Crystal structure of 2-(2,5-dimethoxyphenyl)-benzo[d]thiazole

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The title compound, C_{15}H_{13}NO_{2}S, was synthesized efficiently in the solid state by exploiting pepsin catalysis. The ring systems are nearly coplanar [interplanar angle of 5.38 (2)°] with an associated intramolecular S⋯C=C short contact of 2.7082 (4) Å. The packing involves C—H⋯C=C⋯C, C—H⋯C=C⋯C, and C=C⋯C=C contacts.

1. Chemical context

Although countless synthetic methods are widely available, new and more efficient procedures or approaches are always needed. Enzymes, as ‘green’ catalysts for modern organic synthesis, have attracted increased attention because they may provide alternative and sustainable processes, thus helping to minimize the release of hazardous substances into the environment (Witayakran & Ragauskas, 2009). Pepsin, a kind of hydrolase, belongs to the family of aspartic acid proteases and is involved in chemical digestion of protein (Cooper et al., 1990; Lin et al., 1989). Pepsin-catalysed aldol (and other) reactions have been developed (Li et al., 2010; He et al., 2016; Zongbo et al., 2017).

2-Aryl-benzothiazoles are a class of nitrogen-containing heterocyclic compounds that can be found in a variety of natural and synthetic compounds. In view of their biological and pharmacological characteristics, we are interested in developing synthetic strategies for heterocyclic ring systems containing a benzothiazole moiety; these have shown significant biological activity as novel antiviral and antimicrobial agents. (Azzam et al. 2017a,b, 2020a,b,c; 2021; Elgemeie et al., 2000a,b, 2020). The conventional synthesis of 2-aryl-benzothiazoles, which involves heating a mixture containing 2-aminothiophenol (1), is disadvantageous because 1 is extremely unstable in air and highly toxic. In a continuation of our recent research in developing ‘green’ and simple syntheses of novel heterocyclic compounds (Metwally et al., 2020, 2021a,b), we have now synthesized 2-(2,5-dimethoxyphenyl)benzo[d]thiazole (3) using pepsin as the ‘green’ catalytic reaction. Thus, a mixture of 1 and 2,5-dimethoxybenzaldehyde 2 was ground in a mortar with 0.05 g pepsin for 10 minutes, providing the desired product 3 in 97% yield. The nature of compound 3 was confirmed by spectroscopic analysis and by the single-crystal X-ray structure reported here.

2. Structural commentary

The structure of 3 is shown in Fig. 1. Molecular dimensions may be regarded as normal; a brief selection is presented in...
Table 1. Both ring systems are effectively planar (r.m.s. values of 0.01 Å for the benzothiazole and 0.004 Å for the phenyl ring, respectively), with an interplanar angle of 5.38(2)°. The approximate coplanarity leads to the short intramolecular contacts S1⋯O1 = 2.7082(4) and H16⋯N3 = 2.48 Å; the C16—H16⋯N3 angle is 101°.

3. Supramolecular features

There are no markedly short intermolecular contacts. One borderline ‘weak’ C—H⋯O hydrogen bond can be identified (Table 2), which links molecules via the c-glide operator x, −y + 1, −z + 1/2. This is reinforced by a C—H⋯π contact from H14 to the centroid of the phenyl ring (H14⋯Cg = 2.67 Å, C14—H14⋯Cg = 138°; Cg is the centroid of the C11–C16 ring). Additionally, the molecules are linked in pairs, related by c-axis translation, in which the benzothiazole ring system of one molecule lies opposite the phenyl ring of the other; the intercentroid distances are 3.5651(3) Å for benzo⋯phenyl, and 3.6022(3) Å for thiayole⋯phenyl (phenyl operator x, y, −1 + z). The net effect is to form a somewhat flattened herringbone pattern parallel to the c axis (Fig. 2; the π⋯π interactions are not shown explicitly). The contact C18⋯H18⋯N3 (Table 2), involving a methyl group, connects the chains in the third dimension via the operator −x + 1, −y + 1, −z.

![Figure 1](image1.png)

Figure 1
The molecule of 3 in the crystal. Ellipsoids represent 50% probability levels.

