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# (2-Hydroxyethyl)(propyl)azanium 2-[(2-carboxyphenyl)disulfanyl]benzoate monohydrate

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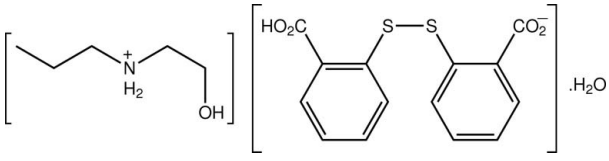
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.068; $wR$ factor = 0.155; data-to-parameter ratio = 13.6.

With the exception of the terminal hydroxy group [N—C—C—O = 53.8 (5)°], the cation of the title salt hydrate, $C_5H_{14}NO^+\cdot C_{14}H_9O_4S_2^-\cdot H_2O$, is a straight chain. A twisted conformation is found for the anion [C—S—S—C = −87.44 (16)°]. In the crystal, the anions self-assemble into a helical supramolecular chain *via* charge-assisted O—H···O$_c$ hydrogen bonds. These chains are connected into a three-dimensional network *via* N—H···O$_c$, N—H···O$_w$, O$_h$—H···O$_{cb}$, and O$_w$—H···O$_c$ hydrogen-bonding interactions (c = carboxylate, w = water, h = hydroxy and cb = carbonyl).

## Related literature

For related studies on co-crystal/salt formation involving 2-[(2-carboxyphenyl)disulfanyl]benzoic acid, see: Broker & Tiekink (2007); Broker *et al.* (2008). For software for searching the Cambridge Structural Database, see: Bruno *et al.* (2002).




## Experimental

### Crystal data

$C_5H_{14}NO^+\cdot C_{14}H_9O_4S_2^-\cdot H_2O$
$M_r$ = 427.52
Monoclinic, $P2_1/n$
$a$ = 8.1207 (16) Å
$b$ = 17.714 (4) Å
$c$ = 14.483 (3) Å
$\beta$ = 99.58 (3)°
$V$ = 2054.3 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.30 mm$^{-1}$
$T$ = 173 K
0.20 × 0.20 × 0.05 mm

### Data collection

Rigaku AFC12/SATURN724 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.803, $T_{max}$ = 1.000
12346 measured reflections
3600 independent reflections
3316 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.051

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.155
$S$ = 1.17
3600 reflections
265 parameters
5 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.11 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.43 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2*o*···O3$^{i}$ | 0.84 | 1.70 | 2.535 (4) | 178 |
| N1—H1n···O4$^{ii}$ | 0.90 | 2.10 | 2.887 (4) | 146 |
| N1—H2n···O1w$^{iii}$ | 0.90 | 1.92 | 2.773 (5) | 158 |
| O5—H5o···O1$^{iv}$ | 0.84 | 1.94 | 2.751 (5) | 162 |
| O1w—H1w···O4$^{v}$ | 0.84 | 1.99 | 2.823 (5) | 174 |
| O1w—H2w···O3 | 0.84 | 2.26 | 3.036 (5) | 154 |

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

Data collection: *CrystalClear* (Rigaku/MSC, 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: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PB2024).

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# supporting information

*Acta Cryst.* (2010). E**66**, o748 [doi:10.1107/S1600536810007658]

# (2-Hydroxyethyl)(propyl)azanium 2-[(2-carboxyphenyl)disulfanyl]benzoate monohydrate

**Grant A. Broker and Edward R. T. Tiekink**

## S1. Comment

The title salt hydrate, (I), was obtained during crystallisation experiments involving various N-containing species with the dicarboxylic acid, 2-[(2-carboxyphenyl)disulfanyl]benzoic acid (Broker & Tiekink, 2007; Broker *et al.*, 2008). The asymmetric unit of (I) comprises an aminium cation, Fig. 1, a uninegative 2,2′-dithio(benzoic acid)benzoate anion, Fig. 2, and a solvent water molecule of crystallisation.

