0.0308 (14)	0.0225 (13)	−0.0071 (12)

Geometric parameters (Å, º)

F1—C1	1.362 (3)	C9—H9A	0.9900
O1—C7	1.204 (3)	C9—H9B	0.9900
O2—C8	1.209 (3)	C10—C11	1.369 (3)
O3—C17	1.402 (2)	C11—H11	0.9500
O3—C13	1.433 (2)	C12—C13	1.515 (3)
O4—C21	1.419 (3)	C12—H12A	0.9900
O4—C14	1.431 (2)	C12—H12B	0.9900
O5—C15	1.425 (3)	C13—C14	1.521 (3)
O5—C21	1.427 (2)	C13—H13	1.0000
O6—C16	1.426 (2)	C14—C15	1.538 (3)
O6—C18	1.430 (3)	C14—H14	1.0000
O7—C17	1.418 (3)	C15—C16	1.517 (3)
O7—C18	1.436 (3)	C15—H15	1.0000
N1—C8	1.360 (3)	C16—C17	1.545 (3)
N1—C4	1.415 (3)	C16—H16	1.0000
N1—C9	1.451 (3)	C17—H17	1.0000
N2—N3	1.315 (3)	C18—C19	1.504 (3)
N2—C10	1.355 (3)	C18—C20	1.521 (3)
N3—N4	1.343 (3)	C19—H19A	0.9800
N4—C11	1.348 (3)	C19—H19B	0.9800
N4—C12	1.455 (3)	C19—H19C	0.9800
C1—C2	1.372 (4)	C20—H20A	0.9800
C1—C6	1.375 (4)	C20—H20B	0.9800
C2—C3	1.383 (3)	C20—H20C	0.9800
C2—H2	0.9500	C21—C22	1.505 (4)
C3—C4	1.400 (3)	C21—C23	1.507 (3)
C3—C7	1.463 (3)	C22—H22A	0.9800
C4—C5	1.375 (3)	C22—H22B	0.9800

C5—C6	1.392 (4)	C22—H22C	0.9800
C5—H5	0.9500	C23—H23A	0.9800
C6—H6	0.9500	C23—H23B	0.9800
C7—C8	1.563 (4)	C23—H23C	0.9800
C9—C10	1.504 (3)		

C17—O3—C13	112.66 (15)	O4—C14—C13	111.41 (17)
C21—O4—C14	110.83 (15)	O4—C14—C15	103.52 (16)
C15—O5—C21	108.35 (15)	C13—C14—C15	112.20 (16)
C16—O6—C18	106.35 (15)	O4—C14—H14	109.8
C17—O7—C18	109.75 (15)	C13—C14—H14	109.8
C8—N1—C4	111.0 (2)	C15—C14—H14	109.8
C8—N1—C9	124.6 (2)	O5—C15—C16	106.43 (16)
C4—N1—C9	124.41 (19)	O5—C15—C14	104.10 (16)
N3—N2—C10	108.69 (18)	C16—C15—C14	114.71 (16)
N2—N3—N4	107.15 (17)	O5—C15—H15	110.4
N3—N4—C11	111.06 (18)	C16—C15—H15	110.4
N3—N4—C12	119.04 (18)	C14—C15—H15	110.4
C11—N4—C12	129.89 (18)	O6—C16—C15	107.93 (16)
F1—C1—C2	118.7 (3)	O6—C16—C17	104.15 (16)
F1—C1—C6	117.8 (2)	C15—C16—C17	113.75 (17)
C2—C1—C6	123.5 (2)	O6—C16—H16	110.3
C1—C2—C3	116.1 (2)	C15—C16—H16	110.3
C1—C2—H2	122.0	C17—C16—H16	110.3
C3—C2—H2	122.0	O3—C17—O7	110.45 (16)
C2—C3—C4	121.6 (2)	O3—C17—C16	114.11 (16)
C2—C3—C7	131.8 (2)	O7—C17—C16	104.05 (17)
C4—C3—C7	106.7 (2)	O3—C17—H17	109.4
C5—C4—C3	121.1 (2)	O7—C17—H17	109.4
C5—C4—N1	127.7 (2)	C16—C17—H17	109.4
C3—C4—N1	111.15 (18)	O6—C18—O7	104.61 (15)
C4—C5—C6	117.4 (3)	O6—C18—C19	108.17 (19)
C4—C5—H5	121.3	O7—C18—C19	109.86 (19)
C6—C5—H5	121.3	O6—C18—C20	110.99 (18)
C1—C6—C5	120.3 (2)	O7—C18—C20	109.60 (18)
C1—C6—H6	119.9	C19—C18—C20	113.3 (2)
C5—C6—H6	119.9	C18—C19—H19A	109.5
O1—C7—C3	131.2 (2)	C18—C19—H19B	109.5
O1—C7—C8	123.4 (2)	H19A—C19—H19B	109.5
C3—C7—C8	105.41 (19)	C18—C19—H19C	109.5
O2—C8—N1	127.8 (3)	H19A—C19—H19C	109.5
O2—C8—C7	126.6 (2)	H19B—C19—H19C	109.5
N1—C8—C7	105.63 (19)	C18—C20—H20A	109.5
N1—C9—C10	112.0 (2)	C18—C20—H20B	109.5
N1—C9—H9A	109.2	H20A—C20—H20B	109.5
C10—C9—H9A	109.2	C18—C20—H20C	109.5
N1—C9—H9B	109.2	H20A—C20—H20C	109.5
C10—C9—H9B	109.2	H20B—C20—H20C	109.5

