organic compounds

(11bR,11cS)-rel-Diethyl 6-bromo-9-methoxy-4,11-dioxo-5,10-dihydro-1H,3H,4H,11H-2-oxa-3a,4a,10a,11a-tetraazabenz[f]indeno[2,1,7-ija]-azulene-11b,11c-dicarboxylate

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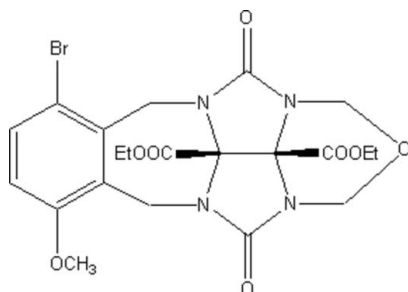
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The title compound, $C_{21}H_{23}BrN_4O_8$, is a glycoluril derivative in which the oxadiazinane six-membered ring displays a normal chair conformation. The methoxy and Br groups attached to the phenyl ring are disordered in a 0.847 (4):0.153 (4) ratio and the pendant ethoxy group is disordered over two positions in a 0.531 (14):0.469 (14) ratio. Intermolecular C—H···O interactions may be effective in the stabilization of the crystal structure.

Related literature

For a related structure, see: Cao *et al.* (2006). For background, see: Branda *et al.* (1995; Elemans *et al.* (1999); Isaac & Witt (2002); Cao *et al.* (2006); Wu *et al.* (2002).

Experimental

Crystal data

$C_{21}H_{23}BrN_4O_8$	$b = 11.6031$ (8) Å
$M_r = 539.34$	$c = 21.4749$ (15) Å
Monoclinic, $P2_1/n$	$\beta = 95.387$ (1)°
$a = 9.2246$ (6) Å	$V = 2288.4$ (3) Å^3

$Z = 4$	$T = 292$ (2) K
Mo $K\alpha$ radiation	$0.15 \times 0.10 \times 0.10$ mm
$\mu = 1.85$ mm^{-1}	

Data collection

Bruker SMART CCD diffractometer	18458 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	4236 independent reflections
$T_{min} = 0.708$, $T_{max} = 0.837$	2704 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.056$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$	5 restraints
$wR(F^2) = 0.143$	H-atom parameters constrained
$S = 1.08$	$\Delta\rho_{max} = 0.44$ e Å^{-3}
4236 reflections	$\Delta\rho_{min} = -0.29$ e Å^{-3}
369 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O6^i	0.93	2.56	3.437 (5)	159
C4—H4···O5^{ii}	0.93	2.43	3.261 (5)	149
C19—H19A···O4^{iii}	0.96	2.47	3.41 (3)	167

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

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: *SHELXTL* (Bruker, 2001).

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supplementary materials

(11b*R*,11c*S*)-*rel*-Diethyl 6-bromo-9-methoxy-4,11-dioxo-5,10-dihydro-1*H*,3*H*,4*H*,11*H*-2-oxa-3a,4a,10a,11a-tetraazabenz[*f*]indeno[2,1,7-*ija*]azulene-11b,11c-dicarboxylate

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Comment

Glycoluril has also become an important building block in supramolecular chemistry (Branda *et al.*, 1995; Elemans *et al.*, 1999; Isaac & Witt, 2002). The widespread interest in glycolurils has led to a variety of crystal structures reported for a number of its derivatives. We report here the structure of the title glycoluril derivative, (I) (Fig. 1), which is an important intermediate for the preparation glycoluril receptors (Wu *et al.*, 2002). The bond lengths and angles in (I) present no unusual features and are similar to those found in similar compounds (Cao *et al.*, 2006).

The C_3N_2O oxadiazinane six-membered ring in (I) displays a normal chair conformation. Weak intermolecular C—H⋯O hydrogen bonds help to establish the packing (Fig. 2).

Experimental

The title compound was synthesized according to the procedure reported by Wu *et al.* (2002). Colourless blocks of (I) were obtained by slow evaporation of a dichloromethane solution at 283 K.

