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# Crystal structure of 2-cyano-3,3-bis(ethylsulfanyl)-*N*-*o*-tolylacrylamide

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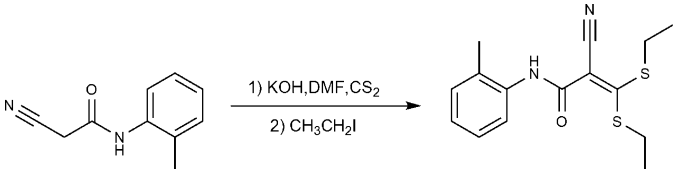
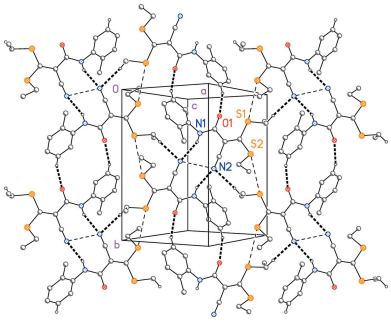
In the molecule of the title compound, $C_{15}H_{18}N_2OS_2$, the central $S_2C{=}C(CN)C$ moiety is planar (r.m.s. deviation = 0.029 Å). The C=O and C—CN groups are *trans* to each other across their common C—C bond. In the crystal, one classical and two 'weak' hydrogen bonds combine with borderline N···N and S···S contacts to form layers parallel to $(10\bar{2})$. One ethyl group is disordered over two positions with relative occupancy 0.721/0.279 (7).

## 1. Chemical context

The synthesis of ketene *S*,*S*-acetals as potential starting materials for the preparation of novel classes of heterocycles has attracted much attention (Elgemeie *et al.* 2009, 2015). As part of a research program for preparing new classes of antimetabolites (Elgemeie *et al.* 2016, 2017*a*), we have recently reported successful approaches for syntheses of pyridine, pyrimidine and mercaptopurine analogues by the reaction of cyanoketene dithioacetals with active methylene compounds (Elgemeie *et al.*, 2003, 2006, 2017*b*). In a continuation of this research, we report here a novel cyanoketene dithioacetal (1). Product (1) was prepared by the reaction of 2-cyano-*N*-(*o*-tolyl)acetamide with carbon disulfide in the presence of sodium ethoxide followed by alkylation with ethyl iodide. The structure of (1) was originally based on its spectroscopic data and elemental analysis (see *Experimental*). In order to establish the structure of the compound unambiguously, the crystal structure was determined.



## 2. Structural commentary

The X-ray analysis confirms the exclusive presence of the form (1) in the solid state (Fig. 1). Molecular dimensions may be regarded as normal [*e.g.* C9—C10 1.3781 (16) and C9—C11 1.4290 (16) Å]. The molecular backbone C1, N1, C8, C9, C10, S1, S2 is planar to within an r.m.s. deviation of 0.029 Å; O1 deviates by 0.063 (1) and C11 by 0.284 (1) Å from this plane. The aromatic ring subtends an angle of 53.30 (3)° with the same plane. The C=O and C—CN groups are *trans* to each

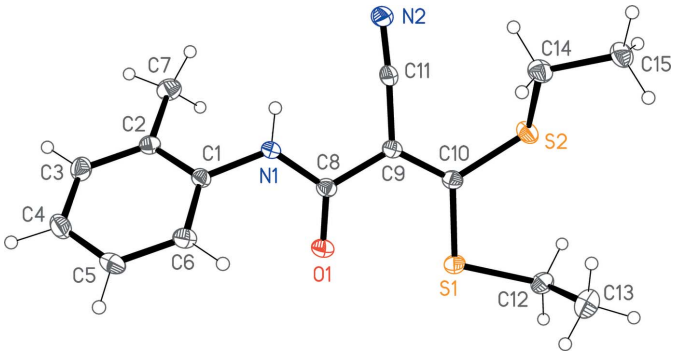



**Figure 1**
The structure of compound (1) in the crystal, with ellipsoids at the 50% probability level. Only one position of the disordered ethyl group C14/C15 is shown.

other across the C8—C9 bond, with a torsion angle of 167.61 (11)°.

