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# 3-(Benzo[*d*]thiazol-2-yl)-2*H*-chromen-2-one

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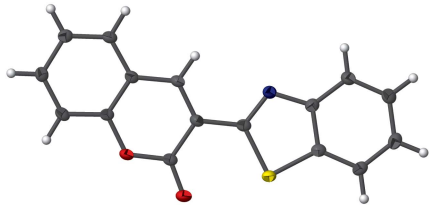
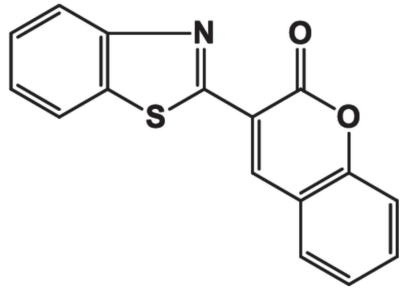
**Structural data**: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_9NO_2S$, the interplanar angle is 6.47 (6)°. An intramolecular S···O=C contact of 2.727 (2) Å is observed. The packing is determined by several types of weak interaction ('weak' hydrogen bonds, S···S contacts and $\pi$–$\pi$ stacking).

**3D view**



**Chemical scheme**




## Structure description

Coumarin (chromen-2-one) derivatives represent a significant class of organic heterocycles; they can be found in various natural or synthetic drug compounds and display a variety of biological activities (Curini *et al.*, 2006), the most noticeable of which are photobiological properties upon irradiation with UV light. Thus, many coumarin derivatives are effective photosensitizers with valuable applications in medicine (Bansal *et al.*, 2013). Because of these photochemical characteristics, together with their practical stability, ease of synthesis and good solubility, coumarins have been widely explored as solar energy collectors, charge-transfer agents and non-linear optical materials for photonic and electronic applications (Kim *et al.*, 2011). Coumarins are one of the most broadly investigated and commercially important classes of organic fluorescent materials. Coumarin dyes fluoresce in the blue–green spectroscopic region and are commonly used in daylight fluorescent pigments, fluorescent probes, and as tunable dye lasers or organic light-emitting diodes (Christie & Lui, 2000). The emission intensity of coumarin chromophores strongly depends on the nature and position of their substituents (Żamojć *et al.*, 2014).

Benzothiazole derivatives also exhibit strong luminescence in solution and in the solid state. Molecules that incorporate benzothiazoles have attracted considerable research interest in the field of organic light-emitting diodes because of their unique electro-optical properties (Wang *et al.*, 2010). Recently, we have synthesized some benzothiazoles

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| S1—C7*A* | 1.733 (2) | C8—C16 | 1.357 (3) |
| S1—C2 | 1.758 (2) | C8—C9 | 1.470 (3) |
| C2—N3 | 1.308 (3) | C9—O1 | 1.365 (3) |
| N3—C3*A* | 1.380 (3) | C10—O1 | 1.379 (2) |
| C3*A*—C7*A* | 1.411 (3) | C15—C16 | 1.434 (3) |
| | | | |
| C7*A*—S1—C2 | 88.87 (10) | N3—C3*A*—C7*A* | 115.15 (19) |
| N3—C2—S1 | 115.63 (16) | C3*A*—C7*A*—S1 | 109.57 (16) |
| C2—N3—C3*A* | 110.75 (18) | | |

(Azzam *et al.*, 2017*a*,*b*, 2020*a*,*b*,*c*, 2021; Elgemeie *et al.*, 2000*a*,*b*) and coumarin derivatives (Elgemeie, 1989; Elgemeie & Elghandour, 1990; Elgemeie *et al.*, 2015).

As a continuation of our research interest in exploiting new coumarin and benzothiazole derivatives for light-emitting materials, we describe here the synthesis and characterization of a benzothiazole-based coumarin compound. The reaction of *N*-[2-(benzo[*d*]thiazol-2-yl)acetyl]benzohydrazide (**1**) with salicylaldehde (**2**) was investigated. This gave a product whose mass spectrum was not consistent with the proposed structure *N*-(3-(benzo[*d*]thiazol-2-yl)-2-oxoquinolin-1(2*H*)-yl)benzamide (**5**). Therefore the X-ray crystal structure was determined, revealing that 3-(benzo[*d*]thiazol-2-yl)-2*H*-chromen-2-one (**4**) is the sole product in the solid state (Fig. 1). The formation of (**4**) is assumed to proceed *via* initial formation of adduct (**3**) and elimination of benzohydrazide rather than water.

