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# 2-(4-Ethoxycarbonyl-5-methyl-1*H*-1,2,3-triazol-1-yl)acetic acid monohydrate

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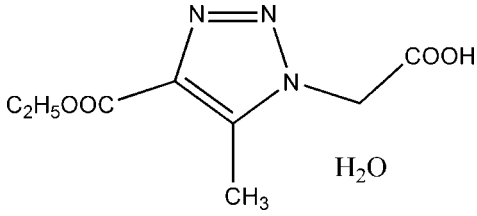
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.066; $wR$ factor = 0.189; data-to-parameter ratio = 16.8.

The title compound, $C_8H_{11}N_3O_4\cdot H_2O$, was synthesized by reaction of 2-azidoacetic acid and ethyl acetylacetate. In the crystal packing, molecules are linked by strong intermolecular O—H···N and O—H···O hydrogen bonds into double layers parallel to the *ab* plane.

## Related literature

For the biological activities of triazole derivatives, see: Olesen *et al.* (2003); Tian *et al.* (2005). For the synthesis, see: El Khadem *et al.* (1968). For related structures, see: Lin *et al.* (2008); Xiao *et al.* (2008); Zhao (2009).




## Experimental

### Crystal data

$C_8H_{11}N_3O_4\cdot H_2O$
$M_r$ = 231.21
Monoclinic, $C2/c$
$a$ = 18.6082 (15) Å
$b$ = 8.2295 (15) Å
$c$ = 14.986 (2) Å
$\beta$ = 92.050 (5)°
$V$ = 2293.4 (6) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 295 K
0.35 × 0.32 × 0.28 mm

### Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min}$ = 0.960, $T_{max}$ = 0.970
10958 measured reflections
2490 independent reflections
1536 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.051

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.189
$S$ = 1.09
2490 reflections
148 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H1$F$···O2$^{i}$ | 0.93 | 1.84 | 2.759 (3) | 176 |
| O1$W$—H1$E$···N3$^{ii}$ | 0.92 | 1.96 | 2.879 (3) | 173 |
| O1—H1···O1$W^{iii}$ | 0.82 | 1.75 | 2.558 (3) | 167 |

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2507).

## References

El Khadem, H., Mansour, H. A. R. & Meshreki, M. H. (1968). *J. Chem. Soc. C*, pp. 1329–1331.
Lin, J. R., Yao, J. Y. & Zhao, H. (2008). *Acta Cryst.* E**64**, o1843.
Olesen, P. H., Sorensen, A. R., Urso, B., Kurtzhals, P., Bowler, A. N., Ehrbar, U. & Hansen, B. F. (2003). *J. Med. Chem.* **46**, 3333–3341.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tian, L., Sun, Y., Li, H., Zheng, X., Cheng, Y., Liu, X. & Qian, B. (2005). *J. Inorg. Biochem.* **99**, 1646–1652.
Xiao, J., Wang, W. X. & Zhao, H. (2008). *Acta Cryst.* E**64**, o2085.
Zhao, H. (2009). *Acta Cryst.* E**65**, o1258.

# supporting information

*Acta Cryst.* (2010). E**66**, o3001 [https://doi.org/10.1107/S1600536810043813]

## 2-(4-Ethoxycarbonyl-5-methyl-1*H*-1,2,3-triazol-1-yl)acetic acid monohydrate

**Gai-Gai Wang and Hong Zhao**

### S1. Comment

Triazole derivatives have attracted considerable attention due to their biological activities (Olesen *et al.*, 2003; Tian *et al.*, 2005). Recently, we have reported the crystal structures of a few triazole compounds (Lin *et al.*, 2008; Xiao *et al.*, 2008; Zhao, 2009). As an extension of our work on the structural characterization of triazole derivatives, the crystal structure of the title compound is reported here.

In the molecule of the title compound (Fig. 1) bond lengths and angles have normal values. The crystal packing is stabilized by strong intermolecular O—H···N and O—H···O hydrogen bonds involving the triazole and water molecules (Fig. 2; Table 1) forming double layers parallel to the *ab* plane.

