Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Uronium 3-carboxy-4-hydroxybenzenesulfonate

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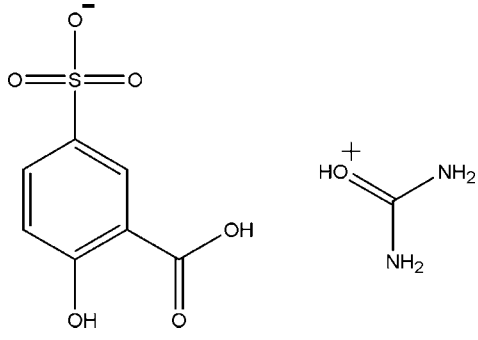
Received 21 September 2013; accepted 25 October 2013

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.110; data-to-parameter ratio = 22.0.

In the title compound, $CH_5N_2O^+\cdot C_7H_5O_6S^-$, the dihedral angle between the benzene ring and the mean plane of the uronium cation is 76.02 (8)°. The carboxyl group in the anion is twisted by 1.47 (9)° from the benzene ring. In the crystal, the cation is linked to the anion by weak O—H···O and N—H···O hydrogen bonds and π–π interactions [centroid–centroid distance = 3.8859 (8) Å], forming a three-dimensional network.

Related literature

For the biological activity of urea derivatives, see: Sliskovic *et al.* (1999); Furlong *et al.* (2000); Houghton *et al.* (1995). For related structures, see: Krishnakumar *et al.* (2012); Sudhahar *et al.* (2013); Worsham & Busing (1969); Nelyubina *et al.* (2007).



Experimental

Crystal data

$CH_5N_2O^+\cdot C_7H_5O_6S^-$
M_r = 278.24
Triclinic, $P\overline{1}$
a = 7.2248 (4) Å
b = 8.7718 (5) Å
c = 10.1549 (5) Å
α = 85.504 (3)°
β = 71.087 (2)°
γ = 68.659 (2)°
V = 566.50 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 295 K
0.30 × 0.24 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.911, T_{max} = 0.939
14246 measured reflections
4079 independent reflections
3432 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.110
S = 1.05
4079 reflections
185 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2	0.82 (1)	1.83 (1)	2.5966 (14)	154 (18)
O7—H7···O6	0.82 (1)	1.85 (1)	2.6629 (14)	169 (18)
N1—H1A···O4	0.84 (1)	1.96 (1)	2.7979 (16)	175 (16)
O1—H1···O5^{i}	0.83 (1)	1.84 (1)	2.6511 (12)	167 (18)
N1—H1B···O5^{ii}	0.85 (1)	1.95 (1)	2.7976 (16)	178 (15)
N2—H2B···O6^{ii}	0.86 (1)	2.22 (1)	3.0712 (16)	172 (15)
N2—H2A···O3^{iii}	0.85 (1)	2.19 (1)	3.0347 (16)	173 (15)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

The authors thank the SAIF, IIT Madras, for the data collection. MKK thanks the Council of Scientific and Industrial Research, New Delhi, India, for providing financial support [project No. 03 (1200)/11/EMR-II].

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2485).

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supporting information

Acta Cryst. (2013). E**69**, o1725 [doi:10.1107/S1600536813029474]

Uronium 3-carboxy-4-hydroxybenzenesulfonate

A. Silambarasan, M. Krishna Kumar, G. Chakkaravarthi, R. Mohan Kumar and P. R. Umarani

S1. Comment

Urea derivatives are known to exhibit anticholesterolemic, herbicidal and antitumor activities (Sliskovic *et al.*, 1999; Furlong *et al.*, 2000; Houghton *et al.*, 1995). Herein, we report the crystal structure of the title compound (I, Fig. 1). The asymmetric unit consists of one $CH_5N_2O^+$ cation and one $C_7H_5O_6S^-$ anion. In the title compound, the geometric parameters of the uronium cation (Worsham & Busing, 1969; Nelyubina *et al.*, 2007) and 3-carboxy-4-hydroxybenzene-sulfonate anion (Krishnakumar *et al.*, 2012; Sudhahar *et al.*, 2013) are comparable with the reported structures.

