IUCrData

ISSN 2414-3146

1-[(1-{[(1*S*,2*R*,6*R*,8*R*,9*S*)-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

Fatima-Zahrae Qachchachi,[a] Joel T. Mague,[b] Youssef Kandri Rodi,[a] Amal Haoudi,[a]* Abdeslem Ben-Tama[a] and El Mokhtar Essassi[c]

[a]Laboratoire de Chimie Organique Appliquée, Université Sidi Mohamed Ben Abdallah, Faculté des Sciences et Techniques, Route d'Imouzzer, BP 2202, Fez, Morocco, [b]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA, and [c]Laboratoire de Chimie Organique Hétérocyclique URAC 21, Pôle de Compétence Pharmacochimie, Av. Ibn Battouta, BP 1014, Faculté des Sciences, Université Mohammed V, Rabat, Morocco. *Correspondence e-mail: amalhaoudi2017@gmail.com

Received 3 May 2017
Accepted 4 May 2017

Edited by M. Bolte, Goethe-Universität Frankfurt, Germany

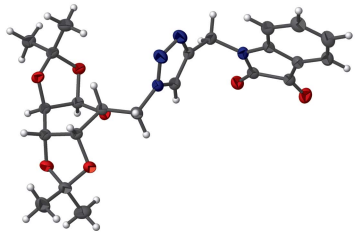
Keywords: crystal structure; isatins; triazole; hydrogen bond.

CCDC reference: 1547809

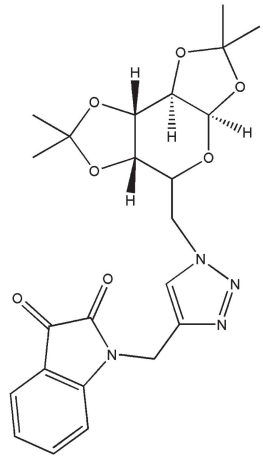
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{23}H_{26}N_4O_7$, adopts a Z-shaped conformation, with a dihedral angle between the mean planes of the dihydroindole and triazole rings of 70.36 (7)°. In the crystal, C—H···O interactions connect the molecules into zigzag chains running along the *b*-axis direction.

3D view



Chemical scheme



Structure description

Isatins are very useful intermediates/subunits for the development of molecules of pharmaceutical or biological interest. The wide variety of biological activities shown by isatin and its derivatives are relevant to applications as insecticides and fungicides and in a broad range of drug therapies, including anticancer drugs, antibiotics and anti-depressants (Bhrigu *et al.*, 2010; da Silva *et al.*, 2001; Ramachandran, 2011; Smitha *et al.*, 2008). As part of our studies in this area, we have undertaken the synthesis and structure determination of the title compound (Fig. 1).

The dihedral angle between the mean planes of the C1–C6 and triazole rings is 70.36 (7)°. A Cremer–Pople puckering analysis of the C13–C17,O3 ring gave parameters Q = 0.6549 (16) Å, θ = 99.92 (15)° and φ = 218.02 (15)°. For the O4,C14,C15,O5,C18 ring, the parameters are $Q(2)$ = 0.2959 (17) Å and $\varphi(2)$ = 307.9 (3)° while for the O6,C16,C17,O7,C21 ring they are $Q(2)$ = 0.2860 (16) Å and $\varphi(2)$ = 196.1 (3)°. The conformation of the O4,C16,C15,O5,C18 ring is twisted on O5—C18, that of the

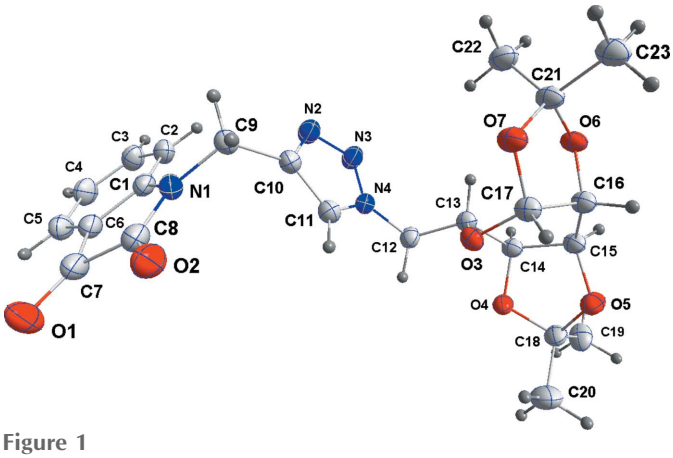


Figure 1
The title molecule with the atom-labelling scheme and 50% probability ellipsoids.