The structure of **4** is shown in Fig. 2. Molecular dimensions may be regarded as normal; a brief selection is presented in Table 1. The ring systems are essentially planar, with r.m.s. deviations of 0.012 Å for the benzothiazole and 0.006 Å for the chromene (including the exocyclic oxygen atom); the

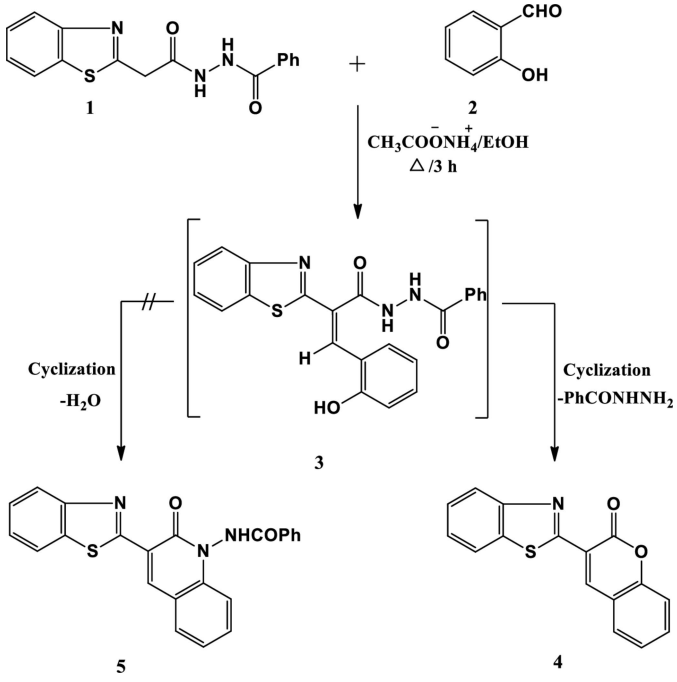



**Figure 1**
Reaction scheme.

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C14—H14···O1$^{i}$ | 0.95 | 2.46 | 3.383 (3) | 163 |
| C11—H11···N3$^{ii}$ | 0.95 | 2.63 | 3.523 (3) | 156 |
| C13—H13···O2$^{iii}$ | 0.95 | 2.65 | 3.314 (3) | 127 |

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

interplanar angle is 6.47 (6)°. The intramolecular contact distance S1···O2 is 2.727 (2) Å.

There are three short intermolecular H···O/H···N contacts (Table 2), the shortest of which could reasonably be regarded as a 'weak' hydrogen bond. Together with an S1···S1 contact [3.626 (1) Å, operator $-x, 1-y, 1-z$], this links the molecules to form corrugated sheets lying parallel to the *bc* plane (Fig. 3). However, this is only one way of considering the packing. The rings also display short contacts between their centroids *via* *x*-axis translation [thiazole···(benzothiazole benzo) 3.6187 (13) Å, thiazole···(coumarin heterocycle) 3.5344 (12) Å, (coumarin heterocycle)···(coumarin benzo) 3.4148 (12) Å], although the stacking is somewhat offset. Viewed parallel to the *c* axis, the rings are seen edge-on in a herringbone pattern.

A search of the Cambridge Database (Version 2021.3.0; Groom *et al.*, 2016) for purely organic coumarin derivatives

