

roughly as composed of two perpendicular halves. The function-group O atoms are readily accessible to reactant molecules or receptor sites.

There is an intermolecular hydrogen bond formed between the basic N of one molecule and the O(18) hydroxyl of another at $(0.25 + x, 0.25 - y, 0.5 - z)$: O(18)—H(18) = 1.01 (4), H(18)···N(5) = 1.84 (4), O(18)···N(5) = 2.693 (6) Å and O(18)—H(18)···N(5) = 139.4 (5)°.

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Structure of 3 α ,7 α -Bis(2-hydroxy-2-propyl)bicyclo[3.3.1]nonane,* C₁₅H₂₈O₂

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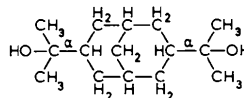
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Abstract. $M_r = 240.39$, orthorhombic, $P2_12_12_1$, $a = 12.700$ (3), $b = 10.474$ (3), $c = 10.771$ (2) Å, $V = 1432.7$ Å³, $Z = 4$, $D_x = 1.12$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.077$ mm⁻¹, $F(000) = 536$, $T = 293$ K. Final $R = 0.043$ for 1559 observed data. The molecule exhibits the double twist-boat conformation. The hydroxypropyl groups have different orientations with respect to the ring system. The molecules are packed by hydrogen bonds of 2.811 and 2.769 Å.

Introduction. For bicyclo[3.3.1]nonane derivatives three groups of conformations must be envisaged: the double-chair, the chair-boats and the double twist-boats. The conformational preference strongly depends on the substituents present on the bicyclic skeleton (Peters, Baas, van de Graaf, van der Toorn & van Bekkum, 1978). Several bicyclo[3.3.1]nonane derivatives occurring in the the double-chair and in the chair-boat conformation have been studied by X-ray

diffraction (Bhattacharjee & Chacko, 1980; van Koningsveld & Peters, 1981; van Koningsveld, 1981), but up till now no such studies have been reported on bicyclo[3.3.1]nonane derivatives existing in the double twist-boat conformation. From ¹H and ¹³C NMR data it was concluded that 3 α ,7 α -bis(2-hydroxy-2-propyl)bicyclo[3.3.1]nonane (HYNONA) exists, at least in solution, predominantly in the double twist-boat conformation (Peters, van der Toorn & van Bekkum, 1975). The structure of HYNONA as determined by X-ray diffraction is reported here.



Experimental. HYNONA (Peters *et al.*, 1975) recrystallized from dimethyl ether at 278 K. A small fragment (approximate size 0.2 × 0.3 × 0.3 mm) mounted on an Enraf–Nonius CAD-4 diffractometer.

* 2,2'-(Bicyclo[3.3.1]nonan-3 α ,7 α -ylene)di-2-propanol.

With Mo $K\alpha$ radiation from a graphite monochromator, unit-cell dimensions and orientation matrix obtained by least squares from setting angles of 25 reflections. Intensities of reflections hkl (range: 0–17, 0–14, 0–15) with $\theta < 30^\circ$ measured in $\omega/2\theta$ scan mode, with variable scan rate and ω -scan angle of $(0.9 + 0.35 \tan \theta)^\circ$. This gave 2370 independent data of which 1559 reflections with $I > \sigma(I)$ were used in subsequent calculations. No correction for absorption. Three standard reflections measured every hour, variation within 2%. Structure solved by direct methods (*MULTAN*, Germain, Main & Woolfson, 1971). Refinement of x, y, z for all atoms, anisotropic temperature factors for heavy atoms and fixed isotropic B values for H atoms (located in a difference map) by full-matrix least squares on F (XRAY72, Stewart, Kruger, Ammon, Dickinson & Hall, 1972) converged at $R = 0.043$, equal weights for all reflections. In final cycle shifts in parameters all $< \sigma$. Final difference map featureless. No secondary-extinction correction applied.

Discussion. Positional and isotropic thermal parameters are given in Table 1.* Bond distances, bond angles and relevant torsion angles are given in Table 2. Fig. 1 shows the molecular structure of HYNONA together with the arbitrary atom numbering used in this report. The molecule exists indeed in the double twist-boat conformation. As far as we know, this is the first double twist-boat bicyclo[3.3.1]nonane crystal structure reported thus far. The orientations of the two hydroxypropyl groups with respect to the ring system are different. In the one group C(11) is nearly antiperiplanar to H(1) [torsion angle C(11)–C(10)–C(1)–H(1) = 177°], whereas in the other group O(2) and H(8) are nearly antiperiplanar [torsion angle O(2)–C(13)–C(8)–H(8) = 171°]. Comparison of the values of the internal

