Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 2-Phenylimidazolium hemi(benzene-1,4-dicarboxylate) trihydrate

**Heng-Da Li**

Department of Chemistry, Jilin Normal University, Siping 136000, People's Republic of China
Correspondence e-mail: lihengdajlsp@yahoo.cn

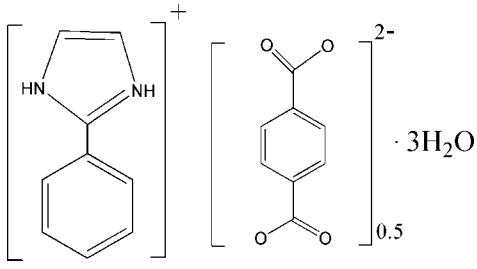
Received 22 October 2011; accepted 27 October 2011

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.051; $wR$ factor = 0.120; data-to-parameter ratio = 12.7.

The asymmetric unit of the title compound, $C_9H_9N_2^+\cdot 0.5C_8H_4O_4^{2-}\cdot 3H_2O$, contains one 2-phenylimidazolium cation, half a benzene-1,4-dicarboxylate anion and three water molecules, which are connected by O—H···O and N—H···O hydrogen bonds into a three-dimensional network.

## Related literature

For the structures of 2-phenylimidazolium nitrate and 2-phenylimidazolium acetate, see: Xia *et al.* (2009); Xia & Yao (2010).




## Experimental

### Crystal data

$C_9H_9N_2^+\cdot 0.5C_8H_4O_4^{2-}\cdot 3H_2O$
$M_r$ = 281.29
Triclinic, $P\overline{1}$
$a$ = 7.208 (1) Å
$b$ = 9.164 (2) Å
$c$ = 11.368 (2) Å
$\alpha$ = 78.506 (1)°
$\beta$ = 75.478 (5)°
$\gamma$ = 86.774 (5)°
$V$ = 712.3 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 293 K
0.17 × 0.15 × 0.12 mm

### Data collection

Bruker APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.55, $T_{max}$ = 0.72
4505 measured reflections
2611 independent reflections
1519 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.044

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.120
$S$ = 0.99
2611 reflections
205 parameters
9 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···O1 | 0.86 | 1.91 | 2.768 (3) | 172 |
| N2—H2$A$···O3$W^{i}$ | 0.86 | 1.82 | 2.663 (3) | 168 |
| O1$W$—H$W$11···O2 | 0.85 (1) | 2.02 (1) | 2.850 (3) | 167 (3) |
| O1$W$—H$W$12···O2$^{ii}$ | 0.85 (1) | 2.33 (3) | 2.955 (3) | 131 (3) |
| O2$W$—H$W$21···O1$^{iii}$ | 0.86 (1) | 2.03 (2) | 2.818 (3) | 153 (3) |
| O2$W$—H$W$22···O2$^{ii}$ | 0.85 (1) | 2.01 (1) | 2.828 (3) | 161 (3) |
| O3$W$—H$W$31···O1$W^{iv}$ | 0.85 (1) | 1.94 (2) | 2.749 (4) | 160 (4) |
| O3$W$—H$W$32···O2$W^{v}$ | 0.85 (1) | 1.89 (1) | 2.723 (4) | 167 (4) |

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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*.

The author thanks Jilin Normal University for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5686).

## References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xia, D.-C., Li, W.-C. & Han, S. (2009). *Acta Cryst.* E**65**, o3283.
Xia, D.-C. & Yao, J.-H. (2010). *Acta Cryst.* E**66**, o649.

# supporting information

*Acta Cryst.* (2011). E**67**, o3156 [https://doi.org/10.1107/S1600536811044953]

## 2-Phenylimidazolium hemi(benzene-1,4-dicarboxylate) trihydrate

**Heng-Da Li**

### S1. Comment

2-Phenylimidazolium nitrate and 2-phenylimidazolium acetate have been reported (Xia *et al.*, 2009; Xia & Yao, 2010). Here, I report the synthesis and crystal structure of the 2-phenylimidazolium hemi-benzene-1,4-dicarboxylate trihydrate.