## 3. Supramolecular features

Hydrogen bonds are given in Table 1, where the operators are also defined. The classical hydrogen bond N1—H01···N2$^{i}$ connects the molecules across inversion centres; associated with this interaction, the N2 atoms of both molecules are forced into a close contact of 3.061 (2) Å. Two further contacts (C—H···N and C—H···O; Table 1) may reasonably be regarded as 'weak' hydrogen bonds on the basis of distance and approximately linear angles at the relevant hydrogen atoms. Finally, a borderline contact S1···S2$^{ii}$ of 3.7488 (4) Å is observed. All these secondary interactions combine to form a layer of molecules parallel to (10$\bar{2}$) (Fig. 2).

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H01···N2$^{i}$ | 0.817 (17) | 2.375 (17) | 3.1346 (15) | 155.0 (15) |
| C12—H12$A$···N2$^{ii}$ | 0.99 | 2.51 | 3.4628 (16) | 160 |
| C5—H5···O1$^{iii}$ | 0.95 | 2.50 | 3.4110 (15) | 161 |

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

## 4. Database survey

A search of the Cambridge Database (Version 1.19; Groom & Allen, 2014; Groom *et al.*, 2016) for the fragment (C—

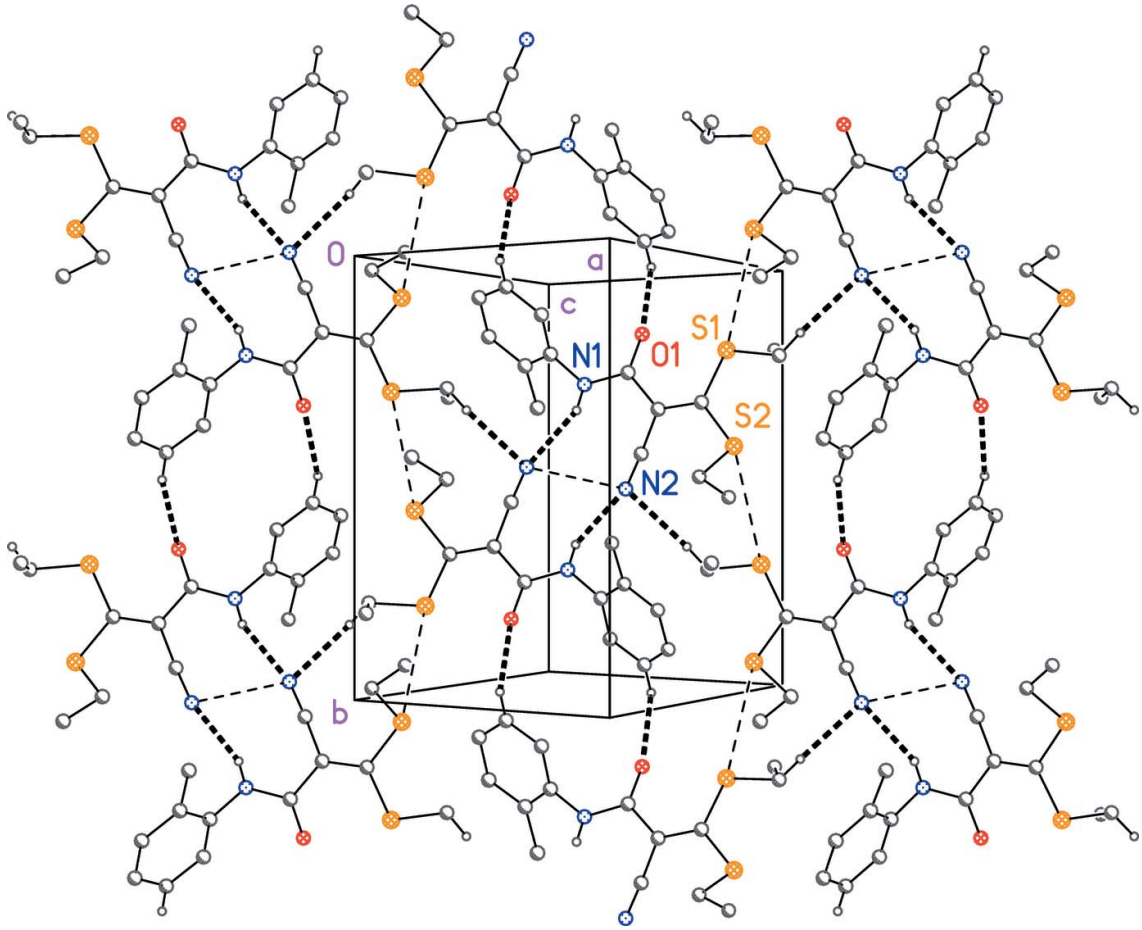



**Figure 2**
Packing diagram of compound (1) viewed perpendicular to (10$\bar{2}$). Hydrogen bonds are drawn as thick dashed bonds, with other contacts (see text) as thin dashed bonds. H atoms not involved in hydrogen bonds have been omitted for clarity.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{15}H_{18}N_2OS_2$ |
| $M_r$ | 306.43 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 100 |
| $a, b, c$ (Å) | 9.4104 (4), 12.8350 (4), 13.0774 (5) |
| $\beta$ (°) | 104.198 (4) |
| $V$ (Å$^3$) | 1531.28 (10) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.35 |
| Crystal size (mm) | 0.35 × 0.35 × 0.30 |
| Data collection | |
| Diffractometer | Oxford Diffraction Xcalibur Eos |
| Absorption correction | Multi-scan (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015) |
