

Crystal structure of *N*-hydroxyquinoline-2-carboxamide monohydrate

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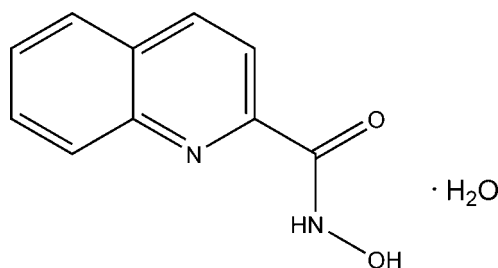
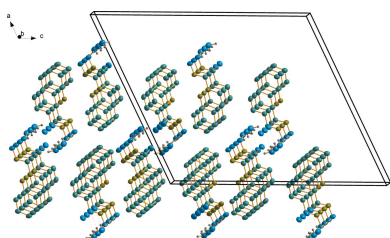
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The title compound, C₁₀H₈N₂O₂·H₂O, consists of an *N*-hydroxyquinoline-2-carboxamide molecule in the keto tautomeric form and a water molecule connected through an O—H···O hydrogen bond. The *N*-hydroxyquinoline-2-carboxamide molecule has a nearly planar structure [maximum deviation = 0.062 (1) Å] and only the hydroxy H atom deviates significantly from the molecule plane. In the crystal, π - π stacking between the aromatic rings [intercentroid distance = 3.887 (1) Å] and intermolecular O—H···O hydrogen bonds organize the crystal components into columns extending along the *b*-axis direction.

1. Chemical context

Hydroxamic acids are important bioligands that exhibit enzyme-inhibitory properties (Marmion *et al.*, 2013) and they have been studied extensively in coordination and bioinorganic chemistry (Ostrowska *et al.*, 2016; Golenya *et al.*, 2012*b*; Świętek-Kozłowska *et al.*, 2000; Dobosz *et al.*, 1999). They are widely used in the preparation of metallacrowns (Golenya *et al.*, 2012*a*; Gumienna-Kontecka *et al.*, 2013; Safyanova *et al.*, 2015) and as building blocks for synthesis of metal-organic frameworks and coordination polymers (Gumienna-Kontecka *et al.*, 2007; Golenya *et al.*, 2014; Pavlishchuk *et al.*, 2010, 2011).

N-Hydroxyquinoline-2-carboxamide, also known as quinoline-2-hydroxamic acid (QuinHA), has been used for the preparation of various metallacrown complexes (Stemmler *et al.*, 1999; Trivedi *et al.*, 2014; Jankolovits *et al.*, 2013). Presently, the Cambridge Structural Database (Groom *et al.*, 2016) contains ten entries on coordination compounds based on *N*-hydroxyquinoline-2-carboxamide, nine of which have been reported within the past four years.



Structural information about the title compound is absent in the literature, however, and this will be useful in controlling the purity of the synthesized ligand and metal complexes by

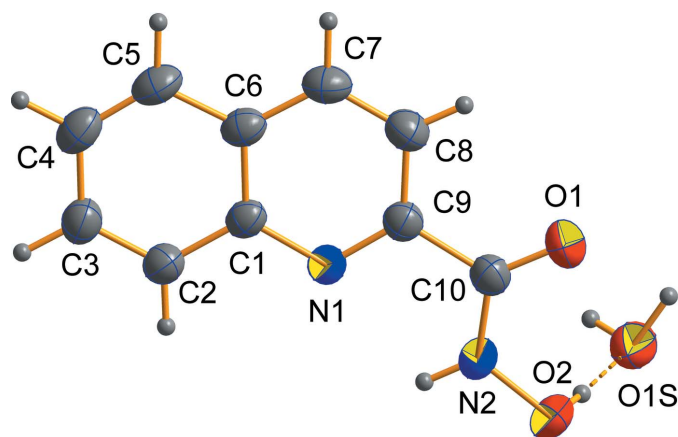


Figure 1
The asymmetric unit of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius. The dashed line indicates a hydrogen bond.

powder diffraction. It is well known that the products of such syntheses can be contaminated with impurities that result from hydrolysis or oxidation of the hydroxamic groups to the carboxylic group. In addition, syntheses of polynuclear complexes are often carried out with various metal-to-ligand ratios, so that in some cases an excessive quantity of the hydroxamic ligand can be present in the isolated samples.