The asymmetric unit of the title compound is composed of one 2-phenylimidazolium cation, half a benzene-1,4-dicarboxylate anion, and three water molecules (Fig. 1) which are connected by O—H···O and N—H···O hydrogen bonds to a three-dimensional network.

### S2. Experimental

A mixture of 2-phenylimidazole (0.5 mmol), benzene-1,4-dicarboxylic acid (0.3 mmol) and $H_2O$ (10 ml) was mixed. After several days, colorless crystals were obtained at room temperature.

### S3. Refinement

All H atoms on C and N atoms were positioned geometrically (N—H = 0.86 Å and C—H = 0.93 Å) and refined as riding, with $U_{iso}$(H)=1.2$U_{eq}$(carrier). Water H atoms were located in a difference Fourier map and refined as riding with the O—H and H···H distances restrainted to 0.85±0.01 and 1.35±0.01 Å, respectively.

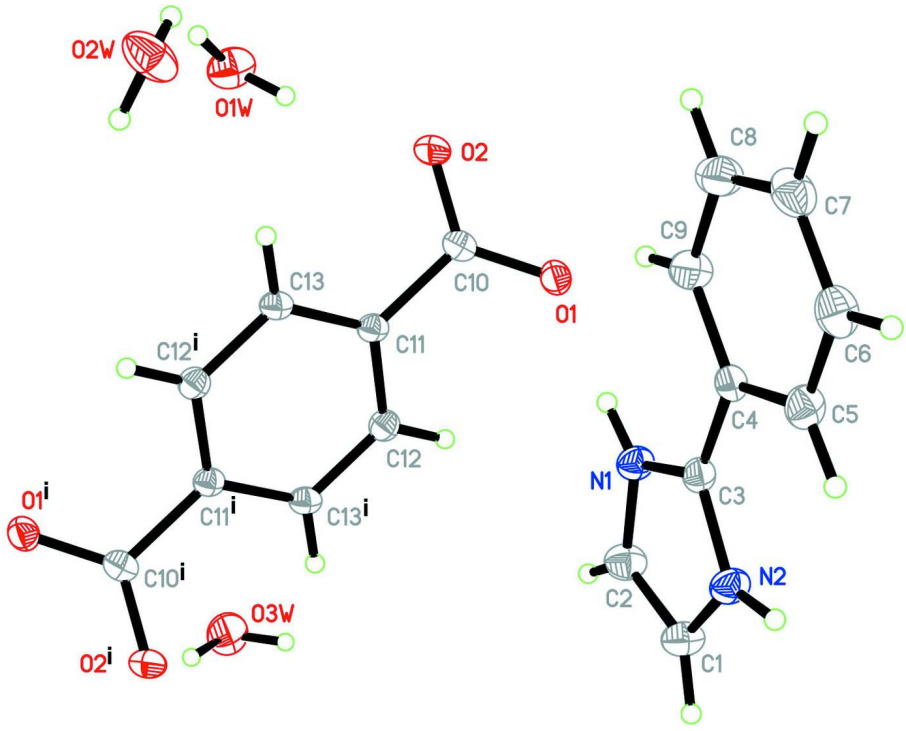



**Figure 1**

The structure of the title compound showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry codes: (i) 1 - *x*, 1 - *y*, 1 - *z*.

## 2-Phenylimidazolium hemi(benzene-1,4-dicarboxylate) trihydrate

*Crystal data*

$C_9H_9N_2^+·0.5C_8H_4O_4^{2-}·3H_2O$
$M_r = 281.29$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.208$ (1) Å
$b = 9.164$ (2) Å
$c = 11.368$ (2) Å
$\alpha = 78.506$ (1)°
$\beta = 75.478$ (5)°
$\gamma = 86.774$ (5)°
$V = 712.3$ (2) Å$^3$
$Z = 2$
$F(000) = 298$
$D_x = 1.311$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4505 reflections
$\theta = 1.9$–25.4°
$\mu = 0.10$ mm$^{-1}$
$T = 293$ K
Block, colorless
0.17 × 0.15 × 0.12 mm

