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(2E)-1-(4-Aminophenyl)-3-(2-thienyl)-prop-2-en-1-one ethanol hemisolvateHoong-Kun Fun,^{a*} ‡ Thawanrat Kobkeatthawin^b and Suchada Chantrapromma^{c§}

^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bDepartment of Chemistry and Center of Excellence for Innovation in Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and ^cCrystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

Correspondence e-mail: hkfun@usm.my

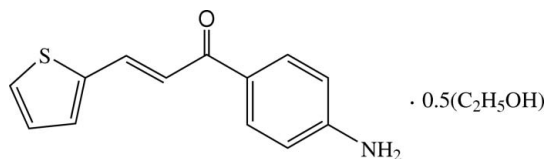
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in main residue; R factor = 0.061; wR factor = 0.205; data-to-parameter ratio = 22.5.

In the title compound, $\text{C}_{13}\text{H}_{11}\text{NOS}\cdot 0.5\text{C}_2\text{H}_6\text{O}$, the chalcone derivative is close to planar, the dihedral angle between the thiophene and 4-aminophenyl rings being $3.1(2)^\circ$. The thiophene ring is disordered over two orientations with occupancies of 0.842 (3) and 0.158 (3). In the crystal structure, molecules are linked into chains along the b axis by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The chains are crosslinked *via* $\text{N}-\text{H}\cdots\pi$ interactions involving the thiophene ring. The ethanol solvent molecule is also disordered over two positions, each with an occupancy of 0.25.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related structures, see: Fun *et al.* (2009); Suwunwong *et al.* (2009). For background and applications of chalcones, see: Dimmock *et al.* (1999); Go *et al.* (2005); Ni *et al.* (2004); Patil & Dharmaprakash (2008). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



‡ Thomson Reuters ResearcherID: A-3561-2009.

§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Experimental

Crystal data

$\text{C}_{13}\text{H}_{11}\text{NOS}\cdot 0.5\text{C}_2\text{H}_6\text{O}$
 $M_r = 252.32$
 Orthorhombic, $P2_12_12_1$
 $a = 5.1413(1)$ Å
 $b = 13.9754(2)$ Å
 $c = 18.2647(2)$ Å

$V = 1312.35(3)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.24$ mm⁻¹
 $T = 100$ K
 $0.56 \times 0.22 \times 0.17$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.879$, $T_{\max} = 0.961$

22258 measured reflections
 4225 independent reflections
 3893 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.205$
 $S = 1.09$
 4225 reflections
 188 parameters
 14 restraints

H-atom parameters constrained
 $\Delta\rho_{\max} = 1.04$ e Å⁻³
 $\Delta\rho_{\min} = -0.31$ e Å⁻³
 Absolute structure: Flack (1983), 1874 Friedel pairs
 Flack parameter: 0.00 (12)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1A}\cdots\text{O1}^{\text{i}}$ | 0.86 | 2.16 | 2.931 (3) | 149 |
| $\text{N1}-\text{H1B}\cdots\text{Cg1}^{\text{ii}}$ | 0.86 | 2.80 | 3.597 (3) | 156 |

Symmetry codes: (i) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{1}{2}, -y, z + \frac{1}{2}$. Cg1 is the centroid of the S1/C10-C13 ring.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2908).

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supporting information

Acta Cryst. (2009). E65, o2532–o2533 [doi:10.1107/S1600536809037933]

(2E)-1-(4-Aminophenyl)-3-(2-thienyl)prop-2-en-1-one ethanol hemisolvate**Hoong-Kun Fun, Thawanrat Kobkeatthawin and Suchada Chantrapromma****S1. Comment**

Chalcones have been reported to be responsible for a variety of biological activities such as analgesic, anti-inflammatory, antibacterial and antimycotic properties (Dimmock *et al.*, 1999; Go *et al.*, 2005; Ni *et al.*, 2004). Some of the synthetic chalcones have also been found to be non-linear optical (NLO) materials (Patil & Dharmaparakash, 2008). These interesting activities have led us to synthesize the title heteroaryl chalcone, (I), in order to study its NLO properties and biological activities. Herein we report the crystal structure of (I). The title compound crystallizes in orthorhombic noncentrosymmetric space group $P2_12_12_1$ and therefore it is expected to exhibit second-order non-linear optic properties.

