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Poly[[aquacalcium(II)]-μ_4-1*H*-imidazole-4,5-dicarboxylato]

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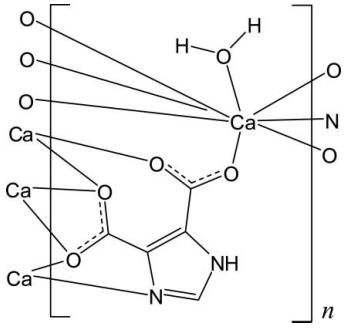
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.069; data-to-parameter ratio = 14.0.

In the title compound, $[Ca(C_5H_2N_2O_4)(H_2O)]_n$, the Ca^{2+} cations are eigthtfold coordinated by six O atoms and one N atom of four symmetry-related anions and one water molecule within an irregular polyhedron. These CaO_7N polyhedra are connected *via* the anions into a three-dimensional network. The anions are additionally linked by N—H···O and O—H···O hydrogen bonding.

Related literature

For general background to metal coordination polymers, see: Kitagawa *et al.* (2004). For related structures, see: Gao *et al.* (2004); Starosta & Leciejewicz (2006).



Experimental

Crystal data

$[Ca(C_5H_2N_2O_4)(H_2O)]$ M_r = 212.18
Monoclinic, $P2_1/n$
a = 6.4752 (4) Å
b = 9.7627 (6) Å
c = 10.9079 (6) Å
β = 103.041 (2)°
V = 671.76 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.92 mm^{-1}
T = 295 K
0.45 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.681, T_{max} = 0.874
6029 measured reflections
1665 independent reflections
1619 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.069
S = 1.08
1665 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O4^{i}	0.85	2.13	2.9552 (14)	162
O5—H5B···O1^{ii}	0.85	2.23	3.0109 (14)	153
N2—H2A···O4^{iii}	0.86	1.86	2.7220 (13)	176

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

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2199).

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supporting information

Acta Cryst. (2010). E**66**, m1382 [https://doi.org/10.1107/S1600536810039000]

Poly[[aquacalcium(II)]-μ_4-1*H*-imidazole-4,5-dicarboxylato]

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S1. Comment

The synthesis of metal coordination polymers has been a intense research due to their interesting topologies and potential applications (Kitagawa, *et al.*, 2004). The imidazole-4,5-dicarboxylic acid (H_3IDC) has been successively applied to construct two calcium complexes (Gao, *et al.*, 2004; Starosta, *et al.*, 2006). In our ongoing investigations in this field we report here the structure of a new Ca compound with the anionic imidazole-4,5-dicarboxylato ligand.

The asymmetric unit of the title compound conists of one Ca atom, one carboxylate ligand and one coordinated water molecule all of them located in general positions (Figure 1). The Ca center is eight-coordinated by six oxygen atoms and one nitrogen atom of four carboxylate ligands and one oxygen atom of a coordinated water molecule within an irregular polyhedron. The Ca—O distances range from 2.3197 (11) to 2.8777 (12) Å and the Ca—N distance amount to 2.4215 (10)Å. The CaO_7N polyhedra are connected via the anions into a three-dimensional network and are further linked by N—H···O and O—H···O hydrogen bonding (Fig. 2 and Table 1).

S2. Experimental

imidazole-4,5-dicarboxylic acid ($C_5H_4N_2O_4$, 0.0752 g, 0.45 mmol) and $Ca(NO_3)_2.4H_2O$ (0.2361 g, 1 mmol) were reacted in 10 mL of H_2O in a Teflon-lined digestion bomb with an internal volume of 23 ml l. The reaction mixture was heated to 453 K for 5 d followed by slow cooling at 6 K/h to room temperature. The product consits of transparent colorless crystals.

S3. Refinement

H atoms were constrained to ideal geometries, with C—H = 0.93 Å, O—H = 0.85 Å and N—H = 0.86 Å and refined with U_{iso}(H) = 1.2U_{eq}(1.5 for water H atoms) using a riding model.