*Data collection*

Bruker APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.55$, $T_{max} = 0.72$
4505 measured reflections
2611 independent reflections
1519 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.9°$
$h = -8 \rightarrow 7$
$k = -10 \rightarrow 11$
$l = -13 \rightarrow 11$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.120$
$S = 0.99$
2611 reflections
205 parameters
9 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0385P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å$^{-3}$
$\Delta\rho_{min} = -0.22$ e Å$^{-3}$

*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.
**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.9035 (4) | 0.2035 (3) | 0.8466 (3) | 0.0553 (7) |
| H1 | 0.9328 | 0.1023 | 0.8563 | 0.066* |
| C2 | 0.9084 (4) | 0.2981 (3) | 0.7392 (3) | 0.0543 (7) |
| H2 | 0.9410 | 0.2750 | 0.6605 | 0.065* |
| C3 | 0.8178 (3) | 0.4256 (2) | 0.8904 (2) | 0.0391 (6) |
| C4 | 0.7537 (3) | 0.5458 (2) | 0.9583 (2) | 0.0386 (6) |
| C5 | 0.7153 (4) | 0.5189 (3) | 1.0870 (2) | 0.0514 (7) |
| H5 | 0.7339 | 0.4241 | 1.1303 | 0.062* |
| C6 | 0.6498 (4) | 0.6324 (3) | 1.1502 (3) | 0.0604 (8) |
| H6 | 0.6230 | 0.6135 | 1.2362 | 0.072* |
| C7 | 0.6238 (4) | 0.7730 (3) | 1.0875 (3) | 0.0641 (9) |
| H7 | 0.5806 | 0.8495 | 1.1306 | 0.077* |
| C8 | 0.6617 (4) | 0.8005 (3) | 0.9604 (3) | 0.0639 (8) |
| H8 | 0.6437 | 0.8958 | 0.9177 | 0.077* |
| C9 | 0.7263 (4) | 0.6880 (3) | 0.8957 (3) | 0.0542 (7) |
| H9 | 0.7515 | 0.7076 | 0.8097 | 0.065* |
| C10 | 0.7373 (3) | 0.7275 (2) | 0.5307 (2) | 0.0387 (6) |
| C11 | 0.6137 (3) | 0.6093 (2) | 0.5159 (2) | 0.0342 (6) |
| C12 | 0.6925 (3) | 0.4735 (2) | 0.4926 (2) | 0.0381 (6) |
| H12 | 0.8223 | 0.4555 | 0.4873 | 0.046* |
| C13 | 0.4193 (3) | 0.6353 (2) | 0.5229 (2) | 0.0373 (6) |
| H13 | 0.3647 | 0.7262 | 0.5380 | 0.045* |
| N1 | 0.8561 (3) | 0.4352 (2) | 0.76772 (18) | 0.0446 (5) |
| H1A | 0.8490 | 0.5154 | 0.7148 | 0.054* |

