IUCrData

ISSN 2414-3146

1-Carboxy-2-hydroxypropan-1-aminium 4-methylbenzenesulfonate monohydrate

P. Prabu,[a] A. Thiruvalluvar[b]* and C. Ramachandra Raja[a]‡

[a]Postgraduate Research Department of Physics, Government Arts College (Autonomous), Kumbakonam 612 001, Tamilnadu, India, and [b]Kunthavai Naacchiyaar Government Arts College for Women (Autonomous), Thanjavur 613 007, Tamilnadu, India. *Correspondence e-mail: thiruvalluvar.a@gmail.com

Received 23 December 2017
Accepted 30 December 2017

Edited by E. R. T. Tiekink, Sunway University, Malaysia

‡ Additional correspondence author, e-mail: crraja_phy@yahoo.com.

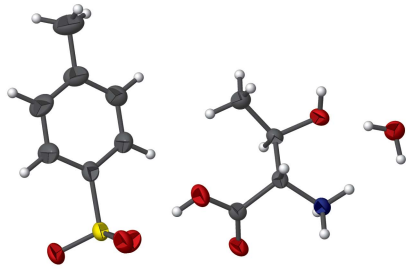
Keywords: crystal structure; hydrated salt; *p*-toluenesulfonate; L-threonine; hydrogen bonding.

CCDC reference: 1814028

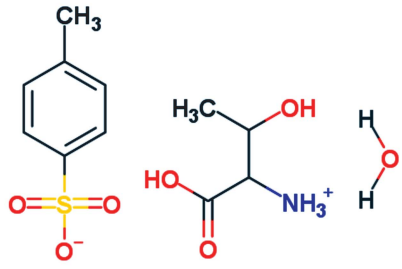
Structural data: full structural data are available from iucrdata.iucr.org

In the title hydrated salt, $C_4H_{10}NO_3^+\cdot C_7H_7O_3S^-\cdot H_2O$, an intramolecular C—H···O hydrogen bond in the cation generates an $S(6)$ loop. In the crystal, carboxyl-O—H···O(sulfonate), hydroxyl-O—H···O(sulfonate), water-O—H···O(sulfonate, hydroxyl) and ammonium-N—H···O(water, carbonyl) hydrogen bonds link the components of the asymmetric unit into supramolecular layers parallel to (001).

3D view



Chemical scheme



Structure description

Having a non-centrosymmetric crystal is an important requisite for second harmonic generation (Etter & Huang, 1992; Sarma *et al.*, 1994). As part of our studies in this area, we now describe the crystal structure of the title hydrated molecular salt (Fig. 1), which crystallizes in the non-centrosymmetric space group $P2_1$. It crystallizes with one independent cation, an anion and a water molecule in the asymmetric unit.

There is an intramolecular C11—H11*B*···O5 hydrogen bond within the cation, which generates an $S(6)$ ring, Table 1. The crystal structure features a variety of hydrogen bonds, as listed in Table 1. As seen from Fig. 2, the hydrogen bonds connect the constituents of the asymmetric unit into supramolecular layers that stack along the *c*-axis direction.

Synthesis and crystallization

p-Toluenesulfonic acid monohydrate (1.902 g, 0.0099 mol) and L-threonine (1.191 g, 0.0099 mol) were mixed in deionized water. The solution was stirred well using a magnetic stirrer for about 4 h to obtain a homogeneous solution. Then, the solution was

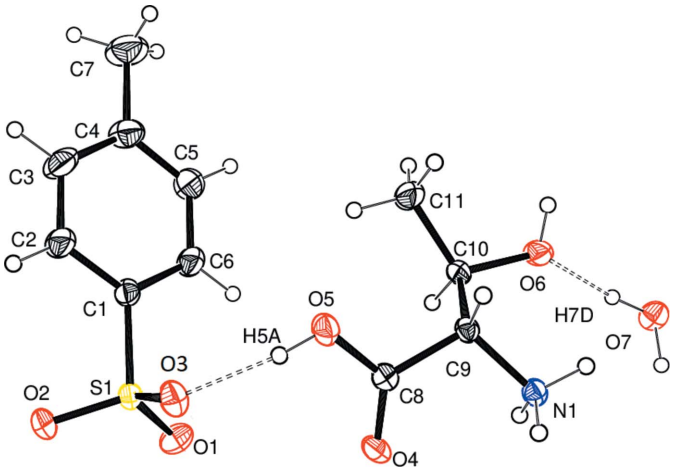


Figure 1
A view of the asymmetric unit showing the atom numbering and displacement ellipsoids drawn at the 30% probability level. Dashed lines indicate hydrogen-bonding interactions.

filtered and left to evaporate slowly. The colourless blocks used for the analysis were harvested after three weeks.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Owing to obstruction from the beam-stop, the (001) reflection was omitted from the final cycles of the refinement.