The dihedral angle between the benzene ring and the mean plane of the uronium fragment is 76.02 (8)°. In the anion, the carboxyl group makes the dihedral angle of 1.47 (9)° with the benzene ring. In the molecular structure, cation and anion are linked by O—H···O and N—H···O hydrogen bonds, and the molecular conformation of the anion is controlled by weak O—H···O hydrogen bond (Fig. 1). The crystal structure exhibits weak intermolecular O—H···O, N—H···O (Table 1 and Fig. 2) and $\pi\cdots\pi$ [Cg1···Cg1^{i} distance: 3.8859 (8) Å; (*i*) *x*, 1-*y*, 1-*z*; Cg1 is the centroid of the ring C1···C6] interactions, to form a three dimensional network.

S2. Experimental

Urea (CH_4N_2O, 3.003 g) and 5-sulfosalicylic acid ($C_7H_6O_6S$, 12.711 g, 1:1 ratio) were mixed in water and the resulting solution was allowed for slow evaporation at room temperature. Good quality crystals, suitable for X-ray intensity data collection, were collected after 2 weeks.

S3. Refinement

H atoms bonded to O and N heteroatoms were placed from difference maps and refined with free coordinates, although restrictions were applied on bond lengths: N—H = 0.86 (1) Å and O—H = 0.82 (1) Å. Isotropic displacement parameters for these H atoms were calculated as U_{iso}(H) = 1.5U_{eq}(O) for O—H bonds and U_{iso}(H) = 1.2U_{eq}(N) for N—H bonds. H atoms for CH groups were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

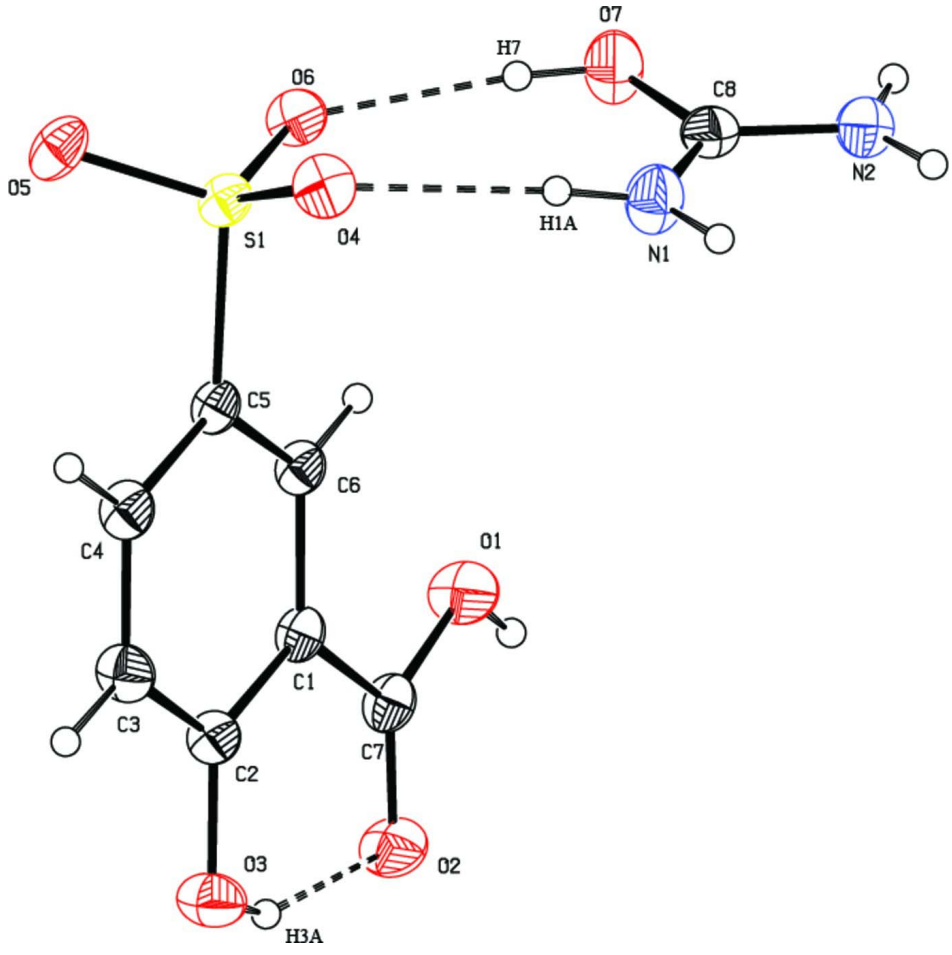

Figure 1

The molecular structure of (I), with 30% probability displacement ellipsoids for non-H atoms. Intramolecular hydrogen bonds are shown as dashed lines.

