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Methyl (1-benzamido-2-methoxy-2-oxoethyl)-tryptophanate

Oumaima Karai,[a] Younas Aouine,[b] Hassane Faraj,[b] Anouar Alami,[b]* Abdelilah El Hallaoui[b] and Hafid Zouihri[c]

[a]Formation Doctorale Molécules Bioactives, Santé et Biotechnologies, Centre d'études Doctorales Sciences et Technologies LCO, Faculté des Sciences Dhar El Marhaz, Fès, Morocco, [b]Laboratoire de Chimie Organique, Faculté des Sciences Dhar el Mahraz, Université Sidi Mohammed Ben Abdellah, Fès, Morocco, and [c]Laboratoire de Chimie des Matériaux et Biotechnologie des Produits Naturels, E.Ma.Me.P.S., Université Moulay Ismail, Faculté des Sciences, Meknès, Morocco. *Correspondence e-mail: anouar.alami@usmba.ac.ma

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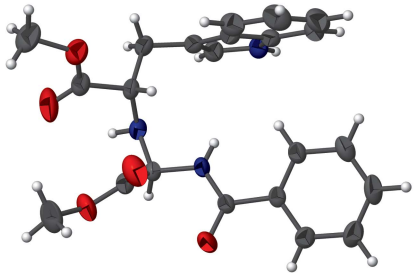
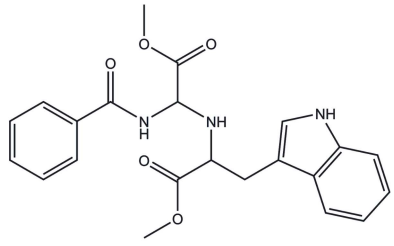
The title molecule, $C_{22}H_{23}N_3O_2$, is U-shaped, with a dihedral angle of 80.76 (9)° between the indole ring system and the phenyl ring. In the crystal, N—H···O hydrogen bonds combine with N—H···π and C—H···π interactions to generate a three-dimensional structure.

3D view



Chemical scheme



Structure description

The molecule of the title compound is approximately U-shaped, Fig. 1, with the indole ring system and the benzene ring linked by a complex alaninate chain and with a dihedral angle of 80.76 (9)° between them. This conformation is supported by an intramolecular C5—H5···*Cg*3 interaction (Table 1).

In the crystal, classical N3—H3*N*···O1 hydrogen bonds together with unusual intermolecular N2—H2*N*···*Cg*2 contacts and C20—H20···*Cg*1 interactions combine to generate a three dimensional network, Fig. 2.

Synthesis and crystallization

To a solution of 2.6 mmol of *N*-protected methyl α-azidoglycinate and 3.12 mmol of diisopropylethylamine (DIEA) in 10 ml of acetone, 2.86 mmol of 2-amino-3-(1*H*-indol-3-yl)propanoate was added. The reaction mixture was stirred at room temperature. The solvent was evaporated under reduced pressure. The residue was quenched with saturated aqueous solution of ammonium chloride (20 ml) and extracted with methylene chloride (3 × 20 ml). The organic layer was dried over sodium sulfate (Na_2SO_4) and the

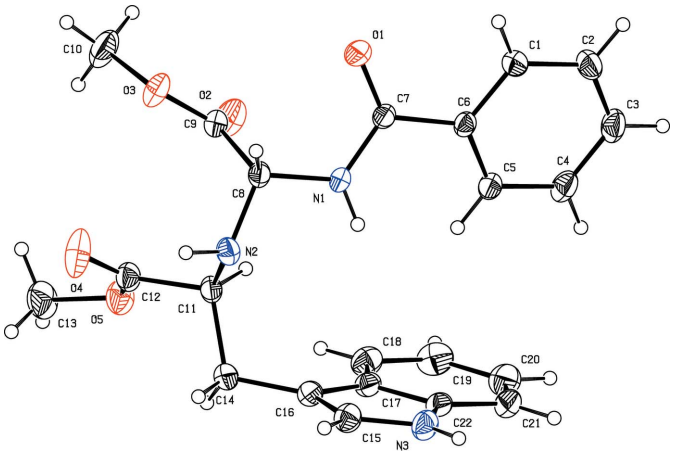


Figure 1
The structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

solvent was removed under reduced pressure. Single crystals of the title compound were obtained by recrystallization from chloroform ($CHCl_3$) solution, yield = 86% (white solid); m.p. = 174–176°C.

