Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diethyl 1,4-dioxo-6-(3-pyridyl)perhydro-2,3,4a,6,7a-pentaazacyclopenta[*cd*]-indene-2a,7b-dicarboxylate 1,2-dichoroethane solvate

Li-ping Cao,[a]* Zhi-guo Wang[b] and Yan Hu[a]

[a]Key Laboratory of Pesticides and Chemical Biology of the Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China, and [b]School of Chemical and Materials Engineering, Huangshi Insititute of Technology, Huangshi 435003, People's Republic of China
Correspondence e-mail: chlpcao@mails.ccnu.edu.cn

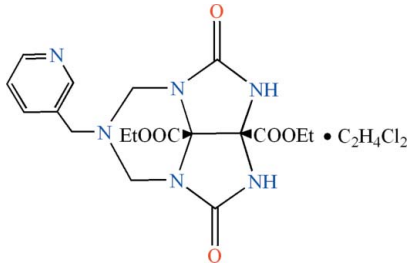
Received 16 October 2007; accepted 30 October 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.072; wR factor = 0.225; data-to-parameter ratio = 11.5.

The main residue of the title compound, $C_{18}H_{22}N_6O_6\cdot C_2H_4Cl_2$, is a derivative of glycoluril. The six-membered heterocycle adopts a chair conformation and shares two N atoms with two five-membered rings of the glycoluril unit to form the flexible sidewalls of a molecular clip. Two ethyl fragments and the solvent molecule are disordered; the site occupancy factors for the ethyl groups are 0.72/0.28 and 0.75/0.25 and for the solvent molecule 0.54/0.46. Intermolecular N—H···O and N—H···N hydrogen bonds link the cyclopenta[*cd*]indene molecules into two-dimensional layers parallel to the *bc* plane.

Related literature

For details of the synthesis, see: Li *et al.* (2006). For general background, see: Behrend *et al.* (1905); Freeman *et al.* (1981); Rebek (2005); Rowan *et al.* (1999); Wu *et al.* (2002).



Experimental

Crystal data

$C_{18}H_{22}N_6O_6\cdot C_2H_4Cl_2$
M_r = 517.37
Monoclinic, $P2_1/c$
a = 12.9023 (9) Å
b = 16.4032 (11) Å
c = 12.1719 (8) Å
β = 114.657 (1)°
V = 2341.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 294 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: none
13883 measured reflections
4114 independent reflections
2684 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.225
S = 1.04
4114 reflections
359 parameters
26 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6···N1^{i}	0.86	2.07	2.892 (4)	160
N5—H5···O1^{ii}	0.86	2.03	2.862 (3)	162

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

We thank Dr Xiang-Gao Meng for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2321).

References

Behrend, R., Meyer, E. & Rusche, F. (1905). *Liebigs Ann. Chem.* **339**, 1–37.
Bruker (2001). *SMART* (Version 5.628) and *SAINT* (Version 6.45). Bruker AXS Inc., Madison, Wisconsin, USA.
Freeman, W. A., Mock, W. L. & Shih, N. Y. (1981). *J. Am. Chem. Soc.* **103**, 7367–7368.
Li, Y., Yin, G., Guo, H., Zhou, B. & Wu, A. (2006). *Synthesis*, **17**, 2897–2902.
Rebek, J. Jr (2005). *Angew. Chem. Int. Ed.* **44**, 2068–2078.
Rowan, A. E., Elemans, J. A. A. W. & Nolte, R. J. M. (1999). *Acc. Chem. Res.* **32**, 995–1006.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wu, A., Fettinger, J. C. & Isaacs, L. (2002). *Tetrahedron*, **58**, 9769–9777.

  doi:10.1107/S1600536807054566

supplementary materials

Acta Cryst. (2007). E**63**, o4540 [**doi:10.1107/S1600536807054566**]

Diethyl 1,4-dioxo-6-(3-pyridyl)perhydro-2,3,4a,6,7a-pentaazacyclopenta[*cd*]indene-2a,7b-dicarboxylate 1,2-dichoroethane solvate

L. Cao, Z. Wang and Y. Hu

Comment

In 1905, Behrend reported that the condensation of glycoluril and formaldehyde in dilute HCl yielded an insoluble polymeric material now known as Behrend's polymer (Behrend *et al.*, 1905). Glycoluril and its derivatives have during the past two decades established an impressive career as building blocks for supramolecular chemistry (Freeman *et al.*, 1981; Rebek, 2005; Rowan *et al.*, 1999; Wu *et al.*, 2002). As a part of our ongoing investigation into glycoluril derivatives (Li *et al.*, 2006), we report here the structure of the title compound, 1,2-dichoroethane solvate of (I) (Fig. 1).

The molecular structure of the main residue, (I), and solvent molecule are shown in Fig. 1. Molecule (I) has three fused rings, namely, two nearly planar imidazole five-membered rings that adopt envelope conformation with the C═O groups at the flap position and one non-planar triazine six-membered ring that adopts a chair conformation.

