



# Crystal structure of 3-(benzo[d]thiazol-2-yl)-6-methyl-2H-chromen-2-one

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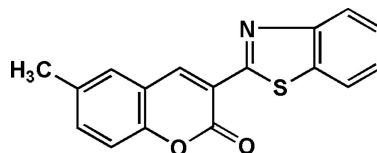
This article is part of a collection of articles to commemorate the founding of the African Crystallographic Association and the 75th anniversary of the IUCr.

**Keywords:** benzothiazole; coumarin; crystal structure.**CCDC reference:** 2256779**Supporting information:** this article has supporting information at journals.iucr.org/e

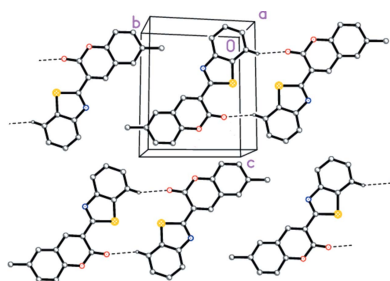
The molecule of the title compound, C<sub>17</sub>H<sub>11</sub>NO<sub>2</sub>S, is almost planar, with an interplanar angle of 3.01 (3)° between the benzothiazole and chromene ring systems. A short intramolecular S···O=C contact of 2.727 (2) Å is observed. The crystal packing involves a layer structure parallel to (211), containing dimeric inversion-symmetric units connected by a 'weak' C—H···O=C hydrogen bond.

## 1. Chemical context

Benzothiazole and its derivatives are important heterocyclic aromatic compounds. Benzothiazole can be readily substituted at the C-2 position of the thiazole ring (Elgemeie *et al.*, 2020). Compounds containing a benzothiazolyl group have found numerous applications in medicine and in nonlinear optics (Sigmundová *et al.*, 2007). Benzothiazole derivatives can also display strong fluorescence and luminescence in the solid state and in solution (Wang *et al.*, 2010). Benzothiazole compounds as incorporated in organic light-emitting diodes have attracted substantial attention because of their notable photovoltaic properties (Ghanavatkar *et al.*, 2020). Recently, we have synthesized novel heterocyclic derivatives involving the benzothiazole moiety, many of which showed significant biological and fluorescence activities (Azzam *et al.*, 2020; Khedr *et al.*, 2022).



Coumarin is a natural product with the systematic name 2H-chromen-2-one. Its derivatives represent an important class of organic heterocycles. Thus, they are constituents of many intensively investigated and commercially important organic fluorescent materials; they also display important biological activities and are found in synthetic drugs (Curini *et al.*, 2006). Furthermore, many coumarin compounds are current photosensitizers with valuable applications in medicinal chemistry (Bansal *et al.*, 2013). Because of their photochemical properties, coumarin compounds have found applications in nonlinear optical materials, solar energy collectors and charge-transfer agents (Kim *et al.*, 2011), and also as daylight fluorescent pigments, tunable dye lasers, fluorescent probes and components of organic light-emitting diodes (Christie & Lui, 2000). The emission intensities of coumarin chromo-



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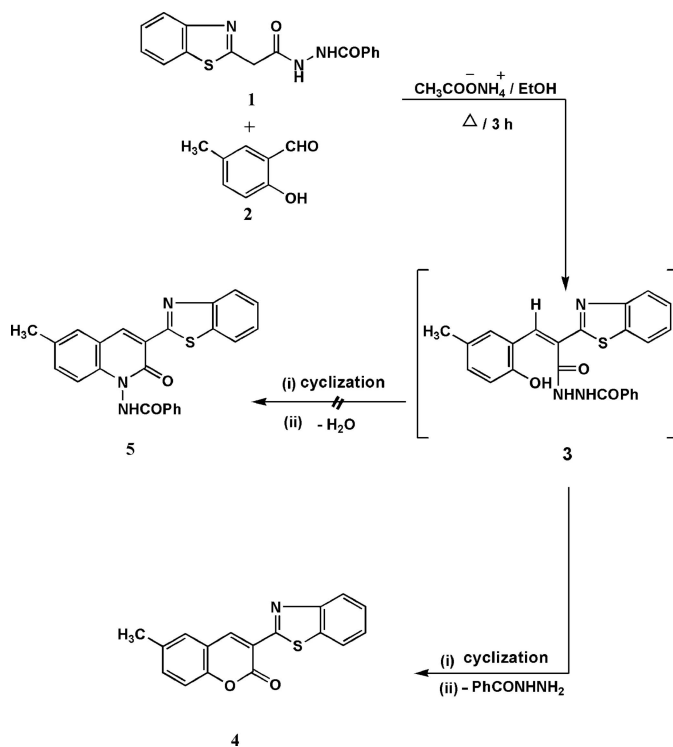
**Table 1**  
Selected geometric parameters (Å, °).