^{1}H NMR (DMSO, δ_H p.p.m.): 2.94–3.02 (*m*, 3H, *NH*—CH—CH_2– and –*CH*$_2$-indol-3-yl); 3.28 (*s*, 3H, –O*CH*$_3$); 3.64 (*s*, 3H, –O*CH*$_3$); 3.67–3.72 (*t*, 1H, N—*CH*—CH_2–, *J* = 6.90 Hz); 5.25–5.31 (*dd*, 1H, N—*CH*—N, J_1 = 10.17 Hz and J_2 = 7.73 Hz); 6.93–7.82 (*m*, 10H, 10H$_{arom}$); 9.1 (*s*, 1H, *NH*Bz); 10.9 (*s*, 1H, *NH*$_{indole}$). ^{13}C NMR (DMSO, δ_C p.p.m.): 29.18 (1 C, –*C*H_2-indol-3-yl); 51.78 and 52.65 (2 C, –O*C*H_3); 59.06 (1 C, –*C*H—CH_2-indol-3-yl); 64.18 (1 C, N—*C*H—N); 109.80–136.48 (14 C, *C*$_{arom}$); 166.61, 170.38 and 174.89 (3 C, *C*O). Calculated for $C_{22}H_{23}N_3O_5$ (%): C, 64.54; H, 5.66; N, 10.26; found (%): C 64.28, H 5.71, N 10.16. MS ESI *m*/*z* (%) = 409.60.

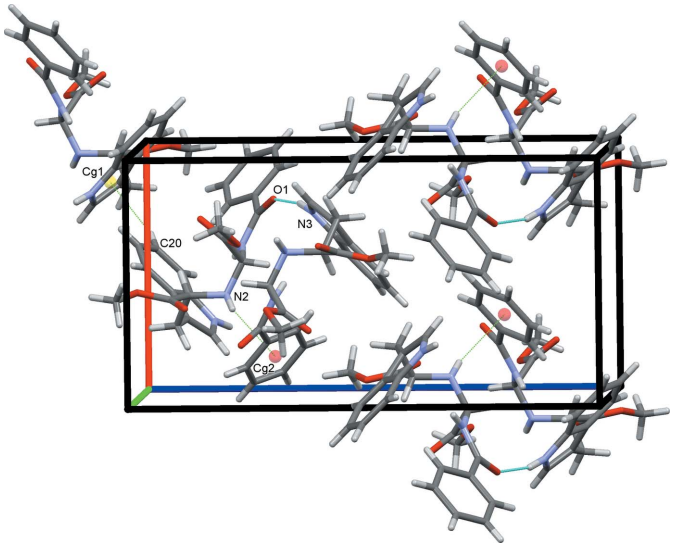


Figure 2
A view of the crystal packing of the title compound along the *b* axis. N—H···O hydrogen bonds are drawn as blue dashed lines with N—H···π and C—H···π contacts shown as dotted green lines. Ring centroids are displayed as coloured spheres.

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

*Cg*1, *Cg*2 and *Cg*3 are the centroids of the N3/C15–C17/C22, C1–C6 and C17–C22 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O1^{i}	0.87 (2)	2.03 (2)	2.862 (2)	162 (3)
N2—H2*N*···*Cg*2^{ii}	0.89 (2)	2.65 (3)	3.4451 (18)	149 (2)
C5—H5···*Cg*3	0.93	2.83	3.662 (2)	149
C20—H20···*Cg*1^{iii}	0.93	2.83	3.684 (3)	154

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{23}N_3O_5$
M_r	409.43
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	293
a, *b*, *c* (Å)	9.5716 (3), 11.6875 (4), 18.1827 (6)
V (Å^3)	2034.06 (12)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.10
Crystal size (mm)	0.23 × 0.21 × 0.14
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2005)
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22029, 4675, 4359
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.034, 0.097, 1.05
No. of reflections	4675
No. of parameters	285
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.22, −0.17
Absolute structure	Flack *x* determined using 1822 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).
Absolute structure parameter	0.0 (2)

Computer programs: *APEX2* and *SAINT* (Bruker, 2005), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. With no heavy atoms in the molecule the absolute structure could not be determined reliably.

