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(E)-N'-(3,4,5-Trimethoxybenzylidene)-2-(8-quinolyloxy)acetohydrazide methanol solvate

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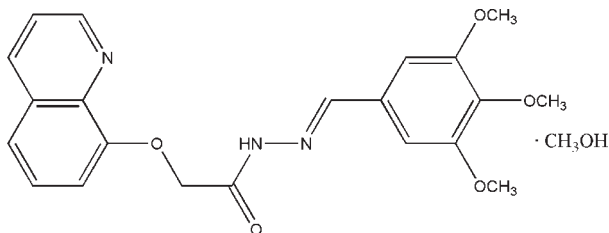
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.043; wR factor = 0.099; data-to-parameter ratio = 6.6.

In the title compound, $\text{C}_{21}\text{H}_{21}\text{N}_3\text{O}_5 \cdot \text{CH}_4\text{O}$, the quinoline plane and the benzene ring form a dihedral angle of $3.6(2)^\circ$. The methanol solvent molecule is linked with the acetohydrazide molecule *via* $\text{O}-\text{H} \cdots \text{N}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds. In the crystal structure, weak intermolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds help to consolidate the crystal packing, which also exhibits $\pi-\pi$ interactions, as indicated by short distances of $3.739(4)$ Å between the centroids of the aromatic rings.

Related literature

For applications of 8-hydroxyquinoline derivatives, see: Park *et al.* (2006); Karmakar *et al.* (2007). For a related structure, see Wang *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{21}\text{H}_{21}\text{N}_3\text{O}_5 \cdot \text{CH}_4\text{O}$ $M_r = 427.45$ Orthorhombic, $Pna2_1$ $a = 13.385(4)$ Å $b = 4.9005(15)$ Å $c = 31.89(1)$ Å $V = 2091.8(11)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.10$ mm⁻¹ $T = 295$ K $0.18 \times 0.15 \times 0.12$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

10056 measured reflections

1879 independent reflections

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $R_{\text{int}} = 0.077$ $T_{\text{min}} = 0.982$, $T_{\text{max}} = 0.988$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.043$ $wR(F^2) = 0.099$ $S = 1.08$

1879 reflections

283 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.15$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O6}-\text{H6} \cdots \text{N1}$	0.82	2.02	2.814 (5)	164
$\text{N2}-\text{H4} \cdots \text{O6}$	0.86	2.30	3.070 (4)	149
$\text{C3}-\text{H3} \cdots \text{O5}^i$	0.93	2.45	3.340 (6)	159
$\text{C5}-\text{H5} \cdots \text{O4}^i$	0.93	2.52	3.411 (6)	160
$\text{C19}-\text{H19A} \cdots \text{O2}^{ii}$	0.96	2.37	3.196 (6)	144
$\text{C21}-\text{H21A} \cdots \text{O3}^{iii}$	0.96	2.57	3.265 (5)	130

Symmetry codes: (i) $-x + 1, -y + 1, z - \frac{1}{2}$; (ii) $x - \frac{1}{2}, -y + \frac{3}{2}, z$; (iii) $x + \frac{1}{2}, -y + \frac{3}{2}, z$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2668).

References

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supplementary materials

Acta Cryst. (2010). E66, o23 [doi:10.1107/S1600536809051113]

(*E*)-*N'*-(3,4,5-Trimethoxybenzylidene)-2-(8-quinolyloxy)acetohydrazide methanol solvate

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Comment

Synthesis of 8-hydroxyquinoline and its derivatives have attracted a great interest due to their biological activities and applications in coordination chemistry (Park *et al.*, 2006; Karmakar *et al.*, 2007). In our search for new extractants of metal ions and biologically active materials, the title compound, (I), has been synthesized. We report here its crystal structure.