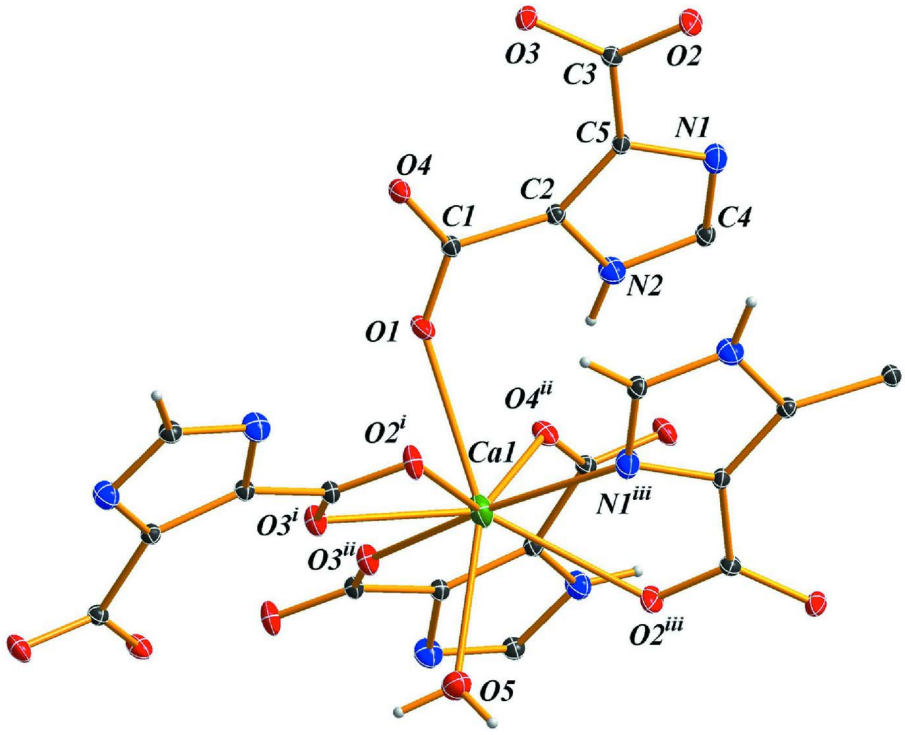

Figure 1

Crystal structure of the title compound with labelling and displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (i) *x* + 1/2, -*y* + 1/2, *z* - 1/2; (ii) -*x* - 1/2, *y* + 1/2, -*z* + 1/2; (iii) -*x*, -*y* + 1, -*z* + 1.