O1—C2	1.3723 (17)	S11—C17A	1.7343 (14)
O1—C8A	1.3765 (18)	S11—C12	1.7515 (15)
O2—C2	1.2077 (19)	C12—N13	1.3076 (18)
C3—C12	1.4698 (19)	N13—C13A	1.3836 (18)
C2—O1—C8A	122.52 (11)	N13—C12—S11	115.94 (11)
O2—C2—O1	116.99 (12)	C3—C12—S11	123.35 (10)
O2—C2—C3	125.69 (14)	N13—C13A—C14	124.90 (13)
O1—C2—C3	117.32 (13)	N13—C13A—C17A	115.23 (12)
C17A—S11—C12	88.94 (7)	C17—C17A—S11	129.05 (12)
N13—C12—C3	120.72 (13)	C13A—C17A—S11	109.42 (11)

phores depend on the nature of their substituents at various sites (Žamojć *et al.*, 2014).

In two recent papers (Abdallah *et al.*, 2022, 2023), we have given a more extensive list of references concerning the properties of benzothiazoles and coumarins, including many of our own publications in these fields.

Some decades ago, we reported the syntheses and characterizations of novel coumarin derivatives that have found applications as laser dyes (Elgemeie, 1989); these included 3-(benzo[*d*]thiazol-2-yl)-2*H*-chromen-2-one, the desmethyl analogue of title compound **4**, a benzothiazole-based coumarin derivative which was synthesized by the reaction of 2-(cyanomethyl)benzothiazole with salicylaldehyde. Afterwards, other research groups utilized essentially the same reaction to synthesize different derivatives of the same heterocyclic framework, including compound **4** (Chao *et al.*, 2010; Makowska *et al.*, 2019).



**Figure 1**  
Reaction scheme showing the attempted synthesis of compound **5**, which led instead to the product **4**.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C17—H17 $\cdots$ O2 <sup>i</sup>	0.95	2.43	3.3235 (19)	157

Symmetry code: (i)  $-x + 2, -y, -z + 1$ .

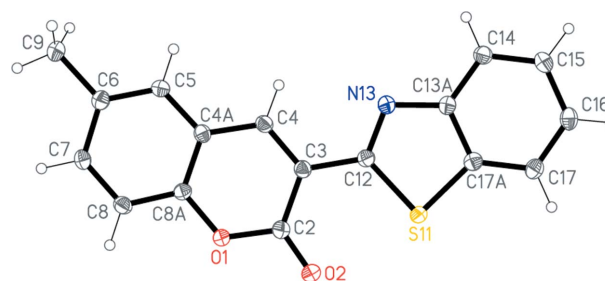
Recently, we attempted to synthesize *N*-[3-(benzo[*d*]thiazol-2-yl)-6-methyl-2-oxoquinolin-1(2*H*)-yl]benzamide (**5**) by the reaction of *N*-[2-(benzo[*d*]thiazol-2-yl)acetyl]benzohydrazide (**1**) (Azzam *et al.*, 2021) with 5-methylsalicylaldehyde (**2**) (Fig. 1). However, the product gave a mass spectrum that was inconsistent with the proposed structure. Therefore, the X-ray crystal structure was determined, indicating the formation of 3-(benzo[*d*]thiazol-2-yl)-6-methyl-2*H*-chromen-2-one (**4**) as the sole product, presumably arising *via* the initial formation of adduct **3** and the subsequent elimination of benzohydrazide rather than water. This is consistent with our recent observation that the desmethyl compound mentioned above is also formed as an unintended product by an exactly analogous reaction (Abdallah *et al.*, 2022). The products thus represent, by coincidence, a continuation of our research on developing new benzothiazole and coumarin derivatives as organic fluorescent constituents (Elgemeie *et al.*, 2015).