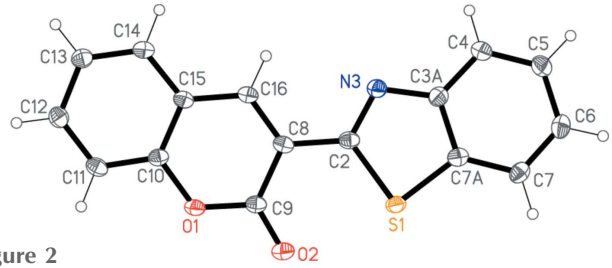



**Figure 2**
The molecule of **4** in the crystal. Ellipsoids represent 50% probability levels.

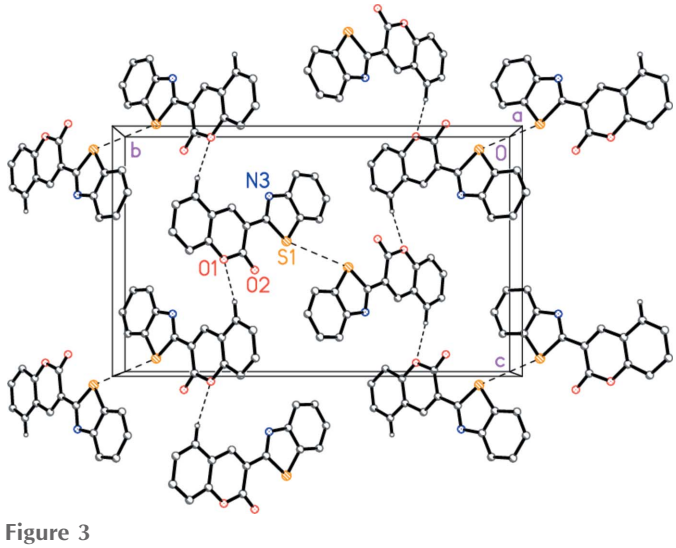



**Figure 3**
Crystal packing of **4** viewed parallel to the short *a* axis. Dashed lines indicate 'weak' hydrogen bonds or S···S contacts. Hydrogen atoms not involved in hydrogen bonding are omitted.

gave 1030 hits with 1299 individual molecules. The mean bond lengths of the coumarin heterocycle (referred to the numbering of **4**) are: C15—C16 = 1.442 (15), C16—C8 =1.356 (19), C8—C9 =1.448 (18), C9—O1 =1.375 (14) and O1—C10 =1.378 (11) Å, and the values in **4** (Table 1) are consistent with these mean values. A more specialized search revealed four compounds with simple coumarin derivatives linked to benzo[*d*]thiazol in the same way as in **4**: DARPIX (Ezeh & Harrop, 2012), VIVWEF and VIWDOX (Shi *et al.*, 2019), WINZAU (Jasinski & Paight, 1995). The first three have been used as fluorescent probes, for the detection of biothiols and the evaluation of anti-cancer agents, respectively.

## Synthesis and crystallization

A mixture of *N*-[2-(benzo[*d*]thiazol-2-yl)acetyl]benzohydrazide **1** (3.11 g, 0.01 mol), salicylaldehyde **2** (1.22 g, 0.01 mol) and ammonium acetate (0.77 g, 0.01 mol) in ethanol (10 mL) was refluxed for 3 h. The precipitate was filtered off and recrystallized from ethanol solution to give pale-yellow crystals in 95% yield, m.p. 501–503 K; IR (KBr, $cm^{-1}$): $\upsilon$ 3048, 3028 (CH-aromatic), 1715 (C═O), 1557 (C═N) and 1602, 1479 (C═C). $^1$H NMR (400 MHz DMSO-$d_6$) $\delta$: 7.46–8.21 (*m*, 8H, $2C_6H_4$), 9.26 (*s*, 1H, CH-pyran). $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 116.7, 119.2, 119.8, 122.7, 123.0, 125.7, 125.9, 127.2, 130.7, 134.2, 136.4, 142.5, 152.4, 153.8 (aromatic carbons), 159.9 (C═N), 160.1 (C═O). MS (EI): *m*/*z* (%) 281 [$M^+$+2] (0.44), 280 [$M^+$+1] (0.96), 279 [$M^+$] (4.06), 278 [$M^+$−1] (0.12), 277 [$M^+$−2] (0.17), 105 (100.00), 77 $[C_6H_5]^+$ (58.06). Analysis: calculated for $C_{16}H_9NO_2S$ (279.31): C 68.80; H 3.25; N 5.01; S 11.48%. Found: C 68.70; H 3.33; N 5.12; S 11.60%.

