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ISSN 2414-3146

4,4′-(Acridine-2,7-diyl)bis(2-methylbut-3-yn-2-ol)

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Received 16 October 2025
Accepted 18 October 2025

Edited by I. Brito, University of Antofagasta, Chile

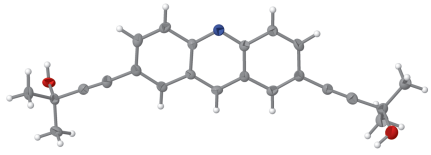
Keywords: acridine; π–π stacking; hydrogen bond; crystal structure.

CCDC reference: 2496569

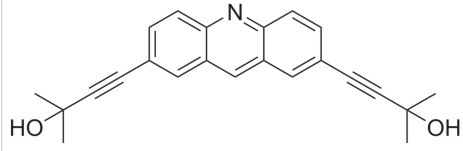
Structural data: full structural data are available from iucrdata.iucr.org

The title acridine derivative, $C_{23}H_{21}NO_2$, which has two 2-methylbut-3-yn-2-ol moieties at the 2,7-positions, was synthesized by Sonogashira coupling reaction. In the crystal, a columnar structure is formed by the π–π stacking of acridine units. The 2-methylbut-3-yn-2-ol moieties form intermolecular hydrogen bonds.

3D view



Chemical scheme



Structure description

Acridine is often used as a luminophore (Ryan *et al.*, 1997) and DNA-intercalator (Lerman, 1963). It is known to crystallize in seven polymorphic forms, in which various intermolecular interactions, *i.e.*, π–π, C—H–π, and C—H–N interactions, are observed (Mei & Wolf, 2004).

The title compound (Fig. 1) was synthesized by the Sonogashira coupling reaction of 2,7-dibromoacridine with 2-methylbut-3-yn-2-ol. The structure of the core acridine unit of the title compound is very similar to those of other 2,7-substituted acridine (Yamamura *et al.*, 2015). All the bond lengths and angles in the acridine unit are in expected ranges. The C12 and C13 atoms in a triple bond are within the least-square plane of the acridine unit (Fig. 2). In contrast, the C16 and C17 atoms in the other triple bond are separated from the plane. The distance of C16 from the plane is 0.179 (3) Å and that of C17 is 0.331 (3) Å.

In the crystal, a columnar structure was observed due to the π–π stacking of acridine units (Fig. 3). The distance between the least-square planes of the acridine units is 3.505 Å. The acridine unit is arranged in anti-fashion toward a neighbor acridine unit. Intermolecular hydrogen bonds also link molecules (Table 1). Two hydroxy groups of **1** form hydrogen bonds with two different molecules, between which another molecule is inserted.

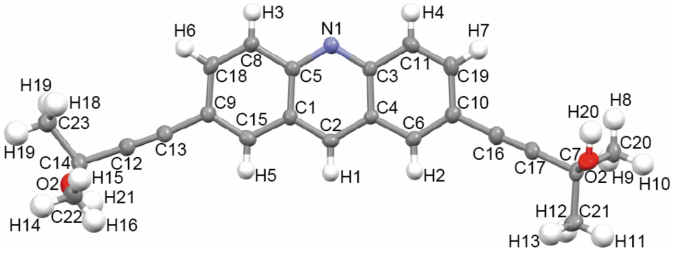


Figure 1
Molecular structure of the title compound with 50% probability ellipsoids.

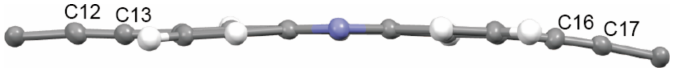


Figure 2
Side view of molecular structure. Terminal methyl and hydroxy groups are omitted for clarify.

Synthesis and crystallization

A mixture of 2,7-dibromoacridine (Vlassa *et al.*, 1995) (0.158 g, 47.0 mmol), 2-methylbut-3-yn-2-ol (0.15 ml, 1.5 mmol), $PdCl_2(PPh_3)_2$ (6.7 mg, 2 mol%), and CuI (1.6 mg, 2 mol%) were refluxed for 15 h in diisopropylamine (45 ml). After evaporation, the residue was extracted with CH_2Cl_2 then washed with water. After evaporation, the crude products were separated by silica-gel column chromatography to give the yellow powder of the title compound in 43% yield. Yellow crystals suitable for X-ray analysis were obtained form a $CHCl_3$/hexane solution.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.62 (*s*, 1H), 8.14 (*d*, J = 8.9 Hz, 2H), 8.09 (*d*, J = 1.7 Hz, 2H), 7.73 (*dd*, J = 1.7, 8.9 Hz, 2H), 2.08 (*s*, 2H), 1.70 (*s*, 12H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.1 (CH), 136.2 (C), 133.9 (CH), 132.3 (CH), 130.2 (CH), 127.1 (CH), 121.3 (C), 84.7 (C), 82.6 (C), 66.3 (C), 31.7 (CH_3); Analysis calculated for $C_{23}H_{21}NO_2$: C, 80.44; H, 6.16; N, 4.08; Found: C, 80.13; H, 5.99; N, 3.89.

