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3-{5-Ethoxycarbonyl-1-[3-(2-oxopyrrolidin-1-yl)propyl]-1*H*-benzimidazol-2-yl}-benzoic acid

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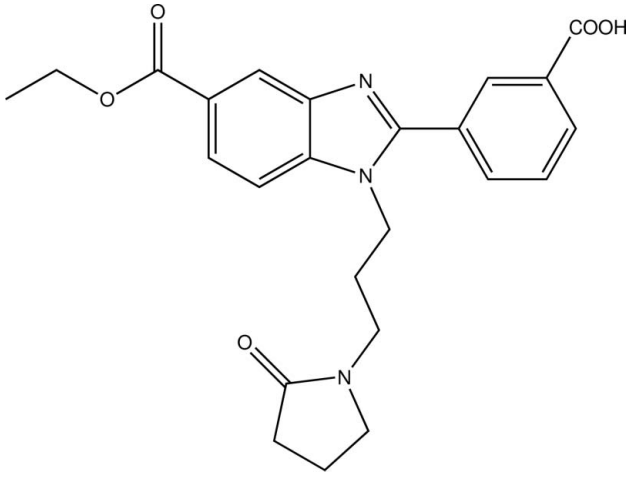
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.064; wR factor = 0.134; data-to-parameter ratio = 19.0.

In the title compound, $C_{24}H_{25}N_3O_5$, the ethoxy group is disordered over two orientations in a 0.853 (14):0.147 (14) ratio. The benzimadazole ring system (r.m.s. deviation = 0.016 Å) makes a dihedral angle of 35.47 (7)° with the attached benzene ring. The pyrrolidine ring adopts an envelope conformation with a methylene C atom as the flap. In the crystal, inversion dimers linked by pairs of O—H···N hydrogen bonds generate $R_2^2(16)$ loops. C—H···O interactions link the dimers into a three-dimensional network.

Related literature

For a related structure and background to benzimidazoles, see: Yoon *et al.* (2012). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{24}H_{25}N_3O_5$
M_r = 435.47
Monoclinic, $P2_1/c$
a = 9.3097 (3) Å
b = 24.8252 (7) Å
c = 9.6761 (3) Å
β = 112.322 (1)°
V = 2068.71 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.39 × 0.23 × 0.08 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.963, T_{max} = 0.992
23636 measured reflections
6010 independent reflections
3706 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.134
S = 1.03
6010 reflections
317 parameters
69 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1*O2*···N2^{i}	1.04 (4)	1.66 (4)	2.675 (2)	166 (3)
C5—H5*A*···O3^{i}	0.95	2.57	3.367 (2)	141
C9—H9*A*···O2^{i}	0.95	2.46	3.177 (2)	132
C15—H15*A*···O3^{ii}	0.99	2.34	3.314 (3)	167
C17—H17*B*···O5^{iii}	0.99	2.51	3.429 (3)	154
C20—H20*B*···O4^{iv}	0.99	2.55	3.340 (3)	137

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7017).

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Yoon, Y. K., Ali, M. A., Choon, T. S., Asik, S. I. J. & Razak, I. A. (2012). *Acta Cryst.* E**68**, o59.

supporting information

Acta Cryst. (2013). E**69**, o304 [doi:10.1107/S1600536813001116]

3-{5-Ethoxycarbonyl-1-[3-(2-oxopyrrolidin-1-yl)propyl]-1*H*-benzimidazol-2-yl}benzoic acid

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S1. Comment

As part of our ongoing structural studies of benzimidazole derivatives (Yoon *et al.*, 2012), we now report the structure of the title compound.

The ethoxy group is disordered over two position with the final refined occupancies of 0.853 (14):0.147 (14). The benzimidazole (N1—N2/C—C7) ring in the title compound, Fig. 1, is almost planar with maximum deviation of 0.02 Å for atom N2 and makes a dihedral angle of 35.63° with the attached benzene ring (C8—C13). The pyrrolidine ring (N3/C18—C21) adopts an envelope conformation with C19 as the flap and puckering parameters Q = 0.262 (3) Å and φ = 64.0 (5)°.

