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Ethyl 2-[(2*E*)-4-decyl-3-oxo-1,2,3,4-tetrahydroquinoxalin-2-ylidene]acetate

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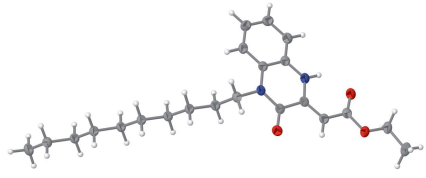
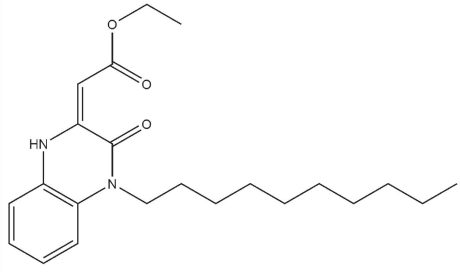
In the title compound, $C_{22}H_{32}N_2O_3$, the tetrahydroquinoxaline unit is planar. The ester substituent is nearly coplanar with this ring system as a result of an intramolecular N—H···O hydrogen bond. In the crystal, C—H···O hydrogen bonds and π-stacking interactions form oblique stacks which are connected into pairs by additional C—H···O hydrogen bonds. These pairs are further linked into thick sheets, with the *n*-decyl chains extending out from both surfaces as a result of a third set of C—H···O hydrogen bonds. Intercalation of the *n*-decyl chains completes the crystal packing.

3D view



Chemical scheme



Structure description

A number of compounds based on nitrogen-containing heterocycles show antimicrobial activity and have been developed for clinical use (Ohkanda & Katoh, 1998). Among the various classes of heterocyclic units, the quinoxaline ring system has frequently been used as a component of various antibiotic molecules, such as hinomycin, levomycin and actindeutin, which inhibit the growth of Gram-positive bacteria and are active against various transplantable tumors (Dell *et al.*, 1975; Bailly *et al.*, 1999; Sato *et al.*, 1967). In addition, many reports describe a variety of biological properties of quinoxaline derivatives, including anticancer, antibacterial, antifungal, antiviral and antiprotozoal activities (Sanna *et al.*, 1999; Rao *et al.*, 2009; Fonseca *et al.*, 2004; Budakoti *et al.*, 2008). The numerous applications of quinoxaline derivatives prompted researchers to develop efficient methods for the synthesis of new quinoxaline derivatives likely to show interesting pharmaceutical activities (Ramli *et al.*, 2011, 2013, 2018; Caleb *et al.*, 2016; Abad *et al.*, 2018). We report here the synthesis and crystal structure of the title tetrahydroquinoxaline compound (Fig. 1).

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.88 (2)	1.99 (2)	2.6885 (18)	134.8 (17)
C3—H3···O1^{i}	0.996 (19)	2.491 (19)	3.278 (2)	135.6 (14)
C11—H11A···O2^{ii}	0.96 (2)	2.59 (2)	3.424 (2)	144.7 (16)
C13—H13B···O1^{iii}	0.983 (18)	2.541 (18)	3.2714 (19)	131.0 (13)

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

The 10-membered ring is planar to within 0.0507 (11) Å (r.m.s. deviation of the fitted atoms is 0.0227 Å), with atom C8 furthest from the mean plane [0.0507 (11) Å] and atom O1 0.162 (2) Å from this plane. The ester substituent is nearly coplanar with the bicyclic core, as indicated by the N1—C7—C9—C10 torsion angle of −1.0 (2)°. This is due to the intramolecular N1—H1···O2 hydrogen bond. In the crystal, molecules form oblique stacks extending along the *b*-axis direction through a combination of C13—H13B···O1^{iii} hydrogen bonds and π-stacking interations between the C1–C6 and C1/C6/N1/C7/C8/N2 rings [centroid–centroid distance = 3.7896 (9) Å; dihedral angle = 1.9 (7)°]. The stacks are connected by C3—H3···O1^{i} hydrogen bonds (Fig. 2). Inversion-related C11—H11A···O2^{ii} hydrogen bonds (Table 1 and Fig. 3) form dimers with $R_2^2(10)$ ring motifs. These combine with the previously mentioned C3—H3···O1^{i} contacts to generate sheets of molecules in the *ac* plane, with the decyl chains intercalated in opposite directions between adjacent dimers (Fig. 3).