O6,C16,C17,O7,C21 ring is twisted on O6—C16 and that of the C13···C17,O3 ring is best described as a twist–boat.

In the crystal, C—H···O interactions (Table 1) connect the molecules into zigzag chains running along the *b*-axis direction. The crystal packing is shown in Fig. 2.

Synthesis and crystallization

To a solution of 1-(prop-2-yn-1-yl)idoline-2,3-dione (0.2 g, 1.08 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*-bis([1,3]-dioxolo)[4,5-*b*:4′,5′-*d*]pyran (0.46 g, 1.62 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 crystals in 62% yield.

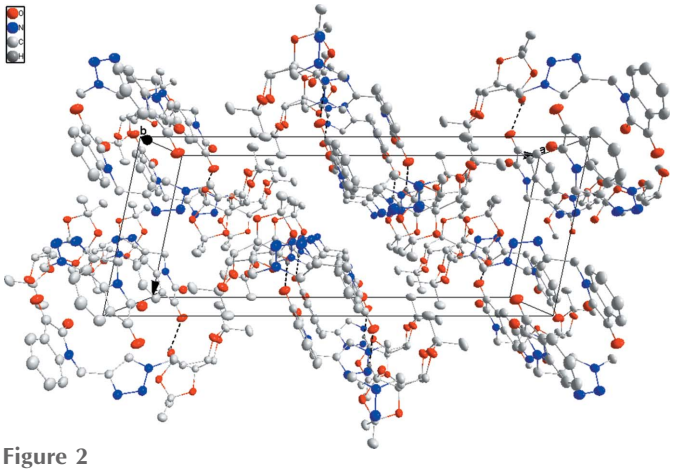


Figure 2
The packing viewed down the *b* axis. C—H···O interactions are shown as dotted lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17···O1^{i}	1.00	2.50	3.386 (2)	147

Symmetry code: (i) $-x+1, y-1, -z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{26}N_4O_7$
M_r	470.48
Crystal system, space group	Monoclinic, *C*2
Temperature (K)	150
a, *b*, *c* (Å)	25.6622 (8), 8.7731 (3), 10.3928 (3)
β (°)	101.847 (1)
V (Å^3)	2289.96 (13)
Z	4
Radiation type	Cu *K*α
μ (mm^{-1})	0.86
Crystal size (mm)	0.18 × 0.18 × 0.09
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.86, 0.92
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13935, 4250, 4142
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.619
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.025, 0.064, 1.02
No. of reflections	4250
No. of parameters	312
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.12
Absolute structure	Flack *x* determined using 1738 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.06 (5)

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

Refinement

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

Acknowledgements

The support of NSF-MRI Grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

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full crystallographic data

IUCrData (2017). **2**, x170670 [https://doi.org/10.1107/S2414314617006708]

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1-[(1-{[(1*S*,2*R*,6*R*,8*R*,9*S*)-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

$C_{23}H_{26}N_4O_7$
M_r = 470.48
Monoclinic, *C*2
a = 25.6622 (8) Å
b = 8.7731 (3) Å
c = 10.3928 (3) Å
β = 101.847 (1)°
V = 2289.96 (13) Å^3
Z = 4
F(000) = 992
D_x = 1.365 Mg m^{-3}
Cu *K*α radiation, λ = 1.54178 Å
Cell parameters from 9918 reflections
θ = 3.5–72.5°
μ = 0.86 mm^{-1}
T = 150 K
Thick plate, yellow
0.18 × 0.18 × 0.09 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} = 0.86, T_{max} = 0.92
13935 measured reflections
4250 independent reflections
4142 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 72.5°, θ_{min} = 3.5°
h = −31→31
k = −10→10
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.064
S = 1.02
4250 reflections
312 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.4737P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: SHELXL-2014/7 (Sheldrick 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00122 (12)

Absolute structure: Flack *x* determined using 1738 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.06 (5)