### S2. Experimental

The title compound was prepared from 2-azidoacetic acid according to the reported method (El Khadem *et al.*, 1968). Colourless prismatic crystal suitable for X-ray analysis were obtained by slow evaporation of a 95% ethanol/water solution.

### S3. Refinement

The water H atoms were located from a difference Fourier map but not refined [$U_{iso}$(H) = 1.5$U_{eq}$(O)]. All other H atoms were fixed geometrically and treated as riding with C—H = 0.96–0.97 Å, O—H = 0.82 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(C, O) for methyl and carboxy H atoms.

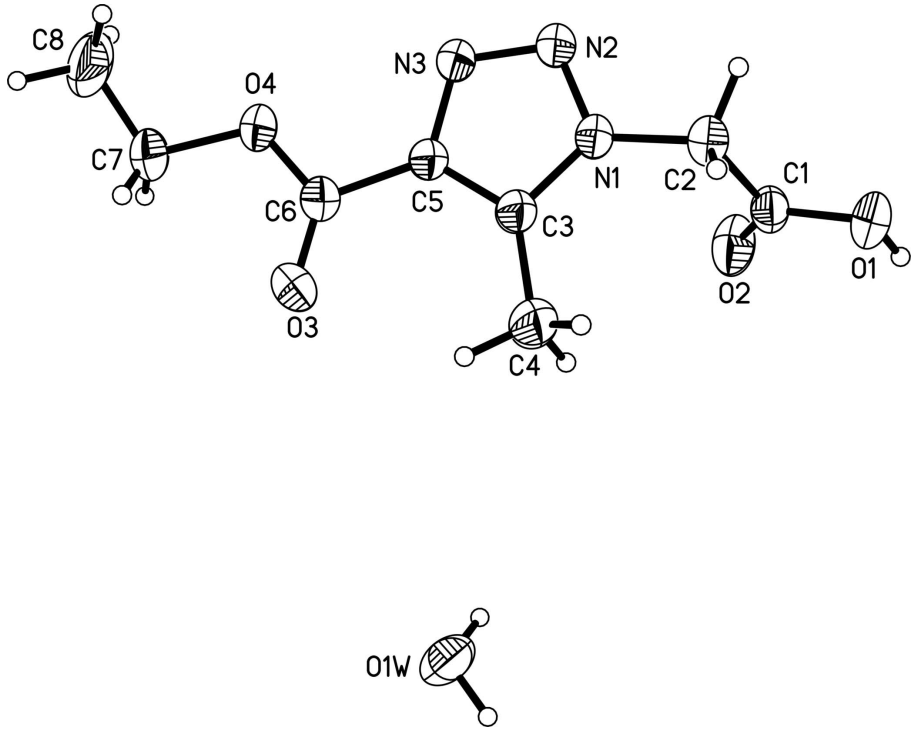


**Figure 1**

The molecular structure of the title compound, showing the atomic numbering scheme. The displacement ellipsoids are drawn at the 30% probability level.