In the crystal, the intermolecular N—H···O and N—H···N hydrogen bonds (Table 1) link the molecules of (I) into two-dimensional layers parallel to *bc* plane.

Experimental

The title compound was synthesized according to the procedure of Li *et al.* (2006) in 45% isolated yield. Crystals for X-ray data collection were obtained by slow evaporation of a 1,2-dichoroethane and methanol solution in a ratio of 4:1 at 293 K.

Refinement

C-bound H atoms were geometrically positioned (C–H= 0.93 Å (aromatic), 0.96 Å (methyl) or 0.97Å (methylene)) and refined as riding, with U_{iso}(H) = 1.2Ueq(C), or =U_{eq}(C) for disordered C atoms. N-bound H atoms were found in difference, but placed in idealized positions (N–H= 0.86 Å), and refined as riding, with U_{iso}(H)=1.2Ueq(N). Two ethyl fragments in (I) were treated as disordered between two orientations each, with the refined occupancies 0.720 (9)/0.280 (9) and 0.754 (11)/0.246 (11), respectively. The solvent molecule was also treated as disordered between two positions, with the refined occupancies 0.458 (10) and 0.542 (10), respectively.

Figures

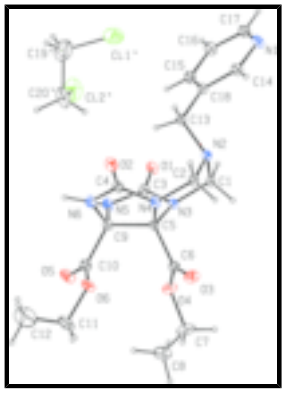


Fig. 1. A content of asymmetric unit of the title compound, showing the atomic numbering and displacement ellipsoids at the 10% probability level. H atoms are represented by spheres of arbitrary radius. Only major parts of disordered fragments are shown.

Diethyl 1,4-dioxo-6-(3-pyridyl)perhydro-2,3,4a,6,7a-pentaazacyclopenta[cd]indene- 2a,7 b-dicarboxylate 1,2-dichoroethane solvate

Crystal data

$C_{18}H_{22}N_6O_6{\cdot}C_2H_4Cl_2$	F_{000} = 1080
M_r = 517.37	D_x = 1.468 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2658 reflections
a = 12.9023 (9) Å	θ = 2.3–21.1º
b = 16.4032 (11) Å	μ = 0.33 mm^{-1}
c = 12.1719 (8) Å	T = 294 (2) K
β = 114.657 (1)º	Block, colourless
V = 2341.2 (3) Å^3	0.20 × 0.10 × 0.10 mm
Z = 4	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	2684 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.058
Monochromator: graphite	θ_{max} = 25.0º
T = 294(2) K	θ_{min} = 1.7º
φ and ω scans	h = −15→12
Absorption correction: none	k = −19→19
13883 measured reflections	l = −14→14
4114 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.073$	H-atom parameters constrained
$wR(F^2) = 0.225$	$w = 1/[\sigma^2(F_o^2) + (0.1319P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
4114 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
359 parameters	$\Delta\rho_{min} = -0.46$ e Å^{-3}
26 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2$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)
C19	0.4234 (19)	0.4751 (19)	0.346 (3)	0.30 (2)	0.458 (10)
H19A	0.4754	0.4958	0.4241	0.30*	0.458 (10)
H19B	0.4679	0.4570	0.3028	0.30*	0.458 (10)
C1	0.5988 (3)	0.24533 (17)	0.1119 (3)	0.0434 (8)	
H1A	0.5250	0.2406	0.0438	0.052*	
H1B	0.6482	0.2039	0.1028	0.052*	
C2	0.6934 (3)	0.24312 (19)	0.3264 (3)	0.0490 (9)	
H2A	0.7476	0.2025	0.3252	0.059*	
H2B	0.6830	0.2356	0.4002	0.059*	
C3	0.5798 (3)	0.39281 (18)	0.0564 (3)	0.0440 (8)	
C4	0.7221 (3)	0.3900 (2)	0.3877 (3)	0.0496 (9)	
C5	0.7473 (3)	0.35055 (18)	0.2168 (3)	0.0398 (7)	
C6	0.8552 (3)	0.3164 (2)	0.2086 (3)	0.0502 (9)	
C7	0.9642 (4)	0.3216 (4)	0.0915 (5)	0.1029 (18)	
H7A	0.9511	0.2693	0.0508	0.10*	0.720 (9)
H7B	1.0312	0.3174	0.1676	0.10*	0.720 (9)
H7C	0.9487	0.3301	0.0072	0.10*	0.280 (9)
H7D	0.9793	0.2641	0.1092	0.10*	0.280 (9)
C8	0.9803 (9)	0.3879 (6)	0.0128 (10)	0.139 (4)	0.720 (9)
H8A	0.9284	0.3790	−0.0699	0.14*	0.720 (9)
H8B	1.0572	0.3863	0.0199	0.14*	0.720 (9)
H8C	0.9656	0.4402	0.0388	0.14*	0.720 (9)
C8'	1.0650 (19)	0.3728 (16)	0.146 (3)	0.139 (4)	0.280 (9)