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 - 1.00 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached carbon atoms.
H atoms were placed in calculated positions (C—H = 0.95–1.00 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}
O1	0.38504 (6)	0.73451 (17)	−0.11559 (13)	0.0422 (3)
O2	0.42118 (5)	0.46504 (16)	0.04958 (13)	0.0361 (3)
O3	0.63848 (4)	0.14283 (14)	0.27385 (11)	0.0256 (3)
O4	0.73597 (5)	0.17670 (15)	0.17487 (12)	0.0318 (3)
O5	0.73377 (5)	−0.07332 (15)	0.22667 (12)	0.0306 (3)
O6	0.70228 (4)	0.00891 (15)	0.54268 (10)	0.0273 (3)
O7	0.61358 (4)	0.03958 (17)	0.45789 (11)	0.0308 (3)
N1	0.46396 (6)	0.65011 (18)	0.19074 (14)	0.0285 (3)
N2	0.57957 (6)	0.5944 (2)	0.41853 (16)	0.0361 (4)
N3	0.62799 (6)	0.5552 (2)	0.40657 (15)	0.0351 (4)
N4	0.62257 (5)	0.46388 (16)	0.30101 (14)	0.0263 (3)
C1	0.46996 (6)	0.8105 (2)	0.18610 (17)	0.0275 (4)
C2	0.50002 (7)	0.9035 (2)	0.28016 (18)	0.0327 (4)
H2	0.5212	0.8633	0.3586	0.039*
C3	0.49773 (7)	1.0598 (2)	0.2540 (2)	0.0364 (4)
H3	0.5180	1.1270	0.3165	0.044*
C4	0.46679 (8)	1.1205 (2)	0.1399 (2)	0.0378 (4)
H4	0.4657	1.2277	0.1267	0.045*
C5	0.43747 (7)	1.0249 (2)	0.04507 (18)	0.0333 (4)
H5	0.4165	1.0651	−0.0336	0.040*
C6	0.43983 (6)	0.8696 (2)	0.06881 (17)	0.0284 (4)
C7	0.41433 (7)	0.7402 (2)	−0.00880 (17)	0.0306 (4)
C8	0.43274 (7)	0.5968 (2)	0.07729 (17)	0.0289 (4)
C9	0.48516 (7)	0.5535 (2)	0.30425 (17)	0.0308 (4)
H9A	0.4664	0.4543	0.2936	0.037*
H9B	0.4780	0.6025	0.3847	0.037*
C10	0.54375 (6)	0.5266 (2)	0.32078 (15)	0.0277 (3)
C11	0.57112 (7)	0.4422 (2)	0.24590 (17)	0.0276 (3)

