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# Crystal structures of two polymorphs of tixocortol pivalate

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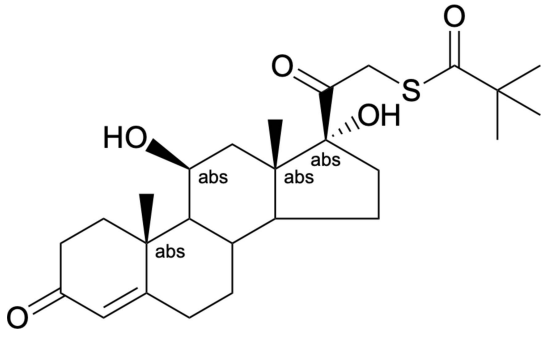
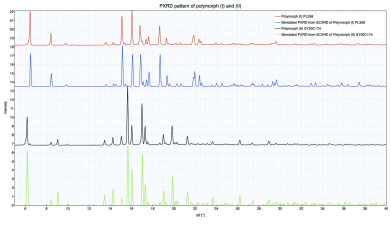
Two polymorphs, (**I**) and (**II**), of (*S*)-{2-[(8*S*,9*S*,10*R*,11*S*,13*S*,14*S*,17*R*)-11,17-dihydroxy-10,13-dimethyl-3-oxo-2,6,7,8,9,11,12,14,15,16-decahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl]-2-oxoethyl} 2,2-dimethylpropanethioate, $C_{26}H_{38}O_5S$, have been identified. They are orthorhombic, non-centrosymmetric ($P2_12_12_1$). The structures display layers of molecules conected *via* O—H···O hydrogen bonds along the *b*-axis direction in polymorph (**I**) and along the *c*-axis direction in polymorph (**II**). The structure of (**II**) exhibits disorder of the main molecule.

## 1. Chemical context

Tixocortol pivalate, also named Pivalone®, is a corticosteroid with local and topical anti-inflammatory activity (Davies *et al.*, 1981; Jezequel *et al.*, 1979; Liddle *et al.*, 1960; Mazauric & Alligier, 1978; Nugent *et al.*, 1963; Uphill, 1981) equal to that of hydrocortisone. As a corticosteroid, Tixocortol pivalate is used topically to relieve contact allergies and is also frequently recommended as a screening test for class A corticosteroids (Bircher *et al.*, 1995; Burden & Beck, 1992; Lauerma, 1991; Bouley, 2013). Surprisingly, the structure of tixocortol pivalate has never been determined. It was therefore of interest to obtain two polymorphs, (**I**) and (**II**), of the title compound prepared by total enantio-selective synthesis.



## 2. Structural commentary

The presence of two polymorphs was confirmed by powder X-ray diffraction (PXRD) and the structures were determined by single crystal X-ray diffraction (SCXRD). The absolute configuration of its seven asymmetric carbons was established. Both polymorphs of the title compound consist of a (*S*)-{2-[(8*S*,9*S*,10*R*,11*S*,13*S*,14*S*,17*R*)-11,17-dihydroxy-10,13-dimethyl-3-oxo-2,6,7,8,9,11,12,14,15,16-decahydro-1*H*-cyclopenta[*a*]-

**Table 1**
Ring puckering parameters.

| Compound | PL358 (**I**) | SY20C174 (**II**) |
|---|---|---|
| C8–C12 | $Q2$ = 0.4847 (18) Å | $Q2$ = 0.441 (5) Å |
| Envelope conformation | $\varphi2$ = 39.4 (2)° | $\varphi2$ = 41.4 (6)° |
| C9/C14–C17/C10 | $Q$ = 0.5519 (17) Å \| Θ = 9.45 (18) ° \| $\varphi2$ = 53.2 (11)° | $Q$ = 0.556 (4) Å \| Θ = 13.4 (4) ° \| $\varphi2$ = 37 (2)° |
| Chair conformation | $Q2$ = 0.0908 (17) Å \| $Q3$ = 54.4444 (17) Å \| $\varphi2$ = 53.2 (11)° | $Q2$ = 0.128 (4) Å \| $Q3$ = 0.541 (4) Å \| $\varphi2$ = 37 (2)° |
| C16/C18–C21/C17 | $Q$ = 0.5450 (17) Å \| Θ = 175.11 (18) ° \| $\varphi2$ = 170 (2)° | $Q$ = 0.538 (4) Å \| Θ = 173.2 (4) ° \| $\varphi2$ = 196 (4)° |
| Chair conformation | $Q2$ = 0.0457 (17) Å \| $Q3$ = −0.5431 (17) Å \| $\varphi2$ = 170 (2)° | $Q2$ = 0.0065 (4) Å \| $Q3$ = −0.534 (4) Å \| $\varphi2$ = 196 (4)° |
| C18/C19/C23–C26 | $Q$ = 0.4724 (18) Å \| Θ = 52.7 (2) ° \| $\varphi2$ = 266.8 (3)° | $Q$ = 0.454 (4) Å \| Θ = 55.6 (5) ° \| $\varphi2$ = 281.9 (7)° |
| Half-chair conformation | $Q2$ = 0.3756 (18) Å \| $Q3$ = 0.2865 (18) Å \| $\varphi2$ = 266.8 (3)° | $Q2$ = 0.375 (4) Å \| $Q3$ = 0.256 (4) Å \| $\varphi2$ = 281.9 (7)° |

phenanthren-17-yl]-2-oxoethyl} 2,2-dimethylpropanethioate molecule in the asymmetric unit (Figs. 1 and 2). The general shape of the molecule is strongly influenced by the conformation of one five-membered ring and three six-membered rings. In both polymorphs (Table 1), the five-membered ring (C8–C12) adopts an envelope form, both central six-membered rings (C9/C14–C17/C10 and C16/C18–C21/C17) adopt chair conformations and the six-membered ring with the double bond (C18/C19/C23–C26) adopts a half-chair conformation (Cremer & Pople, 1975). The superposition of the molecules, with the Automatic Molecule Overlay feature of *Mercury* (Macrae *et al.*, 2020), results in an r.m.s.d. of 0.829 and a maximum deviation of 2.545 Å if no flexibility is allowed and in values of 0.336 and 0.856, respectively, if flexibility is allowed. The main difference is on the dimethyl-sulfanyl-propanone group whose position is imposed by crystal packing.

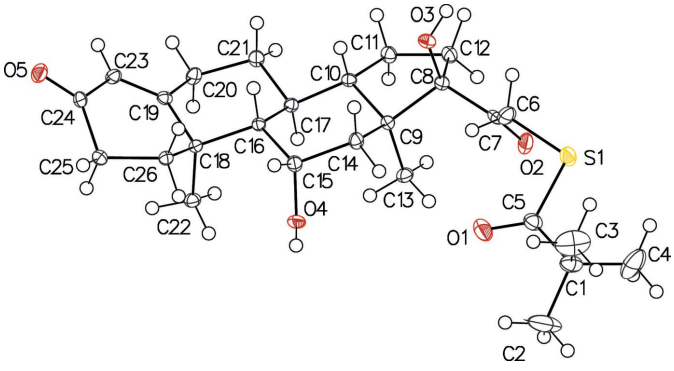



**Figure 1**
*ORTEP* view of polymorph (**I**). Displacement ellipsoids are drawn at the 50% probability level.

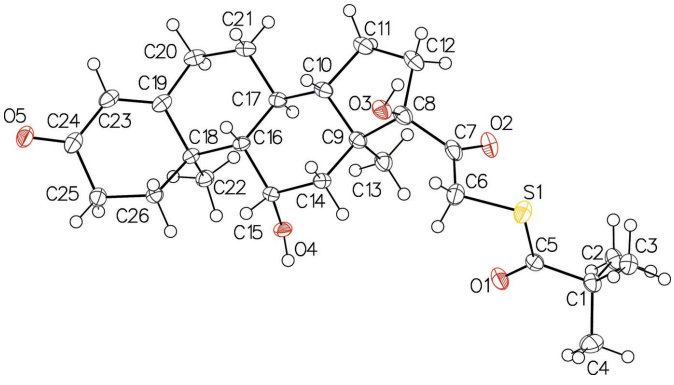



**Figure 2**
*ORTEP* view of polymorph (**II**). Displacement ellipsoids are drawn at the 30% probability level. The minor component of the disorder is omitted for clarity.

**Table 2**
Hydrogen-bond geometry (Å, °) for PL358 (**I**).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O5$^{i}$ | 0.84 | 2.07 | 2.9021 (17) | 169 |

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

**Table 3**
Hydrogen-bond geometry (Å, °) for SY20C174 (**II**).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O5$^{i}$ | 0.84 | 1.96 | 2.802 (4) | 175 |

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

## 3. Supramolecular features

The crystal packing in both structures is stabilized by one O—H···O hydrogen bond (Figs. 3 and 4, Tables 2 and 3) producing layers along (010) for polymorph (**I**) (PL358) and along (001) for polymorph (**II**) (SY20C174). The geometry of these interactions indicates that these are strong hydrogen bonds.

## 4. Morphology prediction

In both polymorphs, it was observed that the same type of hydrogen bonds plays a dominant role in the formation of

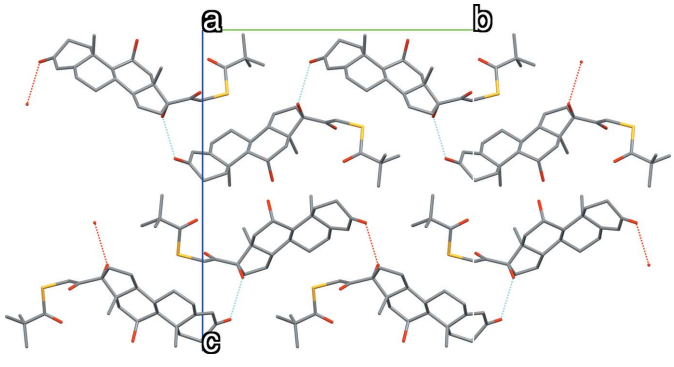



**Figure 3**
View of the hydrogen bond-network in polymorph (**I**).

Table 4
Cell parameters determined from SCXRD and PXRD at different temperatures.

| Compound | PL358 | PL358 | PL358 | SY20C174 | SY20C174 | SY20C174 |
|---|---|---|---|---|---|---|
| XRD measurement | SCXRD | SCXRD | PXRD | SCXRD | SCXRD | PXRD |
| Temperature | 110 K | 295 K | 295 K | 100 K | 298 K | 295 K |
| Space group | $P2_12_12_1$ | $P2_12_12_1$ | $P2_12_12_1$ | $P2_12_12_1$ | $P2_12_12_1$ | $P2_12_12_1$ |
| *a* | 6.4201 (2) | 6.467 (5) | 6.4775 (2) | 6.0146 (2) | 6.157 (9) | 6.1573 (2) |
| *b* | 17.6239 (7) | 17.887 (12) | 17.9583 (7) | 19.2817 (7) | 19.46 (3) | 19.4684 (7) |
| *c* | 20.8997 (8) | 20.897 (15) | 20.9335 (7) | 20.9887 (7) | 20.92 (3) | 20.8859 (9) |
| Volume | 2364.7 (1) | 2417 (5) | 2435.1 (1) | 2434.1 (1) | 2508 (11) | 2503.7 (2) |

hydrogen-bonded networks. However, the arrangements of molecules in the crystal packing of polymorphs (**I**) and (**II**) are different. The different arrangements can also be seen in the external shape and size of the crystals. The theoretical crystal habits of polymorphs (**I**) and (**II**) were predicted based on the BFDH model with *Mercury* (Fig. 5). The morphologies of Pivalone polymorphs (**I**) and (**II**) display significant differences in their main crystal dimension.

