

1-Benzylpiperazine-1,4-dium bis(perchlorate) monohydrate

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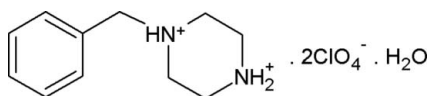
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.066; wR factor = 0.240; data-to-parameter ratio = 23.1.

In the title compound, $\text{C}_{11}\text{H}_{18}\text{N}_2^{2+} \cdot 2\text{ClO}_4^- \cdot \text{H}_2\text{O}$, one perchlorate anion is disordered over two orientations in a 0.66 (3):0.34 (3) ratio. Intermolecular $\text{O}-\text{H} \cdots \text{O}$, $\text{N}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds link the cations, anions and water molecules into ribbons extending along [100].

Related literature

For general background to the properties of perchlorate salts containing organic cations, see: Czarnecki *et al.* (1994); Czupinski *et al.* (2002, 2006). For related structures, see: Antolini *et al.* (1982); Place & Willett (1988).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{18}\text{N}_2^{2+} \cdot 2\text{ClO}_4^- \cdot \text{H}_2\text{O}$	$\gamma = 70.560$ (7)°
$M_r = 395.19$	$V = 838.05$ (12) Å ³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 8.6632$ (6) Å	Mo $K\alpha$ radiation
$b = 10.0197$ (8) Å	$\mu = 0.44$ mm ⁻¹
$c = 10.8831$ (7) Å	$T = 293$ K
$\alpha = 70.184$ (7)°	$0.53 \times 0.40 \times 0.25$ mm
$\beta = 83.946$ (6)°	

Data collection

Oxford Diffraction Xcalibur Atlas Gemini ultra diffractometer	32031 measured reflections
Absorption correction: analytical (<i>CrysAlis PRO</i> ; Oxford Diffraction, 2006)	5885 independent reflections
$T_{\min} = 0.832$, $T_{\max} = 0.907$	3882 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.045$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$	40 restraints
$wR(F^2) = 0.240$	H-atom parameters constrained
$S = 1.12$	$\Delta\rho_{\text{max}} = 1.04$ e Å ⁻³
5885 reflections	$\Delta\rho_{\text{min}} = -0.88$ e Å ⁻³
255 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O9}-\text{H91} \cdots \text{O4A}^i$	0.81	2.27	2.942 (11)	141
$\text{O9}-\text{H92} \cdots \text{O5}^{ii}$	0.82	2.06	2.869 (6)	170
$\text{N16}-\text{H161} \cdots \text{O8}$	0.90	2.15	2.964 (3)	151
$\text{N19}-\text{H191} \cdots \text{O9}^{iii}$	0.89	1.92	2.750 (4)	155
$\text{N19}-\text{H192} \cdots \text{O1A}^{iv}$	0.89	2.08	2.907 (10)	154
$\text{C17}-\text{H172} \cdots \text{O6}^v$	0.97	2.48	3.446 (5)	172
$\text{C20}-\text{H201} \cdots \text{O7}^{iv}$	0.95	2.49	3.406 (4)	160
$\text{C21}-\text{H212} \cdots \text{O3A}$	0.96	2.48	3.130 (15)	125

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2006); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2717).

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supplementary materials

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Comment

Chemists and physicists of the solid state have shown an increasing interest in the study of perchlorate salts containing organic cations in recent years owing to their great interesting properties such as ferroelectric and dielectric behaviours. (Czarnecki *et al.*, 1994; Czupinski *et al.*, 2002; Czupinski *et al.*, 2006). Here, we report the synthesis and the crystal structure of the title compound (I), $[\text{C}_{11}\text{H}_{18}\text{N}_2]^{2+} \cdot 2\text{ClO}_4^- \cdot \text{H}_2\text{O}$.