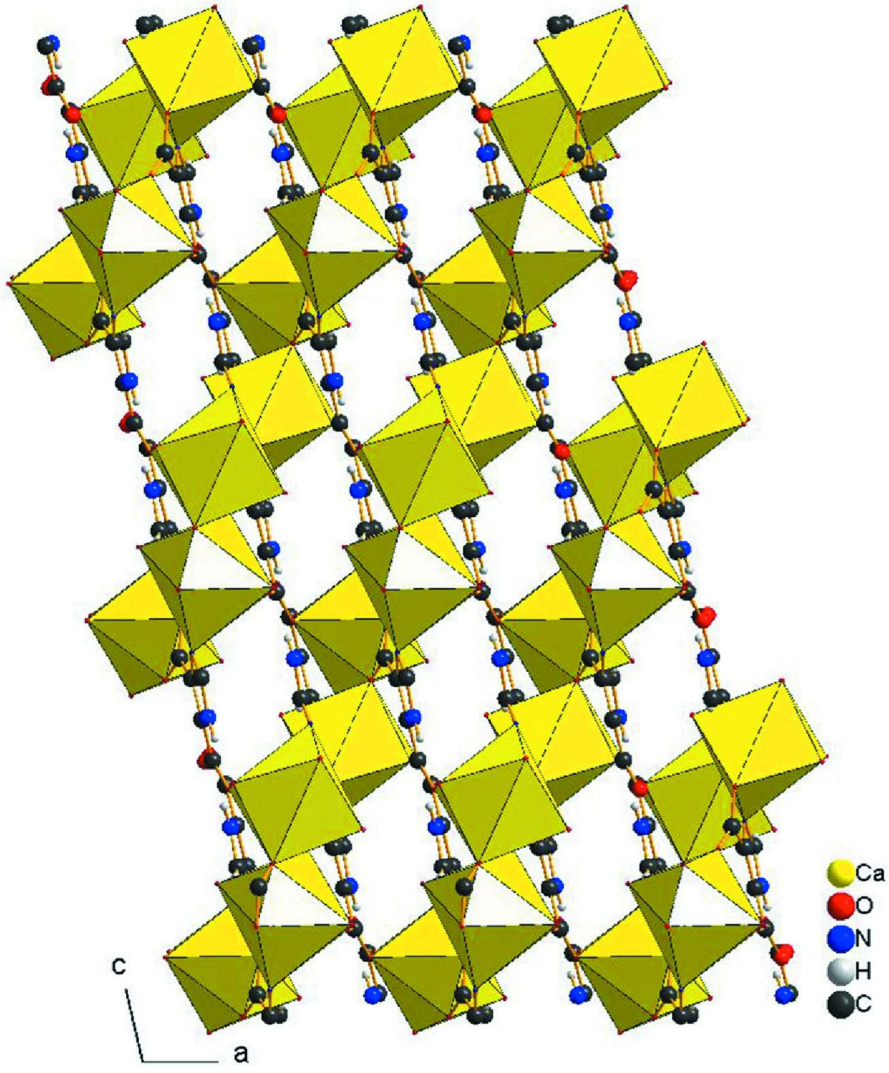

Figure 2

Crystal structure of the title compound with view along the crystallographic *b* axis.

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Crystal data

$[Ca(C_5H_2N_2O_4)(H_2O)]$
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Hall symbol: -P 2yn
$a = 6.4752$ (4) Å
$b = 9.7627$ (6) Å
$c = 10.9079$ (6) Å
$\beta = 103.041$ (2)°
$V = 671.76$ (7) Å^3
$Z = 4$

$F(000) = 432$
$D_x = 2.098$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4091 reflections
$\theta = 2.8$–$28.3°$
$\mu = 0.92$ mm^{-1}
$T = 295$ K
Columnar, colourless
0.45 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.681, T_{max} = 0.874

6029 measured reflections
1665 independent reflections
1619 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.3°, θ_{min} = 2.8°
h = −8→8
k = −12→12
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.069
S = 1.08
1665 reflections
119 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
$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.2825P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.092 (5)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Ca1	0.08356 (4)	0.55299 (2)	0.17359 (2)	0.01629 (12)
O1	−0.22252 (16)	0.39733 (10)	0.20098 (8)	0.0226 (2)
O2	−0.25830 (16)	0.22314 (10)	0.70724 (8)	0.0239 (2)
O3	−0.40728 (15)	0.13090 (9)	0.52331 (8)	0.0202 (2)
O4	−0.30507 (15)	0.19962 (9)	0.28239 (8)	0.0201 (2)
O5	0.38501 (16)	0.60082 (11)	0.08033 (10)	0.0293 (2)
H5A	0.5147	0.6114	0.1164	0.044*
H5B	0.3763	0.5845	0.0028	0.044*
C1	−0.26916 (17)	0.32726 (12)	0.28690 (10)	0.0140 (2)
C2	−0.27309 (17)	0.39902 (11)	0.40685 (10)	0.0132 (2)
C3	−0.31453 (17)	0.22884 (12)	0.58996 (11)	0.0143 (2)
C4	−0.23120 (19)	0.57835 (12)	0.53133 (11)	0.0160 (2)
H4A	−0.2128	0.6687	0.5586	0.019*
C5	−0.27759 (17)	0.35991 (11)	0.52820 (10)	0.0129 (2)