## 5. Synthesis and crystallization

Tixocortol pivalate (Fig. 6) has been produced as follows (Bouley, 2013): in a dry inerted flask, cesium thiopivalate (620 g, 2.48 mol) and tetrahydrofuran (1460 mL) are stirred at room temperature. A hydrocortisone mesylate (995 g, 2.26 mol) solution in THF (4600 mL) is added in 1 h below 293 K. After 16 h of stirring, the reaction mixture is cooled below 283 K and water (12320 mL) is added. After addition, the reaction mixture is stirred for approximately 2 h. The precipitate is filtered and washed with water (10 × 820 mL). After drying under vacuum at 323 K for one night, the product is isolated as a white powder (yield 93%, purity by HPLC 98.5%).

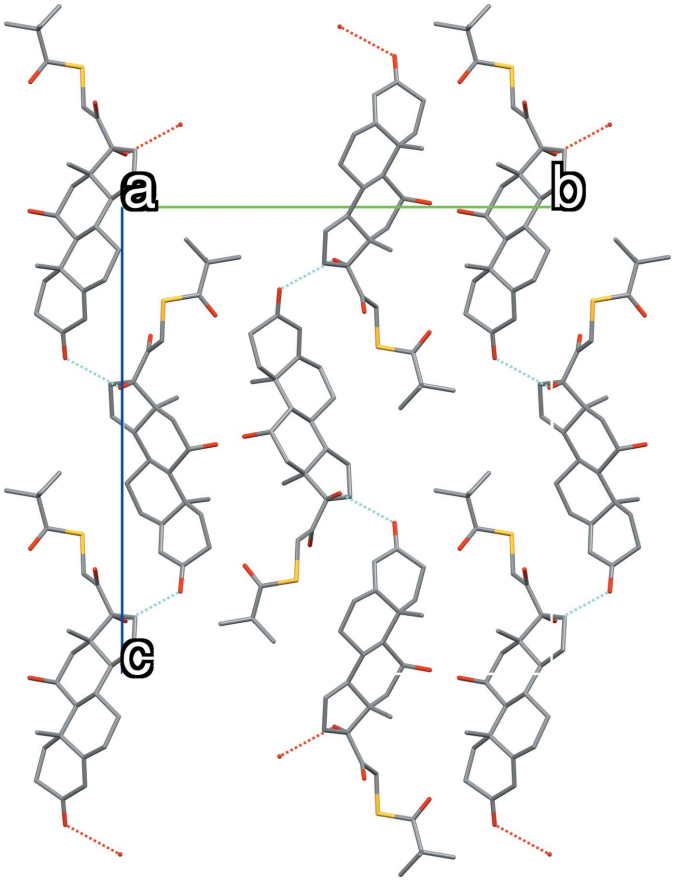



Figure 4
View of the hydrogen-bond network in polymorph (**II**).

## 6. Powder X-ray diffraction (PXRD)

Analyses were performed at room temperature from $2\theta = 3$ to 50° with an increasing step size of 0.02° and a count time of 120 s. The X-ray powder diffraction patterns were registered in transmission mode unless mentioned otherwise. The samples (few milligrams) are introduced without being crushed in 1 mm diameter glass capillaries to avoid preferential orientation. The capillaries are sealed to avoid contact with air. The analysis is performed in transmission mode by using a focusing X-ray mirror with divergence slits and anti-

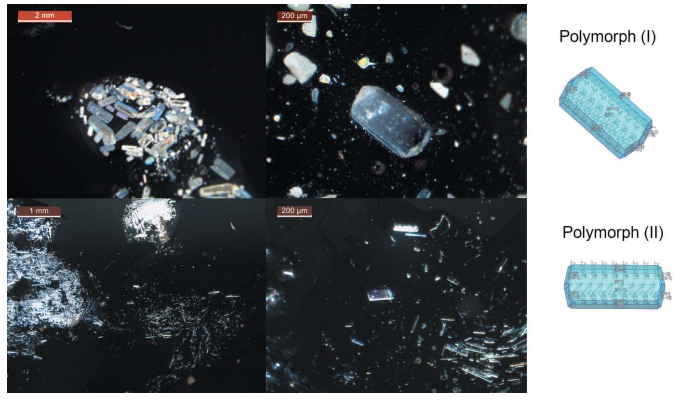



Figure 5
View of the crystal morphology of polymorph (**I**) (top) and (**II**) (bottom).

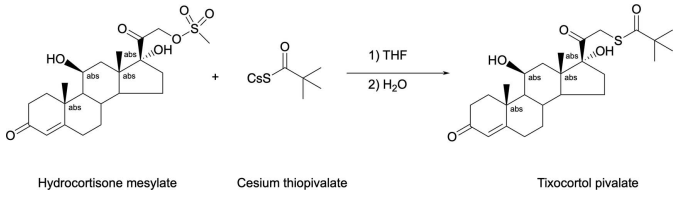



Figure 6
Reaction scheme for the synthesis of tixocortol pivalate.

**Table 5**
Experimental details.

| | PL358 (**I**) | SY20C174 (**II**) |
|---|---|---|
| Crystal data | | |
| Chemical formula | $C_{26}H_{38}O_5S$ | $C_{26}H_{38}O_5S$ |
| $M_r$ | 462.62 | 462.62 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ | Orthorhombic, $P2_12_12_1$ |
| Temperature (K) | 110 | 100 |
| $a$, $b$, $c$ (Å) | 6.4201 (2), 17.6239 (7), 20.8997 (8) | 6.0146 (2), 19.2817 (7), 20.9887 (7) |
| $V$ (Å$^3$) | 2364.74 (15) | 2434.10 (14) |
| $Z$ | 4 | 4 |
| Radiation type | Mo $K\alpha$ | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.17 | 1.46 |
| Crystal size (mm) | 0.46 × 0.25 × 0.24 | 0.18 × 0.06 × 0.05 |
| Data collection | | |
| Diffractometer | Nonius Kappa APEXII | Bruker D8 Venture |
| Absorption correction | Multi-scan (*SADABS*; Krause *et al.*, 2015) | Multi-scan (*SADABS*; Krause *et al.*, 2015) |
| $T_{min}$, $T_{max}$ | 0.912, 0.958 | 0.707, 0.862 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 74424, 5424, 5180 | 30900, 4303, 3803 |
| $R_{int}$ | 0.034 | 0.102 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.028, 0.072, 1.04 | 0.055, 0.130, 1.07 |
| No. of reflections | 5424 | 4303 |
| No. of parameters | 296 | 329 |
| No. of restraints | 0 | 16 |
| H-atom treatment | H-atom parameters constrained | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.26, −0.23 | 0.26, −0.39 |
| Absolute structure | Flack $x$ determined using 2176 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) | Flack $x$ obtained from refinement |
| Absolute structure parameter | 0.027 (13) | 0.11 (4) |

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

scatter slits (aperture 0.5°), on an Empyrean diffractometer from PANalytical Company (PANalytical, 2011) equipped with a copper anticathode tube (wavelength λ $K\alpha1$ = 1.54060 Å/$K\alpha2$ = 1.54443 Å) and with a PIXcel 1D detector with anti-scatter slits of 7.5 mm. The calibration of the analytical instrument is checked before each analytical batch according to quality systems.

Unit-cell parameters were obtained using indexing methods included in *ITO* (Visser, 1969) or *DICVOL* (Boultif & Louër, 2004). Le Bail (Le Bail, 1988) refinement was performed by using *JANA2006* (Petříček *et al.*, 2014) with the most plausible unit cell. The cell parameters found at room temperature were compared to those found from single crystal at different temperatures (Table 4). The cell parameters at low temperature and at ambient temperature found from single crystal and from powder diffraction are similar, confirming that no phase change occurs with different temperatures. The simulated PXRD patterns were calculated (Palmer, 2015) from SCXRD with cell parameters obtained at room temperature (Fig. 7).

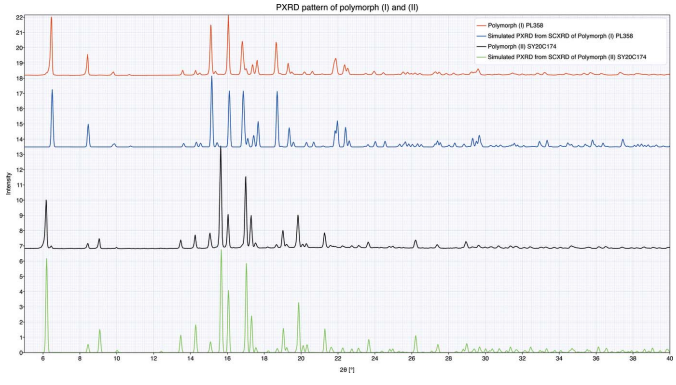



**Figure 7**
PXRD patterns of polymorphs (**I**) and (**II**) and their simulated patterns from the SCXRD study at room temperature.