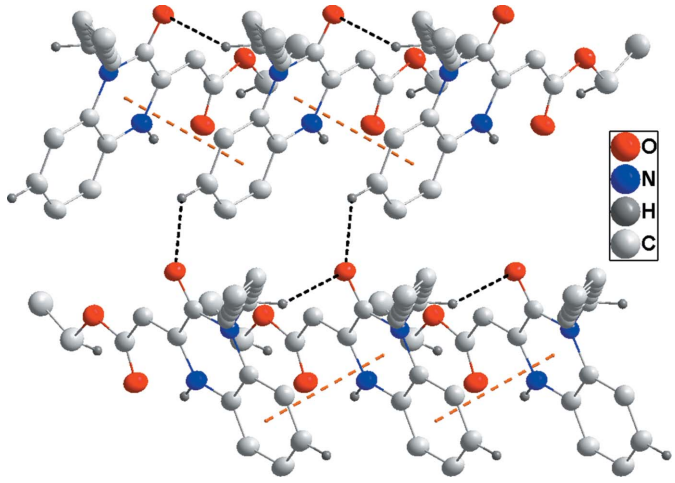


Figure 2
Side view of two stacks projected onto (50$\bar{1}$), with the *b*-axis direction running from left to right. C—H···O hydrogen bonds are shown as black dashed lines, while orange dashed lines show the π-stacking interactions.

Synthesis and crystallization

To a solution of ethyl 2-(3-oxo-3,4-dihydroquinoxalin-2-yl)-acetate (0.5 g, 2.15 mmol) in *N*,*N*-dimethylformamide (20 ml) were added 1-bromodecane (0.45 ml, 2.15 mmol), potassium carbonate (K_2CO_3; 0.3 g, 2.15 mmol) and a catalytic quantity of tetra-*n*-butylammonium bromide (TBAB). The mixture was stirred at room temperature for 48 h. The solution was filtered and the solvent removed under reduced pressure. The residue obtained, after evaporation of solvent, was chromatographed on a silica-gel column using hexane/ethyl acetate (9:1) as eluent. The solid obtained was crystallized from ethanol to afford the title compound as yellow crystals.

