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# 2-Isopropyl-5-methylcyclohexyl 5-acetoxy-1,3-oxathiolane-2-carboxylate

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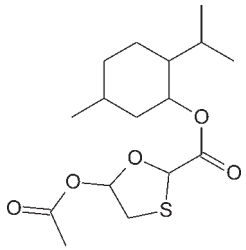
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.028; $wR$ factor = 0.053; data-to-parameter ratio = 13.1.

In the title compound, $C_{16}H_{26}O_5S$, the oxathiolane ring adopts an envelope conformation, with the S atom 0.793 (3) Å out of the mean plane of the remaining four atoms. The cyclohexane ring of the menthol fragment adopts an almost ideal chair conformation, with all substituents in the equatorial positions. In the crystal, relatively strong, short and linear C—H···O hydrogen bonds link the molecules into the chains along [100] direction. The chains are packed into the crystal structure by means of weak dispersive interactions. Intermolecular C—H···S interactions are also observed.

## Related literature

The title compound is a drug intermediate of lamivudine, a reverse transcriptase inhibitor used in the treatment of HIV infections. For the structures of lamivudine and its hydrate have been studied, see: Harris *et al.* (1997). For the identification of lamivudine conformers by Raman scattering measurements and quantum chemical calculations, see: Pereira *et al.* (2007). For asymmetry parameters, see: Duax & Norton (1975). For a description of the Cambridge Structural Database, see: Allen (2002).



## Experimental

### Crystal data

$C_{16}H_{26}O_5S$
$M_r$ = 330.43
Orthorhombic, $P2_12_12_1$
$a$ = 5.329 (1) Å
$b$ = 13.867 (1) Å
$c$ = 23.490 (2) Å
$V$ = 1735.8 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.21 mm$^{-1}$
$T$ = 100 K
0.3 × 0.3 × 0.15 mm

### Data collection

Oxford Diffraction Xcalibur Sapphire2 large Be window diffractometer
Absorption correction: multi-scan (*CrysAlis Pro*; Oxford Diffraction, 2009)
$T_{min}$ = 0.719, $T_{max}$ = 1.000
11503 measured reflections
3632 independent reflections
3175 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.025

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.053
$S$ = 1.03
3632 reflections
277 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$
Absolute structure: Flack (1983), 1435 Friedel pairs
Flack parameter: −0.04 (5)

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C13—H13···O12$^{i}$ | 0.989 (15) | 2.261 (15) | 3.1563 (18) | 150.0 (12) |
| C15—H15$A$···S14$^{ii}$ | 0.961 (16) | 3.033 (15) | 3.7794 (15) | 135.6 (11) |
| C20—H20$B$···O19$^{i}$ | 0.961 (18) | 2.524 (18) | 3.464 (2) | 166.0 (14) |

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

Data collection: *CrysAlis Pro* (Oxford Diffraction, 2009); cell refinement: *CrysAlis Pro*; data reduction: *CrysAlis Pro*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989); software used to prepare material for publication: *SHELXL97*.

CSC thanks the University of Mysore for research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2112).

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# supporting information

*Acta Cryst.* (2009). E**65**, o3169 [doi:10.1107/S1600536809046492]

## 2-Isopropyl-5-methylcyclohexyl 5-acetoxy-1,3-oxathiolane-2-carboxylate

**Grzegorz Dutkiewicz, C. S. Chidan Kumar, H. S. Yathirajan, A. N. Mayekar and Maciej Kubicki**

### S1. Comment

5-Methyl-2-(propan-2-yl)cyclohexyl 5-(acetyloxy)-1,3-oxathiolane-2-carboxylate (I, Scheme 1) is a drug intermediate of lamivudine which is a reverse transcriptase inhibitor used in the treatment of HIV infection alone or in combination with other class of Anti HIV drugs. The crystal stractures of Lamivudine and its hydrate have been studied (Harris *et al.*, 1997). The identification of lamivudine conformers by Raman scattering measurements and quantum chemical calculations is reported (Pereira *et al.*, 2007).