## 2. Structural commentary

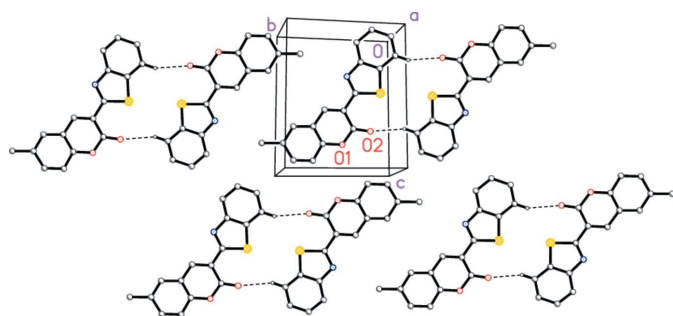
The structure of compound **4** is shown in Fig. 2. Its bond lengths and angles may be regarded as normal; a selection is presented in Table 1. The chromene and benzothiazole ring systems are planar as expected, with respective r.m.s. deviations of 0.020 and 0.015 Å; the angle between these planes is only 3.01 (3)°, so that the whole molecule almost planar. A short intramolecular S11 $\cdots$ O2 contact of 2.792 (1) Å is observed. The desmethyl analogue (Abdallah *et al.*, 2022) has, as expected, a closely similar molecular structure, with an S $\cdots$ O=C contact of 2.727 (2) Å and an interplanar angle of 6.47 (6)°, but is not isotopic to **4**.

## 3. Supramolecular features

The short contact H17 $\cdots$ O2 (at  $-x + 2, -y, -z + 1$ ; see Table 2) may be regarded as a ‘weak’ hydrogen bond. It links



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



**Figure 3**  
Packing diagram of compound **4**, viewed perpendicular to (211). Dashed lines indicate 'weak' hydrogen bonds. Atom labels indicate the asymmetric unit. H atoms other than H17 have been omitted.

the molecules to form inversion-symmetric dimers (Fig. 3) in which the S...S distance is 3.678 (1) Å. Adjacent dimers are related by translation to give an extended structure consisting of layers parallel to (211). The intercentroid distances (in Å) between rings of neighbouring layers, defining rings A–D as thiazole, the arene ring of benzothiazole, pyran and the arene ring of chromene, respectively, are A...C = 3.64, A...D = 3.70 and B...D = 3.61 [all with the symmetry code (−x + 1, −y + 1, −z + 1)], B...C = 3.51 and B...D = 3.66 Å [both with the symmetry code (−x + 2, −y + 1, −z + 1)]. The ring offsets (in the same order) are 1.44, 1.32, 0.80, 0.86 and 1.39 Å.

#### 4. Database survey

The searches employed the routine *ConQuest* (Bruno *et al.*, 2002), part of Version 2022.3.0 of the Cambridge Structural Database (Groom *et al.*, 2016). A search for structures containing both a coumarin and a benzothiazole ring system in the same residue gave 16 hits. After excluding ring systems with more extended annelation and molecules where the ring systems were not directly bonded to each other, 7 hits remained. In all of these, the benzothiazole was bonded *via* its 2-position. The structure with refcode AKUCUG (Bakthadoss & Selvakumar, 2016) involved a linkage *via* the 8-position of the coumarin; the other 6 hits [VIVWEF and VIWDOX (Shi *et al.*, 2019); WINZAU (Jasinski & Paight, 1995); SECSEC (Abdallah *et al.*, 2022); PEGMEX and PEGMIB (Singh *et al.*, 2022)] all had this linkage at the 3-position, as in compound **4**. In all cases, a short intramolecular S...O=C contact was observed, with distances in the range 2.681–2.786 Å.

#### 5. Synthesis and crystallization

5-Methylsalicylaldehyde, **2** (1.36 g, 0.01 mol) and ammonium acetate (0.77 g, 0.01 mol) were added to a solution of *N*-[2-(benzo[*d*]thiazol-2-yl)acetyl]benzohydrazide, **1** (3.11 g, 0.01 mol), in ethanol (10 mL). The reaction mixture was refluxed for *ca* 3 h and the resulting precipitate was collected by filtration and recrystallized from ethanol.

Pale-yellow crystals; yield: 96% (2.82 g); m.p. 495–497 K; IR (KBr, cm<sup>−1</sup>): ν 3062, (CH-aromatic), 2918 (CH<sub>3</sub>), 1710

**Table 3**

Experimental details.

