

Poly[di(μ_2 -2-hydroxypropanoato)cadmium]

Ao-Wei Chen, Rong Shi, Ce Wang, Bi-Bo Shi and Zheng Yin*

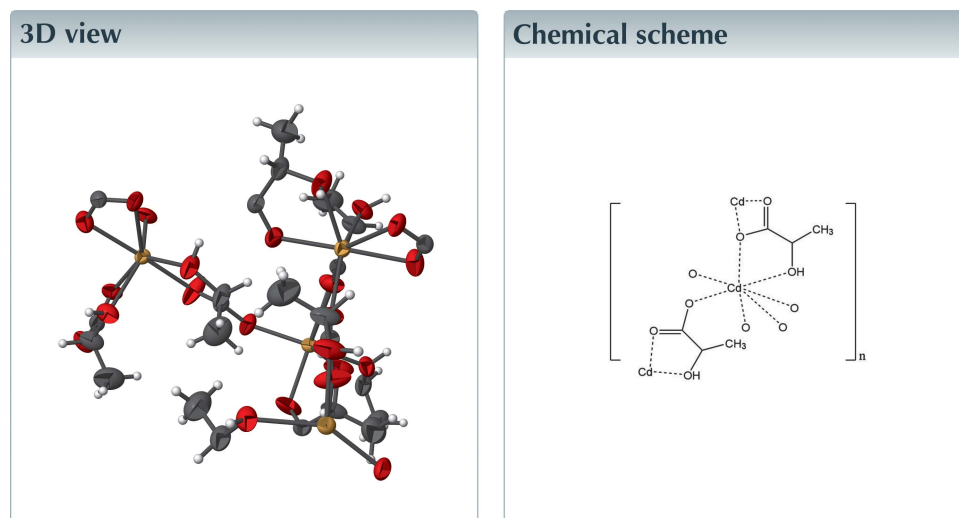
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Received 10 June 2019
Accepted 9 September 2019Edited by M. Weil, Vienna University of
Technology, AustriaKeywords: crystal structure; inorganic–organic
hybrid compound; cadmium; hydrogen
bonding.

CCDC reference: 1921720

Structural data: full structural data are available
from iucrdata.iucr.org

The asymmetric unit of the title inorganic–organic salt, poly[di(μ_2 -2-hydroxypropanoato)cadmium], $[\text{Cd}(\text{C}_3\text{H}_5\text{O}_3)_2]_n$ or $[\text{Cd}(\text{Hlac})_2]_n$ (H_2lac = 2-hydroxypropanoic acid), comprises of a cadmium cation and two 2-hydroxypropanoate anions. The cadmium cation exhibits a distorted pentagonal–bipyramidal coordination environment defined by the hydroxy and carbonyl O atoms of the 2-hydroxypropanoate anions. The coordination mode leads to the formation of layers extending parallel to (010). O–H...O hydrogen bonding between the hydroxy and carbonyl groups stabilizes the structure packing.



Structure description

Compounds with metal–organic framework (MOF) structures with accessible open space have rapidly grown into a major area of chemical research because of their structural diversity and wide applications (Furukawa *et al.*, 2013). Crystal engineering of MOFs has been dominated by single organic units like polycarboxylates, polypyridines, azolate or their derivatives. Recently, mixed-ligand MOFs (Yin *et al.*, 2015), were found to be successful in the rational construction of materials with targeted functionalities. One of the interesting candidates for the construction of mixed-ligand MOFs is 2-hydroxypropanoic acid (H_2lac). Working with the corresponding anion has several advantages: (i) multiple coordination modes by using the hydroxyl and carboxyl groups are possible; (ii) the anion is flexible and a chelating ligand, and thus can facilitate the formation of key building units such as chains or layers; (iii) the terminal methyl group can be replaced by $-\text{H}$, $-\text{C}_2\text{H}_5$, $-\text{Ph}$ and other groups for structural regulation and expansion. As a typical example, the combination of H_2lac and linear pyridine carboxylate generates highly stable rod-spacer MOFs with double π -wall and square nano-channels (Zeng *et al.*, 2010), achieving high-efficiency iodine capture.

