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Methyl 3,4-*O*-isopropylidene-2-*O*-[(methylsulfanyl)thiocarbonyl]-β-L-arabinoside

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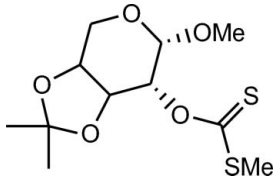
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.100; data-to-parameter ratio = 18.7.

In the title compound, $C_{11}H_{18}O_5S_2$, the six- and five-membered rings adopt a chair and an approximately planar conformation, respectively.

Related literature

For related literature, see: Zhang *et al.* (1999).



Experimental

Crystal data

$C_{11}H_{18}O_5S_2$
M_r = 294.37
Orthorhombic, $P2_12_12_1$
a = 9.1381 (9) Å
b = 11.2898 (11) Å
c = 13.9405 (14) Å
V = 1438.2 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 293 (2) K
0.50 × 0.41 × 0.39 mm

Data collection

Bruker APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.752, T_{max} = 1.000
(expected range = 0.649–0.863)
8467 measured reflections
3115 independent reflections
2487 reflections with $I > 2\sigma(I)$
R_{int} = 0.098

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.099
S = 0.95
3115 reflections
167 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 1306 Friedel pairs
Flack parameter: −0.05 (8)

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2082).

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supporting information

Acta Cryst. (2008). E**64**, o933 [doi:10.1107/S1600536808011458]

Methyl 3,4-*O*-isopropylidene-2-*O*-[(methylsulfanyl)thiocarbonyl]-*β*-L-arabinoside

Hongqi Li, Yanxi Song and Xiumei Li

S1. Comment

The title compound is an important intermediate for synthesis of 2-deoxy-*L*-ribose and was synthesized starting from *L*-arabinose according to the procedures reported by Zhang *et al.* (1999). Herein we present the single-crystal structure of methyl 3,4-*O*-isopropylidene-2-O– [(methylthio)thiocarbonyl]-*β*-*L*-arabinoside.

S2. Experimental

The title compound was prepared from *L*-arabinose according to the procedures reported by Zhang *et al.* (1999). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution in ethyl acetate.

S3. Refinement

All H atoms were placed at calculated positions using a riding model, with C—H = 0.96Å for methyl C—H, 0.97Å for methylene C—H, and 0.98Å for methine C—H and with U_{iso}(H) = 1.2U_{eq}(C) except for methyl groups U_{iso}(H) = 1.5U_{eq}(C).

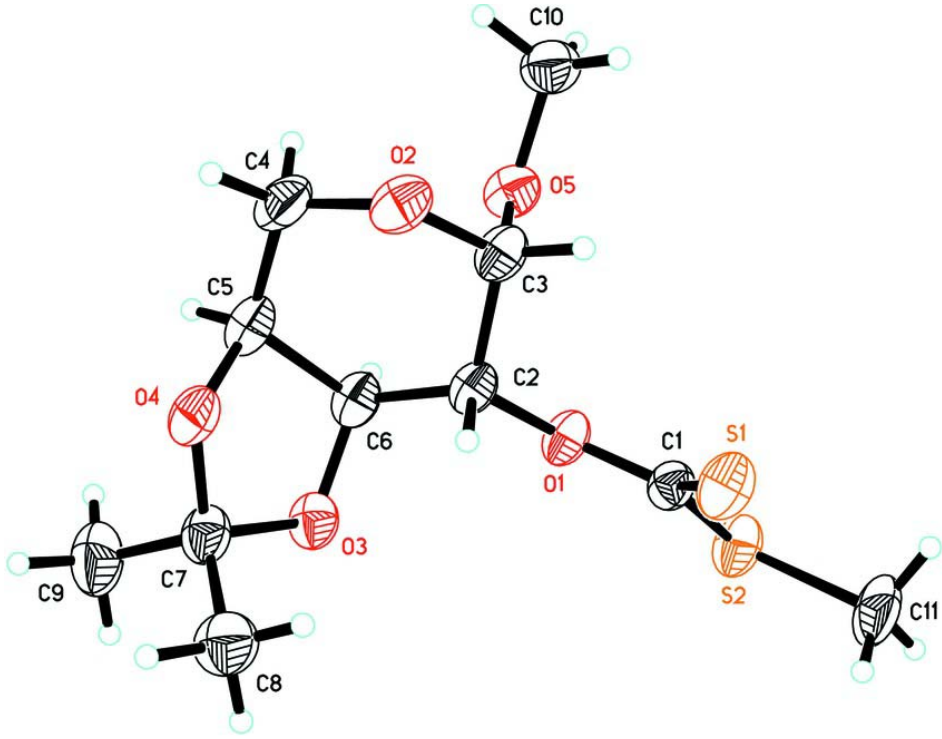

Figure 1

A view of the molecule of the title compound. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