All bond lengths and angles are normal and comparable to those observed in the related compound (*E*)-*N'*-(2,5-dimethoxybenzylidene)-2-(8-quinolyloxy)acetohydrazide methanol solvate (Wang *et al.*, 2009). The molecule is nearly planar, with a dihedral angle of the benzene ring and the quinoline ring of 3.6 (2)°. The methanol solvent molecule forms an O—H···N hydrogen bond to the quinoline ring system and accepts an N—H···O hydrogen bond from the hydrazide NH group. In the crystal structure, weak intermolecular C—H···O hydrogen bonds (Table 1) help to consolidate the crystal packing.

Experimental

3,4,5-Trimethoxybenzaldehyde (0.1 mmol, 19.6 mg) and 2-(quinolin-8-yloxy) acetohydrazide (21.8 mg, 0.1 mmol), were dissolved in methanol (20 ml). The mixture was stirred at room temperature to give a clear colorless solution. Crystals of the title compound were formed by gradual evaporation of the solvent over a period of six days at room temperature.

Refinement

All H atoms were initially located in a difference Fourier map, then placed in idealized positions (C—H 0.93–0.97 Å, O—H 0.82–0.85 Å, N—H 0.86 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ and $1.5U_{\text{eq}}(\text{O})$. In the absence of atoms heavier than Si, the absolute structure can not be reliably determined, so 1784 Friedel pairs were averaged before the final refinement.

Figures

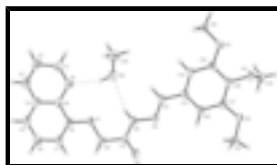


Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. Dashed lines indicate hydrogen bonds.

(*E*)-*N'*-(3,4,5-Trimethoxybenzylidene)-2-(8-quinolyloxy)acetohydrazide methanol solvate

Crystal data

C₂₁H₂₁N₃O₅·CH₄O

$F(000) = 904$

supplementary materials

$M_r = 427.45$

Orthorhombic, $Pna2_1$

Hall symbol: P 2c -2n

$a = 13.385 (4) \text{ \AA}$

$b = 4.9005 (15) \text{ \AA}$

$c = 31.89 (1) \text{ \AA}$

$V = 2091.8 (11) \text{ \AA}^3$

$Z = 4$

$D_x = 1.357 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 786 reflections

$\theta = 2.6\text{--}17.8^\circ$

$\mu = 0.10 \text{ mm}^{-1}$

$T = 295 \text{ K}$

Block, colourless

$0.18 \times 0.15 \times 0.12 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube
graphite

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.982$, $T_{\max} = 0.988$

10056 measured reflections

1879 independent reflections

1263 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.077$

$\theta_{\max} = 25.1^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -15 \rightarrow 13$

$k = -5 \rightarrow 5$

$l = -37 \rightarrow 37$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.099$

$S = 1.08$

1879 reflections

283 parameters

0 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 0.0119P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.15 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.16 \text{ e \AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick, 2008),

$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0111 (15)