In the crystal (Fig. 2), O—H···N and C—H···O interactions (Table 1) link the molecules into a three-dimensional network.

S2. Experimental

Ethyl 3-amino-4-(3(2-oxopyrrolidin-1yl)propylamino)benzoate (0.84 mmol) and sodium metabisulfite adduct of 4-carboxy benzaldehyde (1.68 mmol) were dissolved in DMF. The reaction mixture was reflux at 130 °C for 2 hrs. After completion, the reaction mixture was diluted in Ethyl acetate (20 ml) and washed with water (20 ml). The organic layer was collected, dried over Na_2SO_4 and the evaporated *in vacuo* to yield the product. The product was recrystallized from ethyl acetate as brown plates.

S3. Refinement

O bound H atoms were located from a difference Fourier maps and freely refined. The remaining H atoms were positioned geometrically and refined using a riding model with C—H = 0.95–0.99 Å and U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(*C*-methyl). The ethoxy group disordered over two postion with the final refine occupancies 0.853 (14):0.147 (14). A rigid bond and similar restraint were applied for the disordered component. The same anisotropic displacement are used for the C23X and C24X.

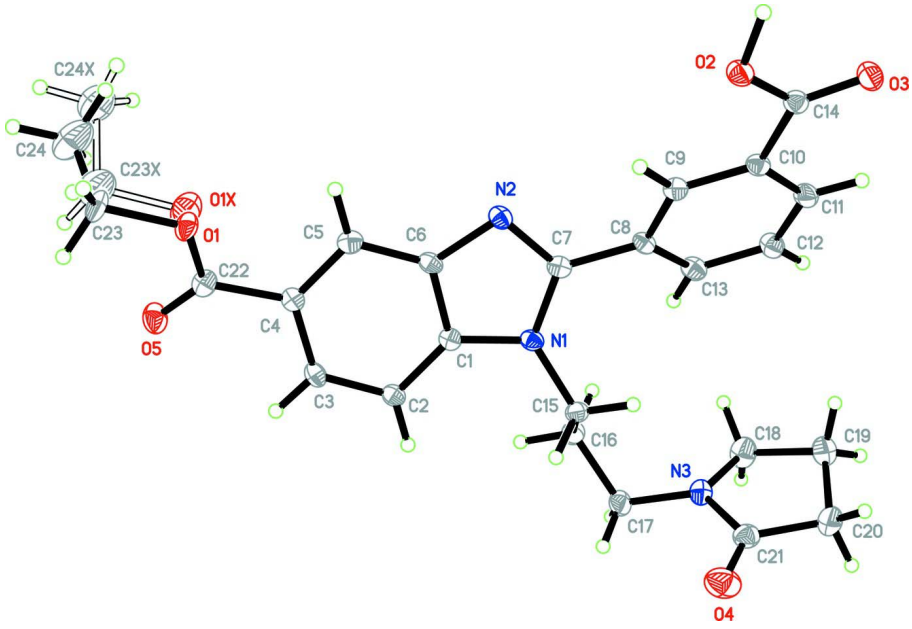

Figure 1

The structure of the title compound, showing 50% probability displacement ellipsoids.