Refinement

The methoxy and Br groups are disordered in a 0.847 (4):0.153 (4) ratio and the pendant ethoxy group is disordered over two positions in a 0.531 (14):0.469 (14) ratio. The H atoms were placed at calculated positions (C—H = 0.93–0.97 Å) and treated as riding atoms with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).
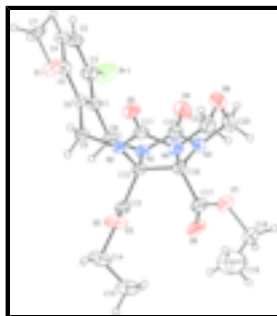
Figures

Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 30% probability level. The minor disorder components are omitted for clarity.
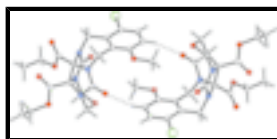

Fig. 2. A packing diagram for (I). C—H⋯O hydrogen bonds are shown as dashed lines.

supplementary materials

(11bR,11cS)-*rel*-Diethyl 6-bromo-9-methoxy-4,11-dioxo-5,10-dihydro- 1H,3*H*,4H,11*H*-2-Oxa-3a,4a,10a,11a-tetraazabenz[*f*]indeno[2,1,7-ija]azulene- 11*b*,11*c*-dicarboxylate

Crystal data

$C_{21}H_{23}BrN_4O_8$	$F_{000} = 1104$
$M_r = 539.34$	$D_x = 1.565$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 2981 reflections
$a = 9.2246$ (6) Å	$\theta = 2.3–19.8º$
$b = 11.6031$ (8) Å	$\mu = 1.85$ mm^{-1}
$c = 21.4749$ (15) Å	$T = 292$ (2) K
$\beta = 95.387$ (1)º	Block, colourless
$V = 2288.4$ (3) Å^3	$0.15 \times 0.10 \times 0.10$ mm
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	4236 independent reflections
Radiation source: fine-focus sealed tube	2704 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.056$
$T = 292(2)$ K	$\theta_{max} = 25.5º$
ω scans	$\theta_{min} = 1.9º$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$h = -11\rightarrow11$
$T_{min} = 0.708$, $T_{max} = 0.837$	$k = -13\rightarrow14$
18458 measured reflections	$l = -25\rightarrow25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.059$	H-atom parameters constrained
$wR(F^2) = 0.143$	$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2 + 3.1185P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
4236 reflections	$\Delta\rho_{max} = 0.44$ e Å^{-3}
369 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
5 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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Br1	0.57294 (17)	0.87084 (9)	1.04001 (5)	0.0952 (4)	0.847 (4)
Br1'	0.8610 (16)	0.3756 (17)	1.0205 (8)	0.089 (5)	0.153 (4)
C1	0.6041 (4)	0.6566 (4)	0.97427 (18)	0.0435 (10)	
C2	0.6485 (6)	0.7233 (5)	1.0268 (2)	0.0609 (14)	
C3	0.7521 (6)	0.6827 (5)	1.0725 (2)	0.0666 (14)	
H3	0.7813	0.7290	1.1067	0.080*	
C4	0.8109 (5)	0.5772 (5)	1.0680 (2)	0.0599 (13)	
H4	0.8812	0.5514	1.0987	0.072*	
C5	0.7665 (5)	0.5063 (4)	1.01703 (19)	0.0513 (11)	
C6	0.6624 (4)	0.5463 (4)	0.97033 (17)	0.0442 (10)	
C7	0.9300 (8)	0.3499 (7)	1.0566 (4)	0.074 (3)	0.794 (11)
H7A	1.0120	0.4011	1.0612	0.111*	0.794 (11)
H7B	0.9607	0.2757	1.0431	0.111*	0.794 (11)
H7C	0.8899	0.3421	1.0961	0.111*	0.794 (11)
O1	0.8177 (16)	0.3973 (15)	1.0096 (8)	0.066 (4)	0.794 (11)
O1'	0.575 (5)	0.801 (4)	1.0325 (19)	0.084 (18)	0.206 (11)
C7'	0.564 (5)	0.904 (4)	1.071 (2)	0.122 (16)	0.206 (11)
H7'1	0.6253	0.8952	1.1099	0.183*	0.206 (11)
H7'2	0.4650	0.9144	1.0806	0.183*	0.206 (11)
H7'3	0.5949	0.9704	1.0494	0.183*	0.206 (11)
C8	0.4914 (4)	0.7006 (4)	0.92392 (18)	0.0469 (10)	
H8A	0.4064	0.6511	0.9224	0.056*	
H8B	0.4614	0.7773	0.9352	0.056*	
C9	0.6138 (4)	0.4681 (3)	0.91544 (17)	0.0422 (10)	
H9A	0.6630	0.3945	0.9214	0.051*	
H9B	0.5101	0.4541	0.9152	0.051*	
C10	0.6426 (5)	0.7862 (4)	0.8483 (2)	0.0528 (11)	
C11	0.7733 (5)	0.5001 (4)	0.83120 (18)	0.0447 (10)	
C12	0.5534 (4)	0.6036 (3)	0.82418 (17)	0.0394 (9)	
C13	0.4013 (4)	0.5554 (4)	0.80176 (18)	0.0428 (10)	
C14	0.2821 (6)	0.3967 (5)	0.7486 (3)	0.0777 (16)	
H14A	0.2993	0.3142	0.7497	0.093*	
H14B	0.2063	0.4135	0.7756	0.093*	