## 7. Structure solution and refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. The dimethyl-sulfanyl-propanone group was found to be disordered over two positions 77 (1)%/23 (1)% in polymotph (**II**). The SAME (Sheldrick, 2015*b*) restraint was employed for the minor disordered part to maintain a reasonable model. All non-hydrogen atoms were refined anisotropically, except the minor disorder component. Hydrogen-atom positions were calculated geometrically and refined using the riding model. All H atoms, on carbon atoms, were placed at calculated positions using a riding model with C—H = 0.95 Å (aromatic), 0.99 Å (methylene) or 1 Å (methine) with $U_{iso}$(H) = 1.2$U_{eq}$(C). H atoms on oxygen atoms were located in difference-Fourier maps. Their positional parameters were refined as an idealized OH group (AFIX

147), (Sheldrick, 2015*b*) with $U_{iso}(H) = 1.5U_{eq}(O)$. The TWIN/BASF instruction was used to refine the Flack parameter.

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# supporting information

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## Crystal structures of two polymorphs of tixocortol pivalate

**Yoann Rousselin, Sylvie Yolka and Alexandre Clavel**

### Computing details

For both structures, data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: ShelXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

**(*S*)-{2-[(8*S*,9*S*,10*R*,11*S*,13*S*,14*S*,17*R*)-11,17-Dihydroxy-10,13-dimethyl-3-oxo-2,6,7,8,9,11,12,14,15,16-decahydro-1*H*-cyclopenta[a]phenanthren-17-yl]-2-oxoethyl} 2,2-dimethylpropanethioate (PL358)**

*Crystal data*

$C_{26}H_{38}O_5S$
$M_r$ = 462.62
Orthorhombic, $P2_12_12_1$
$a$ = 6.4201 (2) Å
$b$ = 17.6239 (7) Å
$c$ = 20.8997 (8) Å
$V$ = 2364.74 (15) Å$^3$
$Z$ = 4
$F(000)$ = 1000
$D_x$ = 1.299 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9539 reflections
$\theta$ = 2.3–27.4°
$\mu$ = 0.17 mm$^{-1}$
$T$ = 110 K
Prism, clear light colourless
0.46 × 0.25 × 0.24 mm

*Data collection*

Nonius Kappa APEXII diffractometer
Radiation source: X-ray tube, Siemens KFF Mo 2K-180
Graphite monochromator
Detector resolution: 512 x 512 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans′
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.912, $T_{max}$ = 0.958
74424 measured reflections
5424 independent reflections
5180 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 3.0°
$h$ = −8→8
$k$ = −22→22
$l$ = −27→27

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.072
$S$ = 1.04
5424 reflections
296 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2 + 0.6195P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

Absolute structure: Flack *x* determined using 2176 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.027 (13)

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.57981 (8) | 0.59132 (2) | 0.30170 (2) | 0.02260 (11) |
| O3 | 0.6410 (2) | 0.35175 (7) | 0.23101 (6) | 0.0154 (2) |
| H3 | 0.671037 | 0.360570 | 0.192609 | 0.023* |
| O5 | 0.2936 (2) | −0.10124 (7) | 0.40054 (6) | 0.0186 (3) |
| O2 | 0.9568 (2) | 0.49062 (7) | 0.30543 (6) | 0.0219 (3) |
| O4 | 0.6656 (3) | 0.25785 (7) | 0.46752 (6) | 0.0243 (3) |
| H4 | 0.576170 | 0.276912 | 0.492065 | 0.036* |
| O1 | 0.4852 (3) | 0.52261 (8) | 0.40864 (7) | 0.0352 (4) |
| C10 | 0.8689 (2) | 0.24334 (9) | 0.30097 (7) | 0.0105 (3) |
| H10 | 0.757227 | 0.229873 | 0.269614 | 0.013* |
| C16 | 0.6649 (2) | 0.16686 (9) | 0.37858 (7) | 0.0101 (3) |
| H16 | 0.572073 | 0.153973 | 0.341722 | 0.012* |
| C24 | 0.4122 (3) | −0.04919 (9) | 0.41447 (8) | 0.0133 (3) |
| C22 | 0.7966 (3) | 0.09961 (10) | 0.48265 (8) | 0.0139 (3) |
| H22A | 0.939136 | 0.110305 | 0.468479 | 0.021* |
| H22B | 0.793474 | 0.050904 | 0.505250 | 0.021* |
| H22C | 0.749690 | 0.139990 | 0.511490 | 0.021* |
| C7 | 0.8049 (3) | 0.45608 (10) | 0.28770 (8) | 0.0152 (3) |
| C18 | 0.6505 (2) | 0.09589 (9) | 0.42376 (7) | 0.0101 (3) |
| C17 | 0.8818 (2) | 0.17768 (9) | 0.34806 (7) | 0.0108 (3) |
| H17 | 0.986748 | 0.189289 | 0.382019 | 0.013* |
| C23 | 0.6075 (3) | −0.03662 (9) | 0.37971 (8) | 0.0133 (3) |
| H23 | 0.657156 | −0.075699 | 0.352367 | 0.016* |
| C26 | 0.4233 (3) | 0.08633 (9) | 0.44563 (7) | 0.0116 (3) |
| H26A | 0.395372 | 0.122300 | 0.480995 | 0.014* |
| H26B | 0.329792 | 0.099703 | 0.409691 | 0.014* |
| C21 | 0.9454 (3) | 0.10574 (9) | 0.31190 (8) | 0.0140 (3) |
| H21A | 0.855784 | 0.100246 | 0.273627 | 0.017* |
| H21B | 1.091004 | 0.111287 | 0.297053 | 0.017* |
| C20 | 0.9280 (3) | 0.03384 (9) | 0.35248 (8) | 0.0138 (3) |
| H20A | 0.950163 | −0.011075 | 0.324867 | 0.017* |
| H20B | 1.038671 | 0.034177 | 0.385458 | 0.017* |
| C9 | 0.8031 (3) | 0.31896 (9) | 0.33168 (7) | 0.0110 (3) |
| C11 | 1.0623 (3) | 0.26354 (9) | 0.26139 (8) | 0.0142 (3) |
| H11A | 1.076384 | 0.229298 | 0.224088 | 0.017* |