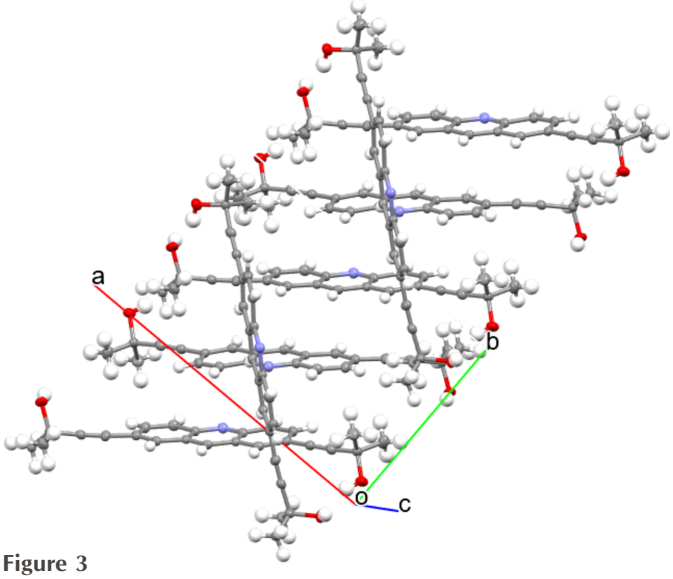


Figure 3
Packing of the title compound

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H20···O2^{i}	0.84	2.04	2.794 (4)	149
O2—H21···O1^{ii}	0.84	1.94	2.735 (4)	158

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{21}NO_2$
M_r	343.41
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	120
a, b, c (Å)	16.023 (13), 9.496 (7), 12.215 (10)
β (°)	99.646 (10)
V (Å^3)	1832 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.20 × 0.20 × 0.05
Data collection	
Diffractometer	Bruler APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.843, 0.915
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10340, 3058, 1605
R_{int}	0.093
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.585
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.122, 1.01
No. of reflections	3058
No. of parameters	241
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.28

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2019*/1 (Sheldrick, 2015*b*) and *Mercury* (Macrae *et al.*, 2020).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

Funding for this research was provided by: MEXT, Japan (grant No. 15H00985).

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full crystallographic data

IUCrData (2025). **10**, x250915 [https://doi.org/10.1107/S2414314625009150]

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Crystal data

$C_{23}H_{21}NO_2$
M_r = 343.41
Monoclinic, $P2_1/c$
a = 16.023 (13) Å
b = 9.496 (7) Å
c = 12.215 (10) Å
β = 99.646 (10)°
V = 1832 (2) Å^3
Z = 4
$F(000)$ = 728
D_x = 1.245 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 476 reflections
θ = 2.5–26.6°
μ = 0.08 mm^{-1}
T = 120 K
Prism, colorless
0.20 × 0.20 × 0.05 mm

