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Diaquabis(5-carboxy-2-ethyl-1*H*-imidazole-5-carboxylato-κ^2N^3,O^4)zinc trihydrate

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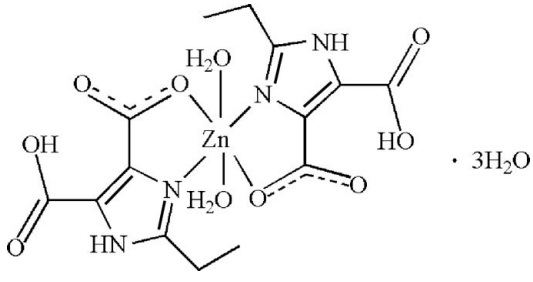
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; disorder in solvent or counterion; R factor = 0.041; wR factor = 0.119; data-to-parameter ratio = 11.7.

In the crystal structure of the title compound, $[Zn(C_7H_7N_2O_4)_2(H_2O)_2]\cdot 3H_2O$, the Zn^{II} ion, located an inversion center, is *N*,*O*-chelated by two 5-carboxy-2-ethyl-1*H*-imidazole-4-carboxylate anions and further coordinated by two water molecules in a distorted octahedral geometry. The carboxy group links with the carboxylate group of the same ligand *via* an intramolecular O—H···O hydrogen bond. An extensive intermolecular N—H···O and O—H···O hydrogen-bonded network exists in the crystal structure. One disordered lattice water molecule is half-occupied and is located close to an inversion center.

Related literature

For coordination polymers built from 2-ethyl-4,5-imidazole-dicarboxylate, see: Li *et al.* (2011); Wang *et al.* (2008); Zhang *et al.* (2010).



Experimental

Crystal data

$[Zn(C_7H_7N_2O_4)_2(H_2O)_2]\cdot 3H_2O$
M_r = 521.74
Triclinic, $P\overline{1}$
a = 7.229 (1) Å
b = 8.8959 (12) Å
c = 9.3541 (15) Å
α = 65.769 (1)°
β = 88.587 (2)°
γ = 70.676 (1)°
V = 513.31 (13) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.27 mm^{-1}
T = 298 K
0.24 × 0.22 × 0.21 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.750, T_{max} = 0.776
2676 measured reflections
1774 independent reflections
1532 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.119
S = 1.09
1774 reflections
152 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

Table 1
Selected bond lengths (Å).

Zn1—N1	2.104 (3)	Zn1—O5	2.116 (3)
Zn1—O1	2.164 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O6^{i}	0.86	1.95	2.778 (4)	161
O3—H3···O2	0.82	1.65	2.465 (4)	172
O5—H5*C*···O3^{ii}	0.85	1.95	2.785 (4)	167
O5—H5*D*···O4^{iii}	0.85	1.88	2.713 (4)	166
O6—H6*E*···O4^{iv}	0.86	2.29	3.145 (5)	175
O6—H6*F*···O7^{v}	0.85	2.09	2.664 (17)	125
O7—H7*F*···O1^{i}	0.85	2.12	2.93 (3)	160
O7—H7*G*···O2^{ii}	0.85	2.24	3.06 (3)	160

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

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5200).

References

Bruker (2004). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
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Zhang, F.-W., Li, Z.-F., Ge, T.-Z., Yao, H.-C., Li, G., Lu, H.-J. & Zhu, Y.-Y. (2010). *Inorg. Chem.* **49**, 3776–3788.

supporting information

Acta Cryst. (2011). E**67**, m717 [doi:10.1107/S160053681101676X]

Diaquabis(5-carboxy-2-ethyl-1*H*-imidazole-5-carboxylato-κ^2N^3,O^4)zinc trihydrate

Gang Zhang

S1. Comment

Self-assembly of supramolecular architectures based on imidazole carboxylate ligands has draw much attention during recent decades. To the best of our knowledge, coordination polymers based on 2-ethyl-4,5-imidazoledicarboxylate has been rarely reported so far (Wang *et al.*, 2008; Zhang *et al.*, 2010; Li *et al.*, 2011). Herein we report the title compound by the reaction of zinc nitrate with 2-ethyl-4,5-imidazoledicarboxylate (H_3EIDC) in an aqueous solution under hydrothermal condition.