C15	0.2343 (8)	0.4321 (7)	0.6847 (3)	0.128 (3)	
H15A	0.2148	0.5134	0.6839	0.192*	
H15B	0.1473	0.3910	0.6703	0.192*	
H15C	0.3094	0.4152	0.6580	0.192*	
C16	0.6491 (4)	0.6436 (3)	0.77189 (18)	0.0441 (10)	
C17	0.5664 (5)	0.6525 (4)	0.7062 (2)	0.0533 (12)	
O6	0.4468 (4)	0.6891 (3)	0.69821 (14)	0.0718 (10)	
O7	0.6602 (12)	0.6425 (10)	0.6639 (4)	0.054 (3)	0.531 (14)
C18	0.604 (2)	0.6630 (10)	0.5969 (6)	0.059 (4)	0.531 (14)
H18A	0.5293	0.7221	0.5941	0.071*	0.531 (14)
H18B	0.6823	0.6875	0.5728	0.071*	0.531 (14)
C19	0.543 (3)	0.5523 (17)	0.5732 (10)	0.160 (10)	0.531 (14)
H19A	0.6145	0.4927	0.5813	0.240*	0.531 (14)
H19B	0.5170	0.5581	0.5290	0.240*	0.531 (14)
H19C	0.4582	0.5341	0.5939	0.240*	0.531 (14)
O7'	0.6316 (18)	0.5954 (13)	0.6630 (5)	0.074 (5)	0.469 (14)
C18'	0.560 (2)	0.615 (2)	0.5979 (9)	0.106 (10)	0.469 (14)
H18C	0.4857	0.5576	0.5871	0.127*	0.469 (14)
H18D	0.5145	0.6910	0.5947	0.127*	0.469 (14)
C19'	0.6783 (18)	0.6071 (13)	0.5562 (5)	0.095 (6)	0.469 (14)
H19D	0.7457	0.6694	0.5650	0.143*	0.469 (14)
H19E	0.6381	0.6116	0.5134	0.143*	0.469 (14)
H19F	0.7282	0.5350	0.5632	0.143*	0.469 (14)
C20	0.8337 (5)	0.7980 (5)	0.7755 (3)	0.0734 (15)	
H20A	0.8293	0.8122	0.7309	0.088*	
H20B	0.8584	0.8699	0.7969	0.088*	
C21	0.9111 (5)	0.6116 (5)	0.7606 (2)	0.0697 (15)	
H21A	0.9882	0.5562	0.7713	0.084*	
H21B	0.9062	0.6254	0.7159	0.084*	
N1	0.5433 (3)	0.7050 (3)	0.86198 (14)	0.0420 (8)	
N2	0.6426 (3)	0.5141 (3)	0.85540 (14)	0.0390 (8)	
N3	0.6924 (4)	0.7591 (3)	0.79149 (16)	0.0500 (9)	
N4	0.7724 (3)	0.5639 (3)	0.77641 (15)	0.0504 (9)	
O2	0.2900 (3)	0.5991 (3)	0.81306 (14)	0.0577 (8)	
O3	0.4160 (3)	0.4573 (3)	0.77150 (14)	0.0596 (9)	
O4	0.6769 (4)	0.8709 (3)	0.87933 (17)	0.0796 (11)	
O5	0.8721 (3)	0.4370 (3)	0.85125 (14)	0.0589 (8)	
O8	0.9437 (3)	0.7148 (4)	0.79252 (18)	0.0794 (11)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1399 (9)	0.0653 (6)	0.0747 (7)	0.0082 (6)	−0.0197 (6)	−0.0237 (5)
Br1'	0.088 (10)	0.088 (6)	0.095 (12)	0.012 (6)	0.031 (9)	0.012 (6)
C1	0.040 (2)	0.053 (3)	0.037 (2)	−0.007 (2)	−0.0008 (18)	0.0016 (18)
C2	0.069 (4)	0.059 (3)	0.053 (3)	−0.014 (3)	−0.006 (3)	−0.003 (2)
C3	0.071 (3)	0.081 (4)	0.045 (3)	−0.029 (3)	−0.013 (2)	−0.007 (3)
C4	0.049 (3)	0.088 (4)	0.040 (3)	−0.016 (3)	−0.011 (2)	0.009 (2)