The molecule of the title heteroaryl chalcone (Fig. 1) exists in an *E* configuration with respect to the C8=C9 double bond [1.346 (3) Å], with C7—C8—C9—C10 torsion angle of 179.1 (2)°. The molecule is essentially planar as indicated by the dihedral angle between thiophene (C10—C13/S1) and 4-aminophenyl rings of 3.1 (2)°. Bond distances (Allen *et al.*, 1987) and angles show normal values and are comparable with those observed in closely related structures (Fun *et al.*, 2009; Suwunwong *et al.*, 2009).

In the crystal, molecules are linked into chains along the *b* axis through N—H···O hydrogen bonds (Fig. 2 and Table 1). The chains are interlinked via N—H··· π interactions (Table 1) involving the C10-C13/S1 ring (Fig.2).

S2. Experimental

The title compound was synthesized by the condensation of 4-aminoacetophenone (0.40 g, 3 mmol) with thiophene-2-carboxaldehyde (0.28 ml, 3 mmol) in ethanol (30 ml) in the presence of 10% NaOH (aq) (5 ml). After stirring for 2 hr at room temperature, the resulting yellow solid was collected by filtration, washed with distilled water, dried and purified by repeated recrystallization from acetone. Yellow plate-shaped single crystals of the title compound suitable for *X*-ray structure determination were grown by slow evaporation of an ethanol solution at room temperature after several days, (m.p. 378–379 K).

S3. Refinement

The thiophene ring of the chalcone is disordered over two orientations with occupancies of 0.842 (3) and 0.158 (3). The same anisotropic displacement parameters were used for atoms pairs C12A/C11, C11A/C12 and C13A/C13. Atoms S1A, C11A, C12A, C13A and C10 were restrained to be coplanar. The ethanol solvent molecule is also disordered over two positions across a center of symmetry. Their occupancies were initially refined to 0.248 (5) and 0.242 (5) and later both were fixed at 0.25. Both disorder components were refined isotropically. The C—O, C—C and O···C distances were restrained to 1.42 (1), 1.51 (1) and 2.43 (1) Å, respectively. All H atoms were placed in calculated positions, with N-H = 0.86 Å, C-H = 0.93–0.97 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl and hydroxyl H atoms and $1.2U_{eq}(C)$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.96 Å from H2B and the deepest hole is located at 0.30 Å from H14B. The

final difference density features indicate that the solvent molecule may be disordered over multiple sites.