| $T_{min}$, $T_{max}$ | 0.986, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 42164, 4682, 3985 |
| $R_{int}$ | 0.043 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.728 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.034, 0.081, 1.03 |
| No. of reflections | 4682 |
| No. of parameters | 197 |
| No. of restraints | 15 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.37, −0.22 |

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *XP* (Siemens, 1994).

S)$_2$C═C(CN)C═O gave six hits (MTBCEY, NUCFEW, SESHUT10, SESHUT11, ZAMQUZ, ZEDJEX). In all cases the C═O and C—CN groups are mutually *trans*, as in the title compound.

## 5. Synthesis and crystallization

2-Cyano-*N*-(*o*-tolyl)acetamide (1 mmol) was added to a stirred solution of potassium hydroxide (2 mmol) in DMF (10 ml). After stirring for 30 min at room temperature, carbon disulfide (1.5 mmol) was added. The solution was left for 12 h at room temperature and then ethyl iodide (2 mmol) was added dropwise. Stirring was continued for a further 6 h. The reaction mixture was poured onto ice–water and the solid product was filtered off, dried and crystallized from ethanol to give yellow crystals, m.p. 93°C (366 K), yield 40%.

IR (KBr), 3430 (NH), 2220 (CN), 1670 (C═O) cm$^{-1}$; $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 1.25 (*t*, $J$ = 8 Hz, 3H, $CH_2CH_3$), 1.31 (*t*, $J$ = 8 Hz, 3H, $CH_2CH_3$), 2.51 (*s*, 3H, $CH_3$), 3.03 (*q*, $J$ = 6.8 Hz, 2H, $CH_2CH_3$), 3.12 (*q*, $J$ = 6.8 Hz, 2H, $CH_2CH_3$), 7.15–7.36 (*m*, 4H, $C_6H_4$), 10.05 (*s*, 1H, NH), Analysis calculated for $C_{15}H_{18}ON_2S_2$ (306.43): C, 58.82; H, 5.88, N, 9.15, S, 20.91%; Found: C, 58.70; H, 5.65, N, 9.00, S, 20.77%.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The ethyl group C14/15 is disordered over two positions with relative occupancy 0.721 (7)/0.279 (7). Appropriate restraints were employed to improve refinement stability, but the dimensions of disordered groups should be interpreted with caution.