H8'1	1.0879	0.3756	0.2317	0.14*	0.280 (9)
H8'2	1.0479	0.4266	0.1121	0.14*	0.280 (9)
H8'3	1.1258	0.3499	0.1299	0.14*	0.280 (9)
C9	0.7366 (3)	0.44625 (18)	0.2187 (3)	0.0439 (8)	
C10	0.8353 (3)	0.4977 (2)	0.2176 (3)	0.0545 (9)	
C11	1.0337 (10)	0.5246 (6)	0.3268 (11)	0.103 (3)	0.754 (11)
H11A	1.0406	0.5275	0.2506	0.10*	0.754 (11)
H11B	1.1006	0.4970	0.3850	0.10*	0.754 (11)
C12	1.0264 (11)	0.6081 (6)	0.3702 (13)	0.192 (5)	0.754 (11)
H12A	0.9529	0.6309	0.3216	0.19*	0.754 (11)
H12B	1.0847	0.6416	0.3641	0.19*	0.754 (11)
H12C	1.0368	0.6056	0.4530	0.19*	0.754 (11)
C11'	1.019 (4)	0.543 (2)	0.333 (3)	0.103 (3)	0.246 (11)
H11C	1.0947	0.5202	0.3755	0.10*	0.246 (11)
H11D	1.0094	0.5860	0.3833	0.10*	0.246 (11)
C12'	1.006 (3)	0.5791 (19)	0.211 (3)	0.192 (5)	0.246 (11)
H12D	0.9722	0.5391	0.1486	0.19*	0.246 (11)
H12E	1.0799	0.5938	0.2157	0.19*	0.246 (11)
H12F	0.9584	0.6265	0.1927	0.19*	0.246 (11)
C13	0.4906 (3)	0.2760 (2)	0.2311 (3)	0.0488 (8)	
H13A	0.4957	0.3327	0.2112	0.059*	
H13B	0.4967	0.2738	0.3132	0.059*	
C14	0.3768 (3)	0.24205 (19)	0.1459 (3)	0.0454 (8)	
C15	0.2997 (3)	0.2838 (2)	0.0490 (3)	0.0596 (10)	
H15	0.3170	0.3358	0.0311	0.072*	
C16	0.1963 (3)	0.2484 (2)	−0.0223 (4)	0.0684 (11)	
H16	0.1429	0.2764	−0.0880	0.082*	
C17	0.1738 (3)	0.1718 (2)	0.0053 (4)	0.0645 (11)	
H17	0.1040	0.1484	−0.0432	0.077*	
C18	0.3455 (3)	0.1646 (2)	0.1670 (3)	0.0559 (9)	
H18	0.3966	0.1359	0.2334	0.067*	
C20	0.348 (2)	0.5359 (12)	0.281 (2)	0.191 (10)	0.458 (10)
H20A	0.3948	0.5781	0.2682	0.19*	0.458 (10)
H20B	0.3185	0.5588	0.3352	0.19*	0.458 (10)
C19'	0.353 (2)	0.4923 (11)	0.3537 (19)	0.177 (9)	0.542 (10)
H19C	0.2866	0.5249	0.3050	0.18*	0.542 (10)
H19D	0.3791	0.5102	0.4370	0.18*	0.542 (10)
C20'	0.4474 (14)	0.5059 (9)	0.3103 (15)	0.145 (7)	0.542 (10)
H20C	0.5008	0.4608	0.3308	0.145*	0.542 (10)
H20D	0.4881	0.5567	0.3397	0.145*	0.542 (10)
Cl1	0.3467 (12)	0.3958 (6)	0.3638 (8)	0.194 (5)	0.458 (10)
Cl2	0.2351 (10)	0.5258 (4)	0.1477 (5)	0.204 (6)	0.458 (10)
Cl1'	0.3153 (13)	0.3950 (6)	0.3439 (13)	0.285 (8)	0.542 (10)
Cl2'	0.3591 (9)	0.5093 (2)	0.1587 (5)	0.177 (4)	0.542 (10)
N1	0.2469 (3)	0.12886 (18)	0.0982 (3)	0.0627 (9)	
N2	0.5853 (2)	0.23011 (15)	0.2236 (2)	0.0428 (7)	
N3	0.6469 (2)	0.32622 (14)	0.1092 (2)	0.0380 (6)	
N4	0.7410 (2)	0.32533 (14)	0.3273 (2)	0.0418 (7)	
N5	0.6352 (2)	0.46129 (16)	0.1113 (3)	0.0544 (8)	