2. Structural commentary

The molecular structure of the title compound is presented in Fig. 1. It consists of an *N*-hydroxyquinoline-2-carboxamide molecule in the keto tautomeric form [which is supported by the C=O [1.227 (2) Å] and C–N [1.317 (2) Å] bond lengths] and a water molecule. The carbonyl group possesses a *Z* conformation against the N1 atom of the quinoline moiety and *E* conformation against the hydroxy oxygen atom [torsion angles O2–N2–C10–O1 = 0.8 (2)° and N1–C9–C10–O1 = –177.33 (14)°]. The *N*-hydroxyquinoline-2-carboxamide

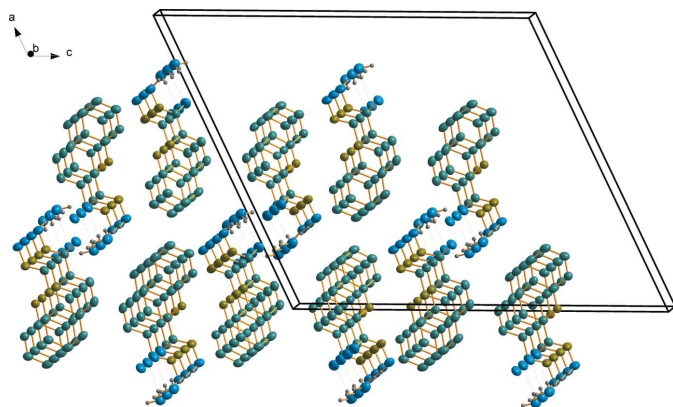


Figure 2
A packing diagram of the title compound. Hydrogen bonds (see Table 1) are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O1S–H1SA···O1 ⁱ	0.85 (2)	2.15 (3)	2.9404 (19)	155 (2)
O1S–H1SA···O2 ⁱⁱ	0.85 (2)	2.54 (2)	3.0850 (18)	124 (2)
O1S–H1SB···O1 ⁱⁱⁱ	0.93 (2)	1.85 (2)	2.7783 (18)	176 (2)
O2–H2···O1S	0.97 (2)	1.67 (2)	2.6407 (18)	175 (2)
C3–H3···O2 ⁱⁱⁱ	0.999 (16)	2.518 (16)	3.493 (2)	165.0 (15)
C4–H4···O2 ^{iv}	0.975 (19)	2.589 (18)	3.273 (2)	127.3 (12)
C5–H5···O1S ^v	0.967 (17)	2.593 (17)	3.547 (2)	169.0 (13)

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

molecule has an almost planar structure (non-hydrogen atoms are planar to within 0.03 Å). Only the H atom of the OH group deviates significantly from the molecular plane: the C–N–O–H torsion angle of –75.1 (13)° is defined by the O–H···O hydrogen bond between hydroxy group and the water molecule. The C–N and C–C bond lengths in the quinoline moiety are typical for 2-substituted pyridine derivatives (Moroz *et al.*, 2012; Strotmeyer *et al.*, 2003; Krämer & Fritsky, 2000).

3. Supramolecular features

In the crystal, molecules form columns along the *b* axis as a result of the π – π stacking interaction between parallel quinoline moieties [symmetry operation $x, y + 1, z$; interplanar separation 3.420 (1) Å, intercentroid distance 3.887 (1) Å, displacement 1.846 (1) Å]. These columns are linked pairwise by the O–H···O hydrogen bonds (Table 1) *via* the bridging water molecules (see Fig. 2). Each water molecule forms two donor hydrogen bonds [H···O1 = 1.85 (2) and 2.15 (2) Å] with the carbonyl oxygen atom O1 and one acceptor hydrogen bond with the O–H group of the hydroxamic function that is the strongest hydrogen bond in the crystal [H···O2 = 1.67 (2) Å]. This latter hydrogen bond results in a shortened H···H contact between the water and hydroxy hydrogen atoms [2.05 (3) Å]. The doubled columns are linked by weak N–H··· π (2.71 Å, 159°) as well as van der Waals interactions. Weak intermolecular C–H···O contacts (Table 1) are also observed in the crystal.