| | | | | |
|---|---|---|---|---|
| H11B | 1.190147 | 0.260105 | 0.287716 | 0.017* |
| C1 | 0.4236 (3) | 0.65833 (11) | 0.41260 (8) | 0.0219 (4) |
| C19 | 0.7196 (2) | 0.02742 (9) | 0.38462 (8) | 0.0112 (3) |
| C15 | 0.5701 (3) | 0.24038 (9) | 0.40657 (8) | 0.0142 (3) |
| H15 | 0.418893 | 0.230653 | 0.414522 | 0.017* |
| C5 | 0.4924 (3) | 0.58328 (10) | 0.38238 (9) | 0.0184 (3) |
| C14 | 0.5861 (3) | 0.30893 (9) | 0.36064 (8) | 0.0134 (3) |
| H14A | 0.483971 | 0.302354 | 0.325584 | 0.016* |
| H14B | 0.548391 | 0.355646 | 0.384264 | 0.016* |
| C25 | 0.3715 (3) | 0.00567 (9) | 0.46830 (8) | 0.0138 (3) |
| H25A | 0.458872 | −0.007601 | 0.505705 | 0.017* |
| H25B | 0.223523 | 0.002816 | 0.481369 | 0.017* |
| C8 | 0.8139 (3) | 0.37093 (9) | 0.27071 (8) | 0.0126 (3) |
| C13 | 0.9608 (3) | 0.34721 (9) | 0.38172 (8) | 0.0155 (3) |
| H13A | 0.975665 | 0.309141 | 0.415590 | 0.023* |
| H13B | 0.911232 | 0.394965 | 0.400299 | 0.023* |
| H13C | 1.096090 | 0.355534 | 0.361145 | 0.023* |
| C12 | 1.0227 (3) | 0.34651 (10) | 0.23951 (8) | 0.0155 (3) |
| H12A | 1.137374 | 0.379980 | 0.253852 | 0.019* |
| H12B | 1.012823 | 0.349257 | 0.192295 | 0.019* |
| C6 | 0.5917 (3) | 0.49264 (10) | 0.28061 (9) | 0.0205 (4) |
| H6A | 0.491554 | 0.464500 | 0.307701 | 0.025* |
| H6B | 0.546071 | 0.487033 | 0.235619 | 0.025* |
| C4 | 0.5645 (4) | 0.72401 (12) | 0.39145 (14) | 0.0431 (6) |
| H4A | 0.708534 | 0.713275 | 0.404017 | 0.065* |
| H4B | 0.518021 | 0.771070 | 0.411956 | 0.065* |
| H4C | 0.556793 | 0.729660 | 0.344859 | 0.065* |
| C3 | 0.2026 (4) | 0.67460 (16) | 0.38883 (13) | 0.0430 (6) |
| H3A | 0.203839 | 0.679901 | 0.342158 | 0.065* |
| H3B | 0.151797 | 0.721734 | 0.408214 | 0.065* |
| H3C | 0.110650 | 0.632594 | 0.400915 | 0.065* |
| C2 | 0.4239 (6) | 0.64969 (16) | 0.48501 (10) | 0.0564 (9) |
| H2A | 0.332983 | 0.607392 | 0.497155 | 0.085* |
| H2B | 0.372756 | 0.696584 | 0.504713 | 0.085* |
| H2C | 0.565977 | 0.639511 | 0.499814 | 0.085* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0352 (3) | 0.01312 (19) | 0.0195 (2) | 0.00396 (19) | 0.01024 (19) | 0.00171 (16) |
| O3 | 0.0179 (6) | 0.0176 (6) | 0.0106 (5) | −0.0006 (5) | −0.0030 (5) | 0.0007 (4) |
| O5 | 0.0195 (6) | 0.0148 (6) | 0.0216 (6) | −0.0049 (5) | 0.0009 (5) | −0.0018 (5) |
| O2 | 0.0258 (7) | 0.0141 (6) | 0.0257 (7) | −0.0061 (5) | −0.0009 (6) | 0.0018 (5) |
| O4 | 0.0479 (9) | 0.0151 (6) | 0.0100 (6) | 0.0015 (6) | 0.0044 (6) | −0.0025 (5) |
| O1 | 0.0617 (11) | 0.0218 (7) | 0.0221 (7) | 0.0018 (7) | 0.0063 (7) | 0.0068 (6) |
| C10 | 0.0096 (7) | 0.0121 (7) | 0.0099 (7) | 0.0001 (6) | 0.0002 (6) | −0.0007 (6) |
| C16 | 0.0111 (7) | 0.0097 (7) | 0.0093 (6) | 0.0006 (6) | 0.0011 (6) | −0.0002 (6) |
| C24 | 0.0172 (8) | 0.0099 (7) | 0.0128 (7) | 0.0012 (6) | −0.0020 (7) | 0.0024 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C22 | 0.0157 (8) | 0.0138 (8) | 0.0122 (7) | 0.0002 (6) | −0.0027 (6) | −0.0015 (6) |
| C7 | 0.0225 (9) | 0.0147 (8) | 0.0082 (7) | 0.0000 (7) | 0.0026 (7) | 0.0028 (6) |
| C18 | 0.0114 (7) | 0.0095 (7) | 0.0094 (7) | −0.0004 (6) | −0.0006 (5) | −0.0010 (6) |
| C17 | 0.0107 (7) | 0.0106 (7) | 0.0111 (7) | 0.0006 (6) | 0.0007 (6) | −0.0006 (6) |
| C23 | 0.0156 (8) | 0.0113 (7) | 0.0132 (7) | 0.0012 (6) | 0.0003 (6) | −0.0023 (6) |
| C26 | 0.0128 (7) | 0.0108 (7) | 0.0111 (7) | −0.0001 (6) | 0.0012 (6) | −0.0009 (6) |
| C21 | 0.0142 (7) | 0.0125 (7) | 0.0154 (7) | 0.0012 (6) | 0.0045 (6) | −0.0003 (6) |
| C20 | 0.0127 (7) | 0.0111 (7) | 0.0177 (8) | 0.0018 (6) | 0.0032 (7) | −0.0018 (6) |
| C9 | 0.0125 (7) | 0.0104 (7) | 0.0102 (7) | −0.0005 (6) | −0.0004 (6) | 0.0007 (6) |
| C11 | 0.0128 (7) | 0.0149 (7) | 0.0149 (7) | −0.0006 (7) | 0.0030 (6) | 0.0006 (6) |
| C1 | 0.0255 (9) | 0.0243 (9) | 0.0158 (8) | 0.0083 (8) | −0.0004 (8) | −0.0027 (7) |
| C19 | 0.0124 (7) | 0.0119 (7) | 0.0092 (7) | 0.0028 (6) | −0.0014 (6) | 0.0004 (6) |
| C15 | 0.0167 (8) | 0.0116 (7) | 0.0142 (7) | 0.0013 (6) | 0.0045 (7) | −0.0001 (6) |
| C5 | 0.0209 (8) | 0.0195 (8) | 0.0146 (7) | 0.0021 (7) | −0.0015 (7) | 0.0016 (7) |
| C14 | 0.0141 (7) | 0.0111 (7) | 0.0151 (7) | 0.0019 (6) | 0.0024 (6) | 0.0010 (6) |
| C25 | 0.0154 (8) | 0.0140 (8) | 0.0119 (7) | −0.0013 (6) | 0.0028 (6) | 0.0000 (6) |
| C8 | 0.0140 (8) | 0.0126 (7) | 0.0110 (7) | −0.0012 (6) | −0.0016 (6) | 0.0007 (6) |
| C13 | 0.0195 (8) | 0.0138 (7) | 0.0133 (7) | −0.0007 (7) | −0.0046 (7) | −0.0013 (6) |
| C12 | 0.0170 (8) | 0.0150 (8) | 0.0143 (8) | −0.0013 (7) | 0.0035 (6) | 0.0012 (6) |
| C6 | 0.0276 (9) | 0.0137 (8) | 0.0203 (8) | 0.0030 (7) | −0.0007 (8) | −0.0039 (6) |
| C4 | 0.0458 (14) | 0.0216 (10) | 0.0621 (16) | 0.0019 (10) | 0.0085 (14) | −0.0157 (10) |
| C3 | 0.0298 (12) | 0.0568 (16) | 0.0424 (13) | 0.0196 (11) | −0.0046 (11) | −0.0140 (12) |
| C2 | 0.105 (3) | 0.0496 (15) | 0.0150 (9) | 0.0370 (18) | −0.0069 (13) | −0.0052 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C5 | 1.7827 (18) | C20—H20A | 0.9900 |
| S1—C6 | 1.7957 (18) | C20—H20B | 0.9900 |
| O3—H3 | 0.8400 | C20—C19 | 1.501 (2) |
| O3—C8 | 1.426 (2) | C9—C14 | 1.529 (2) |
| O5—C24 | 1.227 (2) | C9—C8 | 1.571 (2) |
| O2—C7 | 1.208 (2) | C9—C13 | 1.538 (2) |
| O4—H4 | 0.8400 | C11—H11A | 0.9900 |
| O4—C15 | 1.447 (2) | C11—H11B | 0.9900 |
| O1—C5 | 1.203 (2) | C11—C12 | 1.553 (2) |
| C10—H10 | 1.0000 | C1—C5 | 1.531 (3) |
| C10—C17 | 1.521 (2) | C1—C4 | 1.534 (3) |
| C10—C9 | 1.538 (2) | C1—C3 | 1.531 (3) |
| C10—C11 | 1.534 (2) | C1—C2 | 1.521 (3) |
| C16—H16 | 1.0000 | C15—H15 | 1.0000 |
| C16—C18 | 1.570 (2) | C15—C14 | 1.546 (2) |
| C16—C17 | 1.543 (2) | C14—H14A | 0.9900 |
| C16—C15 | 1.546 (2) | C14—H14B | 0.9900 |
| C24—C23 | 1.466 (2) | C25—H25A | 0.9900 |
| C24—C25 | 1.506 (2) | C25—H25B | 0.9900 |
| C22—H22A | 0.9800 | C8—C12 | 1.552 (2) |
| C22—H22B | 0.9800 | C13—H13A | 0.9800 |
| C22—H22C | 0.9800 | C13—H13B | 0.9800 |

| | | | |
|---|---|---|---|
| C22—C18 | 1.549 (2) | C13—H13C | 0.9800 |
| C7—C8 | 1.543 (2) | C12—H12A | 0.9900 |
| C7—C6 | 1.520 (3) | C12—H12B | 0.9900 |
| C18—C26 | 1.538 (2) | C6—H6A | 0.9900 |
| C18—C19 | 1.524 (2) | C6—H6B | 0.9900 |
| C17—H17 | 1.0000 | C4—H4A | 0.9800 |
| C17—C21 | 1.531 (2) | C4—H4B | 0.9800 |
| C23—H23 | 0.9500 | C4—H4C | 0.9800 |
| C23—C19 | 1.342 (2) | C3—H3A | 0.9800 |
| C26—H26A | 0.9900 | C3—H3B | 0.9800 |
| C26—H26B | 0.9900 | C3—H3C | 0.9800 |
| C26—C25 | 1.535 (2) | C2—H2A | 0.9800 |
| C21—H21A | 0.9900 | C2—H2B | 0.9800 |
| C21—H21B | 0.9900 | C2—H2C | 0.9800 |
| C21—C20 | 1.529 (2) | | |

| | | | |
|---|---|---|---|
| C5—S1—C6 | 99.71 (9) | C5—C1—C4 | 111.30 (16) |
| C8—O3—H3 | 109.5 | C3—C1—C5 | 107.18 (17) |
| C15—O4—H4 | 109.5 | C3—C1—C4 | 108.15 (19) |
| C17—C10—H10 | 106.4 | C2—C1—C5 | 108.89 (16) |
| C17—C10—C9 | 113.83 (13) | C2—C1—C4 | 111.2 (2) |
| C17—C10—C11 | 118.79 (13) | C2—C1—C3 | 110.0 (2) |
| C9—C10—H10 | 106.4 | C23—C19—C18 | 123.42 (15) |
| C11—C10—H10 | 106.4 | C23—C19—C20 | 120.46 (15) |
| C11—C10—C9 | 104.26 (13) | C20—C19—C18 | 116.09 (13) |
| C18—C16—H16 | 104.3 | O4—C15—C16 | 110.17 (13) |
| C17—C16—H16 | 104.3 | O4—C15—H15 | 107.5 |
| C17—C16—C18 | 113.58 (13) | O4—C15—C14 | 110.62 (13) |
| C17—C16—C15 | 114.07 (13) | C16—C15—H15 | 107.5 |
| C15—C16—H16 | 104.3 | C16—C15—C14 | 113.19 (13) |
| C15—C16—C18 | 114.64 (12) | C14—C15—H15 | 107.5 |
| O5—C24—C23 | 121.75 (15) | O1—C5—S1 | 120.96 (15) |
| O5—C24—C25 | 123.32 (16) | O1—C5—C1 | 124.65 (17) |
| C23—C24—C25 | 114.93 (14) | C1—C5—S1 | 114.36 (13) |
| H22A—C22—H22B | 109.5 | C9—C14—C15 | 113.34 (13) |
| H22A—C22—H22C | 109.5 | C9—C14—H14A | 108.9 |
| H22B—C22—H22C | 109.5 | C9—C14—H14B | 108.9 |
| C18—C22—H22A | 109.5 | C15—C14—H14A | 108.9 |
| C18—C22—H22B | 109.5 | C15—C14—H14B | 108.9 |
| C18—C22—H22C | 109.5 | H14A—C14—H14B | 107.7 |
| O2—C7—C8 | 122.04 (17) | C24—C25—C26 | 109.05 (13) |
| O2—C7—C6 | 122.91 (16) | C24—C25—H25A | 109.9 |
| C6—C7—C8 | 115.05 (15) | C24—C25—H25B | 109.9 |
| C22—C18—C16 | 114.12 (13) | C26—C25—H25A | 109.9 |
| C26—C18—C16 | 108.77 (12) | C26—C25—H25B | 109.9 |
| C26—C18—C22 | 110.05 (12) | H25A—C25—H25B | 108.3 |
| C19—C18—C16 | 106.91 (12) | O3—C8—C7 | 109.59 (14) |
| C19—C18—C22 | 106.50 (13) | O3—C8—C9 | 107.43 (13) |