Data collection

Bruler APEXII CCD diffractometer
Radiation source: fine-focus seald tube
φ and ω scan
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.843, T_{max} = 0.915
10340 measured reflections
3058 independent reflections
1605 reflections with $I > 2\sigma(I)$
R_{int} = 0.093
θ_{max} = 24.6°, θ_{min} = 1.3°
h = −18→18
k = −11→11
l = −14→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.122
S = 1.01
3058 reflections
241 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.030P)^2 + 1.P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.05976 (13)	0.6608 (2)	0.54622 (18)	0.0336 (6)
H20	0.046205	0.632400	0.480520	0.050*
O2	0.95459 (13)	−0.0895 (2)	0.8478 (2)	0.0391 (7)
H21	0.936554	−0.016649	0.875071	0.059*
N1	0.49494 (16)	0.2007 (3)	0.3889 (2)	0.0259 (7)
C1	0.57022 (19)	0.1892 (3)	0.5807 (3)	0.0222 (8)
C2	0.51585 (19)	0.2890 (3)	0.6130 (3)	0.0236 (8)
H1	0.523062	0.319641	0.688064	0.028*
C3	0.44299 (19)	0.2968 (3)	0.4223 (3)	0.0221 (8)
C4	0.45037 (19)	0.3441 (3)	0.5345 (3)	0.0201 (8)
C5	0.55663 (19)	0.1465 (3)	0.4664 (3)	0.0227 (8)
C6	0.39012 (19)	0.4417 (3)	0.5630 (3)	0.0238 (8)
H2	0.394231	0.472234	0.637781	0.029*
C7	0.1346 (2)	0.7473 (3)	0.5551 (3)	0.0269 (8)
C8	0.60864 (19)	0.0387 (3)	0.4338 (3)	0.0246 (8)
H3	0.600074	0.008741	0.358542	0.030*
C9	0.68701 (19)	0.0220 (3)	0.6217 (3)	0.0246 (8)
C10	0.3259 (2)	0.4925 (3)	0.4836 (3)	0.0263 (9)
C11	0.3759 (2)	0.3515 (3)	0.3423 (3)	0.0275 (9)
H4	0.370215	0.321267	0.267231	0.033*
C12	0.8143 (2)	−0.1025 (3)	0.7458 (3)	0.0286 (9)
C13	0.7548 (2)	−0.0444 (3)	0.6939 (3)	0.0257 (8)
C14	0.88472 (19)	−0.1841 (3)	0.8114 (3)	0.0277 (8)
C15	0.63762 (19)	0.1265 (3)	0.6553 (3)	0.0251 (8)
H5	0.648782	0.157535	0.730252	0.030*
C16	0.2620 (2)	0.5886 (3)	0.5099 (3)	0.0274 (9)
C17	0.2065 (2)	0.6609 (3)	0.5275 (3)	0.0256 (8)
C18	0.6699 (2)	−0.0219 (3)	0.5076 (3)	0.0274 (9)
H6	0.702629	−0.095674	0.483679	0.033*
C19	0.3198 (2)	0.4463 (3)	0.3715 (3)	0.0293 (9)
H7	0.276038	0.482228	0.316502	0.035*
C20	0.1141 (2)	0.8677 (3)	0.4733 (3)	0.0307 (9)
H8	0.102475	0.830157	0.397582	0.046*
H9	0.162315	0.932392	0.480441	0.046*
H10	0.064129	0.918263	0.489222	0.046*
C21	0.1557 (2)	0.7998 (3)	0.6747 (3)	0.0335 (9)
H11	0.108256	0.855329	0.692606	0.050*
H12	0.206642	0.858645	0.683083	0.050*
H13	0.165849	0.719074	0.725186	0.050*
C22	0.8534 (2)	−0.2498 (3)	0.9101 (3)	0.0352 (9)
H14	0.899577	−0.302739	0.954676	0.053*
H15	0.806286	−0.313714	0.883803	0.053*
H16	0.834173	−0.175585	0.955753	0.053*
C23	0.9165 (2)	−0.2961 (3)	0.7380 (3)	0.0382 (10)
H17	0.941707	−0.249937	0.679546	0.057*