The cation is linear with the exception of the terminal hydroxyl group which is twisted out of the chain as seen in the O5–C15–C16–N1 torsion angle of 53.8 (5) °. Confirmation of protonation of the amine-N1 atom during crystallisation is seen in the pattern of hydrogen bonding interactions, see below. A search of the CSD (Bruno *et al.*, 2002) suggests that this is the first structural characterisation reported for the 2-hydroxyethyl(propyl)aminium cation. The 2,2′-dithio(benzoic acid)benzoate molecule is twisted [C3–S1–S2–C10 = -87.44 (16) °] in accord with expectation with the conformation stabilised by intramolecular S···O interactions of 2.633 (3) Å for S2···O3 and 2.647 (3) Å for S3···O4 (Broker & Tiekink (2007). Confirmation that the C8—O1, O2 residue is in the acid form is found in the disparity of the C1–O1 and C1–O2 bond distances, *i.e.* 1.212 (4) and 1.316 (4) Å, respectively, compared with the equivalence of the C8–O3 and C8–O4 bond distances of 1.266 (4) and 1.247 (4) Å, respectively. The O1-carboxylic acid group is co-planar with the benzene ring to which it is connected with the C3–C2–C1–O1 and torsion angle being -2.9 (5) Å, but the O3-carboxylate group is not: the O3–C8–C9–C10 torsion angle being -21.0 (5) °.

The most distinctive feature of the crystal packing is supramolecular chain formed via self-association between 2,2′-dithio(benzoic acid)benzoate anions, as normally seen for such species (Broker & Tiekink, 2007), Fig. 2 and Table 1. The chain has a helical topology being generated by $2_1$-screw symmetry along the *b* axis of the monoclinic unit cell. The next most prominent O–H···O interaction involves the O5-hydroxyl group and the carbonyl-O1 atom. The remaining carboxylate-O4 associates with the water molecule of crystallization. The water molecule also forms a hydrogen bond with a neighbouring carboxylate-O3 atom and with a centrosymmetrically related water molecule to form a 12-membered $\{\cdots OCO\cdots HOH\}_2$ synthon. The aminium-H2n atom forms a donor interaction to a O1w-water molecule so that the latter participates in three hydrogen bonds. The second ammonium-H1n atom forms a N—H···O hydrogen bond with the carboxylate-O4 atom which is also connected to the O1w-water molecule and therefore closes a 12-membered centrosymmetric $\{\cdots O\cdots HNH\cdots OH\}_2$ synthon. In this way the components of the crystal structure are linked into a 3-D network, Table 1.

## S2. Experimental

The title salt (I) was obtained by dissolving 2-[(2-carboxyphenyl)disulfanyl]benzoic acid (0.100 g, Fluka) in ethanol (20 ml) to which was added the amine in 1:1, 1:2 and 1:3 stoichiometric ratios in three separate experiments. Regardless of the stoichiometry, only crystals of (I) were harvested as proved by multiple unit cell determinations, m.pt. 429–431 K

### S3. Refinement

The H-atoms located from difference maps but placed in their idealised positions (O–H = 0.84 Å, N–H = 0.90 Å, and C–H 0.93-0.97 Å) and were included in the refinement in the riding model approximation with $U_{iso}$(H) set to 1.2-1.5$U_{eq}$(carrier atom). The maximum and minimum residual electron density peaks of 1.11 and 0.43 e Å$^{-3}$, respectively, were located 0.91 Å and 0.60 Å from the H15b and O1w atoms, respectively.

