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(+)-3-{(4*S*,5*S*)-5-[1,1-dimethyl-2-(phenylthio)ethyl]-2,2-dimethyl-1,3-dioxolan-4-yl}prop-2-yn-1-ol

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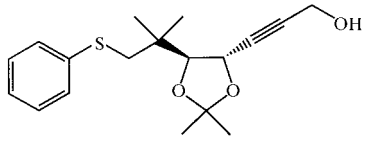
The absolute configuration was determined for the title compound, (+)-$C_{18}H_{24}O_3S$, (I), which was prepared in a synthetic study on the natural products, bryostatins.

Comment

A synthetic study on bryostatin 3 has been reported (Obitsu *et al.*, 1998).



(I)

Experimental

The title optically active compound was prepared by the authors in a synthetic study on bryostatins (Ohmori *et al.*, 2000). The angle of rotation $[\alpha]_D^{21}$ is +20.9 ° (*c* 1.0, $CHCl_3$). Crystals were grown from a hexane/ethyl acetate solution.

Crystal data

$C_{18}H_{24}O_3S$
M_r = 320.45
Orthorhombic, $P2_12_12_1$
a = 8.760 (2) Å
b = 32.090 (2) Å
c = 6.269 (2) Å
V = 1762.3 (6) Å^3
Z = 4
D_x = 1.208 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 29.1–30.0°
μ = 1.706 mm^{-1}
T = 248 (1) K
Prism, colourless
0.5 × 0.5 × 0.3 mm

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
T_{min} = 0.476, T_{max} = 0.636
2633 measured reflections
2147 independent reflections
2055 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 75°
$h = -11 \rightarrow 5$
$k = -20 \rightarrow 40$
$l = -4 \rightarrow 8$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.040
$wR(F^2)$ = 0.108
S = 1.08
2147 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.8338P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0101 (6)
Absolute structure: Flack (1983), 163 Friedel pairs
Flack parameter = 0.03 (3)

Table 1
Selected geometric parameters (Å, °).

S1—C5	1.762 (3)	C17—C18	1.188 (4)
S1—C11	1.813 (3)	C18—C19	1.465 (5)
C16—C17	1.465 (4)		
C5—S1—C11	103.1 (1)	C17—C18—C19	178.9 (3)
C16—C17—C18	177.7 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O3^{i}	0.98 (3)	1.86 (3)	2.816 (3)	164 (3)

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

X-ray intensities were measured for $-h+k+l$ ($\theta < 75°$), $+h+k-l$ ($\theta < 30°$) and $+h-k-l$ ($\theta < 30°$). There were 163 Friedel pairs, which were not averaged. The completeness of symmetry unique reflections ($\theta < 75°$) was 93.3%, which was due to the blind region of the low-temperature apparatus. The hydroxyl H atom was located from a difference synthesis. Their positional parameters were refined with restraint of O—H = 0.98 Å (s.u. 0.01 Å) with U_{iso}(H) = 1.2U_{eq}(parent atom). The other H-atom positional parameters were calculated geometrically and fixed with U_{iso}(H) = 1.2U_{eq}(parent atom). The absolute structure was determined based on a Flack parameter value of 0.03 (3).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN*.

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