| | | | |
|---|---|---|---|
| C19—C18—C26 | 110.42 (13) | O3—C8—C12 | 111.23 (13) |
| C10—C17—C16 | 108.20 (12) | C7—C8—C9 | 112.25 (13) |
| C10—C17—H17 | 109.9 | C7—C8—C12 | 113.50 (14) |
| C10—C17—C21 | 108.97 (13) | C12—C8—C9 | 102.54 (13) |
| C16—C17—H17 | 109.9 | C9—C13—H13A | 109.5 |
| C21—C17—C16 | 110.02 (13) | C9—C13—H13B | 109.5 |
| C21—C17—H17 | 109.9 | C9—C13—H13C | 109.5 |
| C24—C23—H23 | 118.4 | H13A—C13—H13B | 109.5 |
| C19—C23—C24 | 123.21 (15) | H13A—C13—H13C | 109.5 |
| C19—C23—H23 | 118.4 | H13B—C13—H13C | 109.5 |
| C18—C26—H26A | 108.9 | C11—C12—H12A | 110.5 |
| C18—C26—H26B | 108.9 | C11—C12—H12B | 110.5 |
| H26A—C26—H26B | 107.7 | C8—C12—C11 | 106.20 (13) |
| C25—C26—C18 | 113.48 (13) | C8—C12—H12A | 110.5 |
| C25—C26—H26A | 108.9 | C8—C12—H12B | 110.5 |
| C25—C26—H26B | 108.9 | H12A—C12—H12B | 108.7 |
| C17—C21—H21A | 109.0 | S1—C6—H6A | 108.5 |
| C17—C21—H21B | 109.0 | S1—C6—H6B | 108.5 |
| H21A—C21—H21B | 107.8 | C7—C6—S1 | 115.15 (13) |
| C20—C21—C17 | 113.14 (13) | C7—C6—H6A | 108.5 |
| C20—C21—H21A | 109.0 | C7—C6—H6B | 108.5 |
| C20—C21—H21B | 109.0 | H6A—C6—H6B | 107.5 |
| C21—C20—H20A | 109.2 | C1—C4—H4A | 109.5 |
| C21—C20—H20B | 109.2 | C1—C4—H4B | 109.5 |
| H20A—C20—H20B | 107.9 | C1—C4—H4C | 109.5 |
| C19—C20—C21 | 112.07 (14) | H4A—C4—H4B | 109.5 |
| C19—C20—H20A | 109.2 | H4A—C4—H4C | 109.5 |
| C19—C20—H20B | 109.2 | H4B—C4—H4C | 109.5 |
| C10—C9—C8 | 98.89 (12) | C1—C3—H3A | 109.5 |
| C10—C9—C13 | 112.53 (13) | C1—C3—H3B | 109.5 |
| C14—C9—C10 | 108.37 (13) | C1—C3—H3C | 109.5 |
| C14—C9—C8 | 115.37 (13) | H3A—C3—H3B | 109.5 |
| C14—C9—C13 | 111.59 (13) | H3A—C3—H3C | 109.5 |
| C13—C9—C8 | 109.50 (13) | H3B—C3—H3C | 109.5 |
| C10—C11—H11A | 110.9 | C1—C2—H2A | 109.5 |
| C10—C11—H11B | 110.9 | C1—C2—H2B | 109.5 |
| C10—C11—C12 | 104.17 (13) | C1—C2—H2C | 109.5 |
| H11A—C11—H11B | 108.9 | H2A—C2—H2B | 109.5 |
| C12—C11—H11A | 110.9 | H2A—C2—H2C | 109.5 |
| C12—C11—H11B | 110.9 | H2B—C2—H2C | 109.5 |

| | | | |
|---|---|---|---|
| O3—C8—C12—C11 | −88.38 (16) | C26—C18—C19—C23 | −10.2 (2) |
| O5—C24—C23—C19 | 166.22 (16) | C26—C18—C19—C20 | 171.80 (13) |
| O5—C24—C25—C26 | −136.63 (16) | C21—C20—C19—C18 | −52.86 (18) |
| O2—C7—C8—O3 | −159.55 (15) | C21—C20—C19—C23 | 129.03 (16) |
| O2—C7—C8—C9 | 81.2 (2) | C9—C10—C17—C16 | 59.62 (17) |
| O2—C7—C8—C12 | −34.5 (2) | C9—C10—C17—C21 | 179.23 (13) |
| O2—C7—C6—S1 | −0.9 (2) | C9—C10—C11—C12 | −31.87 (16) |

| | | | |
|---|---|---|---|
| O4—C15—C14—C9 | 76.44 (17) | C9—C8—C12—C11 | 26.18 (16) |
| C10—C17—C21—C20 | −170.47 (13) | C11—C10—C17—C16 | −177.02 (13) |
| C10—C9—C14—C15 | 52.98 (17) | C11—C10—C17—C21 | −57.42 (18) |
| C10—C9—C8—O3 | 72.69 (15) | C11—C10—C9—C14 | 168.13 (13) |
| C10—C9—C8—C7 | −166.78 (14) | C11—C10—C9—C8 | 47.53 (15) |
| C10—C9—C8—C12 | −44.62 (14) | C11—C10—C9—C13 | −67.98 (16) |
| C10—C11—C12—C8 | 2.91 (17) | C19—C18—C26—C25 | 41.50 (17) |
| C16—C18—C26—C25 | 158.52 (13) | C15—C16—C18—C22 | −71.13 (17) |
| C16—C18—C19—C23 | −128.32 (16) | C15—C16—C18—C26 | 52.16 (17) |
| C16—C18—C19—C20 | 53.64 (17) | C15—C16—C18—C19 | 171.40 (13) |
| C16—C17—C21—C20 | −51.99 (18) | C15—C16—C17—C10 | −51.38 (17) |
| C16—C15—C14—C9 | −47.76 (19) | C15—C16—C17—C21 | −170.32 (13) |
| C24—C23—C19—C18 | −3.6 (3) | C5—S1—C6—C7 | −95.53 (14) |
| C24—C23—C19—C20 | 174.33 (15) | C14—C9—C8—O3 | −42.61 (18) |
| C22—C18—C26—C25 | −75.79 (17) | C14—C9—C8—C7 | 77.93 (18) |
| C22—C18—C19—C23 | 109.31 (17) | C14—C9—C8—C12 | −159.91 (14) |
| C22—C18—C19—C20 | −68.74 (17) | C25—C24—C23—C19 | −14.5 (2) |
| C7—C8—C12—C11 | 147.50 (14) | C8—C7—C6—S1 | 178.95 (12) |
| C18—C16—C17—C10 | 174.74 (12) | C8—C9—C14—C15 | 162.72 (13) |
| C18—C16—C17—C21 | 55.80 (16) | C13—C9—C14—C15 | −71.47 (17) |
| C18—C16—C15—O4 | 55.85 (18) | C13—C9—C8—O3 | −169.48 (13) |
| C18—C16—C15—C14 | −179.71 (13) | C13—C9—C8—C7 | −48.95 (18) |
| C18—C26—C25—C24 | −59.02 (17) | C13—C9—C8—C12 | 73.22 (15) |
| C17—C10—C9—C14 | −60.92 (16) | C6—S1—C5—O1 | 9.35 (19) |
| C17—C10—C9—C8 | 178.48 (13) | C6—S1—C5—C1 | −168.80 (14) |
| C17—C10—C9—C13 | 62.97 (17) | C6—C7—C8—O3 | 20.62 (19) |
| C17—C10—C11—C12 | −159.84 (14) | C6—C7—C8—C9 | −98.66 (17) |
| C17—C16—C18—C22 | 62.48 (17) | C6—C7—C8—C12 | 145.63 (15) |
| C17—C16—C18—C26 | −174.24 (12) | C4—C1—C5—S1 | −38.1 (2) |
| C17—C16—C18—C19 | −55.00 (16) | C4—C1—C5—O1 | 143.8 (2) |
| C17—C16—C15—O4 | −77.53 (16) | C3—C1—C5—S1 | 79.96 (19) |
| C17—C16—C15—C14 | 46.91 (19) | C3—C1—C5—O1 | −98.1 (2) |
| C17—C21—C20—C19 | 50.36 (19) | C2—C1—C5—S1 | −161.1 (2) |
| C23—C24—C25—C26 | 44.08 (19) | C2—C1—C5—O1 | 20.9 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O5$^{i}$ | 0.84 | 2.07 | 2.9021 (17) | 169 |

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.

**(*S*)-{2-[(8*S*,9*S*,10*R*,11*S*,13*S*,14*S*,17*R*)-11,17-Dihydroxy-10,13-dimethyl-3-oxo-2,6,7,8,9,11,12,14,15,16-decahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl]-2-oxoethyl} 2,2-dimethylpropanethioate (SY20C174)**

*Crystal data*

$C_{26}H_{38}O_5S$
$M_r$ = 462.62

Orthorhombic, $P2_12_12_1$
*a* = 6.0146 (2) Å

$b$ = 19.2817 (7) Å
$c$ = 20.9887 (7) Å
$V$ = 2434.10 (14) Å$^3$
$Z$ = 4
$F$(000) = 1000
$D_x$ = 1.262 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54178 Å

Cell parameters from 6178 reflections
$\theta$ = 3.1–66.5°
$\mu$ = 1.46 mm$^{-1}$
$T$ = 100 K
Plate, clear light colourless
0.18 × 0.06 × 0.05 mm

*Data collection*

Bruker D8 Venture diffractometer
Radiation source: sealed X-ray tube, high brilliance microfocus sealed tube, Cu
QUAZAR MX multilayer optics monochromator
Detector resolution: 1024 x 1024 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans′
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)

$T_{min}$ = 0.707, $T_{max}$ = 0.862
30900 measured reflections
4303 independent reflections
3803 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.102
$\theta_{max}$ = 66.7°, $\theta_{min}$ = 3.1°
$h$ = −7→6
$k$ = −22→22
$l$ = −24→25

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.130
$S$ = 1.07
4303 reflections
329 parameters
16 restraints
Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 1.3292P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$
Absolute structure: Flack $x$ obtained from refinement
Absolute structure parameter: 0.11 (4)