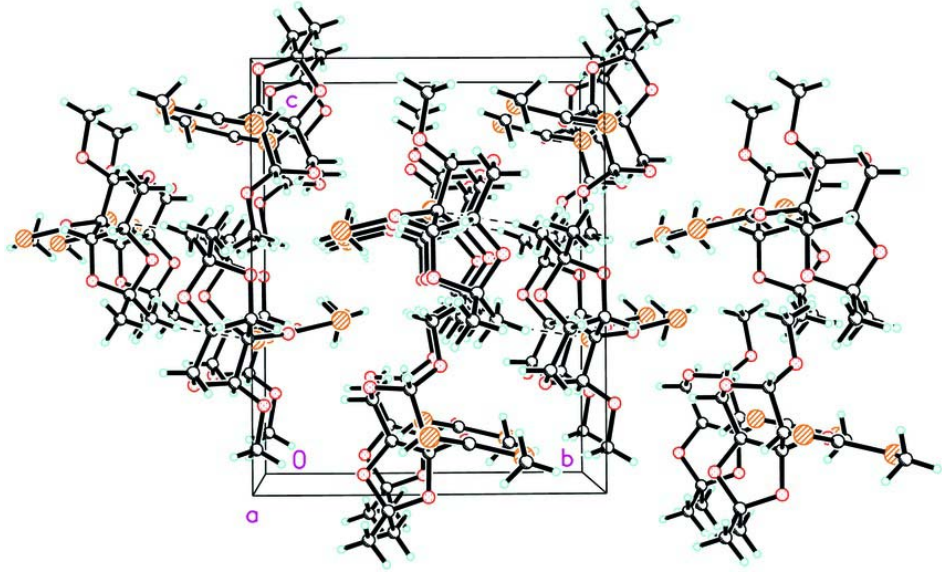

Figure 2

The crystal structure of the title compound, viewed along *a* axis.

Methyl 3,4-*O*-isopropylidene-2-*O*-[(methylsulfanyl)thiocarbonyl]-*β*-L-arabinoside

Crystal data

$C_{11}H_{18}O_5S_2$
$M_r = 294.37$
Orthorhombic, $P2_12_12_1$
$a = 9.1381$ (9) Å
$b = 11.2898$ (11) Å
$c = 13.9405$ (14) Å
$V = 1438.2$ (2) Å^3
$Z = 4$
$F(000) = 624$
$D_x = 1.360$ Mg m^{-3}
Melting point = 403–400 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3384 reflections
$\theta = 4.6$–$47.9°$
$\mu = 0.38$ mm^{-1}
$T = 293$ K
Prismatic, colorless
0.50 × 0.41 × 0.39 mm

Data collection

Bruker APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.752$, $T_{max} = 1.000$
8467 measured reflections
3115 independent reflections
2487 reflections with $I > 2\sigma(I)$
$R_{int} = 0.098$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.3°$
$h = -11 \rightarrow 8$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.099$
$S = 0.95$
3115 reflections
167 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Absolute structure: Flack (1983), 1306 Friedel pairs
Absolute structure parameter: −0.05 (8)

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}
S1	0.98335 (7)	1.00851 (6)	0.84959 (5)	0.0646 (2)
S2	0.91603 (8)	0.75403 (5)	0.89614 (5)	0.0669 (2)
O1	0.72545 (17)	0.90443 (12)	0.86747 (11)	0.0509 (4)