During exploration of the coordination chemistry of H_2lac with different metals and co-ligands, the title compound was obtained as a single-ligand cadmium compound,

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O3—H3···O2 ⁱ	0.87 (1)	1.81 (2)	2.657 (6)	163 (5)
C5—H5···O4 ⁱⁱ	0.98	2.22	3.021 (8)	139
O6—H6···O5 ⁱⁱ	0.85 (1)	2.11 (9)	2.747 (7)	132 (11)

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

notwithstanding the presence of the linear pyrazine co-ligand under the given solvothermal conditions. The asymmetric unit of the title compound comprises of one cadmium cation and two 2-hydroxypropanoate anions (Fig. 1). The Cd1 cation is sevenfold-coordinated by oxygen and adopts a distorted pentagonal-bipyramidal coordination environment. Four oxygen atoms stem from carboxyl groups and three from hydroxyl groups from four different Hlac[−] ligands whereby three ligands are chelating and one is monodentate. This coordination mode leads to the formation of layers extending parallel to (010) (Fig. 2, left). Under consideration of the Cd as nodes, the cations are extended to tapes parallel to [001] consisting of a (4,4) grid (Fig. 2, right). The tapes are stacked along [100] and are linked into a three-dimensional network by more distant nodes [Cd···Cd distances of 6.4014 (14) Å] parallel to [210]. The crystal packing is consolidated by O—H···O hydrogen-bonding interactions of medium strength between hydroxy and carbonyl functions, and additional weak C—H···O interactions (Table 1).

Synthesis and crystallization

A mixture of H₂lac (0.125 mmol), pyrazine (0.1 mmol) and Cd(NO₃)₂·4H₂O (0.2 mmol) in C₂H₅OH (15 ml) was stirred in air with a magnetic stirrer, generating a colourless clear solution after 10 min. The reaction solution was then transferred to a solvothermal PTFE reaction vessel with 25 ml capacity, followed by heating at 393 K for 72 h. The reaction vessel was then cooled to room temperature at a rate of 10 K h^{−1}. The formed crystalline material was filtered to obtain colourless rod-like crystals with a yield of about 52% (based on Cd). The obtained crystals are insoluble in common

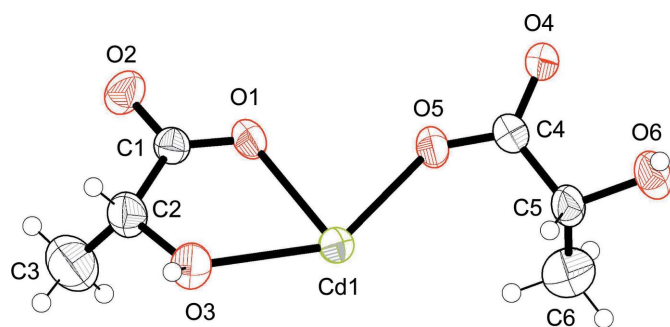


Figure 1
The asymmetric unit of the title compound showing the atom numbering with displacement ellipsoids drawn at the 50% probability level.

Table 2
Experimental details.

Crystal data	
Chemical formula	[Cd(C ₃ H ₅ O ₃) ₂]
<i>M_r</i>	290.54
Crystal system, space group	Orthorhombic, <i>Iba</i> 2
Temperature (K)	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.238 (2), 19.104 (4), 9.463 (2)
<i>V</i> (Å ³)	1850.8 (7)
<i>Z</i>	8
Radiation type	Mo <i>K</i> α
μ (mm ^{−1})	2.36
Crystal size (mm)	0.3 × 0.2 × 0.2
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2016)
<i>T</i> _{min} , <i>T</i> _{max}	0.547, 0.746
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	9606, 2230, 2032
<i>R</i> _{int}	0.031
(sin θ/λ) _{max} (Å ^{−1})	0.672
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.025, 0.065, 1.05
No. of reflections	2230
No. of parameters	127
No. of restraints	18
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ^{−3})	0.88, −0.36
Absolute structure	Flack <i>x</i> determined using 836 quotients [(<i>I</i> ⁺) − (<i>I</i> [−])] / [(<i>I</i> ⁺) + (<i>I</i> [−])] (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.02 (2)

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *OLEX2* (Dolomanov *et al.*, 2009) and *PLATON* (Spek, 2009).

organic solvents such as DMF, CH₃OH, C₂H₅OH, CH₂Cl₂, and acetone. IR (KBr pellets, cm^{−1}): 3044 (*m*), 1626 (*s*), 1563 (*s*), 1451 (*s*), 1370 (*s*), 1092 (*m*). Elemental analysis (%), calculated: C, 24.80; H, 3.47; found: C, 24.71; H, 3.55. The compound is thermally stable up to 533 K under an N₂ atmosphere.