*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.
**Refinement**. Refined as a 2-component inversion twin.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O2 | 0.5899 (6) | 0.4360 (2) | 0.72379 (16) | 0.0542 (10) | |
| O3 | 0.2156 (5) | 0.50617 (16) | 0.61339 (15) | 0.0390 (7) | |
| H3 | 0.209393 | 0.545661 | 0.630189 | 0.059* | |
| O4 | 0.4523 (5) | 0.28363 (14) | 0.49153 (14) | 0.0367 (7) | |
| H4 | 0.369010 | 0.253012 | 0.507049 | 0.055* | |
| O5 | 0.2955 (7) | 0.36601 (17) | 0.17678 (14) | 0.0487 (9) | |
| C19 | 0.5824 (7) | 0.4099 (2) | 0.3207 (2) | 0.0332 (9) | |
| C23 | 0.5075 (8) | 0.4126 (2) | 0.2605 (2) | 0.0384 (10) | |
| H23 | 0.550048 | 0.451088 | 0.235126 | 0.046* | |
| C10 | 0.5508 (7) | 0.4744 (2) | 0.5179 (2) | 0.0324 (9) | |

| | | | | | |
|---|---|---|---|---|---|
| H10 | 0.415291 | 0.501122 | 0.505311 | 0.039* | |
| C26 | 0.2974 (7) | 0.3182 (2) | 0.34159 (18) | 0.0312 (9) | |
| H26A | 0.270014 | 0.275824 | 0.366990 | 0.037* | |
| H26B | 0.167597 | 0.349225 | 0.347184 | 0.037* | |
| C21 | 0.7010 (7) | 0.4915 (2) | 0.4084 (2) | 0.0346 (9) | |
| H21A | 0.832545 | 0.517167 | 0.424124 | 0.042* | |
| H21B | 0.578497 | 0.525229 | 0.402795 | 0.042* | |
| C18 | 0.5052 (7) | 0.3548 (2) | 0.36772 (19) | 0.0288 (9) | |
| C24 | 0.3664 (8) | 0.3607 (2) | 0.2319 (2) | 0.0406 (11) | |
| C15 | 0.3269 (7) | 0.34602 (19) | 0.48133 (19) | 0.0296 (9) | |
| H15 | 0.180184 | 0.332125 | 0.462922 | 0.036* | |
| C8 | 0.4270 (8) | 0.4760 (2) | 0.6258 (2) | 0.0386 (10) | |
| C17 | 0.6327 (7) | 0.4380 (2) | 0.4582 (2) | 0.0292 (9) | |
| H17 | 0.763081 | 0.408045 | 0.468950 | 0.035* | |
| C9 | 0.4772 (7) | 0.4237 (2) | 0.5707 (2) | 0.0322 (10) | |
| C22 | 0.6971 (8) | 0.3019 (2) | 0.3744 (2) | 0.0345 (9) | |
| H22A | 0.751711 | 0.289182 | 0.331957 | 0.052* | |
| H22B | 0.642942 | 0.260357 | 0.396306 | 0.052* | |
| H22C | 0.818350 | 0.322694 | 0.399048 | 0.052* | |
| C11 | 0.7052 (8) | 0.5257 (2) | 0.5512 (2) | 0.0387 (10) | |
| H11A | 0.698529 | 0.571722 | 0.530401 | 0.046* | |
| H11B | 0.860739 | 0.508887 | 0.550330 | 0.046* | |
| C20 | 0.7564 (7) | 0.4589 (2) | 0.3438 (2) | 0.0378 (11) | |
| H20A | 0.775716 | 0.496321 | 0.311979 | 0.045* | |
| H20B | 0.899444 | 0.433782 | 0.347301 | 0.045* | |
| C12 | 0.6174 (8) | 0.5295 (3) | 0.6202 (2) | 0.0448 (12) | |
| H12A | 0.561469 | 0.576745 | 0.629549 | 0.054* | |
| H12B | 0.737654 | 0.518446 | 0.650724 | 0.054* | |
| C6 | 0.2076 (9) | 0.4088 (2) | 0.7138 (2) | 0.0441 (11) | |
| H6AA | 0.196918 | 0.361138 | 0.696528 | 0.053* | 0.770 (4) |
| H6AB | 0.082226 | 0.435978 | 0.696182 | 0.053* | 0.770 (4) |
| H6BC | 0.120508 | 0.394023 | 0.676151 | 0.053* | 0.230 (4) |
| H6BD | 0.119404 | 0.443918 | 0.737153 | 0.053* | 0.230 (4) |
| C13 | 0.6669 (8) | 0.3752 (2) | 0.5918 (2) | 0.0381 (10) | |
| H13A | 0.721108 | 0.348936 | 0.554993 | 0.057* | |
| H13B | 0.611557 | 0.343021 | 0.624265 | 0.057* | |
| H13C | 0.788640 | 0.402879 | 0.609613 | 0.057* | |
| C25 | 0.3151 (8) | 0.2983 (2) | 0.27161 (19) | 0.0373 (10) | |
| H25A | 0.433983 | 0.263261 | 0.266140 | 0.045* | |
| H25B | 0.173190 | 0.277449 | 0.257299 | 0.045* | |
| C14 | 0.2796 (7) | 0.3837 (2) | 0.54478 (19) | 0.0303 (9) | |
| H14A | 0.154299 | 0.416247 | 0.538403 | 0.036* | |
| H14B | 0.232769 | 0.349021 | 0.576884 | 0.036* | |
| C7 | 0.4242 (9) | 0.4410 (2) | 0.6919 (2) | 0.0421 (11) | |
| C16 | 0.4439 (7) | 0.39282 (19) | 0.43159 (19) | 0.0270 (9) | |
| H16 | 0.326901 | 0.426958 | 0.418849 | 0.032* | |
| S1 | 0.1815 (3) | 0.40537 (8) | 0.79935 (7) | 0.0450 (5) | 0.770 (4) |
| O1 | 0.3483 (7) | 0.2850 (2) | 0.76836 (19) | 0.0451 (11) | 0.770 (4) |

