

(1*S*)-(-)- β -Pinene

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Key indicators

Single-crystal X-ray study

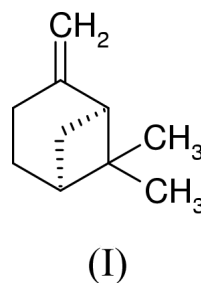
 $T = 185$ KMean $\sigma(\text{C}-\text{C}) = 0.003$ Å R factor = 0.060 wR factor = 0.155

Data-to-parameter ratio = 18.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The crystal structure of (1*S*)-(-)- β -pinene, $\text{C}_{10}\text{H}_{16}$, has been determined at 185 (2) K by *in situ* growth from the liquid.

Comment

β -Pinene, (I), occurs in nature in the majority of the oils in which α -pinene is found; the percentage present however is very variable and is, as a rule, considerably less than that of the latter. For an account of the history of β -pinene and the determination of its structure using the techniques of classical organic chemistry, see Simonsen & Owen (1947). This work forms part of a continuing study devoted to improving the techniques for determining the crystal structures of substances which are liquids at room temperature [see, for example, Davies & Bond (2001)].



Experimental

(1*S*)-(-)- β -Pinene (99%) was obtained from the Aldrich Company and used without further purification. The crystal was grown in a 0.4 mm glass capillary tube at 201 K (a temperature only slightly less than the melting point of the solid in the capillary tube). With the axis of the capillary parallel to the φ axis and horizontal on the instrument, the crystal was obtained by moving a plug of solid material up and down the tube [the movement being controlled with the standard Z (height) adjustment of the goniometer head]. The crystal was cooled subsequently to 185 (2) K for data collection.

Crystal data

$\text{C}_{10}\text{H}_{16}$
 $M_r = 136.23$
 Orthorhombic, $P2_12_12_1$
 $a = 7.5079$ (3) Å
 $b = 9.3361$ (8) Å
 $c = 12.4875$ (11) Å
 $V = 875.30$ (11) Å³
 $Z = 4$
 $D_x = 1.034$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 3825 reflections
 $\theta = 1.0$ – 26.7°
 $\mu = 0.06$ mm⁻¹
 $T = 185$ (2) K
 Cylinder, colourless
 0.20 mm (radius)

Data collection

| | |
|---|------------------------------------|
| Nonius KappaCCD diffractometer | $R_{\text{int}} = 0.081$ |
| Thin-slice ω and φ scans | $\theta_{\text{max}} = 26.8^\circ$ |
| 4164 measured reflections | $h = -9 \rightarrow 9$ |
| 1747 independent reflections | $k = -11 \rightarrow 11$ |
| 1498 reflections with $I > 2\sigma(I)$ | $l = -15 \rightarrow 15$ |

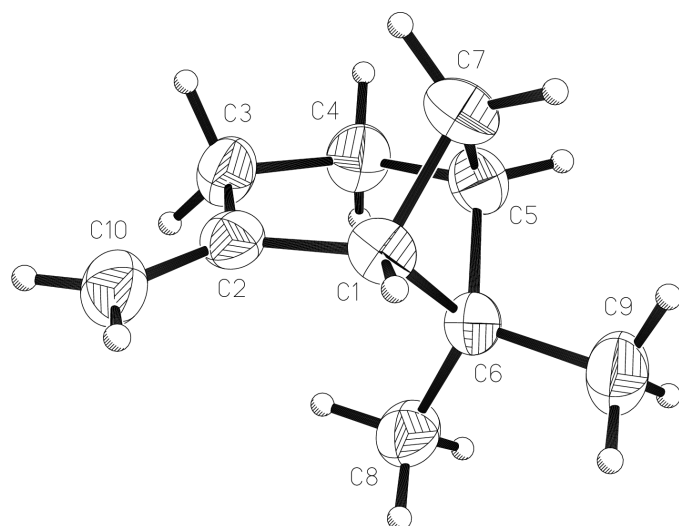


Figure 1
The asymmetric unit in (I) showing displacement ellipsoids at the 50% probability level (*XP*; Sheldrick, 1993).

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.155$
 $S = 1.09$
 1747 reflections
 93 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.2821P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.004$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$

H atoms were placed geometrically and refined using a riding model with an isotropic displacement parameter fixed at $1.2U_{\text{eq}}$ of the carbon to which they are attached. The absolute configuration could not be determined reliably and was assigned according to the known configuration of the sample. Friedel pairs were merged, therefore, prior to merging of other equivalent reflections in $P2_12_12_1$; the reported value of R_{int} corresponds to merging in this space group.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*;

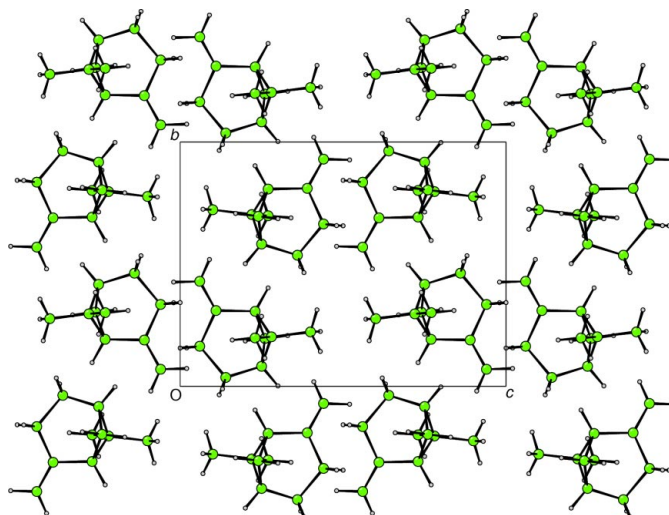


Figure 2
Projection onto (100) of the crystal structure of (I) (*CAMERON*; Watkin *et al.*, 1996).

program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994);
 program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997);
 software used to prepare material for publication: *SHELXL97*.

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References

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