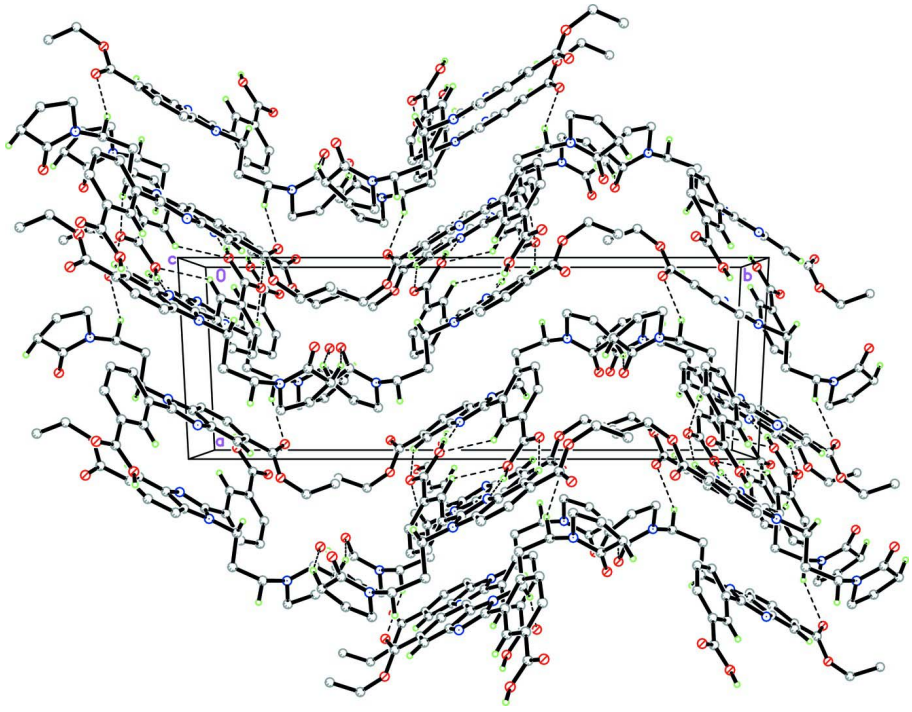

Figure 2

The crystal packing of (I). Dashed lines indicate hydrogen bonds. H atoms not involved in the hydrogen bond interactions have been omitted for clarity.

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c = 9.6761 (3) Å
β = 112.322 (1)°
V = 2068.71 (11) Å^3
Z = 4
$F(000)$ = 920
D_x = 1.398 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3829 reflections
θ = 2.4–29.4°
μ = 0.10 mm^{-1}
T = 100 K
Plate, brown
0.39 × 0.23 × 0.08 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.963, T_{max} = 0.992
23636 measured reflections
6010 independent reflections
3706 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 30.1°, θ_{min} = 2.4°
h = −13→13
k = −34→34
l = −8→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.134
S = 1.03
6010 reflections
317 parameters
69 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.6946P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
O1	−0.1036 (4)	−0.15612 (14)	0.1235 (6)	0.0179 (7)	0.853 (14)
C23	−0.1927 (4)	−0.20029 (16)	0.1526 (4)	0.0216 (8)	0.853 (14)