H11	0.5568	0.3816	0.1713	0.033*
C12	0.66918 (6)	0.3967 (2)	0.26235 (17)	0.0283 (3)
H12A	0.7002	0.4651	0.2885	0.034*
H12B	0.6623	0.3842	0.1657	0.034*
C13	0.68127 (6)	0.2430 (2)	0.32814 (15)	0.0242 (3)
H13	0.6823	0.2549	0.4243	0.029*
C14	0.73352 (6)	0.1746 (2)	0.31067 (15)	0.0252 (3)
H14	0.7641	0.2326	0.3635	0.030*
C15	0.73768 (6)	0.0043 (2)	0.34853 (15)	0.0259 (3)
H15	0.7729	−0.0171	0.4074	0.031*
C16	0.69277 (6)	−0.0520 (2)	0.41186 (15)	0.0253 (3)
H16	0.6923	−0.1660	0.4143	0.030*
C17	0.63744 (6)	0.0097 (2)	0.34884 (15)	0.0251 (3)
H17	0.6167	−0.0712	0.2926	0.030*
C18	0.75196 (7)	0.0290 (2)	0.13954 (17)	0.0307 (4)
C19	0.81202 (8)	0.0232 (3)	0.1570 (2)	0.0419 (4)
H19A	0.8284	0.0425	0.2493	0.063*
H19B	0.8237	0.1011	0.1015	0.063*
H19C	0.8228	−0.0777	0.1314	0.063*
C20	0.72339 (10)	−0.0058 (3)	0.00086 (19)	0.0489 (5)
H20A	0.7321	−0.1096	−0.0224	0.073*
H20B	0.7346	0.0668	−0.0597	0.073*
H20C	0.6849	0.0027	−0.0054	0.073*
C21	0.65171 (6)	0.0218 (2)	0.57957 (15)	0.0286 (4)
C22	0.65193 (8)	0.1628 (3)	0.66276 (19)	0.0388 (4)
H22A	0.6820	0.1581	0.7381	0.058*
H22B	0.6185	0.1685	0.6943	0.058*
H22C	0.6554	0.2533	0.6100	0.058*
C23	0.63892 (7)	−0.1216 (2)	0.64715 (18)	0.0353 (4)
H23A	0.6392	−0.2090	0.5885	0.053*
H23B	0.6036	−0.1119	0.6687	0.053*
H23C	0.6657	−0.1370	0.7281	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0486 (8)	0.0401 (8)	0.0323 (7)	0.0053 (6)	−0.0050 (6)	−0.0013 (6)
O2	0.0386 (7)	0.0316 (7)	0.0380 (7)	0.0035 (5)	0.0075 (5)	−0.0013 (5)
O3	0.0200 (5)	0.0306 (7)	0.0244 (5)	−0.0002 (4)	0.0002 (4)	0.0016 (5)
O4	0.0384 (7)	0.0323 (7)	0.0284 (6)	0.0081 (5)	0.0150 (5)	0.0038 (5)
O5	0.0343 (6)	0.0291 (6)	0.0313 (6)	0.0004 (5)	0.0140 (5)	−0.0033 (5)
O6	0.0210 (5)	0.0396 (7)	0.0210 (5)	0.0008 (5)	0.0035 (4)	0.0016 (5)
O7	0.0204 (5)	0.0467 (8)	0.0254 (5)	0.0041 (5)	0.0051 (4)	0.0023 (5)
N1	0.0249 (7)	0.0316 (8)	0.0286 (7)	0.0045 (6)	0.0044 (5)	0.0036 (6)
N2	0.0306 (7)	0.0428 (10)	0.0335 (8)	0.0077 (7)	0.0035 (6)	−0.0068 (7)
N3	0.0297 (7)	0.0396 (9)	0.0344 (8)	0.0064 (6)	0.0029 (6)	−0.0080 (7)
N4	0.0264 (7)	0.0260 (7)	0.0265 (6)	0.0035 (5)	0.0056 (5)	0.0003 (6)
C1	0.0208 (7)	0.0318 (9)	0.0313 (9)	0.0033 (6)	0.0089 (6)	0.0013 (7)

C2	0.0234 (7)	0.0403 (10)	0.0335 (8)	0.0035 (7)	0.0038 (7)	0.0000 (8)
C3	0.0258 (8)	0.0386 (11)	0.0432 (10)	−0.0026 (7)	0.0036 (7)	−0.0058 (8)
C4	0.0330 (9)	0.0310 (10)	0.0490 (11)	0.0013 (7)	0.0076 (8)	0.0007 (8)
C5	0.0301 (8)	0.0340 (10)	0.0355 (9)	0.0051 (7)	0.0062 (7)	0.0037 (8)
C6	0.0231 (7)	0.0326 (9)	0.0300 (8)	0.0039 (7)	0.0065 (6)	0.0019 (7)
C7	0.0291 (8)	0.0338 (9)	0.0297 (8)	0.0051 (7)	0.0077 (7)	0.0011 (7)
C8	0.0253 (7)	0.0324 (10)	0.0298 (8)	0.0039 (7)	0.0077 (6)	−0.0003 (7)
C9	0.0263 (8)	0.0383 (10)	0.0283 (8)	0.0055 (7)	0.0069 (6)	0.0075 (7)
C10	0.0274 (8)	0.0304 (9)	0.0249 (7)	0.0049 (7)	0.0042 (6)	0.0033 (7)
C11	0.0267 (7)	0.0289 (9)	0.0265 (7)	0.0013 (6)	0.0038 (6)	0.0004 (6)
C12	0.0254 (7)	0.0297 (9)	0.0312 (8)	0.0034 (7)	0.0094 (6)	0.0019 (7)
C13	0.0209 (7)	0.0275 (8)	0.0236 (7)	−0.0003 (6)	0.0036 (6)	−0.0010 (6)
C14	0.0216 (7)	0.0310 (9)	0.0235 (7)	0.0003 (6)	0.0055 (6)	0.0000 (6)
C15	0.0229 (7)	0.0310 (9)	0.0244 (7)	0.0026 (6)	0.0060 (6)	0.0012 (7)
C16	0.0228 (7)	0.0300 (9)	0.0232 (7)	0.0008 (6)	0.0049 (6)	0.0004 (6)
C17	0.0213 (7)	0.0298 (8)	0.0241 (7)	−0.0016 (6)	0.0043 (6)	−0.0008 (7)
C18	0.0342 (8)	0.0318 (9)	0.0294 (8)	0.0047 (7)	0.0141 (7)	0.0002 (7)
C19	0.0354 (9)	0.0421 (11)	0.0542 (11)	0.0077 (8)	0.0235 (8)	0.0085 (10)
C20	0.0599 (13)	0.0568 (14)	0.0307 (9)	0.0035 (11)	0.0108 (9)	−0.0072 (10)
C21	0.0222 (7)	0.0407 (10)	0.0233 (7)	0.0019 (7)	0.0053 (6)	0.0025 (7)
C22	0.0428 (10)	0.0442 (11)	0.0311 (9)	0.0017 (9)	0.0115 (8)	−0.0043 (8)
C23	0.0305 (8)	0.0449 (11)	0.0324 (9)	0.0003 (8)	0.0112 (7)	0.0059 (8)