4. Database survey

A search of the Cambridge Structural Database (Groom *et al.*, 2016) reveals no crystal structures of isomeric *N*-hydroxyquinoline-carboxamides or their homologues. Two independent studies on the crystal structure of *N*-hydroxypicolinamide have been published recently (Chaiyaveij *et al.*, 2015; Safyanova *et al.*, 2016).

5. Synthesis and crystallization

The title compound was obtained by the reaction of a methanol solution of hydroxyamine with a mixture of quinaldic acid and ethyl chloroformate in dry methylene

chloride in the presence of *N*-methylmorpholine according to the reported procedure (Trivedi *et al.*, 2014). Light-yellow crystals suitable for X-ray diffraction were obtained from aqueous solution by slow evaporation at room temperature (yield 76%).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were found from the difference-Fourier maps and refined isotropically.

Acknowledgements

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Table 2

Experimental details.

Crystal data	
Chemical formula	C ₁₀ H ₈ N ₂ O ₂ ·H ₂ O
<i>M_r</i>	206.20
Crystal system, space group	Monoclinic, C2/c
Temperature (K)	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	21.613 (4), 3.8867 (4), 25.081 (5)
β (°)	115.37 (2)
<i>V</i> (Å ³)	1903.7 (6)
<i>Z</i>	8
Radiation type	Mo Kα
μ (mm ⁻¹)	0.11
Crystal size (mm)	0.4 × 0.1 × 0.1
Data collection	
Diffractometer	Agilent Xcalibur, Sapphire3
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2014)
<i>T_{min}</i> , <i>T_{max}</i>	0.730, 1.000
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	7711, 2167, 1387
<i>R_{int}</i>	0.040
(sin θ/λ) _{max} (Å ⁻¹)	0.650
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.040, 0.098, 0.97
No. of reflections	2167
No. of parameters	173
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.14, -0.17

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

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supporting information

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Crystal structure of *N*-hydroxyquinoline-2-carboxamide monohydrate

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

N-hydroxyquinoline-2-carboxamide monohydrate

Crystal data

$C_{10}H_8N_2O_2 \cdot H_2O$
 $M_r = 206.20$
 Monoclinic, *C2/c*
 $a = 21.613$ (4) Å
 $b = 3.8867$ (4) Å
 $c = 25.081$ (5) Å
 $\beta = 115.37$ (2)°
 $V = 1903.7$ (6) Å³
 $Z = 8$

$F(000) = 864$
 $D_x = 1.439$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 1341 reflections
 $\theta = 3.8$ – 27.4 °
 $\mu = 0.11$ mm⁻¹
 $T = 298$ K
 Needle, clear light yellow
 $0.4 \times 0.1 \times 0.1$ mm

Data collection

Agilent Xcalibur, Sapphire3
 diffractometer
 Radiation source: Enhance (Mo) X-ray Source
 Graphite monochromator
 Detector resolution: 16.1827 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (*CrysAlis PRO*; Agilent, 2014)
 $T_{\min} = 0.730$, $T_{\max} = 1.000$

7711 measured reflections
 2167 independent reflections
 1387 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$
 $\theta_{\text{max}} = 27.5$ °, $\theta_{\text{min}} = 3.3$ °
 $h = -28 \rightarrow 28$
 $k = -5 \rightarrow 4$
 $l = -32 \rightarrow 32$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.098$
 $S = 0.97$
 2167 reflections
 173 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 atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0454P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ 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.