H23A	−0.3036	−0.1964	0.0868	0.026*	0.853 (14)
H23B	−0.1847	−0.1983	0.2575	0.026*	0.853 (14)
C24	−0.1357 (4)	−0.25307 (15)	0.1261 (8)	0.0427 (14)	0.853 (14)
H24A	−0.1986	−0.2817	0.1448	0.064*	0.853 (14)
H24B	−0.0270	−0.2575	0.1935	0.064*	0.853 (14)
H24C	−0.1437	−0.2551	0.0223	0.064*	0.853 (14)
O1X	−0.066 (4)	−0.1693 (10)	0.123 (4)	0.043 (6)	0.147 (14)
C23X	−0.148 (5)	−0.2161 (11)	0.153 (3)	0.054 (5)	0.147 (14)
H23C	−0.1018	−0.2268	0.2591	0.064*	0.147 (14)
H23D	−0.2598	−0.2080	0.1259	0.064*	0.147 (14)
C24X	−0.127 (4)	−0.2575 (9)	0.057 (3)	0.054 (5)	0.147 (14)
H24D	−0.1522	−0.2927	0.0879	0.080*	0.147 (14)
H24E	−0.0185	−0.2575	0.0657	0.080*	0.147 (14)
H24F	−0.1952	−0.2504	−0.0466	0.080*	0.147 (14)
O2	−0.02946 (16)	0.06006 (5)	−0.61398 (17)	0.0181 (3)	
O3	0.12028 (16)	0.11302 (5)	−0.69174 (16)	0.0201 (3)	
O4	0.43579 (19)	0.23361 (6)	0.23971 (19)	0.0333 (4)	
O5	0.06150 (17)	−0.15773 (6)	0.36566 (17)	0.0231 (3)	
N1	0.30457 (18)	0.03432 (6)	0.07554 (19)	0.0142 (3)	
N2	0.17804 (18)	−0.02457 (6)	−0.10670 (19)	0.0147 (4)	
N3	0.61523 (19)	0.17298 (6)	0.2256 (2)	0.0184 (4)	
C1	0.2460 (2)	−0.00527 (7)	0.1413 (2)	0.0135 (4)	
C2	0.2562 (2)	−0.01238 (8)	0.2868 (2)	0.0145 (4)	
H2A	0.3106	0.0124	0.3638	0.017*	
C3	0.1832 (2)	−0.05727 (7)	0.3144 (2)	0.0151 (4)	
H3A	0.1897	−0.0640	0.4133	0.018*	
C4	0.0994 (2)	−0.09329 (7)	0.1994 (2)	0.0147 (4)	
C5	0.0890 (2)	−0.08568 (8)	0.0537 (2)	0.0158 (4)	
H5A	0.0315	−0.1097	−0.0240	0.019*	
C6	0.1663 (2)	−0.04131 (7)	0.0261 (2)	0.0139 (4)	
C7	0.2601 (2)	0.02085 (7)	−0.0722 (2)	0.0139 (4)	
C8	0.2977 (2)	0.05271 (7)	−0.1826 (2)	0.0145 (4)	
C9	0.1876 (2)	0.05557 (7)	−0.3289 (2)	0.0148 (4)	
H9A	0.0924	0.0365	−0.3548	0.018*	
C10	0.2155 (2)	0.08584 (7)	−0.4364 (2)	0.0148 (4)	
C11	0.3555 (2)	0.11325 (8)	−0.3997 (2)	0.0178 (4)	
H11A	0.3739	0.1347	−0.4725	0.021*	
C12	0.4683 (2)	0.10921 (8)	−0.2565 (2)	0.0185 (4)	
H12A	0.5650	0.1271	−0.2325	0.022*	
C13	0.4402 (2)	0.07915 (8)	−0.1481 (2)	0.0177 (4)	
H13A	0.5179	0.0765	−0.0504	0.021*	
C14	0.0980 (2)	0.08844 (7)	−0.5930 (2)	0.0153 (4)	
C15	0.3717 (2)	0.08531 (7)	0.1508 (2)	0.0155 (4)	
H15A	0.3073	0.0989	0.2044	0.019*	
H15B	0.3679	0.1123	0.0742	0.019*	
C16	0.5390 (2)	0.07964 (8)	0.2613 (2)	0.0176 (4)	
H16A	0.6042	0.0656	0.2090	0.021*	
H16B	0.5435	0.0536	0.3404	0.021*	