The crystal structure of (I) (Fig.1), contains two ClO_4^- anions, a 1-benzylpiperazine-1,4-dium dication and a water molecule. In its atomic arrangement, the ClO_4^- anions are associated per pair *via* $\text{O}—\text{H}\cdots\text{O}$ hydrogen bonds generated by a water molecule to form $[\text{Cl}_2\text{O}_8\text{H}_2\text{O}]^{2-}$ entities. The 1-benzylpiperazine-1,4-dium dications are associated to these entities and connected them through $\text{N}—\text{H}\cdots\text{O}$ and $\text{C}—\text{H}\cdots\text{O}$ hydrogen bonds, leading to the formation of three dimensional network. As expected, the ClO_4^- anion has typical tetrahedral geometry where the $\text{Cl}—\text{O}$ bond lengths and $\text{O}—\text{Cl}—\text{O}$ angles are not equal to one another but very with the environment around the O atoms. In the title compound, the $\text{Cl}—\text{O}$ bond lengths vary from 1.382 (12) Å to 1.437 (7) Å for Cl1O_4^- anion and from 1.374 (3) Å to 1.484 (4) Å for Cl2O_4^- anion. The $\text{O}—\text{Cl}—\text{O}$ angles range from 104.2 (14) ° to 119.3 (15) ° for the first anion and from 103.1 (2) ° to 118.4 (2) ° for the second one. These values clearly indicate that the coordination geometry of the Cl atom can be regarded as being a distorted tetrahedron. However, for Cl2O_4^- tetrahedron all the oxygen atoms are involved in hydrogen bonds, while only three oxygen atoms acts as acceptors of hydrogen bonds for the Cl1O_4^- tetrahedron.

Refinement

All H atoms were located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry ($\text{C}—\text{H}$ in the range 0.93–0.98, $\text{N}—\text{H}$ in the range 0.86–0.89 $\text{N}—\text{H}$ to 0.86 $\text{O}—\text{H}$ = 0.82 Å) and $U_{\text{iso}}(\text{H})$ (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints. The rotational disorder observed for one perchlorate anion (with Cl1) was modeled using two superimposed molecules with partial occupancies. The molecules were then refined with restraints on the $\text{Cl}—\text{O}$ bonds, $\text{O}—\text{Cl}—\text{O}$ angles and displacement parameters of the oxygen atoms

Figures

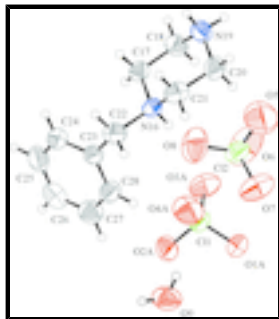


Fig. 1. View of (I), showing 50% probability displacement ellipsoids and arbitrary spheres for the H atoms. For the disordered perchlorate anion, only major part is shown.

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Crystal data

$C_{11}H_{18}N_2^{2+} \cdot 2ClO_4^- \cdot H_2O$

$M_r = 395.19$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 8.6632\ (6)\ \text{\AA}$

$b = 10.0197\ (8)\ \text{\AA}$

$c = 10.8831\ (7)\ \text{\AA}$

$\alpha = 70.184\ (7)^\circ$

$\beta = 83.946\ (6)^\circ$

$\gamma = 70.560\ (7)^\circ$

$V = 838.05\ (12)\ \text{\AA}^3$

$Z = 2$

$F(000) = 412.000$

$D_x = 1.566\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.7107\ \text{\AA}$

Cell parameters from 13879 reflections

$\theta = 3.5\text{--}32.9^\circ$

$\mu = 0.44\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Plate, colourless

$0.53 \times 0.40 \times 0.25\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur Atlas Gemini ultra diffractometer

Radiation source: Enhance (Mo) X-ray Source graphite

Detector resolution: $10.4685\ \text{pixels mm}^{-1}$

$\omega/2\theta$ scans

Absorption correction: analytical (*Crys.Alis PRO*; Oxford Diffraction, 2006)

$T_{\min} = 0.832$, $T_{\max} = 0.907$

32031 measured reflections

5885 independent reflections

3882 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 33.0^\circ$, $\theta_{\min} = 3.5^\circ$