| | | | | |
|---|---|---|---|---|
| N2 | 0.8474 (3) | 0.2839 (2) | 0.93913 (19) | 0.0483 (6) |
| H2A | 0.8333 | 0.2485 | 1.0168 | 0.058* |
| O1 | 0.8703 (2) | 0.68667 (16) | 0.58457 (15) | 0.0455 (5) |
| O2 | 0.7039 (3) | 0.86002 (17) | 0.48727 (17) | 0.0574 (5) |
| O1W | 0.4795 (4) | 0.9746 (2) | 0.31515 (19) | 0.0722 (6) |
| O2W | 0.0067 (4) | 0.9337 (3) | 0.6501 (2) | 0.0830 (7) |
| O3W | 0.7934 (5) | 0.1375 (3) | 0.1721 (2) | 0.0883 (7) |
| HW11 | 0.529 (4) | 0.934 (4) | 0.374 (2) | 0.127 (16)* |
| HW12 | 0.373 (3) | 1.010 (4) | 0.350 (3) | 0.132 (17)* |
| HW21 | 0.004 (4) | 0.858 (2) | 0.616 (3) | 0.106 (13)* |
| HW22 | 0.084 (4) | 0.994 (2) | 0.595 (2) | 0.109 (14)* |
| HW31 | 0.683 (2) | 0.107 (4) | 0.214 (3) | 0.132 (18)* |
| HW32 | 0.862 (4) | 0.129 (4) | 0.224 (3) | 0.120 (15)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0659 (19) | 0.0421 (14) | 0.0546 (19) | 0.0025 (14) | −0.0106 (15) | −0.0078 (14) |
| C2 | 0.0643 (19) | 0.0479 (16) | 0.0483 (18) | 0.0019 (14) | −0.0064 (14) | −0.0133 (14) |
| C3 | 0.0400 (14) | 0.0422 (14) | 0.0330 (15) | −0.0070 (11) | −0.0096 (11) | 0.0002 (11) |
| C4 | 0.0368 (14) | 0.0431 (14) | 0.0368 (15) | −0.0047 (11) | −0.0102 (11) | −0.0075 (11) |
| C5 | 0.0567 (17) | 0.0583 (16) | 0.0380 (17) | −0.0048 (13) | −0.0112 (13) | −0.0061 (13) |
| C6 | 0.0636 (19) | 0.077 (2) | 0.0435 (18) | −0.0051 (16) | −0.0107 (15) | −0.0196 (16) |
| C7 | 0.063 (2) | 0.071 (2) | 0.067 (2) | 0.0036 (16) | −0.0152 (17) | −0.0362 (18) |
| C8 | 0.080 (2) | 0.0494 (17) | 0.065 (2) | 0.0021 (16) | −0.0202 (18) | −0.0141 (15) |
| C9 | 0.0691 (19) | 0.0492 (16) | 0.0425 (17) | −0.0030 (14) | −0.0094 (14) | −0.0092 (13) |
| C10 | 0.0456 (16) | 0.0362 (13) | 0.0327 (15) | −0.0064 (11) | −0.0046 (12) | −0.0076 (11) |
| C11 | 0.0417 (14) | 0.0314 (12) | 0.0288 (14) | −0.0005 (11) | −0.0085 (11) | −0.0045 (10) |
| C12 | 0.0383 (14) | 0.0390 (13) | 0.0363 (15) | 0.0003 (11) | −0.0090 (11) | −0.0056 (11) |
| C13 | 0.0431 (15) | 0.0300 (12) | 0.0389 (15) | 0.0019 (11) | −0.0090 (11) | −0.0083 (10) |
| N1 | 0.0526 (13) | 0.0421 (12) | 0.0362 (13) | −0.0008 (10) | −0.0090 (10) | −0.0027 (9) |
| N2 | 0.0594 (14) | 0.0436 (12) | 0.0375 (13) | −0.0023 (11) | −0.0102 (11) | 0.0011 (10) |
| O1 | 0.0495 (11) | 0.0452 (9) | 0.0447 (11) | −0.0091 (8) | −0.0194 (9) | −0.0032 (8) |
| O2 | 0.0735 (13) | 0.0318 (9) | 0.0722 (14) | −0.0085 (9) | −0.0346 (11) | 0.0010 (9) |
| O1W | 0.0924 (18) | 0.0683 (14) | 0.0497 (14) | 0.0082 (13) | −0.0121 (13) | −0.0059 (11) |
| O2W | 0.127 (2) | 0.0710 (14) | 0.0531 (15) | −0.0447 (15) | −0.0272 (14) | 0.0016 (12) |
| O3W | 0.106 (2) | 0.0951 (18) | 0.0560 (15) | −0.0288 (16) | −0.0322 (17) | 0.0270 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.343 (3) | C9—H9 | 0.9300 |
| C1—N2 | 1.369 (3) | C10—O2 | 1.251 (3) |
| C1—H1 | 0.9300 | C10—O1 | 1.263 (3) |
| C2—N1 | 1.368 (3) | C10—C11 | 1.501 (3) |
| C2—H2 | 0.9300 | C11—C12 | 1.385 (3) |
| C3—N2 | 1.334 (3) | C11—C13 | 1.393 (3) |
| C3—N1 | 1.338 (3) | C12—C13$^{i}$ | 1.381 (3) |
| C3—C4 | 1.458 (3) | C12—H12 | 0.9300 |