Acknowledgements

The authors are grateful to the Sophisticated Analytical Instrument Facility (SAIF) in IITM, Chennai, India, for the single-crystal X-ray diffraction data.

Funding information

The funding for this research work was provided by the Council of Scientific and Industrial Research (CSIR), New Delhi, India (Scheme No. 03(1301)/13/EMR II to author CRR).

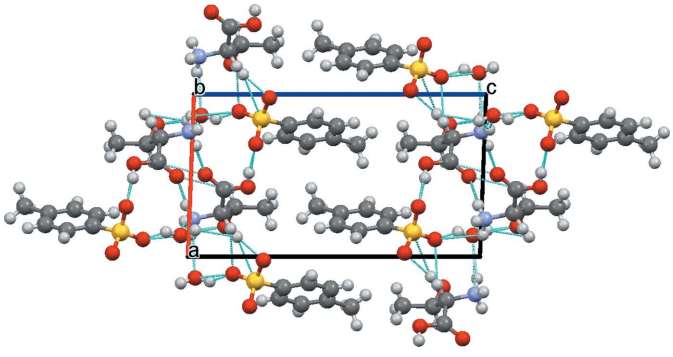


Figure 2
The molecular packing of the title compound, viewed down the *b* axis. The hydrogen bonds are shown as blue lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11*B*···O5	0.96	2.47	3.068 (4)	120
O5—H5*A*···O3	0.82	1.76	2.576 (3)	172
O6—H6*A*···O2^{i}	0.82	1.90	2.713 (3)	172
O7—H7*E*···O1^{ii}	0.88 (2)	1.98 (3)	2.770 (3)	149 (3)
O7—H7*D*···O6	0.88 (2)	1.88 (3)	2.741 (3)	166 (4)
N1—H1*B*···O7^{iii}	0.91 (2)	2.16 (2)	2.973 (3)	149 (2)
N1—H1*C*···O4^{ii}	0.88 (2)	2.01 (2)	2.874 (3)	166 (3)
N1—H1*A*···O7^{iv}	0.90 (2)	1.92 (2)	2.769 (3)	155 (3)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_4H_{10}NO_3^+·C_7H_7O_3S^-·H_2O$
M_r	309.33
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	296
a, b, c (Å)	8.0762 (4), 6.2486 (4), 14.5096 (10)
β (°)	92.161 (2)
V (Å^3)	731.71 (8)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.25
Crystal size (mm)	0.15 × 0.15 × 0.10
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.662, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8712, 3347, 2705
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.658
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.081, 1.02
No. of reflections	3347
No. of parameters	203
No. of restraints	10
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.19
Absolute structure	Flack x determined using 1035 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).
Absolute structure parameter	0.06 (4)

Computer programs: *APEX2*, *SAINT* and *XPREP* (Bruker, 2004), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2018/1* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009), and *publCIF* (Westrip, 2010).

References

Bruker (2004). *APEX2*, *SAINT*, *XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C. & Huang, K. S. (1992). *Chem. Mater.* **4**, 824–827.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Sarma, J. A. R. P., Dhurjati, M. S. K., Ravikumar, K. & Bhanuprakash, K. (1994). *Chem. Mater.* **6**, 1369–1377.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2018). **3**, x171848 [https://doi.org/10.1107/S241431461701848X]