The title compound, $[Zn(C_7H_7N_2O_4)_2(H_2O)_2].3H_2O$, depicted in Fig. 1, has two symmetrical coordination water molecules, three free water molecules and two 2-ethyl-4,5-imidazoledicarboxylate ligands. the Zn^{II} ion, lying on a center of inversion, is surrounded by two terminal water molecules, two nitrogen atoms and two oxygen atoms from two different 2-ethyl-4,5-imidazoledicarboxylate ligands in a slightly distorted octahedral coordination environment. Three solvent water molecules exist *via* hydrogen bonding among the imidazole N atom, the carboxylate O atom and the O atom from water molecule, whose distances and angles are shown in Tab. 1, Each H_2EIDC is bonded to Zn^{II} ion in a μ_2-mode. A three-dimensional supramolecular structure is consolidated by hydrogen-bonding interactions (N—H···O and O—H···O).

S2. Experimental

A mixture of $Zn(NO_3)_2$ (0.5 mmol, 0.110 g) and 2-ethyl-1*H*-imidazole-4,5-dicarboxylic acid (0.5 mmol, 0.95 g) in an aqueous solution (15 ml) was placed in a 23 ml Teflon-lined reactor, which was heated at 423 K for 2 d, and then cooled to room temperature at a rate of 10 K h^{-1}. Crystals of the title compound were obtained by slow evaporation of the solvent at room temperature.

S3. Refinement

Carboxy H atom was located in a difference map and refined with distance constraint of O—H = 0.82 Å, U_{iso}(H) = 1.5U_{eq}(O). Carbon and nitrogen bound H atoms were placed at calculated positions and were treated as riding on the parent C or N atoms with C—H = 0.96 (methyl), 0.97 (methylene) and N—H = 0.86 Å, U_{iso}(H) = 1.2 or 1.5U_{eq}(C,N). H atoms of the O6 water molecule were located in a difference Fourier map and refined as riding in as-found relative positions with U_{iso}(H) = 1.2U_{eq}(O). The O7 atom is located close to an inversion center and is half-occupied in the crystal structure; its H atoms were placed in calculated positions and refined in a riding mode with U_{iso}(H) = 1.2U_{eq}(O).

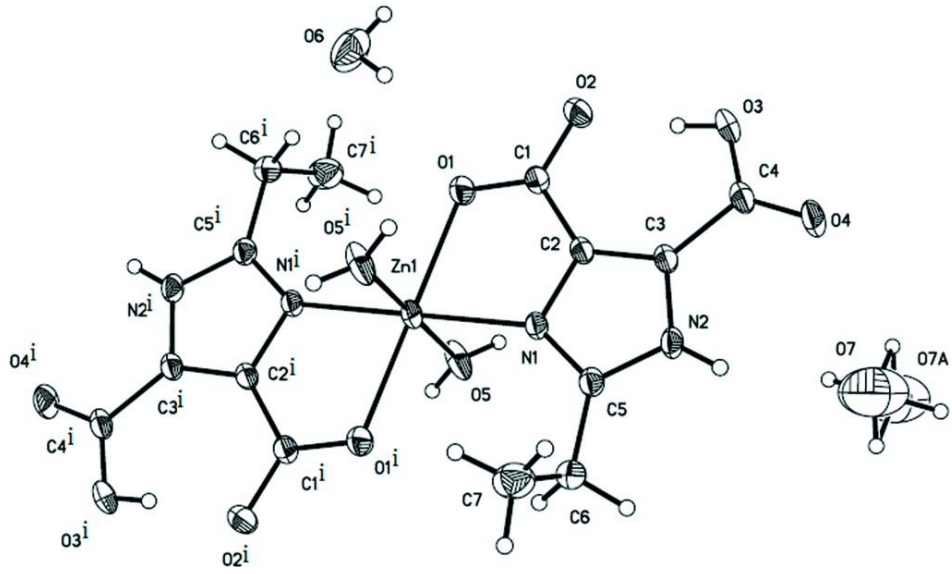

Figure 1

The structure of the title compound, showing the atomic numbering scheme with 30% probability displacement ellipsoids [symmetry codes: i: 1 - *x*, 1 - *y*, 1 - *z*.]