![Figure 2](image2.png)

Figure 2
Crystal packing of 3, viewed perpendicular to (100) in the region x ~ 0.25. Dashed lines indicate ‘weak’ C—H⋯O hydrogen bonds or C—H⋯π contacts. Atom labels correspond to the asymmetric unit.

Table 1
Selected geometric parameters (Å, °).

| Bond/Contact | Distance | Angle |
|--------------|----------|-------|
| S1—C7A       | 1.7327(6)|       |
| S1—C2        | 1.7642(5)|       |
| C2—N3        | 1.3064(7)|       |
| C7A—S1—C2    | 89.40(3) |       |
| N3—C3—C7A    | 115.25(5)|       |

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

| Bond/Contact | D—H—A | D—H | H—A | D—A | D—H···A |
|--------------|--------|-----|-----|-----|---------|
| C13—H13⋯O1i | 0.95   | 2.65| 3.5113(7)| 151 |
| C18—H18C⋯N3ii | 0.98   | 2.60| 3.4867(8)| 151 |

Symmetry codes: (i) x, −y + 1/2, z − 1/2; (ii) −x + 1, −y + 1, −z.
Crystal data

| Characteristic | Value |
|---------------|-------|
| Chemical formula | \(\text{C}_{15}\text{H}_{13}\text{NO}_{2}\text{S} \) |
| \(M_r\) | 271.32 |
| Crystal system, space group | Monoclinic, \(P2_1/c\) |
| Temperature (K) | 100 |
| \(a\), \(b\), \(c\) (Å) | 14.6666 (2), 13.8922 (2), 6.26063 (10) |
| \(\beta\) (°) | 100.1273 (14) |
| \(V\) (Å\(^3\)) | 1255.74 (3) |
| \(Z\) | 4 |
| Radiation type | Mo Kα |
| \(\mu\) (mm\(^-1\)) | 0.25 |
| Crystal size (mm) | 0.22 × 0.22 × 0.15 |

Diffractometer

| Description | Details |
|-------------|---------|
| Data collection | XtaLAB Synergy, HyPix |
| Absorption correction | Multi-scan (CrysAlis PRO; Rigaku OD, 2021) |
| \(T_{\text{min}}, T_{\text{max}}\) | 0.027, 1.00 |
| No. of measured, independent and observed \([I > 2\sigma(I)]\) reflections | 123768, 6759, 6156, 6123 |
| \(R_{\text{int}}\) | 0.023 |
| \(\sin \theta/\lambda_{\text{max}}\) (Å\(^{-1}\)) | 0.871 |

Refinement

| Description | Value |
|-------------|-------|
| \(R(F^2) > 2\sigma(F^2), S\) | 0.027, 0.084, 1.04 |
| No. of reflections | 6759 |
| No. of parameters | 174 |
| H-atom treatment | H-atom parameters constrained |
| \(\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}\) (e Å\(^{-3}\)) | 0.62, −0.19 |

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

4. Database survey

A search of the Cambridge Database (Version 2021.3.0; Groom et al., 2016) gave four hits for purely organic, neutral species in which one benzo[d][1]thiazole is bonded at its 2-position to an aromatic C6 ring with oxygen substituents at the ortho (2-) and meta (5-) positions. These were CEFWOB [Yousuf et al., 2012; 2-hydroxy, 5-methoxy; no S—O contact because of an intramolecular O—H···O hydrogen bond; interplanar angle 1.23 (9)°]; NOYSOM [Wang et al., 2019; 2,5-dimethoxy with an additional 4-(2-pyridyl) substituent; two independent molecules; S···O = 2.650, 2.715 Å; interplanar angles of 6.0, 5.5°]; UFAHUF [Chen, 2007; 2,4,5-trimethoxy; S···O = 2.671 Å, interplanar angle of 4.5 (2)°] and WACPUO (Sakai et al., 2016; 2-hydroxy, 5-methoxy with an additional 3-imidazole substituent; S···O = 2.695 Å, interplanar angle of 1.6°). Where not given in the original publications, these values were calculated using the CCDC program Mercury (Macrae et al., 2020).

