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The absolute configuration of heliespirone B, from sunflower *Helianthus annuus*

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Key indicators

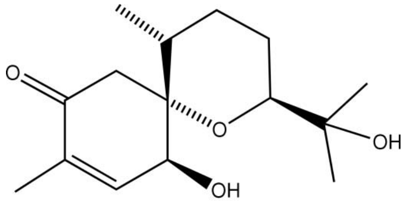
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.087
Data-to-parameter ratio = 12.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The absolute configuration of the title compound [systematic name: 5-hydroxy-8-(1-hydroxyisopropyl)-3,11-dimethyl-7-oxaspiro[5.5]undec-3-en-2-one], $C_{15}H_{24}O_4$, has been determined using Cu *Kα* data at 100 K. The configurations at its four asymmetric C centers are 1*R*, 2*S*, 7*R* and 10*S*. There are two independent molecules in the asymmetric unit, and they form hydrogen-bonded chains in the [100] direction using only the OH groups.

Comment

Isolation of the title compound, (I), from the sunflower (*Helianthus annuus* L.) has recently been reported (Macías *et al.*, 2006), including its crystal structure based on Mo *Kα* data. Determination of its absolute configuration was not possible, but only the relative configurations of its four asymmetric C atoms. It is important to know the absolute configuration of (I), as this allows assignment of the absolute configurations of a series of oxaspirocyclic sesquiterpenes isolated from the same plant. These compounds are bioactive, with potential application as natural herbicides (Macías *et al.*, 1998).



(I)

Thus, a low-temperature data set was obtained using Cu *Kα* radiation, and refinement of the Flack (1983) parameter demonstrates the configuration at these centers to be 1*R*, 2*S*, 7*R*, 10*S* (Fig. 1). By chance, the configuration arbitrarily chosen in the original publication is correct. This configuration has also been confirmed using a data set collected at *T* = 150 K on a Rigaku SPIDER curved image-plate diffractometer using Cu *Kα* radiation.

The two independent molecules in the asymmetric unit of (I) form hydrogen-bonded chains (Fig. 2) in the [100] direction, using only the OH groups. The OH groups from both independent molecules alternate along the chain with pattern (O1*B*···O1*A*···O4*B*···O4*A*···)$_n$, and all O—H···O angles are nearly linear (Table 2).

Experimental

The isolation of the title compound from the leaves of *Helianthus annuus* has been described previously by Macías *et al.* (2006). It was crystallized from *n*-hexane–ethyl acetate (1:8 *v*/*v*). $[\alpha]_D$ +19.6; *c* = 0.1, $CHCl_3$, 298 K.

Crystal data

$C_{15}H_{24}O_4$
$M_r = 268.34$
Monoclinic, $P2_1$
$a = 10.3142$ (4) Å
$b = 11.9229$ (4) Å
$c = 12.2124$ (4) Å
$\beta = 107.223$ (2)°
$V = 1434.48$ (9) Å^3
$Z = 4$
Cu $K\alpha$ radiation
$\mu = 0.72$ mm^{-1}
$T = 100$ K
0.38 × 0.35 × 0.32 mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.772$, $T_{max} = 0.802$
13509 measured reflections
4578 independent reflections
4573 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.04$
4578 reflections
365 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Absolute structure: Flack (1983), with 2065 Friedel pairs
Flack parameter: 0.08 (12)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*A*···O1*B*	0.87 (3)	1.96 (3)	2.8278 (19)	177 (2)
O4*A*—H4*A*···O4*B*	0.81 (3)	2.24 (3)	3.0440 (19)	173 (2)
O1*B*—H1*B*···O4*A*	0.89 (3)	1.83 (3)	2.7091 (18)	172 (2)
O4*B*—H4*B*···O1*A*^{i}	0.82 (3)	2.36 (3)	3.1726 (18)	174 (2)

Symmetry code: (i) $x+1, y, z$.

H atoms on C were placed in idealized positions, with C—H = 0.95–1.00 Å, and thereafter treated as riding. A torsional parameter was refined for each methyl group. Displacement parameters for H atoms were assigned as $U_{iso}(H) = 1.2U_{eq}(C)$, or $1.5U_{eq}(C)$ for methyl H. The coordinates of O-bound H atoms were refined.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *XPREP* (Sheldrick, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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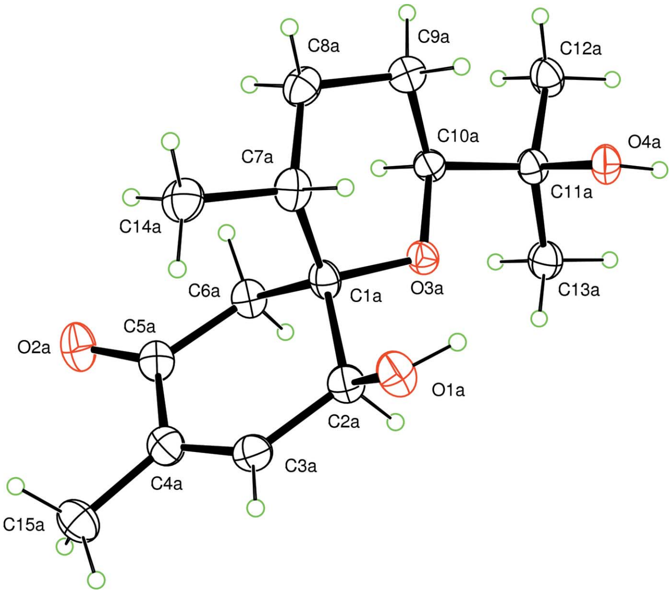

Figure 1
The structure of one of the two independent molecules of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% level.

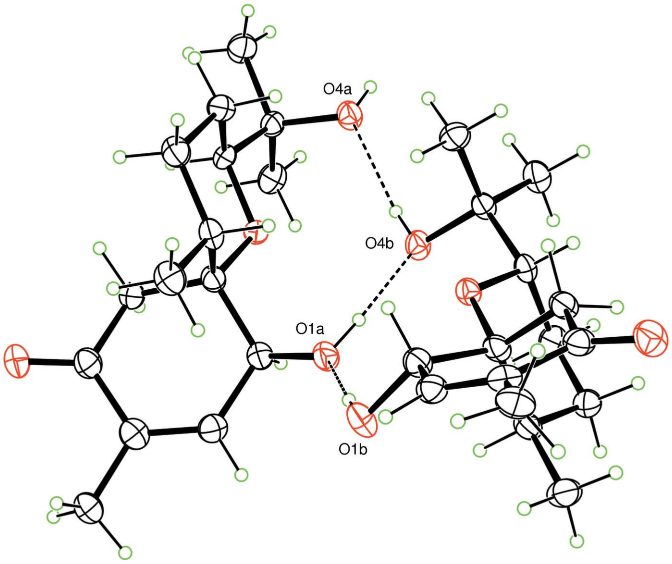

Figure 2
The asymmetric unit of (I), showing the hydrogen bonding (dashed lines).

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