The NH hydrogen was refined freely. Methyl H atoms were refined as idealized rigid groups (C—H 0.98 Å, H—C—H 109.5°) allowed to rotate but not tip (exception: minor disorder component at C15′, set ideally staggered with AFIX 33). Other hydrogen atoms were included using a riding model starting from calculated positions, with $C_{arom}$—H 0.95, $C_{methylene}$—H 0.99 Å, with $U_{iso}$(H) = 1.5$U_{eq}$(C-methyl) and 1.2$U_{eq}$(C) for other H atoms.

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# supporting information

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## Crystal structure of 2-cyano-3,3-bis(ethylsulfanyl)-*N*-*o*-tolylacrylamide

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### Computing details

Data collection: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); cell refinement: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); data reduction: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

### 2-Cyano-3,3-bis(ethylsulfanyl)-*N*-(2-methylphenyl)prop-2-enamide

*Crystal data*

$C_{15}H_{18}N_2OS_2$
$M_r = 306.43$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.4104$ (4) Å
$b = 12.8350$ (4) Å
$c = 13.0774$ (5) Å
$\beta = 104.198$ (4)°
$V = 1531.28$ (10) Å$^3$
$Z = 4$
$F(000) = 648$
$D_x = 1.329$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10087 reflections
$\theta = 2.7$–$30.4°$
$\mu = 0.35$ mm$^{-1}$
$T = 100$ K
Block, pale yellow
0.35 × 0.35 × 0.30 mm