Refinement

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

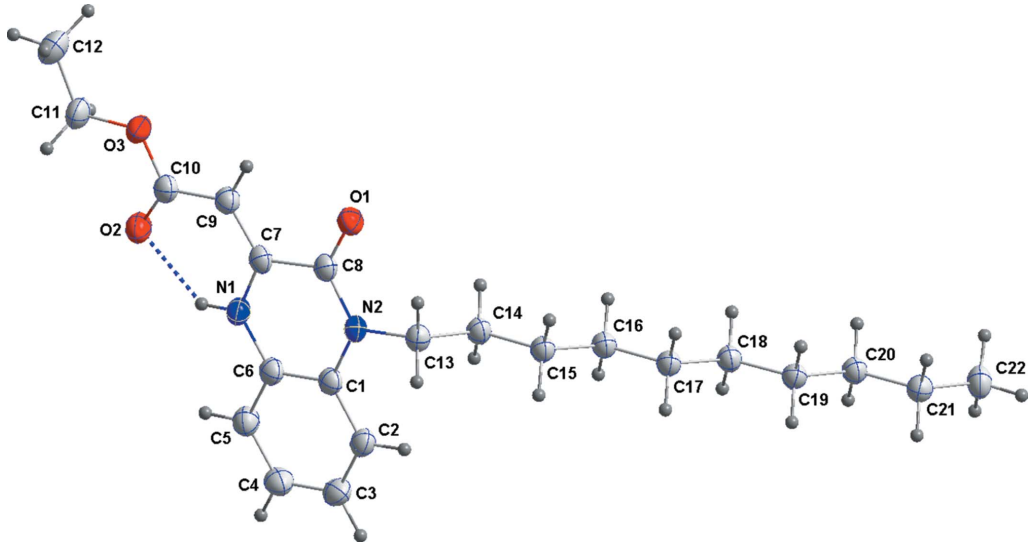


Figure 1
The title molecule, showing the labeling scheme and 50% probability displacement ellipsoids. The intramolecular N—H···O hydrogen bond is shown as a dashed line.

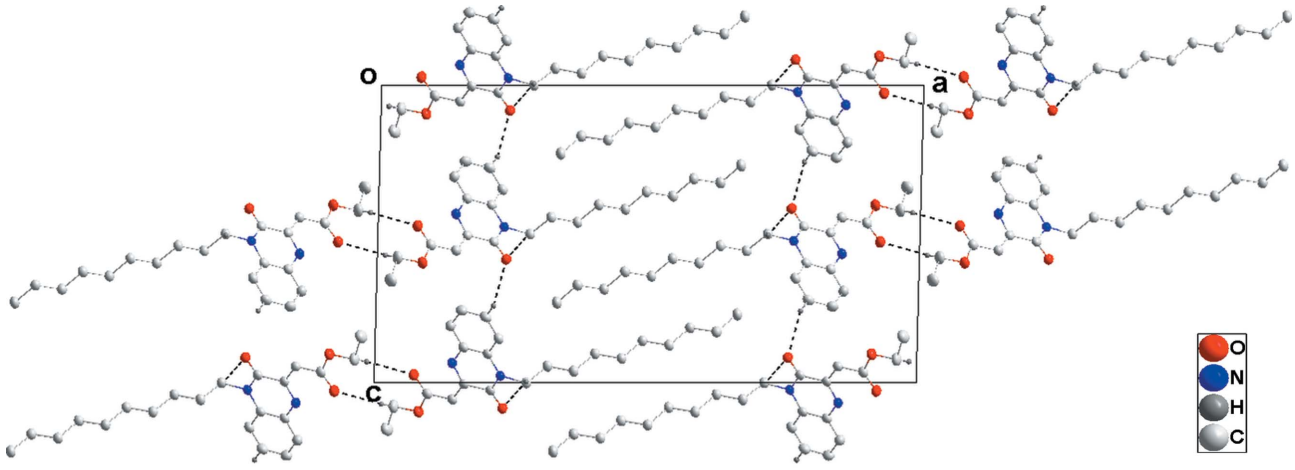


Figure 3
The packing, viewed along the *b*-axis direction, with C—H···O hydrogen bonds shown as dashed lines.

Acknowledgements

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Funding information

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{32}N_2O_3$
M_r	372.49
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, b, c (Å)	28.0610 (8), 4.7650 (1), 15.3667 (4)
β (°)	91.503 (1)
V (Å^3)	2053.98 (9)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.63
Crystal size (mm)	0.21 × 0.07 × 0.03
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.86, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16668, 4009, 3075
R_{int}	0.049
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.102, 1.04
No. of reflections	4009
No. of parameters	372
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.17

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Bruker, 2016).

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full crystallographic data

IUCrData (2018). **3**, x180680 [https://doi.org/10.1107/S2414314618006806]