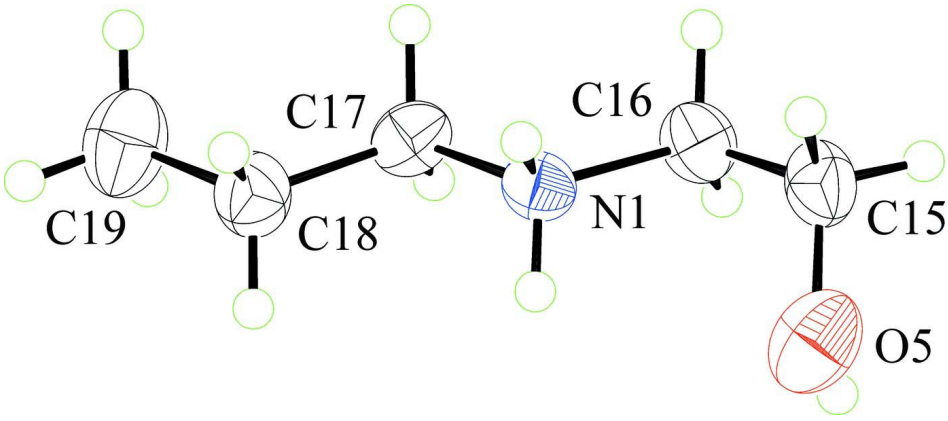


**Figure 1**

Molecular structure of the cation in (I) showing atom-labelling scheme and displacement ellipsoids at the 70% probability level.

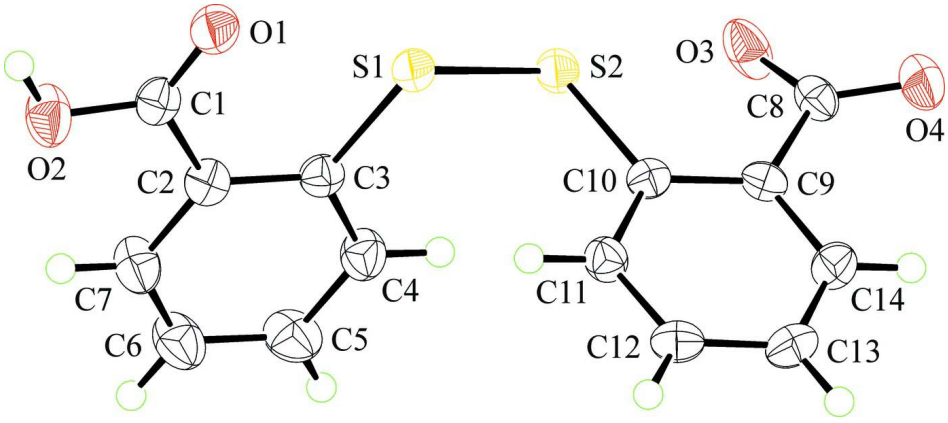


**Figure 2**

Molecular structure of the anion in (I) showing atom-labelling scheme and displacement ellipsoids at the 70% probability level.

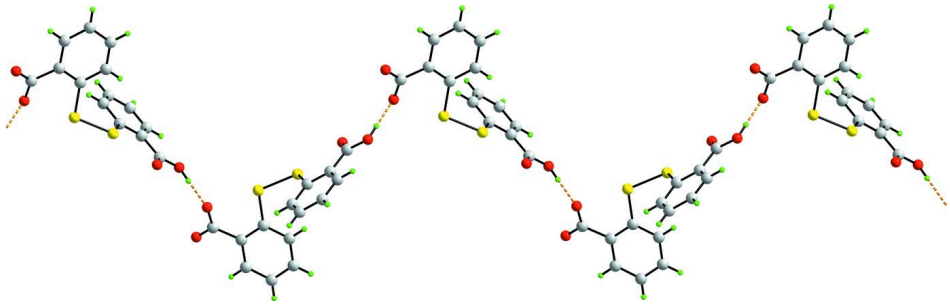

**Figure 3**

Supramolecular chain formation in (I) mediated by charge-assisted O–H···O$^-$ (orange dashed lines) hydrogen bonding. Colour code: S, yellow; O, red; N, blue; C, grey; H, green.