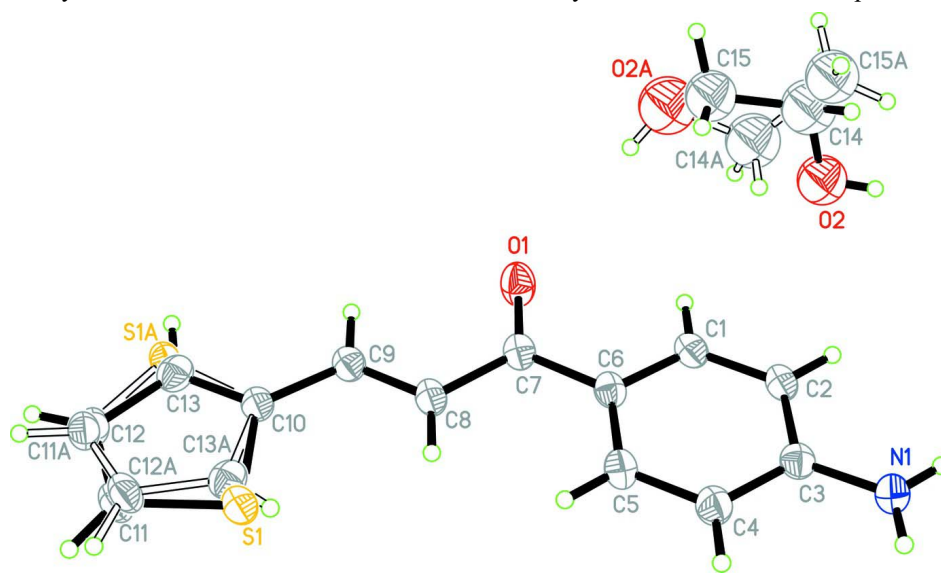
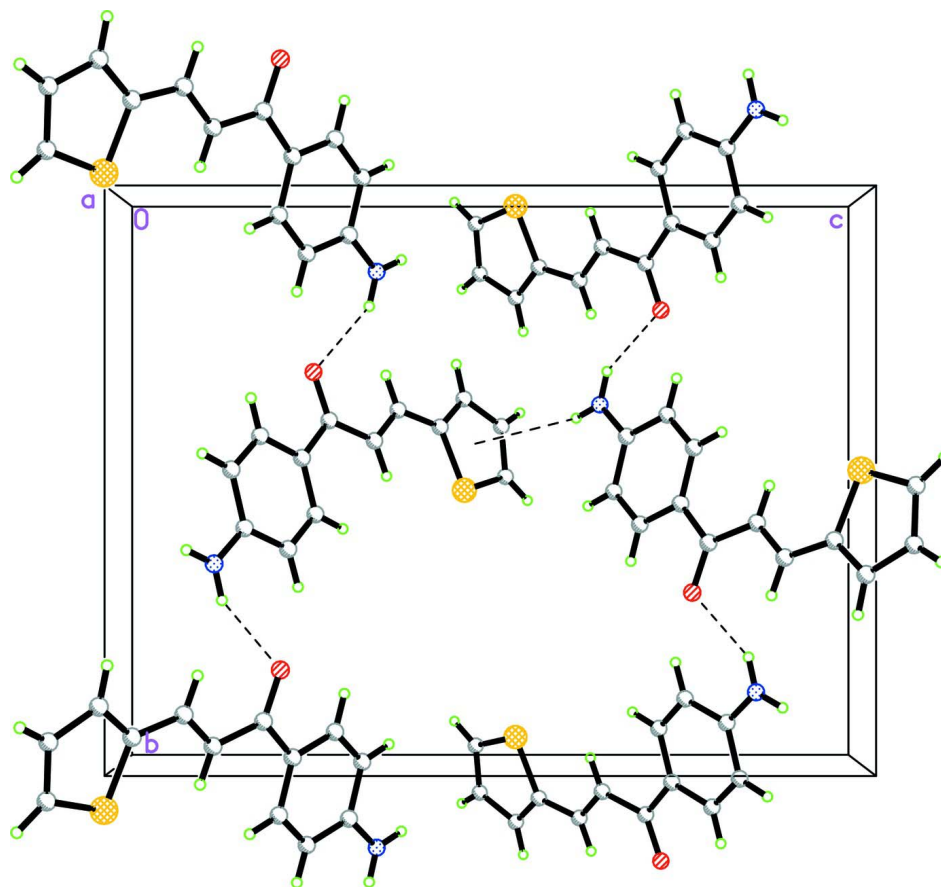


Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. The minor disorder components are shown in open bonds.

**Figure 2**

The crystal packing of the title compound, viewed along the *a* axis, showing chains running along the *b* axis. N—H···O hydrogen bonds and N—H··· π interactions are shown as dashed lines. Only the major disorder component is shown. For clarity, the disordered ethanol solvent molecules are not shown.