5. Synthesis and crystallization

A mixture of o-aminophenol 1 (0.01 mol), 2,5-dimethoxybenzaldehyde 2 (0.01 mol) and pepsin (0.05 g) was ground together at room temperature for 10 min. The viscous mixture was poured onto ice–water; the solid that formed was filtered off and recrystallized from ethanol to give pale-yellow crystals of 3 in 97% yield, m.p. 414 K; IR (KBr, cm\(^{-1}\)): \(\nu_{\text{max}}\) 1581 (C==N); \(^1\)H NMR (DMSO-d\(_6\)): \(\delta = 3.82\) (s, 3H, OCH\(_3\)), 4.0 (s, 3H, OCH\(_3\)), 7.13–7.15 (m, 1H, Ar), 7.22 (d, 1H, J = 8.8 Hz, Ar), 7.41 (1H, J = 7.6 Hz, Ar), 7.51 (1H, J = 7.6 Hz, Ar), 7.96 (s, 1H, Ar), 8.07 (dd, 2H, J = 8.0 Hz, Ar), \(^{13}\)C NMR (DMSO-d\(_6\)): \(\delta = 56.0, 57.0, 112.5, 114.7, 119.1, 122.1, 122.2, 129.5, 126.7, 136.0, 151.9, 153.8, 154.4, 162.3\); \(m/z\) = 271 (M\(^+\), 100%), 238 (61.4%), 185 (27.6%), 136 (79.0%); Analysis: calculated for \(\text{C}_{15}\text{H}_{13}\text{NO}_{2}\text{S}\) (271.33) C 66.58, H 4.65, N 5.39, S 11.68%.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Methyl groups were refined as idealized rigid groups allowed to rotate but not tip (AFIX 137), with C—H = 0.98 Å, H—C—H = 109.5°. Other hydrogen atoms were included using a riding model starting from calculated positions (C—H\(_{\text{aromatic}}\) = 0.95 Å). The U(H) values were fixed at 1.5 or 1.2 \(\times\) \(U_{eq}\) of the parent carbon atoms for methyl and aromatic hydrogens, respectively.

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Crystal data

C_{15}H_{13}NO_{2}S  
Mr = 271.32  
Monoclinic, P2_{1}/c  
a = 14.6666 (2) Å  
b = 13.8922 (2) Å  
c = 6.26063 (10) Å  
\( \beta = 100.1273 (14)^{\circ} \)  
V = 1255.74 (3) Å^{3}  
Z = 4

\( F(000) = 568 \)  
\( D_{\text{r}} = 1.435 \text{ Mg m}^{-3} \)  
Mo K\( \alpha \) radiation, \( \lambda = 0.71073 \text{ Å} \)  
Cell parameters from 88513 reflections  
\( \theta = 2.8–38.4^\circ \)  
\( \mu = 0.25 \text{ mm}^{-1} \)  
\( T = 100 \text{ K} \)  
Block, colourless  
0.22 \times 0.22 \times 0.15 \text{ mm}

Data collection

XtaLAB Synergy, HyPix  
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)  
\( T_{\text{min}} = 0.927, T_{\text{max}} = 1.000 \)

Refinement

Refinement on \( F^{2} \)  
Least-squares matrix: full  
\( R[F^{2} > 2\sigma(F^{2})] = 0.027 \)  
w\( R(F^{2}) = 0.084 \)  
\( S = 1.03 \)  
6759 reflections  
174 parameters  
0 restraints  
Primary atom site location: dual  
Hydrogen site location: inferred from neighbouring sites  
H-atom parameters constrained  
\( w = 1/[\sigma^{2}(F_{o}^{2}) + (0.0489P)^{2} + 0.2455P] \)  
where \( P = (F_{o}^{2} + 2F_{c}^{2})/3 \)  
\( \Delta\sigma_{\text{max}} = 0.001 \)  
\( \Delta\rho_{\text{max}} = 0.62 \text{ e Å}^{-3} \)  
\( \Delta\rho_{\text{min}} = -0.19 \text{ e Å}^{-3} \)
**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)

\[-0.2606 (0.0025) x + 11.3686 (0.0009) y - 3.5208 (0.0006) z = 4.3561 (0.0009)\]

\[* -0.0113 (0.0003) S1 * 0.0122 (0.0004) C2 * 0.0073 (0.0004) N3 * -0.0022 (0.0005) C3A * -0.0178 (0.0004) C4 * 0.0029 (0.0005) C5 * 0.0151 (0.0005) C6 * -0.0033 (0.0005) C7 * -0.0029 (0.0005) C7A\]