### (2-Hydroxyethyl)(propyl)azanium 2-[(2-carboxyphenyl)disulfanyl]benzoate monohydrate

*Crystal data*

$C_5H_{14}NO^+·C_{14}H_9O_4S_2^-·H_2O$
$M_r$ = 427.52
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a$ = 8.1207 (16) Å
$b$ = 17.714 (4) Å
$c$ = 14.483 (3) Å
$\beta$ = 99.58 (3)°
$V$ = 2054.3 (7) Å$^3$
$Z$ = 4
$F(000)$ = 904
$D_x$ = 1.382 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 6664 reflections
$\theta$ = 3.4–30.5°
$\mu$ = 0.30 mm$^{-1}$
$T$ = 173 K
Prism, colourless
0.20 × 0.20 × 0.05 mm

*Data collection*

Rigaku AFC12K/SATURN724 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.803, $T_{max}$ = 1.000
12346 measured reflections
3600 independent reflections
3316 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.051
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 3.4°
$h$ = −9→9
$k$ = −21→19
$l$ = −17→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.155
$S$ = 1.17
3600 reflections
265 parameters
5 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
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 2.9819P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.11 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.43 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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.69957 (11) | 0.89493 (5) | 0.68210 (6) | 0.0297 (3) |
| S2 | 0.63482 (11) | 0.78938 (5) | 0.62977 (6) | 0.0301 (3) |
| O1 | 0.8096 (3) | 1.01510 (14) | 0.78455 (18) | 0.0366 (6) |
| O2 | 1.0446 (3) | 1.07927 (16) | 0.7855 (2) | 0.0442 (7) |
| H2o | 1.0216 | 1.1032 | 0.8317 | 0.066* |
| O3 | 0.5300 (3) | 0.65217 (14) | 0.5766 (2) | 0.0436 (7) |
| O4 | 0.2893 (3) | 0.62652 (15) | 0.48483 (19) | 0.0426 (7) |
| O5 | 0.4552 (5) | 0.9986 (2) | 1.1203 (3) | 0.0670 (10) |
| H5O | 0.3620 | 0.9926 | 1.1373 | 0.101* |
| O1W | 0.6571 (5) | 0.5265 (2) | 0.4645 (3) | 0.0763 (11) |
| H1W | 0.6755 | 0.4821 | 0.4838 | 0.114* |
| H2W | 0.5987 | 0.5504 | 0.4974 | 0.114* |
| N1 | 0.4341 (4) | 0.87963 (18) | 0.9850 (2) | 0.0387 (8) |
| H1N | 0.5317 | 0.8935 | 0.9685 | 0.046* |
| H2N | 0.3630 | 0.9187 | 0.9728 | 0.046* |
| C1 | 0.9391 (4) | 1.0248 (2) | 0.7550 (2) | 0.0300 (8) |
| C2 | 0.9888 (4) | 0.9776 (2) | 0.6796 (2) | 0.0298 (8) |
| C3 | 0.8909 (4) | 0.9169 (2) | 0.6417 (2) | 0.0274 (7) |
| C4 | 0.9445 (5) | 0.8745 (2) | 0.5709 (3) | 0.0353 (9) |
| H4 | 0.8798 | 0.8342 | 0.5443 | 0.042* |
| C5 | 1.0921 (5) | 0.8913 (2) | 0.5397 (3) | 0.0428 (10) |
| H5 | 1.1263 | 0.8624 | 0.4928 | 0.051* |
| C6 | 1.1889 (5) | 0.9512 (2) | 0.5783 (3) | 0.0444 (10) |
| H6 | 1.2884 | 0.9627 | 0.5575 | 0.053* |
| C7 | 1.1376 (5) | 0.9937 (2) | 0.6473 (3) | 0.0386 (9) |
| H7 | 1.2030 | 1.0339 | 0.6731 | 0.046* |
| C8 | 0.4090 (4) | 0.66936 (19) | 0.5131 (2) | 0.0286 (8) |
| C9 | 0.4176 (4) | 0.74599 (19) | 0.4703 (2) | 0.0264 (7) |
| C10 | 0.5159 (4) | 0.80430 (18) | 0.5153 (2) | 0.0238 (7) |
| C11 | 0.5186 (4) | 0.8742 (2) | 0.4707 (2) | 0.0292 (8) |
| H11 | 0.5838 | 0.9132 | 0.5001 | 0.035* |
| C12 | 0.4245 (4) | 0.8856 (2) | 0.3827 (2) | 0.0324 (8) |
| H12 | 0.4263 | 0.9325 | 0.3540 | 0.039* |
| C13 | 0.3281 (5) | 0.8281 (2) | 0.3371 (2) | 0.0352 (9) |
| H13 | 0.2664 | 0.8357 | 0.2777 | 0.042* |