The sample consisted of a mass of long, extremely fine needles with an overall matt appearance. Careful separation and cutting provided a single crystal, which proved to be measurable using a high-intensity X-ray source.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{16}H_9NO_2S$ |
| $M_r$ | 279.30 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 100 |
| $a$, $b$, $c$ (Å) | 4.60717 (11), 20.7275 (5), 12.6444 (3) |
| $\beta$ (°) | 91.911 (2) |
| $V$ (Å$^3$) | 1206.81 (5) |
| $Z$ | 4 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ ($mm^{-1}$) | 2.39 |
| Crystal size (mm) | 0.04 × 0.01 × 0.01 |
| | |
| Data collection | |
| Diffractometer | XtaLAB Synergy |
| Absorption correction | Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021) |
| $T_{min}$, $T_{max}$ | 0.696, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 49602, 2547, 2528 |
| $R_{int}$ | 0.068 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.634 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.049, 0.120, 1.25 |
| No. of reflections | 2547 |
| No. of parameters | 181 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.45, −0.43 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXT* (Sheldrick, 2015*b*), *SHELXL2018/3* (Sheldrick, 2015*a*) and *XP* (Siemens, 1994).

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# full crystallographic data

*IUCrData* (2022). **7**, x220332 [https://doi.org/10.1107/S2414314622003327]

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*Crystal data*

$C_{16}H_9NO_2S$
$M_r$ = 279.30
Monoclinic, $P2_1/c$
$a$ = 4.60717 (11) Å
$b$ = 20.7275 (5) Å
$c$ = 12.6444 (3) Å
$\beta$ = 91.911 (2)°
$V$ = 1206.81 (5) Å$^3$
$Z$ = 4
$F(000)$ = 576
$D_x$ = 1.537 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54184 Å
Cell parameters from 20622 reflections
$\theta$ = 4.1–76.8°
$\mu$ = 2.38 mm$^{-1}$
$T$ = 100 K
Needle, pale yellow
0.04 × 0.01 × 0.01 mm

*Data collection*

XtaLAB Synergy diffractometer
Radiation source: micro-focus sealed X-ray tube
Detector resolution: 10.0000 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2021)
$T_{min}$ = 0.696, $T_{max}$ = 1.000
49602 measured reflections
2547 independent reflections
2528 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.068
$\theta_{max}$ = 77.9°, $\theta_{min}$ = 4.1°
$h$ = −5→5
$k$ = −24→26
$l$ = −15→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.120
$S$ = 1.25
2547 reflections
181 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 1.2289P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.45 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.
Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)
- 3.3898 (0.0014) x + 13.7552 (0.0080) y + 2.0177 (0.0070) z = 8.9676 (0.0037)
* 0.0227 (0.0011) S1 * 0.0005 (0.0015) C2 * -0.0077 (0.0015) N3 * -0.0083 (0.0019) C3A * 0.0016 (0.0017) C4 * 0.0176 (0.0017) C5 * 0.0019 (0.0017) C6 * -0.0169 (0.0016) C7 * -0.0115 (0.0018) C7A
Rms deviation of fitted atoms = 0.0124
- 3.2516 (0.0012) x + 14.6654 (0.0053) y + 0.7477 (0.0068) z = 9.1325 (0.0065)
Angle to previous plane (with approximate esd) = 6.467 ( 0.060 )
* -0.0054 (0.0017) C8 * -0.0016 (0.0018) C9 * 0.0001 (0.0019) C10 * -0.0072 (0.0017) C11 * 0.0011 (0.0017) C12 * -0.0035 (0.0017) C13 * -0.0017 (0.0017) C14 * 0.0079 (0.0019) C15 * 0.0022 (0.0016) C16 * 0.0136 (0.0014) O1 * -0.0054 (0.0014) O2
Rms deviation of fitted atoms = 0.0059
#==========
Short contact:
3.6259 (0.0010) S1 - S1_$4
Operator $4: -x, 1-y, 1-z