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Crystal data

$C_{22}H_{32}N_2O_3$
M_r = 372.49
Monoclinic, $P2_1/c$
a = 28.0610 (8) Å
b = 4.7650 (1) Å
c = 15.3667 (4) Å
β = 91.503 (1)°
V = 2053.98 (9) Å^3
Z = 4
$F(000)$ = 808
D_x = 1.205 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9673 reflections
θ = 3.2–72.2°
μ = 0.63 mm^{-1}
T = 150 K
Plate, yellow
0.21 × 0.07 × 0.03 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*; Krause *et al.*, 2015)
T_{min} = 0.86, T_{max} = 0.98
16668 measured reflections
4009 independent reflections
3075 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 72.2°, θ_{min} = 4.7°
h = −34→34
k = −5→5
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.102
S = 1.04
4009 reflections
372 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2 + 0.6332P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.23816 (4)	0.7156 (2)	0.58536 (7)	0.0334 (3)
O2	0.07295 (4)	0.9468 (3)	0.47288 (7)	0.0392 (3)
O3	0.08353 (4)	1.1955 (2)	0.59667 (7)	0.0354 (3)
N1	0.14315 (5)	0.5888 (3)	0.43313 (8)	0.0320 (3)
H1	0.1148 (7)	0.660 (4)	0.4198 (12)	0.044 (5)*
N2	0.23287 (4)	0.3938 (3)	0.47640 (8)	0.0278 (3)
C1	0.20821 (5)	0.2817 (3)	0.40277 (9)	0.0284 (3)
C2	0.22774 (6)	0.0724 (3)	0.35065 (10)	0.0326 (3)
H2	0.2596 (7)	0.002 (4)	0.3653 (11)	0.037 (5)*
C3	0.20276 (6)	−0.0267 (4)	0.27830 (11)	0.0369 (4)
H3	0.2168 (7)	−0.175 (4)	0.2412 (12)	0.044 (5)*
C4	0.15807 (6)	0.0801 (4)	0.25643 (11)	0.0377 (4)
H4	0.1405 (7)	0.016 (4)	0.2052 (13)	0.044 (5)*
C5	0.13824 (6)	0.2857 (4)	0.30769 (11)	0.0358 (4)
H5	0.1070 (6)	0.364 (3)	0.2933 (11)	0.031 (4)*
C6	0.16306 (6)	0.3855 (3)	0.38092 (10)	0.0301 (3)
C7	0.16541 (5)	0.7038 (3)	0.50423 (9)	0.0283 (3)
C8	0.21484 (5)	0.6064 (3)	0.52594 (9)	0.0279 (3)
C9	0.14518 (6)	0.9036 (3)	0.55540 (10)	0.0303 (3)
H9	0.1634 (6)	0.973 (4)	0.6043 (11)	0.031 (4)*
C10	0.09832 (6)	1.0108 (3)	0.53625 (10)	0.0316 (3)
C11	0.03737 (6)	1.3213 (4)	0.57921 (12)	0.0412 (4)
H11A	0.0146 (8)	1.172 (4)	0.5712 (13)	0.050 (6)*
H11B	0.0399 (7)	1.439 (4)	0.5231 (14)	0.051 (6)*
C12	0.02591 (8)	1.5045 (5)	0.65563 (14)	0.0478 (5)
H12A	−0.0044 (9)	1.597 (5)	0.6452 (14)	0.062 (6)*
H12B	0.0512 (8)	1.658 (5)	0.6648 (13)	0.057 (6)*
H12C	0.0249 (8)	1.389 (5)	0.7103 (15)	0.065 (7)*
C13	0.28087 (5)	0.2893 (3)	0.50025 (10)	0.0300 (3)
H13A	0.2863 (6)	0.342 (3)	0.5639 (11)	0.029 (4)*
H13B	0.2804 (6)	0.084 (4)	0.4959 (11)	0.033 (4)*
C14	0.31933 (6)	0.4135 (3)	0.44375 (11)	0.0299 (3)
H14A	0.3206 (6)	0.615 (4)	0.4553 (11)	0.031 (4)*
H14B	0.3101 (6)	0.399 (4)	0.3814 (12)	0.035 (4)*
C15	0.36764 (5)	0.2769 (3)	0.46053 (10)	0.0292 (3)
H15A	0.3783 (7)	0.302 (4)	0.5226 (13)	0.044 (5)*
H15B	0.3649 (6)	0.071 (4)	0.4518 (11)	0.035 (5)*
C16	0.40639 (6)	0.3910 (3)	0.40243 (11)	0.0298 (3)
H16A	0.4091 (6)	0.598 (4)	0.4114 (11)	0.036 (5)*
H16B	0.3965 (6)	0.366 (4)	0.3403 (12)	0.035 (5)*