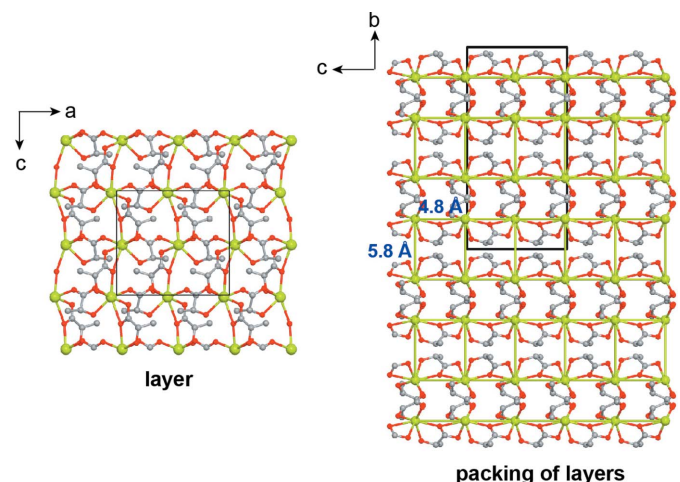


Figure 2
Left: formation of polymeric layers extending parallel to (010); right: bands of (4,4) grids between Cd nodes extending parallel to [001]. H atoms have been omitted for clarity.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank Shaanxi University of Science and Technology for supporting this work.

Funding information

Funding for this research was provided by: the National Science Foundation of China (No. 21601116), the NSF of Shaanxi Province (Nos. 2017JQ2008, 17JK0113), the China Postdoctoral Science Foundation (2016M592736), and the College students' innovation and entrepreneurship training program (No. 201710708017).

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full crystallographic data

IUCrData (2019). 4, x191255 [https://doi.org/10.1107/S2414314619012550]

Poly[di(μ_2 -2-hydroxypropanoato)cadmium]

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Poly[di(μ_2 -2-hydroxypropanoato)cadmium]*Crystal data*

[Cd(C₃H₅O₃)₂]

$M_r = 290.54$

Orthorhombic, *Iba*2

$a = 10.238$ (2) Å

$b = 19.104$ (4) Å

$c = 9.463$ (2) Å

$V = 1850.8$ (7) Å³

$Z = 8$

$F(000) = 1136$

$D_x = 2.085$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9045 reflections

$\theta = 3.1$ – 28.5°

$\mu = 2.36$ mm⁻¹

$T = 298$ K

Block, clear light colourless

$0.3 \times 0.2 \times 0.2$ mm

Data collection

Bruker APEXII CCD

diffractometer

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2016)

$T_{\min} = 0.547$, $T_{\max} = 0.746$

9606 measured reflections

2230 independent reflections

2032 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 28.5^\circ$, $\theta_{\min} = 3.6^\circ$

$h = -13 \rightarrow 13$

$k = -20 \rightarrow 25$

$l = -10 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.065$

$S = 1.05$

2230 reflections

127 parameters

18 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2 + 2.8326P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.88$ e Å⁻³

$\Delta\rho_{\min} = -0.36$ e Å⁻³

Absolute structure: Flack x determined using

836 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)

Absolute structure parameter: 0.02 (2)

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. The H atoms (H3 and H6) bound to the O3 and O6 atoms were located from a difference-Fourier map. The O—H bond lengths were restrained by *DFIX* command to be 0.85 Å. The *DANG* command was used for H3 and H6 to restrain their orientation. Due to unresolved disorder of the methyl groups involving C3 and C6, the latter atoms were treated with *ISOR* commands.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cd1	0.45111 (3)	0.35060 (2)	0.51898 (10)	0.03129 (11)
O1	0.4657 (3)	0.3771 (3)	0.2739 (5)	0.0439 (10)
O2	0.3965 (5)	0.4340 (2)	0.0914 (5)	0.0512 (11)
O3	0.3247 (5)	0.4498 (2)	0.4582 (5)	0.0458 (10)
H3	0.332 (5)	0.4896 (11)	0.502 (3)	0.069*
O4	0.8064 (5)	0.2269 (3)	0.4374 (6)	0.0697 (18)
O5	0.6209 (4)	0.2835 (2)	0.4492 (5)	0.0461 (10)
O6	0.7918 (5)	0.1741 (3)	0.6884 (5)	0.0573 (13)
C1	0.3958 (5)	0.4223 (3)	0.2229 (6)	0.0344 (11)
C2	0.3081 (7)	0.4690 (4)	0.3124 (7)	0.0514 (16)
H2	0.3376	0.5174	0.3010	0.062*
C3	0.1765 (9)	0.4651 (6)	0.2688 (12)	0.086 (3)
H3A	0.1440	0.4186	0.2847	0.129*
H3B	0.1706	0.4761	0.1700	0.129*
H3C	0.1254	0.4980	0.3220	0.129*
C4	0.7044 (5)	0.2430 (3)	0.5010 (10)	0.0370 (17)
C5	0.6793 (7)	0.2125 (4)	0.6430 (6)	0.0497 (16)
H5	0.6663	0.2513	0.7093	0.060*
C6	0.5661 (9)	0.1679 (6)	0.6511 (14)	0.086 (3)
H6A	0.5790	0.1275	0.5923	0.129*
H6B	0.4906	0.1932	0.6193	0.129*
H6C	0.5532	0.1532	0.7472	0.129*
H6	0.843 (6)	0.202 (2)	0.732 (11)	0.12 (5)*