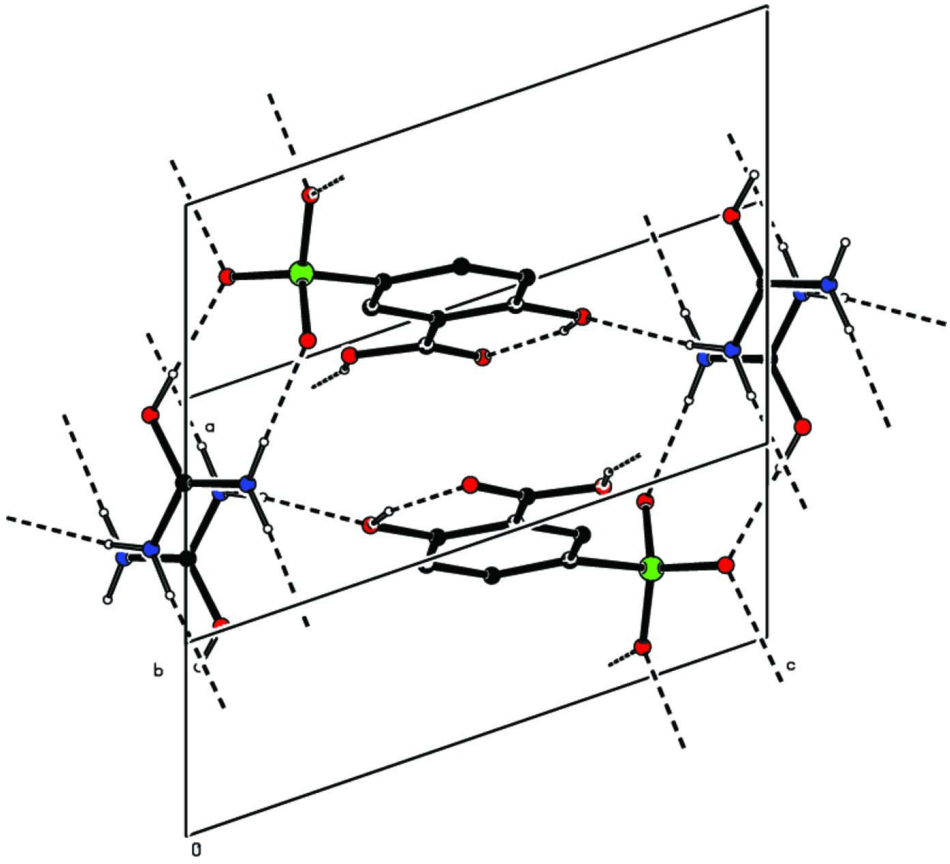

Figure 2

The packing of (I), viewed down *b* axis. Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Uronium 3-carboxy-4-hydroxybenzenesulfonate

Crystal data

$CH_5N_2O^+ \cdot C_7H_5O_6S^-$
$M_r = 278.24$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.2248$ (4) Å
$b = 8.7718$ (5) Å
$c = 10.1549$ (5) Å
$\alpha = 85.504$ (3)°
$\beta = 71.087$ (2)°
$\gamma = 68.659$ (2)°
$V = 566.50$ (5) Å^3
$Z = 2$
$F(000) = 288$
$D_x = 1.631$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7318 reflections
$\theta = 2.1$–32.8°
$\mu = 0.32$ mm^{-1}
$T = 295$ K
Block, colourless
0.30 × 0.24 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.911$, $T_{max} = 0.939$
14246 measured reflections
4079 independent reflections
3432 reflections with $I > 2\sigma(I)$

