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Crystal structure of 4-sulfamoylanilinium dihydrogen phosphate

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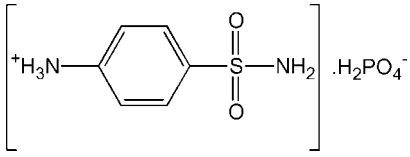
In the crystal structure of the title molecular salt, $C_6H_9N_2O_2S^+\cdot H_2PO_4^-$, the sulfomylalinium cations and the dihydrogen phosphate anions form independent [100] chains through N_s—H···O (s = sulfamoyl) and O—H···O hydrogen bonds, respectively. The chains are cross-linked by N_a—H···O (a = amine) hydrogen bonds, generating (010) sheets. Two C—H···O hydrogen bonds involving diametrically opposite C atoms in the benzene ring of the cation as donors form chains parallel to [202] in which P=O and P—OH groups are acceptors. Together, these interactions lead to a three-dimensional network.

Keywords: crystal structure; 4-sulfamoylanilinium; dihydrogen phosphate; hydrogen bonding; sulfanilamide derivatives; sulfa drugs.

CCDC reference: 1016950

1. Related literature

For background to sulfa drugs, see: Topacli & Kesimli (2001); Gelbrich *et al.* (2007). For structures of other molecular salts of the same cation, see: Anitha *et al.* (2013); Ravikumar *et al.* (2013); Pandiarajan *et al.* (2011); Zaouali Zgolli *et al.* (2010); Gelbrich *et al.* (2008); Chatterjee *et al.* (1981).



2. Experimental

2.1. Crystal data

$C_6H_9N_2O_2S^+\cdot H_2O_4P^-$ M_r = 270.20

Monoclinic, *Pc*
a = 4.8041 (7) Å
b = 10.8564 (15) Å
c = 10.3862 (15) Å
β = 101.067 (2)°
V = 531.62 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 294 K
0.28 × 0.18 × 0.10 mm

2.2. Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.94, T_{max} = 0.99
6033 measured reflections
2512 independent reflections
2502 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.055
S = 1.06
2512 reflections
174 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: Flack x determined using 1209 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons & Flack, 2004)
Absolute structure parameter: 0.069 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O5^{i}	0.87 (3)	1.89 (3)	2.760 (2)	174 (3)
N1—H1B···O6^{ii}	0.90 (4)	1.96 (4)	2.856 (2)	170 (3)
N1—H1C···O6^{iii}	0.86 (4)	2.00 (4)	2.855 (2)	175 (3)
N2—H2A···O3^{iv}	0.86 (3)	2.25 (4)	3.076 (3)	161 (3)
N2—H2B···O2^{v}	0.88 (4)	2.10 (4)	2.886 (3)	149 (3)
O3—H3A···O1^{vi}	0.78 (2)	1.97 (2)	2.750 (3)	176 (4)
O4—H4A···O5^{vi}	0.80 (3)	1.76 (3)	2.500 (2)	154 (5)
C3—H3···O6^{v}	0.93	2.59	3.283 (3)	132
C6—H6···O4^{i}	0.93	2.42	3.288 (3)	156

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2013*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7251).

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supporting information

Acta Cryst. (2014). E**70**, o997–o998 [doi:10.1107/S1600536814017462]

Crystal structure of 4-sulfamoylanilinium dihydrogen phosphate

C. Muthuselvi, N. Mala, N. Srinivasan, S. Pandiarajan and R. V. Krishnakumar

S1. Comment

Sulfanilamide (4-sulfamoyl aniline) and its derivatives known as sulfa drugs were used to treat bacterial infections (e.g. Gelbrich *et al.*, 2007). Sulfa drugs were successfully deployed as chemotherapeutic agents (Topacli & Kesimli, 2001).

The perchlorate (Anitha, *et al.*, 2013); nitrate, (Pandiarajan, *et al.*, 2011); sulfate (Ravikumar *et al.*, 2013) and hydrogen chloride (Zaouali Zgolli *et al.*, 2010) complexes of sulfanilamide were already been reported. The present report on phosphate salt of sulfanilamide is part of a series of x-ray investigations being carried out on sulfanilamide-inorganic acid complexes.

