Monoclinic
$C2/c$
$a = 23.9096\,(7)$ Å
$b = 9.5192\,(3)$ Å
$c = 11.9629\,(3)$ Å
$\beta = 112.136\,(1)°$
$V = 2522.07\,(13)$ Å^3
$Z = 8$
$D_x = 1.387$ Mg m^{-3}
D_m not measured

Cell parameters from 6375
   reflections
$\theta = 1.5–33.0°$
$\mu = 0.256$ mm^{-1}
$T = 293\,(2)$ K
Block
$0.52 \times 0.36 \times 0.30$ mm
Colourless

Data collection

Siemens SMART CCD area-
   detector diffractometer
ω scans
Absorption correction:
   empirical (*SADABS*;
   Sheldrick, 1996a)
   $T_{min} = 0.871$, $T_{max} = 0.955$
8060 measured reflections

2898 independent reflections
2474 reflections with
   $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 27.5°$
$h = -30 \rightarrow 28$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.102$
$S = 1.058$
2897 reflections
215 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 1.4298P]$
   where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.218$ e Å^{-3}
$\Delta\rho_{min} = -0.280$ e Å^{-3}
Extinction correction: none
Scattering factors from
   *International Tables for
   Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S—O2	1.4259 (13)	S—C7	1.760 (2)
S—O3	1.4347 (12)	O1—C1	1.371 (2)
S—N1	1.6214 (13)	N1—C5	1.432 (2)
O2—S—O3	119.55 (8)	O3—S—C7	107.62 (8)
O2—S—N1	108.65 (8)	N1—S—C7	107.96 (7)
O3—S—N1	104.59 (7)	C5—N1—S	123.62 (11)
O2—S—C7	107.97 (8)		
C4—C5—N1—S	52.0 (2)	N1—S—C7—C8	−74.3 (2)
C5—N1—S—C7	64.19 (14)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1O1⋯O3^i	0.79 (3)	1.95 (3)	2.727 (2)	171 (2)
N1—H1N1⋯O1^{ii}	0.85 (2)	2.14 (2)	2.983 (2)	174 (2)

Symmetry codes: (i) $x, 1 + y, z$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay, monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, was found to be negligible. The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *SMART* (Siemens, 1996b). Cell refinement: *SAINT* (Siemens, 1996a). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996b). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Cyclodecanol

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Abstract

Cyclodecanol crystals, $C_{10}H_{20}O$, grown by slow sublimation, occur in the monoclinic system in space group

$P2_1/n$. The carbocycle has the more stable diamond-lattice boat–chair–boat conformation and the hydroxy group is in the 2e position. Molecules are linked by hydrogen bonding between hydroxy groups, forming infinite chains along **a**, although the hydroxy hydrogen is apparently conformationally disordered, with O$\cdots$O distances of 2.862 (4) and 2.893 (4) Å.

Comment

Conformations of medium carbocycles have been of long-standing interest because of the diverse structures encountered during conformational interconversion, and the theoretical and experimental challenges to understanding the dynamics of these processes. The boat–chair–boat (BCB) conformation has been found by diffraction for solid cyclodecane near 173 K (Shenhav & Schaeffer, 1981) and it has been calculated to be the most stable form for the cycle (Hendrickson, 1967b; Saunders, 1991; Kolossváry & Guida, 1993; Senderowitz et al., 1995) for which the C atoms are essentially superimposed upon the interstices of a diamond lattice. At 403 K, the BCB form is the major conformer in the gas phase, though probably co-existing with several other lower energy forms (Hilderbrandt et al., 1973). The BCB form is the likely conformer in solid cyclodecane at 145 K (Drotloff, 1987), and the major conformer of 1,1-difluorocyclodecane in solution at 109 K (Noe & Roberts, 1972), as determined by NMR spectroscopy. Diffraction studies of solid cyclodecane derivatives have usually found the ring in the BCB conformation except under some steric circumstances (Mladeck & Nowacki, 1964; Dunitz, 1971) and packing environments (Ermer et al., 1989).

Crystallographic determinations of structures of monosubstituted cyclodecanes are still relatively few in number. The ring in cyclodecylammonium chloride has the BCB conformation with the substituent in the 3e position (Mladeck & Nowacki, 1964). In the present study, cyclodecanol molecules, (I), have the BCB con-

(I)
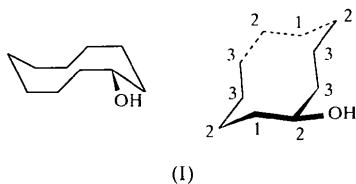
formation (Fig. 1) and atoms occupy general positions. Packing in cyclodecanol is similar to that in cyclodecane, which also crystallizes in space group $P2_1/n$ ($Z = 2$); cyclodecane molecules display approximate 2/m site symmetry and surround inversion centers. Cyclodecanol molecules pack between inversion centers with the hydroxy group linking molecules in chains along the *a* axis; hydroxy groups in alternate layers along **b** have opposing orientations. To accommodate the hy-

