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trans-Decahydronaphthalene (decalin) from powder diffraction data

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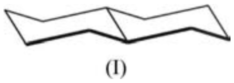
The title compound, $C_{10}H_{18}$, a decalin stereoisomer, crystallizes with $Z' = 0.5$ in the space group $P2_1/n$. The *trans*-decalin molecule is located on an inversion centre with both rings in a chair conformation, making for a quasi-flat overall shape. Despite the absence of hydrogen bonds, it crystallizes easily. In this work the unknown crystal structure of *trans*-decalin has been solved and refined using X-ray powder diffraction data.

Comment

Decahydronaphthalene, or decalin, (I), is the hydrogen-saturated analogue of naphthalene. It has a wide range of applications in research and industry, from multipurpose solvent, prospective use as a jet fuel for high-Mach aircraft (Lai & Song, 1996) to a hydrogen-storage medium for fuel cells (Tsuji *et al.*, 2007). As a solvent it is typically used for index matching in optical studies of polymers or colloids due to its high refractive index of n = 1.47. Its two isomers, *cis* and *trans* (Fig. 3), have different thermodynamic properties (Lal & Swinton, 1969), which makes it a system of choice for studies of the effect of stereoisomerism on physical properties in liquids.


(I)

In the field of glass physics, the influence of the isomers on the behaviour of decalin recently caused interest. The fragility, a property quantifying the 'rapidity' of solidification as a function of temperature at the glass transition point, of decalin has been investigated. It was found to have one of the highest fragility values for molecular liquids at a mixing ratio of 1:1, while the fragility of *cis*-decalin was found to be moderate (Wang *et al.*, 2002; Duvvuri & Richert, 2002). This would imply dependence of the fragility on isomeric composition, which is unusual in this domain and so prompted research on the microscopic origin of the effect (Dalle-Ferrier *et al.*, 2007). The study of anharmonicity of vibrational modes in solidified *cis*-decalin investigated that microscopic origin (Plazanet & Schober, 2008) and also led to this work.

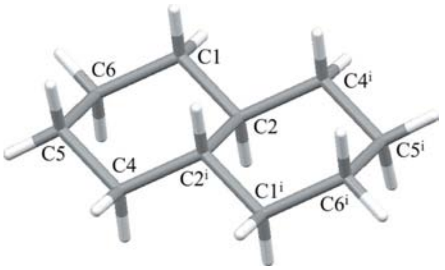


Figure 1
The structure of the *trans*-decalin molecule. [Symmetry code: (i) $-x, -y, -z$.]

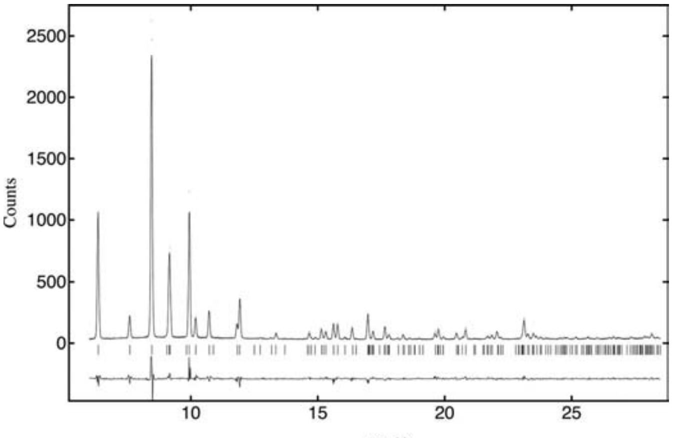


Figure 2
The final Rietveld refinement, showing observed, calculated and difference profiles for *trans*-decalin.

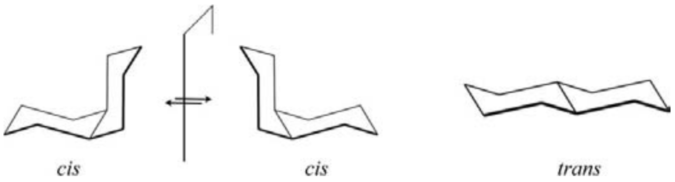


Figure 3
Decalin exists as a *cis* or *trans* isomer, with the relative position of the H atoms to the two common C atoms as a distinction criterion. The ground state of *trans*-decalin is not chirally split as it is for *cis*-decalin.

The two known isomers are distinct in their conformational freedom. *trans*-Decalin is rigid and confined to one conformation with a centre of symmetry at the centre of mass, whereas *cis*-decalin can interchange between several conformations, with two chiral chair–chair ground states (Gerig & Roberts, 1966). Experiments on a differential scanning calorimeter with high cooling rate show a very pronounced exothermic signal on crystallization of *trans*-decalin with no further features appearing down to liquid nitrogen tempera-

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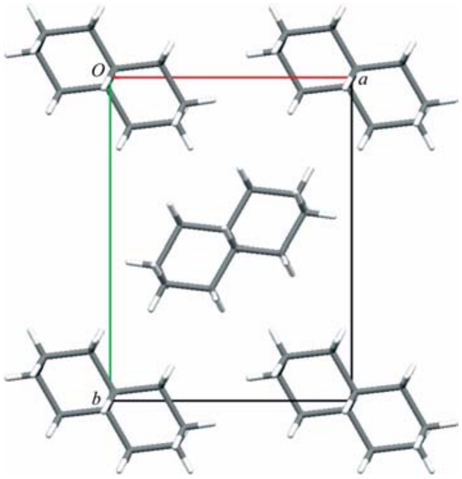

Figure 4
The packing diagram for crystalline *trans*-decalin, viewed along the *c* axis.

ture. Observations made during experiments that involved cooling past the melting temperature confirm this finding in showing quick crystallization and a crystal structure that is stable and independent of cooling history.