* Lists of structure factors, anisotropic thermal parameters, C–H distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38869 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates with their e.s.d.'s ($\times 10^4$, for H $\times 10^3$) and isotropic thermal parameters ($\times 10^3$)

	x	y	z	$U_{eq}^*/U_{iso}^\dagger(\text{\AA}^2)$
C(1)	4003 (2)	2236 (3)	4863 (3)	39
C(2)	3595 (3)	1010 (3)	5503 (3)	42
C(3)	3599 (2)	1120 (3)	6934 (3)	43
C(4)	4543 (3)	1921 (3)	7307 (4)	53
C(5)	4272 (2)	3316 (3)	7002 (3)	48
C(6)	3803 (3)	3425 (3)	5672 (3)	47
C(7)	2566 (2)	1719 (3)	7436 (3)	44
C(8)	2721 (2)	2760 (3)	8425 (3)	42
C(9)	3485 (3)	3806 (3)	7979 (3)	49
C(10)	3589 (2)	2390 (3)	3528 (3)	46
C(11)	2388 (3)	2421 (5)	3438 (4)	64
C(12)	4062 (4)	3571 (4)	2913 (4)	67
C(13)	1659 (2)	3303 (3)	8878 (3)	46
C(14)	991 (3)	2275 (4)	9498 (4)	62
C(15)	1805 (4)	4423 (4)	9767 (4)	67
O(1)	3905 (2)	1288 (2)	2821 (2)	52
O(2)	1051 (2)	3728 (2)	7820 (2)	48
H(1)	487 (2)	217 (3)	480 (3)	51
H(21)	286 (2)	79 (3)	520 (3)	51
H(22)	403 (2)	24 (3)	521 (3)	51
H(3)	367 (2)	21 (3)	733 (3)	51
H(41)	471 (2)	182 (3)	816 (3)	51
H(42)	519 (2)	161 (3)	688 (3)	51
H(5)	490 (2)	387 (3)	696 (3)	51
H(61)	412 (2)	423 (3)	528 (3)	51
H(62)	299 (2)	359 (3)	569 (3)	51
H(71)	216 (2)	103 (3)	778 (3)	51
H(72)	213 (2)	206 (3)	673 (3)	51
H(8)	300 (2)	238 (3)	918 (3)	51
H(91)	306 (2)	459 (3)	761 (3)	51
H(92)	385 (2)	417 (3)	877 (3)	51
H(111)	209 (3)	317 (3)	393 (3)	70
H(112)	220 (3)	254 (3)	265 (3)	70
H(113)	200 (3)	153 (3)	390 (3)	70
H(121)	486 (3)	364 (3)	307 (3)	70
H(122)	391 (3)	362 (3)	197 (3)	70
H(123)	379 (3)	442 (3)	325 (3)	70
H(141)	81 (3)	146 (3)	901 (3)	70
H(142)	132 (3)	196 (3)	1024 (3)	70
H(143)	29 (3)	269 (4)	974 (3)	70
H(151)	224 (3)	425 (3)	1040 (3)	70
H(152)	214 (3)	513 (4)	935 (3)	70
H(153)	115 (3)	473 (3)	1006 (3)	70
H(O1)	448 (2)	126 (3)	260 (3)	61
H(O2)	123 (3)	432 (3)	761 (3)	61

* $U_{eq} = \frac{1}{3} \text{trace } \bar{U}$.

† U_{iso} for H atoms were held fixed.

Table 2. Bond distances (\AA), bond angles and relevant torsion angles ($^\circ$) involving non-H atoms, and hydrogen-bonding scheme

E.s.d.'s for bonds are 0.004–0.005 \AA and for angles 0.2–0.3 $^\circ$ unless otherwise stated.