C17	0.6019 (3)	0.13412 (8)	0.3320 (2)	0.0212 (5)
H17A	0.5322	0.1489	0.3786	0.025*
H17B	0.7054	0.1287	0.4121	0.025*
C18	0.7451 (3)	0.17112 (9)	0.1760 (3)	0.0262 (5)
H18A	0.7494	0.1360	0.1293	0.031*
H18B	0.8451	0.1774	0.2604	0.031*
C19	0.7087 (3)	0.21703 (9)	0.0613 (3)	0.0283 (5)
H19A	0.8051	0.2348	0.0645	0.034*
H19B	0.6505	0.2036	−0.0411	0.034*
C20	0.6095 (3)	0.25556 (8)	0.1106 (3)	0.0280 (5)
H20A	0.6740	0.2846	0.1743	0.034*
H20B	0.5269	0.2720	0.0230	0.034*
C21	0.5400 (2)	0.22093 (8)	0.1978 (2)	0.0206 (5)
C22	0.0213 (2)	−0.13958 (8)	0.2405 (2)	0.0181 (4)
H1O2	−0.085 (4)	0.0523 (13)	−0.727 (4)	0.088 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0142 (12)	0.0183 (13)	0.0183 (12)	−0.0053 (9)	0.0027 (10)	0.0015 (11)
C23	0.0192 (16)	0.0166 (16)	0.0258 (16)	−0.0079 (11)	0.0050 (13)	0.0021 (12)
C24	0.0372 (19)	0.0253 (16)	0.064 (4)	−0.0002 (13)	0.018 (2)	−0.0081 (19)
O1X	0.062 (15)	0.034 (9)	0.021 (8)	−0.021 (9)	0.004 (11)	0.003 (9)
C23X	0.090 (13)	0.025 (8)	0.054 (10)	−0.008 (8)	0.037 (10)	−0.004 (8)
C24X	0.090 (13)	0.025 (8)	0.054 (10)	−0.008 (8)	0.037 (10)	−0.004 (8)
O2	0.0158 (7)	0.0229 (7)	0.0141 (8)	−0.0050 (6)	0.0041 (6)	−0.0017 (6)
O3	0.0208 (7)	0.0225 (7)	0.0168 (8)	−0.0016 (6)	0.0069 (6)	0.0022 (6)
O4	0.0335 (9)	0.0364 (9)	0.0320 (11)	0.0089 (7)	0.0148 (8)	−0.0014 (8)
O5	0.0236 (8)	0.0253 (8)	0.0175 (8)	−0.0030 (6)	0.0047 (7)	0.0059 (6)
N1	0.0123 (8)	0.0176 (8)	0.0125 (9)	−0.0010 (6)	0.0045 (7)	−0.0019 (7)
N2	0.0119 (8)	0.0194 (8)	0.0115 (9)	−0.0008 (6)	0.0029 (7)	0.0002 (7)
N3	0.0173 (9)	0.0187 (8)	0.0185 (10)	−0.0009 (7)	0.0060 (8)	0.0018 (7)
C1	0.0100 (9)	0.0159 (9)	0.0143 (10)	0.0005 (7)	0.0042 (8)	−0.0003 (8)
C2	0.0134 (9)	0.0174 (9)	0.0113 (10)	0.0008 (7)	0.0031 (8)	0.0002 (7)
C3	0.0132 (9)	0.0192 (10)	0.0132 (10)	0.0017 (8)	0.0053 (8)	0.0028 (8)
C4	0.0124 (9)	0.0169 (9)	0.0144 (10)	0.0010 (7)	0.0046 (8)	0.0011 (8)
C5	0.0120 (9)	0.0178 (9)	0.0159 (11)	0.0001 (7)	0.0033 (8)	−0.0019 (8)
C6	0.0122 (9)	0.0183 (9)	0.0097 (10)	0.0015 (7)	0.0024 (8)	0.0003 (8)
C7	0.0105 (9)	0.0166 (9)	0.0128 (10)	0.0021 (7)	0.0026 (8)	−0.0002 (8)
C8	0.0146 (10)	0.0172 (9)	0.0130 (10)	0.0014 (7)	0.0067 (8)	−0.0001 (8)
C9	0.0125 (9)	0.0162 (9)	0.0156 (11)	−0.0004 (7)	0.0052 (8)	−0.0014 (8)
C10	0.0154 (9)	0.0154 (9)	0.0139 (10)	0.0018 (7)	0.0058 (8)	−0.0010 (8)
C11	0.0178 (10)	0.0206 (10)	0.0177 (11)	−0.0034 (8)	0.0096 (9)	−0.0018 (8)
C12	0.0151 (10)	0.0234 (10)	0.0180 (11)	−0.0038 (8)	0.0076 (9)	−0.0011 (9)
C13	0.0145 (10)	0.0223 (10)	0.0145 (11)	−0.0004 (8)	0.0036 (9)	−0.0009 (8)
C14	0.0160 (10)	0.0154 (9)	0.0152 (11)	0.0010 (8)	0.0066 (9)	−0.0007 (8)
C15	0.0168 (10)	0.0145 (9)	0.0158 (11)	−0.0007 (8)	0.0069 (9)	−0.0024 (8)
C16	0.0175 (10)	0.0176 (10)	0.0149 (11)	−0.0006 (8)	0.0032 (9)	0.0007 (8)