Atomic displacement parameters (Å²)

	<i>U</i> ¹¹	<i>U</i> ²²	<i>U</i> ³³	<i>U</i> ¹²	<i>U</i> ¹³	<i>U</i> ²³
Cd1	0.03011 (16)	0.03302 (16)	0.03074 (17)	−0.00204 (12)	−0.0024 (2)	−0.0012 (2)
O1	0.039 (2)	0.049 (2)	0.043 (2)	0.0177 (17)	0.004 (2)	0.004 (2)
O2	0.073 (3)	0.043 (2)	0.037 (2)	0.015 (2)	0.012 (2)	0.0042 (18)
O3	0.061 (3)	0.044 (2)	0.0328 (18)	0.011 (2)	−0.0017 (18)	−0.0084 (18)
O4	0.052 (3)	0.096 (4)	0.061 (3)	0.036 (3)	0.021 (2)	0.046 (3)
O5	0.045 (2)	0.053 (2)	0.040 (2)	0.0210 (19)	0.0023 (18)	0.0086 (19)
O6	0.059 (3)	0.084 (3)	0.029 (2)	0.036 (3)	−0.004 (2)	−0.002 (2)
C1	0.035 (3)	0.030 (2)	0.039 (3)	0.001 (2)	0.002 (2)	0.000 (2)
C2	0.060 (4)	0.058 (4)	0.037 (3)	0.021 (3)	0.002 (3)	−0.003 (3)
C3	0.070 (4)	0.111 (5)	0.078 (5)	0.025 (4)	−0.001 (4)	0.001 (4)
C4	0.033 (2)	0.037 (2)	0.041 (5)	0.0039 (18)	−0.002 (2)	−0.001 (2)
C5	0.052 (4)	0.069 (4)	0.028 (3)	0.029 (3)	0.002 (2)	0.007 (3)
C6	0.072 (4)	0.092 (4)	0.094 (5)	0.002 (4)	0.013 (4)	0.028 (4)

Geometric parameters (Å, °)