Bond distances			
C(1)–C(2)	1.546	C(10)–O(1)	1.440
C(2)–C(3)	1.545	C(3)–C(7)	1.551
C(3)–C(4)	1.518	C(7)–C(8)	1.537
C(4)–C(5)	1.536	C(8)–C(9)	1.540
C(5)–C(6)	1.556	C(9)–C(5)	1.539
C(6)–C(1)	1.541	C(8)–C(13)	1.544
C(1)–C(10)	1.539	C(13)–C(14)	1.525
C(10)–C(11)	1.528	C(13)–C(15)	1.526
C(10)–C(12)	1.526	C(13)–O(2)	1.446
Bond angles			
C(10)–C(1)–C(2)	112.9	C(9)–C(8)–C(13)	112.8
C(10)–C(1)–C(6)	112.8	C(8)–C(9)–C(5)	112.6
C(2)–C(1)–C(6)	111.4	C(1)–C(10)–C(11)	113.7
C(1)–C(2)–C(3)	112.4	C(1)–C(10)–C(12)	110.9
C(2)–C(3)–C(4)	107.9	C(1)–C(10)–O(1)	108.4
C(2)–C(3)–C(7)	112.0	C(11)–C(10)–O(1)	110.4
C(4)–C(3)–C(7)	110.6	C(11)–C(10)–C(12)	105.2
C(3)–C(4)–C(5)	107.0	O(1)–C(10)–C(12)	108.1
C(4)–C(5)–C(6)	110.7	C(8)–C(13)–C(14)	111.4
C(4)–C(5)–C(9)	108.4	C(8)–C(13)–C(15)	112.0
C(6)–C(5)–C(9)	110.9	C(8)–C(13)–O(2)	109.3
C(5)–C(6)–C(1)	113.4	C(14)–C(13)–C(15)	109.6
C(3)–C(7)–C(8)	114.8	C(14)–C(13)–O(2)	105.4
C(7)–C(8)–C(9)	111.7	C(15)–C(13)–O(2)	108.9
C(7)–C(8)–C(13)	111.6		
Selected torsion angles			
HYNONA			
C(6)–C(1)–C(2)–C(3)	27.7	C(5)–C(9)–C(8)–C(7)	26.3
C(1)–C(2)–C(3)–C(4)	34.7	C(4)–C(5)–C(9)–C(8)	34.3
C(2)–C(3)–C(4)–C(5)	–75.2	C(3)–C(4)–C(5)–C(9)	–74.7
C(3)–C(4)–C(5)–C(6)	47.1	C(7)–C(3)–C(4)–C(5)	47.7
C(4)–C(5)–C(6)–C(1)	16.0	C(8)–C(7)–C(3)–C(4)	13.8
C(5)–C(6)–C(1)–C(2)	–55.1	C(9)–C(8)–C(7)–C(3)	–52.9
Cyclohexane			
			30.0
			30.0
			–62.0
			30.0
			30.0
			–62.0
Hydrogen-bonding scheme			
$X-H \cdots Y$	$X \cdots Y$ (\AA)	$H \cdots Y$ (\AA)	$X-H \cdots Y$ ($^\circ$)
O(1)–H(O1)–O(2)	2.811	2.05 (3)	174 (4)
O(2)–H(O2)–O(1 ⁱⁱ)	2.769	2.12 (3)	155 (4)
Symmetry code: (i) $x + \frac{1}{2}, -y, -z + 1$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, -z + 1$.			

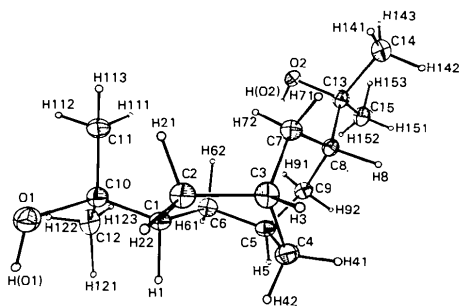


Fig. 1. ORTEP plot (Johnson, 1965) of HYNONA showing atom numbering and thermal ellipsoids (50% probability level) of the non-H atoms. The H atoms were given an arbitrary 0.07 Å radius and were numbered to correspond to their carrier atom followed by a sequence number.

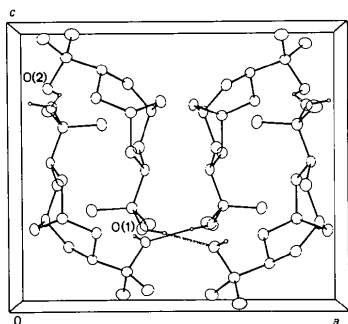


Fig. 2. Drawing of the unit-cell contents viewed down *b*. Short O...O contacts are indicated by open bonds.

torsion angles with those of the twist-boat conformation of cyclohexane as calculated by molecular mechanics (Table 2) (van de Graaf, Baas & van Veen, 1980, and references cited therein) shows that both wings of the bicyclo[3.3.1]nonane skeleton are flattened. Similar geometries were calculated for several analogous 3 α ,7 α -disubstituted bicyclo[3.3.1]nonanes with the use of molecular mechanics (Peters *et al.*, 1978).

The packing of the molecules is illustrated in Fig. 2. There are two short intermolecular O...O contacts which can be considered as (weak) hydrogen bonds; their geometrical details are added in Table 2.

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Bis[2-(*o*-methoxyphenoxy)ethyl] Ether–Urea (1:1) Complex, C₁₈H₂₂O₅·CH₄N₂O

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Dedicated to Professor Friedrich Cramer on the occasion of his 60th birthday

Abstract. $M_r = 378.2$, monoclinic, $C2$, $a = 26.084$ (8), $b = 4.732$ (2), $c = 7.612$ (3) Å, $\beta = 101.75$ (5)°, $Z = 2$, $D_m = 1.32$ Mg m⁻³, $D_x = 1.315$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu(\text{Cu } K\alpha) = 0.83$ mm⁻¹, $F(000) = 404$, room temperature. The structure of the linear

polyether–urea complex was solved from 774 diffractometer-measured reflections and refined to an R of 0.052. The polyether ligand, which has five O atoms in the chain, does not wrap circularly around the urea molecule to have the expected ‘monomer’ type of