H18	0.868982	−0.355098	0.704005	0.057*
H19	0.959236	−0.354900	0.783358	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0267 (13)	0.0324 (14)	0.0409 (18)	−0.0101 (11)	0.0031 (12)	−0.0138 (12)
O2	0.0330 (15)	0.0263 (14)	0.0553 (19)	−0.0053 (12)	−0.0010 (13)	−0.0019 (13)
N1	0.0270 (16)	0.0263 (15)	0.0247 (19)	0.0009 (14)	0.0048 (14)	0.0022 (13)
C1	0.0202 (18)	0.0209 (17)	0.026 (2)	−0.0004 (15)	0.0052 (16)	0.0019 (16)
C2	0.0246 (19)	0.0260 (18)	0.020 (2)	−0.0046 (16)	0.0036 (16)	−0.0035 (16)
C3	0.0223 (19)	0.0203 (18)	0.024 (2)	0.0011 (15)	0.0054 (16)	0.0012 (16)
C4	0.0214 (18)	0.0194 (17)	0.021 (2)	−0.0019 (15)	0.0069 (16)	−0.0002 (15)
C5	0.0240 (19)	0.0218 (18)	0.023 (2)	−0.0006 (16)	0.0048 (17)	0.0032 (16)
C6	0.025 (2)	0.0231 (18)	0.025 (2)	−0.0008 (16)	0.0082 (17)	−0.0020 (16)
C7	0.0214 (19)	0.0197 (17)	0.039 (2)	−0.0002 (15)	0.0037 (16)	−0.0033 (16)
C8	0.025 (2)	0.0248 (18)	0.025 (2)	0.0012 (16)	0.0070 (17)	0.0004 (16)
C9	0.0196 (19)	0.0246 (19)	0.030 (3)	−0.0018 (16)	0.0048 (17)	0.0004 (17)
C10	0.025 (2)	0.0237 (18)	0.031 (3)	0.0000 (16)	0.0081 (18)	0.0005 (17)
C11	0.030 (2)	0.0279 (19)	0.023 (2)	−0.0008 (17)	−0.0002 (17)	0.0030 (16)
C12	0.033 (2)	0.0233 (18)	0.031 (2)	0.0005 (17)	0.0073 (18)	0.0015 (17)
C13	0.025 (2)	0.0222 (18)	0.030 (2)	−0.0017 (17)	0.0034 (17)	−0.0004 (16)
C14	0.0227 (19)	0.0238 (18)	0.035 (2)	−0.0020 (16)	0.0001 (16)	−0.0005 (17)
C15	0.026 (2)	0.0258 (19)	0.023 (2)	−0.0051 (16)	0.0016 (16)	0.0018 (16)
C16	0.023 (2)	0.0285 (19)	0.032 (2)	0.0013 (17)	0.0071 (17)	0.0038 (17)
C17	0.027 (2)	0.0202 (18)	0.029 (2)	−0.0016 (16)	0.0020 (16)	0.0013 (15)
C18	0.026 (2)	0.0225 (19)	0.036 (3)	0.0017 (16)	0.0114 (18)	−0.0033 (17)
C19	0.024 (2)	0.030 (2)	0.034 (3)	0.0030 (16)	0.0052 (17)	0.0085 (18)
C20	0.0251 (19)	0.0263 (19)	0.040 (2)	0.0039 (15)	0.0053 (17)	0.0047 (17)
C21	0.039 (2)	0.0278 (19)	0.034 (2)	−0.0010 (17)	0.0065 (18)	−0.0082 (17)
C22	0.040 (2)	0.035 (2)	0.032 (2)	0.0013 (18)	0.0117 (18)	0.0105 (18)
C23	0.029 (2)	0.045 (2)	0.040 (3)	0.0041 (18)	0.0023 (18)	−0.0062 (19)

Geometric parameters (Å, º)

O1—C7	1.442 (4)	C10—C19	1.425 (5)
O1—H20	0.8400	C10—C16	1.448 (4)
O2—C14	1.446 (4)	C11—C19	1.362 (4)
O2—H21	0.8400	C11—H4	0.9500
N1—C3	1.344 (4)	C12—C13	1.189 (4)
N1—C5	1.351 (4)	C12—C14	1.487 (5)
C1—C2	1.388 (4)	C14—C22	1.516 (4)
C1—C15	1.422 (4)	C14—C23	1.533 (4)
C1—C5	1.435 (4)	C15—H5	0.9500
C2—C4	1.400 (4)	C16—C17	1.171 (4)
C2—H1	0.9500	C18—H6	0.9500
C3—C11	1.424 (4)	C19—H7	0.9500
C3—C4	1.427 (4)	C20—H8	0.9800

C4—C6	1.423 (4)	C20—H9	0.9800
C5—C8	1.419 (4)	C20—H10	0.9800
C6—C10	1.377 (4)	C21—H11	0.9800
C6—H2	0.9500	C21—H12	0.9800
C7—C17	1.498 (4)	C21—H13	0.9800
C7—C20	1.518 (4)	C22—H14	0.9800
C7—C21	1.527 (4)	C22—H15	0.9800
C8—C18	1.346 (4)	C22—H16	0.9800
C8—H3	0.9500	C23—H17	0.9800
C9—C15	1.374 (4)	C23—H18	0.9800
C9—C13	1.427 (4)	C23—H19	0.9800
C9—C18	1.436 (4)		