(2*E*)-1-(4-Aminophenyl)-3-(2-thienyl)prop-2-en-1-one ethanol hemisolvate

Crystal data

$C_{13}H_{11}NOS \cdot 0.5C_2H_6O$

$M_r = 252.32$

Orthorhombic, $P2_12_12_1$

Hall symbol: $P\ 2ac\ 2ab$

$a = 5.1413\ (1)\ \text{\AA}$

$b = 13.9754\ (2)\ \text{\AA}$

$c = 18.2647\ (2)\ \text{\AA}$

$V = 1312.35\ (3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 532$

$D_x = 1.277\ \text{Mg m}^{-3}$

Melting point = 378–379 K

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 4225 reflections

$\theta = 1.8\text{--}31.1^\circ$

$\mu = 0.24\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Plate, yellow

$0.56 \times 0.22 \times 0.17\ \text{mm}$

Data collection

Bruker APEXII CCD area-detector
diffractometer

Radiation source: sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2005)

$T_{\min} = 0.879$, $T_{\max} = 0.961$

22258 measured reflections

4225 independent reflections

3893 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\text{max}} = 31.1^\circ$, $\theta_{\text{min}} = 1.8^\circ$

$h = -7 \rightarrow 7$
 $k = -20 \rightarrow 15$
 $l = -26 \rightarrow 24$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.205$
 $S = 1.09$
 4225 reflections
 188 parameters
 14 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
 $w = 1/[\sigma^2(F_o^2) + (0.1552P)^2 + 0.2319P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.04 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.31 \text{ e } \text{\AA}^{-3}$
 Absolute structure: Flack (1983), 1874 Friedel
 pairs
 Absolute structure parameter: 0.00 (12)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|-------------|---------------|--------------|----------------------------------|-----------|
| O1 | 0.4331 (4) | -0.19026 (12) | 0.23694 (11) | 0.0354 (4) | |
| N1 | -0.3267 (4) | 0.13702 (15) | 0.34900 (11) | 0.0303 (4) | |
| H1A | -0.3311 | 0.1971 | 0.3390 | 0.036* | |
| H1B | -0.4325 | 0.1135 | 0.3807 | 0.036* | |
| C1 | 0.0494 (4) | -0.07627 (15) | 0.29945 (11) | 0.0260 (4) | |
| H1 | 0.0568 | -0.1408 | 0.3116 | 0.031* | |
| C2 | -0.1363 (4) | -0.01898 (16) | 0.33217 (11) | 0.0257 (4) | |
| H2 | -0.2512 | -0.0453 | 0.3659 | 0.031* | |
| C3 | -0.1526 (4) | 0.07911 (15) | 0.31464 (11) | 0.0249 (4) | |
| C4 | 0.0225 (5) | 0.11577 (16) | 0.26252 (12) | 0.0296 (4) | |
| H4 | 0.0135 | 0.1800 | 0.2495 | 0.035* | |
| C5 | 0.2078 (4) | 0.05764 (16) | 0.23027 (13) | 0.0284 (4) | |
| H5 | 0.3214 | 0.0835 | 0.1960 | 0.034* | |
| C6 | 0.2273 (4) | -0.03956 (15) | 0.24835 (10) | 0.0232 (4) | |
| C7 | 0.4185 (4) | -0.10551 (15) | 0.21554 (11) | 0.0244 (4) | |
| C8 | 0.5911 (4) | -0.07082 (15) | 0.15699 (12) | 0.0256 (4) | |
| H8 | 0.5733 | -0.0082 | 0.1405 | 0.031* | |
| C9 | 0.7742 (4) | -0.12715 (15) | 0.12666 (11) | 0.0251 (4) | |