Special details

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.6229 (2)	0.7115 (6)	0.13448 (8)	0.0497 (8)
O2	0.6522 (3)	1.1590 (8)	0.22093 (11)	0.0819 (12)
O3	0.1718 (2)	0.3242 (6)	0.34960 (8)	0.0495 (8)
O4	0.2323 (2)	0.6336 (6)	0.41318 (8)	0.0448 (8)
O5	0.3852 (2)	0.9783 (6)	0.40569 (9)	0.0515 (9)
O6	0.4246 (3)	0.4033 (8)	0.16176 (12)	0.0776 (12)
H6	0.4642	0.3998	0.1421	0.116*
N1	0.5266 (3)	0.3563 (9)	0.08495 (12)	0.0543 (11)
N2	0.5580 (3)	0.7798 (8)	0.21461 (10)	0.0475 (10)
H4	0.5427	0.6407	0.1995	0.057*
N3	0.5127 (3)	0.8173 (8)	0.25337 (10)	0.0449 (10)
C1	0.4809 (4)	0.1830 (12)	0.06048 (17)	0.0679 (16)
H1	0.4270	0.0872	0.0716	0.081*
C2	0.5070 (5)	0.1317 (12)	0.01916 (17)	0.0679 (16)
H2	0.4720	0.0042	0.0033	0.082*
C3	0.5852 (4)	0.2727 (12)	0.00229 (15)	0.0635 (15)
H3	0.6037	0.2437	-0.0255	0.076*
C4	0.6372 (4)	0.4592 (10)	0.02669 (14)	0.0481 (12)
C5	0.7184 (4)	0.6099 (12)	0.01122 (15)	0.0610 (15)
H5	0.7386	0.5885	-0.0165	0.073*
C6	0.7676 (4)	0.7867 (11)	0.03669 (15)	0.0597 (14)
H6A	0.8220	0.8836	0.0263	0.072*
C7	0.7376 (4)	0.8255 (11)	0.07854 (14)	0.0530 (13)
H7	0.7724	0.9470	0.0955	0.064*
C8	0.6581 (3)	0.6872 (10)	0.09433 (12)	0.0436 (12)
C9	0.6056 (3)	0.4985 (10)	0.06885 (13)	0.0439 (12)
C10	0.6703 (3)	0.9191 (9)	0.15890 (14)	0.0486 (12)
H10A	0.6671	1.0901	0.1436	0.058*
H10B	0.7402	0.8724	0.1624	0.058*
C11	0.6250 (3)	0.9590 (11)	0.20091 (14)	0.0479 (12)
C12	0.4417 (3)	0.6516 (10)	0.26137 (14)	0.0463 (12)
H12	0.4241	0.5212	0.2415	0.056*
C13	0.3880 (3)	0.6625 (10)	0.30083 (12)	0.0417 (12)
C14	0.3068 (3)	0.4905 (9)	0.30537 (12)	0.0421 (11)
H14	0.2883	0.3768	0.2833	0.050*
C15	0.2527 (3)	0.4860 (9)	0.34250 (12)	0.0389 (11)
C16	0.2808 (3)	0.6550 (10)	0.37560 (12)	0.0374 (11)
C17	0.3629 (3)	0.8264 (9)	0.37111 (12)	0.0405 (11)
C18	0.4170 (3)	0.8321 (9)	0.33362 (12)	0.0409 (12)
H18	0.4717	0.9477	0.3306	0.049*
C19	0.1410 (4)	0.1460 (10)	0.31644 (14)	0.0520 (12)
H19A	0.1206	0.2525	0.2927	0.078*