The conformation of the oxathiolane ring is close to an envelope (Fig. 1), with four atoms C13, C15, C16 and O17 almost coplanar (maximum deviation from the least-squares plane of 0.0469 (11) Å) while the fifth atom (S14) is significantly, by 0.793 (3) Å out of this plane. Also the asymmetry parameter (Duax & Norton, 1975), which describes the deviation from the ideal symmetry (in this case $C_s$), has relatively low value of 6.0°. Similar conformation was observed in the majority of the structures with not fused oxathiolane rings found in the Cambridge Structural Database (Allen, 2002), however different atoms occupy the out-of-plane position. The acetyloxy substituent occupies the *quasi*-axial position with respect to the oxathiolane ring (C13—O17—C16—O18 torsion angle is -109.18 (13) °, S14—C15—C16—O18 84.13 (12) °), the position of carboxylate group is also close to the axial one (C16—O17—C13—C12 -97.66 (14) °, C15—S14—C13—C12 84.89 (10) °). The cyclohexyl ring is close to the typical chair conformation (maximum and minimum values of the asymmetry parameters are 0.74° for $\Delta C_s^5$, and 3.96° $\Delta C_2^{1-6}$), all substituents: methyl, isopropyl and carboxylate are in equatorial positions.

In the crystal structure relatively strong (short and directional) C13—H13···O12$^{i}$ hydrogen bonds join the molecules into the infinite chains along [100]. These chain in turn are organized into the crystal structure by weak van der Waals - type interactions (Table 1, Fig. 2).

### S2. Experimental

To a mixture of *L*-menthyl-5-hydroxy-1,3-oxathiolane-2-carboxylate (1.5 g, 5.2 m mol) in pyridine (30 ml), acetic anhydride (6.4 ml) was added slowly at 273 K (Fig. 4). The mixture was allowed to attain room temperature and stirred over night, then quenched to ice cold water and extracted with ethyl acetate. The organic layer was concentrated under vacuum to obtain the product. X-ray quality crystals were grown from slow evaporation of methanol solution (m.p.: 333–335 K).

### S3. Refinement

Positional parameters of the hydrogen atoms were freely refined, the $U_{iso}$ values of these atoms were set at 1.2 (1.5 for methyl groups) times $U_{eq}$ of their carrier carbon atom.

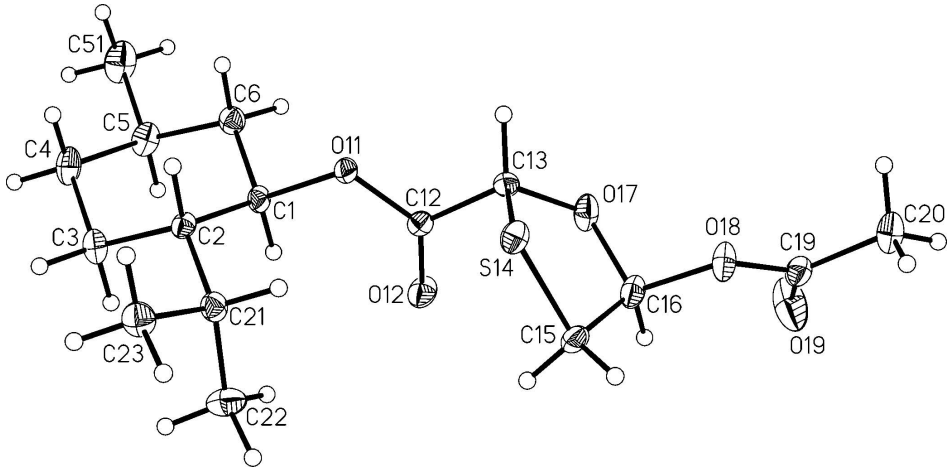


**Figure 1**

Anisotropic ellipsoid representation of the compound **I** together with atom labelling scheme. The ellipsoids are drawn at 50% probability level, hydrogen atoms are depicted as spheres with arbitrary radii.