| | | | | | |
|------|--------------|---------------|--------------|-------------|-----------|
| H9 | 0.7867 | -0.1897 | 0.1436 | 0.030* | |
| C10 | 0.9527 (4) | -0.09779 (15) | 0.06978 (11) | 0.0241 (4) | |
| S1 | 0.95430 (16) | 0.01724 (6) | 0.03611 (4) | 0.0312 (2) | 0.842 (3) |
| C11 | 1.2101 (13) | -0.0077 (4) | -0.0214 (2) | 0.0304 (8) | 0.842 (3) |
| H11 | 1.2848 | 0.0368 | -0.0529 | 0.037* | 0.842 (3) |
| C12 | 1.2907 (12) | -0.1014 (4) | -0.0157 (4) | 0.0293 (6) | 0.842 (3) |
| H12 | 1.4268 | -0.1277 | -0.0425 | 0.035* | 0.842 (3) |
| C13 | 1.1404 (12) | -0.1509 (4) | 0.0372 (4) | 0.0291 (11) | 0.842 (3) |
| H13 | 1.1694 | -0.2150 | 0.0481 | 0.035* | 0.842 (3) |
| S1A | 1.1592 (16) | -0.1790 (5) | 0.0320 (5) | 0.0257 (13) | 0.158 (3) |
| C11A | 1.312 (7) | -0.095 (2) | -0.020 (2) | 0.0293 (6) | 0.16 |
| H11A | 1.4432 | -0.1078 | -0.0536 | 0.035* | 0.158 (3) |
| C12A | 1.208 (7) | -0.006 (2) | -0.0088 (18) | 0.0304 (8) | 0.16 |
| H12A | 1.2513 | 0.0496 | -0.0341 | 0.037* | 0.158 (3) |
| C13A | 1.005 (4) | -0.0026 (14) | 0.0424 (12) | 0.0291 (11) | 0.16 |
| H13A | 0.9294 | 0.0517 | 0.0628 | 0.035* | 0.158 (3) |
| O2 | -0.2058 (19) | -0.2501 (8) | 0.4350 (6) | 0.060 (3)* | 0.25 |
| H2B | -0.3410 | -0.2368 | 0.4565 | 0.089* | 0.25 |
| C14 | 0.013 (2) | -0.2424 (15) | 0.4817 (8) | 0.070 (4)* | 0.25 |
| H14A | 0.0016 | -0.2951 | 0.5161 | 0.084* | 0.25 |
| H14B | 0.0012 | -0.1832 | 0.5093 | 0.084* | 0.25 |
| C15 | 0.272 (2) | -0.2486 (12) | 0.4437 (9) | 0.058 (3)* | 0.25 |
| H15A | 0.3970 | -0.2793 | 0.4748 | 0.087* | 0.25 |
| H15B | 0.3290 | -0.1848 | 0.4328 | 0.087* | 0.25 |
| H15C | 0.2548 | -0.2842 | 0.3990 | 0.087* | 0.25 |
| O2A | 0.333 (2) | -0.2650 (10) | 0.4138 (7) | 0.075 (3)* | 0.25 |
| H2AA | 0.3521 | -0.2536 | 0.3700 | 0.113* | 0.25 |
| C14A | 0.059 (3) | -0.2529 (15) | 0.4304 (7) | 0.070 (4)* | 0.25 |
| H14C | -0.0254 | -0.2859 | 0.3909 | 0.084* | 0.25 |
| H14D | 0.0526 | -0.1870 | 0.4158 | 0.084* | 0.25 |
| C15A | 0.012 (3) | -0.2491 (14) | 0.5119 (7) | 0.064 (4)* | 0.25 |
| H15G | -0.1703 | -0.2413 | 0.5218 | 0.096* | 0.25 |
| H15D | 0.0720 | -0.3078 | 0.5333 | 0.096* | 0.25 |
| H15E | 0.1065 | -0.1963 | 0.5324 | 0.096* | 0.25 |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|-------------|-------------|-------------|
| O1 | 0.0388 (9) | 0.0271 (7) | 0.0402 (9) | -0.0006 (7) | 0.0127 (8) | -0.0014 (6) |
| N1 | 0.0301 (9) | 0.0343 (9) | 0.0266 (8) | 0.0049 (7) | 0.0037 (7) | -0.0008 (7) |
| C1 | 0.0271 (9) | 0.0298 (9) | 0.0211 (8) | -0.0024 (8) | 0.0010 (7) | 0.0000 (7) |
| C2 | 0.0262 (8) | 0.0299 (9) | 0.0210 (8) | -0.0017 (8) | 0.0025 (7) | -0.0004 (7) |
| C3 | 0.0232 (8) | 0.0309 (9) | 0.0207 (8) | -0.0009 (7) | -0.0029 (7) | -0.0030 (7) |
| C4 | 0.0300 (10) | 0.0293 (9) | 0.0295 (10) | 0.0002 (8) | 0.0042 (8) | 0.0015 (8) |
| C5 | 0.0260 (9) | 0.0317 (10) | 0.0275 (9) | -0.0011 (8) | 0.0057 (8) | -0.0003 (8) |
| C6 | 0.0227 (8) | 0.0274 (9) | 0.0194 (8) | -0.0032 (7) | -0.0006 (6) | -0.0045 (6) |
| C7 | 0.0228 (9) | 0.0271 (8) | 0.0234 (9) | -0.0036 (7) | 0.0010 (7) | -0.0033 (7) |
| C8 | 0.0233 (9) | 0.0317 (9) | 0.0217 (8) | -0.0027 (7) | 0.0019 (7) | -0.0016 (7) |