C17	0.45527 (5)	0.2572 (3)	0.41760 (10)	0.0293 (3)
H17A	0.4655 (6)	0.286 (3)	0.4785 (12)	0.031 (4)*
H17B	0.4527 (6)	0.051 (4)	0.4078 (11)	0.040 (5)*
C18	0.49321 (5)	0.3742 (3)	0.35852 (10)	0.0297 (3)
H18A	0.4951 (6)	0.584 (4)	0.3682 (10)	0.031 (4)*
H18B	0.4824 (6)	0.349 (4)	0.2957 (12)	0.038 (5)*
C19	0.54237 (5)	0.2458 (3)	0.37324 (10)	0.0295 (3)
H19A	0.5527 (6)	0.273 (3)	0.4355 (11)	0.030 (4)*
H19B	0.5404 (6)	0.041 (4)	0.3637 (11)	0.040 (5)*
C20	0.58003 (6)	0.3680 (3)	0.31472 (10)	0.0299 (3)
H20A	0.5820 (6)	0.577 (4)	0.3249 (11)	0.036 (5)*
H20B	0.5696 (7)	0.343 (4)	0.2525 (13)	0.042 (5)*
C21	0.62922 (6)	0.2390 (3)	0.32837 (11)	0.0328 (3)
H21A	0.6397 (6)	0.260 (4)	0.3904 (12)	0.039 (5)*
H21B	0.6269 (7)	0.034 (4)	0.3165 (12)	0.045 (5)*
C22	0.66655 (6)	0.3645 (4)	0.26983 (12)	0.0399 (4)
H22A	0.6573 (7)	0.346 (4)	0.2060 (14)	0.049 (5)*
H22B	0.6706 (7)	0.567 (5)	0.2824 (13)	0.053 (6)*
H22C	0.6984 (8)	0.272 (4)	0.2793 (13)	0.052 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0292 (6)	0.0390 (6)	0.0319 (5)	−0.0010 (5)	−0.0008 (5)	−0.0027 (5)
O2	0.0287 (6)	0.0504 (7)	0.0384 (6)	0.0026 (5)	−0.0003 (5)	−0.0066 (5)
O3	0.0289 (6)	0.0391 (6)	0.0382 (6)	0.0057 (5)	0.0032 (5)	−0.0059 (5)
N1	0.0250 (7)	0.0387 (7)	0.0322 (7)	0.0021 (6)	0.0015 (6)	−0.0035 (6)
N2	0.0235 (6)	0.0314 (6)	0.0285 (6)	−0.0005 (5)	0.0033 (5)	0.0011 (5)
C1	0.0271 (8)	0.0315 (7)	0.0268 (7)	−0.0040 (6)	0.0053 (6)	0.0010 (6)
C2	0.0289 (8)	0.0351 (8)	0.0342 (8)	0.0007 (7)	0.0068 (7)	0.0002 (7)
C3	0.0388 (9)	0.0393 (9)	0.0330 (8)	−0.0023 (7)	0.0083 (7)	−0.0056 (7)
C4	0.0387 (9)	0.0436 (9)	0.0308 (8)	−0.0041 (7)	0.0019 (7)	−0.0058 (7)
C5	0.0300 (9)	0.0432 (9)	0.0340 (8)	0.0000 (7)	0.0004 (7)	−0.0019 (7)
C6	0.0281 (8)	0.0337 (8)	0.0286 (7)	−0.0033 (6)	0.0054 (6)	−0.0003 (6)
C7	0.0254 (8)	0.0328 (8)	0.0269 (7)	−0.0035 (6)	0.0036 (6)	0.0032 (6)
C8	0.0260 (8)	0.0306 (7)	0.0274 (7)	−0.0028 (6)	0.0048 (6)	0.0029 (6)
C9	0.0277 (8)	0.0341 (8)	0.0293 (8)	−0.0020 (6)	0.0033 (6)	−0.0007 (6)
C10	0.0280 (8)	0.0341 (8)	0.0327 (8)	−0.0027 (6)	0.0052 (6)	−0.0002 (6)
C11	0.0280 (9)	0.0474 (10)	0.0482 (10)	0.0084 (8)	0.0026 (8)	−0.0055 (8)
C12	0.0422 (11)	0.0522 (11)	0.0492 (11)	0.0140 (9)	0.0085 (9)	−0.0040 (9)
C13	0.0256 (8)	0.0319 (8)	0.0325 (8)	0.0010 (6)	0.0023 (6)	0.0021 (6)
C14	0.0268 (8)	0.0285 (8)	0.0346 (8)	−0.0010 (6)	0.0034 (6)	0.0015 (6)
C15	0.0257 (8)	0.0289 (8)	0.0332 (8)	−0.0009 (6)	0.0035 (6)	0.0004 (6)
C16	0.0262 (8)	0.0300 (8)	0.0335 (8)	−0.0011 (6)	0.0028 (6)	−0.0005 (6)
C17	0.0261 (8)	0.0291 (8)	0.0328 (8)	−0.0001 (6)	0.0039 (6)	−0.0004 (6)
C18	0.0261 (8)	0.0301 (8)	0.0330 (8)	−0.0011 (6)	0.0020 (6)	0.0007 (6)
C19	0.0277 (8)	0.0278 (8)	0.0332 (8)	−0.0001 (6)	0.0041 (6)	−0.0006 (6)
C20	0.0272 (8)	0.0297 (8)	0.0329 (8)	−0.0011 (6)	0.0029 (6)	0.0007 (6)