Rms deviation of fitted atoms = 0.0101

-1.6150 (0.0031) x + 11.4261 (0.0017) y - 3.3180 (0.0011) z = 4.1087 (0.0012)

Angle to previous plane (with approximate esd) = 5.381 ( 0.023 )

\[* -0.0035 (0.0003) C11 * 0.0058 (0.0004) C12 * -0.0025 (0.0004) C13 * -0.0031 (0.0004) C14 * 0.0054 (0.0004) C15 * -0.0020 (0.0004) C16 * -0.0003 (0.0008) O1 0.0801 (0.0011) C17 0.0285 (0.0008) O2 0.1636 (0.0010) C18\]

Rms deviation of fitted atoms = 0.0040

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

| Atom | x   | y   | z   | Uiso* Ueq |
|------|-----|-----|-----|-----------|
| S1   | 0.13465 (2) | 0.49165 (2) | 0.34350 (2) | 0.01451 (4) |
| C2   | 0.24674 (4) | 0.47726 (4) | 0.28209 (8) | 0.01188 (8) |
| N3   | 0.31247 (3) | 0.52002 (3) | 0.41668 (7) | 0.01347 (7) |
| C3A  | 0.27752 (4) | 0.56826 (4) | 0.57771 (8) | 0.01360 (8) |
| C4   | 0.33134 (4) | 0.62033 (4) | 0.74630 (9) | 0.01695 (9) |
| H4   | 0.396755 | 0.623486 | 0.758537 | 0.020* |
| C5   | 0.28692 (5) | 0.66707 (4) | 0.89466 (9) | 0.01963 (10) |
| H5   | 0.322334 | 0.703577 | 1.008219 | 0.024* |
| C6   | 0.19042 (5) | 0.66133 (5) | 0.87976 (10) | 0.02095 (11) |
| C7   | 0.13610 (5) | 0.60805 (4) | 0.71696 (10) | 0.01924 (10) |
| H7   | 0.071002 | 0.603042 | 0.709129 | 0.023* |
| C7A  | 0.18087 (4) | 0.56196 (4) | 0.56475 (9) | 0.01468 (8) |
| C11  | 0.26660 (3) | 0.42388 (3) | 0.09270 (8) | 0.01131 (7) |
| C12  | 0.19879 (3) | 0.37320 (4) | -0.05164 (8) | 0.01179 (8) |
| C13  | 0.22303 (4) | 0.32478 (4) | -0.22766 (8) | 0.01295 (8) |
| H13  | 0.177114 | 0.290066 | -0.323243 | 0.016* |
| C14  | 0.31402 (4) | 0.32648 (4) | -0.26592 (8) | 0.01283 (8) |
| H14  | 0.329903 | 0.293120 | -0.386674 | 0.015* |
| C15  | 0.38132 (3) | 0.37743 (4) | -0.12577 (8) | 0.01215 (8) |
| C16  | 0.35765 (4) | 0.42522 (4) | 0.05254 (8) | 0.01240 (8) |
| H16  | 0.404004 | 0.459248 | 0.148423 | 0.015* |
| O1   | 0.11061 (3) | 0.37347 (3) | -0.00594 (7) | 0.01587 (7) |
| C17  | 0.04056 (4) | 0.32676 (5) | -0.15693 (10) | 0.01998 (10) |
| H17A | 0.036645 | 0.356648 | -0.300050 | 0.030* |
| H17B | -0.019153 | 0.333071 | -0.108453 | 0.030* |
| H17C | 0.056005 | 0.258430 | -0.166186 | 0.030* |
| O2   | 0.47191 (3) | 0.38509 (3) | -0.15047 (7) | 0.01691 (8) |
| C18  | 0.49545 (4) | 0.34434 (5) | -0.34295 (10) | 0.01952 (10) |
| H18A | 0.454902 | 0.371139 | -0.470203 | 0.029* |
Atomic displacement parameters (Å²)