The crystal structure of the title compound contains a cation with a protonated amino group and the dihydrogen phosphate anion $C_6H_9N_2O_2S^+.H_2PO_4^-$(Fig. 1). The crystal structure features a three-dimensional hydrogen bonding network formed through N—H···O, O—H···O and C—H···O hydrogen bonds. The sulfomylalinium cations and the phosphate anions form independent chains through N—H···O [N1—H1A···O5 ; N1—H1B··· O6 ; N1—H1C···O6 ; N2—H2A···O3 and N2—H2B···O2]. O—H···O [viz., O3—H3A,,,O1 (-1+x, y, z) and O4—H4A···O5 (-1+x, y, z)] hydrogen bonds along the shortest a-axis. Two C—H···O hydrogen bonds viz., C3—H3···O6 (1+x, y, z); C6—H6···O4 (x, y, -1+z) involving diametricaly opposite aryl carbon atoms in the benzene ring of the 4-sulfomylanilinum cation act as donors to form chains parallel to (202) in which P = O and P—OH are acceptors . This one dimensional chain is extended into a two dimensional layer parallel to the ac-plane through the same P—OH linking the adjacent phosophate anion and another P=O.

The overall picture of the complex intermolecular interactions may be visualized in terms of sinple graph-set motifs; viz., $R_2^2(9)$ motif generated through O3—H3A···O1 (-1+x, y, z) and C3—H3···O6 (1+x, y, z) hydrogen bonds, two $R_6^6(26)$ motifs generated involving O3—H3A···O1 (-1+x, y, z) and C6—H6···O4(x, y, -1+z); O4—H4A···O5(-1+x,y,z) hydrogen bond interactions (Fig. 2).

S2. Synthesis and crystallization

The title compound was synthesised by heating a mixture of sulphanilamide (3.4 g) and phosphoric acid (0.5 ml of 98% concentration) in 20 ml of water as the stoichiometric ratio of 2:1 (at 60°C) under reflux for 1 h. The solution upon allowing to evaporate under room temperature yielded colourless needles of the title salt.

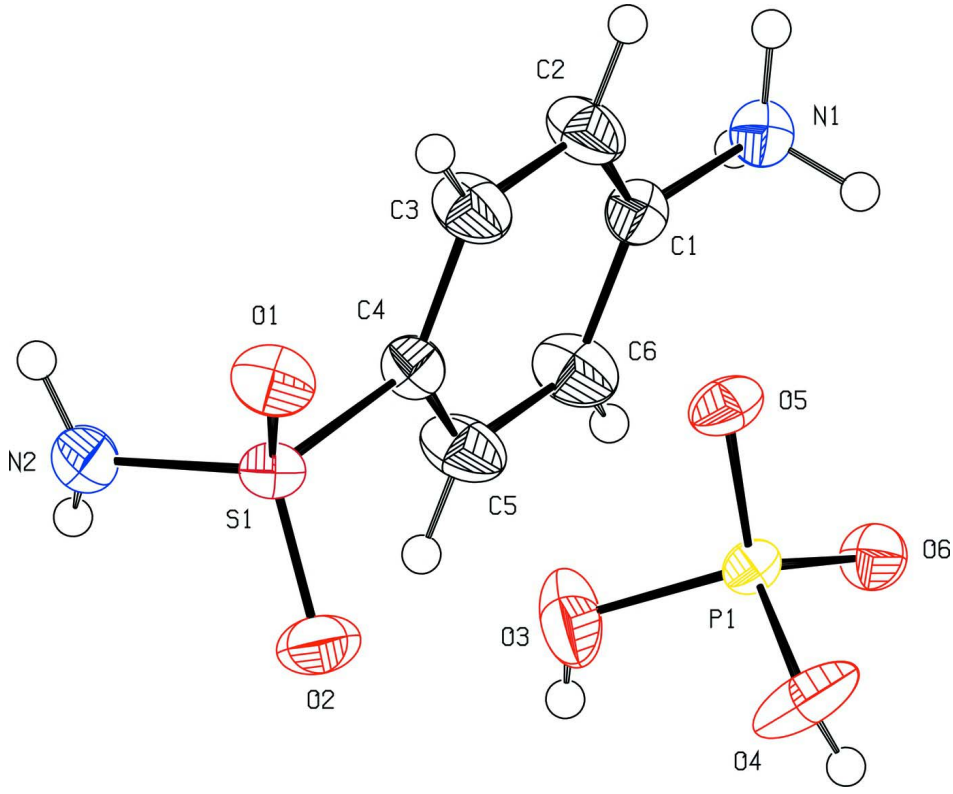

Figure 1

Molecular structure of (I) showing displacement ellipsoids drawn at the 50% probability level.