C5	0.046 (3)	0.064 (3)	0.043 (2)	−0.009 (2)	−0.001 (2)	0.013 (2)
C6	0.041 (2)	0.055 (3)	0.036 (2)	−0.009 (2)	−0.0036 (18)	0.0078 (18)
C7	0.064 (5)	0.096 (6)	0.057 (4)	0.022 (4)	−0.028 (4)	0.030 (4)
O1	0.073 (8)	0.076 (9)	0.047 (4)	0.008 (5)	−0.008 (5)	0.005 (4)
O1'	0.19 (4)	0.07 (5)	0.09 (4)	0.00 (4)	0.03 (3)	−0.01 (4)
C7'	0.12 (3)	0.12 (4)	0.12 (4)	0.00 (3)	0.01 (3)	0.00 (3)
C8	0.043 (2)	0.054 (3)	0.044 (2)	0.001 (2)	0.0007 (19)	−0.001 (2)
C9	0.038 (2)	0.044 (2)	0.042 (2)	−0.0020 (19)	−0.0067 (18)	0.0040 (18)
C10	0.058 (3)	0.043 (3)	0.054 (3)	−0.002 (2)	−0.009 (2)	0.008 (2)
C11	0.042 (3)	0.047 (2)	0.042 (2)	−0.001 (2)	−0.008 (2)	−0.0007 (19)
C12	0.036 (2)	0.045 (2)	0.036 (2)	0.0023 (18)	−0.0062 (17)	0.0029 (17)
C13	0.034 (2)	0.050 (3)	0.043 (2)	0.002 (2)	−0.0029 (19)	0.0007 (19)
C14	0.059 (3)	0.076 (4)	0.094 (4)	−0.016 (3)	−0.010 (3)	−0.024 (3)
C15	0.110 (5)	0.171 (7)	0.094 (5)	−0.063 (5)	−0.040 (4)	0.001 (5)
C16	0.043 (2)	0.048 (2)	0.041 (2)	0.004 (2)	−0.0023 (18)	0.0087 (18)
C17	0.045 (3)	0.071 (3)	0.042 (2)	0.009 (2)	−0.002 (2)	0.004 (2)
O6	0.051 (2)	0.112 (3)	0.0494 (19)	0.018 (2)	−0.0089 (16)	0.0163 (18)
O7	0.062 (5)	0.059 (7)	0.041 (5)	0.010 (5)	0.000 (4)	0.004 (4)
C18	0.067 (9)	0.064 (8)	0.044 (6)	0.004 (7)	0.000 (6)	0.006 (5)
C19	0.154 (19)	0.16 (2)	0.16 (2)	−0.004 (16)	−0.022 (18)	−0.012 (15)
O7'	0.083 (9)	0.096 (11)	0.041 (5)	0.027 (8)	−0.009 (5)	−0.018 (6)
C18'	0.12 (2)	0.11 (2)	0.086 (14)	0.007 (18)	−0.001 (14)	0.001 (15)
C19'	0.117 (13)	0.107 (12)	0.063 (8)	0.029 (10)	0.018 (8)	0.018 (8)
C20	0.064 (3)	0.076 (4)	0.080 (4)	−0.018 (3)	0.006 (3)	0.030 (3)
C21	0.040 (3)	0.101 (4)	0.069 (3)	0.008 (3)	0.011 (2)	0.029 (3)
N1	0.042 (2)	0.0398 (18)	0.0427 (19)	0.0002 (16)	−0.0017 (15)	0.0024 (15)
N2	0.0350 (19)	0.0446 (19)	0.0358 (17)	0.0039 (15)	−0.0046 (14)	0.0040 (14)
N3	0.045 (2)	0.054 (2)	0.050 (2)	−0.0035 (17)	0.0019 (17)	0.0092 (17)
N4	0.0330 (19)	0.067 (2)	0.051 (2)	0.0159 (18)	0.0033 (16)	0.0132 (18)
O2	0.0376 (18)	0.073 (2)	0.0610 (19)	0.0082 (16)	−0.0034 (15)	−0.0094 (16)
O3	0.0381 (17)	0.064 (2)	0.074 (2)	−0.0034 (15)	−0.0060 (15)	−0.0232 (17)
O4	0.105 (3)	0.054 (2)	0.079 (2)	−0.024 (2)	0.006 (2)	−0.0073 (18)
O5	0.0460 (18)	0.068 (2)	0.0600 (19)	0.0149 (16)	−0.0078 (15)	0.0149 (16)
O8	0.046 (2)	0.097 (3)	0.094 (3)	−0.015 (2)	−0.0009 (18)	0.031 (2)