H9A—C9—H9B	107.9	O4—C21—O5	106.43 (16)
N2—C10—C11	108.8 (2)	O4—C21—C22	109.0 (2)
N2—C10—C9	119.9 (2)	O5—C21—C22	107.85 (19)
C11—C10—C9	131.2 (2)	O4—C21—C23	108.8 (2)
N4—C11—C10	104.28 (19)	O5—C21—C23	110.09 (19)
N4—C11—H11	127.9	C22—C21—C23	114.3 (2)
C10—C11—H11	127.9	C21—C22—H22A	109.5
N4—C12—C13	111.82 (16)	C21—C22—H22B	109.5
N4—C12—H12A	109.3	H22A—C22—H22B	109.5
C13—C12—H12A	109.3	C21—C22—H22C	109.5
N4—C12—H12B	109.3	H22A—C22—H22C	109.5
C13—C12—H12B	109.3	H22B—C22—H22C	109.5
H12A—C12—H12B	107.9	C21—C23—H23A	109.5
O3—C13—C12	107.23 (16)	C21—C23—H23B	109.5
O3—C13—C14	110.57 (16)	H23A—C23—H23B	109.5
C12—C13—C14	110.90 (16)	C21—C23—H23C	109.5
O3—C13—H13	109.4	H23A—C23—H23C	109.5
C12—C13—H13	109.4	H23B—C23—H23C	109.5
C14—C13—H13	109.4		

C10—N2—N3—N4	−0.3 (2)	C11—N4—C12—C13	64.1 (3)
N2—N3—N4—C11	0.4 (2)	C17—O3—C13—C12	−168.58 (16)
N2—N3—N4—C12	−179.12 (17)	C17—O3—C13—C14	70.4 (2)
F1—C1—C2—C3	179.1 (2)	N4—C12—C13—O3	57.2 (2)
C6—C1—C2—C3	0.3 (4)	N4—C12—C13—C14	177.97 (17)
C1—C2—C3—C4	1.1 (3)	C21—O4—C14—C13	−111.73 (19)
C1—C2—C3—C7	178.9 (2)	C21—O4—C14—C15	9.1 (2)
C2—C3—C4—C5	−1.6 (4)	O3—C13—C14—O4	79.27 (19)
C7—C3—C4—C5	−179.9 (2)	C12—C13—C14—O4	−39.5 (2)
C2—C3—C4—N1	178.1 (2)	O3—C13—C14—C15	−36.3 (2)
C7—C3—C4—N1	−0.2 (2)	C12—C13—C14—C15	−155.10 (17)
C8—N1—C4—C5	177.1 (2)	C21—O5—C15—C16	148.22 (17)
C9—N1—C4—C5	0.0 (4)	C21—O5—C15—C14	26.7 (2)
C8—N1—C4—C3	−2.6 (3)	O4—C14—C15—O5	−21.5 (2)
C9—N1—C4—C3	−179.7 (2)	C13—C14—C15—O5	98.75 (18)
C3—C4—C5—C6	0.7 (4)	O4—C14—C15—C16	−137.38 (18)
N1—C4—C5—C6	−178.9 (2)	C13—C14—C15—C16	−17.1 (2)
F1—C1—C6—C5	−180.0 (3)	C18—O6—C16—C15	151.56 (16)
C2—C1—C6—C5	−1.1 (4)	C18—O6—C16—C17	30.35 (19)
C4—C5—C6—C1	0.6 (4)	O5—C15—C16—O6	172.38 (15)
C2—C3—C7—O1	4.5 (4)	C14—C15—C16—O6	−73.1 (2)
C4—C3—C7—O1	−177.5 (2)	O5—C15—C16—C17	−72.6 (2)
C2—C3—C7—C8	−175.5 (2)	C14—C15—C16—C17	41.9 (2)
C4—C3—C7—C8	2.5 (2)	C13—O3—C17—O7	73.2 (2)
C4—N1—C8—O2	−174.8 (3)	C13—O3—C17—C16	−43.6 (2)
C9—N1—C8—O2	2.2 (4)	C18—O7—C17—O3	−128.04 (17)
C4—N1—C8—C7	4.0 (2)	C18—O7—C17—C16	−5.2 (2)
C9—N1—C8—C7	−178.9 (2)	O6—C16—C17—O3	105.06 (18)

O1—C7—C8—O2	−5.2 (4)	C15—C16—C17—O3	−12.2 (3)
C3—C7—C8—O2	174.8 (2)	O6—C16—C17—O7	−15.4 (2)
O1—C7—C8—N1	176.0 (2)	C15—C16—C17—O7	−132.63 (17)
C3—C7—C8—N1	−4.1 (2)	C16—O6—C18—O7	−33.89 (19)
C8—N1—C9—C10	112.8 (2)	C16—O6—C18—C19	−150.96 (19)
C4—N1—C9—C10	−70.5 (3)	C16—O6—C18—C20	84.2 (2)
N3—N2—C10—C11	0.1 (2)	C17—O7—C18—O6	23.9 (2)
N3—N2—C10—C9	178.42 (19)	C17—O7—C18—C19	139.75 (19)
N1—C9—C10—N2	173.84 (19)	C17—O7—C18—C20	−95.2 (2)
N1—C9—C10—C11	−8.3 (4)	C14—O4—C21—O5	6.9 (2)
N3—N4—C11—C10	−0.3 (2)	C14—O4—C21—C22	123.0 (2)
C12—N4—C11—C10	179.12 (19)	C14—O4—C21—C23	−111.7 (2)
N2—C10—C11—N4	0.1 (2)	C15—O5—C21—O4	−21.6 (2)
C9—C10—C11—N4	−177.9 (2)	C15—O5—C21—C22	−138.4 (2)
N3—N4—C12—C13	−116.6 (2)	C15—O5—C21—C23	96.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O5^{i}	0.95	2.24	3.184 (3)	170
C15—H15···N2^{ii}	1.00	2.53	3.338 (3)	138

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