Diaquabis(5-carboxy-2-ethyl-1*H*-imidazole-5-carboxylato- κ^2N^3,O^4)zinc trihydrate

Crystal data

$[Zn(C_7H_7N_2O_4)_2(H_2O)_2]\cdot 3H_2O$
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Hall symbol: -P 1
$a = 7.229$ (1) Å
$b = 8.8959$ (12) Å
$c = 9.3541$ (15) Å
$\alpha = 65.769$ (1)°
$\beta = 88.587$ (2)°
$\gamma = 70.676$ (1)°
$V = 513.31$ (13) Å^3
$Z = 1$
$F(000) = 270$
$D_x = 1.688$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1329 reflections
$\theta = 2.4$–26.5°
$\mu = 1.27$ mm^{-1}
$T = 298$ K
Block, colorless
0.24 × 0.22 × 0.21 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.750$, $T_{max} = 0.776$
2676 measured reflections
1774 independent reflections
1532 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.4°$
$h = -8\rightarrow 8$
$k = -8\rightarrow 10$
$l = -10\rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.119$
$S = 1.09$
1774 reflections
152 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2 + 0.7163P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.45$ e Å^{-3}
	$\Delta\rho_{min} = -0.71$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Zn1	0.5000	0.5000	0.5000	0.0323 (2)	
N1	0.3641 (4)	0.6476 (4)	0.6256 (3)	0.0279 (7)	
N2	0.2272 (4)	0.7940 (4)	0.7637 (4)	0.0323 (7)	
H2	0.1798	0.8155	0.8413	0.039*	
O1	0.4472 (4)	0.7653 (3)	0.3230 (3)	0.0387 (6)	
O2	0.3323 (5)	1.0446 (4)	0.2846 (3)	0.0471 (8)	
O3	0.1810 (4)	1.2103 (3)	0.4395 (3)	0.0459 (7)	
H3	0.2405	1.1501	0.3944	0.069*	
O4	0.0686 (4)	1.1604 (4)	0.6715 (4)	0.0461 (7)	
O5	0.7784 (4)	0.4784 (4)	0.5890 (4)	0.0511 (8)	
H5C	0.8109	0.5636	0.5848	0.061*	
H5D	0.8821	0.3872	0.6159	0.061*	
O6	0.1656 (5)	0.8359 (5)	0.0415 (4)	0.0734 (11)	
H6E	0.1063	0.8394	0.1212	0.088*	
H6F	0.1745	0.9376	−0.0026	0.088*	
O7	0.439 (3)	0.987 (4)	0.987 (3)	0.169 (9)	0.50
H7F	0.4629	0.9037	1.0795	0.202*	0.50
H7G	0.5168	0.9516	0.9288	0.202*	0.50
C1	0.3691 (5)	0.8809 (5)	0.3702 (4)	0.0310 (8)	
C2	0.3209 (5)	0.8245 (4)	0.5336 (4)	0.0274 (8)	
C3	0.2351 (5)	0.9175 (5)	0.6180 (4)	0.0290 (8)	
C4	0.1541 (6)	1.1095 (5)	0.5770 (5)	0.0343 (9)	
C5	0.3068 (5)	0.6320 (5)	0.7650 (4)	0.0304 (8)	
C6	0.3164 (6)	0.4663 (5)	0.9015 (5)	0.0386 (9)	
H6A	0.2978	0.4874	0.9956	0.046*	
H6B	0.4466	0.3783	0.9188	0.046*	
C7	0.1624 (8)	0.3961 (7)	0.8768 (6)	0.0579 (13)	
H7A	0.1874	0.3649	0.7895	0.087*	
H7B	0.0336	0.4849	0.8544	0.087*	
H7C	0.1681	0.2938	0.9706	0.087*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0374 (4)	0.0234 (3)	0.0368 (4)	−0.0042 (3)	0.0041 (3)	−0.0184 (3)
N1	0.0294 (16)	0.0241 (15)	0.0311 (16)	−0.0060 (12)	0.0031 (12)	−0.0152 (13)
N2	0.0336 (17)	0.0309 (17)	0.0357 (17)	−0.0051 (14)	0.0058 (13)	−0.0222 (14)
O1	0.0490 (17)	0.0309 (14)	0.0351 (15)	−0.0090 (12)	0.0111 (12)	−0.0174 (12)
O2	0.072 (2)	0.0276 (15)	0.0381 (16)	−0.0160 (14)	0.0135 (15)	−0.0120 (13)
O3	0.0598 (19)	0.0240 (14)	0.0548 (19)	−0.0083 (13)	0.0056 (15)	−0.0226 (14)
O4	0.0492 (18)	0.0328 (15)	0.0566 (18)	−0.0010 (13)	0.0059 (14)	−0.0298 (14)
O5	0.0344 (16)	0.0329 (16)	0.090 (2)	−0.0026 (12)	−0.0071 (15)	−0.0364 (17)
O6	0.073 (2)	0.095 (3)	0.055 (2)	−0.014 (2)	0.0122 (18)	−0.047 (2)
O7	0.15 (2)	0.154 (14)	0.125 (11)	−0.022 (16)	0.017 (16)	−0.013 (10)
C1	0.034 (2)	0.0264 (19)	0.034 (2)	−0.0091 (16)	0.0034 (16)	−0.0152 (16)
C2	0.0266 (18)	0.0230 (18)	0.0333 (19)	−0.0067 (14)	−0.0006 (14)	−0.0139 (15)
C3	0.0283 (19)	0.0248 (18)	0.0352 (19)	−0.0064 (15)	0.0006 (15)	−0.0162 (16)
C4	0.032 (2)	0.0264 (19)	0.046 (2)	−0.0056 (16)	−0.0028 (17)	−0.0206 (19)
C5	0.0291 (19)	0.0295 (19)	0.034 (2)	−0.0064 (15)	0.0017 (15)	−0.0170 (16)
C6	0.045 (2)	0.033 (2)	0.033 (2)	−0.0097 (18)	0.0042 (18)	−0.0130 (17)
C7	0.069 (3)	0.058 (3)	0.048 (3)	−0.034 (3)	0.004 (2)	−0.014 (2)