supplementary materials

H19B	0.0861	0.0359	0.3258	0.078*
H19C	0.1957	0.0301	0.3086	0.078*
C20	0.1689 (4)	0.8559 (10)	0.42331 (16)	0.0624 (15)
H20A	0.2052	1.0238	0.4203	0.094*
H20B	0.1462	0.8378	0.4517	0.094*
H20C	0.1125	0.8567	0.4047	0.094*
C21	0.4632 (3)	1.1764 (10)	0.40269 (14)	0.0513 (13)
H21A	0.5264	1.0852	0.4002	0.077*
H21B	0.4633	1.2881	0.4274	0.077*
H21C	0.4524	1.2889	0.3785	0.077*
C22	0.3780 (4)	0.1473 (12)	0.1656 (2)	0.0755 (16)
H22A	0.4269	0.0056	0.1624	0.113*
H22B	0.3280	0.1286	0.1442	0.113*
H22C	0.3472	0.1329	0.1927	0.113*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0574 (19)	0.061 (2)	0.0310 (16)	-0.0138 (17)	0.0085 (14)	-0.0099 (15)
O2	0.104 (3)	0.092 (3)	0.050 (2)	-0.048 (2)	0.0258 (19)	-0.030 (2)
O3	0.052 (2)	0.058 (2)	0.0388 (17)	-0.0123 (18)	0.0051 (14)	-0.0016 (16)
O4	0.0519 (19)	0.052 (2)	0.0299 (16)	0.0046 (16)	0.0083 (14)	0.0080 (15)
O5	0.0585 (19)	0.061 (2)	0.0350 (17)	-0.0140 (19)	0.0038 (15)	-0.0064 (16)
O6	0.088 (3)	0.080 (3)	0.064 (3)	-0.023 (2)	0.018 (2)	-0.018 (2)
N1	0.056 (2)	0.059 (3)	0.048 (2)	-0.009 (2)	0.000 (2)	-0.008 (2)
N2	0.058 (2)	0.054 (3)	0.031 (2)	-0.001 (2)	0.0097 (17)	-0.0052 (18)
N3	0.051 (2)	0.057 (3)	0.0264 (19)	0.003 (2)	0.0121 (18)	-0.0002 (18)
C1	0.063 (3)	0.080 (4)	0.060 (3)	-0.016 (3)	0.000 (3)	-0.019 (3)
C2	0.080 (4)	0.073 (4)	0.052 (3)	-0.001 (4)	-0.011 (3)	-0.022 (3)
C3	0.077 (4)	0.076 (4)	0.037 (3)	0.017 (3)	-0.007 (3)	-0.010 (3)
C4	0.064 (3)	0.048 (3)	0.033 (3)	0.014 (3)	-0.001 (2)	-0.005 (2)
C5	0.077 (4)	0.070 (4)	0.036 (3)	0.013 (3)	0.010 (3)	-0.007 (3)
C6	0.067 (3)	0.069 (4)	0.043 (3)	0.004 (3)	0.017 (3)	0.002 (3)
C7	0.058 (3)	0.065 (4)	0.036 (3)	-0.004 (3)	0.012 (2)	-0.008 (2)
C8	0.052 (3)	0.052 (3)	0.027 (2)	-0.001 (3)	0.005 (2)	-0.003 (2)
C9	0.051 (3)	0.043 (3)	0.038 (3)	0.007 (3)	0.002 (2)	0.000 (2)
C10	0.052 (3)	0.060 (3)	0.034 (2)	-0.013 (3)	0.005 (2)	-0.010 (2)
C11	0.046 (3)	0.062 (4)	0.035 (3)	-0.010 (3)	0.006 (2)	-0.009 (3)
C12	0.051 (3)	0.055 (3)	0.033 (2)	-0.005 (3)	0.008 (2)	-0.003 (2)
C13	0.041 (3)	0.052 (3)	0.032 (2)	0.002 (2)	0.005 (2)	0.001 (2)
C14	0.047 (3)	0.047 (3)	0.033 (2)	-0.001 (2)	0.0023 (19)	-0.003 (2)
C15	0.037 (3)	0.043 (3)	0.037 (3)	0.001 (2)	0.0049 (19)	0.010 (2)
C16	0.041 (3)	0.042 (3)	0.028 (2)	0.003 (2)	0.0052 (19)	0.002 (2)
C17	0.046 (3)	0.047 (3)	0.029 (2)	0.007 (2)	-0.0021 (19)	0.001 (2)
C18	0.043 (3)	0.047 (3)	0.032 (2)	0.001 (2)	0.004 (2)	0.004 (2)
C19	0.057 (3)	0.058 (3)	0.041 (3)	-0.015 (3)	-0.005 (2)	-0.001 (3)
C20	0.070 (3)	0.058 (4)	0.059 (3)	0.002 (3)	0.025 (3)	0.002 (3)
C21	0.050 (3)	0.050 (3)	0.053 (3)	-0.007 (3)	-0.003 (2)	-0.010 (2)

C22 0.069 (4) 0.069 (4) 0.088 (4) -0.001 (3) -0.003 (3) 0.008 (3)

Geometric parameters (Å, °)

