

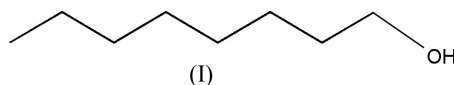
n-OctanolHoward A. Shallard-Brown,*
David J. Watkin and Andrew R.
CowleyChemical Crystallography Laboratory, Central
Chemistry Laboratory, Mansfield Road, Oxford
University, Oxford OX1 3TA, EnglandCorrespondence e-mail:
howard.shallard-brown@lmh.ox.ac.uk

Key indicators

Single-crystal X-ray study
 $T = 190$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.125
 wR factor = 0.156
Data-to-parameter ratio = 22.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The structure of *n*-octanol, $\text{C}_8\text{H}_{17}\text{OH}$, at 150 K consists of infinite hydrogen-bonded chains forming a ribbon parallel to the *b* axis.Received 8 December 2004
Accepted 10 December 2004
Online 8 January 2005

Comment

The low-molecular-weight aliphatic monoalcohols are liquid at room temperature. Methanol (CH_3OH ; Allan *et al.*, 1998), ethanol ($\text{C}_2\text{H}_5\text{OH}$; Jönsson, 1976; Allan & Clark, 1999) and cyclobutanol ($\text{C}_4\text{H}_{10}\text{OH}$; McGregor *et al.*, 2003) form planar hydrogen-bonded ribbons in the solid state, while the bulkier tertiary butanol [$(\text{CH}_3)_3\text{COH}$; Steininger *et al.*, 1989] forms threefold helical hydrogen-bonded chains. At ambient pressure, phenol also forms threefold helical chains, while at 0.16 GPa and just above its normal melting point (313 K), it forms planar ribbons (Allan *et al.*, 2002). As part of a programme aimed at simplifying the growth of crystals from materials which are liquid at room temperature, we have looked at *n*-heptanol ($\text{C}_7\text{H}_{15}\text{OH}$) and *n*-octanol ($\text{C}_8\text{H}_{17}\text{OH}$). *n*-Heptanol could only be zone-crystallized, by a modification of the Bridgman technique (Bridgman, 1925), to an unindexable polycrystalline mass. *n*-Octanol, (I), was obtained as 'fair quality' single crystals accompanied by small satellite crystals. A previous examination of *n*-octanol crystals (Dunoyer & Ribaud, 1951) reported, on the basis of Debye–Scherrer photographs, a low-symmetry form just below the melting point, passing to an hexagonal form ($a = 4.468$ Å, $c = 7.282$ Å; ice I has $a = 4.5$ Å and $c = 7.3$ Å) between 248 and 215 K, after which the original low-symmetry cell reappeared.



In the present experiment, *n*-octanol was grown as a single crystal just below its melting point, and the temperature was then lowered to 150 K at a rate of 360 K per hour. There was no evidence of a phase transition.

In the low-temperature and ambient-pressure form of ethanol, the molecules form hydrogen-bonded ribbons, with the methyl group oriented somewhat towards the hydrogen-bonded backbone. This leads to a narrow ribbon with strained hydrogen-bonding angles. At ambient temperature and 3.0 GPa, the methyl groups of ethanol are coplanar with the backbone, lying fully extended on alternate sides. In *n*-octanol, the aliphatic chains are also coplanar, with the hydrogen-bonded backbone forming infinite wide ribbons parallel to the *b* axis. These ribbons pack side-by-side, with the terminal ethyl

groups parallel and in close contact, forming sheets of molecules.

Experimental

A single crystal of (I), which is a liquid at room temperature, was grown by keeping the compound under a cold nitrogen stream at just below its melting point, and slowly moving a small liquid zone up and down the sample. The temperature was then lowered for the main data collection.