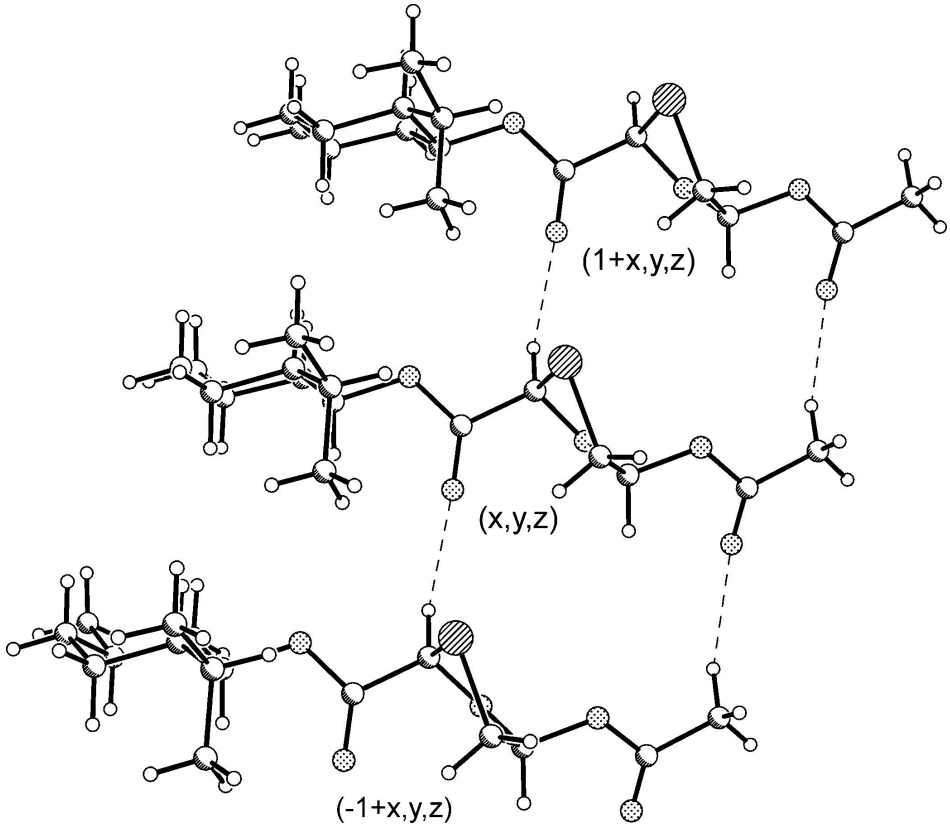


**Figure 2**

The hydrogen-bonded chain of molecules of **I**. Hydrogen bonds are shown as dashed lines.

