

the diol with Si—O—Si link angles of 147.4 (3), 157.1 (4) and 162.5 (4)° (Hossain, 1980; Hossain & Hursthouse, 1987). In the tetraisopropyl derivative the two independent molecules present have link angles of 163.8 (1) and 164.1 (1)° (Clegg, 1983).

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Acta Cryst. (1988). C44, 1320–1321

Structure of a Hydroxyiodolactam

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(Received 8 December 1987; accepted 15 March 1988)

Abstract. 1-(1-Hydroxymethylpropyl)-5-iodomethyl-2-pyrrolidone, $C_9H_{16}INO_2$, $M_r = 297.14$, monoclinic, $P2_1/c$, $a = 7.419$ (1), $b = 19.562$ (3), $c = 8.156$ (1) Å, $\beta = 114.58$ (1)°, $V = 1076.5$ (3) Å³, $Z = 4$, D_x (130 K) = 1.83 g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 29.1$ cm⁻¹, $F(000) = 584$, $T = 130$ (1) K, $R = 0.045$ for 1953 unique reflections. The molecules crystallize as intermolecular hydrogen-bonded pairs. The O(2)···O(1') distance is 2.760 (9) Å, $(i) = -1 + x, \frac{1}{2} - y, z - \frac{1}{2}$. There is an I···I intermolecular contact of 3.942 (4) Å. The bond lengths and angles are all normal.

Experimental. Compound prepared by iodolactamization of 2-(3-butenyl)-4,5-dihydro-4-ethylloxazole as a mixture of (5*R**,1'*R**)- and (5*R**,1'*S**)-isomers [(1) and (2), respectively]. Purification by radial chromatography (LiChroprepTM Si60 PF, EtOAc:cyclohexane 1:1) gave (1) as a crystalline solid and (2) as an oil. Recrystallization of (1) from cyclohexane gave colorless needles and plates; crystal dimensions 0.25 × 0.25 × 1.00 mm; Syntex $P2_1$ diffractometer, locally modified LT-1 apparatus, $T = 130$ K, Mo $K\alpha$ radiation, graphite monochromator; cell dimensions from least-squares fit of 10 reflections with $17 < 2\theta < 26^\circ$; space group $P2_1/c$ based on conditions $h0l, l = 2n, 0k0, k = 2n$, range of absorption correction factors 1.83–2.45; data collected to $2\theta_{max} = 50^\circ$ with hkl ranges 0 to 8, 0 to 23, -9 to 9, respectively; ω scans, 2.0° range, 60° min⁻¹ speed, 1.8° offset for background; two check reflections monitored every 200 reflections displayed only random fluctuations with a maximum deviation from the average

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters (Å² × 10³)

Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
I	444 (1)	4678 (1)	2908 (1)	31 (1)
O(1)	6607 (8)	2690 (3)	1824 (7)	33 (2)
O(2)	484 (8)	2601 (3)	-886 (7)	32 (2)
N	4022 (9)	3445 (3)	839 (7)	21 (2)
C(1)	5726 (12)	3182 (4)	2088 (10)	25 (3)
C(2)	6356 (12)	3581 (4)	3830 (10)	29 (3)
C(3)	5146 (11)	4240 (4)	3157 (10)	28 (3)
C(4)	3309 (11)	4026 (4)	1534 (10)	23 (3)
C(5)	1591 (12)	3821 (4)	1996 (11)	27 (3)
C(6)	3186 (12)	3226 (4)	-1076 (10)	28 (3)
C(7)	3724 (13)	3719 (5)	-2226 (11)	36 (3)
C(8)	5877 (12)	3887 (5)	-1535 (10)	31 (3)
C(9)	993 (13)	3098 (4)	-1869 (10)	32 (3)

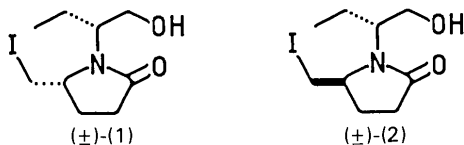
intensity of less than 2%; 2108 reflections measured, 1953 unique data, $R_{int} = 0.008$, 1758 observed [$I > 3\sigma(I)$] used in the solution and refinement (based on F); structure solved by heavy-atom methods; absorption correction [program *XABS*, a method which obtains an absorption tensor from $F_o - F_c$ differences; Hope & Moezzi (1987)] applied, blocked-cascade least-squares refinement, 124 parameters; all non-H atoms with anisotropic thermal parameters. H atoms bonded to C were included using a riding model with C—H of 0.96 Å and isotropic thermal parameters set equal to 1.2 times the equivalent isotropic thermal parameter of the bonded C atom (this requires the use of three additional parameters in order to define the methyl group); the hydroxyl H atom was refined using the constraint that it be staggered with respect to the C in a Newman projection down the O—C bond and U_{iso}