H5	0.6118	0.5094	0.0844	0.065*
N6	0.7249 (2)	0.45955 (16)	0.3290 (2)	0.0533 (8)
H6	0.7201	0.5073	0.3556	0.064*
O1	0.4855 (2)	0.38855 (13)	−0.0294 (2)	0.0559 (7)
O2	0.7055 (3)	0.38493 (16)	0.4792 (2)	0.0753 (9)
O3	0.9188 (2)	0.26940 (19)	0.2803 (3)	0.0830 (9)
O4	0.8647 (2)	0.34583 (18)	0.1125 (2)	0.0712 (8)
O5	0.8239 (3)	0.54950 (18)	0.1454 (3)	0.0846 (10)
O6	0.9320 (2)	0.47937 (16)	0.3110 (3)	0.0705 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C19	0.31 (6)	0.30 (7)	0.29 (5)	0.00 (4)	0.14 (5)	−0.01 (5)
C1	0.0462 (19)	0.0367 (16)	0.0449 (18)	−0.0002 (14)	0.0168 (16)	−0.0029 (14)
C2	0.053 (2)	0.0409 (17)	0.0445 (19)	−0.0002 (15)	0.0114 (17)	0.0092 (14)
C3	0.045 (2)	0.0413 (17)	0.0375 (18)	0.0022 (14)	0.0093 (17)	0.0071 (14)
C4	0.054 (2)	0.053 (2)	0.0408 (19)	−0.0112 (16)	0.0190 (18)	−0.0049 (15)
C5	0.0342 (17)	0.0398 (16)	0.0399 (17)	0.0013 (13)	0.0101 (14)	0.0041 (13)
C6	0.042 (2)	0.054 (2)	0.052 (2)	0.0006 (16)	0.0157 (18)	0.0011 (17)
C7	0.076 (3)	0.150 (5)	0.108 (4)	0.018 (3)	0.063 (3)	0.007 (3)
C8	0.134 (9)	0.160 (8)	0.176 (9)	−0.044 (6)	0.116 (8)	−0.033 (7)
C8'	0.134 (9)	0.160 (8)	0.176 (9)	−0.044 (6)	0.116 (8)	−0.033 (7)
C9	0.0421 (19)	0.0399 (16)	0.0415 (18)	−0.0001 (14)	0.0092 (15)	0.0056 (13)
C10	0.053 (2)	0.0460 (19)	0.057 (2)	−0.0039 (16)	0.0147 (19)	0.0068 (17)
C11	0.055 (4)	0.088 (6)	0.139 (5)	−0.020 (5)	0.014 (3)	0.023 (4)
C12	0.157 (9)	0.148 (8)	0.236 (11)	−0.009 (7)	0.048 (9)	−0.033 (8)
C11'	0.055 (4)	0.088 (6)	0.139 (5)	−0.020 (5)	0.014 (3)	0.023 (4)
C12'	0.157 (9)	0.148 (8)	0.236 (11)	−0.009 (7)	0.048 (9)	−0.033 (8)
C13	0.056 (2)	0.0442 (17)	0.049 (2)	−0.0032 (15)	0.0244 (18)	−0.0043 (15)
C14	0.047 (2)	0.0416 (17)	0.0496 (19)	−0.0017 (15)	0.0218 (17)	0.0000 (15)
C15	0.059 (2)	0.0439 (18)	0.069 (2)	0.0009 (17)	0.020 (2)	0.0131 (17)
C16	0.056 (3)	0.057 (2)	0.072 (3)	0.0049 (19)	0.006 (2)	0.014 (2)
C17	0.046 (2)	0.061 (2)	0.069 (3)	−0.0062 (18)	0.007 (2)	0.000 (2)
C18	0.055 (2)	0.052 (2)	0.054 (2)	−0.0024 (17)	0.0152 (19)	0.0075 (16)
C20	0.19 (3)	0.19 (3)	0.19 (3)	0.01 (2)	0.08 (2)	−0.003 (18)
C19'	0.19 (2)	0.16 (2)	0.180 (19)	0.026 (17)	0.079 (17)	−0.027 (15)
C20'	0.165 (15)	0.060 (7)	0.187 (18)	−0.044 (9)	0.051 (14)	−0.009 (8)
Cl1	0.292 (10)	0.208 (9)	0.166 (5)	0.138 (8)	0.180 (6)	0.093 (5)
Cl2	0.250 (11)	0.163 (5)	0.131 (4)	−0.074 (6)	0.013 (5)	0.025 (3)
Cl1'	0.368 (13)	0.199 (9)	0.458 (17)	0.014 (7)	0.342 (13)	0.091 (8)
Cl2'	0.290 (10)	0.112 (3)	0.167 (4)	−0.010 (3)	0.134 (6)	0.017 (2)
N1	0.058 (2)	0.0494 (17)	0.070 (2)	−0.0098 (15)	0.0156 (18)	0.0085 (15)
N2	0.0431 (16)	0.0396 (14)	0.0403 (15)	−0.0009 (11)	0.0119 (13)	0.0013 (11)
N3	0.0371 (14)	0.0378 (13)	0.0338 (13)	0.0005 (11)	0.0095 (12)	0.0037 (11)
N4	0.0445 (16)	0.0393 (14)	0.0339 (14)	−0.0030 (11)	0.0085 (12)	0.0045 (11)
N5	0.0477 (17)	0.0368 (14)	0.0565 (18)	0.0019 (12)	−0.0004 (14)	0.0115 (13)
N6	0.063 (2)	0.0413 (15)	0.0565 (18)	−0.0040 (13)	0.0256 (16)	−0.0062 (13)