| | | | | | |
|---|---|---|---|---|---|
| C1 | 0.3521 (12) | 0.3028 (4) | 0.8813 (3) | 0.0361 (18) | 0.770 (4) |
| C2 | 0.6090 (12) | 0.3076 (8) | 0.8878 (8) | 0.039 (3) | 0.770 (4) |
| H2A | 0.652836 | 0.294448 | 0.931111 | 0.058* | 0.770 (4) |
| H2B | 0.657117 | 0.355274 | 0.879249 | 0.058* | 0.770 (4) |
| H2C | 0.679074 | 0.276143 | 0.857122 | 0.058* | 0.770 (4) |
| C4 | 0.272 (2) | 0.2291 (5) | 0.8928 (5) | 0.058 (3) | 0.770 (4) |
| H4A | 0.339000 | 0.198135 | 0.861101 | 0.087* | 0.770 (4) |
| H4B | 0.110117 | 0.227401 | 0.889187 | 0.087* | 0.770 (4) |
| H4C | 0.316890 | 0.214251 | 0.935628 | 0.087* | 0.770 (4) |
| C3 | 0.2438 (11) | 0.3521 (3) | 0.9302 (3) | 0.0404 (15) | 0.770 (4) |
| H3A | 0.278848 | 0.336261 | 0.973456 | 0.061* | 0.770 (4) |
| H3B | 0.082257 | 0.352166 | 0.924197 | 0.061* | 0.770 (4) |
| H3C | 0.301585 | 0.399154 | 0.924132 | 0.061* | 0.770 (4) |
| C5 | 0.3022 (10) | 0.3230 (3) | 0.8119 (3) | 0.0332 (12) | 0.770 (4) |
| S1A | 0.2553 (10) | 0.3349 (3) | 0.7649 (3) | 0.055 (2)* | 0.230 (4) |
| O1A | 0.297 (2) | 0.4314 (7) | 0.8496 (6) | 0.042 (4)* | 0.230 (4) |
| C1A | 0.355 (4) | 0.3187 (10) | 0.8972 (9) | 0.023 (7)* | 0.230 (4) |
| C2A | 0.615 (5) | 0.309 (3) | 0.899 (3) | 0.06 (2)* | 0.230 (4) |
| H2AA | 0.683993 | 0.351239 | 0.915726 | 0.089* | 0.230 (4) |
| H2AB | 0.669427 | 0.300462 | 0.855557 | 0.089* | 0.230 (4) |
| H2AC | 0.652259 | 0.269623 | 0.926175 | 0.089* | 0.230 (4) |
| C3A | 0.283 (5) | 0.3463 (15) | 0.9609 (11) | 0.056 (8)* | 0.230 (4) |
| H3AA | 0.340463 | 0.316272 | 0.994771 | 0.084* | 0.230 (4) |
| H3AB | 0.120195 | 0.347280 | 0.962866 | 0.084* | 0.230 (4) |
| H3AC | 0.341046 | 0.393392 | 0.966587 | 0.084* | 0.230 (4) |
| C4A | 0.260 (6) | 0.2455 (13) | 0.8820 (15) | 0.038 (8)* | 0.230 (4) |
| H4AA | 0.330574 | 0.227462 | 0.843296 | 0.058* | 0.230 (4) |
| H4AB | 0.099349 | 0.248796 | 0.875531 | 0.058* | 0.230 (4) |
| H4AC | 0.291241 | 0.214194 | 0.917714 | 0.058* | 0.230 (4) |
| C5A | 0.310 (3) | 0.3695 (9) | 0.8412 (8) | 0.041 (5)* | 0.230 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O2 | 0.057 (2) | 0.066 (2) | 0.0398 (19) | −0.0032 (19) | −0.0148 (17) | −0.0134 (17) |
| O3 | 0.0435 (18) | 0.0303 (16) | 0.0434 (18) | 0.0078 (14) | −0.0008 (14) | −0.0107 (13) |
| O4 | 0.056 (2) | 0.0208 (14) | 0.0328 (16) | −0.0010 (14) | 0.0056 (15) | 0.0014 (12) |
| O5 | 0.077 (2) | 0.0398 (18) | 0.0297 (17) | 0.0135 (18) | −0.0042 (17) | 0.0032 (13) |
| C19 | 0.034 (2) | 0.031 (2) | 0.035 (2) | 0.0098 (18) | 0.0098 (18) | 0.0075 (18) |
| C23 | 0.049 (3) | 0.030 (2) | 0.036 (2) | 0.009 (2) | 0.011 (2) | 0.008 (2) |
| C10 | 0.029 (2) | 0.025 (2) | 0.043 (2) | 0.0000 (17) | −0.0052 (19) | −0.0017 (19) |
| C26 | 0.033 (2) | 0.030 (2) | 0.031 (2) | −0.0031 (18) | 0.0027 (19) | 0.0025 (17) |
| C21 | 0.031 (2) | 0.026 (2) | 0.047 (3) | −0.0033 (18) | 0.000 (2) | 0.0037 (18) |
| C18 | 0.032 (2) | 0.026 (2) | 0.029 (2) | 0.0006 (17) | 0.0017 (17) | 0.0055 (17) |
| C24 | 0.053 (3) | 0.038 (2) | 0.031 (2) | 0.017 (2) | 0.007 (2) | 0.0035 (19) |
| C15 | 0.036 (2) | 0.022 (2) | 0.030 (2) | −0.0032 (18) | −0.0003 (18) | −0.0013 (16) |
| C8 | 0.040 (2) | 0.032 (2) | 0.044 (3) | 0.005 (2) | −0.005 (2) | −0.009 (2) |
| C17 | 0.026 (2) | 0.0237 (19) | 0.038 (2) | 0.0022 (16) | −0.0028 (17) | 0.0020 (17) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9 | 0.033 (2) | 0.027 (2) | 0.037 (2) | 0.0015 (17) | −0.0020 (18) | −0.0033 (18) |
| C22 | 0.042 (2) | 0.028 (2) | 0.033 (2) | 0.003 (2) | 0.004 (2) | 0.0018 (17) |
| C11 | 0.036 (2) | 0.030 (2) | 0.050 (3) | −0.0019 (19) | −0.007 (2) | −0.006 (2) |
| C20 | 0.036 (2) | 0.031 (2) | 0.047 (3) | −0.0004 (18) | 0.008 (2) | 0.010 (2) |
| C12 | 0.046 (3) | 0.037 (2) | 0.051 (3) | −0.001 (2) | −0.007 (2) | −0.015 (2) |
| C6 | 0.058 (3) | 0.041 (3) | 0.033 (2) | 0.012 (2) | −0.002 (2) | 0.000 (2) |
| C13 | 0.043 (3) | 0.034 (2) | 0.037 (2) | 0.005 (2) | −0.006 (2) | −0.0056 (19) |
| C25 | 0.044 (2) | 0.036 (2) | 0.032 (2) | 0.004 (2) | −0.002 (2) | 0.0002 (18) |
| C14 | 0.032 (2) | 0.025 (2) | 0.034 (2) | −0.0018 (17) | 0.0041 (17) | 0.0008 (17) |
| C7 | 0.052 (3) | 0.038 (2) | 0.037 (3) | 0.004 (2) | −0.004 (2) | −0.016 (2) |
| C16 | 0.026 (2) | 0.0223 (19) | 0.033 (2) | 0.0016 (16) | −0.0008 (17) | 0.0049 (16) |
| S1 | 0.0628 (10) | 0.0413 (9) | 0.0309 (8) | 0.0206 (8) | 0.0025 (7) | −0.0008 (6) |
| O1 | 0.052 (3) | 0.042 (2) | 0.042 (2) | 0.007 (2) | −0.015 (2) | −0.013 (2) |
| C1 | 0.041 (4) | 0.031 (4) | 0.036 (4) | 0.008 (3) | −0.001 (3) | −0.008 (3) |
| C2 | 0.033 (5) | 0.044 (6) | 0.039 (5) | 0.005 (3) | −0.007 (3) | −0.007 (4) |
| C4 | 0.071 (6) | 0.037 (4) | 0.066 (6) | 0.001 (4) | −0.013 (5) | 0.012 (4) |
| C3 | 0.041 (4) | 0.043 (4) | 0.037 (4) | 0.006 (3) | −0.001 (3) | −0.001 (3) |
| C5 | 0.032 (3) | 0.030 (3) | 0.037 (3) | 0.001 (2) | 0.000 (2) | −0.004 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O2—C7 | 1.204 (6) | C6—H6AA | 0.9900 |
| O3—H3 | 0.8400 | C6—H6AB | 0.9900 |
| O3—C8 | 1.422 (6) | C6—H6BC | 0.9900 |
| O4—H4 | 0.8400 | C6—H6BD | 0.9900 |
| O4—C15 | 1.436 (5) | C6—C7 | 1.514 (7) |
| O5—C24 | 1.238 (5) | C6—S1 | 1.804 (4) |
| C19—C23 | 1.341 (6) | C6—S1A | 1.807 (5) |
| C19—C18 | 1.523 (6) | C13—H13A | 0.9800 |
| C19—C20 | 1.491 (6) | C13—H13B | 0.9800 |
| C23—H23 | 0.9500 | C13—H13C | 0.9800 |
| C23—C24 | 1.443 (7) | C25—H25A | 0.9900 |
| C10—H10 | 1.0000 | C25—H25B | 0.9900 |
| C10—C17 | 1.519 (6) | C14—H14A | 0.9900 |
| C10—C9 | 1.542 (6) | C14—H14B | 0.9900 |
| C10—C11 | 1.525 (6) | C16—H16 | 1.0000 |
| C26—H26A | 0.9900 | S1—C5 | 1.767 (6) |
| C26—H26B | 0.9900 | O1—C5 | 1.203 (6) |
| C26—C18 | 1.536 (6) | C1—C2 | 1.554 (9) |
| C26—C25 | 1.522 (5) | C1—C4 | 1.519 (11) |
| C21—H21A | 0.9900 | C1—C3 | 1.543 (9) |
| C21—H21B | 0.9900 | C1—C5 | 1.538 (9) |
| C21—C17 | 1.524 (6) | C2—H2A | 0.9800 |
| C21—C20 | 1.531 (6) | C2—H2B | 0.9800 |
| C18—C22 | 1.547 (6) | C2—H2C | 0.9800 |
| C18—C16 | 1.572 (6) | C4—H4A | 0.9800 |
| C24—C25 | 1.495 (6) | C4—H4B | 0.9800 |
| C15—H15 | 1.0000 | C4—H4C | 0.9800 |

| | | | |
|---|---|---|---|
| C15—C14 | 1.543 (6) | C3—H3A | 0.9800 |
| C15—C16 | 1.549 (5) | C3—H3B | 0.9800 |
| C8—C9 | 1.565 (6) | C3—H3C | 0.9800 |
| C8—C12 | 1.547 (7) | S1A—C5A | 1.767 (16) |
| C8—C7 | 1.543 (7) | O1A—C5A | 1.209 (17) |
| C17—H17 | 1.0000 | C1A—C2A | 1.57 (2) |
| C17—C16 | 1.536 (6) | C1A—C3A | 1.50 (2) |
| C9—C13 | 1.540 (6) | C1A—C4A | 1.55 (2) |
| C9—C14 | 1.518 (6) | C1A—C5A | 1.553 (19) |
| C22—H22A | 0.9800 | C2A—H2AA | 0.9800 |
| C22—H22B | 0.9800 | C2A—H2AB | 0.9800 |
| C22—H22C | 0.9800 | C2A—H2AC | 0.9800 |
| C11—H11A | 0.9900 | C3A—H3AA | 0.9800 |
| C11—H11B | 0.9900 | C3A—H3AB | 0.9800 |
| C11—C12 | 1.544 (7) | C3A—H3AC | 0.9800 |
| C20—H20A | 0.9900 | C4A—H4AA | 0.9800 |
| C20—H20B | 0.9900 | C4A—H4AB | 0.9800 |
| C12—H12A | 0.9900 | C4A—H4AC | 0.9800 |
| C12—H12B | 0.9900 | | |

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|---|---|---|---|
| C8—O3—H3 | 109.5 | C7—C6—S1 | 113.1 (4) |
| C15—O4—H4 | 109.5 | C7—C6—S1A | 111.5 (4) |
| C23—C19—C18 | 122.2 (4) | S1—C6—H6AA | 109.0 |
| C23—C19—C20 | 121.2 (4) | S1—C6—H6AB | 109.0 |
| C20—C19—C18 | 116.4 (4) | S1A—C6—H6BC | 109.3 |
| C19—C23—H23 | 117.9 | S1A—C6—H6BD | 109.3 |
| C19—C23—C24 | 124.3 (4) | C9—C13—H13A | 109.5 |
| C24—C23—H23 | 117.9 | C9—C13—H13B | 109.5 |
| C17—C10—H10 | 106.5 | C9—C13—H13C | 109.5 |
| C17—C10—C9 | 113.1 (3) | H13A—C13—H13B | 109.5 |
| C17—C10—C11 | 118.6 (4) | H13A—C13—H13C | 109.5 |
| C9—C10—H10 | 106.5 | H13B—C13—H13C | 109.5 |
| C11—C10—H10 | 106.5 | C26—C25—H25A | 109.6 |
| C11—C10—C9 | 104.9 (4) | C26—C25—H25B | 109.6 |
| H26A—C26—H26B | 107.7 | C24—C25—C26 | 110.4 (4) |
| C18—C26—H26A | 108.8 | C24—C25—H25A | 109.6 |
| C18—C26—H26B | 108.8 | C24—C25—H25B | 109.6 |
| C25—C26—H26A | 108.8 | H25A—C25—H25B | 108.1 |
| C25—C26—H26B | 108.8 | C15—C14—H14A | 108.8 |
| C25—C26—C18 | 113.8 (4) | C15—C14—H14B | 108.8 |
| H21A—C21—H21B | 107.8 | C9—C14—C15 | 113.8 (3) |
| C17—C21—H21A | 109.0 | C9—C14—H14A | 108.8 |
| C17—C21—H21B | 109.0 | C9—C14—H14B | 108.8 |
| C17—C21—C20 | 112.8 (3) | H14A—C14—H14B | 107.7 |
| C20—C21—H21A | 109.0 | O2—C7—C8 | 121.7 (5) |
| C20—C21—H21B | 109.0 | O2—C7—C6 | 120.7 (5) |
| C19—C18—C26 | 109.7 (3) | C6—C7—C8 | 117.5 (4) |
| C19—C18—C22 | 106.9 (3) | C18—C16—H16 | 104.1 |