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full crystallographic data

IUCrData (2017). **2**, x171547 [https://doi.org/10.1107/S2414314617015474]

Methyl (1-benzamido-2-methoxy-2-oxoethyl)tryptophanate

Oumaima Karai, Younas Aouine, Hassane Faraj, Anouar Alami, Abdelilah El Hallaoui and Hafid Zouihri

Methyl 3-(1*H*-indol-3-yl)-2-{[2-methoxy-2-oxo-1-(phenylformamido)ethyl]amino}propanoate

Crystal data

$C_{22}H_{23}N_3O_5$
M_r = 409.43
Orthorhombic, $P2_12_12_1$
a = 9.5716 (3) Å
b = 11.6875 (4) Å
c = 18.1827 (6) Å
V = 2034.06 (12) Å^3
Z = 4
$F(000)$ = 864
D_x = 1.337 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 275 reflections
θ = 1.3–57°
μ = 0.10 mm^{-1}
T = 293 K
Prism, colourless
0.23 × 0.21 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scan
Absorption correction: multi-scan (SADABS; Bruker, 2005)
22029 measured reflections
4675 independent reflections
4359 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°, θ_{min} = 2.1°
h = −12→12
k = −15→15
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.097
S = 1.05
4675 reflections
285 parameters
3 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.2737P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Absolute structure: Flack x determined using 1822 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).
Absolute structure parameter: 0.0 (2)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C7	0.71253 (18)	0.07261 (14)	0.23815 (10)	0.0313 (3)
C6	0.77399 (19)	−0.04380 (14)	0.22646 (10)	0.0309 (3)
C5	0.7366 (2)	−0.11460 (15)	0.16841 (10)	0.0361 (4)
H5	0.6644	−0.0934	0.1370	0.043*
C1	0.8802 (2)	−0.07750 (17)	0.27415 (11)	0.0393 (4)
H1	0.9056	−0.0306	0.3133	0.047*
C4	0.8070 (2)	−0.21720 (17)	0.15728 (13)	0.0464 (5)
H4	0.7830	−0.2640	0.1179	0.056*
C2	0.9483 (2)	−0.18102 (19)	0.26330 (14)	0.0486 (5)
H2	1.0180	−0.2041	0.2957	0.058*
C3	0.9124 (2)	−0.24964 (19)	0.20450 (14)	0.0510 (5)
H3	0.9596	−0.3181	0.1967	0.061*
N1	0.58969 (17)	0.09702 (13)	0.20564 (9)	0.0349 (3)
C8	0.52998 (19)	0.21167 (15)	0.21117 (10)	0.0327 (4)
H8	0.5171	0.2306	0.2632	0.039*
C9	0.6299 (2)	0.29926 (15)	0.17664 (11)	0.0365 (4)
O1	0.77431 (17)	0.14492 (12)	0.27462 (9)	0.0497 (4)
O3	0.61318 (19)	0.40054 (12)	0.20853 (9)	0.0517 (4)
O2	0.70594 (19)	0.28145 (14)	0.12588 (10)	0.0582 (4)
C10	0.6864 (3)	0.4950 (2)	0.17486 (19)	0.0675 (7)
H10A	0.6760	0.5621	0.2049	0.101*
H10B	0.6480	0.5094	0.1270	0.101*
H10C	0.7837	0.4764	0.1704	0.101*
N2	0.39521 (17)	0.21076 (14)	0.17551 (9)	0.0352 (3)
C12	0.3859 (2)	0.33133 (17)	0.06305 (11)	0.0401 (4)
C11	0.3936 (2)	0.21081 (15)	0.09530 (10)	0.0340 (4)
H11	0.4796	0.1744	0.0777	0.041*
C13	0.3913 (3)	0.4341 (2)	−0.04853 (15)	0.0638 (7)
H13A	0.4554	0.4918	−0.0320	0.096*
H13B	0.2972	0.4595	−0.0403	0.096*
H13C	0.4052	0.4205	−0.1001	0.096*
O5	0.41550 (19)	0.32938 (14)	−0.00816 (9)	0.0514 (4)
O4	0.3559 (3)	0.41515 (15)	0.09638 (11)	0.0743 (6)
N3	0.2625 (2)	−0.15885 (14)	0.13318 (10)	0.0431 (4)
C17	0.3845 (2)	−0.05961 (16)	0.04926 (10)	0.0370 (4)
C16	0.2847 (2)	0.01589 (16)	0.08328 (11)	0.0379 (4)
C14	0.2676 (2)	0.14084 (17)	0.06700 (12)	0.0413 (4)
H14A	0.1828	0.1686	0.0902	0.050*
H14B	0.2583	0.1516	0.0143	0.050*
C15	0.2145 (2)	−0.04813 (17)	0.13370 (11)	0.0422 (4)
H15	0.1441	−0.0208	0.1642	0.051*
C22	0.3673 (2)	−0.16815 (16)	0.08245 (11)	0.0381 (4)
C18	0.4861 (2)	−0.0466 (2)	−0.00585 (12)	0.0480 (5)
H18	0.4993	0.0237	−0.0288	0.058*
C21	0.4464 (3)	−0.26306 (19)	0.06147 (12)	0.0481 (5)