1-Carboxy-2-hydroxypropan-1-aminium 4-methylbenzenesulfonate monohydrate

P. Prabu, A. Thiruvalluvar and C. Ramachandra Raja

1-Carboxy-2-hydroxypropan-1-aminium 4-methylbenzenesulfonate monohydrate

Crystal data

$C_4H_{10}NO_3^+·C_7H_7O_3S^-·H_2O$
M_r = 309.33
Monoclinic, $P2_1$
a = 8.0762 (4) Å
b = 6.2486 (4) Å
c = 14.5096 (10) Å
β = 92.161 (2)°
V = 731.71 (8) Å^3
Z = 2
$F(000)$ = 328
D_x = 1.404 Mg m^{-3}
Melting point: 356 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3667 reflections
θ = 2.8–26.3°
μ = 0.25 mm^{-1}
T = 296 K
Block, colourless
0.15 × 0.15 × 0.10 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.662, T_{max} = 0.746
8712 measured reflections
3347 independent reflections
2705 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.9°, θ_{min} = 2.5°
h = −10→10
k = −8→8
l = −19→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.081
S = 1.02
3347 reflections
203 parameters
10 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0332P)^2 + 0.1196P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack x determined using 1035 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).
Absolute structure parameter: 0.06 (4)

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 carbon-bound H-atoms were placed in calculated positions (C—H = 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2–1.5U_{equiv}(C). The oxygen-bound H-atoms were either fixed with O—H = 0.82 or refined with a distance restraint of O—H = 0.82±0.02 Å, and with U_{iso}(H) set to 1.5U_{equiv}(O). The nitrogen-bound H-atoms were refined with a distance restraint of N—H = 0.90±0.02 Å, and with U_{iso}(H) set to 1.2U_{equiv}(N).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1902 (4)	0.2622 (5)	0.3298 (2)	0.0322 (7)
C2	0.1609 (5)	0.1904 (6)	0.4177 (2)	0.0482 (9)
H2	0.116375	0.054961	0.426354	0.058*
C3	0.1979 (5)	0.3202 (7)	0.4928 (3)	0.0586 (11)
H3	0.176111	0.271176	0.551625	0.070*
C4	0.2667 (5)	0.5213 (6)	0.4826 (3)	0.0498 (10)
C5	0.2923 (4)	0.5914 (7)	0.3942 (2)	0.0488 (9)
H5	0.336603	0.726971	0.385569	0.059*
C6	0.2539 (4)	0.4652 (6)	0.3181 (2)	0.0428 (8)
H6	0.270923	0.516931	0.259131	0.051*
C7	0.3102 (7)	0.6609 (7)	0.5653 (3)	0.0764 (14)
H7A	0.299896	0.808749	0.548036	0.115*
H7B	0.422038	0.632250	0.586385	0.115*
H7C	0.236118	0.630136	0.613767	0.115*
C8	0.5860 (4)	0.2830 (5)	0.1146 (2)	0.0267 (7)
C9	0.7313 (3)	0.4344 (5)	0.1101 (2)	0.0241 (6)
H9	0.827748	0.370261	0.142508	0.029*
C10	0.6941 (4)	0.6526 (4)	0.1541 (2)	0.0287 (7)
H10	0.583673	0.699233	0.132162	0.034*
C11	0.6977 (5)	0.6436 (6)	0.2586 (2)	0.0496 (10)
H11A	0.804857	0.595975	0.281004	0.074*
H11B	0.614560	0.545714	0.278236	0.074*
H11C	0.676108	0.783559	0.282673	0.074*
O1	0.1064 (3)	0.2289 (4)	0.15625 (16)	0.0479 (6)
O2	0.0228 (3)	−0.0530 (5)	0.25880 (16)	0.0517 (7)
O3	0.3066 (3)	−0.0201 (3)	0.21803 (16)	0.0404 (6)
O4	0.4950 (3)	0.2474 (4)	0.04880 (15)	0.0439 (6)
O5	0.5713 (3)	0.2015 (4)	0.19660 (15)	0.0410 (6)
H5A	0.483804	0.135286	0.198317	0.061*
O6	0.8126 (3)	0.8011 (3)	0.12263 (15)	0.0414 (6)
H6A	0.877061	0.833342	0.165426	0.062*
O7	0.8692 (3)	1.0534 (3)	−0.02744 (18)	0.0394 (6)
S1	0.15216 (9)	0.09312 (13)	0.23372 (5)	0.0331 (2)
N1	0.7689 (3)	0.4662 (4)	0.01176 (17)	0.0268 (6)