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| C19—C18—C16 | 107.4 (3) | C15—C16—C18 | 114.2 (3) |
| C26—C18—C22 | 109.7 (3) | C15—C16—H16 | 104.1 |
| C26—C18—C16 | 109.1 (3) | C17—C16—C18 | 113.6 (3) |
| C22—C18—C16 | 113.9 (3) | C17—C16—C15 | 114.9 (3) |
| O5—C24—C23 | 122.3 (4) | C17—C16—H16 | 104.1 |
| O5—C24—C25 | 121.1 (5) | C5—S1—C6 | 98.4 (2) |
| C23—C24—C25 | 116.6 (4) | C4—C1—C2 | 110.8 (8) |
| O4—C15—H15 | 107.3 | C4—C1—C3 | 109.7 (6) |
| O4—C15—C14 | 111.2 (3) | C4—C1—C5 | 109.0 (7) |
| O4—C15—C16 | 110.5 (3) | C3—C1—C2 | 109.0 (9) |
| C14—C15—H15 | 107.3 | C5—C1—C2 | 105.2 (7) |
| C14—C15—C16 | 113.0 (3) | C5—C1—C3 | 113.1 (5) |
| C16—C15—H15 | 107.3 | C1—C2—H2A | 109.5 |
| O3—C8—C9 | 107.5 (3) | C1—C2—H2B | 109.5 |
| O3—C8—C12 | 112.0 (4) | C1—C2—H2C | 109.5 |
| O3—C8—C7 | 109.5 (4) | H2A—C2—H2B | 109.5 |
| C12—C8—C9 | 103.3 (4) | H2A—C2—H2C | 109.5 |
| C7—C8—C9 | 112.7 (4) | H2B—C2—H2C | 109.5 |
| C7—C8—C12 | 111.7 (4) | C1—C4—H4A | 109.5 |
| C10—C17—C21 | 109.9 (3) | C1—C4—H4B | 109.5 |
| C10—C17—H17 | 109.5 | C1—C4—H4C | 109.5 |
| C10—C17—C16 | 108.8 (3) | H4A—C4—H4B | 109.5 |
| C21—C17—H17 | 109.5 | H4A—C4—H4C | 109.5 |
| C21—C17—C16 | 109.5 (3) | H4B—C4—H4C | 109.5 |
| C16—C17—H17 | 109.5 | C1—C3—H3A | 109.5 |
| C10—C9—C8 | 100.3 (3) | C1—C3—H3B | 109.5 |
| C13—C9—C10 | 112.3 (4) | C1—C3—H3C | 109.5 |
| C13—C9—C8 | 108.7 (3) | H3A—C3—H3B | 109.5 |
| C14—C9—C10 | 106.9 (3) | H3A—C3—H3C | 109.5 |
| C14—C9—C8 | 116.2 (4) | H3B—C3—H3C | 109.5 |
| C14—C9—C13 | 112.0 (3) | O1—C5—S1 | 121.9 (4) |
| C18—C22—H22A | 109.5 | O1—C5—C1 | 121.4 (5) |
| C18—C22—H22B | 109.5 | C1—C5—S1 | 116.6 (4) |
| C18—C22—H22C | 109.5 | C5A—S1A—C6 | 105.7 (6) |
| H22A—C22—H22B | 109.5 | C3A—C1A—C2A | 108 (3) |
| H22A—C22—H22C | 109.5 | C3A—C1A—C4A | 113.4 (19) |
| H22B—C22—H22C | 109.5 | C3A—C1A—C5A | 113.4 (17) |
| C10—C11—H11A | 110.8 | C4A—C1A—C2A | 105 (3) |
| C10—C11—H11B | 110.8 | C5A—C1A—C2A | 105 (2) |
| C10—C11—C12 | 104.6 (4) | C5A—C1A—C4A | 110.7 (17) |
| H11A—C11—H11B | 108.9 | C1A—C2A—H2AA | 109.5 |
| C12—C11—H11A | 110.8 | C1A—C2A—H2AB | 109.5 |
| C12—C11—H11B | 110.8 | C1A—C2A—H2AC | 109.5 |
| C19—C20—C21 | 113.3 (4) | H2AA—C2A—H2AB | 109.5 |
| C19—C20—H20A | 108.9 | H2AA—C2A—H2AC | 109.5 |
| C19—C20—H20B | 108.9 | H2AB—C2A—H2AC | 109.5 |
| C21—C20—H20A | 108.9 | C1A—C3A—H3AA | 109.5 |
| C21—C20—H20B | 108.9 | C1A—C3A—H3AB | 109.5 |

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| H20A—C20—H20B | 107.7 | C1A—C3A—H3AC | 109.5 |
| C8—C12—H12A | 110.3 | H3AA—C3A—H3AB | 109.5 |
| C8—C12—H12B | 110.3 | H3AA—C3A—H3AC | 109.5 |
| C11—C12—C8 | 107.0 (4) | H3AB—C3A—H3AC | 109.5 |
| C11—C12—H12A | 110.3 | C1A—C4A—H4AA | 109.5 |
| C11—C12—H12B | 110.3 | C1A—C4A—H4AB | 109.5 |
| H12A—C12—H12B | 108.6 | C1A—C4A—H4AC | 109.5 |
| H6AA—C6—H6AB | 107.8 | H4AA—C4A—H4AB | 109.5 |
| H6BC—C6—H6BD | 108.0 | H4AA—C4A—H4AC | 109.5 |
| C7—C6—H6AA | 109.0 | H4AB—C4A—H4AC | 109.5 |
| C7—C6—H6AB | 109.0 | O1A—C5A—S1A | 119.5 (13) |
| C7—C6—H6BC | 109.3 | O1A—C5A—C1A | 121.7 (15) |
| C7—C6—H6BD | 109.3 | C1A—C5A—S1A | 118.6 (12) |

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| O3—C8—C9—C10 | 78.4 (4) | C11—C10—C9—C13 | −71.3 (4) |
| O3—C8—C9—C13 | −163.7 (4) | C11—C10—C9—C14 | 165.5 (3) |
| O3—C8—C9—C14 | −36.2 (5) | C20—C19—C23—C24 | 169.9 (4) |
| O3—C8—C12—C11 | −92.8 (4) | C20—C19—C18—C26 | 169.2 (4) |
| O3—C8—C7—O2 | −150.6 (4) | C20—C19—C18—C22 | −72.0 (4) |
| O3—C8—C7—C6 | 33.2 (5) | C20—C19—C18—C16 | 50.7 (5) |
| O4—C15—C14—C9 | 77.5 (4) | C20—C21—C17—C10 | −172.5 (3) |
| O4—C15—C16—C18 | 51.7 (4) | C20—C21—C17—C16 | −53.0 (5) |
| O4—C15—C16—C17 | −82.2 (4) | C12—C8—C9—C10 | −40.1 (4) |
| O5—C24—C25—C26 | −147.6 (4) | C12—C8—C9—C13 | 77.8 (4) |
| C19—C23—C24—O5 | 177.9 (4) | C12—C8—C9—C14 | −154.8 (4) |
| C19—C23—C24—C25 | −4.1 (7) | C12—C8—C7—O2 | −25.9 (6) |
| C19—C18—C16—C15 | 170.8 (3) | C12—C8—C7—C6 | 157.8 (4) |
| C19—C18—C16—C17 | −54.7 (4) | C6—S1—C5—O1 | −8.4 (6) |
| C23—C19—C18—C26 | −14.2 (5) | C6—S1—C5—C1 | 169.9 (5) |
| C23—C19—C18—C22 | 104.7 (5) | C6—S1A—C5A—O1A | 4 (2) |
| C23—C19—C18—C16 | −132.7 (4) | C6—S1A—C5A—C1A | 179.1 (15) |
| C23—C19—C20—C21 | 133.4 (4) | C13—C9—C14—C15 | −67.5 (5) |
| C23—C24—C25—C26 | 34.4 (6) | C25—C26—C18—C19 | 45.3 (5) |
| C10—C17—C16—C18 | 177.5 (3) | C25—C26—C18—C22 | −71.9 (4) |
| C10—C17—C16—C15 | −48.4 (4) | C25—C26—C18—C16 | 162.7 (3) |
| C10—C9—C14—C15 | 55.9 (4) | C14—C15—C16—C18 | 177.0 (3) |
| C10—C11—C12—C8 | 4.4 (5) | C14—C15—C16—C17 | 43.2 (5) |
| C26—C18—C16—C15 | 52.0 (4) | C7—C8—C9—C10 | −160.8 (4) |
| C26—C18—C16—C17 | −173.5 (3) | C7—C8—C9—C13 | −42.9 (5) |
| C21—C17—C16—C18 | 57.3 (4) | C7—C8—C9—C14 | 84.5 (5) |
| C21—C17—C16—C15 | −168.5 (3) | C7—C8—C12—C11 | 143.9 (4) |
| C18—C19—C23—C24 | −6.6 (7) | C7—C6—S1—C5 | −90.2 (4) |
| C18—C19—C20—C21 | −49.9 (5) | C7—C6—S1A—C5A | 81.0 (8) |
| C18—C26—C25—C24 | −56.1 (5) | C16—C15—C14—C9 | −47.5 (5) |
| C8—C9—C14—C15 | 166.8 (3) | S1—C6—C7—O2 | 30.1 (6) |
| C17—C10—C9—C8 | 174.8 (3) | S1—C6—C7—C8 | −153.6 (3) |
| C17—C10—C9—C13 | 59.5 (5) | C2—C1—C5—S1 | −106.9 (8) |
| C17—C10—C9—C14 | −63.7 (4) | C2—C1—C5—O1 | 71.4 (10) |

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| C17—C10—C11—C12 | −158.0 (4) | C4—C1—C5—S1 | 134.2 (6) |
| C17—C21—C20—C19 | 49.7 (5) | C4—C1—C5—O1 | −47.5 (9) |
| C9—C10—C17—C21 | 179.7 (3) | C3—C1—C5—S1 | 11.9 (7) |
| C9—C10—C17—C16 | 59.8 (4) | C3—C1—C5—O1 | −169.8 (6) |
| C9—C10—C11—C12 | −30.4 (4) | S1A—C6—C7—O2 | −27.2 (6) |
| C9—C8—C12—C11 | 22.6 (5) | S1A—C6—C7—C8 | 149.1 (4) |
| C9—C8—C7—O2 | 89.9 (5) | C2A—C1A—C5A—S1A | 92 (3) |
| C9—C8—C7—C6 | −86.4 (5) | C2A—C1A—C5A—O1A | −93 (3) |
| C22—C18—C16—C15 | −70.9 (4) | C3A—C1A—C5A—S1A | −149.7 (18) |
| C22—C18—C16—C17 | 63.5 (4) | C3A—C1A—C5A—O1A | 25 (3) |
| C11—C10—C17—C21 | −56.7 (5) | C4A—C1A—C5A—S1A | −21 (2) |
| C11—C10—C17—C16 | −176.6 (4) | C4A—C1A—C5A—O1A | 154 (2) |
| C11—C10—C9—C8 | 44.0 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O5$^{i}$ | 0.84 | 1.96 | 2.802 (4) | 175 |

Symmetry code: (i) −*x*+1/2, −*y*+1, *z*+1/2.