$h = -13 \rightarrow 13$

$k = -15 \rightarrow 15$

$l = -15 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.066$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$wR(F^2) = 0.240$	$w = 1/[\sigma^2(F_o^2) + (0.15P)^2 + 0.05P]$
$S = 1.12$	where $P = (F_o^2 + 2F_c^2)/3$
5885 reflections	$(\Delta/\sigma)_{\max} < 0.001$
255 parameters	$\Delta\rho_{\max} = 1.04 \text{ e } \text{\AA}^{-3}$
40 restraints	$\Delta\rho_{\min} = -0.88 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ Extinction coefficient: 0.062 (11)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N16	0.2509 (2)	0.3896 (2)	0.78548 (16)	0.0377 (4)	
H161	0.1556	0.3721	0.7879	0.045*	
N19	0.1505 (3)	0.7088 (2)	0.6477 (2)	0.0507 (5)	
H191	0.2431	0.7311	0.6393	0.061*	
H192	0.0729	0.7939	0.6086	0.061*	
C17	0.2348 (3)	0.5002 (3)	0.8544 (2)	0.0419 (4)	
H171	0.2003	0.4603	0.9431	0.050*	
H172	0.3416	0.5114	0.8562	0.050*	
C18	0.1106 (3)	0.6496 (3)	0.7878 (2)	0.0490 (5)	
H181	0.1085	0.7216	0.8271	0.059*	
H182	0.0026	0.6393	0.7937	0.059*	
C20	0.1645 (4)	0.5982 (3)	0.5795 (2)	0.0560 (6)	
H201	0.1960	0.6349	0.4910	0.067*	
H202	0.0599	0.5802	0.5822	0.067*	
C21	0.2923 (4)	0.4523 (3)	0.6457 (2)	0.0502 (6)	
H211	0.3980	0.4667	0.6411	0.060*	
H212	0.3022	0.3805	0.6022	0.060*	
C22	0.3782 (3)	0.2403 (3)	0.8511 (3)	0.0480 (5)	
H221	0.3960	0.1769	0.7969	0.058*	
H222	0.4798	0.2588	0.8566	0.058*	
C23	0.3262 (3)	0.1631 (2)	0.9849 (2)	0.0423 (5)	
C24	0.3844 (4)	0.1675 (3)	1.0962 (3)	0.0563 (6)	
H241	0.4594	0.2183	1.0883	0.068*	
C25	0.3348 (4)	0.0959 (3)	1.2180 (3)	0.0658 (8)	
H251	0.3758	0.1010	1.2910	0.079*	
C26	0.2275 (4)	0.0170 (3)	1.2299 (3)	0.0629 (7)	
H261	0.1933	-0.0309	1.3131	0.075*	
C27	0.1682 (4)	0.0116 (3)	1.1200 (3)	0.0606 (7)	