**Refinement**. The hydrogen atoms were included using a riding model starting from calculated positions (C—$H_{aromatic}$ 0.95 Å). The *U*(H) values were fixed at 1.2 times the equivalent $U_{iso}$ value of the parent carbon atoms.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | −0.03347 (11) | 0.58024 (2) | 0.44385 (4) | 0.01871 (16) |
| C2 | 0.1290 (5) | 0.63202 (10) | 0.35289 (16) | 0.0179 (4) |
| N3 | 0.0429 (4) | 0.62471 (9) | 0.25396 (14) | 0.0190 (4) |
| C3A | −0.1604 (5) | 0.57600 (10) | 0.24410 (17) | 0.0197 (4) |
| C4 | −0.3012 (5) | 0.55593 (11) | 0.14936 (17) | 0.0227 (5) |
| H4 | −0.257833 | 0.575871 | 0.084112 | 0.027* |
| C5 | −0.5025 (5) | 0.50703 (11) | 0.15244 (18) | 0.0238 (5) |
| H5 | −0.600561 | 0.493791 | 0.088821 | 0.029* |
| C6 | −0.5657 (5) | 0.47627 (11) | 0.24818 (18) | 0.0230 (5) |
| H6 | −0.704244 | 0.442317 | 0.248094 | 0.028* |
| C7 | −0.4295 (5) | 0.49470 (10) | 0.34205 (18) | 0.0208 (4) |
| H7 | −0.471183 | 0.473769 | 0.406635 | 0.025* |
| C7A | −0.2284 (5) | 0.54501 (10) | 0.33950 (16) | 0.0189 (4) |
| C8 | 0.3479 (4) | 0.67984 (10) | 0.38551 (16) | 0.0174 (4) |
| C9 | 0.4134 (5) | 0.68882 (10) | 0.49914 (16) | 0.0186 (4) |
| C10 | 0.7662 (5) | 0.76940 (10) | 0.45552 (16) | 0.0174 (4) |
| C11 | 0.9720 (5) | 0.81249 (10) | 0.49547 (16) | 0.0200 (4) |
| H11 | 1.009311 | 0.816281 | 0.569554 | 0.024* |
| C12 | 1.1223 (5) | 0.84998 (10) | 0.42467 (17) | 0.0208 (4) |
| H12 | 1.262170 | 0.880282 | 0.450509 | 0.025* |
| C13 | 1.0693 (5) | 0.84351 (10) | 0.31510 (17) | 0.0202 (4) |
| H13 | 1.174863 | 0.869054 | 0.267249 | 0.024* |
| C14 | 0.8647 (5) | 0.80021 (10) | 0.27695 (16) | 0.0191 (4) |
| H14 | 0.830301 | 0.796009 | 0.202773 | 0.023* |