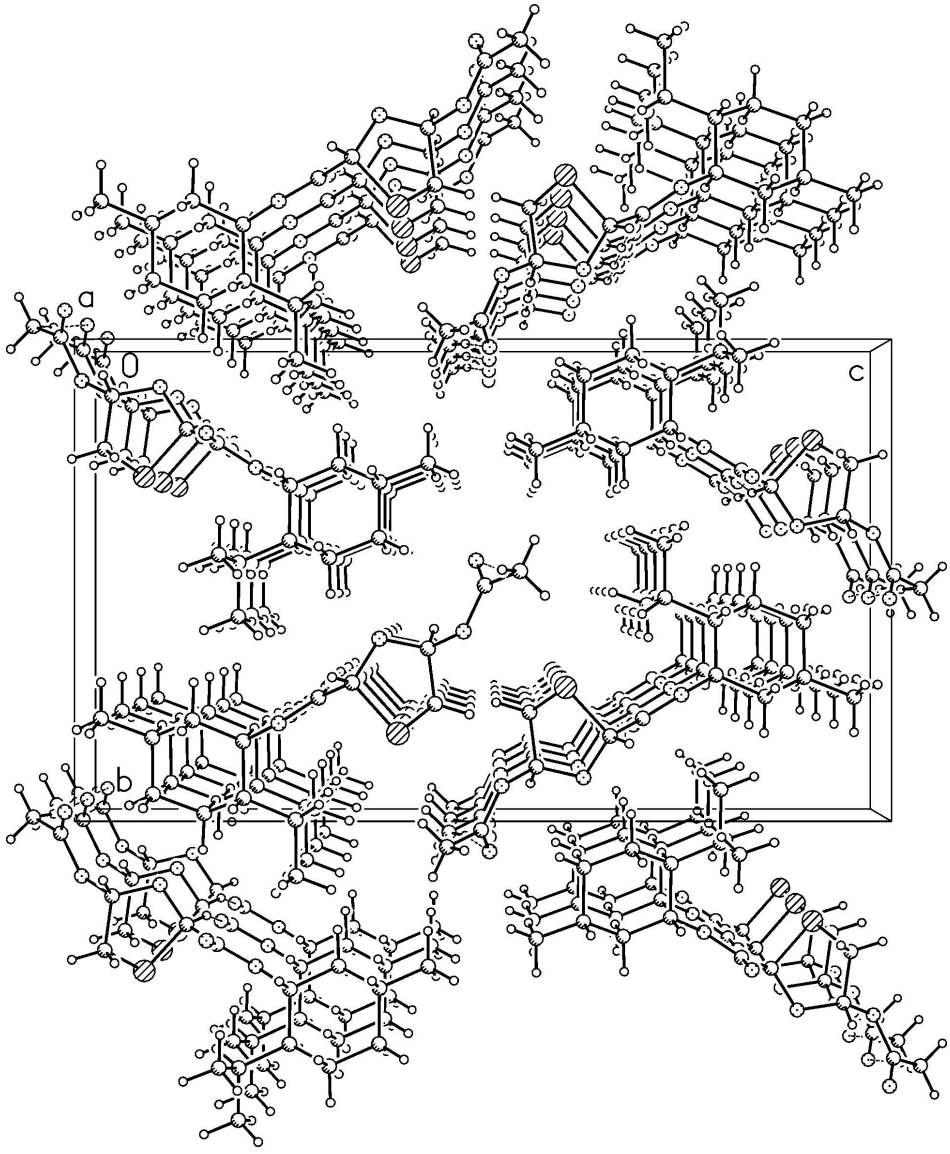


**Figure 3**

The crystal packing as seen along the chain direction, *i.e.* along [100].

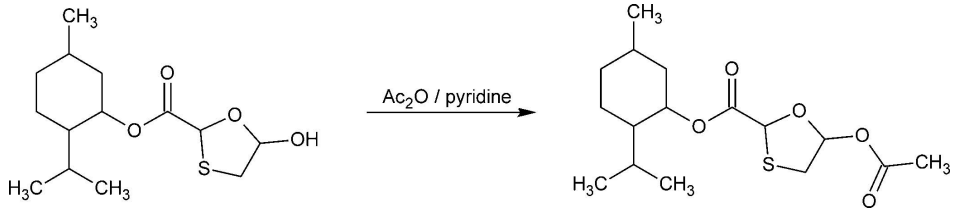


**Figure 4**

The preparation of the title compound.

**2-Isopropyl-5-methylcyclohexyl 5-acetoxy-1,3-oxathiolane-2-carboxylate**

*Crystal data*

$C_{16}H_{26}O_5S$
$M_r = 330.43$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.329$ (1) Å
$b = 13.867$ (1) Å
$c = 23.490$ (2) Å
$V = 1735.8$ (4) Å$^3$
$Z = 4$
$F(000) = 712$
$D_x = 1.264$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3953 reflections
$\theta = 3.0$–$75.3°$
$\mu = 0.21$ mm$^{-1}$
$T = 100$ K
Prism, colourless
0.3 × 0.3 × 0.15 mm

*Data collection*

Oxford Diffraction Xcalibur Sapphire2 large Be window diffractometer
Radiation source: Nova (Mo) X-ray Source
Graphite monochromator
Detector resolution: 5.2679 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min} = 0.719$, $T_{max} = 1.000$
11503 measured reflections
3632 independent reflections
3175 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 27.8°$, $\theta_{min} = 2.9°$
$h = -6 \rightarrow 6$
$k = -17 \rightarrow 19$
$l = -18 \rightarrow 28$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.053$
$S = 1.03$
3632 reflections
277 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.026P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ e Å$^{-3}$
Absolute structure: Flack (1983), 1435 Friedel pairs
Absolute structure parameter: −0.04 (5)

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.2221 (3) | 0.81153 (11) | 0.22603 (6) | 0.0144 (3) |
| H1 | 0.055 (3) | 0.7890 (12) | 0.2363 (5) | 0.017* |
| C2 | 0.2271 (3) | 0.92125 (11) | 0.22689 (6) | 0.0133 (3) |