O1	0.0483 (15)	0.0497 (13)	0.0466 (14)	0.0023 (11)	−0.0032 (12)	0.0108 (10)
O2	0.105 (2)	0.0729 (18)	0.0578 (17)	−0.0234 (15)	0.0442 (17)	−0.0146 (13)
O3	0.0560 (18)	0.093 (2)	0.093 (2)	0.0292 (15)	0.0242 (16)	0.0342 (17)
O4	0.0566 (17)	0.095 (2)	0.0707 (18)	0.0124 (14)	0.0348 (15)	0.0108 (15)
O5	0.078 (2)	0.0799 (19)	0.087 (2)	−0.0123 (15)	0.0253 (18)	0.0347 (17)
O6	0.0419 (15)	0.0689 (17)	0.0802 (19)	−0.0131 (12)	0.0053 (14)	0.0163 (14)

Geometric parameters (Å, °)

C19—C20	1.388 (18)	C10—O6	1.325 (4)
C19—Cl1	1.70 (2)	C11—O6	1.449 (9)
C19—H19A	0.9700	C11—C12	1.485 (9)
C19—H19B	0.9700	C11—H11A	0.9700
C1—N2	1.463 (4)	C11—H11B	0.9700
C1—N3	1.471 (4)	C12—H12A	0.9600
C1—H1A	0.9700	C12—H12B	0.9600
C1—H1B	0.9700	C12—H12C	0.9600
C2—N2	1.449 (4)	C11'—O6	1.48 (2)
C2—N4	1.480 (4)	C11'—C12'	1.542 (11)
C2—H2A	0.9700	C11'—H11C	0.9700
C2—H2B	0.9700	C11'—H11D	0.9700
C3—O1	1.231 (4)	C12'—H12D	0.9600
C3—N5	1.349 (4)	C12'—H12E	0.9600
C3—N3	1.375 (4)	C12'—H12F	0.9600
C4—O2	1.222 (4)	C13—N2	1.470 (4)
C4—N6	1.355 (4)	C13—C14	1.507 (5)
C4—N4	1.368 (4)	C13—H13A	0.9700
C5—N4	1.442 (4)	C13—H13B	0.9700
C5—N3	1.462 (4)	C14—C15	1.368 (5)
C5—C6	1.542 (5)	C14—C18	1.389 (5)
C5—C9	1.577 (4)	C15—C16	1.380 (5)
C6—O3	1.197 (4)	C15—H15	0.9300
C6—O4	1.318 (4)	C16—C17	1.362 (5)
C7—C8'	1.455 (16)	C16—H16	0.9300
C7—O4	1.464 (5)	C17—N1	1.333 (5)
C7—C8	1.519 (9)	C17—H17	0.9300
C7—H7A	0.9700	C18—N1	1.332 (4)
C7—H7B	0.9700	C18—H18	0.9300
C7—H7C	0.9700	C20—Cl2	1.672 (17)
C7—H7D	0.9700	C20—H20A	0.9700
C8—H7C	1.0229	C20—H20B	0.9700
C8—H8A	0.9600	C19'—C20'	1.538 (17)
C8—H8B	0.9600	C19'—Cl1'	1.658 (17)
C8—H8C	0.9600	C19'—H19C	0.9700
C8'—H8'1	0.9600	C19'—H19D	0.9700
C8'—H8'2	0.9600	C20'—Cl2'	1.718 (16)
C8'—H8'3	0.9600	C20'—H20C	0.9700
C9—N6	1.428 (4)	C20'—H20D	0.9700
C9—N5	1.433 (4)	N5—H5	0.8600