| | | | | |
|---|---|---|---|---|
| C15 | 0.7059 (4) | 0.76208 (10) | 0.34688 (16) | 0.0173 (4) |
| C16 | 0.4906 (4) | 0.71565 (10) | 0.31393 (16) | 0.0173 (4) |
| H16 | 0.447058 | 0.709837 | 0.240584 | 0.021* |
| O1 | 0.6178 (3) | 0.73370 (7) | 0.52821 (11) | 0.0198 (3) |
| O2 | 0.2995 (4) | 0.65973 (8) | 0.56960 (12) | 0.0247 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0214 (3) | 0.0199 (3) | 0.0150 (3) | −0.00055 (19) | 0.00235 (18) | 0.00232 (18) |
| C2 | 0.0205 (10) | 0.0179 (10) | 0.0156 (9) | 0.0036 (8) | 0.0022 (7) | 0.0008 (7) |
| N3 | 0.0211 (9) | 0.0194 (9) | 0.0166 (8) | −0.0004 (7) | 0.0017 (7) | −0.0005 (7) |
| C3A | 0.0209 (10) | 0.0180 (10) | 0.0203 (10) | 0.0004 (8) | 0.0033 (8) | −0.0002 (8) |
| C4 | 0.0265 (11) | 0.0241 (11) | 0.0175 (10) | −0.0013 (9) | 0.0009 (8) | −0.0015 (8) |
| C5 | 0.0268 (11) | 0.0234 (11) | 0.0213 (11) | −0.0010 (9) | 0.0017 (8) | −0.0048 (8) |
| C6 | 0.0212 (11) | 0.0182 (10) | 0.0300 (12) | −0.0016 (8) | 0.0042 (9) | −0.0026 (9) |
| C7 | 0.0212 (10) | 0.0180 (10) | 0.0237 (11) | 0.0006 (8) | 0.0059 (8) | 0.0014 (8) |
| C7A | 0.0210 (10) | 0.0180 (10) | 0.0178 (10) | 0.0034 (8) | 0.0026 (8) | 0.0011 (8) |
| C8 | 0.0190 (10) | 0.0190 (10) | 0.0142 (9) | 0.0034 (8) | 0.0002 (7) | −0.0005 (7) |
| C9 | 0.0207 (10) | 0.0204 (10) | 0.0149 (9) | 0.0024 (8) | 0.0021 (8) | −0.0003 (8) |
| C10 | 0.0200 (10) | 0.0171 (10) | 0.0153 (9) | 0.0017 (8) | 0.0026 (7) | 0.0004 (7) |
| C11 | 0.0236 (11) | 0.0206 (10) | 0.0158 (9) | 0.0034 (8) | −0.0005 (8) | −0.0034 (8) |
| C12 | 0.0217 (10) | 0.0191 (10) | 0.0214 (10) | 0.0002 (8) | −0.0010 (8) | −0.0023 (8) |
| C13 | 0.0221 (10) | 0.0193 (10) | 0.0194 (10) | 0.0017 (8) | 0.0024 (8) | 0.0013 (8) |
| C14 | 0.0229 (10) | 0.0199 (10) | 0.0145 (9) | 0.0021 (8) | 0.0012 (8) | 0.0008 (8) |
| C15 | 0.0201 (10) | 0.0169 (10) | 0.0147 (10) | 0.0027 (8) | −0.0004 (7) | 0.0001 (7) |
| C16 | 0.0192 (10) | 0.0197 (10) | 0.0130 (9) | 0.0026 (8) | 0.0007 (7) | −0.0002 (7) |
| O1 | 0.0242 (8) | 0.0227 (8) | 0.0125 (7) | −0.0010 (6) | 0.0010 (6) | 0.0005 (6) |
| O2 | 0.0303 (8) | 0.0281 (8) | 0.0157 (7) | −0.0034 (7) | 0.0030 (6) | 0.0022 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C7A | 1.733 (2) | C10—C11 | 1.385 (3) |
| S1—C2 | 1.758 (2) | C10—C15 | 1.401 (3) |
| C2—N3 | 1.308 (3) | C11—C12 | 1.388 (3) |
| C2—C8 | 1.464 (3) | C12—C13 | 1.405 (3) |
| N3—C3A | 1.380 (3) | C13—C14 | 1.377 (3) |
| C3A—C4 | 1.406 (3) | C14—C15 | 1.409 (3) |
| C3A—C7A | 1.411 (3) | C15—C16 | 1.434 (3) |
| C4—C5 | 1.375 (3) | C4—H4 | 0.9500 |
| C5—C6 | 1.407 (3) | C5—H5 | 0.9500 |
| C6—C7 | 1.378 (3) | C6—H6 | 0.9500 |
| C7—C7A | 1.396 (3) | C7—H7 | 0.9500 |
| C8—C16 | 1.357 (3) | C11—H11 | 0.9500 |
| C8—C9 | 1.470 (3) | C12—H12 | 0.9500 |
| C9—O2 | 1.210 (3) | C13—H13 | 0.9500 |
| C9—O1 | 1.365 (3) | C14—H14 | 0.9500 |
| C10—O1 | 1.379 (2) | C16—H16 | 0.9500 |

| | | | |
|---|---|---|---|
| C7A—S1—C2 | 88.87 (10) | C14—C13—C12 | 120.1 (2) |
| N3—C2—C8 | 122.13 (19) | C13—C14—C15 | 120.63 (19) |
| N3—C2—S1 | 115.63 (16) | C10—C15—C14 | 117.67 (19) |
| C8—C2—S1 | 122.24 (15) | C10—C15—C16 | 118.07 (19) |
| C2—N3—C3A | 110.75 (18) | C14—C15—C16 | 124.25 (19) |
| N3—C3A—C4 | 125.9 (2) | C8—C16—C15 | 121.26 (19) |
| N3—C3A—C7A | 115.15 (19) | C9—O1—C10 | 122.61 (16) |
| C4—C3A—C7A | 119.0 (2) | C5—C4—H4 | 120.5 |
| C5—C4—C3A | 119.1 (2) | C3A—C4—H4 | 120.5 |
| C4—C5—C6 | 121.2 (2) | C4—C5—H5 | 119.4 |
| C7—C6—C5 | 120.9 (2) | C6—C5—H5 | 119.4 |
| C6—C7—C7A | 118.1 (2) | C7—C6—H6 | 119.5 |
| C7—C7A—C3A | 121.7 (2) | C5—C6—H6 | 119.5 |
| C7—C7A—S1 | 128.67 (17) | C6—C7—H7 | 121.0 |
| C3A—C7A—S1 | 109.57 (16) | C7A—C7—H7 | 121.0 |
| C16—C8—C2 | 121.80 (18) | C10—C11—H11 | 120.8 |
| C16—C8—C9 | 119.64 (19) | C12—C11—H11 | 120.8 |
| C2—C8—C9 | 118.56 (18) | C11—C12—H12 | 119.7 |
| O2—C9—O1 | 116.96 (18) | C13—C12—H12 | 119.7 |
| O2—C9—C8 | 125.2 (2) | C14—C13—H13 | 119.9 |
| O1—C9—C8 | 117.81 (18) | C12—C13—H13 | 119.9 |
| O1—C10—C11 | 116.83 (18) | C13—C14—H14 | 119.7 |
| O1—C10—C15 | 120.59 (19) | C15—C14—H14 | 119.7 |
| C11—C10—C15 | 122.58 (19) | C8—C16—H16 | 119.4 |
| C10—C11—C12 | 118.41 (19) | C15—C16—H16 | 119.4 |
| C11—C12—C13 | 120.6 (2) | | |