*Data collection*

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: fine-focus sealed X-ray tube
Graphite monochromator
Detector resolution: 16.1419 pixels mm$^{-1}$
$\omega$ scan
Absorption correction: multi-scan (CrysAlis PRO; Rigaku Oxford Diffraction, 2015)
$T_{min} = 0.986$, $T_{max} = 1.000$
42164 measured reflections
4682 independent reflections
3985 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 31.2°$, $\theta_{min} = 2.2°$
$h = -13 \rightarrow 13$
$k = -18 \rightarrow 18$
$l = -18 \rightarrow 18$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.081$
$S = 1.03$
4682 reflections
197 parameters
15 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
$w = 1/[\sigma^2(F_o^2) + (0.0309P)^2 + 0.7259P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.37$ e Å$^{-3}$
$\Delta\rho_{min} = -0.22$ 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.
Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)
7.3265 (0.0012) x + 3.0782 (0.0029) y - 9.8498 (0.0015) z = 0.3852 (0.0018)
* -0.0120 (0.0007) C1 * 0.0262 (0.0010) C8 * 0.0574 (0.0010) C9 * -0.0136 (0.0009) C10 * -0.0055 (0.0004) S1 * -0.0220 (0.0005) S2 * -0.0304 (0.0009) N1 0.0632 (0.0010) O1 0.2839 (0.0014) C11
Rms deviation of fitted atoms = 0.0287
8.1736 (0.0024) x - 6.3013 (0.0055) y - 3.6411 (0.0064) z = 1.1855 (0.0026)
Angle to previous plane (with approximate esd) = 53.30 ( 0.03 )
* -0.0174 (0.0008) C1 * 0.0106 (0.0008) C2 * 0.0039 (0.0009) C3 * -0.0118 (0.0009) C4 * 0.0052 (0.0009) C5 * 0.0095 (0.0008) C6
Rms deviation of fitted atoms = 0.0107
**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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.48134 (12) | 0.21516 (9) | 0.38738 (9) | 0.0144 (2) | |
| C2 | 0.46128 (12) | 0.24547 (9) | 0.28216 (9) | 0.0156 (2) | |
| C3 | 0.37643 (14) | 0.18128 (10) | 0.20464 (9) | 0.0206 (2) | |
| H3 | 0.3601 | 0.2008 | 0.1326 | 0.025* | |
| C4 | 0.31546 (14) | 0.08976 (10) | 0.23047 (10) | 0.0222 (2) | |
| H4 | 0.2564 | 0.0480 | 0.1765 | 0.027* | |
| C5 | 0.34046 (14) | 0.05902 (9) | 0.33513 (10) | 0.0200 (2) | |
| H5 | 0.3005 | −0.0045 | 0.3529 | 0.024* | |
| C6 | 0.42423 (13) | 0.12172 (9) | 0.41347 (9) | 0.0173 (2) | |
| H6 | 0.4427 | 0.1008 | 0.4852 | 0.021* | |
| C7 | 0.53257 (14) | 0.34242 (10) | 0.25430 (10) | 0.0214 (2) | |
| H7A | 0.4999 | 0.4026 | 0.2886 | 0.032* | |
| H7B | 0.5051 | 0.3522 | 0.1776 | 0.032* | |
| H7C | 0.6393 | 0.3357 | 0.2784 | 0.032* | |
| C8 | 0.67932 (12) | 0.25551 (9) | 0.54338 (9) | 0.0148 (2) | |
| C9 | 0.74786 (13) | 0.33969 (9) | 0.61750 (9) | 0.0157 (2) | |
| C10 | 0.86223 (13) | 0.32278 (9) | 0.70449 (9) | 0.0156 (2) | |
| C11 | 0.69858 (13) | 0.44369 (9) | 0.59035 (9) | 0.0176 (2) | |
| S1 | 0.94594 (3) | 0.20139 (2) | 0.72800 (2) | 0.01904 (8) | |
| C12 | 1.07266 (14) | 0.21116 (10) | 0.85705 (10) | 0.0222 (2) | |
| H12A | 1.1520 | 0.1594 | 0.8617 | 0.027* | |
| H12B | 1.1179 | 0.2813 | 0.8647 | 0.027* | |
| C13 | 1.00032 (18) | 0.19311 (12) | 0.94728 (11) | 0.0324 (3) | |
| H13A | 0.9294 | 0.2489 | 0.9481 | 0.049* | |
| H13B | 1.0752 | 0.1930 | 1.0142 | 0.049* | |
| H13C | 0.9496 | 0.1258 | 0.9378 | 0.049* | |
| S2 | 0.93498 (3) | 0.42489 (2) | 0.79137 (2) | 0.02058 (8) | |