Geometric parameters (Å, °)

Br1—C2	1.880 (6)	C13—O2	1.190 (5)
Br1'—C5	1.748 (16)	C13—O3	1.324 (5)
C1—C6	1.395 (6)	C14—C15	1.460 (8)
C1—C2	1.397 (6)	C14—O3	1.466 (5)
C1—C8	1.516 (5)	C14—H14A	0.9700
C2—O1'	1.14 (4)	C14—H14B	0.9700
C2—C3	1.386 (7)	C15—H15A	0.9600
C3—C4	1.347 (7)	C15—H15B	0.9600
C3—H3	0.9300	C15—H15C	0.9600
C4—C5	1.399 (6)	C16—N3	1.449 (5)
C4—H4	0.9300	C16—N4	1.462 (5)
C5—O1	1.365 (18)	C16—C17	1.544 (6)

C5—C6	1.401 (5)	C17—O6	1.180 (5)
C6—C9	1.521 (5)	C17—O7	1.317 (8)
C7—O1	1.482 (10)	C17—O7'	1.328 (8)
C7—H7A	0.9600	O7—C18	1.501 (10)
C7—H7B	0.9600	C18—C19	1.472 (16)
C7—H7C	0.9600	C18—H18A	0.9700
O1'—C7'	1.474 (14)	C18—H18B	0.9700
C7'—H7'1	0.9600	C19—H19A	0.9600
C7'—H7'2	0.9600	C19—H19B	0.9600
C7'—H7'3	0.9600	C19—H19C	0.9600
C8—N1	1.456 (5)	O7'—C18'	1.509 (11)
C8—H8A	0.9700	C18'—C19'	1.481 (16)
C8—H8B	0.9700	C18'—H18C	0.9700
C9—N2	1.443 (5)	C18'—H18D	0.9700
C9—H9A	0.9700	C19'—H19D	0.9600
C9—H9B	0.9700	C19'—H19E	0.9600
C10—O4	1.212 (5)	C19'—H19F	0.9600
C10—N1	1.365 (5)	C20—O8	1.423 (6)
C10—N3	1.380 (6)	C20—N3	1.451 (6)
C11—O5	1.216 (5)	C20—H20A	0.9700
C11—N2	1.367 (5)	C20—H20B	0.9700
C11—N4	1.390 (5)	C21—O8	1.399 (6)
C12—N1	1.437 (5)	C21—N4	1.463 (6)
C12—N2	1.450 (5)	C21—H21A	0.9700
C12—C13	1.545 (5)	C21—H21B	0.9700
C12—C16	1.563 (6)		
C6—C1—C2	118.1 (4)	C14—C15—H15A	109.5
C6—C1—C8	120.6 (3)	C14—C15—H15B	109.5
C2—C1—C8	121.3 (4)	H15A—C15—H15B	109.5
O1'—C2—C3	125 (2)	C14—C15—H15C	109.5
O1'—C2—C1	113 (2)	H15A—C15—H15C	109.5
C3—C2—C1	121.0 (5)	H15B—C15—H15C	109.5
C3—C2—Br1	116.3 (4)	N3—C16—N4	112.0 (3)
C1—C2—Br1	122.8 (4)	N3—C16—C17	107.6 (3)
C4—C3—C2	120.9 (5)	N4—C16—C17	114.7 (3)
C4—C3—H3	119.6	N3—C16—C12	103.0 (3)
C2—C3—H3	119.6	N4—C16—C12	104.6 (3)
C3—C4—C5	120.0 (4)	C17—C16—C12	114.4 (3)
C3—C4—H4	120.0	O6—C17—O7	126.7 (7)
C5—C4—H4	120.0	O6—C17—O7'	123.9 (8)
O1—C5—C4	123.8 (7)	O6—C17—C16	122.1 (4)
O1—C5—C6	116.4 (8)	O7—C17—C16	108.9 (7)
C4—C5—C6	119.8 (5)	O7'—C17—C16	112.8 (8)
C4—C5—Br1'	111.6 (7)	C17—O7—C18	117.2 (12)
C6—C5—Br1'	128.6 (7)	C19—C18—O7	106.2 (11)
C1—C6—C5	120.2 (4)	C19—C18—H18A	110.5
C1—C6—C9	120.5 (3)	O7—C18—H18A	110.5
C5—C6—C9	119.3 (4)	C19—C18—H18B	110.5
C5—O1—C7	119.5 (15)	O7—C18—H18B	110.5