H21	0.4324	−0.3344	0.0829	0.058*
C19	0.5659 (3)	−0.1399 (2)	−0.02529 (14)	0.0584 (6)
H19	0.6345	−0.1318	−0.0611	0.070*
C20	0.5456 (3)	−0.2470 (2)	0.00800 (14)	0.0564 (6)
H20	0.6007	−0.3085	−0.0065	0.068*
H2N	0.343 (3)	0.2689 (19)	0.1905 (14)	0.047 (7)*
H1N	0.537 (2)	0.0475 (18)	0.1878 (12)	0.036 (6)*
H3N	0.233 (3)	−0.212 (2)	0.1624 (15)	0.070 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C7	0.0333 (8)	0.0256 (7)	0.0350 (8)	−0.0013 (7)	0.0002 (7)	−0.0008 (6)
C6	0.0307 (8)	0.0247 (7)	0.0373 (8)	−0.0019 (6)	0.0032 (7)	0.0033 (6)
C5	0.0382 (9)	0.0278 (8)	0.0422 (10)	−0.0006 (7)	0.0000 (8)	0.0005 (7)
C1	0.0341 (8)	0.0367 (9)	0.0472 (10)	−0.0005 (8)	−0.0037 (8)	0.0032 (8)
C4	0.0507 (11)	0.0310 (9)	0.0574 (12)	−0.0001 (8)	0.0075 (9)	−0.0088 (8)
C2	0.0337 (9)	0.0427 (11)	0.0694 (14)	0.0071 (8)	−0.0038 (9)	0.0087 (10)
C3	0.0427 (11)	0.0310 (9)	0.0793 (15)	0.0081 (8)	0.0102 (10)	−0.0008 (10)
N1	0.0366 (8)	0.0219 (6)	0.0463 (8)	0.0003 (6)	−0.0065 (7)	−0.0013 (6)
C8	0.0366 (9)	0.0256 (8)	0.0358 (8)	0.0032 (7)	−0.0010 (7)	0.0002 (7)
C9	0.0394 (9)	0.0290 (8)	0.0411 (9)	0.0009 (7)	−0.0015 (8)	0.0024 (7)
O1	0.0485 (8)	0.0338 (7)	0.0669 (9)	0.0035 (6)	−0.0163 (7)	−0.0159 (7)
O3	0.0640 (10)	0.0268 (7)	0.0643 (9)	−0.0053 (7)	0.0080 (8)	−0.0036 (6)
O2	0.0665 (10)	0.0450 (8)	0.0630 (9)	−0.0052 (8)	0.0244 (9)	−0.0024 (7)
C10	0.0796 (18)	0.0320 (11)	0.0910 (19)	−0.0118 (11)	0.0063 (16)	0.0059 (12)
N2	0.0335 (7)	0.0330 (7)	0.0391 (8)	0.0057 (6)	0.0023 (6)	0.0022 (6)
C12	0.0409 (10)	0.0335 (9)	0.0460 (10)	0.0008 (8)	0.0002 (8)	0.0089 (8)
C11	0.0343 (8)	0.0279 (8)	0.0396 (9)	0.0044 (7)	−0.0009 (7)	0.0047 (7)
C13	0.0725 (16)	0.0576 (15)	0.0614 (14)	−0.0001 (13)	−0.0020 (13)	0.0283 (12)
O5	0.0655 (10)	0.0446 (8)	0.0442 (8)	0.0014 (7)	−0.0002 (7)	0.0135 (6)
O4	0.1215 (18)	0.0361 (8)	0.0655 (11)	0.0202 (10)	0.0198 (11)	0.0083 (8)
N3	0.0521 (10)	0.0345 (8)	0.0429 (9)	−0.0053 (7)	0.0003 (8)	0.0085 (7)
C17	0.0414 (10)	0.0336 (9)	0.0360 (9)	−0.0049 (8)	−0.0063 (8)	0.0004 (7)
C16	0.0416 (10)	0.0335 (9)	0.0386 (9)	−0.0027 (8)	−0.0074 (8)	0.0023 (7)
C14	0.0405 (10)	0.0345 (9)	0.0488 (11)	−0.0003 (8)	−0.0099 (9)	0.0080 (8)
C15	0.0457 (10)	0.0388 (9)	0.0421 (10)	−0.0012 (9)	0.0009 (9)	0.0026 (8)
C22	0.0417 (10)	0.0340 (9)	0.0386 (9)	−0.0050 (8)	−0.0091 (8)	−0.0005 (8)
C18	0.0519 (12)	0.0469 (12)	0.0451 (11)	−0.0085 (10)	0.0022 (9)	0.0012 (9)
C21	0.0572 (12)	0.0339 (10)	0.0534 (12)	−0.0001 (9)	−0.0111 (10)	−0.0056 (9)
C19	0.0559 (14)	0.0651 (15)	0.0543 (13)	−0.0040 (12)	0.0085 (11)	−0.0120 (11)
C20	0.0565 (13)	0.0512 (12)	0.0614 (13)	0.0065 (11)	−0.0012 (11)	−0.0177 (11)