H1B	0.865 (3)	0.542 (4)	0.007 (2)	0.033 (9)*
H1C	0.683 (3)	0.535 (5)	−0.013 (2)	0.060 (13)*
H1A	0.782 (4)	0.338 (4)	−0.016 (2)	0.050 (11)*
H7E	0.848 (4)	0.980 (5)	−0.0782 (17)	0.048 (11)*
H7D	0.836 (5)	0.983 (7)	0.0212 (19)	0.088 (17)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0281 (16)	0.0385 (17)	0.0298 (17)	−0.0019 (14)	−0.0013 (13)	0.0071 (14)
C2	0.072 (3)	0.0402 (19)	0.0329 (19)	−0.0149 (19)	0.0013 (17)	0.0055 (17)
C3	0.089 (3)	0.059 (3)	0.027 (2)	−0.008 (2)	0.001 (2)	0.0055 (19)
C4	0.065 (2)	0.045 (2)	0.039 (2)	−0.0006 (18)	−0.0045 (18)	−0.0046 (17)
C5	0.063 (2)	0.0364 (18)	0.047 (2)	−0.010 (2)	0.0043 (17)	−0.001 (2)
C6	0.055 (2)	0.0408 (19)	0.0325 (19)	−0.0082 (18)	0.0049 (16)	0.0056 (16)
C7	0.113 (4)	0.067 (3)	0.048 (3)	−0.013 (3)	−0.007 (2)	−0.016 (2)
C8	0.0254 (15)	0.0237 (15)	0.0310 (17)	0.0018 (13)	0.0013 (13)	−0.0007 (13)
C9	0.0197 (14)	0.0263 (14)	0.0263 (16)	0.0000 (12)	−0.0005 (12)	0.0022 (13)
C10	0.0295 (16)	0.0270 (16)	0.0296 (16)	−0.0069 (12)	0.0021 (12)	−0.0037 (12)
C11	0.064 (2)	0.052 (2)	0.0335 (18)	−0.0228 (19)	0.0100 (16)	−0.0100 (16)
O1	0.0577 (15)	0.0567 (15)	0.0288 (13)	0.0011 (13)	−0.0049 (11)	0.0106 (12)
O2	0.0422 (13)	0.0725 (17)	0.0404 (14)	−0.0327 (13)	0.0031 (11)	−0.0002 (14)
O3	0.0371 (12)	0.0332 (12)	0.0513 (15)	−0.0043 (11)	0.0068 (11)	0.0016 (11)
O4	0.0415 (13)	0.0551 (15)	0.0346 (13)	−0.0247 (12)	−0.0061 (11)	0.0012 (11)
O5	0.0400 (13)	0.0454 (13)	0.0374 (13)	−0.0153 (11)	−0.0015 (10)	0.0100 (11)
O6	0.0561 (15)	0.0369 (13)	0.0311 (13)	−0.0210 (12)	0.0001 (11)	−0.0012 (11)
O7	0.0497 (14)	0.0287 (13)	0.0400 (14)	0.0014 (11)	0.0068 (11)	−0.0032 (12)
S1	0.0304 (4)	0.0405 (4)	0.0281 (4)	−0.0097 (4)	−0.0003 (3)	0.0048 (4)
N1	0.0252 (14)	0.0244 (13)	0.0310 (14)	−0.0014 (12)	0.0036 (11)	−0.0016 (12)

Geometric parameters (Å, º)

C1—C2	1.381 (4)	C9—C10	1.539 (4)
C1—C6	1.381 (5)	C9—H9	0.9800
C1—S1	1.767 (3)	C10—O6	1.420 (3)
C2—C3	1.382 (5)	C10—C11	1.517 (4)
C2—H2	0.9300	C10—H10	0.9800
C3—C4	1.384 (5)	C11—H11A	0.9600
C3—H3	0.9300	C11—H11B	0.9600
C4—C5	1.378 (5)	C11—H11C	0.9600
C4—C7	1.513 (5)	O1—S1	1.445 (2)
C5—C6	1.383 (5)	O2—S1	1.445 (2)
C5—H5	0.9300	O3—S1	1.459 (2)
C6—H6	0.9300	O5—H5A	0.8200
C7—H7A	0.9600	O6—H6A	0.8200
C7—H7B	0.9600	O7—H7E	0.88 (2)
C7—H7C	0.9600	O7—H7D	0.88 (2)
C8—O4	1.204 (3)	N1—H1B	0.913 (19)