Geometric parameters (Å, º)

Zn1—N1	2.104 (3)	O5—H5D	0.8499
Zn1—N1^{i}	2.104 (3)	O6—H6E	0.8578
Zn1—O1	2.164 (3)	O6—H6F	0.8502
Zn1—O1^{i}	2.164 (3)	O7—O7^{ii}	1.05 (3)
Zn1—O5	2.116 (3)	O7—H7F	0.8500
Zn1—O5^{i}	2.116 (3)	O7—H7G	0.8500
N1—C5	1.324 (5)	C1—C2	1.473 (5)
N1—C2	1.375 (4)	C2—C3	1.366 (5)
N2—C5	1.358 (5)	C3—C4	1.490 (5)
N2—C3	1.369 (5)	C5—C6	1.483 (5)
N2—H2	0.8600	C6—C7	1.509 (6)
O1—C1	1.243 (4)	C6—H6A	0.9700
O2—C1	1.277 (4)	C6—H6B	0.9700
O3—C4	1.286 (5)	C7—H7A	0.9600
O3—H3	0.8200	C7—H7B	0.9600
O4—C4	1.218 (5)	C7—H7C	0.9600
O5—H5C	0.8501		
N1—Zn1—N1^{i}	180.0	H7F—O7—H7G	108.8
N1—Zn1—O5	88.97 (11)	O1—C1—O2	123.1 (3)
N1^{i}—Zn1—O5	91.03 (11)	O1—C1—C2	118.0 (3)
N1—Zn1—O5^{i}	91.03 (11)	O2—C1—C2	118.9 (3)
N1^{i}—Zn1—O5^{i}	88.97 (11)	C3—C2—N1	109.7 (3)
O5—Zn1—O5^{i}	180.00 (16)	C3—C2—C1	131.9 (3)
N1—Zn1—O1	78.99 (10)	N1—C2—C1	118.5 (3)