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H271	0.0944	-0.0426	1.1291	0.073*	
C28	0.2175 (3)	0.0838 (3)	0.9975 (2)	0.0496 (5)	
H281	0.1822	0.0763	0.9236	0.059*	
Cl1	0.19840 (7)	0.07672 (7)	0.62088 (5)	0.0459 (2)	
O1A	0.0878 (13)	0.0586 (10)	0.5438 (9)	0.071 (2)	0.66 (3)
O2A	0.2349 (11)	-0.0512 (9)	0.7360 (6)	0.0630 (17)	0.66 (3)
O3A	0.1333 (19)	0.2126 (12)	0.6492 (15)	0.095 (4)	0.66 (3)
O4A	0.3513 (9)	0.0646 (13)	0.5527 (11)	0.086 (3)	0.66 (3)
O1B	0.122 (3)	0.082 (3)	0.510 (2)	0.101 (7)	0.34 (3)
O2B	0.190 (4)	-0.047 (2)	0.724 (2)	0.105 (6)	0.34 (3)
O3B	0.099 (2)	0.2007 (17)	0.660 (2)	0.064 (4)	0.34 (3)
O4B	0.3506 (16)	0.098 (2)	0.585 (2)	0.080 (3)	0.34 (3)
Cl2	-0.23663 (8)	0.44332 (8)	0.79535 (8)	0.0570 (2)	
O5	-0.2245 (5)	0.5724 (5)	0.6812 (5)	0.1421 (15)	
O6	-0.3698 (4)	0.5028 (5)	0.8630 (4)	0.1273 (13)	
O7	-0.2735 (4)	0.3526 (4)	0.7389 (3)	0.0951 (9)	
O8	-0.0873 (3)	0.3889 (4)	0.8630 (3)	0.0991 (10)	
O9	0.4548 (3)	-0.2670 (3)	0.5678 (3)	0.0757 (7)	
H91	0.4625	-0.1874	0.5194	0.114*	
H92	0.5408	-0.3200	0.6072	0.114*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N16	0.0336 (8)	0.0422 (9)	0.0420 (9)	-0.0174 (7)	0.0030 (7)	-0.0149 (7)
N19	0.0463 (11)	0.0441 (10)	0.0544 (11)	-0.0149 (8)	-0.0041 (9)	-0.0050 (8)
C17	0.0433 (11)	0.0430 (10)	0.0421 (10)	-0.0128 (9)	-0.0001 (8)	-0.0181 (9)
C18	0.0464 (12)	0.0436 (11)	0.0537 (12)	-0.0111 (9)	0.0049 (10)	-0.0162 (10)
C20	0.0661 (17)	0.0639 (15)	0.0410 (11)	-0.0303 (13)	-0.0058 (11)	-0.0096 (10)
C21	0.0606 (15)	0.0569 (14)	0.0429 (11)	-0.0285 (12)	0.0128 (10)	-0.0222 (10)
C22	0.0369 (10)	0.0437 (11)	0.0629 (14)	-0.0112 (9)	0.0065 (10)	-0.0200 (10)
C23	0.0362 (10)	0.0365 (9)	0.0527 (11)	-0.0076 (8)	-0.0031 (9)	-0.0154 (8)
C24	0.0560 (14)	0.0472 (12)	0.0656 (15)	-0.0145 (11)	-0.0176 (12)	-0.0147 (11)
C25	0.077 (2)	0.0540 (15)	0.0563 (15)	-0.0025 (14)	-0.0229 (14)	-0.0163 (12)
C26	0.0640 (17)	0.0517 (14)	0.0544 (14)	-0.0049 (12)	0.0031 (12)	-0.0081 (11)
C27	0.0546 (15)	0.0554 (14)	0.0694 (17)	-0.0229 (12)	0.0045 (13)	-0.0132 (13)
C28	0.0476 (12)	0.0539 (13)	0.0515 (12)	-0.0201 (10)	-0.0020 (10)	-0.0177 (10)
Cl1	0.0433 (3)	0.0519 (3)	0.0484 (3)	-0.0204 (2)	-0.0019 (2)	-0.0177 (2)
O1A	0.079 (4)	0.062 (3)	0.078 (3)	-0.032 (3)	-0.033 (3)	-0.010 (3)
O2A	0.073 (4)	0.065 (3)	0.044 (2)	-0.030 (2)	-0.007 (2)	0.0004 (17)
O3A	0.123 (8)	0.077 (4)	0.112 (6)	-0.038 (4)	-0.016 (5)	-0.050 (4)
O4A	0.074 (3)	0.085 (5)	0.088 (5)	-0.039 (3)	0.015 (3)	-0.005 (3)
O1B	0.088 (10)	0.134 (16)	0.104 (11)	-0.017 (9)	-0.025 (9)	-0.078 (10)
O2B	0.130 (15)	0.093 (10)	0.125 (12)	-0.088 (11)	0.070 (9)	-0.047 (8)
O3B	0.060 (6)	0.054 (5)	0.071 (6)	0.003 (5)	-0.005 (4)	-0.029 (5)
O4B	0.060 (5)	0.094 (7)	0.116 (9)	-0.047 (5)	0.029 (5)	-0.057 (7)
Cl2	0.0410 (3)	0.0587 (4)	0.0838 (5)	-0.0185 (3)	-0.0003 (3)	-0.0358 (3)
O5	0.122 (3)	0.127 (3)	0.160 (3)	-0.076 (2)	-0.022 (2)	0.018 (2)

O6	0.0690 (18)	0.192 (3)	0.159 (3)	-0.029 (2)	0.0164 (18)	-0.120 (3)
O7	0.111 (2)	0.112 (2)	0.1031 (19)	-0.0633 (19)	0.0164 (17)	-0.0613 (18)
O8	0.0527 (14)	0.133 (3)	0.103 (2)	-0.0300 (15)	-0.0070 (13)	-0.0256 (19)
O9	0.0635 (14)	0.0607 (12)	0.1013 (17)	-0.0305 (11)	0.0107 (12)	-0.0167 (12)

Geometric parameters (Å, °)