O1—C8	1.369 (5)	C7—C8	1.358 (6)
O1—C10	1.429 (5)	C7—H7	0.9300
O2—C11	1.225 (5)	C8—C9	1.418 (6)
O3—C15	1.360 (5)	C10—C11	1.483 (6)
O3—C19	1.432 (5)	C10—H10A	0.9700
O4—C16	1.367 (5)	C10—H10B	0.9700
O4—C20	1.418 (5)	C12—C13	1.450 (6)
O5—C17	1.364 (5)	C12—H12	0.9300
O5—C21	1.429 (5)	C13—C14	1.382 (6)
O6—C22	1.407 (6)	C13—C18	1.391 (6)
O6—H6	0.8200	C14—C15	1.389 (5)
N1—C1	1.306 (6)	C14—H14	0.9300
N1—C9	1.366 (6)	C15—C16	1.394 (6)
N2—C11	1.329 (5)	C16—C17	1.390 (6)
N2—N3	1.389 (5)	C17—C18	1.398 (6)
N2—H4	0.8600	C18—H18	0.9300
N3—C12	1.275 (5)	C19—H19A	0.9600
C1—C2	1.386 (7)	C19—H19B	0.9600
C1—H1	0.9300	C19—H19C	0.9600
C2—C3	1.365 (8)	C20—H20A	0.9600
C2—H2	0.9300	C20—H20B	0.9600
C3—C4	1.387 (7)	C20—H20C	0.9600
C3—H3	0.9300	C21—H21A	0.9600
C4—C5	1.404 (7)	C21—H21B	0.9600
C4—C9	1.422 (6)	C21—H21C	0.9600
C5—C6	1.358 (7)	C22—H22A	0.9600
C5—H5	0.9300	C22—H22B	0.9600
C6—C7	1.407 (6)	C22—H22C	0.9600
C6—H6A	0.9300		
C8—O1—C10	114.8 (3)	N3—C12—C13	121.3 (4)
C15—O3—C19	117.5 (3)	N3—C12—H12	119.4
C16—O4—C20	115.1 (3)	C13—C12—H12	119.4
C17—O5—C21	118.5 (3)	C14—C13—C18	120.3 (4)
C22—O6—H6	109.5	C14—C13—C12	117.3 (4)
C1—N1—C9	118.0 (4)	C18—C13—C12	122.4 (4)
C11—N2—N3	120.0 (4)	C13—C14—C15	120.6 (4)
C11—N2—H4	120.0	C13—C14—H14	119.7
N3—N2—H4	120.0	C15—C14—H14	119.7
C12—N3—N2	114.8 (4)	O3—C15—C14	124.5 (4)
N1—C1—C2	124.6 (5)	O3—C15—C16	115.8 (4)
N1—C1—H1	117.7	C14—C15—C16	119.7 (4)
C2—C1—H1	117.7	O4—C16—C17	120.8 (4)
C3—C2—C1	118.4 (5)	O4—C16—C15	119.3 (4)
C3—C2—H2	120.8	C17—C16—C15	119.6 (3)
C1—C2—H2	120.8	O5—C17—C16	114.8 (3)