Geometric parameters (Å, º)

O1—C7	1.207 (2)	C9—H9A	0.9900
O2—C8	1.213 (2)	C9—H9B	0.9900
O3—C17	1.408 (2)	C10—C11	1.368 (2)
O3—C13	1.4290 (19)	C11—H11	0.9500
O4—C14	1.4256 (19)	C12—C13	1.515 (2)
O4—C18	1.430 (2)	C12—H12A	0.9900
O5—C18	1.420 (2)	C12—H12B	0.9900
O5—C15	1.423 (2)	C13—C14	1.514 (2)
O6—C21	1.4313 (18)	C13—H13	1.0000
O6—C16	1.4344 (19)	C14—C15	1.543 (2)
O7—C17	1.4189 (19)	C14—H14	1.0000
O7—C21	1.4396 (19)	C15—C16	1.522 (2)
N1—C8	1.365 (2)	C15—H15	1.0000
N1—C1	1.418 (2)	C16—C17	1.535 (2)
N1—C9	1.464 (2)	C16—H16	1.0000
N2—N3	1.319 (2)	C17—H17	1.0000
N2—C10	1.359 (2)	C18—C20	1.508 (3)
N3—N4	1.343 (2)	C18—C19	1.515 (2)
N4—C11	1.341 (2)	C19—H19A	0.9800
N4—C12	1.462 (2)	C19—H19B	0.9800
C1—C2	1.381 (3)	C19—H19C	0.9800
C1—C6	1.402 (2)	C20—H20A	0.9800
C2—C3	1.396 (3)	C20—H20B	0.9800

C2—H2	0.9500	C20—H20C	0.9800
C3—C4	1.391 (3)	C21—C22	1.508 (3)
C3—H3	0.9500	C21—C23	1.510 (3)
C4—C5	1.392 (3)	C22—H22A	0.9800
C4—H4	0.9500	C22—H22B	0.9800
C5—C6	1.384 (3)	C22—H22C	0.9800
C5—H5	0.9500	C23—H23A	0.9800
C6—C7	1.467 (3)	C23—H23B	0.9800
C7—C8	1.560 (3)	C23—H23C	0.9800
C9—C10	1.497 (2)		