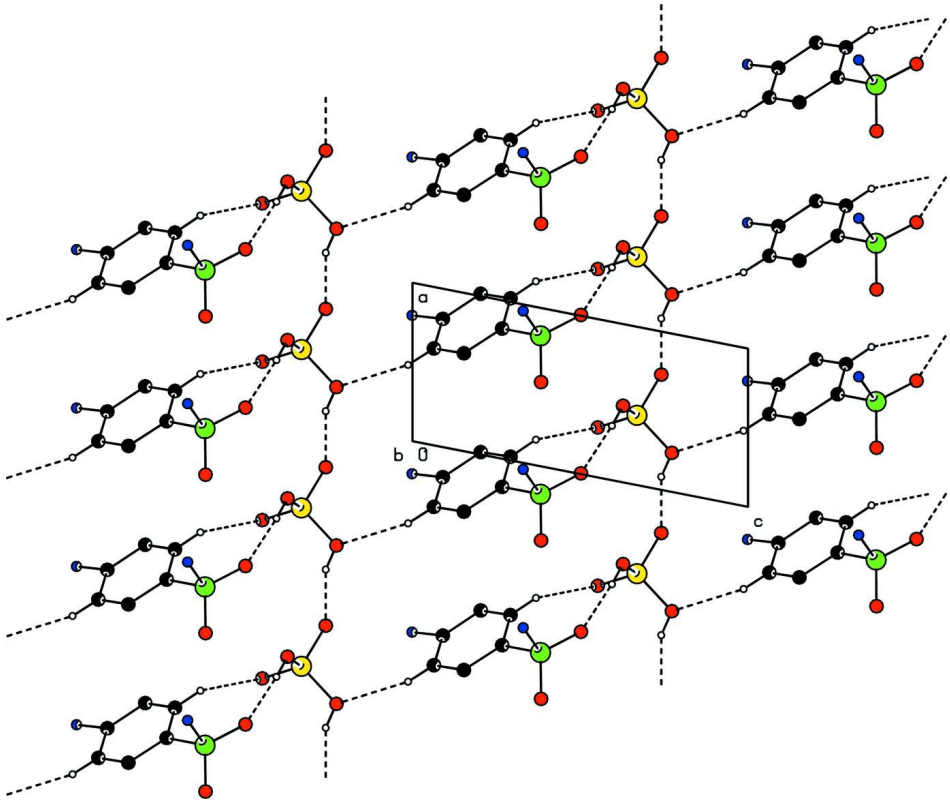

Figure 2

Hydrogen-bonding environment of the dihydrogen phosphate anion viewed along the b-axis. Other hydrogen bonds involving N atom and non-participating H atoms have been omitted for clarity.

