KAFTORY, M. & DUNITZ, J. D. (1975). *Acta Cryst.* B31, 2917–2918.
KAFTORY, M. & DUNITZ, J. D. (1976). *Acta Cryst.* B32, 1–4.
LEISEROWITZ, L. (1976). *Acta Cryst.* B32, 775–802.
SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* B24, 63–76.
SCHWEIZER, W. B. (1977). Dissertation No. 5948, Federal Institute of Technology (ETH), Zürich, Switzerland.
STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1972). The XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1978). B**34**, 968–970

3-Amino-3-hydroxy-*trans*-bicyclo[4.4.0]decane-1-carboxylic Acid Lactam (*trans*-8a,2-Decahydro-2-naphtholcarbolactam)

BY DEREK J. CHADWICK AND JACK D. DUNITZ

Organic Chemistry Laboratory, Swiss Federal Institute of Technology, ETH-Zentrum, 8092 *Zürich, Switzerland*

(*Received* 12 *September* 1977; *accepted* 26 *October* 1977)

Abstract. $C_{11}H_{17}NO_2$, $M_r = 195{\cdot}14$, monoclinic, $C2/c$, $a = 28{\cdot}159\ (28)$, $b = 7{\cdot}404\ (7)$, $c = 10{\cdot}635\ (11)$ Å, $\beta = 70{\cdot}35\ (2)°$, $Z = 8$, $D_x = 1{\cdot}24$ g cm^{-3}. The title compound is usually named as the corresponding carboxamide–ketone isomer [octahydro-3-oxo-4a(2*H*)-naphthalenecarboxamide] but it exists both in the crystalline state and in solution as the lactam–alcohol. An appreciable out-of-plane distortion of the amide group can be detected. The hydroxyl and amide H atoms both participate in intermolecular hydrogen bonds.

Introduction. The compound (m.p. 165·5–166 °C) was synthesized by the method of Meyer & Schnautz (1962). Crystals were obtained by slow cooling of a saturated solution (1:1 ethyl acetate–cyclohexane). Data collection, structure analysis and refinement followed much the same lines as in Chadwick & Dunitz (1978); the crystal used was 0·3 × 0·3 × 0·2 mm on edge, and 2276 reflexions ($2\theta < 54°$) were measured, 1787 with $I \geq 3\sigma(I)$. The final R was 0·040 (no extinction correction required). Final positional and mean thermal parameters are given in Table 1.* Fig. 1 shows the molecular structure, Fig. 2 bond lengths,

Table 1. *Atomic coordinates and isotropic U values* (× 10^4 *for* C *and* O, × 10^3 *for* H *atoms; e.s.d.'s in parentheses*)

Non-hydrogen atoms were refined anisotropically.

	x	y	z	$\bar{U}$ (Å^2)
C(1)	9391 (1)	5532 (4)	934 (3)	469 (17)
C(2)	9581 (1)	7209 (5)	1472 (3)	486 (18)
C(3)	9314 (1)	8898 (4)	1257 (3)	371 (14)
C(4)	8750 (1)	8712 (4)	1965 (3)	400 (15)
C(5)	8047 (1)	7276 (5)	1262 (3)	547 (19)
C(6)	7897 (1)	5729 (6)	523 (4)	675 (24)
C(7)	8101 (1)	3930 (5)	783 (4)	625 (22)
C(8)	8668 (1)	4017 (4)	462 (3)	488 (18)
C(9)	8819 (1)	5488 (4)	1257 (3)	368 (14)
C(10)	8611 (1)	7366 (4)	1050 (3)	343 (13)
C(11)	8944 (1)	8055 (4)	−317 (3)	387 (14)
O(1)	8884 (1)	7771 (3)	−1394 (2)	557 (13)
O(2)	9547 (1)	10437 (3)	1571 (2)	548 (13)
N	9326 (1)	8988 (3)	−136 (2)	387 (12)