C17—O3—C13	113.08 (12)	O4—C14—C13	109.50 (13)
C14—O4—C18	108.40 (13)	O4—C14—C15	104.49 (13)
C18—O5—C15	107.02 (13)	C13—C14—C15	111.71 (13)
C21—O6—C16	107.23 (11)	O4—C14—H14	110.3
C17—O7—C21	110.78 (11)	C13—C14—H14	110.3
C8—N1—C1	111.06 (14)	C15—C14—H14	110.3
C8—N1—C9	123.58 (16)	O5—C15—C16	107.94 (13)
C1—N1—C9	125.25 (15)	O5—C15—C14	104.35 (12)
N3—N2—C10	108.73 (15)	C16—C15—C14	113.93 (13)
N2—N3—N4	106.91 (14)	O5—C15—H15	110.1
C11—N4—N3	111.19 (14)	C16—C15—H15	110.1
C11—N4—C12	127.95 (15)	C14—C15—H15	110.1
N3—N4—C12	120.80 (14)	O6—C16—C15	107.00 (13)
C2—C1—C6	121.68 (17)	O6—C16—C17	103.62 (12)
C2—C1—N1	127.68 (17)	C15—C16—C17	114.66 (14)
C6—C1—N1	110.63 (16)	O6—C16—H16	110.4
C1—C2—C3	116.58 (17)	C15—C16—H16	110.4
C1—C2—H2	121.7	C17—C16—H16	110.4
C3—C2—H2	121.7	O3—C17—O7	110.42 (14)
C4—C3—C2	122.39 (18)	O3—C17—C16	114.07 (13)
C4—C3—H3	118.8	O7—C17—C16	103.74 (12)
C2—C3—H3	118.8	O3—C17—H17	109.5
C3—C4—C5	120.29 (19)	O7—C17—H17	109.5
C3—C4—H4	119.9	C16—C17—H17	109.5
C5—C4—H4	119.9	O5—C18—O4	105.05 (12)
C6—C5—C4	118.00 (17)	O5—C18—C20	108.82 (16)
C6—C5—H5	121.0	O4—C18—C20	108.66 (16)
C4—C5—H5	121.0	O5—C18—C19	111.23 (15)
C5—C6—C1	121.01 (17)	O4—C18—C19	109.75 (16)
C5—C6—C7	131.76 (17)	C20—C18—C19	112.99 (16)
C1—C6—C7	107.22 (16)	C18—C19—H19A	109.5
O1—C7—C6	131.38 (18)	C18—C19—H19B	109.5
O1—C7—C8	123.52 (18)	H19A—C19—H19B	109.5
C6—C7—C8	105.10 (14)	C18—C19—H19C	109.5
O2—C8—N1	127.09 (17)	H19A—C19—H19C	109.5
O2—C8—C7	126.99 (16)	H19B—C19—H19C	109.5
N1—C8—C7	105.92 (15)	C18—C20—H20A	109.5

N1—C9—C10	112.49 (14)	C18—C20—H20B	109.5
N1—C9—H9A	109.1	H20A—C20—H20B	109.5
C10—C9—H9A	109.1	C18—C20—H20C	109.5
N1—C9—H9B	109.1	H20A—C20—H20C	109.5
C10—C9—H9B	109.1	H20B—C20—H20C	109.5
H9A—C9—H9B	107.8	O6—C21—O7	105.27 (11)
N2—C10—C11	108.34 (15)	O6—C21—C22	108.76 (15)
N2—C10—C9	121.73 (16)	O7—C21—C22	109.68 (16)
C11—C10—C9	129.88 (16)	O6—C21—C23	110.75 (15)
N4—C11—C10	104.82 (15)	O7—C21—C23	108.99 (14)
N4—C11—H11	127.6	C22—C21—C23	113.10 (14)
C10—C11—H11	127.6	C21—C22—H22A	109.5
N4—C12—C13	109.77 (13)	C21—C22—H22B	109.5
N4—C12—H12A	109.7	H22A—C22—H22B	109.5
C13—C12—H12A	109.7	C21—C22—H22C	109.5
N4—C12—H12B	109.7	H22A—C22—H22C	109.5
C13—C12—H12B	109.7	H22B—C22—H22C	109.5
H12A—C12—H12B	108.2	C21—C23—H23A	109.5
O3—C13—C14	109.68 (14)	C21—C23—H23B	109.5
O3—C13—C12	107.43 (13)	H23A—C23—H23B	109.5
C14—C13—C12	113.77 (13)	C21—C23—H23C	109.5
O3—C13—H13	108.6	H23A—C23—H23C	109.5
C14—C13—H13	108.6	H23B—C23—H23C	109.5
C12—C13—H13	108.6		