Geometric parameters (Å, º)

C7—O1	1.226 (2)	C12—O5	1.326 (3)
C7—N1	1.347 (2)	C12—C11	1.528 (2)
C7—C6	1.497 (2)	C11—C14	1.546 (3)

C6—C5	1.388 (3)	C11—H11	0.9800
C6—C1	1.393 (3)	C13—O5	1.446 (3)
C5—C4	1.391 (3)	C13—H13A	0.9600
C5—H5	0.9300	C13—H13B	0.9600
C1—C2	1.388 (3)	C13—H13C	0.9600
C1—H1	0.9300	N3—C22	1.367 (3)
C4—C3	1.378 (3)	N3—C15	1.373 (3)
C4—H4	0.9300	N3—H3N	0.87 (2)
C2—C3	1.380 (3)	C17—C18	1.405 (3)
C2—H2	0.9300	C17—C22	1.414 (3)
C3—H3	0.9300	C17—C16	1.440 (3)
N1—C8	1.460 (2)	C16—C15	1.361 (3)
N1—H1N	0.831 (19)	C16—C14	1.499 (3)
C8—N2	1.444 (2)	C14—H14A	0.9700
C8—C9	1.535 (3)	C14—H14B	0.9700
C8—H8	0.9800	C15—H15	0.9300
C9—O2	1.194 (3)	C22—C21	1.396 (3)
C9—O3	1.328 (2)	C18—C19	1.377 (4)
O3—C10	1.444 (3)	C18—H18	0.9300
C10—H10A	0.9600	C21—C20	1.372 (4)
C10—H10B	0.9600	C21—H21	0.9300
C10—H10C	0.9600	C19—C20	1.404 (4)
N2—C11	1.459 (2)	C19—H19	0.9300
N2—H2N	0.89 (2)	C20—H20	0.9300
C12—O4	1.187 (3)		