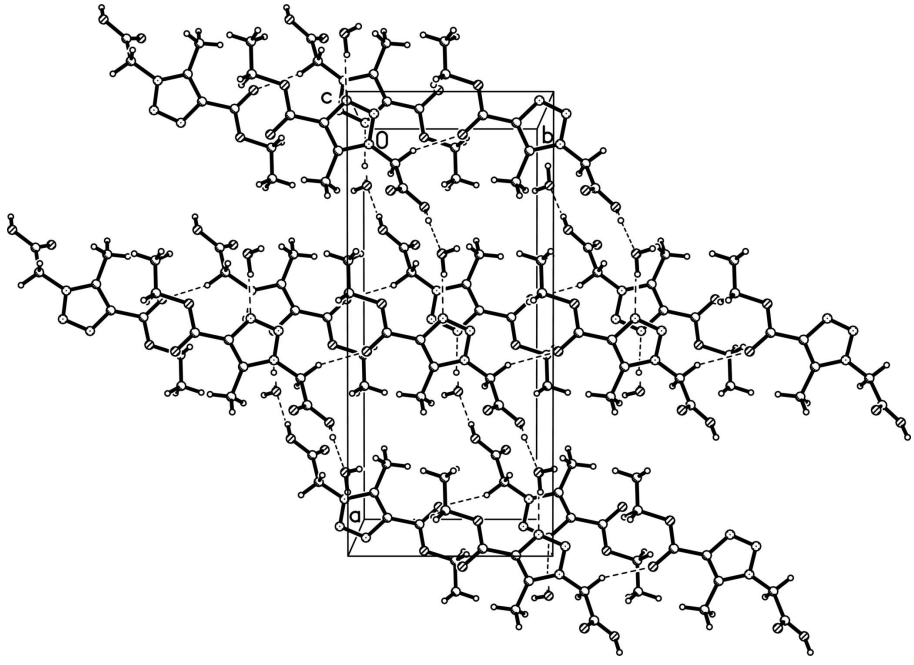


**Figure 2**

Packing diagram of the title compound, showing the structure along the *c* axis.

## 2-(4-Ethoxycarbonyl-5-methyl-1*H*-1,2,3-triazol-1-yl)acetic acid monohydrate

*Crystal data*

$C_8H_{11}N_3O_4·H_2O$
$M_r = 231.21$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 18.6082$ (15) Å
$b = 8.2295$ (15) Å
$c = 14.986$ (2) Å
$\beta = 92.050$ (5)°
$V = 2293.4$ (6) Å$^3$
$Z = 8$
$F(000) = 976$
$D_x = 1.339$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2099 reflections
$\theta = 2.7$–27.5°
$\mu = 0.11$ mm$^{-1}$
$T = 295$ K
Prism, colourless
0.35 × 0.32 × 0.28 mm

*Data collection*

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm$^{-1}$
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.960$, $T_{max} = 0.970$
10958 measured reflections
2490 independent reflections
1536 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.7°$
$h = -23\rightarrow23$
$k = -10\rightarrow10$
$l = -19\rightarrow19$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.189$
$S = 1.09$
2490 reflections
148 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0861P)^2 + 0.6922P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ 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 | 0.81442 (14) | −0.2524 (3) | 0.37722 (19) | 0.0552 (6) |
| C2 | 0.87508 (14) | −0.2098 (3) | 0.44180 (18) | 0.0611 (7) |
| H2A | 0.8554 | −0.1835 | 0.4991 | 0.073* |
| H2B | 0.9059 | −0.3041 | 0.4502 | 0.073* |