N1	−0.24822 (16)	0.47378 (10)	0.60526 (9)	0.0152 (2)
N2	−0.24362 (16)	0.53860 (10)	0.41286 (10)	0.0147 (2)
H2A	−0.2347	0.5911	0.3510	0.018*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ca1	0.02062 (16)	0.01716 (16)	0.01057 (16)	0.00256 (8)	0.00247 (10)	−0.00080 (8)
O1	0.0348 (5)	0.0199 (5)	0.0159 (4)	−0.0034 (4)	0.0118 (4)	0.0003 (3)
O2	0.0332 (5)	0.0246 (5)	0.0121 (4)	−0.0058 (4)	0.0009 (4)	0.0035 (3)
O3	0.0297 (5)	0.0141 (4)	0.0160 (4)	−0.0067 (3)	0.0032 (4)	−0.0002 (3)
O4	0.0305 (5)	0.0138 (4)	0.0184 (4)	−0.0033 (3)	0.0103 (4)	−0.0037 (3)
O5	0.0252 (5)	0.0310 (6)	0.0326 (5)	−0.0034 (4)	0.0082 (4)	−0.0045 (4)
C1	0.0155 (5)	0.0141 (5)	0.0128 (5)	0.0003 (4)	0.0038 (4)	−0.0012 (4)
C2	0.0151 (5)	0.0111 (5)	0.0133 (5)	−0.0002 (4)	0.0032 (4)	−0.0001 (4)
C3	0.0159 (5)	0.0141 (5)	0.0130 (5)	−0.0004 (4)	0.0033 (4)	0.0015 (4)
C4	0.0197 (5)	0.0125 (5)	0.0157 (5)	−0.0007 (4)	0.0041 (4)	−0.0023 (4)
C5	0.0146 (5)	0.0120 (5)	0.0116 (5)	−0.0011 (4)	0.0020 (4)	−0.0014 (4)
N1	0.0194 (5)	0.0131 (4)	0.0129 (5)	−0.0015 (4)	0.0030 (4)	−0.0026 (4)
N2	0.0196 (5)	0.0115 (5)	0.0138 (5)	−0.0003 (3)	0.0054 (4)	0.0009 (3)

Geometric parameters (Å, º)

Ca1—O3^{i}	2.3211 (9)	O3—Ca1^{v}	2.4408 (9)
Ca1—N1^{ii}	2.4215 (10)	O4—C1	1.2665 (14)
Ca1—O4^{i}	2.4336 (9)	O4—Ca1^{vi}	2.4336 (9)
Ca1—O3^{iii}	2.4408 (9)	O5—H5A	0.8497
Ca1—O5	2.4411 (11)	O5—H5B	0.8496
Ca1—O1	2.5679 (10)	C1—C2	1.4895 (15)
Ca1—O2^{ii}	2.6614 (10)	C2—N2	1.3755 (14)
Ca1—O2^{iii}	2.8776 (10)	C2—C5	1.3842 (15)
Ca1—C3^{iii}	3.0181 (12)	C3—C5	1.4904 (16)
Ca1—Ca1^{iv}	3.8384 (5)	C3—Ca1^{v}	3.0182 (12)
O1—C1	1.2511 (14)	C4—N1	1.3209 (16)
O2—C3	1.2498 (14)	C4—N2	1.3342 (15)
O2—Ca1^{ii}	2.6613 (10)	C4—H4A	0.9300
O2—Ca1^{v}	2.8777 (10)	C5—N1	1.3806 (14)
O3—C3	1.2675 (14)	N1—Ca1^{ii}	2.4215 (10)
O3—Ca1^{vi}	2.3212 (9)	N2—H2A	0.8600
O3^{i}—Ca1—N1^{ii}	166.21 (3)	O5—Ca1—Ca1^{iv}	73.40 (3)
O3^{i}—Ca1—O4^{i}	76.01 (3)	O1—Ca1—Ca1^{iv}	84.38 (2)
N1^{ii}—Ca1—O4^{i}	92.65 (3)	O2^{ii}—Ca1—Ca1^{iv}	134.22 (2)
O3^{i}—Ca1—O3^{iii}	72.60 (3)	O2^{iii}—Ca1—Ca1^{iv}	83.473 (19)
N1^{ii}—Ca1—O3^{iii}	121.18 (3)	C3^{iii}—Ca1—Ca1^{iv}	59.22 (2)
O4^{i}—Ca1—O3^{iii}	134.15 (3)	C1—O1—Ca1	137.21 (8)
O3^{i}—Ca1—O5	79.88 (4)	C3—O2—Ca1^{ii}	117.19 (8)
N1^{ii}—Ca1—O5	102.83 (4)	C3—O2—Ca1^{v}	84.15 (7)