| | | | |
|---|---|---|---|
| C4—C9 | 1.384 (3) | C13—C12$^{i}$ | 1.381 (3) |
| C4—C5 | 1.393 (3) | C13—H13 | 0.9300 |
| C5—C6 | 1.376 (3) | N1—H1A | 0.8600 |
| C5—H5 | 0.9300 | N2—H2A | 0.8600 |
| C6—C7 | 1.371 (4) | O1W—HW11 | 0.852 (10) |
| C6—H6 | 0.9300 | O1W—HW12 | 0.850 (10) |
| C7—C8 | 1.375 (4) | O2W—HW21 | 0.859 (10) |
| C7—H7 | 0.9300 | O2W—HW22 | 0.854 (10) |
| C8—C9 | 1.377 (3) | O3W—HW31 | 0.850 (10) |
| C8—H8 | 0.9300 | O3W—HW32 | 0.847 (10) |
| | | | |
| C2—C1—N2 | 107.1 (2) | C8—C9—C4 | 120.2 (3) |
| C2—C1—H1 | 126.4 | C8—C9—H9 | 119.9 |
| N2—C1—H1 | 126.4 | C4—C9—H9 | 119.9 |
| C1—C2—N1 | 106.9 (2) | O2—C10—O1 | 124.1 (2) |
| C1—C2—H2 | 126.6 | O2—C10—C11 | 117.9 (2) |
| N1—C2—H2 | 126.6 | O1—C10—C11 | 118.0 (2) |
| N2—C3—N1 | 106.71 (19) | C12—C11—C13 | 119.0 (2) |
| N2—C3—C4 | 126.4 (2) | C12—C11—C10 | 120.3 (2) |
| N1—C3—C4 | 126.8 (2) | C13—C11—C10 | 120.69 (19) |
| C9—C4—C5 | 119.0 (2) | C13$^{i}$—C12—C11 | 120.8 (2) |
| C9—C4—C3 | 120.4 (2) | C13$^{i}$—C12—H12 | 119.6 |
| C5—C4—C3 | 120.6 (2) | C11—C12—H12 | 119.6 |
| C6—C5—C4 | 120.1 (2) | C12$^{i}$—C13—C11 | 120.2 (2) |
| C6—C5—H5 | 120.0 | C12$^{i}$—C13—H13 | 119.9 |
| C4—C5—H5 | 120.0 | C11—C13—H13 | 119.9 |
| C7—C6—C5 | 120.6 (3) | C3—N1—C2 | 109.7 (2) |
| C7—C6—H6 | 119.7 | C3—N1—H1A | 125.2 |
| C5—C6—H6 | 119.7 | C2—N1—H1A | 125.2 |
| C6—C7—C8 | 119.7 (2) | C3—N2—C1 | 109.6 (2) |
| C6—C7—H7 | 120.2 | C3—N2—H2A | 125.2 |
| C8—C7—H7 | 120.2 | C1—N2—H2A | 125.2 |
| C7—C8—C9 | 120.5 (3) | HW11—O1W—HW12 | 104.8 (15) |
| C7—C8—H8 | 119.7 | HW21—O2W—HW22 | 103.8 (14) |
| C9—C8—H8 | 119.7 | HW31—O3W—HW32 | 106.0 (16) |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···O1 | 0.86 | 1.91 | 2.768 (3) | 172 |
| N2—H2*A*···O3*W*$^{ii}$ | 0.86 | 1.82 | 2.663 (3) | 168 |
| O1*W*—H*W*11···O2 | 0.85 (1) | 2.02 (1) | 2.850 (3) | 167 (3) |
| O1*W*—H*W*12···O2$^{iii}$ | 0.85 (1) | 2.33 (3) | 2.955 (3) | 131 (3) |
| O2*W*—H*W*21···O1$^{iv}$ | 0.86 (1) | 2.03 (2) | 2.818 (3) | 153 (3) |
| O2*W*—H*W*22···O2$^{iii}$ | 0.85 (1) | 2.01 (1) | 2.828 (3) | 161 (3) |

| | | | | |
|---|---|---|---|---|
| O3*W*—H*W*31···O1*W*$^{v}$ | 0.85 (1) | 1.94 (2) | 2.749 (4) | 160 (4) |
| O3*W*—H*W*32···O2*W*$^{i}$ | 0.85 (1) | 1.89 (1) | 2.723 (4) | 167 (4) |

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