C9—C10	1.532 (5)	N6—H6	0.8600
C10—O5	1.187 (4)		
C20—C19—Cl1	108.4 (18)	O6—C11—H11A	109.8
C20—C19—H19A	110.0	C12—C11—H11A	109.8
Cl1—C19—H19A	110.0	O6—C11—H11B	109.8
C20—C19—H19B	110.0	C12—C11—H11B	109.8
Cl1—C19—H19B	110.0	H11A—C11—H11B	108.3
H19A—C19—H19B	108.4	O6—C11'—C12'	109.0 (17)
N2—C1—N3	112.9 (2)	O6—C11'—H11C	109.9
N2—C1—H1A	109.0	C12'—C11'—H11C	109.9
N3—C1—H1A	109.0	O6—C11'—H11D	109.9
N2—C1—H1B	109.0	C12'—C11'—H11D	109.9
N3—C1—H1B	109.0	H11C—C11'—H11D	108.3
H1A—C1—H1B	107.8	C11'—C12'—H12D	109.5
N2—C2—N4	112.8 (2)	C11'—C12'—H12E	109.5
N2—C2—H2A	109.0	H12D—C12'—H12E	109.5
N4—C2—H2A	109.0	C11'—C12'—H12F	109.5
N2—C2—H2B	109.0	H12D—C12'—H12F	109.5
N4—C2—H2B	109.0	H12E—C12'—H12F	109.5
H2A—C2—H2B	107.8	N2—C13—C14	111.4 (3)
O1—C3—N5	126.8 (3)	N2—C13—H13A	109.4
O1—C3—N3	123.9 (3)	C14—C13—H13A	109.4
N5—C3—N3	109.2 (3)	N2—C13—H13B	109.4
O2—C4—N6	126.2 (3)	C14—C13—H13B	109.4
O2—C4—N4	125.1 (3)	H13A—C13—H13B	108.0
N6—C4—N4	108.7 (3)	C15—C14—C18	117.0 (3)
N4—C5—N3	112.6 (2)	C15—C14—C13	123.9 (3)
N4—C5—C6	111.1 (3)	C18—C14—C13	119.1 (3)
N3—C5—C6	109.2 (2)	C14—C15—C16	119.8 (3)
N4—C5—C9	103.5 (2)	C14—C15—H15	120.1
N3—C5—C9	103.6 (2)	C16—C15—H15	120.1
C6—C5—C9	116.7 (3)	C17—C16—C15	118.8 (3)
O3—C6—O4	126.4 (3)	C17—C16—H16	120.6
O3—C6—C5	123.3 (3)	C15—C16—H16	120.6
O4—C6—C5	110.2 (3)	N1—C17—C16	123.3 (4)
C8'—C7—O4	116.6 (11)	N1—C17—H17	118.4
C8'—C7—C8	62.8 (12)	C16—C17—H17	118.4
O4—C7—C8	106.1 (5)	N1—C18—C14	124.2 (3)
C8'—C7—H7A	132.4	N1—C18—H18	117.9
O4—C7—H7A	110.5	C14—C18—H18	117.9
C8—C7—H7A	110.5	C19—C20—Cl2	127 (2)
C8'—C7—H7B	48.3	C19—C20—H20A	105.6
O4—C7—H7B	110.5	Cl2—C20—H20A	105.6
C8—C7—H7B	110.5	C19—C20—H20B	105.6
H7A—C7—H7B	108.7	Cl2—C20—H20B	105.6
C8'—C7—H7C	98.6	H20A—C20—H20B	106.1
O4—C7—H7C	109.7	C20'—C19'—Cl1'	111.3 (12)
C8—C7—H7C	41.6	C20'—C19'—H19C	109.4
H7A—C7—H7C	70.7	Cl1'—C19'—H19C	109.4