C17	0.0256 (11)	0.0217 (10)	0.0130 (11)	−0.0055 (9)	0.0037 (9)	0.0013 (8)
C18	0.0201 (11)	0.0253 (11)	0.0361 (15)	0.0011 (9)	0.0141 (11)	0.0038 (10)
C19	0.0347 (13)	0.0267 (11)	0.0276 (13)	−0.0046 (10)	0.0165 (11)	0.0015 (10)
C20	0.0372 (14)	0.0202 (11)	0.0266 (13)	−0.0011 (10)	0.0120 (11)	0.0029 (9)
C21	0.0219 (11)	0.0209 (10)	0.0169 (11)	0.0007 (8)	0.0048 (9)	−0.0016 (8)
C22	0.0143 (10)	0.0172 (9)	0.0216 (12)	0.0001 (8)	0.0056 (9)	0.0005 (9)

Geometric parameters (Å, º)

O1—C22	1.343 (5)	C3—H3A	0.9500
O1—C23	1.465 (3)	C4—C5	1.388 (3)
C23—C24	1.472 (4)	C4—C22	1.492 (3)
C23—H23A	0.9900	C5—C6	1.396 (3)
C23—H23B	0.9900	C5—H5A	0.9500
C24—H24A	0.9800	C7—C8	1.475 (3)
C24—H24B	0.9800	C8—C9	1.399 (3)
C24—H24C	0.9800	C8—C13	1.402 (3)
O1X—C22	1.34 (3)	C9—C10	1.386 (3)
O1X—C23X	1.478 (17)	C9—H9A	0.9500
C23X—C24X	1.446 (17)	C10—C11	1.392 (3)
C23X—H23C	0.9900	C10—C14	1.495 (3)
C23X—H23D	0.9900	C11—C12	1.388 (3)
C24X—H24D	0.9800	C11—H11A	0.9500
C24X—H24E	0.9800	C12—C13	1.390 (3)
C24X—H24F	0.9800	C12—H12A	0.9500
O2—C14	1.328 (2)	C13—H13A	0.9500
O2—H1O2	1.03 (4)	C15—C16	1.525 (3)
O3—C14	1.216 (2)	C15—H15A	0.9900
O4—C21	1.226 (2)	C15—H15B	0.9900
O5—C22	1.211 (2)	C16—C17	1.528 (3)
N1—C7	1.370 (2)	C16—H16A	0.9900
N1—C1	1.390 (2)	C16—H16B	0.9900
N1—C15	1.475 (2)	C17—H17A	0.9900
N2—C7	1.331 (2)	C17—H17B	0.9900
N2—C6	1.394 (2)	C18—C19	1.536 (3)
N3—C21	1.355 (3)	C18—H18A	0.9900
N3—C17	1.450 (3)	C18—H18B	0.9900
N3—C18	1.462 (3)	C19—C20	1.527 (3)
C1—C2	1.386 (3)	C19—H19A	0.9900
C1—C6	1.401 (3)	C19—H19B	0.9900
C2—C3	1.383 (3)	C20—C21	1.511 (3)
C2—H2A	0.9500	C20—H20A	0.9900
C3—C4	1.410 (3)	C20—H20B	0.9900
C22—O1—C23	116.1 (4)	C12—C11—C10	119.99 (19)
O1—C23—C24	111.4 (3)	C12—C11—H11A	120.0
O1—C23—H23A	109.3	C10—C11—H11A	120.0
C24—C23—H23A	109.3	C11—C12—C13	120.27 (19)