The crystallization of *trans*-decalin (Fig. 1) happens very quickly and is hard to avoid. In the course of experiments, attempts have been made to vitrify *trans*-decalin by quenching it in liquid nitrogen. Quantities as small as a few milligrams quenched from room temperature in aluminium containers did not show any signature of amorphicity during further investigation. In the course of crystal structure determination of *trans*-decalin, difficulties arose from the quick crystallization behaviour. Measurements of an *in situ* crystallized sample on the high-resolution X-ray powder diffractometer ID31 at ESRF (Fitch, 2004) showed very narrow peaks and suggested large grain sizes. Although the sample was spun for angular averaging during data acquisition, the grain sizes seemed to be too big for accurate sampling of peak intensities. Hence, a further investigation of the crystallization behaviour was conducted on the X-ray diffractometer SNBL BM01 on a bending magnet at ESRF, which operates a two-dimensional mar345 detector. The two-dimensional pattern showed strongly inhomogeneous angular intensity distribution and so proved the intensities first collected on ID31 to be insufficient for accurate refinement. During further treatment the one-dimensional pattern was used to determine cell parameters, while the integrated two-dimensional pattern was used for intensity-sensitive evaluation and structure solution.

The molecules of (I) are located on crystallographic centres of inversion. Both rings have the chair conformation. The packing of the molecules in the unit cell is shown in Fig. 4.

Experimental

trans-Decalin was purchased from Sigma Aldrich with a purity higher than 99% and used without further treatment. For both measurements, glass capillaries with a diameter of 1 mm for ID31 and 0.5 mm for SNBL BM01A were used as sample containers. The sample was mounted on the instruments at room temperature, where it is liquid, and then cooled to 100 K using a cryostream cold N_2 gas blower. An *in situ* phase change from liquid to crystalline powder could thus be achieved. The measurement on ID31 was performed at a wavelength of 0.79984 (4) Å; for the SNBL BM01A measurement, a wavelength of 0.694 (1) Å was used.

Crystal data

$C_{10}H_{18}$
$M_r = 138.25$
Monoclinic, $P2_1/n$
$a = 7.8101$ (5) Å
$b = 10.4690$ (12) Å
$c = 5.2638$ (3) Å
$\beta = 90.990$ (7)°
$V = 430.33$ (6) Å^3
$Z = 2$
Synchrotron radiation
$\lambda = 0.694$ (1) Å
$\mu = 0.03$ mm^{-1}
$T = 100$ K
Specimen shape: cylinder
10 × 0.5 × 0.5 mm
Specimen prepared at 100 kPa
Specimen prepared at 293 K
Particle morphology: plate-like, colourless

Data collection

SNBL diffractometer
Specimen mounting: *in situ* crystallised liquid in capillary
Specimen mounted in transmission mode
Scan method: continuous
$2\theta_{\min} = 0$, $2\theta_{\max} = 36°$
Increment in $2\theta = 0.02°$

Refinement

$R_{wp} = 0.093$
$R_B = 0.039$
Excluded region(s): none
Profile function: Thompson–Cox–Hastings (Thompson *et al.*, 1987)
H atoms treated by a mixture of independent and constrained refinement
Preferred orientation correction: spherical harmonics of order four (Järvinen, 1993)

The high-resolution powder diffraction pattern from ID31 was indexed using the program *DICVOL* (Boultif & Louër, 2004), implemented in the program package *DASH* (David *et al.*, 2006) *via* the positions of 25 diffraction peaks. Systematic absences suggested space group $P2_1/n$. The resulting unit cell holds two molecules situated on centres of symmetry with half a molecule as an asymmetric unit. A molecular structure of *trans*-decalin was optimized using the *ab initio* code DMol3 (Delley, 1990), then converted into *z*-matrix format and its centre fixed on the centre of symmetry (0,0,0) of the cell. Data from SNBL BM01A were reduced using the *Fit2D* (Hammersley *et al.*, 1996) package. With the simulated annealing feature of the program *TOPAS* (Coelho, 2000), the three degrees of freedom corresponding to the orientations of the molecule were optimized to obtain the best agreement with the two-dimensional data from SNBL BM01A. The solved structure was refined using *TOPAS*, with H atoms fixed at a distance of 0.96 Å from C atoms. In the refinement (Rietveld, 1969) (Fig. 2), a total of four parameters, three orientational degrees of freedom and $B_{\rm overall}$, were fitted to reflections contributing to the profile. Peak shapes were modelled using a Thompson–Cox–Hastings function (Thompson *et al.*, 1987) and residual preferred orientation was described by spherical harmonics of order four. Standard uncertainties were calculated by the bootstrap method implemented in *TOPAS*.

Data collection: ESRF: ID31 and BM01A using *spec* (Certified Scientific Software, 2003); cell refinement: *DASH* (David *et al.*, 2006); data reduction: *Fit2D* (Hammersley *et al.*, 1996); program(s) used to solve structure: *TOPAS* (Coelho, 2000); program(s) used to refine structure: *TOPAS*; molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK3312). Services for accessing these data are described at the back of the journal.

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