H7B—C7—H7C	136.7	C20'—C19'—H19D	109.4
C8'—C7—H7D	113.7	Cl1'—C19'—H19D	109.4
O4—C7—H7D	109.6	H19C—C19'—H19D	108.0
C8—C7—H7D	140.2	C19'—C20'—Cl2'	96.2 (12)
H7A—C7—H7D	39.5	C19'—C20'—H20C	112.5
H7B—C7—H7D	72.6	Cl2'—C20'—H20C	112.5
H7C—C7—H7D	107.9	C19'—C20'—H20D	112.5
C7—C8—H7C	39.1	Cl2'—C20'—H20D	112.5
C7—C8—H8A	109.5	H20C—C20'—H20D	110.0
H7C—C8—H8A	72.8	C18—N1—C17	116.9 (3)
C7—C8—H8B	109.5	C2—N2—C1	109.4 (3)
H7C—C8—H8B	110.5	C2—N2—C13	113.4 (3)
C7—C8—H8C	109.5	C1—N2—C13	113.2 (2)
H7C—C8—H8C	136.2	C3—N3—C5	110.6 (2)
C7—C8'—H8'1	109.5	C3—N3—C1	122.5 (3)
C7—C8'—H8'2	109.5	C5—N3—C1	116.8 (2)
H8'1—C8'—H8'2	109.5	C4—N4—C5	111.7 (2)
C7—C8'—H8'3	109.5	C4—N4—C2	123.0 (3)
H8'1—C8'—H8'3	109.5	C5—N4—C2	116.0 (2)
H8'2—C8'—H8'3	109.5	C3—N5—C9	113.7 (2)
N6—C9—N5	114.6 (3)	C3—N5—H5	123.2
N6—C9—C10	109.8 (3)	C9—N5—H5	123.2
N5—C9—C10	110.1 (3)	C4—N6—C9	113.8 (3)
N6—C9—C5	102.1 (2)	C4—N6—H6	123.1
N5—C9—C5	102.1 (2)	C9—N6—H6	123.1
C10—C9—C5	118.0 (3)	C6—O4—C7	118.0 (3)
O5—C10—O6	125.4 (3)	C10—O6—C11	118.3 (5)
O5—C10—C9	123.6 (3)	C10—O6—C11'	111.6 (16)
O6—C10—C9	110.8 (3)	C11—O6—C11'	15 (3)
O6—C11—C12	109.3 (9)		
N4—C5—C6—O3	6.1 (5)	N4—C5—N3—C3	107.4 (3)
N3—C5—C6—O3	−118.7 (4)	C6—C5—N3—C3	−128.6 (3)
C9—C5—C6—O3	124.4 (4)	C9—C5—N3—C3	−3.7 (3)
N4—C5—C6—O4	−174.5 (3)	N4—C5—N3—C1	−38.9 (3)
N3—C5—C6—O4	60.7 (3)	C6—C5—N3—C1	85.0 (3)
C9—C5—C6—O4	−56.3 (4)	C9—C5—N3—C1	−150.0 (3)
N4—C5—C9—N6	−0.6 (3)	N2—C1—N3—C3	−94.8 (3)
N3—C5—C9—N6	117.1 (2)	N2—C1—N3—C5	47.3 (3)
C6—C5—C9—N6	−122.9 (3)	O2—C4—N4—C5	174.7 (3)
N4—C5—C9—N5	−119.4 (3)	N6—C4—N4—C5	−5.2 (4)
N3—C5—C9—N5	−1.7 (3)	O2—C4—N4—C2	29.4 (5)
C6—C5—C9—N5	118.3 (3)	N6—C4—N4—C2	−150.5 (3)
N4—C5—C9—C10	119.8 (3)	N3—C5—N4—C4	−107.7 (3)
N3—C5—C9—C10	−122.5 (3)	C6—C5—N4—C4	129.4 (3)
C6—C5—C9—C10	−2.5 (4)	C9—C5—N4—C4	3.5 (3)
N6—C9—C10—O5	−118.2 (4)	N3—C5—N4—C2	40.1 (3)
N5—C9—C10—O5	8.9 (5)	C6—C5—N4—C2	−82.7 (3)
C5—C9—C10—O5	125.5 (4)	C9—C5—N4—C2	151.3 (3)
N6—C9—C10—O6	58.3 (4)	N2—C2—N4—C4	93.3 (4)