C2—O1'—C7'	141 (5)	H18A—C18—H18B	108.7
O1'—C7'—H7'1	109.5	C17—O7'—C18'	112.4 (13)
O1'—C7'—H7'2	109.5	C19'—C18'—O7'	105.3 (12)
H7'1—C7'—H7'2	108	C19'—C18'—H18C	110.7
O1'—C7'—H7'3	109.5	O7'—C18'—H18C	110.7
H7'1—C7'—H7'3	109.5	C19'—C18'—H18D	110.7
H7'2—C7'—H7'3	109.5	O7'—C18'—H18D	110.7
N1—C8—C1	113.6 (3)	H18C—C18'—H18D	108.8
N1—C8—H8A	108.8	C18'—C19'—H19D	109.5
C1—C8—H8A	108.8	C18'—C19'—H19E	109.5
N1—C8—H8B	108.8	H19D—C19'—H19E	109.5
C1—C8—H8B	108.8	C18'—C19'—H19F	109.5
H8A—C8—H8B	107.7	H19D—C19'—H19F	109.5
N2—C9—C6	114.0 (3)	H19E—C19'—H19F	109.5
N2—C9—H9A	108.8	O8—C20—N3	111.3 (4)
C6—C9—H9A	108.8	O8—C20—H20A	109.4
N2—C9—H9B	108.8	N3—C20—H20A	109.4
C6—C9—H9B	108.8	O8—C20—H20B	109.4
H9A—C9—H9B	107.7	N3—C20—H20B	109.4
O4—C10—N1	126.0 (5)	H20A—C20—H20B	108.0
O4—C10—N3	125.4 (4)	O8—C21—N4	111.3 (4)
N1—C10—N3	108.6 (4)	O8—C21—H21A	109.4
O5—C11—N2	126.6 (4)	N4—C21—H21A	109.4
O5—C11—N4	124.4 (4)	O8—C21—H21B	109.4
N2—C11—N4	108.8 (3)	N4—C21—H21B	109.4
N1—C12—N2	112.9 (3)	H21A—C21—H21B	108.0
N1—C12—C13	111.5 (3)	C10—N1—C12	111.3 (3)
N2—C12—C13	110.1 (3)	C10—N1—C8	120.3 (3)
N1—C12—C16	103.5 (3)	C12—N1—C8	122.1 (3)
N2—C12—C16	102.2 (3)	C11—N2—C9	122.6 (3)
C13—C12—C16	116.2 (3)	C11—N2—C12	113.1 (3)
O2—C13—O3	126.7 (4)	C9—N2—C12	122.3 (3)
O2—C13—C12	123.9 (4)	C10—N3—C16	111.2 (3)
O3—C13—C12	109.4 (3)	C10—N3—C20	121.3 (4)
C15—C14—O3	110.5 (5)	C16—N3—C20	116.6 (4)
C15—C14—H14A	109.5	C11—N4—C16	109.6 (3)
O3—C14—H14A	109.5	C11—N4—C21	117.5 (3)
C15—C14—H14B	109.5	C16—N4—C21	115.9 (4)
O3—C14—H14B	109.5	C13—O3—C14	117.1 (4)
H14A—C14—H14B	108.1	C21—O8—C20	109.8 (4)
C6—C1—C2—O1'	−165 (3)	C16—C17—O7—C18	−172.9 (7)
C8—C1—C2—O1'	13 (3)	C17—O7—C18—C19	−86 (2)
C6—C1—C2—C3	2.5 (7)	O6—C17—O7'—C18'	18.8 (17)
C8—C1—C2—C3	−179.7 (4)	O7—C17—O7'—C18'	−87 (3)
C6—C1—C2—Br1	−177.3 (3)	C16—C17—O7'—C18'	−173.5 (11)
C8—C1—C2—Br1	0.5 (6)	C17—O7'—C18'—C19'	148.8 (19)
O1'—C2—C3—C4	165 (3)	O4—C10—N1—C12	−166.4 (4)
C1—C2—C3—C4	−1.0 (8)	N3—C10—N1—C12	16.7 (4)
Br1—C2—C3—C4	178.8 (4)	O4—C10—N1—C8	−13.7 (6)