4-Sulfamoylanilinium dihydrogen phosphate

Crystal data

$C_6H_9N_2O_2S^+·H_2O_4P^-$
M_r = 270.20
Monoclinic, *Pc*
a = 4.8041 (7) Å
b = 10.8564 (15) Å
c = 10.3862 (15) Å
β = 101.067 (2)°
V = 531.62 (13) Å^3
Z = 2
$F(000)$ = 280
D_x = 1.688 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
μ = 0.47 mm^{-1}
T = 294 K
Needle, colourless
0.28 × 0.18 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.94, T_{max} = 0.99
6033 measured reflections
2512 independent reflections
2502 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 28.3°, θ_{min} = 1.9°
h = −6→6
k = −14→14
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.055$
$S = 1.06$
2512 reflections
174 parameters
4 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 0.0212P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL2013* (Sheldrick, 2013), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.099 (8)
Absolute structure: Flack *x* determined using 1209 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons & Flack, 2004)
Absolute structure parameter: 0.069 (16)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8098 (4)	0.14923 (16)	0.09044 (19)	0.0265 (3)
C2	1.0153 (5)	0.1497 (2)	0.2035 (2)	0.0365 (5)
H2	1.1473	0.0862	0.2199	0.044*
C3	1.0231 (5)	0.2455 (2)	0.2922 (2)	0.0364 (5)
H3	1.1623	0.2475	0.3680	0.044*
C4	0.8220 (4)	0.33816 (17)	0.26728 (19)	0.0271 (4)
C5	0.6183 (5)	0.3379 (2)	0.1532 (2)	0.0381 (5)
H5	0.4862	0.4013	0.1368	0.046*
C6	0.6116 (5)	0.2433 (2)	0.0639 (2)	0.0379 (5)
H6	0.4759	0.2426	−0.0132	0.046*
N1	0.7938 (4)	0.04600 (14)	−0.00122 (17)	0.0271 (3)
N2	0.9589 (4)	0.57959 (17)	0.3299 (2)	0.0352 (4)
O6	0.3160 (3)	0.08705 (13)	0.55239 (13)	0.0288 (3)
O5	0.7279 (3)	0.12348 (14)	0.74157 (13)	0.0298 (3)
O3	0.4839 (5)	0.30112 (15)	0.62757 (19)	0.0504 (5)
O4	0.2474 (3)	0.1676 (2)	0.77341 (17)	0.0506 (5)
O1	1.0051 (4)	0.42220 (15)	0.50222 (15)	0.0364 (3)
O2	0.5320 (3)	0.48636 (17)	0.38326 (17)	0.0400 (4)
P1	0.44104 (7)	0.16369 (4)	0.66908 (4)	0.02196 (12)
S1	0.82319 (8)	0.45858 (4)	0.38175 (5)	0.02634 (13)
H1A	0.760 (7)	0.072 (3)	−0.082 (3)	0.039 (7)*
H1B	0.639 (8)	0.001 (3)	0.006 (3)	0.046 (8)*
H1C	0.946 (8)	0.003 (3)	0.018 (3)	0.046 (8)*
H2A	0.855 (8)	0.611 (3)	0.261 (3)	0.050 (9)*
H2B	1.143 (8)	0.578 (3)	0.333 (3)	0.042 (8)*
H3A	0.343 (6)	0.334 (3)	0.594 (4)	0.052 (10)*
H4A	0.083 (6)	0.164 (4)	0.742 (4)	0.080 (14)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0272 (8)	0.0273 (8)	0.0245 (8)	−0.0007 (7)	0.0037 (7)	0.0024 (6)
C2	0.0368 (11)	0.0306 (9)	0.0372 (10)	0.0102 (8)	−0.0053 (9)	−0.0037 (8)
C3	0.0362 (11)	0.0335 (10)	0.0336 (10)	0.0084 (8)	−0.0084 (8)	−0.0030 (8)
C4	0.0288 (9)	0.0255 (8)	0.0270 (8)	0.0011 (6)	0.0052 (7)	0.0007 (6)
C5	0.0395 (11)	0.0363 (10)	0.0344 (10)	0.0145 (8)	−0.0036 (9)	0.0009 (8)
C6	0.0402 (11)	0.0400 (11)	0.0288 (9)	0.0105 (8)	−0.0053 (7)	−0.0011 (8)
N1	0.0287 (8)	0.0275 (7)	0.0243 (7)	−0.0007 (6)	0.0035 (6)	0.0016 (6)
N2	0.0310 (9)	0.0275 (8)	0.0462 (10)	0.0021 (7)	0.0050 (7)	0.0048 (7)
O6	0.0273 (6)	0.0314 (7)	0.0262 (6)	−0.0009 (5)	0.0014 (5)	−0.0035 (5)
O5	0.0189 (6)	0.0402 (8)	0.0290 (6)	0.0020 (5)	0.0017 (5)	0.0063 (6)
O3	0.0514 (10)	0.0261 (7)	0.0608 (11)	−0.0079 (7)	−0.0212 (8)	0.0091 (7)
O4	0.0195 (7)	0.0980 (15)	0.0357 (9)	−0.0113 (7)	0.0086 (6)	−0.0243 (8)
O1	0.0389 (8)	0.0414 (8)	0.0276 (6)	0.0079 (7)	0.0031 (6)	0.0013 (6)
O2	0.0250 (7)	0.0473 (8)	0.0492 (9)	0.0050 (6)	0.0107 (6)	−0.0059 (7)
P1	0.01703 (19)	0.0251 (2)	0.0230 (2)	−0.00087 (15)	0.00186 (14)	−0.00013 (15)
S1	0.0230 (2)	0.0275 (2)	0.0287 (2)	0.00361 (16)	0.00527 (14)	0.00025 (15)