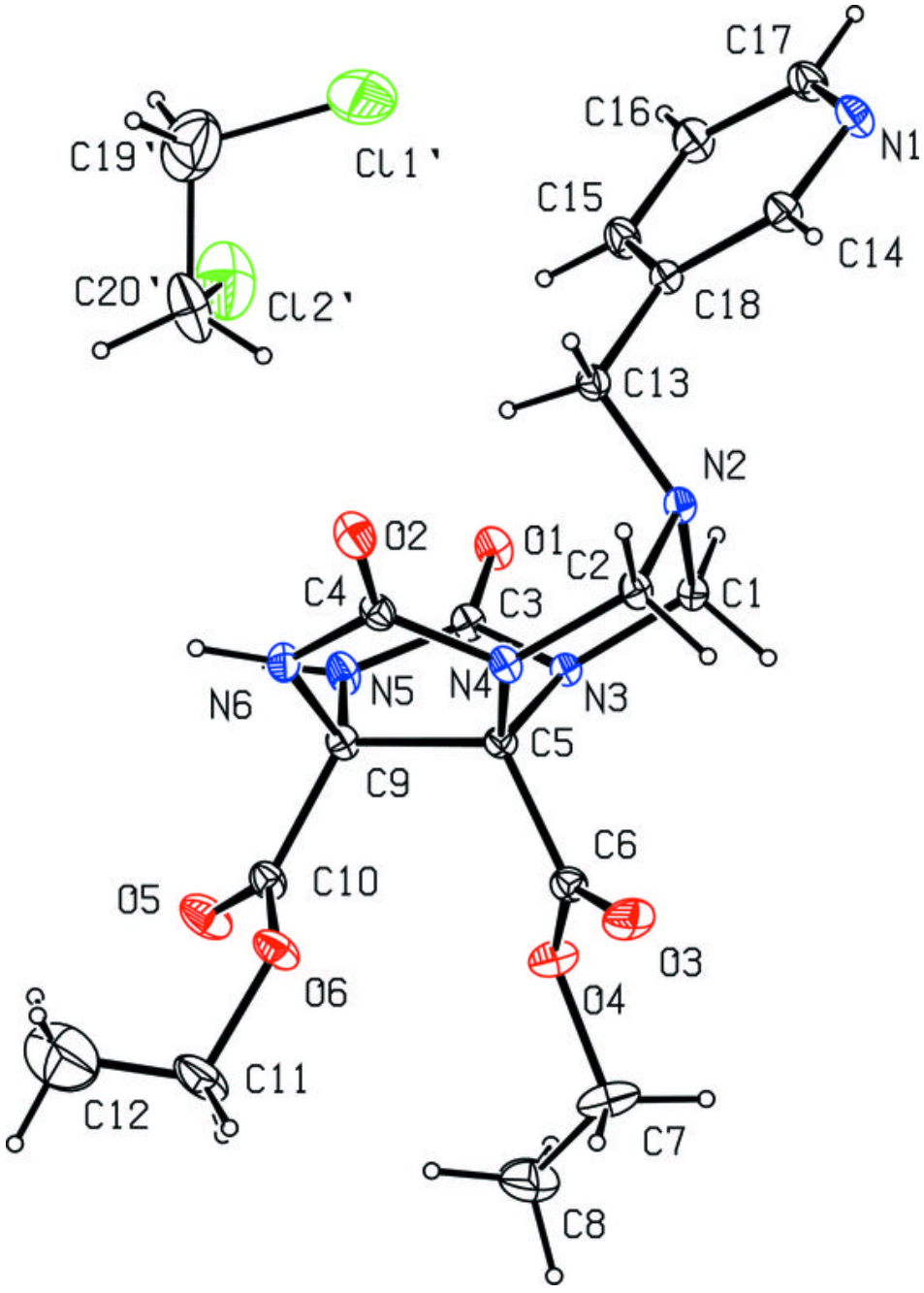
N5—C9—C10—O6	−174.6 (3)	N2—C2—N4—C5	−50.7 (4)
C5—C9—C10—O6	−58.0 (4)	O1—C3—N5—C9	172.1 (3)
N2—C13—C14—C15	−114.8 (4)	N3—C3—N5—C9	−9.6 (4)
N2—C13—C14—C18	66.8 (4)	N6—C9—N5—C3	−102.7 (3)
C18—C14—C15—C16	0.1 (5)	C10—C9—N5—C3	133.0 (3)
C13—C14—C15—C16	−178.3 (3)	C5—C9—N5—C3	6.8 (4)
C14—C15—C16—C17	−0.6 (6)	O2—C4—N6—C9	−175.0 (4)
C15—C16—C17—N1	0.0 (7)	N4—C4—N6—C9	4.9 (4)
C15—C14—C18—N1	1.0 (5)	N5—C9—N6—C4	107.0 (3)
C13—C14—C18—N1	179.6 (3)	C10—C9—N6—C4	−128.6 (3)
Cl1—C19—C20—Cl2	−53 (5)	C5—C9—N6—C4	−2.6 (3)
Cl1'—C19'—C20'—Cl2'	82.1 (15)	O3—C6—O4—C7	−2.2 (6)
C14—C18—N1—C17	−1.6 (6)	C5—C6—O4—C7	178.5 (3)
C16—C17—N1—C18	1.1 (6)	C8'—C7—O4—C6	−89.6 (15)
N4—C2—N2—C1	55.9 (3)	C8—C7—O4—C6	−156.8 (5)
N4—C2—N2—C13	−71.5 (3)	O5—C10—O6—C11	−1.3 (8)
N3—C1—N2—C2	−54.3 (3)	C9—C10—O6—C11	−177.7 (6)
N3—C1—N2—C13	73.2 (3)	O5—C10—O6—C11'	14 (2)
C14—C13—N2—C2	−162.3 (3)	C9—C10—O6—C11'	−163 (2)
C14—C13—N2—C1	72.3 (3)	C12—C11—O6—C10	73.4 (10)
O1—C3—N3—C5	−173.6 (3)	C12—C11—O6—C11'	6(5)
N5—C3—N3—C5	8.1 (4)	C12'—C11'—O6—C10	−35 (4)
O1—C3—N3—C1	−29.5 (5)	C12'—C11'—O6—C11	84 (6)
N5—C3—N3—C1	152.1 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1A···O1	0.97	2.56	2.918 (4)	102
C19'—H19D···O2^{i}	0.97	2.47	3.166 (17)	128
C1—H1B···O2^{ii}	0.97	2.42	3.305 (4)	152
N6—H6···N1^{iii}	0.86	2.07	2.892 (4)	160
N5—H5···O1^{iv}	0.86	2.03	2.862 (3)	162

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

Fig. 1


C17
C16
N1
C15
C14
C18
C19'
Cl1'
C20'
Cl2'
C13
N2
O2
O1
C2
C4
C3
C1
N6
N5
N4
N3
C5
C9
C6
O5
C10
O3
O6
O4
C11
C12
C7
C8