| | | | | |
|---|---|---|---|---|
| H2 | 0.402 (3) | 0.9383 (11) | 0.2176 (6) | 0.016* |
| C21 | 0.1670 (3) | 0.96521 (11) | 0.28540 (6) | 0.0163 (3) |
| H21 | 0.277 (3) | 0.9324 (11) | 0.3133 (7) | 0.020* |
| C22 | −0.1041 (3) | 0.95024 (13) | 0.30376 (8) | 0.0248 (4) |
| H22A | −0.139 (3) | 0.9755 (13) | 0.3442 (8) | 0.037* |
| H22B | −0.154 (3) | 0.8837 (14) | 0.3016 (7) | 0.037* |
| H22C | −0.216 (3) | 0.9878 (14) | 0.2804 (8) | 0.037* |
| C23 | 0.2308 (3) | 1.07241 (12) | 0.28675 (7) | 0.0207 (4) |
| H23A | 0.126 (3) | 1.1091 (13) | 0.2621 (7) | 0.031* |
| H23B | 0.396 (3) | 1.0834 (12) | 0.2739 (7) | 0.031* |
| H23C | 0.207 (3) | 1.0990 (12) | 0.3264 (7) | 0.031* |
| C3 | 0.0559 (3) | 0.95554 (11) | 0.17848 (6) | 0.0193 (3) |
| H3A | −0.122 (3) | 0.9338 (12) | 0.1888 (7) | 0.023* |
| H3B | 0.049 (3) | 1.0270 (12) | 0.1771 (6) | 0.023* |
| C4 | 0.1377 (3) | 0.91614 (12) | 0.12123 (7) | 0.0222 (4) |
| H4A | 0.300 (3) | 0.9392 (12) | 0.1134 (7) | 0.027* |
| H4B | 0.025 (3) | 0.9406 (12) | 0.0913 (7) | 0.027* |
| C5 | 0.1436 (3) | 0.80595 (12) | 0.12053 (6) | 0.0199 (4) |
| H5 | −0.025 (3) | 0.7843 (12) | 0.1282 (6) | 0.024* |
| C51 | 0.2389 (4) | 0.76739 (13) | 0.06413 (8) | 0.0305 (4) |
| H51A | 0.398 (4) | 0.7932 (14) | 0.0557 (7) | 0.046* |
| H51B | 0.137 (3) | 0.7914 (15) | 0.0335 (8) | 0.046* |
| H51C | 0.245 (3) | 0.6975 (14) | 0.0650 (8) | 0.046* |
| C6 | 0.3052 (3) | 0.76948 (11) | 0.16982 (6) | 0.0172 (3) |
| H6A | 0.478 (3) | 0.7877 (12) | 0.1612 (6) | 0.021* |
| H6B | 0.305 (3) | 0.6970 (12) | 0.1714 (6) | 0.021* |
| O11 | 0.39944 (16) | 0.77425 (7) | 0.26904 (4) | 0.0146 (2) |
| C12 | 0.3058 (3) | 0.72616 (10) | 0.31299 (6) | 0.0134 (3) |
| O12 | 0.08816 (18) | 0.70999 (9) | 0.32180 (4) | 0.0214 (2) |
| C13 | 0.5134 (3) | 0.69492 (11) | 0.35302 (6) | 0.0146 (3) |
| H13 | 0.672 (3) | 0.6830 (11) | 0.3325 (6) | 0.018* |
| S14 | 0.56357 (7) | 0.79072 (3) | 0.404699 (15) | 0.01686 (9) |
| C15 | 0.3109 (3) | 0.73820 (11) | 0.44548 (6) | 0.0176 (3) |
| H15A | 0.327 (3) | 0.7581 (11) | 0.4845 (7) | 0.021* |
| H15B | 0.150 (3) | 0.7576 (11) | 0.4298 (7) | 0.021* |
| C16 | 0.3439 (3) | 0.63111 (12) | 0.43767 (6) | 0.0189 (3) |
| H16 | 0.175 (3) | 0.5948 (11) | 0.4441 (6) | 0.023* |
| O17 | 0.4379 (2) | 0.61176 (7) | 0.38320 (4) | 0.0205 (2) |
| O18 | 0.52454 (18) | 0.59729 (7) | 0.47832 (4) | 0.0206 (2) |
| C19 | 0.4838 (3) | 0.50989 (11) | 0.50215 (6) | 0.0193 (3) |
| O19 | 0.3034 (2) | 0.46223 (9) | 0.49229 (6) | 0.0418 (4) |
| C20 | 0.6923 (3) | 0.48274 (13) | 0.54115 (7) | 0.0252 (4) |
| H20A | 0.709 (3) | 0.5287 (14) | 0.5686 (8) | 0.038* |
| H20B | 0.850 (3) | 0.4807 (13) | 0.5212 (8) | 0.038* |
| H20C | 0.659 (3) | 0.4227 (13) | 0.5576 (7) | 0.038* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0124 (7) | 0.0178 (9) | 0.0129 (8) | −0.0003 (7) | −0.0033 (6) | 0.0033 (6) |