Crystal data	
Chemical formula	C <sub>17</sub> H <sub>11</sub> NO <sub>2</sub> S
<i>M<sub>r</sub></i>	293.33
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.1592 (2), 9.0048 (2), 10.8678 (3)
α, β, γ (°)	82.779 (2), 76.016 (2), 75.078 (2)
<i>V</i> (Å <sup>3</sup> )	655.42 (3)
<i>Z</i>	2
Radiation type	Cu <i>K</i> α
μ (mm <sup>−1</sup> )	2.22
Crystal size (mm)	0.2 × 0.08 × 0.03
Data collection	
Diffractometer	Rigaku XtaLAB Synergy
Absorption correction	Multi-scan ( <i>CrysAlis PRO</i> ; Rigaku OD, 2022)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.391, 1.000
No. of measured, independent and observed [ <i>I</i> > 2σ( <i>I</i> )] reflections	4408, 4408, 4313
<i>R<sub>int</sub></i>	See <i>Refinement</i>
(sin θ/λ) <sub>max</sub> (Å <sup>−1</sup> )	0.634
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.035, 0.102, 1.07
No. of reflections	4408
No. of parameters	192
H-atom treatment	H-atom parameters constrained
Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>−3</sup> )	0.36, −0.28

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022), *SHELXT* (Sheldrick, 2015a), *SHELXL2018* (Sheldrick, 2015b) and *XP* (Siemens, 1994).

(C=O), 1582 (C=N) and 1619, 1485 (C=C). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>): δ 2.40 (*s*, 3H, CH<sub>3</sub>), 7.42–8.18 (*m*, 7H, C<sub>6</sub>H<sub>4</sub>, C<sub>6</sub>H<sub>3</sub>), 9.19 (*s*, 1H, CH-pyran). <sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>6</sub>): δ 20.8 (CH<sub>3</sub>), 116.5, 119.0, 120.0, 122.7, 123.0, 124.5, 125.9, 127.2, 128.6, 130.2, 135.2, 136.4, 142.4, 152.4 (aromatic C atoms, pyran ring), 160.0 (C=N), 160.4 (C=O). MS (EI): *m/z* (%) 293 [*M*<sup>+</sup>] (100.0). Analysis calculated (%) for C<sub>17</sub>H<sub>11</sub>NO<sub>2</sub>S: C 69.61, H 3.78, N 4.77, S 10.93; found: C 69.42, H 3.90, N 4.66, S 10.99.

#### 6. Refinement

The title crystal was a non-merohedral two-component twin. The orientations are related by a 180° rotation around the reciprocal axis *c*\*. The structure was refined using the HKLF5 method, whereby the relative volume of the smaller twin component refined to 0.387 (1). For non-merohedral twins thus refined, *R<sub>int</sub>* is not a valid concept, and the number of reflections should be interpreted with caution, because the equivalent reflections in the intensity file have already been merged.

Crystal data, data collection and structure refinement details are summarized in Table 3. The methyl group was included as an idealized rigid group allowed to rotate but not tip (C–H = 0.98 Å and H–C–H = 109.5°). Other H atoms were included using a riding model starting from calculated positions (aromatic C–H = 0.95 Å). The *U*<sub>iso</sub>(H) values were fixed at 1.5 times *U*<sub>eq</sub> of the parent C atoms for methyl groups and at 1.2 times *U*<sub>eq</sub> for the other H atoms.

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## supporting information

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## Crystal structure of 3-(benzo[d]thiazol-2-yl)-6-methyl-2H-chromen-2-one

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## Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2022); cell refinement: *CrysAlis PRO* (Rigaku OD, 2022); data reduction: *CrysAlis PRO* (Rigaku OD, 2022); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL2018* (Sheldrick, 2015b).

## 3-(Benzo[d]thiazol-2-yl)-6-methyl-2H-chromen-2-one

## Crystal data

$C_{17}H_{11}NO_2S$

$M_r = 293.33$

Triclinic,  $P\bar{1}$

$a = 7.1592$  (2) Å

$b = 9.0048$  (2) Å

$c = 10.8678$  (3) Å

$\alpha = 82.779$  (2)°

$\beta = 76.016$  (2)°

$\gamma = 75.078$  (2)°

$V = 655.42$  (3) Å<sup>3</sup>

$Z = 2$

$F(000) = 304$

$D_x = 1.486$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54184$  Å

Cell parameters from 15669 reflections

$\theta = 5.1\text{--}76.9^\circ$

$\mu = 2.22$  mm<sup>-1</sup>

$T = 100$  K

Lath, yellow

$0.2 \times 0.08 \times 0.03$  mm

## Data collection

Rigaku XtaLAB Synergy  
diffractometer

Radiation source: micro-focus sealed X-ray  
tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Rigaku OD, 2022)