| | | | | |
|---|---|---|---|---|
| C14 | 0.3250 (4) | 0.7591 (2) | 0.3812 (2) | 0.0330 (8) |
| H14 | 0.2598 | 0.7204 | 0.3510 | 0.040* |
| C15 | 0.5417 (6) | 0.9308 (3) | 1.1412 (3) | 0.0498 (11) |
| H15A | 0.5489 | 0.9203 | 1.2075 | 0.060* |
| H15B | 0.6546 | 0.9367 | 1.1286 | 0.060* |
| C16 | 0.4617 (6) | 0.8648 (2) | 1.0865 (3) | 0.0474 (11) |
| H16A | 0.5326 | 0.8207 | 1.0999 | 0.057* |
| H16B | 0.3555 | 0.8538 | 1.1059 | 0.057* |
| C17 | 0.3663 (5) | 0.8148 (2) | 0.9263 (3) | 0.0427 (10) |
| H17A | 0.2652 | 0.7974 | 0.9469 | 0.051* |
| H17B | 0.4467 | 0.7739 | 0.9356 | 0.051* |
| C18 | 0.3278 (6) | 0.8329 (3) | 0.8244 (3) | 0.0527 (11) |
| H18A | 0.4288 | 0.8499 | 0.8032 | 0.063* |
| H18B | 0.2471 | 0.8737 | 0.8146 | 0.063* |
| C19 | 0.2579 (7) | 0.7645 (3) | 0.7669 (4) | 0.0674 (14) |
| H19A | 0.2345 | 0.7779 | 0.7019 | 0.101* |
| H19B | 0.1568 | 0.7481 | 0.7870 | 0.101* |
| H19C | 0.3383 | 0.7243 | 0.7757 | 0.101* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0292 (5) | 0.0334 (5) | 0.0270 (5) | −0.0037 (4) | 0.0063 (4) | −0.0028 (4) |
| S2 | 0.0311 (5) | 0.0289 (5) | 0.0283 (5) | −0.0037 (4) | −0.0004 (4) | 0.0062 (4) |
| O1 | 0.0369 (15) | 0.0395 (15) | 0.0347 (14) | −0.0020 (11) | 0.0097 (11) | −0.0078 (12) |
| O2 | 0.0370 (15) | 0.0421 (16) | 0.0548 (18) | −0.0075 (12) | 0.0114 (13) | −0.0189 (14) |
| O3 | 0.0340 (15) | 0.0321 (14) | 0.0610 (18) | −0.0064 (11) | −0.0027 (13) | 0.0199 (14) |
| O4 | 0.0427 (16) | 0.0370 (15) | 0.0471 (16) | −0.0134 (12) | 0.0048 (13) | 0.0007 (13) |
| O5 | 0.075 (2) | 0.066 (2) | 0.068 (2) | −0.011 (2) | 0.0340 (19) | −0.0046 (19) |
| O1W | 0.077 (3) | 0.049 (2) | 0.109 (3) | −0.0073 (19) | 0.033 (2) | −0.017 (2) |
| N1 | 0.0402 (19) | 0.0356 (18) | 0.0403 (18) | 0.0005 (14) | 0.0066 (14) | 0.0047 (15) |
| C1 | 0.0293 (19) | 0.0285 (19) | 0.0306 (19) | 0.0029 (15) | 0.0004 (15) | 0.0008 (16) |
| C2 | 0.0290 (19) | 0.0286 (18) | 0.0312 (19) | 0.0028 (14) | 0.0034 (15) | 0.0002 (16) |
| C3 | 0.0264 (18) | 0.0294 (18) | 0.0261 (17) | 0.0026 (14) | 0.0038 (14) | 0.0017 (15) |
| C4 | 0.037 (2) | 0.031 (2) | 0.038 (2) | −0.0029 (16) | 0.0060 (16) | −0.0062 (17) |
| C5 | 0.042 (2) | 0.043 (2) | 0.048 (2) | 0.0028 (18) | 0.0229 (19) | −0.006 (2) |
| C6 | 0.038 (2) | 0.043 (2) | 0.056 (3) | −0.0059 (18) | 0.0198 (19) | −0.008 (2) |
| C7 | 0.032 (2) | 0.035 (2) | 0.050 (2) | −0.0036 (16) | 0.0111 (17) | −0.0084 (19) |
| C8 | 0.0268 (18) | 0.0259 (18) | 0.0354 (19) | −0.0021 (14) | 0.0116 (15) | −0.0018 (16) |
| C9 | 0.0260 (17) | 0.0273 (18) | 0.0276 (17) | 0.0015 (14) | 0.0095 (14) | 0.0026 (15) |
| C10 | 0.0225 (17) | 0.0258 (17) | 0.0238 (16) | 0.0048 (13) | 0.0061 (13) | −0.0007 (14) |