supplementary materials

C2—C3—C4—C5	−0.8 (8)	N3—C10—N1—C8	169.4 (3)
C3—C4—C5—O1	−178.3 (10)	N2—C12—N1—C10	95.9 (4)
C3—C4—C5—C6	1.0 (7)	C13—C12—N1—C10	−139.4 (3)
C3—C4—C5—Br1'	178.6 (7)	C16—C12—N1—C10	−13.7 (4)
C2—C1—C6—C5	−2.3 (6)	N2—C12—N1—C8	−56.3 (5)
C8—C1—C6—C5	180.0 (4)	C13—C12—N1—C8	68.4 (4)
C2—C1—C6—C9	177.7 (4)	C16—C12—N1—C8	−165.9 (3)
C8—C1—C6—C9	−0.1 (6)	C1—C8—N1—C10	−71.8 (5)
O1—C5—C6—C1	179.9 (9)	C1—C8—N1—C12	77.9 (4)
C4—C5—C6—C1	0.5 (6)	O5—C11—N2—C9	10.5 (6)
Br1'—C5—C6—C1	−176.6 (8)	N4—C11—N2—C9	−173.8 (3)
O1—C5—C6—C9	−0.1 (10)	O5—C11—N2—C12	174.6 (4)
C4—C5—C6—C9	−179.4 (4)	N4—C11—N2—C12	−9.7 (4)
Br1'—C5—C6—C9	3.5 (9)	C6—C9—N2—C11	85.6 (4)
C4—C5—O1—C7	−2 (2)	C6—C9—N2—C12	−77.1 (4)
C6—C5—O1—C7	178.9 (11)	N1—C12—N2—C11	−108.3 (4)
Br1'—C5—O1—C7	12 (5)	C13—C12—N2—C11	126.3 (3)
C3—C2—O1'—C7'	19 (8)	C16—C12—N2—C11	2.2 (4)
C1—C2—O1'—C7'	−174 (6)	N1—C12—N2—C9	55.9 (5)
Br1—C2—O1'—C7'	−40 (6)	C13—C12—N2—C9	−69.5 (4)
C6—C1—C8—N1	−60.5 (5)	C16—C12—N2—C9	166.4 (3)
C2—C1—C8—N1	121.8 (4)	O4—C10—N3—C16	170.7 (4)
C1—C6—C9—N2	60.5 (5)	N1—C10—N3—C16	−12.4 (4)
C5—C6—C9—N2	−119.5 (4)	O4—C10—N3—C20	27.7 (7)
N1—C12—C13—O2	2.0 (5)	N1—C10—N3—C20	−155.4 (4)
N2—C12—C13—O2	128.2 (4)	N4—C16—N3—C10	−108.3 (4)
C16—C12—C13—O2	−116.3 (4)	C17—C16—N3—C10	124.8 (4)
N1—C12—C13—O3	−175.0 (3)	C12—C16—N3—C10	3.6 (4)
N2—C12—C13—O3	−48.8 (4)	N4—C16—N3—C20	36.6 (5)
C16—C12—C13—O3	66.7 (4)	C17—C16—N3—C20	−90.3 (4)
N1—C12—C16—N3	5.8 (3)	C12—C16—N3—C20	148.5 (3)
N2—C12—C16—N3	−111.7 (3)	O8—C20—N3—C10	91.9 (5)
C13—C12—C16—N3	128.4 (3)	O8—C20—N3—C16	−49.1 (6)
N1—C12—C16—N4	123.0 (3)	O5—C11—N4—C16	−170.8 (4)
N2—C12—C16—N4	5.5 (4)	N2—C11—N4—C16	13.4 (4)
C13—C12—C16—N4	−114.3 (4)	O5—C11—N4—C21	−35.6 (6)
N1—C12—C16—C17	−110.7 (4)	N2—C11—N4—C21	148.6 (4)
N2—C12—C16—C17	131.9 (3)	N3—C16—N4—C11	99.3 (4)
C13—C12—C16—C17	12.0 (5)	C17—C16—N4—C11	−137.7 (4)
N3—C16—C17—O6	−73.9 (6)	C12—C16—N4—C11	−11.5 (4)
N4—C16—C17—O6	160.8 (4)	N3—C16—N4—C21	−36.7 (5)
C12—C16—C17—O6	39.9 (6)	C17—C16—N4—C21	86.3 (5)
N3—C16—C17—O7	90.1 (7)	C12—C16—N4—C21	−147.5 (4)
N4—C16—C17—O7	−35.2 (7)	O8—C21—N4—C11	−82.3 (5)
C12—C16—C17—O7	−156.1 (6)	O8—C21—N4—C16	50.1 (5)
N3—C16—C17—O7'	118.2 (9)	O2—C13—O3—C14	1.0 (6)
N4—C16—C17—O7'	−7.1 (10)	C12—C13—O3—C14	177.9 (4)
C12—C16—C17—O7'	−128.0 (9)	C15—C14—O3—C13	91.6 (6)
O6—C17—O7—C18	−9.8 (13)	N4—C21—O8—C20	−61.0 (5)