| C2 | 0.0115 (7) | 0.0157 (8) | 0.0127 (8) | −0.0026 (6) | −0.0008 (6) | 0.0021 (6) |
| C21 | 0.0179 (8) | 0.0171 (8) | 0.0137 (8) | 0.0017 (7) | −0.0020 (6) | 0.0002 (6) |
| C22 | 0.0232 (9) | 0.0248 (10) | 0.0265 (10) | −0.0013 (8) | 0.0080 (8) | −0.0066 (7) |
| C23 | 0.0247 (9) | 0.0181 (9) | 0.0192 (10) | 0.0005 (7) | −0.0012 (7) | −0.0027 (7) |
| C3 | 0.0246 (9) | 0.0149 (8) | 0.0184 (8) | 0.0007 (8) | −0.0050 (7) | 0.0025 (6) |
| C4 | 0.0310 (10) | 0.0200 (9) | 0.0157 (8) | −0.0019 (7) | −0.0068 (7) | 0.0038 (7) |
| C5 | 0.0255 (8) | 0.0188 (9) | 0.0155 (8) | −0.0053 (7) | −0.0037 (6) | −0.0011 (7) |
| C51 | 0.0474 (12) | 0.0260 (11) | 0.0180 (9) | −0.0037 (10) | −0.0045 (8) | −0.0035 (8) |
| C6 | 0.0198 (8) | 0.0142 (9) | 0.0175 (8) | 0.0005 (7) | 0.0004 (6) | −0.0005 (6) |
| O11 | 0.0124 (5) | 0.0178 (6) | 0.0137 (5) | 0.0008 (5) | −0.0006 (4) | 0.0037 (4) |
| C12 | 0.0159 (8) | 0.0115 (8) | 0.0126 (8) | −0.0005 (6) | 0.0010 (6) | −0.0021 (6) |
| O12 | 0.0138 (5) | 0.0287 (6) | 0.0218 (6) | −0.0059 (5) | −0.0009 (4) | 0.0072 (5) |
| C13 | 0.0158 (8) | 0.0156 (8) | 0.0124 (7) | 0.0031 (6) | 0.0013 (5) | −0.0005 (6) |
| S14 | 0.01923 (18) | 0.01732 (18) | 0.01403 (18) | −0.00361 (17) | −0.00206 (15) | 0.00048 (16) |
| C15 | 0.0176 (8) | 0.0250 (9) | 0.0101 (8) | 0.0018 (7) | 0.0015 (6) | 0.0009 (6) |
| C16 | 0.0186 (8) | 0.0236 (9) | 0.0145 (8) | −0.0040 (8) | −0.0029 (6) | 0.0058 (7) |
| O17 | 0.0326 (6) | 0.0136 (5) | 0.0153 (5) | 0.0006 (5) | −0.0039 (5) | 0.0012 (4) |
| O18 | 0.0229 (6) | 0.0205 (6) | 0.0183 (5) | −0.0065 (5) | −0.0073 (4) | 0.0079 (4) |
| C19 | 0.0224 (8) | 0.0180 (8) | 0.0174 (8) | −0.0002 (7) | 0.0028 (6) | 0.0046 (6) |
| O19 | 0.0291 (6) | 0.0330 (8) | 0.0632 (9) | −0.0137 (6) | −0.0157 (6) | 0.0248 (7) |
| C20 | 0.0335 (10) | 0.0230 (10) | 0.0193 (9) | −0.0006 (8) | −0.0049 (8) | 0.0050 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O11 | 1.4768 (16) | C51—H51A | 0.94 (2) |
| C1—C6 | 1.510 (2) | C51—H51B | 0.961 (18) |
| C1—C2 | 1.522 (2) | C51—H51C | 0.970 (19) |
| C1—H1 | 0.975 (15) | C6—H6A | 0.975 (15) |
| C2—C3 | 1.533 (2) | C6—H6B | 1.006 (16) |
| C2—C21 | 1.537 (2) | O11—C12 | 1.3264 (16) |
| C2—H2 | 0.986 (15) | C12—O12 | 1.1991 (16) |
| C21—C22 | 1.522 (2) | C12—C13 | 1.5155 (19) |
| C21—C23 | 1.525 (2) | C13—O17 | 1.4122 (17) |
| C21—H21 | 0.990 (16) | C13—S14 | 1.8193 (15) |
| C22—H22A | 1.029 (19) | C13—H13 | 0.989 (15) |
| C22—H22B | 0.961 (19) | S14—C15 | 1.8059 (16) |
| C22—H22C | 0.965 (18) | C15—C16 | 1.507 (2) |
| C23—H23A | 0.951 (17) | C15—H15A | 0.961 (16) |
| C23—H23B | 0.945 (18) | C15—H15B | 0.973 (15) |
| C23—H23C | 1.009 (17) | C16—O17 | 1.4000 (18) |
| C3—C4 | 1.515 (2) | C16—O18 | 1.4345 (17) |
| C3—H3A | 1.024 (16) | C16—H16 | 1.042 (16) |
| C3—H3B | 0.992 (16) | O18—C19 | 1.3526 (17) |
| C4—C5 | 1.528 (2) | C19—O19 | 1.1891 (17) |