$T_{\min} = 0.391$ ,  $T_{\max} = 1.000$

4408 measured reflections

4408 independent reflections

4313 reflections with  $I > 2\sigma(I)$

$\theta_{\max} = 77.7^\circ$ ,  $\theta_{\min} = 4.2^\circ$

$h = -9 \rightarrow 9$

$k = -11 \rightarrow 11$

$l = -13 \rightarrow 13$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.102$

$S = 1.07$

4408 reflections

192 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.069P)^2 + 0.1477P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.36$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.28$  e Å<sup>-3</sup>

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.51103 (16)	0.48246 (12)	0.81168 (9)	0.0190 (2)
O2	0.6896 (2)	0.26471 (13)	0.73105 (10)	0.0284 (3)
C2	0.6325 (2)	0.40210 (18)	0.71141 (13)	0.0193 (3)
C3	0.6808 (2)	0.48993 (17)	0.58998 (12)	0.0165 (3)
C4	0.6030 (2)	0.64361 (17)	0.57955 (12)	0.0167 (3)
H4	0.632732	0.699040	0.499800	0.020*
C4A	0.4771 (2)	0.72457 (17)	0.68581 (13)	0.0163 (3)
C5	0.3967 (2)	0.88415 (17)	0.68151 (13)	0.0180 (3)
H5	0.421660	0.943350	0.603163	0.022*
C6	0.2821 (2)	0.95747 (18)	0.78852 (13)	0.0186 (3)
C7	0.2475 (2)	0.86682 (17)	0.90335 (13)	0.0179 (3)
H7	0.169855	0.915469	0.977831	0.022*
C8A	0.4369 (2)	0.63944 (17)	0.80150 (13)	0.0163 (3)
C8	0.3231 (2)	0.70939 (17)	0.91095 (13)	0.0185 (3)
H8	0.297826	0.650194	0.989268	0.022*
C9	0.1992 (3)	1.12900 (19)	0.78301 (15)	0.0253 (3)
H9A	0.109385	1.158236	0.864314	0.038*
H9B	0.126469	1.159123	0.714667	0.038*
H9C	0.307944	1.181373	0.766476	0.038*
S11	0.92276 (5)	0.21156 (4)	0.48745 (3)	0.01826 (13)
C12	0.8130 (2)	0.40930 (16)	0.48039 (13)	0.0163 (3)
N13	0.85618 (18)	0.48415 (14)	0.37018 (11)	0.0170 (3)
C13A	0.9840 (2)	0.38535 (17)	0.28225 (13)	0.0167 (3)
C14	1.0636 (2)	0.42978 (18)	0.15610 (13)	0.0189 (3)
H14	1.029684	0.533983	0.123985	0.023*
C15	1.1927 (2)	0.31842 (18)	0.07949 (13)	0.0200 (3)
H15	1.249720	0.347072	-0.005730	0.024*
C16	1.2408 (2)	0.16354 (18)	0.12571 (14)	0.0205 (3)
H16	1.327948	0.089102	0.070564	0.025*
C17A	1.0364 (2)	0.23021 (17)	0.32803 (13)	0.0177 (3)
C17	1.1641 (2)	0.11734 (18)	0.24965 (14)	0.0198 (3)
H17	1.196910	0.012568	0.280584	0.024*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0238 (6)	0.0148 (5)	0.0158 (5)	-0.0030 (4)	-0.0012 (4)	-0.0006 (4)
O2	0.0399 (7)	0.0162 (6)	0.0212 (5)	-0.0006 (5)	0.0006 (5)	0.0007 (4)
C2	0.0228 (7)	0.0171 (7)	0.0174 (6)	-0.0044 (6)	-0.0029 (5)	-0.0018 (5)