droxy group, the *b* axis of cyclodecanol is slightly inflated above a value about twice that in cyclodecane, while the other cell dimensions are quite similar [cyclodecane: $a = 5.283$ (4), $b = 12.221$ (7), $c = 7.090$ (5) Å and $\beta = 103.23$ (3)°; Shenhav & Schaeffer, 1981]. Intra-ring torsion angles are given in Table 1, and show the eight *gauche* and two *anti* conformations within the ring which are close both to the theoretical values (Hendrickson, 1967a; Senderowitz et al., 1995) and to those found for solid cyclodecane (Shenhav & Schaeffer, 1981).
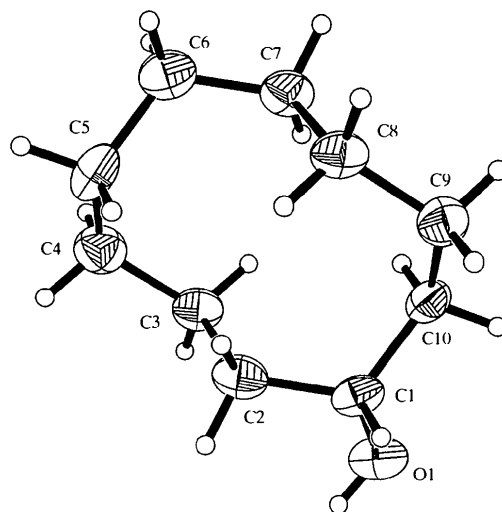

Fig. 1. Displacement ellipsoid plot (50% probability) of cyclodecanol.

The hydroxy group of cyclodecanol is in the 2e position; its oxygen is almost equidistant and at hydrogen-bonding distances from those in either direction along **a**; donor–acceptor O$\cdots$O distances are 2.862 (4) and 2.893 (4) Å. This arrangement is consistent with chain hydrogen bonding of hydroxy groups (Brock & Duncan, 1994). Difference Fourier maps revealed a region of electron density about 1.0 Å from oxygen in an intermediate direction relative to neighboring O atoms. An averaged model for an ordered hydrogen was adopted to fit this density. Since this hydrogen position produces an unusually short H$\cdots$H distance, hydroxy H atoms are apparently disordered over positions representative of hydrogen bonding to oxygen acceptors in each direction (Jeffrey & Saenger, 1991).

Experimental

Crystals of cyclodecanol were grown over a period of four months from the microcrystalline solid (pure by gas chromatography) in a closed vial placed in a temperature gradient between 298 and 306 K. A representative colorless crystal was wedged and sealed in a 0.1 mm glass capillary.

Crystal data

$C_{10}H_{20}O$	Mo $K\alpha$ radiation
$M_r = 156.26$	$\lambda = 0.71073$ Å

Monoclinic
$P2_1/n$
$a = 5.2789\,(12)$ Å
$b = 25.266\,(11)$ Å
$c = 7.139\,(3)$ Å
$\beta = 103.49\,(3)°$
$V = 925.9\,(6)$ Å^3
$Z = 4$
$D_x = 1.121$ Mg m^{-3}
D_m not measured

Cell parameters from 26
    reflections
$\theta = 8–20°$
$\mu = 0.069$ mm^{-1}
$T = 293\,(2)$ K
Thin rod
$0.50 \times 0.10 \times 0.10$ mm
Colorless

Data collection

Siemens *P3* diffractometer,
    scintillation counter pulse-
    height analysis
ω scans
Absorption correction: none
2333 measured reflections
2123 independent reflections
505 reflections with
    $I > 2\sigma(I)$

$R_{int} = 0.120$
$\theta_{max} = 27.50°$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 32$
$l = -9 \rightarrow 9$
3 standard reflections
    every 97 reflections
    intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.138$
$S = 0.60$
2123 reflections
102 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.000$

$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction:
    SHELXL97
Extinction coefficient:
    0.023 (4)
Scattering factors from
    International Tables for
    Crystallography (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C1	1.443 (4)		
O1—C1—C2—C3	−70.5 (4)	C5—C6—C7—C8	−55.7 (5)
C10—C1—C2—C3	53.0 (4)	C6—C7—C8—C9	151.4 (3)
C1—C2—C3—C4	−151.2 (3)	C7—C8—C9—C10	−56.8 (5)
C2—C3—C4—C5	55.1 (5)	O1—C1—C10—C9	−167.8 (3)
C3—C4—C5—C6	65.7 (5)	C2—C1—C10—C9	67.3 (4)
C4—C5—C6—C7	−65.2 (5)	C8—C9—C10—C1	−64.4 (5)

H atoms on C atoms were refined as riding, while those on O atoms were refined as riding with the C—C—O—H torsion angle refined.

Data collection: *P3* (Siemens, 1989*a*). Cell refinement: *P3*. Data reduction: *XDISK* (Siemens, 1989*b*). Program(s) used to solve structure: *SHELXS*93 (Sheldrick, 1993). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *XP* (Siemens, 1989*c*). Software used to prepare material for publication: *SHELXL*97.

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2-(4-Bromo-2-nitrophenyl)-1-(4-dimethyl-aminophenyl)-1-ethanone†

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Abstract

The title compound, $C_{16}H_{15}BrN_2O_3$, is a key intermediate in the synthesis of indole-substituted Hoechst 33258 analogues. Interaction between the dimethylamino substituent and the ketone through the aromatic ring is evident from the short N2—C4 distance, and the slightly lengthened C7—O3 bond.

† Alternative name: 4-bromo-2-nitrobenzyl 4-dimethylaminophenyl ketone.