| | | | | | | |
|------|-------------|-------------|-------------|--------------|--------------|--------------|
| C9 | 0.0221 (8) | 0.0294 (9) | 0.0237 (9) | -0.0041 (7) | -0.0008 (7) | -0.0007 (7) |
| C10 | 0.0209 (8) | 0.0298 (8) | 0.0216 (8) | -0.0026 (7) | -0.0017 (7) | -0.0006 (7) |
| S1 | 0.0333 (4) | 0.0323 (4) | 0.0279 (3) | 0.0023 (3) | 0.0062 (3) | 0.0037 (3) |
| C11 | 0.0314 (11) | 0.0398 (12) | 0.020 (2) | -0.0038 (9) | 0.0043 (14) | 0.0031 (14) |
| C12 | 0.0222 (15) | 0.0416 (14) | 0.0243 (13) | -0.0012 (10) | 0.0021 (10) | 0.0018 (10) |
| C13 | 0.0248 (16) | 0.033 (2) | 0.0298 (17) | 0.0007 (17) | -0.0037 (12) | -0.0028 (19) |
| S1A | 0.0227 (19) | 0.031 (3) | 0.023 (2) | -0.003 (2) | 0.0025 (15) | 0.003 (2) |
| C11A | 0.0222 (15) | 0.0416 (14) | 0.0243 (13) | -0.0012 (10) | 0.0021 (10) | 0.0018 (10) |
| C12A | 0.0314 (11) | 0.0398 (12) | 0.020 (2) | -0.0038 (9) | 0.0043 (14) | 0.0031 (14) |
| C13A | 0.0248 (16) | 0.033 (2) | 0.0298 (17) | 0.0007 (17) | -0.0037 (12) | -0.0028 (19) |

Geometric parameters (Å, °)