| C11 | 0.0290 (18) | 0.0268 (18) | 0.0311 (19) | −0.0013 (14) | 0.0034 (14) | 0.0023 (16) |
| C12 | 0.038 (2) | 0.0316 (19) | 0.0288 (18) | 0.0047 (16) | 0.0087 (15) | 0.0064 (16) |
| C13 | 0.039 (2) | 0.041 (2) | 0.0242 (18) | 0.0041 (17) | 0.0021 (15) | 0.0026 (17) |
| C14 | 0.036 (2) | 0.034 (2) | 0.0294 (19) | −0.0019 (16) | 0.0056 (15) | −0.0054 (17) |
| C15 | 0.048 (3) | 0.065 (3) | 0.037 (2) | −0.002 (2) | 0.0095 (19) | 0.002 (2) |
| C16 | 0.058 (3) | 0.047 (3) | 0.039 (2) | 0.001 (2) | 0.013 (2) | 0.012 (2) |
| C17 | 0.047 (2) | 0.033 (2) | 0.050 (2) | −0.0032 (17) | 0.0146 (19) | −0.0003 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C18 | 0.063 (3) | 0.047 (3) | 0.047 (2) | −0.002 (2) | 0.004 (2) | −0.004 (2) |
| C19 | 0.070 (3) | 0.072 (3) | 0.062 (3) | −0.019 (3) | 0.014 (3) | −0.016 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C3 | 1.791 (3) | C7—H7 | 0.9300 |
| S1—S2 | 2.0528 (13) | C8—C9 | 1.498 (5) |
| S2—C10 | 1.793 (3) | C9—C10 | 1.399 (5) |
| O1—C1 | 1.212 (4) | C9—C14 | 1.400 (5) |
| O2—C1 | 1.316 (4) | C10—C11 | 1.398 (5) |
| O2—H2O | 0.8400 | C11—C12 | 1.387 (5) |
| O3—C8 | 1.266 (4) | C11—H11 | 0.9300 |
| O4—C8 | 1.247 (4) | C12—C13 | 1.384 (5) |
| O5—C15 | 1.400 (6) | C12—H12 | 0.9300 |
| O5—H5O | 0.8401 | C13—C14 | 1.381 (5) |
| O1W—H1W | 0.8401 | C13—H13 | 0.9300 |
| O1W—H2W | 0.8401 | C14—H14 | 0.9300 |
| N1—C16 | 1.473 (5) | C15—C16 | 1.499 (6) |
| N1—C17 | 1.479 (5) | C15—H15A | 0.9700 |
| N1—H1N | 0.9000 | C15—H15B | 0.9700 |
| N1—H2N | 0.9000 | C16—H16A | 0.9700 |
| C1—C2 | 1.485 (5) | C16—H16B | 0.9700 |
| C2—C3 | 1.394 (5) | C17—C18 | 1.491 (6) |
| C2—C7 | 1.395 (5) | C17—H17A | 0.9700 |
| C3—C4 | 1.399 (5) | C17—H17B | 0.9700 |
| C4—C5 | 1.381 (5) | C18—C19 | 1.526 (6) |
| C4—H4 | 0.9300 | C18—H18A | 0.9700 |
| C5—C6 | 1.383 (6) | C18—H18B | 0.9700 |
| C5—H5 | 0.9300 | C19—H19A | 0.9600 |
| C6—C7 | 1.369 (5) | C19—H19B | 0.9600 |
| C6—H6 | 0.9300 | C19—H19C | 0.9600 |
| | | | |
| C3—S1—S2 | 105.07 (12) | C12—C11—H11 | 119.8 |
| C10—S2—S1 | 105.81 (12) | C10—C11—H11 | 119.8 |
| C1—O2—H2O | 114.7 | C13—C12—C11 | 120.8 (3) |
| C15—O5—H5O | 105.7 | C13—C12—H12 | 119.6 |
| H1W—O1W—H2W | 111.6 | C11—C12—H12 | 119.6 |
| C16—N1—C17 | 114.5 (3) | C14—C13—C12 | 118.9 (3) |
| C16—N1—H1N | 108.6 | C14—C13—H13 | 120.6 |
| C17—N1—H1N | 108.6 | C12—C13—H13 | 120.6 |
| C16—N1—H2N | 108.6 | C13—C14—C9 | 121.7 (3) |
| C17—N1—H2N | 108.6 | C13—C14—H14 | 119.2 |
| H1N—N1—H2N | 107.6 | C9—C14—H14 | 119.2 |
| O1—C1—O2 | 122.7 (3) | O5—C15—C16 | 113.4 (4) |
| O1—C1—C2 | 122.5 (3) | O5—C15—H15A | 108.9 |
| O2—C1—C2 | 114.7 (3) | C16—C15—H15A | 108.9 |
| C3—C2—C7 | 119.7 (3) | O5—C15—H15B | 108.9 |
| C3—C2—C1 | 121.1 (3) | C16—C15—H15B | 108.9 |