C3	0.0178 (7)	0.0169 (7)	0.0151 (6)	-0.0046 (5)	-0.0036 (5)	-0.0016 (5)
C4	0.0188 (7)	0.0177 (7)	0.0141 (6)	-0.0050 (6)	-0.0036 (5)	-0.0008 (5)
C4A	0.0165 (7)	0.0178 (7)	0.0153 (6)	-0.0044 (6)	-0.0037 (5)	-0.0021 (5)
C5	0.0196 (7)	0.0173 (7)	0.0163 (6)	-0.0043 (6)	-0.0034 (5)	0.0009 (5)
C6	0.0179 (7)	0.0181 (7)	0.0198 (6)	-0.0034 (6)	-0.0046 (5)	-0.0024 (5)
C7	0.0157 (7)	0.0205 (7)	0.0167 (6)	-0.0029 (5)	-0.0017 (5)	-0.0041 (5)
C8A	0.0170 (7)	0.0149 (7)	0.0174 (6)	-0.0047 (5)	-0.0039 (5)	-0.0004 (5)
C8	0.0198 (7)	0.0197 (7)	0.0158 (6)	-0.0060 (6)	-0.0024 (5)	0.0002 (5)
C9	0.0317 (8)	0.0180 (8)	0.0217 (7)	-0.0002 (6)	-0.0027 (6)	-0.0034 (5)
S11	0.0232 (2)	0.01407 (19)	0.01565 (19)	-0.00208 (14)	-0.00330 (13)	-0.00092 (13)
C12	0.0172 (6)	0.0160 (7)	0.0162 (6)	-0.0040 (5)	-0.0046 (5)	-0.0011 (5)
N13	0.0174 (6)	0.0172 (6)	0.0160 (5)	-0.0038 (5)	-0.0031 (4)	-0.0018 (4)
C13A	0.0157 (7)	0.0168 (7)	0.0180 (6)	-0.0038 (5)	-0.0037 (5)	-0.0027 (5)
C14	0.0190 (7)	0.0190 (7)	0.0184 (6)	-0.0047 (6)	-0.0038 (5)	-0.0003 (5)
C15	0.0186 (7)	0.0240 (8)	0.0175 (6)	-0.0067 (6)	-0.0017 (5)	-0.0028 (5)
C16	0.0185 (7)	0.0227 (8)	0.0211 (7)	-0.0041 (6)	-0.0036 (5)	-0.0076 (6)
C17A	0.0186 (7)	0.0171 (7)	0.0186 (6)	-0.0052 (6)	-0.0051 (5)	-0.0016 (5)
C17	0.0206 (7)	0.0176 (7)	0.0216 (7)	-0.0036 (6)	-0.0054 (5)	-0.0039 (5)

*Geometric parameters (Å, °)*

O1—C2	1.3723 (17)	C13A—C14	1.4022 (19)
O1—C8A	1.3765 (18)	C13A—C17A	1.409 (2)
O2—C2	1.2077 (19)	C14—C15	1.383 (2)
C2—C3	1.4645 (19)	C15—C16	1.406 (2)
C3—C4	1.354 (2)	C16—C17	1.381 (2)
C3—C12	1.4698 (19)	C17A—C17	1.399 (2)
C4—C4A	1.4310 (19)	C4—H4	0.9500
C4A—C8A	1.3956 (19)	C5—H5	0.9500
C4A—C5	1.403 (2)	C7—H7	0.9500
C5—C6	1.384 (2)	C8—H8	0.9500
C6—C7	1.4083 (19)	C9—H9A	0.9800
C6—C9	1.504 (2)	C9—H9B	0.9800
C7—C8	1.381 (2)	C9—H9C	0.9800
C8A—C8	1.3897 (19)	C14—H14	0.9500
S11—C17A	1.7343 (14)	C15—H15	0.9500
S11—C12	1.7515 (15)	C16—H16	0.9500
C12—N13	1.3076 (18)	C17—H17	0.9500
N13—C13A	1.3836 (18)		
C2—O1—C8A	122.52 (11)	C14—C15—C16	121.07 (13)
O2—C2—O1	116.99 (12)	C17—C16—C15	121.37 (14)
O2—C2—C3	125.69 (14)	C17—C17A—C13A	121.53 (13)
O1—C2—C3	117.32 (13)	C17—C17A—S11	129.05 (12)
C4—C3—C2	119.91 (13)	C13A—C17A—S11	109.42 (11)
C4—C3—C12	120.78 (12)	C16—C17—C17A	117.68 (14)
C2—C3—C12	119.31 (13)	C3—C4—H4	119.2
C3—C4—C4A	121.57 (12)	C4A—C4—H4	119.2