O2	0.5721 (2)	1.16759 (15)	0.75190 (13)	0.0639 (5)
O3	0.51511 (18)	1.00383 (13)	1.00502 (12)	0.0567 (4)
O4	0.44353 (18)	1.18675 (13)	0.95542 (11)	0.0593 (4)
O5	0.5692 (2)	0.97000 (14)	0.70389 (12)	0.0605 (4)
C1	0.8723 (3)	0.90034 (18)	0.86865 (16)	0.0473 (5)
C2	0.6525 (2)	1.01781 (17)	0.85773 (15)	0.0457 (5)
H2	0.7075	1.0776	0.8937	0.055*
C3	0.6417 (3)	1.0560 (2)	0.7547 (2)	0.0544 (6)
H3	0.7408	1.0643	0.7286	0.065*
C4	0.4237 (3)	1.1616 (3)	0.78384 (19)	0.0674 (7)
H4A	0.3675	1.1162	0.7376	0.081*
H4B	0.3837	1.2411	0.7852	0.081*
C5	0.4044 (3)	1.1071 (2)	0.88035 (19)	0.0601 (6)
H5	0.3020	1.0833	0.8883	0.072*
C6	0.5033 (2)	1.00210 (18)	0.90391 (17)	0.0522 (5)
H6	0.4582	0.9280	0.8824	0.063*
C7	0.4676 (3)	1.1180 (2)	1.03915 (17)	0.0576 (6)
C8	0.5872 (3)	1.1738 (2)	1.0974 (2)	0.0746 (8)
H8A	0.6729	1.1831	1.0584	0.112*
H8B	0.6097	1.1240	1.1513	0.112*
H8C	0.5555	1.2499	1.1200	0.112*
C9	0.3288 (3)	1.1016 (3)	1.0968 (2)	0.0898 (10)
H9A	0.2923	1.1776	1.1163	0.115*
H9B	0.3494	1.0546	1.1525	0.115*
H9C	0.2567	1.0624	1.0581	0.115*
C10	0.5657 (3)	0.9914 (3)	0.60277 (18)	0.0719 (7)
H10A	0.4948	1.0517	0.5889	0.108*
H10B	0.5395	0.9198	0.5699	0.108*
H10C	0.6605	1.0171	0.5816	0.108*
C11	1.1125 (3)	0.7582 (3)	0.8980 (2)	0.0867 (10)
H11A	1.1483	0.7774	0.8351	0.110*
H11B	1.1496	0.6822	0.9170	0.110*
H11C	1.1446	0.8173	0.9428	0.110*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0495 (3)	0.0544 (3)	0.0899 (5)	−0.0063 (3)	−0.0028 (3)	0.0012 (3)
S2	0.0618 (4)	0.0451 (3)	0.0938 (5)	0.0156 (3)	0.0026 (4)	0.0094 (4)
O1	0.0464 (8)	0.0338 (7)	0.0726 (10)	0.0039 (6)	−0.0027 (8)	0.0054 (7)
O2	0.0733 (12)	0.0430 (8)	0.0754 (11)	0.0076 (9)	−0.0087 (10)	0.0124 (8)
O3	0.0616 (10)	0.0418 (8)	0.0667 (9)	0.0057 (9)	0.0069 (8)	0.0086 (7)
O4	0.0588 (10)	0.0438 (8)	0.0753 (10)	0.0135 (8)	−0.0043 (9)	0.0031 (8)
O5	0.0642 (11)	0.0504 (8)	0.0668 (10)	0.0004 (8)	−0.0101 (8)	−0.0006 (8)
C1	0.0475 (12)	0.0417 (11)	0.0526 (13)	0.0062 (9)	−0.0033 (10)	−0.0031 (10)
C2	0.0447 (11)	0.0299 (9)	0.0626 (14)	0.0015 (9)	−0.0038 (10)	0.0039 (10)
C3	0.0484 (13)	0.0420 (11)	0.0726 (16)	0.0007 (11)	−0.0045 (11)	0.0051 (11)
C4	0.0679 (17)	0.0574 (14)	0.0767 (18)	0.0202 (15)	−0.0196 (15)	0.0030 (13)

C5	0.0428 (12)	0.0521 (12)	0.0854 (18)	0.0067 (11)	−0.0077 (13)	0.0011 (13)
C6	0.0465 (12)	0.0382 (10)	0.0719 (15)	−0.0009 (11)	−0.0028 (11)	0.0009 (11)
C7	0.0583 (16)	0.0469 (12)	0.0676 (15)	0.0092 (11)	0.0070 (13)	0.0079 (11)
C8	0.0845 (19)	0.0596 (15)	0.0799 (18)	0.0064 (16)	−0.0079 (17)	−0.0018 (14)
C9	0.070 (2)	0.095 (2)	0.104 (2)	0.0147 (18)	0.0279 (18)	0.005 (2)
C10	0.0788 (18)	0.0741 (17)	0.0629 (15)	0.0076 (16)	−0.0103 (15)	−0.0025 (14)
C11	0.0576 (16)	0.085 (2)	0.117 (2)	0.0362 (16)	0.0070 (16)	0.017 (2)

Geometric parameters (Å, º)