| | | | | |
|---|---|---|---|---|
| C3 | 0.90266 (14) | 0.0868 (3) | 0.41202 (18) | 0.0557 (6) |
| C4 | 0.83230 (16) | 0.1554 (4) | 0.4359 (3) | 0.0936 (12) |
| H4A | 0.7981 | 0.1402 | 0.3871 | 0.140* |
| H4B | 0.8376 | 0.2694 | 0.4481 | 0.140* |
| H4C | 0.8156 | 0.1012 | 0.4880 | 0.140* |
| C5 | 0.96537 (13) | 0.1568 (3) | 0.38496 (18) | 0.0542 (6) |
| C6 | 0.98182 (15) | 0.3313 (3) | 0.3765 (2) | 0.0631 (7) |
| N1 | 0.91816 (11) | −0.0742 (2) | 0.41279 (14) | 0.0552 (6) |
| N2 | 0.98618 (12) | −0.1025 (3) | 0.38793 (17) | 0.0658 (6) |
| N3 | 1.01479 (11) | 0.0386 (2) | 0.37055 (17) | 0.0629 (6) |
| O1 | 0.77303 (11) | −0.3646 (2) | 0.41090 (13) | 0.0687 (6) |
| H1 | 0.7401 | −0.3861 | 0.3750 | 0.103* |
| O2 | 0.80534 (12) | −0.1933 (3) | 0.30435 (15) | 0.0828 (7) |
| O3 | 0.93857 (12) | 0.4367 (2) | 0.38956 (19) | 0.0963 (8) |
| O4 | 1.04841 (10) | 0.3565 (2) | 0.35401 (16) | 0.0787 (7) |
| O1W | 0.66222 (12) | 0.5429 (3) | 0.31857 (17) | 0.1016 (9) |
| H1E | 0.6157 | 0.5328 | 0.3366 | 0.152* |
| H1F | 0.6724 | 0.4673 | 0.2751 | 0.152* |
| C7 | 1.07274 (18) | 0.5254 (4) | 0.3491 (3) | 0.0975 (13) |
| H7A | 1.0559 | 0.5733 | 0.2931 | 0.117* |
| H7B | 1.0531 | 0.5877 | 0.3975 | 0.117* |
| C8 | 1.1478 (2) | 0.5300 (5) | 0.3554 (4) | 0.1357 (19) |
| H8A | 1.1645 | 0.4680 | 0.4063 | 0.204* |
| H8B | 1.1635 | 0.6406 | 0.3620 | 0.204* |
| H8C | 1.1669 | 0.4846 | 0.3022 | 0.204* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0569 (15) | 0.0444 (13) | 0.0649 (16) | −0.0084 (11) | 0.0111 (12) | −0.0044 (12) |
| C2 | 0.0590 (16) | 0.0525 (15) | 0.0720 (17) | −0.0113 (12) | 0.0030 (13) | 0.0027 (13) |
| C3 | 0.0483 (14) | 0.0482 (14) | 0.0705 (16) | −0.0026 (11) | 0.0034 (12) | −0.0085 (12) |
| C4 | 0.0614 (19) | 0.068 (2) | 0.154 (3) | −0.0031 (16) | 0.031 (2) | −0.017 (2) |
| C5 | 0.0462 (14) | 0.0448 (13) | 0.0713 (16) | −0.0019 (11) | −0.0012 (12) | −0.0070 (12) |
| C6 | 0.0556 (16) | 0.0482 (14) | 0.085 (2) | −0.0050 (13) | −0.0067 (14) | −0.0044 (13) |
| N1 | 0.0482 (12) | 0.0445 (11) | 0.0729 (14) | −0.0073 (9) | 0.0042 (10) | −0.0021 (10) |
| N2 | 0.0493 (13) | 0.0470 (12) | 0.1013 (18) | −0.0040 (10) | 0.0066 (12) | 0.0008 (12) |
| N3 | 0.0462 (12) | 0.0470 (12) | 0.0956 (17) | −0.0039 (9) | 0.0059 (11) | −0.0033 (11) |
| O1 | 0.0652 (12) | 0.0675 (12) | 0.0739 (12) | −0.0251 (10) | 0.0078 (9) | −0.0006 (10) |
| O2 | 0.0906 (16) | 0.0848 (15) | 0.0724 (13) | −0.0321 (12) | −0.0074 (11) | 0.0157 (12) |
| O3 | 0.0744 (15) | 0.0479 (11) | 0.167 (2) | 0.0032 (11) | 0.0108 (15) | −0.0080 (13) |
| O4 | 0.0532 (11) | 0.0499 (11) | 0.1330 (19) | −0.0134 (9) | 0.0037 (11) | 0.0032 (11) |
| O1W | 0.0557 (13) | 0.122 (2) | 0.128 (2) | −0.0154 (12) | 0.0168 (13) | −0.0639 (16) |