C7—O1—H20	109.5	O2—C14—C12	108.7 (3)
C14—O2—H21	109.5	O2—C14—C22	110.7 (3)
C3—N1—C5	117.6 (3)	C12—C14—C22	108.7 (3)
C2—C1—C15	123.3 (3)	O2—C14—C23	107.1 (2)
C2—C1—C5	118.0 (3)	C12—C14—C23	110.0 (3)
C15—C1—C5	118.7 (3)	C22—C14—C23	111.5 (3)
C1—C2—C4	119.6 (3)	C9—C15—C1	121.6 (3)
C1—C2—H1	120.2	C9—C15—H5	119.2
C4—C2—H1	120.2	C1—C15—H5	119.2
N1—C3—C11	118.2 (3)	C17—C16—C10	175.8 (4)
N1—C3—C4	123.3 (3)	C16—C17—C7	176.7 (3)
C11—C3—C4	118.4 (3)	C8—C18—C9	121.7 (3)
C2—C4—C6	122.5 (3)	C8—C18—H6	119.2
C2—C4—C3	118.2 (3)	C9—C18—H6	119.2
C6—C4—C3	119.3 (3)	C11—C19—C10	120.9 (3)
N1—C5—C8	118.3 (3)	C11—C19—H7	119.5
N1—C5—C1	123.3 (3)	C10—C19—H7	119.5
C8—C5—C1	118.4 (3)	C7—C20—H8	109.5
C10—C6—C4	120.9 (3)	C7—C20—H9	109.5
C10—C6—H2	119.6	H8—C20—H9	109.5
C4—C6—H2	119.6	C7—C20—H10	109.5
O1—C7—C17	109.4 (2)	H8—C20—H10	109.5
O1—C7—C20	107.1 (3)	H9—C20—H10	109.5
C17—C7—C20	110.8 (3)	C7—C21—H11	109.5
O1—C7—C21	107.8 (3)	C7—C21—H12	109.5
C17—C7—C21	109.8 (3)	H11—C21—H12	109.5
C20—C7—C21	111.9 (3)	C7—C21—H13	109.5
C18—C8—C5	121.2 (3)	H11—C21—H13	109.5
C18—C8—H3	119.4	H12—C21—H13	109.5
C5—C8—H3	119.4	C14—C22—H14	109.5
C15—C9—C13	123.5 (3)	C14—C22—H15	109.5
C15—C9—C18	118.3 (3)	H14—C22—H15	109.5
C13—C9—C18	118.3 (3)	C14—C22—H16	109.5
C6—C10—C19	119.4 (3)	H14—C22—H16	109.5
C6—C10—C16	122.5 (3)	H15—C22—H16	109.5

C19—C10—C16	118.0 (3)	C14—C23—H17	109.5
C19—C11—C3	121.0 (3)	C14—C23—H18	109.5
C19—C11—H4	119.5	H17—C23—H18	109.5
C3—C11—H4	119.5	C14—C23—H19	109.5
C13—C12—C14	175.9 (3)	H17—C23—H19	109.5
C12—C13—C9	174.1 (4)	H18—C23—H19	109.5
C15—C1—C2—C4	−179.2 (3)	C3—C4—C6—C10	−1.1 (4)
C5—C1—C2—C4	0.0 (4)	N1—C5—C8—C18	−178.4 (3)
C5—N1—C3—C11	−178.0 (3)	C1—C5—C8—C18	−0.5 (4)
C5—N1—C3—C4	0.4 (4)	C4—C6—C10—C19	0.1 (4)
C1—C2—C4—C6	177.0 (3)	C4—C6—C10—C16	178.1 (3)
C1—C2—C4—C3	−1.1 (4)	N1—C3—C11—C19	178.3 (3)
N1—C3—C4—C2	1.0 (4)	C4—C3—C11—C19	−0.2 (4)
C11—C3—C4—C2	179.3 (3)	C13—C9—C15—C1	−179.6 (3)
N1—C3—C4—C6	−177.3 (3)	C18—C9—C15—C1	0.9 (4)
C11—C3—C4—C6	1.1 (4)	C2—C1—C15—C9	176.1 (3)
C3—N1—C5—C8	176.1 (3)	C5—C1—C15—C9	−3.1 (4)
C3—N1—C5—C1	−1.6 (4)	C5—C8—C18—C9	−1.7 (5)
C2—C1—C5—N1	1.4 (4)	C15—C9—C18—C8	1.6 (5)
C15—C1—C5—N1	−179.3 (3)	C13—C9—C18—C8	−178.0 (3)
C2—C1—C5—C8	−176.3 (3)	C3—C11—C19—C10	−0.8 (5)
C15—C1—C5—C8	2.9 (4)	C6—C10—C19—C11	0.8 (5)
C2—C4—C6—C10	−179.2 (3)	C16—C10—C19—C11	−177.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H20···O2^{i}	0.84	2.04	2.794 (4)	149
O2—H21···O1^{ii}	0.84	1.94	2.735 (4)	158

Symmetry codes: (i) $x-1, -y+1/2, z-1/2$; (ii) $-x+1, y-1/2, -z+3/2$.