O7'—C17—O7—C18	84 (3)	N3—C20—O8—C21	60.6 (5)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O6[i]	0.93	2.56	3.437 (5)	159
C4—H4···O5[ii]	0.93	2.43	3.261 (5)	149
C19—H19A···O4[iii]	0.96	2.47	3.41 (3)	167

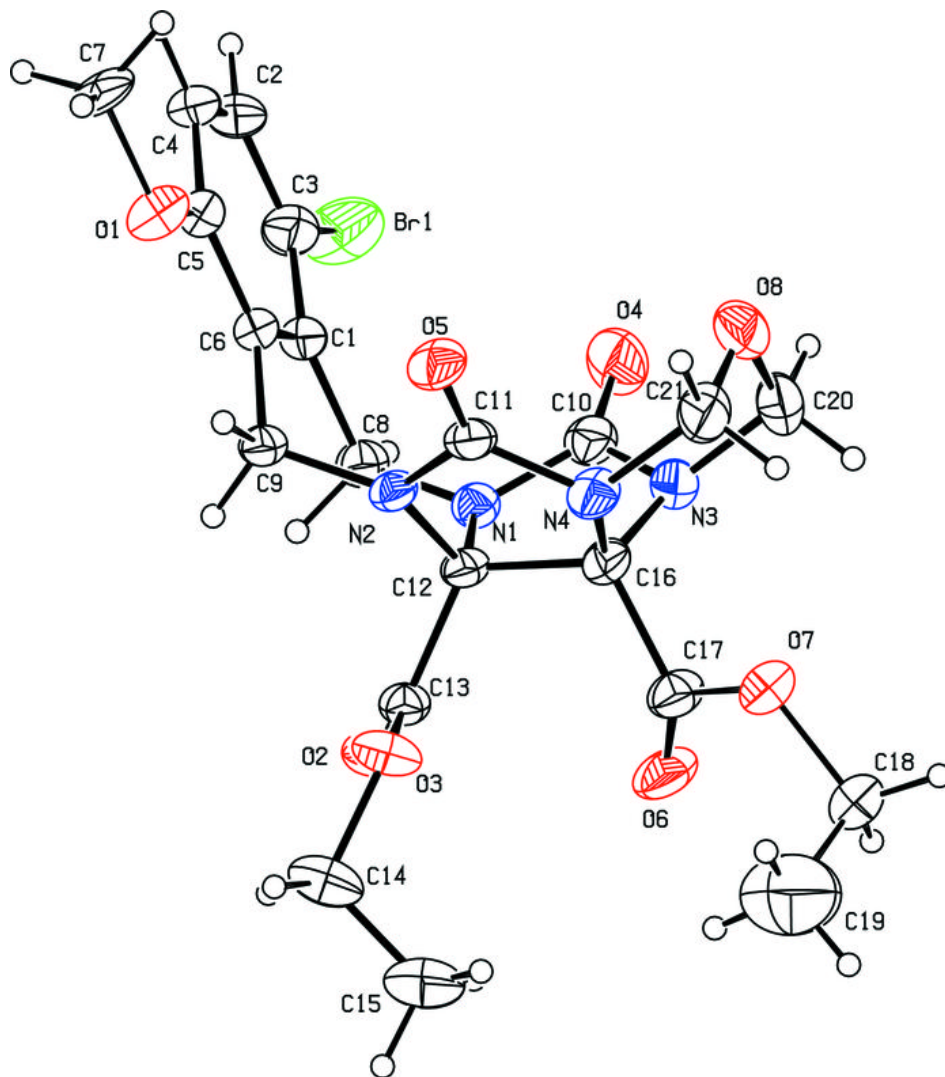Symmetry codes: (i) $x+1/2$, $-y+3/2$, $z+1/2$; (ii) $-x+2$, $-y+1$, $-z+2$; (iii) $-x+3/2$, $y-1/2$, $-z+3/2$.

Fig. 1

Fig. 2