| | | | |
|------------|-------------|----------------|------------|
| O1—C7 | 1.250 (3) | C11—H11 | 0.93 |
| N1—C3 | 1.360 (3) | C12—C13 | 1.418 (8) |
| N1—H1A | 0.86 | C12—H12 | 0.93 |
| N1—H1B | 0.86 | C13—H13 | 0.93 |
| C1—C2 | 1.382 (3) | S1A—C11A | 1.70 (2) |
| C1—C6 | 1.404 (3) | C11A—C12A | 1.373 (17) |
| C1—H1 | 0.93 | C11A—H11A | 0.93 |
| C2—C3 | 1.410 (3) | C12A—C13A | 1.40 (2) |
| C2—H2 | 0.93 | C12A—H12A | 0.93 |
| C3—C4 | 1.407 (3) | C13A—H13A | 0.93 |
| C4—C5 | 1.384 (3) | O2—C14 | 1.416 (9) |
| C4—H4 | 0.93 | O2—H2B | 0.82 |
| C5—C6 | 1.402 (3) | C14—C15 | 1.504 (10) |
| C5—H5 | 0.93 | C14—H14A | 0.97 |
| C6—C7 | 1.475 (3) | C14—H14B | 0.97 |
| C7—C8 | 1.472 (3) | C15—H15A | 0.96 |
| C8—C9 | 1.346 (3) | C15—H15B | 0.96 |
| C8—H8 | 0.93 | C15—H15C | 0.96 |
| C9—C10 | 1.446 (3) | O2A—C14A | 1.449 (9) |
| C9—H9 | 0.93 | O2A—H2AA | 0.82 |
| C10—C13 | 1.355 (6) | C14A—C15A | 1.509 (9) |
| C10—C13A | 1.45 (2) | C14A—H14C | 0.96 |
| C10—S1A | 1.701 (8) | C14A—H14D | 0.96 |
| C10—S1 | 1.721 (2) | C15A—H15G | 0.96 |
| S1—C11 | 1.719 (5) | C15A—H15D | 0.96 |
| C11—C12 | 1.377 (5) | C15A—H15E | 0.96 |
| C3—N1—H1A | 120.0 | C11—C12—H12 | 124.2 |
| C3—N1—H1B | 120.0 | C13—C12—H12 | 125.2 |
| H1A—N1—H1B | 120.0 | C10—C13—C12 | 114.8 (5) |
| C2—C1—C6 | 121.7 (2) | C10—C13—H13 | 123.3 |
| C2—C1—H1 | 119.1 | C12—C13—H13 | 121.9 |
| C6—C1—H1 | 119.1 | C11A—S1A—C10 | 93.1 (12) |
| C1—C2—C3 | 120.41 (19) | C12A—C11A—S1A | 111 (2) |
| C1—C2—H2 | 119.8 | C12A—C11A—H11A | 123.4 |