| | | | |
|---|---|---|---|
| C4—H4A | 0.941 (18) | C19—C20 | 1.489 (2) |
| C4—H4B | 0.983 (16) | C20—H20A | 0.911 (19) |
| C5—C51 | 1.516 (2) | C20—H20B | 0.961 (18) |
| C5—C6 | 1.529 (2) | C20—H20C | 0.936 (18) |
| C5—H5 | 0.965 (15) | | |

| | | | |
|---|---|---|---|
| O11—C1—C6 | 106.00 (11) | C6—C5—H5 | 106.3 (9) |
| O11—C1—C2 | 109.26 (12) | C5—C51—H51A | 110.6 (11) |
| C6—C1—C2 | 113.12 (13) | C5—C51—H51B | 109.9 (11) |
| O11—C1—H1 | 107.7 (8) | H51A—C51—H51B | 102.8 (15) |
| C6—C1—H1 | 111.1 (8) | C5—C51—H51C | 110.2 (11) |
| C2—C1—H1 | 109.5 (10) | H51A—C51—H51C | 110.8 (17) |
| C1—C2—C3 | 106.85 (12) | H51B—C51—H51C | 112.3 (17) |
| C1—C2—C21 | 113.86 (13) | C1—C6—C5 | 111.69 (12) |
| C3—C2—C21 | 114.59 (13) | C1—C6—H6A | 111.0 (9) |
| C1—C2—H2 | 104.7 (9) | C5—C6—H6A | 106.8 (9) |
| C3—C2—H2 | 108.9 (8) | C1—C6—H6B | 110.7 (9) |
| C21—C2—H2 | 107.4 (8) | C5—C6—H6B | 111.0 (9) |
| C22—C21—C23 | 109.79 (14) | H6A—C6—H6B | 105.5 (13) |
| C22—C21—C2 | 113.39 (13) | C12—O11—C1 | 117.88 (11) |
| C23—C21—C2 | 111.01 (13) | O12—C12—O11 | 126.32 (13) |
| C22—C21—H21 | 108.3 (9) | O12—C12—C13 | 123.07 (13) |
| C23—C21—H21 | 107.6 (9) | O11—C12—C13 | 110.60 (11) |
| C2—C21—H21 | 106.5 (9) | O17—C13—C12 | 109.67 (12) |
| C21—C22—H22A | 112.7 (9) | O17—C13—S14 | 107.65 (9) |
| C21—C22—H22B | 112.2 (10) | C12—C13—S14 | 108.21 (10) |
| H22A—C22—H22B | 109.0 (15) | O17—C13—H13 | 110.7 (9) |
| C21—C22—H22C | 110.8 (11) | C12—C13—H13 | 111.7 (9) |
| H22A—C22—H22C | 103.2 (14) | S14—C13—H13 | 108.8 (9) |
| H22B—C22—H22C | 108.5 (15) | C15—S14—C13 | 87.13 (7) |
| C21—C23—H23A | 112.2 (10) | C16—C15—S14 | 104.23 (11) |
| C21—C23—H23B | 111.0 (11) | C16—C15—H15A | 112.8 (9) |
| H23A—C23—H23B | 105.6 (14) | S14—C15—H15A | 108.9 (9) |
| C21—C23—H23C | 110.4 (10) | C16—C15—H15B | 109.2 (9) |
| H23A—C23—H23C | 107.0 (14) | S14—C15—H15B | 110.3 (9) |
| H23B—C23—H23C | 110.5 (14) | H15A—C15—H15B | 111.1 (13) |
| C4—C3—C2 | 112.04 (14) | O17—C16—O18 | 107.80 (12) |
| C4—C3—H3A | 111.7 (9) | O17—C16—C15 | 109.99 (12) |
| C2—C3—H3A | 106.5 (9) | O18—C16—C15 | 108.64 (12) |
| C4—C3—H3B | 110.0 (8) | O17—C16—H16 | 110.4 (8) |
| C2—C3—H3B | 110.9 (9) | O18—C16—H16 | 109.0 (8) |
| H3A—C3—H3B | 105.5 (13) | C15—C16—H16 | 111.0 (8) |
| C3—C4—C5 | 112.08 (13) | C16—O17—C13 | 113.85 (11) |
| C3—C4—H4A | 108.4 (10) | C19—O18—C16 | 117.42 (11) |
| C5—C4—H4A | 108.6 (10) | O19—C19—O18 | 123.18 (13) |
| C3—C4—H4B | 109.6 (9) | O19—C19—C20 | 125.64 (15) |
| C5—C4—H4B | 110.5 (9) | O18—C19—C20 | 111.18 (13) |
| H4A—C4—H4B | 107.6 (13) | C19—C20—H20A | 109.4 (11) |