| | | | |
|---|---|---|---|
| C7—C2—C1 | 119.2 (3) | H15A—C15—H15B | 107.7 |
| C2—C3—C4 | 118.4 (3) | N1—C16—C15 | 111.7 (3) |
| C2—C3—S1 | 120.6 (3) | N1—C16—H16A | 109.3 |
| C4—C3—S1 | 121.1 (3) | C15—C16—H16A | 109.3 |
| C5—C4—C3 | 121.1 (3) | N1—C16—H16B | 109.3 |
| C5—C4—H4 | 119.4 | C15—C16—H16B | 109.3 |
| C3—C4—H4 | 119.4 | H16A—C16—H16B | 108.0 |
| C4—C5—C6 | 120.0 (4) | N1—C17—C18 | 113.5 (3) |
| C4—C5—H5 | 120.0 | N1—C17—H17A | 108.9 |
| C6—C5—H5 | 120.0 | C18—C17—H17A | 108.9 |
| C7—C6—C5 | 119.6 (4) | N1—C17—H17B | 108.9 |
| C7—C6—H6 | 120.2 | C18—C17—H17B | 108.9 |
| C5—C6—H6 | 120.2 | H17A—C17—H17B | 107.7 |
| C6—C7—C2 | 121.2 (4) | C17—C18—C19 | 111.6 (4) |
| C6—C7—H7 | 119.4 | C17—C18—H18A | 109.3 |
| C2—C7—H7 | 119.4 | C19—C18—H18A | 109.3 |
| O4—C8—O3 | 124.1 (3) | C17—C18—H18B | 109.3 |
| O4—C8—C9 | 120.2 (3) | C19—C18—H18B | 109.3 |
| O3—C8—C9 | 115.7 (3) | H18A—C18—H18B | 108.0 |
| C10—C9—C14 | 119.0 (3) | C18—C19—H19A | 109.5 |
| C10—C9—C8 | 122.6 (3) | C18—C19—H19B | 109.5 |
| C14—C9—C8 | 118.4 (3) | H19A—C19—H19B | 109.5 |
| C11—C10—C9 | 119.3 (3) | C18—C19—H19C | 109.5 |
| C11—C10—S2 | 120.7 (3) | H19A—C19—H19C | 109.5 |
| C9—C10—S2 | 120.0 (3) | H19B—C19—H19C | 109.5 |
| C12—C11—C10 | 120.4 (3) | | |