| | | | |
|--------------|--------------|-------------------|-------------|
| C3—C2—H2 | 119.8 | S1A—C11A—H11A | 125.1 |
| N1—C3—C4 | 121.1 (2) | C11A—C12A—H12A | 126.4 |
| N1—C3—C2 | 120.8 (2) | C13A—C12A—H12A | 118.7 |
| C4—C3—C2 | 118.0 (2) | C12A—C13A—C10 | 109.7 (16) |
| C5—C4—C3 | 121.0 (2) | C12A—C13A—H13A | 127.1 |
| C5—C4—H4 | 119.5 | C10—C13A—H13A | 122.4 |
| C3—C4—H4 | 119.5 | C14—O2—H2B | 111.5 |
| C4—C5—C6 | 121.2 (2) | O2—C14—C15 | 114.9 (10) |
| C4—C5—H5 | 119.4 | O2—C14—H14A | 106.6 |
| C6—C5—H5 | 119.4 | C15—C14—H14A | 108.0 |
| C5—C6—C1 | 117.7 (2) | O2—C14—H14B | 109.2 |
| C5—C6—C7 | 123.89 (19) | C15—C14—H14B | 110.1 |
| C1—C6—C7 | 118.42 (19) | H14A—C14—H14B | 107.9 |
| O1—C7—C8 | 120.21 (19) | C14—C15—H15A | 110.2 |
| O1—C7—C6 | 120.36 (19) | C14—C15—H15B | 108.2 |
| C8—C7—C6 | 119.43 (18) | H15A—C15—H15B | 109.5 |
| C9—C8—C7 | 121.9 (2) | C14—C15—H15C | 109.9 |
| C9—C8—H8 | 119.1 | H15A—C15—H15C | 109.5 |
| C7—C8—H8 | 119.1 | H15B—C15—H15C | 109.5 |
| C8—C9—C10 | 125.0 (2) | C14A—O2A—H2AA | 107.3 |
| C8—C9—H9 | 117.5 | O2A—C14A—C15A | 111.5 (9) |
| C10—C9—H9 | 117.5 | O2A—C14A—H14C | 103.1 |
| C13—C10—C9 | 127.7 (3) | C15A—C14A—H14C | 133.2 |
| C13A—C10—C9 | 128.8 (8) | O2A—C14A—H14D | 95.1 |
| C13A—C10—S1A | 111.0 (8) | C15A—C14A—H14D | 103.6 |
| C9—C10—S1A | 119.9 (3) | H14C—C14A—H14D | 103.7 |
| C13—C10—S1 | 110.6 (3) | C14A—C15A—H15G | 110.4 |
| C9—C10—S1 | 121.65 (17) | C14A—C15A—H15D | 108.7 |
| C11—S1—C10 | 91.9 (2) | H15G—C15A—H15D | 109.5 |
| C12—C11—S1 | 112.1 (4) | C14A—C15A—H15E | 109.4 |
| C12—C11—H11 | 124.0 | H15G—C15A—H15E | 109.5 |
| S1—C11—H11 | 123.9 | H15D—C15A—H15E | 109.5 |
| C11—C12—C13 | 110.6 (5) | | |
| | | | |
| C6—C1—C2—C3 | 0.2 (3) | C13A—C10—S1—C11 | 42 (5) |
| C1—C2—C3—N1 | -177.0 (2) | C9—C10—S1—C11 | 178.1 (3) |
| C1—C2—C3—C4 | 0.9 (3) | S1A—C10—S1—C11 | -4.5 (4) |
| N1—C3—C4—C5 | 176.8 (2) | C10—S1—C11—C12 | 0.6 (3) |
| C2—C3—C4—C5 | -1.0 (3) | S1—C11—C12—C13 | -0.7 (5) |
| C3—C4—C5—C6 | 0.0 (4) | C13A—C10—C13—C12 | -7.3 (10) |
| C4—C5—C6—C1 | 1.0 (3) | C9—C10—C13—C12 | -178.3 (4) |
| C4—C5—C6—C7 | 179.4 (2) | S1A—C10—C13—C12 | 154 (4) |
| C2—C1—C6—C5 | -1.1 (3) | S1—C10—C13—C12 | -0.2 (6) |
| C2—C1—C6—C7 | -179.63 (19) | C11—C12—C13—C10 | 0.6 (7) |
| C5—C6—C7—O1 | 176.5 (2) | C13—C10—S1A—C11A | -20 (4) |
| C1—C6—C7—O1 | -5.2 (3) | C13A—C10—S1A—C11A | -0.8 (17) |
| C5—C6—C7—C8 | -3.9 (3) | C9—C10—S1A—C11A | -175.2 (14) |
| C1—C6—C7—C8 | 174.47 (19) | S1—C10—S1A—C11A | 7.3 (14) |

| | | | |
|----------------|-------------|--------------------|------------|
| O1—C7—C8—C9 | -2.4 (3) | C10—S1A—C11A—C12A | 0.0 (13) |
| C6—C7—C8—C9 | 178.02 (19) | S1A—C11A—C12A—C13A | 0.7 (16) |
| C7—C8—C9—C10 | -179.1 (2) | C11A—C12A—C13A—C10 | -1 (2) |
| C8—C9—C10—C13 | 180.0 (4) | C13—C10—C13A—C12A | 4 (2) |
| C8—C9—C10—C13A | 11.2 (11) | C9—C10—C13A—C12A | 175.1 (18) |
| C8—C9—C10—S1A | -175.4 (4) | S1A—C10—C13A—C12A | 1 (2) |
| C8—C9—C10—S1 | 2.0 (3) | S1—C10—C13A—C12A | -136 (6) |
| C13—C10—S1—C11 | -0.2 (4) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N1—H1A \cdots O1 ⁱ | 0.86 | 2.16 | 2.931 (3) | 149 |
| N1—H1B \cdots Cg1 ⁱⁱ | 0.86 | 2.80 | 3.597 (3) | 156 |

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