|    | U¹¹ | U¹² | U¹³ | U¹² | U¹³ | U¹³ |
|----|-----|-----|-----|-----|-----|-----|
| S1 | 0.01415 (6) | 0.01602 (6) | 0.01373 (6) | 0.00148 (4) | 0.00352 (4) | −0.00175 (4) |
| C2 | 0.01436 (18) | 0.01083 (17) | 0.01076 (17) | 0.00014 (14) | 0.00307 (14) | 0.00011 (13) |
| N3 | 0.01641 (18) | 0.01302 (17) | 0.01139 (16) | −0.00157 (13) | 0.00354 (13) | −0.00175 (13) |
| C3A | 0.0193 (2) | 0.01086 (18) | 0.01120 (17) | −0.00042 (15) | 0.00426 (15) | −0.00038 (14) |
| C4 | 0.0236 (2) | 0.0142 (2) | 0.01336 (19) | −0.00312 (17) | 0.00403 (17) | −0.00254 (15) |
| C5 | 0.0314 (3) | 0.0140 (2) | 0.0142 (2) | −0.00201 (19) | 0.00606 (19) | −0.00317 (16) |
| C6 | 0.0321 (3) | 0.0161 (2) | 0.0167 (2) | 0.0025 (2) | 0.0097 (2) | −0.00306 (17) |
| C7 | 0.0239 (2) | 0.0180 (2) | 0.0176 (2) | 0.00356 (19) | 0.00846 (19) | −0.00219 (18) |
| C7A | 0.0190 (2) | 0.01267 (19) | 0.01312 (18) | 0.00175 (15) | 0.00489 (15) | −0.00059 (15) |
| C11 | 0.01340 (17) | 0.01037 (17) | 0.01029 (16) | 0.00010 (13) | 0.00241 (13) | −0.00034 (13) |
| C12 | 0.01206 (17) | 0.01134 (18) | 0.01187 (17) | 0.00064 (13) | 0.00182 (14) | −0.00025 (13) |
| C13 | 0.01318 (18) | 0.01267 (18) | 0.01268 (18) | 0.00015 (14) | 0.00138 (14) | −0.00222 (14) |
| C14 | 0.01418 (18) | 0.01223 (18) | 0.01217 (18) | 0.00012 (14) | 0.00257 (14) | −0.00183 (14) |
| C15 | 0.01276 (18) | 0.01177 (18) | 0.01232 (17) | −0.00073 (14) | 0.00333 (14) | −0.00073 (14) |
| C16 | 0.01365 (18) | 0.01212 (18) | 0.01161 (17) | −0.00128 (14) | 0.00273 (14) | −0.00118 (14) |
| O1 | 0.01170 (15) | 0.01927 (18) | 0.01672 (16) | −0.00048 (12) | 0.00270 (12) | −0.00411 (13) |
| C17 | 0.01240 (19) | 0.0272 (3) | 0.0192 (2) | −0.00078 (18) | −0.00011 (17) | 0.0035 (2) |
| O2 | 0.01379 (16) | 0.02132 (19) | 0.01681 (17) | −0.00358 (13) | 0.00599 (13) | −0.00600 (14) |
| C18 | 0.0175 (2) | 0.0238 (3) | 0.0190 (2) | −0.00194 (19) | 0.00813 (18) | −0.00580 (19) |