C21	0.0286 (8)	0.0351 (8)	0.0348 (8)	0.0009 (7)	0.0022 (7)	−0.0017 (7)
C22	0.0269 (9)	0.0503 (11)	0.0426 (10)	−0.0017 (8)	0.0041 (7)	−0.0001 (8)

Geometric parameters (Å, º)

O1—C8	1.2253 (18)	C13—C14	1.522 (2)
O2—C10	1.2294 (19)	C13—H13A	1.017 (17)
O3—C10	1.3524 (19)	C13—H13B	0.983 (18)
O3—C11	1.446 (2)	C14—C15	1.520 (2)
N1—C7	1.3595 (19)	C14—H14A	0.977 (17)
N1—C6	1.385 (2)	C14—H14B	0.989 (18)
N1—H1	0.88 (2)	C15—C16	1.525 (2)
N2—C8	1.3720 (19)	C15—H15A	1.00 (2)
N2—C1	1.4153 (19)	C15—H15B	0.991 (18)
N2—C13	1.4731 (19)	C16—C17	1.525 (2)
C1—C6	1.393 (2)	C16—H16A	0.998 (18)
C1—C2	1.400 (2)	C16—H16B	0.994 (18)
C2—C3	1.382 (2)	C17—C18	1.523 (2)
C2—H2	0.976 (19)	C17—H17A	0.982 (18)
C3—C4	1.387 (3)	C17—H17B	0.997 (19)
C3—H3	0.995 (19)	C18—C19	1.521 (2)
C4—C5	1.383 (2)	C18—H18A	1.013 (17)
C4—H4	0.967 (19)	C18—H18B	1.011 (18)
C5—C6	1.392 (2)	C19—C20	1.521 (2)
C5—H5	0.973 (18)	C19—H19A	1.000 (17)
C7—C9	1.368 (2)	C19—H19B	0.988 (19)
C7—C8	1.492 (2)	C20—C21	1.521 (2)
C9—C10	1.434 (2)	C20—H20A	1.010 (18)
C9—H9	0.957 (17)	C20—H20B	1.000 (19)
C11—C12	1.505 (3)	C21—C22	1.521 (2)
C11—H11A	0.96 (2)	C21—H21A	0.996 (19)
C11—H11B	1.03 (2)	C21—H21B	1.00 (2)
C12—H12A	0.97 (2)	C22—H22A	1.01 (2)
C12—H12B	1.03 (2)	C22—H22B	0.99 (2)
C12—H12C	1.01 (2)	C22—H22C	1.00 (2)
C10—O3—C11	115.58 (13)	C15—C14—C13	112.28 (13)
C7—N1—C6	124.32 (14)	C15—C14—H14A	111.2 (10)
C7—N1—H1	115.1 (13)	C13—C14—H14A	107.5 (10)
C6—N1—H1	120.5 (13)	C15—C14—H14B	110.1 (10)
C8—N2—C1	122.85 (13)	C13—C14—H14B	110.7 (10)
C8—N2—C13	117.36 (12)	H14A—C14—H14B	104.7 (14)
C1—N2—C13	119.74 (12)	C14—C15—C16	113.22 (13)
C6—C1—C2	118.83 (14)	C14—C15—H15A	110.7 (11)
C6—C1—N2	118.78 (13)	C16—C15—H15A	108.5 (11)
C2—C1—N2	122.39 (14)	C14—C15—H15B	109.5 (10)
C3—C2—C1	120.40 (16)	C16—C15—H15B	109.1 (10)
C3—C2—H2	120.7 (10)	H15A—C15—H15B	105.5 (15)