| | | | |
|---|---|---|---|
| C3—S1—S2—C10 | −87.44 (16) | O4—C8—C9—C14 | −20.8 (5) |
| O1—C1—C2—C3 | −2.9 (5) | O3—C8—C9—C14 | 158.1 (3) |
| O2—C1—C2—C3 | 178.7 (3) | C14—C9—C10—C11 | 0.4 (5) |
| O1—C1—C2—C7 | 178.1 (3) | C8—C9—C10—C11 | 179.5 (3) |
| O2—C1—C2—C7 | −0.2 (5) | C14—C9—C10—S2 | 179.6 (3) |
| C7—C2—C3—C4 | −1.0 (5) | C8—C9—C10—S2 | −1.3 (4) |
| C1—C2—C3—C4 | −180.0 (3) | S1—S2—C10—C11 | 15.5 (3) |
| C7—C2—C3—S1 | 179.9 (3) | S1—S2—C10—C9 | −163.7 (2) |
| C1—C2—C3—S1 | 0.9 (5) | C9—C10—C11—C12 | 0.1 (5) |
| S2—S1—C3—C2 | −166.8 (3) | S2—C10—C11—C12 | −179.1 (3) |
| S2—S1—C3—C4 | 14.1 (3) | C10—C11—C12—C13 | −0.8 (5) |
| C2—C3—C4—C5 | 0.9 (6) | C11—C12—C13—C14 | 0.9 (5) |
| S1—C3—C4—C5 | −180.0 (3) | C12—C13—C14—C9 | −0.4 (5) |
| C3—C4—C5—C6 | −0.4 (6) | C10—C9—C14—C13 | −0.2 (5) |
| C4—C5—C6—C7 | −0.1 (7) | C8—C9—C14—C13 | −179.3 (3) |
| C5—C6—C7—C2 | 0.0 (6) | C17—N1—C16—C15 | 175.9 (3) |
| C3—C2—C7—C6 | 0.5 (6) | O5—C15—C16—N1 | 53.8 (5) |
| C1—C2—C7—C6 | 179.5 (4) | C16—N1—C17—C18 | 175.8 (4) |
| O4—C8—C9—C10 | 160.1 (3) | N1—C17—C18—C19 | −179.8 (4) |
| O3—C8—C9—C10 | −21.0 (5) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2*o*···O3$^{i}$ | 0.84 | 1.70 | 2.535 (4) | 178 |
| N1—H1n···O4$^{ii}$ | 0.90 | 2.10 | 2.887 (4) | 146 |
| N1—H2n···O1w$^{iii}$ | 0.90 | 1.92 | 2.773 (5) | 158 |
| O5—H5o···O1$^{iv}$ | 0.84 | 1.94 | 2.751 (5) | 162 |
| O1w—H1w···O4$^{v}$ | 0.84 | 1.99 | 2.823 (5) | 174 |
| O1w—H2w···O3 | 0.84 | 2.26 | 3.036 (5) | 154 |

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