supplementary materials

C2—C3—C4	119.8 (5)	O5—C17—C18	124.5 (4)
C2—C3—H3	120.1	C16—C17—C18	120.6 (4)
C4—C3—H3	120.1	C13—C18—C17	119.1 (4)
C3—C4—C5	122.5 (5)	C13—C18—H18	120.5
C3—C4—C9	118.1 (5)	C17—C18—H18	120.5
C5—C4—C9	119.4 (5)	O3—C19—H19A	109.5
C6—C5—C4	120.1 (5)	O3—C19—H19B	109.5
C6—C5—H5	120.0	H19A—C19—H19B	109.5
C4—C5—H5	120.0	O3—C19—H19C	109.5
C5—C6—C7	121.0 (5)	H19A—C19—H19C	109.5
C5—C6—H6A	119.5	H19B—C19—H19C	109.5
C7—C6—H6A	119.5	O4—C20—H20A	109.5
C8—C7—C6	120.6 (5)	O4—C20—H20B	109.5
C8—C7—H7	119.7	H20A—C20—H20B	109.5
C6—C7—H7	119.7	O4—C20—H20C	109.5
C7—C8—O1	124.9 (4)	H20A—C20—H20C	109.5
C7—C8—C9	120.1 (4)	H20B—C20—H20C	109.5
O1—C8—C9	115.0 (4)	O5—C21—H21A	109.5
N1—C9—C8	120.1 (4)	O5—C21—H21B	109.5
N1—C9—C4	121.1 (4)	H21A—C21—H21B	109.5
C8—C9—C4	118.9 (4)	O5—C21—H21C	109.5
O1—C10—C11	113.9 (4)	H21A—C21—H21C	109.5
O1—C10—H10A	108.8	H21B—C21—H21C	109.5
C11—C10—H10A	108.8	O6—C22—H22A	109.5
O1—C10—H10B	108.8	O6—C22—H22B	109.5
C11—C10—H10B	108.8	H22A—C22—H22B	109.5
H10A—C10—H10B	107.7	O6—C22—H22C	109.5
O2—C11—N2	123.9 (4)	H22A—C22—H22C	109.5
O2—C11—C10	117.0 (4)	H22B—C22—H22C	109.5
N2—C11—C10	119.1 (4)		
C11—N2—N3—C12	172.4 (4)	O1—C10—C11—O2	-169.6 (4)
C9—N1—C1—C2	0.2 (8)	O1—C10—C11—N2	10.2 (7)
N1—C1—C2—C3	-0.7 (9)	N2—N3—C12—C13	179.2 (4)
C1—C2—C3—C4	0.8 (8)	N3—C12—C13—C14	174.7 (4)
C2—C3—C4—C5	179.8 (5)	N3—C12—C13—C18	-7.0 (7)
C2—C3—C4—C9	-0.6 (7)	C18—C13—C14—C15	0.5 (7)
C3—C4—C5—C6	-179.0 (5)	C12—C13—C14—C15	178.9 (4)
C9—C4—C5—C6	1.5 (7)	C19—O3—C15—C14	0.2 (6)
C4—C5—C6—C7	-1.0 (8)	C19—O3—C15—C16	-179.5 (4)
C5—C6—C7—C8	-0.3 (8)	C13—C14—C15—O3	179.8 (4)
C6—C7—C8—O1	-179.7 (4)	C13—C14—C15—C16	-0.4 (6)
C6—C7—C8—C9	1.0 (7)	C20—O4—C16—C17	76.2 (5)
C10—O1—C8—C7	6.0 (6)	C20—O4—C16—C15	-109.2 (5)
C10—O1—C8—C9	-174.7 (4)	O3—C15—C16—O4	5.0 (6)
C1—N1—C9—C8	-179.4 (5)	C14—C15—C16—O4	-174.7 (4)
C1—N1—C9—C4	0.0 (7)	O3—C15—C16—C17	179.7 (4)
C7—C8—C9—N1	179.0 (4)	C14—C15—C16—C17	0.0 (6)
O1—C8—C9—N1	-0.3 (6)	C21—O5—C17—C16	-174.9 (4)
C7—C8—C9—C4	-0.5 (6)	C21—O5—C17—C18	6.1 (6)

O1—C8—C9—C4	-179.8 (4)	O4—C16—C17—O5	-4.0 (6)
C3—C4—C9—N1	0.2 (6)	C15—C16—C17—O5	-178.6 (4)
C5—C4—C9—N1	179.7 (5)	O4—C16—C17—C18	175.1 (4)
C3—C4—C9—C8	179.7 (4)	C15—C16—C17—C18	0.5 (6)
C5—C4—C9—C8	-0.8 (6)	C14—C13—C18—C17	-0.1 (7)
C8—O1—C10—C11	174.8 (4)	C12—C13—C18—C17	-178.4 (4)
N3—N2—C11—O2	1.5 (7)	O5—C17—C18—C13	178.6 (4)
N3—N2—C11—C10	-178.3 (4)	C16—C17—C18—C13	-0.4 (6)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O6—H6...N1	0.82	2.02	2.814 (5)	164.
N2—H4...O6	0.86	2.30	3.070 (4)	149.
C3—H3...O5 ⁱ	0.93	2.45	3.340 (6)	159.
C5—H5...O4 ⁱ	0.93	2.52	3.411 (6)	160.
C19—H19A...O2 ⁱⁱ	0.96	2.37	3.196 (6)	144.
C21—H21A...O3 ⁱⁱⁱ	0.96	2.57	3.265 (5)	130.

Symmetry codes: (i) $-x+1, -y+1, z-1/2$; (ii) $x-1/2, -y+3/2, z$; (iii) $x+1/2, -y+3/2, z$.

Fig. 1