C1—C2—H2	118.9 (10)	C17—C16—C15	114.44 (13)
C2—C3—C4	120.39 (16)	C17—C16—H16A	109.1 (10)
C2—C3—H3	120.2 (11)	C15—C16—H16A	108.9 (10)
C4—C3—H3	119.4 (11)	C17—C16—H16B	108.9 (10)
C5—C4—C3	119.79 (16)	C15—C16—H16B	109.6 (10)
C5—C4—H4	118.8 (11)	H16A—C16—H16B	105.5 (14)
C3—C4—H4	121.5 (11)	C18—C17—C16	113.33 (13)
C4—C5—C6	120.15 (16)	C18—C17—H17A	109.0 (10)
C4—C5—H5	121.0 (10)	C16—C17—H17A	109.0 (10)
C6—C5—H5	118.8 (10)	C18—C17—H17B	108.6 (11)
N1—C6—C5	120.44 (14)	C16—C17—H17B	109.2 (11)
N1—C6—C1	119.13 (14)	H17A—C17—H17B	107.5 (14)
C5—C6—C1	120.43 (14)	C19—C18—C17	114.19 (13)
N1—C7—C9	123.62 (14)	C19—C18—H18A	109.3 (10)
N1—C7—C8	117.33 (13)	C17—C18—H18A	108.1 (10)
C9—C7—C8	119.05 (13)	C19—C18—H18B	110.0 (10)
O1—C8—N2	121.97 (14)	C17—C18—H18B	109.2 (10)
O1—C8—C7	120.64 (14)	H18A—C18—H18B	105.8 (14)
N2—C8—C7	117.38 (13)	C18—C19—C20	113.55 (13)
C7—C9—C10	121.49 (14)	C18—C19—H19A	109.2 (10)
C7—C9—H9	118.2 (10)	C20—C19—H19A	109.2 (10)
C10—C9—H9	120.3 (10)	C18—C19—H19B	109.2 (11)
O2—C10—O3	121.56 (14)	C20—C19—H19B	109.1 (11)
O2—C10—C9	125.67 (15)	H19A—C19—H19B	106.3 (14)
O3—C10—C9	112.78 (13)	C21—C20—C19	113.94 (13)
O3—C11—C12	107.73 (15)	C21—C20—H20A	109.4 (10)
O3—C11—H11A	107.8 (12)	C19—C20—H20A	108.7 (10)
C12—C11—H11A	112.0 (12)	C21—C20—H20B	108.9 (11)
O3—C11—H11B	107.5 (11)	C19—C20—H20B	109.3 (11)
C12—C11—H11B	110.8 (11)	H20A—C20—H20B	106.2 (14)
H11A—C11—H11B	110.8 (16)	C20—C21—C22	113.34 (14)
C11—C12—H12A	109.9 (13)	C20—C21—H21A	109.7 (11)
C11—C12—H12B	111.1 (12)	C22—C21—H21A	109.6 (11)
H12A—C12—H12B	107.5 (18)	C20—C21—H21B	108.4 (11)
C11—C12—H12C	110.2 (13)	C22—C21—H21B	108.7 (11)
H12A—C12—H12C	110.2 (18)	H21A—C21—H21B	106.9 (15)
H12B—C12—H12C	108.0 (18)	C21—C22—H22A	112.0 (11)
N2—C13—C14	112.57 (12)	C21—C22—H22B	110.2 (12)
N2—C13—H13A	105.6 (9)	H22A—C22—H22B	107.5 (16)
C14—C13—H13A	111.2 (9)	C21—C22—H22C	111.5 (12)
N2—C13—H13B	108.1 (10)	H22A—C22—H22C	107.9 (16)
C14—C13—H13B	110.9 (10)	H22B—C22—H22C	107.6 (17)
H13A—C13—H13B	108.1 (14)		