O1—C7—N1	120.83 (16)	N2—C11—C12	112.63 (16)
O1—C7—C6	120.90 (16)	N2—C11—C14	109.92 (16)
N1—C7—C6	118.24 (15)	C12—C11—C14	108.79 (15)
C5—C6—C1	119.54 (17)	N2—C11—H11	108.5
C5—C6—C7	123.24 (16)	C12—C11—H11	108.5
C1—C6—C7	117.08 (16)	C14—C11—H11	108.5
C6—C5—C4	119.97 (18)	O5—C13—H13A	109.5
C6—C5—H5	120.0	O5—C13—H13B	109.5
C4—C5—H5	120.0	H13A—C13—H13B	109.5
C2—C1—C6	120.0 (2)	O5—C13—H13C	109.5
C2—C1—H1	120.0	H13A—C13—H13C	109.5
C6—C1—H1	120.0	H13B—C13—H13C	109.5
C3—C4—C5	120.1 (2)	C12—O5—C13	116.56 (19)
C3—C4—H4	119.9	C22—N3—C15	108.96 (16)
C5—C4—H4	119.9	C22—N3—H3N	127 (2)
C3—C2—C1	120.0 (2)	C15—N3—H3N	124 (2)
C3—C2—H2	120.0	C18—C17—C22	118.80 (19)
C1—C2—H2	120.0	C18—C17—C16	134.39 (19)
C4—C3—C2	120.33 (19)	C22—C17—C16	106.81 (18)
C4—C3—H3	119.8	C15—C16—C17	106.25 (17)
C2—C3—H3	119.8	C15—C16—C14	127.9 (2)
C7—N1—C8	120.39 (15)	C17—C16—C14	125.78 (19)

C7—N1—H1N	123.4 (16)	C16—C14—C11	111.37 (16)
C8—N1—H1N	115.5 (16)	C16—C14—H14A	109.4
N2—C8—N1	108.18 (15)	C11—C14—H14A	109.4
N2—C8—C9	112.20 (15)	C16—C14—H14B	109.4
N1—C8—C9	109.88 (15)	C11—C14—H14B	109.4
N2—C8—H8	108.8	H14A—C14—H14B	108.0
N1—C8—H8	108.8	C16—C15—N3	110.40 (19)
C9—C8—H8	108.8	C16—C15—H15	124.8
O2—C9—O3	124.51 (19)	N3—C15—H15	124.8
O2—C9—C8	125.43 (18)	N3—C22—C21	130.20 (19)
O3—C9—C8	109.93 (16)	N3—C22—C17	107.58 (17)
C9—O3—C10	115.99 (19)	C21—C22—C17	122.2 (2)
O3—C10—H10A	109.5	C19—C18—C17	118.8 (2)
O3—C10—H10B	109.5	C19—C18—H18	120.6
H10A—C10—H10B	109.5	C17—C18—H18	120.6
O3—C10—H10C	109.5	C20—C21—C22	117.4 (2)
H10A—C10—H10C	109.5	C20—C21—H21	121.3
H10B—C10—H10C	109.5	C22—C21—H21	121.3
C8—N2—C11	117.27 (15)	C18—C19—C20	121.2 (2)
C8—N2—H2N	111.2 (17)	C18—C19—H19	119.4
C11—N2—H2N	107.5 (16)	C20—C19—H19	119.4
O4—C12—O5	124.37 (19)	C21—C20—C19	121.6 (2)
O4—C12—C11	125.23 (19)	C21—C20—H20	119.2
O5—C12—C11	110.40 (17)	C19—C20—H20	119.2

Hydrogen-bond geometry (Å, º)

Cg1, Cg2 and Cg3 are the centroids of the N3/C15–C17/C22, C1–C6 and C17–C22 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O1^{i}	0.87 (2)	2.03 (2)	2.862 (2)	162 (3)
N2—H2*N*···*Cg*2^{ii}	0.89 (2)	2.65 (3)	3.4451 (18)	149 (2)
C5—H5···*Cg*3	0.93	2.83	3.662 (2)	149
C20—H20···*Cg*1^{iii}	0.93	2.83	3.684 (3)	154

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