| | | | | | |
|---|---|---|---|---|---|
| C14 | 0.77925 (19) | 0.4973 (2) | 0.81670 (17) | 0.0211 (6) | 0.721 (7) |
| H14A | 0.7472 | 0.5518 | 0.7624 | 0.025* | 0.721 (7) |
| H14B | 0.6959 | 0.4496 | 0.8145 | 0.025* | 0.721 (7) |
| C15 | 0.8290 (3) | 0.5466 (2) | 0.9252 (2) | 0.0234 (5) | 0.721 (7) |
| H15A | 0.8431 | 0.4922 | 0.9793 | 0.035* | 0.721 (7) |
| H15B | 0.7544 | 0.5959 | 0.9358 | 0.035* | 0.721 (7) |
| H15C | 0.9217 | 0.5836 | 0.9306 | 0.035* | 0.721 (7) |
| C14′ | 0.7812 (5) | 0.4548 (6) | 0.8430 (5) | 0.0206 (13)* | 0.279 (7) |
| H14C | 0.6907 | 0.4573 | 0.7853 | 0.025* | 0.279 (7) |
| H14D | 0.7689 | 0.4007 | 0.8941 | 0.025* | 0.279 (7) |
| C15′ | 0.8087 (9) | 0.5593 (6) | 0.8967 (7) | 0.029 (2)* | 0.279 (7) |
| H15D | 0.7263 | 0.5771 | 0.9269 | 0.044* | 0.279 (7) |
| H15E | 0.8186 | 0.6125 | 0.8451 | 0.044* | 0.279 (7) |
| H15F | 0.8991 | 0.5563 | 0.9530 | 0.044* | 0.279 (7) |
| N1 | 0.55847 (11) | 0.28375 (8) | 0.46806 (8) | 0.0169 (2) | |
| H01 | 0.5279 (18) | 0.3435 (14) | 0.4655 (12) | 0.026 (4)* | |
| N2 | 0.65583 (12) | 0.52547 (8) | 0.56292 (9) | 0.0244 (2) | |
| O1 | 0.73077 (10) | 0.16749 (7) | 0.55038 (7) | 0.01999 (18) | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0152 (5) | 0.0127 (5) | 0.0153 (5) | 0.0007 (4) | 0.0036 (4) | −0.0020 (4) |
| C2 | 0.0157 (5) | 0.0146 (5) | 0.0170 (5) | 0.0021 (4) | 0.0048 (4) | 0.0008 (4) |
| C3 | 0.0233 (6) | 0.0228 (6) | 0.0149 (5) | 0.0019 (5) | 0.0032 (4) | −0.0014 (4) |
| C4 | 0.0219 (6) | 0.0191 (6) | 0.0236 (6) | −0.0007 (5) | 0.0018 (5) | −0.0073 (5) |
| C5 | 0.0205 (6) | 0.0130 (5) | 0.0272 (6) | −0.0016 (4) | 0.0074 (5) | −0.0019 (4) |
| C6 | 0.0208 (6) | 0.0144 (5) | 0.0179 (5) | 0.0010 (4) | 0.0071 (4) | 0.0005 (4) |
| C7 | 0.0230 (6) | 0.0202 (6) | 0.0221 (6) | −0.0010 (5) | 0.0075 (5) | 0.0048 (5) |
| C8 | 0.0166 (5) | 0.0134 (5) | 0.0153 (5) | −0.0007 (4) | 0.0055 (4) | 0.0004 (4) |
| C9 | 0.0170 (5) | 0.0121 (5) | 0.0172 (5) | 0.0008 (4) | 0.0025 (4) | −0.0005 (4) |
| C10 | 0.0150 (5) | 0.0141 (5) | 0.0179 (5) | 0.0010 (4) | 0.0046 (4) | −0.0003 (4) |
| C11 | 0.0173 (5) | 0.0155 (5) | 0.0175 (5) | −0.0013 (4) | −0.0007 (4) | −0.0032 (4) |
| S1 | 0.02006 (15) | 0.01621 (14) | 0.01943 (14) | 0.00640 (11) | 0.00213 (11) | −0.00021 (10) |
| C12 | 0.0184 (6) | 0.0217 (6) | 0.0232 (6) | 0.0053 (5) | −0.0010 (4) | 0.0018 (5) |
| C13 | 0.0391 (8) | 0.0344 (8) | 0.0218 (6) | 0.0005 (6) | 0.0036 (6) | 0.0036 (6) |
| S2 | 0.01549 (14) | 0.01860 (15) | 0.02518 (15) | 0.00178 (10) | 0.00028 (11) | −0.00709 (11) |
| C14 | 0.0192 (9) | 0.0211 (12) | 0.0238 (9) | 0.0043 (7) | 0.0069 (6) | −0.0028 (8) |
| C15 | 0.0326 (12) | 0.0214 (10) | 0.0185 (11) | 0.0020 (8) | 0.0105 (10) | −0.0036 (9) |
| N1 | 0.0210 (5) | 0.0107 (4) | 0.0167 (4) | 0.0021 (4) | 0.0001 (4) | −0.0014 (4) |
| N2 | 0.0239 (6) | 0.0151 (5) | 0.0278 (5) | −0.0002 (4) | −0.0061 (4) | −0.0021 (4) |
| O1 | 0.0224 (4) | 0.0132 (4) | 0.0227 (4) | 0.0037 (3) | 0.0025 (3) | −0.0014 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.3905 (16) | C3—H3 | 0.9500 |
| C1—C2 | 1.3975 (15) | C4—H4 | 0.9500 |
| C1—N1 | 1.4282 (14) | C5—H5 | 0.9500 |