C8—O5	1.304 (3)	N1—H1C	0.88 (2)
C8—C9	1.511 (4)	N1—H1A	0.90 (2)
C9—N1	1.484 (4)		
C2—C1—C6	119.3 (3)	N1—C9—H9	109.1
C2—C1—S1	120.2 (3)	C8—C9—H9	109.1
C6—C1—S1	120.5 (2)	C10—C9—H9	109.1
C1—C2—C3	119.9 (3)	O6—C10—C11	110.9 (2)
C1—C2—H2	120.1	O6—C10—C9	107.5 (2)
C3—C2—H2	120.1	C11—C10—C9	112.6 (3)
C2—C3—C4	121.6 (3)	O6—C10—H10	108.6
C2—C3—H3	119.2	C11—C10—H10	108.6
C4—C3—H3	119.2	C9—C10—H10	108.6
C5—C4—C3	117.5 (4)	C10—C11—H11A	109.5
C5—C4—C7	121.1 (4)	C10—C11—H11B	109.5
C3—C4—C7	121.4 (4)	H11A—C11—H11B	109.5
C4—C5—C6	121.7 (4)	C10—C11—H11C	109.5
C4—C5—H5	119.2	H11A—C11—H11C	109.5
C6—C5—H5	119.2	H11B—C11—H11C	109.5
C1—C6—C5	119.9 (3)	C8—O5—H5A	109.5
C1—C6—H6	120.0	C10—O6—H6A	109.5
C5—C6—H6	120.0	H7E—O7—H7D	111 (3)
C4—C7—H7A	109.5	O2—S1—O1	113.63 (15)
C4—C7—H7B	109.5	O2—S1—O3	111.30 (15)
H7A—C7—H7B	109.5	O1—S1—O3	111.00 (14)
C4—C7—H7C	109.5	O2—S1—C1	106.57 (15)
H7A—C7—H7C	109.5	O1—S1—C1	107.12 (16)
H7B—C7—H7C	109.5	O3—S1—C1	106.79 (14)
O4—C8—O5	125.3 (3)	C9—N1—H1B	109.9 (19)
O4—C8—C9	122.3 (3)	C9—N1—H1C	106 (2)
O5—C8—C9	112.4 (3)	H1B—N1—H1C	112 (2)
N1—C9—C8	108.2 (2)	C9—N1—H1A	110 (2)
N1—C9—C10	109.3 (2)	H1B—N1—H1A	108 (2)
C8—C9—C10	111.9 (2)	H1C—N1—H1A	110 (3)
C6—C1—C2—C3	−1.1 (5)	O4—C8—C9—C10	102.5 (3)
S1—C1—C2—C3	177.1 (3)	O5—C8—C9—C10	−76.5 (3)
C1—C2—C3—C4	−1.0 (6)	N1—C9—C10—O6	−42.8 (3)
C2—C3—C4—C5	2.0 (6)	C8—C9—C10—O6	−162.6 (2)
C2—C3—C4—C7	−178.7 (4)	N1—C9—C10—C11	−165.3 (2)
C3—C4—C5—C6	−1.1 (6)	C8—C9—C10—C11	74.9 (3)
C7—C4—C5—C6	179.7 (4)	C2—C1—S1—O2	27.8 (3)
C2—C1—C6—C5	2.0 (5)	C6—C1—S1—O2	−154.0 (3)
S1—C1—C6—C5	−176.2 (3)	C2—C1—S1—O1	149.7 (3)
C4—C5—C6—C1	−0.9 (5)	C6—C1—S1—O1	−32.1 (3)
O4—C8—C9—N1	−17.9 (4)	C2—C1—S1—O3	−91.3 (3)
O5—C8—C9—N1	163.1 (2)	C6—C1—S1—O3	86.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···O5	0.96	2.47	3.068 (4)	120
O5—H5*A*···O3	0.82	1.76	2.576 (3)	172
O6—H6*A*···O2^{i}	0.82	1.90	2.713 (3)	172
O7—H7*E*···O1^{ii}	0.88 (2)	1.98 (3)	2.770 (3)	149 (3)
O7—H7*D*···O6	0.88 (2)	1.88 (3)	2.741 (3)	166 (4)
N1—H1*B*···O7^{iii}	0.91 (2)	2.16 (2)	2.973 (3)	149 (2)
N1—H1*C*···O4^{ii}	0.88 (2)	2.01 (2)	2.874 (3)	166 (3)
N1—H1*A*···O7^{iv}	0.90 (2)	1.92 (2)	2.769 (3)	155 (3)

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