C10—N2—N3—N4	0.4 (2)	C17—O3—C13—C14	71.36 (16)
N2—N3—N4—C11	−0.8 (2)	C17—O3—C13—C12	−164.51 (12)
N2—N3—N4—C12	−178.34 (16)	N4—C12—C13—O3	67.01 (16)
C8—N1—C1—C2	−177.55 (17)	N4—C12—C13—C14	−171.38 (14)
C9—N1—C1—C2	6.2 (3)	C18—O4—C14—C13	−131.51 (15)
C8—N1—C1—C6	3.03 (19)	C18—O4—C14—C15	−11.73 (16)
C9—N1—C1—C6	−173.24 (15)	O3—C13—C14—O4	69.64 (17)
C6—C1—C2—C3	1.7 (3)	C12—C13—C14—O4	−50.70 (19)
N1—C1—C2—C3	−177.68 (17)	O3—C13—C14—C15	−45.62 (17)
C1—C2—C3—C4	0.3 (3)	C12—C13—C14—C15	−165.96 (13)
C2—C3—C4—C5	−1.4 (3)	C18—O5—C15—C16	147.57 (14)
C3—C4—C5—C6	0.6 (3)	C18—O5—C15—C14	26.05 (16)
C4—C5—C6—C1	1.3 (3)	O4—C14—C15—O5	−8.66 (16)
C4—C5—C6—C7	179.91 (18)	C13—C14—C15—O5	109.63 (14)
C2—C1—C6—C5	−2.5 (3)	O4—C14—C15—C16	−126.14 (13)
N1—C1—C6—C5	176.93 (16)	C13—C14—C15—C16	−7.85 (18)
C2—C1—C6—C7	178.58 (15)	C21—O6—C16—C15	152.78 (14)
N1—C1—C6—C7	−1.97 (19)	C21—O6—C16—C17	31.24 (16)
C5—C6—C7—O1	1.4 (3)	O5—C15—C16—O6	171.12 (12)
C1—C6—C7—O1	−179.84 (19)	C14—C15—C16—O6	−73.50 (16)
C5—C6—C7—C8	−178.37 (18)	O5—C15—C16—C17	−74.59 (18)
C1—C6—C7—C8	0.35 (18)	C14—C15—C16—C17	40.79 (19)
C1—N1—C8—O2	178.09 (17)	C13—O3—C17—O7	80.43 (15)

C9—N1—C8—O2	−5.6 (3)	C13—O3—C17—C16	−35.92 (18)
C1—N1—C8—C7	−2.64 (17)	C21—O7—C17—O3	−114.66 (15)
C9—N1—C8—C7	173.71 (14)	C21—O7—C17—C16	7.95 (19)
O1—C7—C8—O2	0.8 (3)	O6—C16—C17—O3	96.50 (15)
C6—C7—C8—O2	−179.34 (17)	C15—C16—C17—O3	−19.7 (2)
O1—C7—C8—N1	−178.44 (17)	O6—C16—C17—O7	−23.67 (16)
C6—C7—C8—N1	1.39 (17)	C15—C16—C17—O7	−139.92 (14)
C8—N1—C9—C10	107.47 (19)	C15—O5—C18—O4	−33.89 (16)
C1—N1—C9—C10	−76.7 (2)	C15—O5—C18—C20	−150.09 (15)
N3—N2—C10—C11	0.1 (2)	C15—O5—C18—C19	84.80 (17)
N3—N2—C10—C9	−177.57 (17)	C14—O4—C18—O5	28.08 (17)
N1—C9—C10—N2	107.04 (19)	C14—O4—C18—C20	144.40 (16)
N1—C9—C10—C11	−70.1 (3)	C14—O4—C18—C19	−91.59 (16)
N3—N4—C11—C10	0.9 (2)	C16—O6—C21—O7	−26.73 (18)
C12—N4—C11—C10	178.17 (16)	C16—O6—C21—C22	−144.20 (14)
N2—C10—C11—N4	−0.6 (2)	C16—O6—C21—C23	90.93 (16)
C9—C10—C11—N4	176.84 (18)	C17—O7—C21—O6	10.9 (2)
C11—N4—C12—C13	−86.2 (2)	C17—O7—C21—C22	127.73 (15)
N3—N4—C12—C13	90.85 (19)	C17—O7—C21—C23	−107.96 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17···O1^{i}	1.00	2.50	3.386 (2)	147

Symmetry code: (i) −*x*+1, *y*−1, −*z*.