O1—C23—H23B	109.3	C11—C12—H12A	119.9
C24—C23—H23B	109.3	C13—C12—H12A	119.9
H23A—C23—H23B	108.0	C12—C13—C8	120.24 (19)
C22—O1X—C23X	117 (2)	C12—C13—H13A	119.9
C24X—C23X—O1X	103.0 (18)	C8—C13—H13A	119.9
C24X—C23X—H23C	111.2	O3—C14—O2	123.72 (19)
O1X—C23X—H23C	111.2	O3—C14—C10	122.72 (18)
C24X—C23X—H23D	111.2	O2—C14—C10	113.53 (17)
O1X—C23X—H23D	111.2	N1—C15—C16	113.28 (15)
H23C—C23X—H23D	109.1	N1—C15—H15A	108.9
C23X—C24X—H24D	109.5	C16—C15—H15A	108.9
C23X—C24X—H24E	109.5	N1—C15—H15B	108.9
H24D—C24X—H24E	109.5	C16—C15—H15B	108.9
C23X—C24X—H24F	109.5	H15A—C15—H15B	107.7
H24D—C24X—H24F	109.5	C15—C16—C17	110.36 (16)
H24E—C24X—H24F	109.5	C15—C16—H16A	109.6
C14—O2—H1O2	108.8 (19)	C17—C16—H16A	109.6
C7—N1—C1	106.81 (15)	C15—C16—H16B	109.6
C7—N1—C15	128.88 (16)	C17—C16—H16B	109.6
C1—N1—C15	123.18 (16)	H16A—C16—H16B	108.1
C7—N2—C6	105.04 (16)	N3—C17—C16	113.13 (17)
C21—N3—C17	123.25 (18)	N3—C17—H17A	109.0
C21—N3—C18	113.08 (17)	C16—C17—H17A	109.0
C17—N3—C18	121.13 (17)	N3—C17—H17B	109.0
C2—C1—N1	131.88 (18)	C16—C17—H17B	109.0
C2—C1—C6	122.52 (18)	H17A—C17—H17B	107.8
N1—C1—C6	105.60 (17)	N3—C18—C19	103.34 (17)
C3—C2—C1	116.74 (18)	N3—C18—H18A	111.1
C3—C2—H2A	121.6	C19—C18—H18A	111.1
C1—C2—H2A	121.6	N3—C18—H18B	111.1
C2—C3—C4	121.57 (19)	C19—C18—H18B	111.1
C2—C3—H3A	119.2	H18A—C18—H18B	109.1
C4—C3—H3A	119.2	C20—C19—C18	103.47 (18)
C5—C4—C3	121.29 (18)	C20—C19—H19A	111.1
C5—C4—C22	121.47 (18)	C18—C19—H19A	111.1
C3—C4—C22	117.23 (18)	C20—C19—H19B	111.1
C4—C5—C6	117.34 (18)	C18—C19—H19B	111.1
C4—C5—H5A	121.3	H19A—C19—H19B	109.0
C6—C5—H5A	121.3	C21—C20—C19	104.83 (17)
N2—C6—C5	129.66 (18)	C21—C20—H20A	110.8
N2—C6—C1	109.85 (17)	C19—C20—H20A	110.8
C5—C6—C1	120.48 (19)	C21—C20—H20B	110.8
N2—C7—N1	112.70 (17)	C19—C20—H20B	110.8
N2—C7—C8	122.94 (18)	H20A—C20—H20B	108.9
N1—C7—C8	124.36 (17)	O4—C21—N3	125.0 (2)
C9—C8—C13	118.74 (18)	O4—C21—C20	126.77 (19)
C9—C8—C7	118.40 (17)	N3—C21—C20	108.20 (18)
C13—C8—C7	122.84 (19)	O5—C22—O1X	120.1 (11)