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| C2—C3 | 1.3953 (16) | C6—H6 | 0.9500 |
| C2—C7 | 1.5004 (16) | C7—H7A | 0.9800 |
| C3—C4 | 1.3850 (18) | C7—H7B | 0.9800 |
| C4—C5 | 1.3879 (18) | C7—H7C | 0.9800 |
| C5—C6 | 1.3865 (17) | C12—H12A | 0.9900 |
| C8—O1 | 1.2236 (14) | C12—H12B | 0.9900 |
| C8—N1 | 1.3585 (15) | C13—H13A | 0.9800 |
| C8—C9 | 1.4888 (16) | C13—H13B | 0.9800 |
| C9—C10 | 1.3781 (16) | C13—H13C | 0.9800 |
| C9—C11 | 1.4290 (16) | C14—H14A | 0.9900 |
| C10—S1 | 1.7390 (12) | C14—H14B | 0.9900 |
| C10—S2 | 1.7594 (12) | C15—H15A | 0.9800 |
| C11—N2 | 1.1496 (16) | C15—H15B | 0.9800 |
| S1—C12 | 1.8162 (13) | C15—H15C | 0.9800 |
| C12—C13 | 1.518 (2) | C14′—H14C | 0.9900 |
| S2—C14′ | 1.783 (5) | C14′—H14D | 0.9900 |
| S2—C14 | 1.8326 (18) | C15′—H15D | 0.9800 |
| C14—C15 | 1.519 (3) | C15′—H15E | 0.9800 |
| C14′—C15′ | 1.507 (9) | C15′—H15F | 0.9800 |
| N1—H01 | 0.817 (17) | | |

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| C6—C1—C2 | 120.98 (10) | C2—C7—H7A | 109.5 |
| C6—C1—N1 | 120.49 (10) | C2—C7—H7B | 109.5 |
| C2—C1—N1 | 118.50 (10) | H7A—C7—H7B | 109.5 |
| C3—C2—C1 | 117.77 (11) | C2—C7—H7C | 109.5 |
| C3—C2—C7 | 121.57 (11) | H7A—C7—H7C | 109.5 |
| C1—C2—C7 | 120.63 (10) | H7B—C7—H7C | 109.5 |
| C4—C3—C2 | 121.40 (11) | C13—C12—H12A | 108.9 |
| C3—C4—C5 | 120.10 (11) | S1—C12—H12A | 108.9 |
| C6—C5—C4 | 119.46 (11) | C13—C12—H12B | 108.9 |
| C5—C6—C1 | 120.21 (11) | S1—C12—H12B | 108.9 |
| O1—C8—N1 | 123.11 (11) | H12A—C12—H12B | 107.7 |
| O1—C8—C9 | 121.40 (10) | C12—C13—H13A | 109.5 |
| N1—C8—C9 | 115.49 (10) | C12—C13—H13B | 109.5 |
| C10—C9—C11 | 119.50 (10) | H13A—C13—H13B | 109.5 |
| C10—C9—C8 | 123.30 (10) | C12—C13—H13C | 109.5 |
| C11—C9—C8 | 116.99 (10) | H13A—C13—H13C | 109.5 |
| C9—C10—S1 | 121.02 (9) | H13B—C13—H13C | 109.5 |
| C9—C10—S2 | 121.14 (9) | C15—C14—H14A | 110.2 |
| S1—C10—S2 | 117.75 (7) | S2—C14—H14A | 110.2 |
| N2—C11—C9 | 176.23 (12) | C15—C14—H14B | 110.2 |
| C10—S1—C12 | 105.56 (6) | S2—C14—H14B | 110.2 |
| C13—C12—S1 | 113.23 (10) | H14A—C14—H14B | 108.5 |
| C10—S2—C14′ | 100.46 (17) | C15′—C14′—H14C | 110.1 |
| C10—S2—C14 | 107.01 (7) | S2—C14′—H14C | 110.1 |
| C15—C14—S2 | 107.65 (16) | C15′—C14′—H14D | 110.1 |
| C15′—C14′—S2 | 107.8 (5) | S2—C14′—H14D | 110.1 |
| C8—N1—C1 | 123.68 (10) | H14C—C14′—H14D | 108.5 |