| | | | |
|---|---|---|---|
| C7A—S1—C2—N3 | 1.07 (17) | C16—C8—C9—O2 | 179.9 (2) |
| C7A—S1—C2—C8 | −178.49 (18) | C2—C8—C9—O2 | −0.2 (3) |
| C8—C2—N3—C3A | 179.04 (18) | C16—C8—C9—O1 | 0.2 (3) |
| S1—C2—N3—C3A | −0.5 (2) | C2—C8—C9—O1 | −179.90 (17) |
| C2—N3—C3A—C4 | 179.1 (2) | O1—C10—C11—C12 | −178.97 (18) |
| C2—N3—C3A—C7A | −0.5 (3) | C15—C10—C11—C12 | 0.4 (3) |
| N3—C3A—C4—C5 | −179.1 (2) | C10—C11—C12—C13 | −0.9 (3) |
| C7A—C3A—C4—C5 | 0.5 (3) | C11—C12—C13—C14 | 0.7 (3) |
| C3A—C4—C5—C6 | −1.1 (3) | C12—C13—C14—C15 | 0.1 (3) |
| C4—C5—C6—C7 | 0.6 (3) | O1—C10—C15—C14 | 179.76 (18) |
| C5—C6—C7—C7A | 0.4 (3) | C11—C10—C15—C14 | 0.5 (3) |
| C6—C7—C7A—C3A | −1.0 (3) | O1—C10—C15—C16 | −1.3 (3) |
| C6—C7—C7A—S1 | 177.72 (17) | C11—C10—C15—C16 | 179.42 (19) |
| N3—C3A—C7A—C7 | −179.79 (19) | C13—C14—C15—C10 | −0.7 (3) |
| C4—C3A—C7A—C7 | 0.6 (3) | C13—C14—C15—C16 | −179.6 (2) |
| N3—C3A—C7A—S1 | 1.3 (2) | C2—C8—C16—C15 | −179.62 (19) |
| C4—C3A—C7A—S1 | −178.36 (16) | C9—C8—C16—C15 | 0.3 (3) |
| C2—S1—C7A—C7 | 179.9 (2) | C10—C15—C16—C8 | 0.2 (3) |
| C2—S1—C7A—C3A | −1.24 (16) | C14—C15—C16—C8 | 179.1 (2) |
| N3—C2—C8—C16 | −5.4 (3) | O2—C9—O1—C10 | 179.04 (18) |

| | | | |
|---|---|---|---|
| S1—C2—C8—C16 | 174.10 (16) | C8—C9—O1—C10 | −1.2 (3) |
| N3—C2—C8—C9 | 174.66 (19) | C11—C10—O1—C9 | −178.83 (18) |
| S1—C2—C8—C9 | −5.8 (3) | C15—C10—O1—C9 | 1.8 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C14—H14···O1$^{i}$ | 0.95 | 2.46 | 3.383 (3) | 163 |
| C11—H11···N3$^{ii}$ | 0.95 | 2.63 | 3.523 (3) | 156 |
| C13—H13···O2$^{iii}$ | 0.95 | 2.65 | 3.314 (3) | 127 |

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