S1—C1	1.610 (2)	C4—H4B	0.9700
S2—C1	1.742 (2)	C5—C6	1.526 (3)
S2—C11	1.796 (3)	C5—H5	0.9800
O1—C1	1.343 (3)	C6—H6	0.9800
O1—C2	1.450 (2)	C7—C8	1.501 (4)
O2—C3	1.412 (3)	C7—C9	1.513 (4)
O2—C4	1.429 (3)	C8—H8A	0.9600
O3—C6	1.414 (3)	C8—H8B	0.9600
O3—C7	1.441 (3)	C8—H8C	0.9600
O4—C7	1.419 (3)	C9—H9A	0.9600
O4—C5	1.426 (3)	C9—H9B	0.9600
O5—C3	1.372 (3)	C9—H9C	0.9600
O5—C10	1.431 (3)	C10—H10A	0.9600
C2—C3	1.503 (3)	C10—H10B	0.9600
C2—C6	1.518 (3)	C10—H10C	0.9600
C2—H2	0.9800	C11—H11A	0.9600
C3—H3	0.9800	C11—H11B	0.9600
C4—C5	1.490 (4)	C11—H11C	0.9600
C4—H4A	0.9700		
C1—S2—C11	101.97 (14)	C2—C6—C5	110.47 (18)
C1—O1—C2	119.41 (16)	O3—C6—H6	110.4
C3—O2—C4	112.1 (2)	C2—C6—H6	110.4
C6—O3—C7	108.58 (16)	C5—C6—H6	110.4
C7—O4—C5	107.30 (18)	O4—C7—O3	105.34 (18)
C3—O5—C10	113.5 (2)	O4—C7—C8	109.2 (2)
O1—C1—S1	127.00 (16)	O3—C7—C8	109.6 (2)
O1—C1—S2	105.33 (15)	O4—C7—C9	111.9 (2)
S1—C1—S2	127.66 (14)	O3—C7—C9	108.6 (2)
O1—C2—C3	111.91 (17)	C8—C7—C9	112.0 (2)
O1—C2—C6	105.68 (16)	C7—C8—H8A	109.5
C3—C2—C6	112.33 (18)	C7—C8—H8B	109.5
O1—C2—H2	108.9	H8A—C8—H8B	109.5
C3—C2—H2	108.9	C7—C8—H8C	109.5
C6—C2—H2	108.9	H8A—C8—H8C	109.5
O5—C3—O2	113.57 (19)	H8B—C8—H8C	109.5
O5—C3—C2	108.75 (18)	C7—C9—H9A	109.5
O2—C3—C2	108.2 (2)	C7—C9—H9B	109.5

O5—C3—H3	108.8	H9A—C9—H9B	109.5
O2—C3—H3	108.8	C7—C9—H9C	109.5
C2—C3—H3	108.8	H9A—C9—H9C	109.5
O2—C4—C5	114.4 (2)	H9B—C9—H9C	109.5
O2—C4—H4A	108.7	O5—C10—H10A	109.5
C5—C4—H4A	108.7	O5—C10—H10B	109.5
O2—C4—H4B	108.7	H10A—C10—H10B	109.5
C5—C4—H4B	108.7	O5—C10—H10C	109.5
H4A—C4—H4B	107.6	H10A—C10—H10C	109.5
O4—C5—C4	111.9 (2)	H10B—C10—H10C	109.5
O4—C5—C6	100.59 (17)	S2—C11—H11A	109.5
C4—C5—C6	116.4 (2)	S2—C11—H11B	109.5
O4—C5—H5	109.2	H11A—C11—H11B	109.5
C4—C5—H5	109.2	S2—C11—H11C	109.5
C6—C5—H5	109.2	H11A—C11—H11C	109.5
O3—C6—C2	110.65 (18)	H11B—C11—H11C	109.5
O3—C6—C5	104.42 (18)		

C2—O1—C1—S1	−6.7 (3)	O2—C4—C5—C6	−38.1 (3)
C2—O1—C1—S2	172.78 (14)	C7—O3—C6—C2	102.62 (19)
C11—S2—C1—O1	−179.09 (17)	C7—O3—C6—C5	−16.2 (2)
C11—S2—C1—S1	0.4 (2)	O1—C2—C6—O3	76.4 (2)
C1—O1—C2—C3	83.2 (2)	C3—C2—C6—O3	−161.29 (17)
C1—O1—C2—C6	−154.27 (19)	O1—C2—C6—C5	−168.44 (17)
C10—O5—C3—O2	65.6 (3)	C3—C2—C6—C5	−46.1 (2)
C10—O5—C3—C2	−173.94 (19)	O4—C5—C6—O3	32.2 (2)
C4—O2—C3—O5	55.4 (3)	C4—C5—C6—O3	153.3 (2)
C4—O2—C3—C2	−65.4 (2)	O4—C5—C6—C2	−86.8 (2)
O1—C2—C3—O5	57.3 (2)	C4—C5—C6—C2	34.3 (3)
C6—C2—C3—O5	−61.4 (2)	C5—O4—C7—O3	28.4 (2)
O1—C2—C3—O2	−178.93 (17)	C5—O4—C7—C8	146.0 (2)
C6—C2—C3—O2	62.4 (2)	C5—O4—C7—C9	−89.5 (3)
C3—O2—C4—C5	54.0 (3)	C6—O3—C7—O4	−6.3 (2)
C7—O4—C5—C4	−161.4 (2)	C6—O3—C7—C8	−123.6 (2)
C7—O4—C5—C6	−37.1 (2)	C6—O3—C7—C9	113.8 (2)
O2—C4—C5—O4	76.8 (3)		