Geometric parameters (Å, º)

C1—C2	1.381 (3)	N1—H1B	0.90 (4)
C1—C6	1.387 (3)	N1—H1C	0.86 (4)
C1—N1	1.463 (2)	N2—S1	1.6048 (19)
C2—C3	1.386 (3)	N2—H2A	0.86 (3)
C2—H2	0.9300	N2—H2B	0.88 (4)
C3—C4	1.384 (3)	O6—P1	1.4975 (14)
C3—H3	0.9300	O5—P1	1.5027 (13)
C4—C5	1.384 (3)	O3—P1	1.5774 (17)
C4—S1	1.7665 (19)	O3—H3A	0.78 (2)
C5—C6	1.380 (3)	O4—P1	1.5583 (16)
C5—H5	0.9300	O4—H4A	0.80 (3)
C6—H6	0.9300	O1—S1	1.4371 (16)
N1—H1A	0.87 (3)	O2—S1	1.4339 (15)
C2—C1—C6	121.19 (18)	C1—N1—H1C	109 (2)
C2—C1—N1	119.70 (18)	H1A—N1—H1C	113 (3)
C6—C1—N1	119.08 (18)	H1B—N1—H1C	111 (3)
C1—C2—C3	119.45 (19)	S1—N2—H2A	113 (2)
C1—C2—H2	120.3	S1—N2—H2B	116 (2)
C3—C2—H2	120.3	H2A—N2—H2B	117 (3)
C4—C3—C2	119.49 (19)	P1—O3—H3A	114 (3)
C4—C3—H3	120.3	P1—O4—H4A	113 (3)
C2—C3—H3	120.3	O6—P1—O5	115.53 (9)
C3—C4—C5	120.79 (18)	O6—P1—O4	112.17 (9)
C3—C4—S1	120.00 (16)	O5—P1—O4	105.78 (9)
C5—C4—S1	119.21 (15)	O6—P1—O3	110.94 (9)

C6—C5—C4	119.90 (19)	O5—P1—O3	104.87 (10)
C6—C5—H5	120.0	O4—P1—O3	106.92 (13)
C4—C5—H5	120.0	O2—S1—O1	118.66 (10)
C5—C6—C1	119.15 (19)	O2—S1—N2	106.94 (11)
C5—C6—H6	120.4	O1—S1—N2	107.39 (11)
C1—C6—H6	120.4	O2—S1—C4	106.61 (10)
C1—N1—H1A	110.5 (19)	O1—S1—C4	107.79 (10)
C1—N1—H1B	108 (2)	N2—S1—C4	109.21 (10)
H1A—N1—H1B	105 (3)		

C6—C1—C2—C3	0.3 (4)	C2—C1—C6—C5	−0.9 (3)
N1—C1—C2—C3	−177.7 (2)	N1—C1—C6—C5	177.0 (2)
C1—C2—C3—C4	1.0 (4)	C3—C4—S1—O2	−142.11 (19)
C2—C3—C4—C5	−1.7 (4)	C5—C4—S1—O2	37.8 (2)
C2—C3—C4—S1	178.21 (19)	C3—C4—S1—O1	−13.7 (2)
C3—C4—C5—C6	1.1 (4)	C5—C4—S1—O1	166.24 (18)
S1—C4—C5—C6	−178.88 (19)	C3—C4—S1—N2	102.68 (19)
C4—C5—C6—C1	0.3 (4)	C5—C4—S1—N2	−77.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O5^{i}	0.87 (3)	1.89 (3)	2.760 (2)	174 (3)
N1—H1*B*···O6^{ii}	0.90 (4)	1.96 (4)	2.856 (2)	170 (3)
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N2—H2*B*···O2^{v}	0.88 (4)	2.10 (4)	2.886 (3)	149 (3)
O3—H3*A*···O1^{vi}	0.78 (2)	1.97 (2)	2.750 (3)	176 (4)
O4—H4*A*···O5^{vi}	0.80 (3)	1.76 (3)	2.500 (2)	154 (5)
C3—H3···O6^{v}	0.93	2.59	3.283 (3)	132
C6—H6···O4^{i}	0.93	2.42	3.288 (3)	156

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