N1^{i}—Zn1—O1	101.01 (10)	C2—C3—N2	105.4 (3)
O5—Zn1—O1	91.97 (11)	C2—C3—C4	132.7 (4)
O5^{i}—Zn1—O1	88.03 (11)	N2—C3—C4	121.8 (3)
N1—Zn1—O1^{i}	101.01 (10)	O4—C4—O3	124.9 (4)
N1^{i}—Zn1—O1^{i}	78.99 (10)	O4—C4—C3	119.8 (4)
O5—Zn1—O1^{i}	88.03 (11)	O3—C4—C3	115.4 (3)
O5^{i}—Zn1—O1^{i}	91.97 (11)	N1—C5—N2	109.7 (3)
O1—Zn1—O1^{i}	180.00 (11)	N1—C5—C6	126.4 (3)
C5—N1—C2	106.7 (3)	N2—C5—C6	123.8 (3)
C5—N1—Zn1	142.7 (3)	C5—C6—C7	112.4 (3)
C2—N1—Zn1	110.7 (2)	C5—C6—H6A	109.1
C5—N2—C3	108.6 (3)	C7—C6—H6A	109.1
C5—N2—H2	125.7	C5—C6—H6B	109.1
C3—N2—H2	125.7	C7—C6—H6B	109.1
C1—O1—Zn1	113.8 (2)	H6A—C6—H6B	107.9
C4—O3—H3	109.5	C6—C7—H7A	109.5
Zn1—O5—H5C	125.6	C6—C7—H7B	109.5
Zn1—O5—H5D	124.4	H7A—C7—H7B	109.5
H5C—O5—H5D	108.7	C6—C7—H7C	109.5
H6E—O6—H6F	102.5	H7A—C7—H7C	109.5
O7^{ii}—O7—H7F	88.8	H7B—C7—H7C	109.5
O7^{ii}—O7—H7G	79.9		

N1^{i}—Zn1—N1—C5	55 (100)	O2—C1—C2—C3	1.8 (6)
O5—Zn1—N1—C5	−87.4 (4)	O1—C1—C2—N1	−0.1 (5)
O5^{i}—Zn1—N1—C5	92.6 (4)	O2—C1—C2—N1	−178.9 (3)
O1—Zn1—N1—C5	−179.6 (4)	N1—C2—C3—N2	0.2 (4)
O1^{i}—Zn1—N1—C5	0.4 (4)	C1—C2—C3—N2	179.6 (4)
N1^{i}—Zn1—N1—C2	−127 (100)	N1—C2—C3—C4	−177.8 (4)
O5—Zn1—N1—C2	90.9 (2)	C1—C2—C3—C4	1.5 (7)
O5^{i}—Zn1—N1—C2	−89.1 (2)	C5—N2—C3—C2	0.0 (4)
O1—Zn1—N1—C2	−1.3 (2)	C5—N2—C3—C4	178.3 (3)
O1^{i}—Zn1—N1—C2	178.7 (2)	C2—C3—C4—O4	173.7 (4)
N1—Zn1—O1—C1	1.4 (3)	N2—C3—C4—O4	−4.1 (5)
N1^{i}—Zn1—O1—C1	−178.6 (3)	C2—C3—C4—O3	−5.9 (6)
O5—Zn1—O1—C1	−87.2 (3)	N2—C3—C4—O3	176.3 (3)
O5^{i}—Zn1—O1—C1	92.8 (3)	C2—N1—C5—N2	0.4 (4)
O1^{i}—Zn1—O1—C1	168 (100)	Zn1—N1—C5—N2	178.7 (3)
Zn1—O1—C1—O2	177.7 (3)	C2—N1—C5—C6	177.6 (3)
Zn1—O1—C1—C2	−1.1 (4)	Zn1—N1—C5—C6	−4.1 (6)
C5—N1—C2—C3	−0.4 (4)	C3—N2—C5—N1	−0.3 (4)
Zn1—N1—C2—C3	−179.3 (2)	C3—N2—C5—C6	−177.5 (3)
C5—N1—C2—C1	−179.8 (3)	N1—C5—C6—C7	−73.4 (5)
Zn1—N1—C2—C1	1.3 (4)	N2—C5—C6—C7	103.4 (4)
O1—C1—C2—C3	−179.4 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O6^{iii}	0.86	1.95	2.778 (4)	161
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O5—H5*C*···O3^{iv}	0.85	1.95	2.785 (4)	167
O5—H5*D*···O4^{v}	0.85	1.88	2.713 (4)	166
O6—H6*E*···O4^{vi}	0.86	2.29	3.145 (5)	175
O6—H6*F*···O7^{vii}	0.85	2.09	2.664 (17)	125
O7—H7*F*···O1^{iii}	0.85	2.12	2.93 (3)	160
O7—H7*G*···O2^{iv}	0.85	2.24	3.06 (3)	160

Symmetry codes: (iii) $x, y, z+1$; (iv) $-x+1, -y+2, -z+1$; (v) $x+1, y-1, z$; (vi) $-x, -y+2, -z+1$; (vii) $x, y, z-1$.