N16—C21	1.491 (3)	C24—C25	1.377 (4)
N16—C17	1.499 (3)	C24—H241	0.9313
N16—C22	1.519 (3)	C25—C26	1.379 (5)
N16—H161	0.8954	C25—H251	0.9269
N19—C18	1.486 (3)	C26—C27	1.374 (5)
N19—C20	1.498 (4)	C26—H261	0.9388
N19—H191	0.8898	C27—C28	1.384 (4)
N19—H192	0.8904	C27—H271	0.9459
C17—C18	1.510 (3)	C28—H281	0.9242
C17—H171	0.9685	C11—O2B	1.382 (12)
C17—H172	0.9713	C11—O4B	1.398 (11)
C18—H181	0.9500	C11—O1B	1.409 (12)
C18—H182	0.9681	C11—O3A	1.416 (8)
C20—C21	1.503 (4)	C11—O2A	1.427 (5)
C20—H201	0.9532	C11—O3B	1.430 (10)
C20—H202	0.9768	C11—O1A	1.430 (6)
C21—H211	0.9671	C11—O4A	1.437 (7)
C21—H212	0.9645	C12—O6	1.374 (3)
C22—C23	1.500 (3)	C12—O7	1.386 (3)
C22—H221	0.9721	C12—O8	1.405 (3)
C22—H222	0.9695	C12—O5	1.484 (4)
C23—C24	1.380 (3)	O9—H91	0.8131
C23—C28	1.390 (3)	O9—H92	0.8189
C21—N16—C17	109.70 (17)	N16—C22—H222	108.0
C21—N16—C22	110.81 (18)	H221—C22—H222	108.4
C17—N16—C22	111.48 (17)	C24—C23—C28	118.9 (2)
C21—N16—H161	107.9	C24—C23—C22	121.7 (2)
C17—N16—H161	109.9	C28—C23—C22	119.3 (2)
C22—N16—H161	107.0	C25—C24—C23	120.8 (3)
C18—N19—C20	110.79 (19)	C25—C24—H241	120.0
C18—N19—H191	110.7	C23—C24—H241	119.2
C20—N19—H191	110.0	C24—C25—C26	120.0 (3)
C18—N19—H192	110.1	C24—C25—H251	118.8
C20—N19—H192	109.1	C26—C25—H251	121.1
H191—N19—H192	106.1	C27—C26—C25	119.9 (3)
N16—C17—C18	111.57 (19)	C27—C26—H261	120.3
N16—C17—H171	108.2	C25—C26—H261	119.7
C18—C17—H171	109.2	C26—C27—C28	120.1 (3)
N16—C17—H172	108.2	C26—C27—H271	119.3
C18—C17—H172	110.4	C28—C27—H271	120.5
H171—C17—H172	109.2	C27—C28—C23	120.2 (2)
N19—C18—C17	111.1 (2)	C27—C28—H281	120.4

supplementary materials

N19—C18—H181	107.3	C23—C28—H281	119.4
C17—C18—H181	111.0	O2B—C11—O4B	119.3 (15)
N19—C18—H182	108.7	O2B—C11—O1B	109.0 (11)
C17—C18—H182	109.9	O4B—C11—O1B	109.0 (12)
H181—C18—H182	108.7	O3A—C11—O2A	112.5 (8)
N19—C20—C21	110.0 (2)	O2B—C11—O3B	104.2 (14)
N19—C20—H201	110.1	O4B—C11—O3B	107.6 (11)
C21—C20—H201	108.4	O1B—C11—O3B	107.0 (13)
N19—C20—H202	110.0	O3A—C11—O1A	111.9 (7)
C21—C20—H202	108.6	O2A—C11—O1A	107.1 (4)
H201—C20—H202	109.7	O3A—C11—O4A	112.7 (6)
N16—C21—C20	111.0 (2)	O2A—C11—O4A	104.5 (5)
N16—C21—H211	109.0	O1A—C11—O4A	107.8 (6)
C20—C21—H211	110.0	O6—C12—O7	109.7 (2)
N16—C21—H212	109.1	O6—C12—O8	114.0 (2)
C20—C21—H212	110.5	O7—C12—O8	118.4 (2)
H211—C21—H212	107.1	O6—C12—O5	104.5 (3)
C23—C22—N16	111.90 (18)	O7—C12—O5	103.1 (2)
C23—C22—H221	109.6	O8—C12—O5	105.4 (2)
N16—C22—H221	108.8	H91—O9—H92	111.3
C23—C22—H222	110.1		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O9—H91 \cdots O4A ⁱ	0.81	2.27	2.942 (11)	141
O9—H92 \cdots O5 ⁱⁱ	0.82	2.06	2.869 (6)	170
N16—H161 \cdots O8	0.90	2.15	2.964 (3)	151
N19—H191 \cdots O9 ⁱⁱⁱ	0.89	1.92	2.750 (4)	155
N19—H192 \cdots O1A ^{iv}	0.89	2.08	2.907 (10)	154
C17—H172 \cdots O6 ^v	0.97	2.48	3.446 (5)	172
C20—H201 \cdots O7 ^{iv}	0.95	2.49	3.406 (4)	160
C21—H212 \cdots O3A	0.96	2.48	3.130 (15)	125

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

Fig. 1