| C7 | 0.075 (2) | 0.0488 (17) | 0.168 (4) | −0.0179 (15) | −0.011 (2) | 0.014 (2) |
| C8 | 0.095 (3) | 0.094 (3) | 0.216 (5) | −0.049 (2) | −0.031 (3) | 0.050 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O2 | 1.202 (3) | C6—O3 | 1.204 (3) |
| C1—O1 | 1.315 (3) | C6—O4 | 1.312 (3) |
| C1—C2 | 1.502 (4) | N1—N2 | 1.352 (3) |
| C2—N1 | 1.450 (3) | N2—N3 | 1.307 (3) |
| C2—H2A | 0.9700 | O1—H1 | 0.8200 |
| C2—H2B | 0.9700 | O4—C7 | 1.465 (3) |
| C3—N1 | 1.356 (3) | O1W—H1E | 0.9194 |
| C3—C5 | 1.375 (3) | O1W—H1F | 0.9251 |
| C3—C4 | 1.481 (4) | C7—C8 | 1.396 (5) |
| C4—H4A | 0.9600 | C7—H7A | 0.9700 |
| C4—H4B | 0.9600 | C7—H7B | 0.9700 |
| C4—H4C | 0.9600 | C8—H8A | 0.9600 |
| C5—N3 | 1.361 (3) | C8—H8B | 0.9600 |
| C5—C6 | 1.475 (3) | C8—H8C | 0.9600 |
| | | | |
| O2—C1—O1 | 124.6 (3) | O3—C6—C5 | 123.0 (3) |
| O2—C1—C2 | 124.7 (2) | O4—C6—C5 | 112.1 (2) |
| O1—C1—C2 | 110.7 (2) | N2—N1—C3 | 111.5 (2) |
| N1—C2—C1 | 113.4 (2) | N2—N1—C2 | 118.9 (2) |
| N1—C2—H2A | 108.9 | C3—N1—C2 | 129.4 (2) |
| C1—C2—H2A | 108.9 | N3—N2—N1 | 107.1 (2) |
| N1—C2—H2B | 108.9 | N2—N3—C5 | 108.7 (2) |
| C1—C2—H2B | 108.9 | C1—O1—H1 | 109.5 |
| H2A—C2—H2B | 107.7 | C6—O4—C7 | 117.3 (2) |
| N1—C3—C5 | 103.2 (2) | H1E—O1W—H1F | 111.4 |
| N1—C3—C4 | 124.1 (2) | C8—C7—O4 | 109.4 (3) |
| C5—C3—C4 | 132.7 (2) | C8—C7—H7A | 109.8 |
| C3—C4—H4A | 109.5 | O4—C7—H7A | 109.8 |
| C3—C4—H4B | 109.5 | C8—C7—H7B | 109.8 |
| H4A—C4—H4B | 109.5 | O4—C7—H7B | 109.8 |
| C3—C4—H4C | 109.5 | H7A—C7—H7B | 108.2 |
| H4A—C4—H4C | 109.5 | C7—C8—H8A | 109.5 |
| H4B—C4—H4C | 109.5 | C7—C8—H8B | 109.5 |
| N3—C5—C3 | 109.4 (2) | H8A—C8—H8B | 109.5 |
| N3—C5—C6 | 122.6 (2) | C7—C8—H8C | 109.5 |
| C3—C5—C6 | 127.9 (2) | H8A—C8—H8C | 109.5 |
| O3—C6—O4 | 124.8 (3) | H8B—C8—H8C | 109.5 |
| | | | |
| O2—C1—C2—N1 | 7.1 (4) | C5—C3—N1—C2 | 175.1 (3) |
| O1—C1—C2—N1 | −173.6 (2) | C4—C3—N1—C2 | −5.1 (4) |
| N1—C3—C5—N3 | 0.2 (3) | C1—C2—N1—N2 | −110.4 (3) |
| C4—C3—C5—N3 | −179.6 (3) | C1—C2—N1—C3 | 74.9 (3) |
| N1—C3—C5—C6 | −176.4 (3) | C3—N1—N2—N3 | −0.4 (3) |
| C4—C3—C5—C6 | 3.8 (5) | C2—N1—N2—N3 | −176.0 (2) |
| N3—C5—C6—O3 | −179.6 (3) | N1—N2—N3—C5 | 0.5 (3) |
| C3—C5—C6—O3 | −3.4 (5) | C3—C5—N3—N2 | −0.4 (3) |

| | | | |
|---|---|---|---|
| N3—C5—C6—O4 | −0.1 (4) | C6—C5—N3—N2 | 176.4 (3) |
| C3—C5—C6—O4 | 176.1 (3) | O3—C6—O4—C7 | 3.4 (5) |
| C5—C3—N1—N2 | 0.1 (3) | C5—C6—O4—C7 | −176.1 (3) |
| C4—C3—N1—N2 | 179.9 (3) | C6—O4—C7—C8 | 159.6 (4) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H1*F*···O2$^{i}$ | 0.93 | 1.84 | 2.759 (3) | 176 |
| O1*W*—H1*E*···N3$^{ii}$ | 0.92 | 1.96 | 2.879 (3) | 173 |
| O1—H1···O1*W*$^{iii}$ | 0.82 | 1.75 | 2.558 (3) | 167 |

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+1/2; (ii) *x*−1/2, *y*+1/2, *z*; (iii) *x*, *y*−1, *z*.