R_{int} = 0.028
θ_{max} = 33.6°, θ_{min} = 2.1°
h = −11→11
k = −13→13
l = −14→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.110
S = 1.05
4079 reflections
185 parameters
7 restraints
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.1089P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Extinction correction: *SHELXL97*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.105 (7)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.22603 (17)	0.52320 (13)	0.56728 (12)	0.0301 (2)
C2	0.28443 (18)	0.45643 (14)	0.43261 (12)	0.0324 (2)
C3	0.3015 (2)	0.29542 (15)	0.41388 (13)	0.0376 (3)
H3	0.3363	0.2524	0.3246	0.045*
C4	0.26724 (19)	0.20039 (14)	0.52622 (13)	0.0347 (2)
H4	0.2801	0.0928	0.5132	0.042*
C5	0.21286 (17)	0.26557 (12)	0.66054 (12)	0.0288 (2)
C6	0.19033 (17)	0.42571 (13)	0.68131 (12)	0.0301 (2)
H6	0.1514	0.4687	0.7712	0.036*
C7	0.20231 (19)	0.69528 (14)	0.58615 (14)	0.0343 (2)
C8	0.5164 (2)	0.26399 (16)	1.00316 (13)	0.0378 (3)
N1	0.6134 (2)	0.16284 (17)	0.89415 (14)	0.0474 (3)
N2	0.6150 (2)	0.32507 (16)	1.05987 (13)	0.0431 (3)
O1	0.1437 (2)	0.74360 (11)	0.71788 (11)	0.0490 (3)
O2	0.23382 (18)	0.78366 (11)	0.48953 (11)	0.0473 (2)
O3	0.32586 (18)	0.54310 (12)	0.31773 (10)	0.0459 (2)
O4	0.39451 (15)	0.02256 (11)	0.79035 (11)	0.0417 (2)
O5	0.05090 (15)	0.05458 (11)	0.78561 (11)	0.0417 (2)
O6	0.08949 (15)	0.24191 (11)	0.92865 (9)	0.0380 (2)
O7	0.31385 (16)	0.31219 (15)	1.06119 (12)	0.0513 (3)
S1	0.18626 (4)	0.13680 (3)	0.80111 (3)	0.03090 (10)
H1A	0.551 (2)	0.1153 (18)	0.8654 (16)	0.037*
H1B	0.7463 (14)	0.1303 (19)	0.8589 (15)	0.037*
H2A	0.543 (2)	0.3875 (17)	1.1326 (12)	0.037*
H2B	0.7490 (14)	0.2929 (19)	1.0274 (16)	0.037*
H1	0.129 (3)	0.8404 (12)	0.7270 (18)	0.046*
H3A	0.303 (3)	0.6342 (14)	0.3493 (18)	0.046*
H7	0.253 (3)	0.278 (2)	1.0221 (17)	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0310 (5)	0.0228 (4)	0.0369 (6)	−0.0100 (4)	−0.0100 (4)	−0.0027 (4)
C2	0.0333 (5)	0.0287 (5)	0.0338 (6)	−0.0102 (4)	−0.0095 (4)	−0.0006 (4)
C3	0.0466 (6)	0.0317 (5)	0.0328 (6)	−0.0125 (5)	−0.0105 (5)	−0.0068 (4)
C4	0.0412 (6)	0.0245 (5)	0.0382 (6)	−0.0114 (4)	−0.0110 (5)	−0.0070 (4)
C5	0.0300 (4)	0.0222 (4)	0.0341 (5)	−0.0099 (4)	−0.0083 (4)	−0.0037 (4)
C6	0.0342 (5)	0.0237 (4)	0.0327 (5)	−0.0115 (4)	−0.0082 (4)	−0.0053 (4)
C7	0.0361 (5)	0.0244 (5)	0.0434 (6)	−0.0117 (4)	−0.0123 (5)	−0.0015 (4)
C8	0.0418 (6)	0.0372 (6)	0.0358 (6)	−0.0163 (5)	−0.0127 (5)	0.0067 (5)
N1	0.0385 (6)	0.0541 (7)	0.0480 (7)	−0.0165 (5)	−0.0089 (5)	−0.0101 (5)
N2	0.0458 (6)	0.0439 (6)	0.0433 (6)	−0.0191 (5)	−0.0152 (5)	0.0014 (5)
O1	0.0766 (7)	0.0265 (4)	0.0465 (5)	−0.0241 (5)	−0.0149 (5)	−0.0053 (4)
O2	0.0630 (6)	0.0306 (4)	0.0512 (6)	−0.0213 (4)	−0.0176 (5)	0.0064 (4)
O3	0.0634 (6)	0.0374 (5)	0.0356 (5)	−0.0196 (5)	−0.0130 (4)	0.0036 (4)
O4	0.0428 (5)	0.0284 (4)	0.0487 (5)	−0.0056 (4)	−0.0155 (4)	−0.0018 (4)
O5	0.0445 (5)	0.0310 (4)	0.0515 (5)	−0.0216 (4)	−0.0064 (4)	−0.0068 (4)
O6	0.0445 (5)	0.0331 (4)	0.0337 (4)	−0.0155 (4)	−0.0052 (3)	−0.0066 (3)
O7	0.0402 (5)	0.0632 (7)	0.0490 (6)	−0.0176 (5)	−0.0095 (4)	−0.0137 (5)
S1	0.03538 (15)	0.02207 (13)	0.03448 (16)	−0.01185 (10)	−0.00736 (11)	−0.00307 (9)