| | | | |
|---|---|---|---|
| C51—C5—C4 | 111.65 (14) | C19—C20—H20B | 111.1 (10) |
| C51—C5—C6 | 110.89 (13) | H20A—C20—H20B | 106.0 (16) |
| C4—C5—C6 | 109.53 (13) | C19—C20—H20C | 109.6 (11) |
| C51—C5—H5 | 111.4 (9) | H20A—C20—H20C | 110.5 (16) |
| C4—C5—H5 | 106.9 (10) | H20B—C20—H20C | 110.1 (15) |
| | | | |
| O11—C1—C2—C3 | 175.68 (11) | C1—O11—C12—O12 | −0.5 (2) |
| C6—C1—C2—C3 | 57.86 (16) | C1—O11—C12—C13 | −179.08 (11) |
| O11—C1—C2—C21 | −56.78 (16) | O12—C12—C13—O17 | 27.68 (19) |
| C6—C1—C2—C21 | −174.59 (12) | O11—C12—C13—O17 | −153.67 (11) |
| C1—C2—C21—C22 | −68.34 (17) | O12—C12—C13—S14 | −89.49 (16) |
| C3—C2—C21—C22 | 55.10 (18) | O11—C12—C13—S14 | 89.16 (12) |
| C1—C2—C21—C23 | 167.51 (14) | O17—C13—S14—C15 | −33.57 (10) |
| C3—C2—C21—C23 | −69.05 (17) | C12—C13—S14—C15 | 84.90 (11) |
| C1—C2—C3—C4 | −57.56 (17) | C13—S14—C15—C16 | 37.16 (10) |
| C21—C2—C3—C4 | 175.32 (13) | S14—C15—C16—O17 | −33.65 (15) |
| C2—C3—C4—C5 | 57.98 (18) | S14—C15—C16—O18 | 84.12 (12) |
| C3—C4—C5—C51 | −176.96 (15) | O18—C16—O17—C13 | −109.17 (13) |
| C3—C4—C5—C6 | −53.74 (19) | C15—C16—O17—C13 | 9.12 (17) |
| O11—C1—C6—C5 | −177.46 (12) | C12—C13—O17—C16 | −97.69 (14) |
| C2—C1—C6—C5 | −57.75 (17) | S14—C13—O17—C16 | 19.84 (14) |
| C51—C5—C6—C1 | 176.82 (14) | O17—C16—O18—C19 | −98.38 (14) |
| C4—C5—C6—C1 | 53.16 (17) | C15—C16—O18—C19 | 142.46 (13) |
| C6—C1—O11—C12 | −123.41 (13) | C16—O18—C19—O19 | −2.4 (2) |
| C2—C1—O11—C12 | 114.39 (14) | C16—O18—C19—C20 | 177.29 (13) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C13—H13···O12$^{i}$ | 0.989 (15) | 2.261 (15) | 3.1563 (18) | 150.0 (12) |
| C15—H15*A*···S14$^{ii}$ | 0.961 (16) | 3.033 (15) | 3.7794 (15) | 135.6 (11) |
| C20—H20*B*···O19$^{i}$ | 0.961 (18) | 2.524 (18) | 3.464 (2) | 166.0 (14) |

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