C10—C9—C8	120.84 (18)	O5—C22—O1	124.7 (2)
C10—C9—H9A	119.6	O1X—C22—O1	20.7 (16)
C8—C9—H9A	119.6	O5—C22—C4	123.81 (19)
C9—C10—C11	119.84 (19)	O1X—C22—C4	113.4 (11)
C9—C10—C14	120.71 (17)	O1—C22—C4	111.4 (2)
C11—C10—C14	119.43 (18)		
C22—O1—C23—C24	−90.3 (5)	C10—C11—C12—C13	−1.9 (3)
C22—O1X—C23X—C24X	−138 (3)	C11—C12—C13—C8	0.0 (3)
C7—N1—C1—C2	179.4 (2)	C9—C8—C13—C12	2.4 (3)
C15—N1—C1—C2	−11.8 (3)	C7—C8—C13—C12	−179.14 (18)
C7—N1—C1—C6	−0.3 (2)	C9—C10—C14—O3	−177.43 (18)
C15—N1—C1—C6	168.44 (16)	C11—C10—C14—O3	1.2 (3)
N1—C1—C2—C3	−179.75 (19)	C9—C10—C14—O2	0.5 (3)
C6—C1—C2—C3	0.0 (3)	C11—C10—C14—O2	179.16 (17)
C1—C2—C3—C4	−1.5 (3)	C7—N1—C15—C16	−113.5 (2)
C2—C3—C4—C5	1.2 (3)	C1—N1—C15—C16	80.4 (2)
C2—C3—C4—C22	−178.28 (18)	N1—C15—C16—C17	178.68 (17)
C3—C4—C5—C6	0.8 (3)	C21—N3—C17—C16	120.6 (2)
C22—C4—C5—C6	−179.84 (17)	C18—N3—C17—C16	−78.7 (2)
C7—N2—C6—C5	178.0 (2)	C15—C16—C17—N3	−65.6 (2)
C7—N2—C6—C1	−1.1 (2)	C21—N3—C18—C19	−20.0 (2)
C4—C5—C6—N2	178.75 (18)	C17—N3—C18—C19	177.57 (18)
C4—C5—C6—C1	−2.2 (3)	N3—C18—C19—C20	26.0 (2)
C2—C1—C6—N2	−178.87 (17)	C18—C19—C20—C21	−23.6 (2)
N1—C1—C6—N2	0.9 (2)	C17—N3—C21—O4	−11.8 (3)
C2—C1—C6—C5	2.0 (3)	C18—N3—C21—O4	−173.9 (2)
N1—C1—C6—C5	−178.27 (17)	C17—N3—C21—C20	166.91 (18)
C6—N2—C7—N1	0.9 (2)	C18—N3—C21—C20	4.9 (2)
C6—N2—C7—C8	−179.02 (17)	C19—C20—C21—O4	−168.8 (2)
C1—N1—C7—N2	−0.4 (2)	C19—C20—C21—N3	12.5 (2)
C15—N1—C7—N2	−168.29 (17)	C23X—O1X—C22—O5	18 (3)
C1—N1—C7—C8	179.56 (17)	C23X—O1X—C22—O1	−91 (4)
C15—N1—C7—C8	11.6 (3)	C23X—O1X—C22—C4	−179.9 (15)
N2—C7—C8—C9	35.6 (3)	C23—O1—C22—O5	−1.6 (4)
N1—C7—C8—C9	−144.28 (19)	C23—O1—C22—O1X	82 (4)
N2—C7—C8—C13	−142.87 (19)	C23—O1—C22—C4	−177.9 (2)
N1—C7—C8—C13	37.2 (3)	C5—C4—C22—O5	156.8 (2)
C13—C8—C9—C10	−2.8 (3)	C3—C4—C22—O5	−23.7 (3)
C7—C8—C9—C10	178.65 (17)	C5—C4—C22—O1X	−4.5 (18)
C8—C9—C10—C11	0.9 (3)	C3—C4—C22—O1X	174.9 (18)
C8—C9—C10—C14	179.48 (17)	C5—C4—C22—O1	−26.8 (3)
C9—C10—C11—C12	1.5 (3)	C3—C4—C22—O1	152.6 (2)
C14—C10—C11—C12	−177.12 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1*O*2···N2^{i}	1.04 (4)	1.66 (4)	2.675 (2)	166 (3)
C5—H5*A*···O3^{i}	0.95	2.57	3.367 (2)	141
C9—H9*A*···O2^{i}	0.95	2.46	3.177 (2)	132
C15—H15*A*···O3^{ii}	0.99	2.34	3.314 (3)	167
C17—H17*B*···O5^{iii}	0.99	2.51	3.429 (3)	154
C20—H20*B*···O4^{iv}	0.99	2.55	3.340 (3)	137

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