Geometric parameters (Å, º)

C1—C6	1.3966 (16)	C8—O7	1.3029 (16)
C1—C2	1.4010 (16)	C8—N1	1.3047 (17)
C1—C7	1.4766 (15)	C8—N2	1.3104 (18)
C2—O3	1.3523 (15)	N1—H1A	0.841 (9)
C2—C3	1.3951 (17)	N1—H1B	0.852 (9)
C3—C4	1.3695 (18)	N2—H2A	0.850 (9)
C3—H3	0.9300	N2—H2B	0.858 (9)
C4—C5	1.3976 (15)	O1—H1	0.826 (9)
C4—H4	0.9300	O3—H3A	0.824 (9)
C5—C6	1.3791 (14)	O4—S1	1.4468 (10)
C5—S1	1.7544 (12)	O5—S1	1.4602 (9)
C6—H6	0.9300	O6—S1	1.4654 (9)
C7—O2	1.2165 (16)	O7—H7	0.821 (9)
C7—O1	1.3189 (16)		
C6—C1—C2	119.27 (10)	O1—C7—C1	113.41 (11)
C6—C1—C7	121.23 (10)	O7—C8—N1	121.64 (12)
C2—C1—C7	119.50 (11)	O7—C8—N2	115.88 (12)
O3—C2—C3	117.87 (11)	N1—C8—N2	122.48 (13)
O3—C2—C1	122.19 (10)	C8—N1—H1A	121.2 (11)
C3—C2—C1	119.95 (11)	C8—N1—H1B	120.5 (11)
C4—C3—C2	120.39 (11)	H1A—N1—H1B	117.5 (15)
C4—C3—H3	119.8	C8—N2—H2A	117.9 (11)
C2—C3—H3	119.8	C8—N2—H2B	118.8 (11)

C3—C4—C5	119.82 (10)	H2A—N2—H2B	123.0 (15)
C3—C4—H4	120.1	C7—O1—H1	112.4 (12)
C5—C4—H4	120.1	C2—O3—H3A	103.8 (13)
C6—C5—C4	120.59 (11)	C8—O7—H7	115.2 (13)
C6—C5—S1	120.71 (9)	O4—S1—O5	112.06 (6)
C4—C5—S1	118.63 (8)	O4—S1—O6	112.49 (6)
C5—C6—C1	119.95 (10)	O5—S1—O6	111.64 (5)
C5—C6—H6	120.0	O4—S1—C5	106.92 (6)
C1—C6—H6	120.0	O5—S1—C5	106.35 (6)
O2—C7—O1	123.37 (11)	O6—S1—C5	106.94 (5)
O2—C7—C1	123.22 (11)		

C6—C1—C2—O3	−178.47 (11)	C7—C1—C6—C5	179.73 (10)
C7—C1—C2—O3	1.84 (18)	C6—C1—C7—O2	179.30 (12)
C6—C1—C2—C3	1.51 (17)	C2—C1—C7—O2	−1.02 (18)
C7—C1—C2—C3	−178.18 (11)	C6—C1—C7—O1	−0.71 (16)
O3—C2—C3—C4	178.12 (12)	C2—C1—C7—O1	178.98 (11)
C1—C2—C3—C4	−1.86 (19)	C6—C5—S1—O4	−106.66 (10)
C2—C3—C4—C5	0.64 (19)	C4—C5—S1—O4	70.32 (10)
C3—C4—C5—C6	0.94 (18)	C6—C5—S1—O5	133.45 (9)
C3—C4—C5—S1	−176.05 (10)	C4—C5—S1—O5	−49.56 (10)
C4—C5—C6—C1	−1.28 (17)	C6—C5—S1—O6	14.04 (11)
S1—C5—C6—C1	175.65 (8)	C4—C5—S1—O6	−168.98 (9)
C2—C1—C6—C5	0.05 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2	0.82 (1)	1.83 (1)	2.5966 (14)	154 (18)
O7—H7···O6	0.82 (1)	1.85 (1)	2.6629 (14)	169 (18)
N1—H1*A*···O4	0.84 (1)	1.96 (1)	2.7979 (16)	175 (16)
O1—H1···O5^{i}	0.83 (1)	1.84 (1)	2.6511 (12)	167 (18)
N1—H1*B*···O5^{ii}	0.85 (1)	1.95 (1)	2.7976 (16)	178 (15)
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N2—H2*A*···O3^{iii}	0.85 (1)	2.19 (1)	3.0347 (16)	173 (15)

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