C8—N2—C1—C6	−2.0 (2)	C13—N2—C8—C7	−177.40 (13)
C13—N2—C1—C6	−179.39 (13)	N1—C7—C8—O1	174.79 (13)
C8—N2—C1—C2	177.41 (14)	C9—C7—C8—O1	−4.5 (2)
C13—N2—C1—C2	0.0 (2)	N1—C7—C8—N2	−4.6 (2)

C6—C1—C2—C3	0.7 (2)	C9—C7—C8—N2	176.12 (13)
N2—C1—C2—C3	−178.67 (14)	N1—C7—C9—C10	−1.0 (2)
C1—C2—C3—C4	0.0 (2)	C8—C7—C9—C10	178.21 (14)
C2—C3—C4—C5	−0.4 (3)	C11—O3—C10—O2	−3.0 (2)
C3—C4—C5—C6	0.1 (3)	C11—O3—C10—C9	177.34 (14)
C7—N1—C6—C5	−177.99 (15)	C7—C9—C10—O2	−2.9 (3)
C7—N1—C6—C1	2.2 (2)	C7—C9—C10—O3	176.72 (14)
C4—C5—C6—N1	−179.18 (15)	C10—O3—C11—C12	176.51 (15)
C4—C5—C6—C1	0.7 (2)	C8—N2—C13—C14	−99.13 (15)
C2—C1—C6—N1	178.79 (14)	C1—N2—C13—C14	78.44 (17)
N2—C1—C6—N1	−1.8 (2)	N2—C13—C14—C15	−172.45 (13)
C2—C1—C6—C5	−1.1 (2)	C13—C14—C15—C16	178.02 (13)
N2—C1—C6—C5	178.34 (14)	C14—C15—C16—C17	−179.84 (13)
C6—N1—C7—C9	−179.69 (14)	C15—C16—C17—C18	179.79 (13)
C6—N1—C7—C8	1.1 (2)	C16—C17—C18—C19	179.39 (13)
C1—N2—C8—O1	−174.27 (13)	C17—C18—C19—C20	−179.12 (13)
C13—N2—C8—O1	3.2 (2)	C18—C19—C20—C21	−179.46 (14)
C1—N2—C8—C7	5.1 (2)	C19—C20—C21—C22	−179.64 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.88 (2)	1.99 (2)	2.6885 (18)	134.8 (17)
C3—H3···O1^{i}	0.996 (19)	2.491 (19)	3.278 (2)	135.6 (14)
C11—H11*A*···O2^{ii}	0.96 (2)	2.59 (2)	3.424 (2)	144.7 (16)
C13—H13*B*···O1^{iii}	0.983 (18)	2.541 (18)	3.2714 (19)	131.0 (13)

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