Cd1—O1 ⁱ	2.607 (5)	O6—Cd1 ^{iv}	2.335 (5)
Cd1—O1	2.379 (5)	O6—C5	1.432 (7)
Cd1—O2 ⁱ	2.333 (5)	O6—H6	0.848 (14)
Cd1—O3	2.366 (4)	C1—C2	1.523 (8)
Cd1—O4 ⁱⁱ	2.232 (5)	C2—H2	0.9800
Cd1—O5	2.259 (4)	C2—C3	1.411 (11)
Cd1—O6 ⁱⁱ	2.335 (5)	C3—H3A	0.9600
O1—Cd1 ⁱⁱⁱ	2.607 (5)	C3—H3B	0.9600
O1—C1	1.221 (7)	C3—H3C	0.9600
O2—Cd1 ⁱⁱⁱ	2.333 (5)	C4—C5	1.488 (10)
O2—C1	1.264 (7)	C5—H5	0.9800
O3—H3	0.868 (13)	C5—C6	1.440 (12)
O3—C2	1.438 (8)	C6—H6A	0.9600
O4—Cd1 ^{iv}	2.232 (5)	C6—H6B	0.9600
O4—C4	1.244 (8)	C6—H6C	0.9600
O5—C4	1.252 (7)		
O1—Cd1—O1 ⁱ	147.17 (16)	C5—O6—H6	109 (3)
O2 ⁱ —Cd1—O1 ⁱ	51.48 (14)	O1—C1—O2	120.7 (5)
O2 ⁱ —Cd1—O1	95.71 (16)	O1—C1—C2	122.7 (5)
O2 ⁱ —Cd1—O3	83.72 (18)	O2—C1—C2	116.6 (5)
O2 ⁱ —Cd1—O6 ⁱⁱ	113.78 (18)	O3—C2—C1	108.4 (5)
O3—Cd1—O1 ⁱ	104.37 (15)	O3—C2—H2	108.1
O3—Cd1—O1	68.10 (14)	C1—C2—H2	108.1
O4 ⁱⁱ —Cd1—O1	81.1 (2)	C3—C2—O3	112.3 (7)
O4 ⁱⁱ —Cd1—O1 ⁱ	131.71 (18)	C3—C2—C1	111.7 (7)
O4 ⁱⁱ —Cd1—O2 ⁱ	176.8 (2)	C3—C2—H2	108.1
O4 ⁱⁱ —Cd1—O3	94.8 (2)	C2—C3—H3A	109.5
O4 ⁱⁱ —Cd1—O5	91.90 (19)	C2—C3—H3B	109.5
O4 ⁱⁱ —Cd1—O6 ⁱⁱ	68.90 (17)	C2—C3—H3C	109.5
O5—Cd1—O1	77.72 (15)	H3A—C3—H3B	109.5
O5—Cd1—O1 ⁱ	97.42 (15)	H3A—C3—H3C	109.5
O5—Cd1—O2 ⁱ	87.63 (19)	H3B—C3—H3C	109.5
O5—Cd1—O3	143.58 (16)	O4—C4—O5	122.5 (8)
O5—Cd1—O6 ⁱⁱ	128.6 (2)	O4—C4—C5	119.0 (6)
O6 ⁱⁱ —Cd1—O1	139.13 (16)	O5—C4—C5	118.5 (6)
O6 ⁱⁱ —Cd1—O1 ⁱ	68.43 (15)	O6—C5—C4	109.5 (5)
O6 ⁱⁱ —Cd1—O3	86.9 (2)	O6—C5—H5	107.7
Cd1—O1—Cd1 ⁱⁱⁱ	151.79 (19)	O6—C5—C6	109.2 (7)
C1—O1—Cd1 ⁱⁱⁱ	87.9 (4)	C4—C5—H5	107.7
C1—O1—Cd1	119.9 (4)	C6—C5—C4	114.8 (7)
C1—O2—Cd1 ⁱⁱⁱ	99.9 (4)	C6—C5—H5	107.7
Cd1—O3—H3	123 (2)	C5—C6—H6A	109.5
C2—O3—Cd1	120.1 (4)	C5—C6—H6B	109.5
C2—O3—H3	104 (2)	C5—C6—H6C	109.5
C4—O4—Cd1 ^{iv}	123.7 (5)	H6A—C6—H6B	109.5

C4—O5—Cd1	139.6 (5)	H6A—C6—H6C	109.5
Cd1 ^{iv} —O6—H6	92 (8)	H6B—C6—H6C	109.5
C5—O6—Cd1 ^{iv}	117.4 (3)		
Cd1—O1—C1—O2	-175.5 (4)	Cd1—O5—C4—C5	19.2 (10)
Cd1 ⁱⁱⁱ —O1—C1—O2	-0.4 (6)	Cd1 ^{iv} —O6—C5—C4	-13.4 (8)
Cd1 ⁱⁱⁱ —O1—C1—C2	-178.1 (5)	Cd1 ^{iv} —O6—C5—C6	113.0 (7)
Cd1—O1—C1—C2	6.7 (8)	O1—C1—C2—O3	0.2 (9)
Cd1 ⁱⁱⁱ —O2—C1—O1	0.5 (6)	O1—C1—C2—C3	-124.1 (8)
Cd1 ⁱⁱⁱ —O2—C1—C2	178.3 (5)	O2—C1—C2—O3	-177.6 (6)
Cd1—O3—C2—C1	-7.1 (7)	O2—C1—C2—C3	58.1 (9)
Cd1—O3—C2—C3	116.8 (7)	O4—C4—C5—O6	6.9 (9)
Cd1 ^{iv} —O4—C4—O5	-175.6 (5)	O4—C4—C5—C6	-116.2 (8)
Cd1 ^{iv} —O4—C4—C5	3.4 (9)	O5—C4—C5—O6	-174.0 (6)
Cd1—O5—C4—O4	-161.7 (6)	O5—C4—C5—C6	62.8 (9)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 \cdots O2 ^v	0.87 (1)	1.81 (2)	2.657 (6)	163 (5)
C5—H5 \cdots O4 ^{vi}	0.98	2.22	3.021 (8)	139
O6—H6 \cdots O5 ^{vi}	0.85 (1)	2.11 (9)	2.747 (7)	132 (11)

Symmetry codes: (v) $x, -y+1, z+1/2$; (vi) $-x+3/2, -y+1/2, z+1/2$.