O4^{i}—Ca1—O5	131.87 (3)	Ca1^{ii}—O2—Ca1^{v}	158.66 (4)
O3^{iii}—Ca1—O5	73.62 (3)	C3—O3—Ca1^{vi}	148.04 (8)
O3^{i}—Ca1—O1	94.03 (3)	C3—O3—Ca1^{v}	104.46 (7)
N1^{ii}—Ca1—O1	89.89 (3)	Ca1^{vi}—O3—Ca1^{v}	107.40 (3)
O4^{i}—Ca1—O1	72.51 (3)	C1—O4—Ca1^{vi}	135.14 (8)
O3^{iii}—Ca1—O1	77.25 (3)	Ca1—O5—H5A	129.0
O5—Ca1—O1	150.74 (3)	Ca1—O5—H5B	119.9
O3^{i}—Ca1—O2^{ii}	104.41 (3)	H5A—O5—H5B	108.7
N1^{ii}—Ca1—O2^{ii}	63.83 (3)	O1—C1—O4	125.60 (10)
O4^{i}—Ca1—O2^{ii}	70.90 (3)	O1—C1—C2	117.11 (10)
O3^{iii}—Ca1—O2^{ii}	149.31 (3)	O4—C1—C2	117.22 (10)
O5—Ca1—O2^{ii}	75.80 (3)	N2—C2—C5	105.09 (10)
O1—Ca1—O2^{ii}	133.11 (3)	N2—C2—C1	118.56 (10)
O3^{i}—Ca1—O2^{iii}	120.77 (3)	C5—C2—C1	135.93 (10)
N1^{ii}—Ca1—O2^{iii}	73.01 (3)	O2—C3—O3	122.99 (11)
O4^{i}—Ca1—O2^{iii}	141.64 (3)	O2—C3—C5	117.48 (10)
O3^{iii}—Ca1—O2^{iii}	48.30 (3)	O3—C3—C5	119.48 (10)
O5—Ca1—O2^{iii}	86.41 (3)	O2—C3—Ca1^{v}	71.53 (7)
O1—Ca1—O2^{iii}	72.09 (3)	O3—C3—Ca1^{v}	51.54 (6)
O2^{ii}—Ca1—O2^{iii}	127.387 (11)	C5—C3—Ca1^{v}	170.99 (8)
O3^{i}—Ca1—C3^{iii}	96.57 (3)	N1—C4—N2	111.80 (11)
N1^{ii}—Ca1—C3^{iii}	97.22 (3)	N1—C4—H4A	124.1
O4^{i}—Ca1—C3^{iii}	144.90 (3)	N2—C4—H4A	124.1
O3^{iii}—Ca1—C3^{iii}	23.99 (3)	N1—C5—C2	109.29 (10)
O5—Ca1—C3^{iii}	78.38 (3)	N1—C5—C3	115.48 (10)
O1—Ca1—C3^{iii}	73.91 (3)	C2—C5—C3	135.17 (10)
O2^{ii}—Ca1—C3^{iii}	142.98 (3)	C4—N1—C5	105.64 (10)
O2^{iii}—Ca1—C3^{iii}	24.33 (3)	C4—N1—Ca1^{ii}	127.80 (8)
O3^{i}—Ca1—Ca1^{iv}	37.36 (2)	C5—N1—Ca1^{ii}	119.26 (7)
N1^{ii}—Ca1—Ca1^{iv}	156.42 (3)	C4—N2—C2	108.16 (10)
O4^{i}—Ca1—Ca1^{iv}	107.26 (2)	C4—N2—H2A	125.9
O3^{iii}—Ca1—Ca1^{iv}	35.24 (2)	C2—N2—H2A	125.9

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*A*···O4^{vii}	0.85	2.13	2.9552 (14)	162
O5—H5*B*···O1^{iv}	0.85	2.23	3.0109 (14)	153
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