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| C4—C3—H3 | 119.3 | C14′—C15′—H15D | 109.5 |
| C2—C3—H3 | 119.3 | C14′—C15′—H15E | 109.5 |
| C3—C4—H4 | 120.0 | H15D—C15′—H15E | 109.5 |
| C5—C4—H4 | 120.0 | C14′—C15′—H15F | 109.5 |
| C6—C5—H5 | 120.3 | H15D—C15′—H15F | 109.5 |
| C4—C5—H5 | 120.3 | H15E—C15′—H15F | 109.5 |
| C5—C6—H6 | 119.9 | C8—N1—H01 | 120.2 (11) |
| C1—C6—H6 | 119.9 | C1—N1—H01 | 116.0 (11) |
| | | | |
| C6—C1—C2—C3 | 2.91 (17) | C11—C9—C10—S2 | 7.34 (16) |
| N1—C1—C2—C3 | −175.34 (11) | C8—C9—C10—S2 | −178.08 (9) |
| C6—C1—C2—C7 | −175.44 (11) | C9—C10—S1—C12 | −173.24 (10) |
| N1—C1—C2—C7 | 6.31 (16) | S2—C10—S1—C12 | 10.20 (9) |
| C1—C2—C3—C4 | −0.85 (18) | C10—S1—C12—C13 | 83.40 (11) |
| C7—C2—C3—C4 | 177.48 (12) | C9—C10—S2—C14′ | 64.9 (3) |
| C2—C3—C4—C5 | −1.27 (19) | S1—C10—S2—C14′ | −118.6 (3) |
| C3—C4—C5—C6 | 1.35 (19) | C9—C10—S2—C14 | 44.96 (14) |
| C4—C5—C6—C1 | 0.68 (18) | S1—C10—S2—C14 | −138.48 (11) |
| C2—C1—C6—C5 | −2.86 (18) | C10—S2—C14—C15 | 152.32 (19) |
| N1—C1—C6—C5 | 175.35 (11) | C14′—S2—C14—C15 | 78.5 (5) |
| O1—C8—C9—C10 | −7.09 (18) | C10—S2—C14′—C15′ | −164.9 (5) |
| N1—C8—C9—C10 | 173.60 (11) | C14—S2—C14′—C15′ | −53.9 (6) |
| O1—C8—C9—C11 | 167.61 (11) | O1—C8—N1—C1 | −1.97 (18) |
| N1—C8—C9—C11 | −11.70 (15) | C9—C8—N1—C1 | 177.32 (10) |
| C11—C9—C10—S1 | −169.10 (9) | C6—C1—N1—C8 | 57.60 (16) |
| C8—C9—C10—S1 | 5.47 (16) | C2—C1—N1—C8 | −124.15 (12) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H01···N2$^{i}$ | 0.817 (17) | 2.375 (17) | 3.1346 (15) | 155.0 (15) |
| C12—H12*A*···N2$^{ii}$ | 0.99 | 2.51 | 3.4628 (16) | 160 |
| C5—H5···O1$^{iii}$ | 0.95 | 2.50 | 3.4110 (15) | 161 |

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