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.32060 (5)	0.1130 (3)	0.55333 (5)	0.0582 (3)
O2	0.27884 (5)	0.3220 (3)	0.63530 (5)	0.0604 (4)
N1	0.46916 (5)	0.5267 (3)	0.65622 (5)	0.0381 (3)
N2	0.34314 (6)	0.3877 (4)	0.63808 (6)	0.0523 (4)
H2A	0.3705 (8)	0.508 (4)	0.6691 (8)	0.060 (5)*
H2	0.2492 (10)	0.479 (5)	0.6052 (10)	0.090*
H1SA	0.2274 (11)	0.899 (6)	0.5506 (10)	0.090*
H1SB	0.1922 (10)	0.628 (5)	0.5141 (10)	0.090*
C1	0.53537 (6)	0.6016 (3)	0.66788 (6)	0.0359 (3)
C2	0.57630 (7)	0.7759 (4)	0.72050 (7)	0.0424 (4)
H2B	0.5567 (7)	0.834 (4)	0.7473 (7)	0.050 (4)*
C3	0.64285 (8)	0.8497 (4)	0.73422 (8)	0.0469 (4)
H3	0.6734 (8)	0.966 (4)	0.7719 (7)	0.060 (5)*
C4	0.67118 (8)	0.7562 (4)	0.69502 (8)	0.0491 (4)
H4	0.7190 (8)	0.810 (4)	0.7053 (7)	0.055 (4)*
C5	0.63321 (8)	0.5923 (4)	0.64395 (8)	0.0476 (4)
H5	0.6504 (8)	0.527 (4)	0.6156 (7)	0.059 (5)*
C6	0.56373 (7)	0.5090 (3)	0.62831 (7)	0.0391 (3)
C7	0.52127 (8)	0.3386 (4)	0.57617 (7)	0.0461 (4)
H7	0.5385 (8)	0.275 (4)	0.5474 (7)	0.058 (5)*
C8	0.45526 (8)	0.2675 (4)	0.56466 (7)	0.0453 (4)
H8	0.4245 (8)	0.161 (4)	0.5306 (7)	0.054 (5)*
C9	0.43182 (7)	0.3633 (3)	0.60679 (6)	0.0380 (3)
C10	0.35991 (7)	0.2754 (4)	0.59651 (7)	0.0409 (4)
O1S	0.20068 (6)	0.7378 (3)	0.54961 (6)	0.0609 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0489 (6)	0.0750 (8)	0.0461 (7)	−0.0163 (5)	0.0158 (6)	−0.0158 (6)
O2	0.0396 (6)	0.0884 (9)	0.0583 (8)	−0.0119 (5)	0.0259 (6)	0.0006 (6)
N1	0.0342 (6)	0.0446 (7)	0.0360 (7)	−0.0008 (5)	0.0153 (5)	0.0006 (6)
N2	0.0363 (7)	0.0751 (10)	0.0480 (9)	−0.0148 (7)	0.0206 (7)	−0.0130 (8)
C1	0.0326 (7)	0.0384 (7)	0.0380 (8)	0.0029 (6)	0.0163 (6)	0.0065 (6)
C2	0.0378 (8)	0.0501 (9)	0.0412 (9)	−0.0006 (7)	0.0187 (7)	0.0014 (7)
C3	0.0376 (8)	0.0518 (9)	0.0488 (11)	−0.0046 (7)	0.0162 (8)	0.0013 (8)

C4	0.0363 (8)	0.0525 (10)	0.0616 (12)	0.0015 (7)	0.0240 (8)	0.0078 (8)
C5	0.0442 (9)	0.0510 (9)	0.0588 (11)	0.0057 (7)	0.0328 (9)	0.0065 (8)
C6	0.0395 (7)	0.0395 (8)	0.0430 (9)	0.0062 (6)	0.0221 (7)	0.0062 (7)
C7	0.0522 (9)	0.0505 (9)	0.0438 (10)	0.0055 (7)	0.0282 (8)	0.0008 (7)
C8	0.0481 (9)	0.0490 (9)	0.0385 (9)	-0.0015 (7)	0.0182 (8)	-0.0058 (7)
C9	0.0374 (7)	0.0387 (7)	0.0371 (9)	0.0003 (6)	0.0154 (7)	0.0025 (6)
C10	0.0391 (8)	0.0453 (8)	0.0357 (9)	-0.0023 (6)	0.0135 (7)	0.0020 (7)
O1S	0.0549 (7)	0.0724 (8)	0.0607 (8)	-0.0107 (6)	0.0298 (7)	-0.0151 (7)

Geometric parameters (Å, °)