Geometric parameters (Å, °)

|    |    |    |    |    |    |    |
|----|----|----|----|----|----|----|
| S1 | C7A | 1.7327 (6) | C15—O2 | 1.3686 (6) |
| S1 | C2  | 1.7642 (5) | C15—C16 | 1.3941 (7) |
| C2 | N3  | 1.3064 (7) | C1O—C17 | 1.4243 (7) |
| C2 | C11 | 1.4704 (7) | C2—C18 | 1.4276 (7) |
| N3 | C3A | 1.3820 (7) | C4—H4 | 0.9500 |
| C3A | C4  | 1.4027 (8) | C5—H5 | 0.9500 |
| C3A | C7A | 1.4082 (8) | C6—H6 | 0.9500 |
| C4 | C5  | 1.3862 (8) | C7—H7 | 0.9500 |
| C5 | C6  | 1.4043 (10) | C13—H13 | 0.9500 |
| C6 | C7  | 1.3912 (9) | C14—H14 | 0.9500 |
| C7 | C7A | 1.4032 (8) | C16—H16 | 0.9500 |
| C11 | C16 | 1.4017 (7) | C17—H17A | 0.9800 |
| C11 | C12 | 1.4091 (7) | C17—H17B | 0.9800 |
| C12 | O1  | 1.3728 (6) | C17—H17C | 0.9800 |
| C12 | C13 | 1.3896 (7) | C18—H18A | 0.9800 |
| C13 | C14 | 1.3969 (7) | C18—H18B | 0.9800 |
| C14 | C15 | 1.3930 (7) | C18—H18C | 0.9800 |
| C7A—S1—C2 | 89.40 (3) | C15—O2—C18 | 116.63 (4) |
| N3—C2—C11 | 121.40 (5) | C5—C4—H4 | 120.7 |

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sup-3
N3—C2—S1 114.72 (4) C3A—C4—H4 120.7
C11—C2—S1 123.86 (4) C4—C5—H5 119.5
C2—N3—C3A 111.39 (5) C6—C5—H5 119.5
N3—C3A—C4 124.55 (5) C7—C6—H6 119.4
N3—C3A—C7A 115.25 (5) C5—C6—H6 119.4
C4—C3A—C7A 120.20 (5) C6—C7—H7 121.2
C5—C4—C3A 118.50 (6) C7A—C7—H7 121.2
C4—C5—C6 121.05 (6) C12—C13—H13 119.6
C7—C6—C5 121.30 (5) C14—C13—H13 119.6
C6—C7—C7A 117.66 (6) C15—C14—H14 120.2
C7—C7A—S1 109.23 (4) C11—C16—H16 119.5
C3A—C7A—S1 116.71 (4) C11—C16—H16 119.5
C16—C11—C12 123.47 (4) H17A—C17—H17B 109.5
C12—C11—C2 117.91 (4) O1—C12—C13 123.25 (5)
C12—C11—C16 120.02 (5) C16—C11—C2 123.26 (5)
O1—C12—C13 116.61 (4) O1—C17—H17A 109.5
O1—C12—C11 118.76 (4) O1—C17—H17B 109.5
C16—C11—C12 120.88 (5) C12—C11—C16 118.76 (4)
C15—C14—C13 119.54 (5) C12—C11—C12 116.61 (4)
O2—C15—C14 124.24 (5) O2—C18—H18A 109.5
O2—C15—C16 115.90 (4) O2—C18—H18B 109.5
C14—C15—C16 119.85 (5) O2—C18—H18C 109.5
C13—C12—O1—C17 −4.35 (8)
C13—C12—C11—C12 −3.97 (7)
C13—C12—C11—C16 178.93 (5)
C13—C12—C11—C16 −178.93 (5)
C13—C12—O1—C17 176.78 (5)
C13—C12—C11—C12 −178.93 (5)
C13—C12—C11—C16 176.78 (5)
C13—C12—C11—C16 −178.93 (5)
C13—C12—O1—C17 −4.35 (8)
C13—C12—C11—C16 176.78 (5)
C13—C12—C11—C16 −178.93 (5)
C13—C12—O1—C17 176.78 (5)
C13—C12—C11—C16 176.78 (5)
C13—C12—C11—C16 −178.93 (5)
Hydrogen-bond geometry (Å, °)

| D—H···A   | D—H | H···A | D···A      | D—H···A |
|-----------|------|-------|------------|---------|
| C13—H13···O1\(^i\) | 0.95 | 2.65  | 3.5113 (7) | 151     |
| C18—H18C···N3\(^ii\) | 0.98 | 2.60  | 3.4867 (8) | 151     |

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