Crystal data

$C_8H_{18}O$	$D_x = 1.019 \text{ Mg m}^{-3}$
$M_r = 130.23$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1584 reflections
$a = 4.2065 (2) \text{ \AA}$	$\theta = 5-27^\circ$
$b = 5.1845 (2) \text{ \AA}$	$\mu = 0.06 \text{ mm}^{-1}$
$c = 38.9371 (18) \text{ \AA}$	$T = 190 \text{ K}$
$\beta = 91.723 (2)^\circ$	Cylinder, colourless
$V = 848.78 (7) \text{ \AA}^3$	$0.80 \times 0.30 \times 0.30 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD area-detector diffractometer	1854 independent reflections
ω scans	1011 reflections with $I \geq 2\sigma(I)$
Absorption correction: multi-scan, <i>DENZO</i> and <i>SCALEPACK</i> (Otwinowski & Minor, 1997)	$R_{\text{int}} = 0.042$
$T_{\text{min}} = 0.75$, $T_{\text{max}} = 0.98$	$\theta_{\text{max}} = 27.4^\circ$
11 803 measured reflections	$h = -5 \rightarrow 5$
	$k = -6 \rightarrow 5$
	$l = -49 \rightarrow 50$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.125$	$w = 1/[\sigma^2(F) + 0.062 + 0.164P]$,
$wR(F^2) = 0.157$	where $P = [\max(F_o^2, 0) + 2F_c^2]/3$
$S = 0.99$	$(\Delta/\sigma)_{\text{max}} = 0.001$
1854 reflections	$\Delta\rho_{\text{max}} = 0.45 \text{ e \AA}^{-3}$
82 parameters	$\Delta\rho_{\text{min}} = -0.42 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1–C2	1.4352 (19)	C5–C6	1.526 (2)
C2–C3	1.511 (2)	C6–C7	1.524 (2)
C3–C4	1.530 (2)	C7–C8	1.526 (2)
C4–C5	1.524 (2)	C8–C9	1.518 (3)
O1–C2–C3	108.99 (14)	C5–C6–C7	114.18 (15)
C2–C3–C4	112.43 (14)	C6–C7–C8	113.72 (15)
C3–C4–C5	113.66 (14)	C7–C8–C9	113.58 (15)
C4–C5–C6	113.79 (14)		

The ‘multi-scan’ corrections applied by *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997) will also contain a contribution due to changes in the illuminated volume of the cylindrical sample. All H atoms were seen in a difference electron-density map. The hydroxyl H atom was placed as found, and the others were placed geometrically with U_{iso} values related to the adjacent atoms. The H

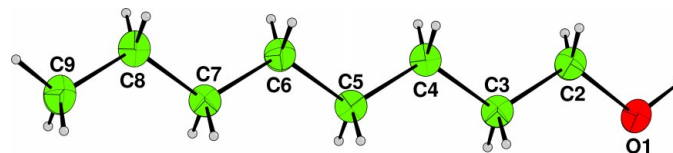


Figure 1
The title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

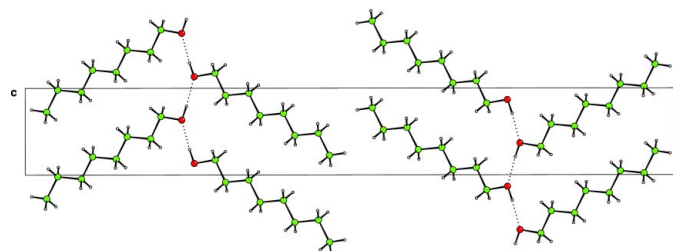


Figure 2
A packing diagram for (I), viewed along the a axis. The molecules are linked into ribbons by hydrogen bonds (dashed lines).

atoms were initially refined with soft restraints on the bond lengths and angles to regularise their geometry ($C-H = 0.93-0.98 \text{ \AA}$) and $U_{\text{iso}}(H)$ values of 1.2–1.5 times U_{eq} of the adjacent atom, after which they were refined with riding constraints.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK*; data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

References

- Allan, D. R. & Clark, S. J. (1999). *Phys. Rev. B*, **60**, 6328–6334.
 Allan, D. R., Clark, S. J., Brugmans, M. J. P., Ackland, G. J. & Vos, W. L. (1998). *Phys. Rev. B*, **58**, 809–812.
 Allan, D. R., Clark, S. J., Dawson, A., McGregor, P. A. & Parsons, S. (2002). *Acta Cryst.* **B58**, 1018–1024.
 Altomare, A., Casciarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
 Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, C. K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
 Bridgman, P. W. (1925). *Proc. Am. Acad. Arts Sci.* **60**, 305–315.
 Dunoyer, J.-M. & Ribaud, G. (1951). *C. R. Acad. Sci.* **233**, 41–42.
 Jönsson, P.-G. (1976). *Acta Cryst.* **B32**, 232–235.
 McGregor, P. A., Allan, D. R., Clark, S. J. & Parsons, S. (2003). In preparation.
 Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, Macromolecular Crystallography, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
 Steinger, R., Bilgram, J. H., Gramlich, V. & Petter, W. (1989). *Z. Kristallogr.* **187**, 1–13.
 Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.