O1—C10	1.2266 (17)	C4—H4	0.974 (16)
O2—N2	1.3851 (15)	C4—C5	1.349 (2)
O2—H2	0.97 (2)	C5—H5	0.966 (16)
N1—C1	1.3635 (17)	C5—C6	1.417 (2)
N1—C9	1.3169 (17)	C6—C7	1.401 (2)
N2—H2A	0.884 (18)	C7—H7	0.976 (17)
N2—C10	1.316 (2)	C7—C8	1.357 (2)
C1—C2	1.408 (2)	C8—H8	0.927 (16)
C1—C6	1.4183 (19)	C8—C9	1.404 (2)
C2—H2B	0.962 (16)	C9—C10	1.5017 (19)
C2—C3	1.358 (2)	O1S—H1SA	0.84 (2)
C3—H3	0.999 (17)	O1S—H1SB	0.93 (2)
C3—C4	1.410 (2)		
N2—O2—H2	103.8 (12)	C4—C5—C6	120.80 (15)
C9—N1—C1	117.93 (12)	C6—C5—H5	115.6 (10)
O2—N2—H2A	114.9 (11)	C5—C6—C1	118.21 (14)
C10—N2—O2	120.66 (14)	C7—C6—C1	117.81 (13)
C10—N2—H2A	124.5 (11)	C7—C6—C5	123.98 (14)
N1—C1—C2	118.95 (13)	C6—C7—H7	120.4 (9)
N1—C1—C6	121.61 (13)	C8—C7—C6	120.22 (14)
C2—C1—C6	119.44 (13)	C8—C7—H7	119.4 (9)
C1—C2—H2B	118.7 (9)	C7—C8—H8	124.1 (10)
C3—C2—C1	120.73 (15)	C7—C8—C9	118.11 (15)
C3—C2—H2B	120.6 (9)	C9—C8—H8	117.8 (10)
C2—C3—H3	122.3 (9)	N1—C9—C8	124.30 (13)
C2—C3—C4	119.85 (16)	N1—C9—C10	116.29 (12)
C4—C3—H3	117.8 (9)	C8—C9—C10	119.41 (14)
C3—C4—H4	119.2 (9)	O1—C10—N2	123.24 (14)
C5—C4—C3	120.96 (15)	O1—C10—C9	122.90 (13)
C5—C4—H4	119.8 (9)	N2—C10—C9	113.86 (13)
C4—C5—H5	123.6 (10)	H1SA—O1S—H1SB	102.7 (19)
O2—N2—C10—O1	0.8 (2)	C2—C3—C4—C5	-0.3 (2)
O2—N2—C10—C9	-178.96 (12)	C3—C4—C5—C6	-0.1 (2)
N1—C1—C2—C3	178.81 (13)	C4—C5—C6—C1	-0.2 (2)
N1—C1—C6—C5	-179.24 (12)	C4—C5—C6—C7	-179.86 (15)

N1—C1—C6—C7	0.4 (2)	C5—C6—C7—C8	179.76 (14)
N1—C9—C10—O1	-177.33 (14)	C6—C1—C2—C3	-1.3 (2)
N1—C9—C10—N2	2.48 (19)	C6—C7—C8—C9	-1.2 (2)
C1—N1—C9—C8	-1.4 (2)	C7—C8—C9—N1	1.9 (2)
C1—N1—C9—C10	178.08 (11)	C7—C8—C9—C10	-177.53 (13)
C1—C2—C3—C4	1.0 (2)	C8—C9—C10—O1	2.2 (2)
C1—C6—C7—C8	0.1 (2)	C8—C9—C10—N2	-178.00 (14)
C2—C1—C6—C5	0.91 (19)	C9—N1—C1—C2	-179.94 (12)
C2—C1—C6—C7	-179.44 (13)	C9—N1—C1—C6	0.20 (19)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1 <i>S</i> —H1 <i>SA</i> ...O1 ⁱ	0.85 (2)	2.15 (3)	2.9404 (19)	155 (2)
O1 <i>S</i> —H1 <i>SA</i> ...O2 ⁱ	0.85 (2)	2.54 (2)	3.0850 (18)	124 (2)
O1 <i>S</i> —H1 <i>SB</i> ...O1 ⁱⁱ	0.93 (2)	1.85 (2)	2.7783 (18)	176 (2)
O2—H2...O1 <i>S</i>	0.97 (2)	1.67 (2)	2.6407 (18)	175 (2)
C3—H3...O2 ⁱⁱⁱ	0.999 (16)	2.518 (16)	3.493 (2)	165.0 (15)
C4—H4...O2 ^{iv}	0.975 (19)	2.589 (18)	3.273 (2)	127.3 (12)
C5—H5...O1 <i>S</i> ^v	0.967 (17)	2.593 (17)	3.547 (2)	169.0 (13)

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