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Table 2. Bond lengths (Å) and angles (°)

I—C(5)	2.147 (9)	O(1)—C(1)	1.231 (11)
O(2)—C(9)	1.407 (12)	N—C(1)	1.353 (9)
N—C(4)	1.464 (11)	N—C(6)	1.484 (9)
C(1)—C(2)	1.514 (11)	C(2)—C(3)	1.537 (11)
C(3)—C(4)	1.512 (9)	C(4)—C(5)	1.526 (14)
C(6)—C(7)	1.510 (14)	C(6)—C(9)	1.501 (12)
C(7)—C(8)	1.493 (13)		
C(1)—N—C(4)	112.3 (6)	C(1)—N—C(6)	122.2 (7)
C(4)—N—C(6)	124.9 (5)	O(1)—C(1)—N	124.3 (7)
O(1)—C(1)—C(2)	126.0 (6)	N—C(1)—C(2)	109.7 (7)
C(1)—C(2)—C(3)	101.0 (6)	C(2)—C(3)—C(4)	105.1 (6)
N—C(4)—C(3)	102.4 (6)	N—C(4)—C(5)	111.4 (6)
C(3)—C(4)—C(5)	113.5 (7)	I—C(5)—C(4)	111.9 (5)
N—C(6)—C(7)	111.5 (7)	N—C(6)—C(9)	113.9 (8)
C(7)—C(6)—C(9)	111.0 (6)	C(6)—C(7)—C(8)	115.1 (6)
O(2)—C(9)—C(6)	111.7 (6)		

= 0.03 Å²; $R = 0.045$, $wR = 0.052$, $w = 1/[\sigma^2(F_o)]$, $S = 3.06$, $(\Delta/\sigma)_{\max} = 0.006$ for U_{33} of O(1); $(\Delta/\sigma)_{\text{av}} = 0.006$; $\Delta\rho$ excursions 1.02 and -0.98 e Å^{-3} , ca 1.0 Å from I; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); computer programs from *SHELXTL* (Sheldrick, 1985). The final atomic coordinates and isotropic thermal parameters for compound (1) are given in Table 1.* Bond lengths and angles for the iodolactam (1) are given in Table 2 and a drawing with the numbering scheme is given in Fig. 1.



* Lists of structure factors, anisotropic thermal parameters and calculated H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44856 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). **C44**, 1321–1323

Structure of 2-Neopentyl-2H-isoindole

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(Received 1 February 1988; accepted 15 March 1988)

Abstract. C₁₃H₁₇N, $M_r = 187.28$, monoclinic, $P2_1/c$, $a = 5.989 (5)$, $b = 16.870 (9)$, $c = 11.214 (7)$ Å, $\beta = 97.37 (5)^\circ$, $V = 1124 (1)$ Å³, $Z = 4$, $D_x = 1.107 \text{ Mg m}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu =$

0108-2701/88/071321-03\$03.00

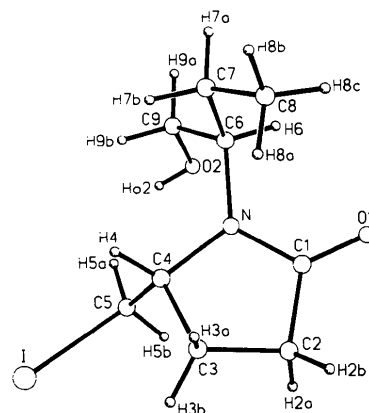


Fig. 1. A computer-generated drawing of (1) showing the relative stereochemistry and atom-numbering scheme.

Related literature. Two relevant crystal structures are those of 5-(1-methyl-2-oxacyclopropyl)pyrrolidin-2-one (Fray, Thomas & Wallis, 1983) and 2-(3-methyl-1-oxoisindolin-2-yl)butyramide (Carlström, Hacksell, Jönsson & Söderholm, 1983).

We thank the National Science Foundation for financial support of this work.

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0.06 mm⁻¹, $F(000) = 408$, $T = 291 (1)$ K, final $R = 0.044$ for 890 unique observed [$F \geq 3.0\sigma(F)$] diffractometer data. 2-Neopentyl-2H-isoindole is a typical exponent of the labile parent N heterocycle with a

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