C8A—C4A—C5	118.28 (13)	C6—C5—H5	119.2
C8A—C4A—C4	117.62 (13)	C4A—C5—H5	119.2
C5—C4A—C4	124.07 (12)	C8—C7—H7	119.0
C6—C5—C4A	121.65 (13)	C6—C7—H7	119.0
C5—C6—C7	117.94 (14)	C7—C8—H8	120.7
C5—C6—C9	121.11 (13)	C8A—C8—H8	120.7
C7—C6—C9	120.94 (13)	C6—C9—H9A	109.5
C8—C7—C6	122.00 (13)	C6—C9—H9B	109.5
O1—C8A—C8	117.40 (12)	H9A—C9—H9B	109.5
O1—C8A—C4A	121.01 (13)	C6—C9—H9C	109.5
C8—C8A—C4A	121.59 (14)	H9A—C9—H9C	109.5
C7—C8—C8A	118.53 (13)	H9B—C9—H9C	109.5
C17A—S11—C12	88.94 (7)	C15—C14—H14	120.8
N13—C12—C3	120.72 (13)	C13A—C14—H14	120.8
N13—C12—S11	115.94 (11)	C14—C15—H15	119.5
C3—C12—S11	123.35 (10)	C16—C15—H15	119.5
C12—N13—C13A	110.47 (12)	C17—C16—H16	119.3
N13—C13A—C14	124.90 (13)	C15—C16—H16	119.3
N13—C13A—C17A	115.23 (12)	C16—C17—H17	121.2
C14—C13A—C17A	119.86 (13)	C17A—C17—H17	121.2
C15—C14—C13A	118.48 (14)		
C8A—O1—C2—O2	179.74 (13)	C4A—C8A—C8—C7	0.5 (2)
C8A—O1—C2—C3	-0.6 (2)	C4—C3—C12—N13	-0.9 (2)
O2—C2—C3—C4	178.35 (15)	C2—C3—C12—N13	178.82 (13)
O1—C2—C3—C4	-1.3 (2)	C4—C3—C12—S11	178.79 (11)
O2—C2—C3—C12	-1.3 (2)	C2—C3—C12—S11	-1.53 (19)
O1—C2—C3—C12	178.98 (12)	C17A—S11—C12—N13	-0.22 (11)
C2—C3—C4—C4A	1.5 (2)	C17A—S11—C12—C3	-179.88 (12)
C12—C3—C4—C4A	-178.79 (13)	C3—C12—N13—C13A	179.37 (12)
C3—C4—C4A—C8A	0.1 (2)	S11—C12—N13—C13A	-0.31 (15)
C3—C4—C4A—C5	178.18 (13)	C12—N13—C13A—C14	-178.01 (13)
C8A—C4A—C5—C6	0.7 (2)	C12—N13—C13A—C17A	0.85 (17)
C4—C4A—C5—C6	-177.38 (13)	N13—C13A—C14—C15	178.86 (13)
C4A—C5—C6—C7	0.0 (2)	C17A—C13A—C14—C15	0.1 (2)
C4A—C5—C6—C9	179.11 (13)	C13A—C14—C15—C16	1.1 (2)
C5—C6—C7—C8	-0.4 (2)	C14—C15—C16—C17	-1.2 (2)
C9—C6—C7—C8	-179.54 (14)	N13—C13A—C17A—C17	179.81 (13)
C2—O1—C8A—C8	-177.01 (13)	C14—C13A—C17A—C17	-1.3 (2)
C2—O1—C8A—C4A	2.3 (2)	N13—C13A—C17A—S11	-1.00 (16)
C5—C4A—C8A—O1	179.81 (12)	C14—C13A—C17A—S11	177.92 (11)
C4—C4A—C8A—O1	-2.0 (2)	C12—S11—C17A—C17	179.76 (15)
C5—C4A—C8A—C8	-1.0 (2)	C12—S11—C17A—C13A	0.66 (11)
C4—C4A—C8A—C8	177.23 (13)	C15—C16—C17—C17A	0.0 (2)
C6—C7—C8—C8A	0.2 (2)	C13A—C17A—C17—C16	1.2 (2)
O1—C8A—C8—C7	179.80 (12)	S11—C17A—C17—C16	-177.78 (11)



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*Hydrogen-bond geometry* ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C17-H17\cdots O2^i$	0.95	2.43	3.3235 (19)	157

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Symmetry code: (i)  $-x+2, -y, -z+1$ .