	x	y	z	U (Å^2)
H(1)C(1)	956 (1)	549 (3)	−11 (3)	39 (7)
H(2)C(1)	953 (1)	441 (4)	118 (3)	67 (10)
H(1)C(2)	996 (1)	730 (4)	102 (2)	49 (8)
H(2)C(2)	954 (1)	712 (4)	243 (3)	58 (8)
H(1)C(4)	856 (1)	991 (4)	197 (3)	49 (8)
H(2)C(4)	866 (1)	830 (4)	292 (3)	45 (8)
H(1)C(5)	794 (1)	841 (5)	98 (4)	88 (11)
H(2)C(5)	788 (1)	710 (4)	225 (3)	53 (8)
H(1)C(6)	801 (1)	602 (4)	−43 (3)	76 (9)
H(2)C(6)	753 (2)	575 (6)	69 (4)	98 (13)
H(1)C(7)	800 (1)	295 (5)	27 (4)	79 (11)
H(2)C(7)	794 (1)	366 (5)	174 (3)	67 (10)
H(1)C(8)	882 (1)	429 (4)	−51 (3)	53 (8)
H(2)C(8)	883 (1)	280 (5)	57 (3)	73 (10)
HC(9)	864 (1)	528 (3)	224 (3)	38 (7)
HN	963 (1)	925 (4)	−81 (3)	62 (8)
HO(2)	928 (1)	1104 (5)	223 (4)	102 (11)

---

* Lists of structure factors and anisotropic thermal parameters (for C, N and O atoms) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33125 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

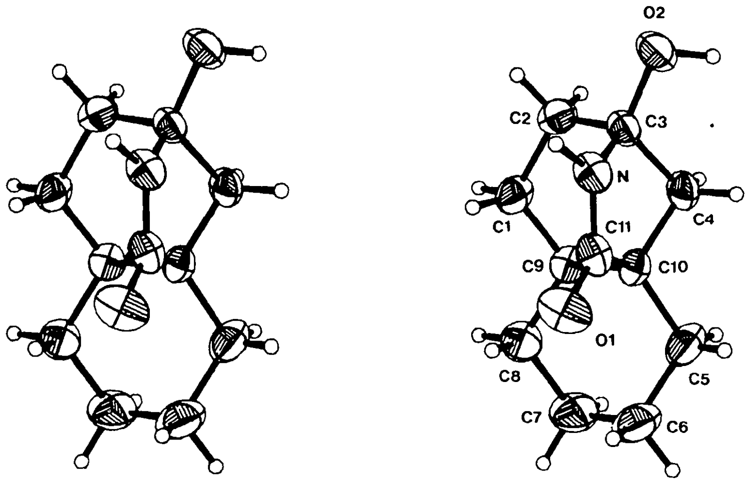
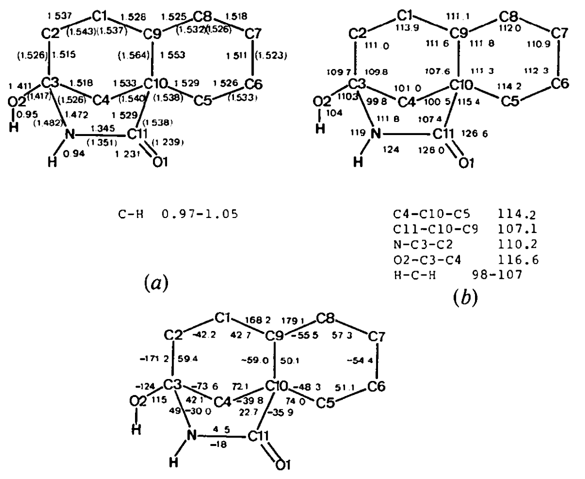


Fig. 1. Stereoscopic view of the molecule drawn by *ORTEP* (Johnson, 1965). The thermal-vibration ellipsoids are at the 50% probability level.



C-H 0.97-1.05

(*a*)

C4-C10-C5	114.2
C11-C10-C9	107.1
N-C3-C2	110.2
O2-C3-C4	116.6
H-C-H	98-107

(*b*)

C1-C2-C3-N	-49.6
C1-C9-C10-C5	175.2
C1-C9-C10-C11	48.3
C2-C3-N-C11	85.4
C3-C4-C10-C5	-163.9
C3-N-C11-O1	-173.6
C4-C10-C9-C8	175.9
C4-C10-C5-C6	-170.4
C4-C10-C11-O1	-159.2
C5-C10-C11-N	145.9
C8-C9-C10-C11	-76.8
C9-C10-C11-O1	88.6
C9-C10-C11-N	-89.6
C10-C4-C3-O2	160.8
C11-N-C3-O2	-153.3
C2-C3-N-H	-72
C4-C3-O2-H	2
C4-C3-N-H	173
C10-C11-N-H	161

(*c*)

Fig. 2. (*a*) Bond lengths (Å) (values corrected for molecular libration in brackets), (*b*) bond angles (°), and (*c*) torsion angles (°). E.s.d.'s are 0·003–0·006 Å for bond lengths, 0·2–0·4° for bond angles not involving H atoms and somewhat larger for torsion angles.

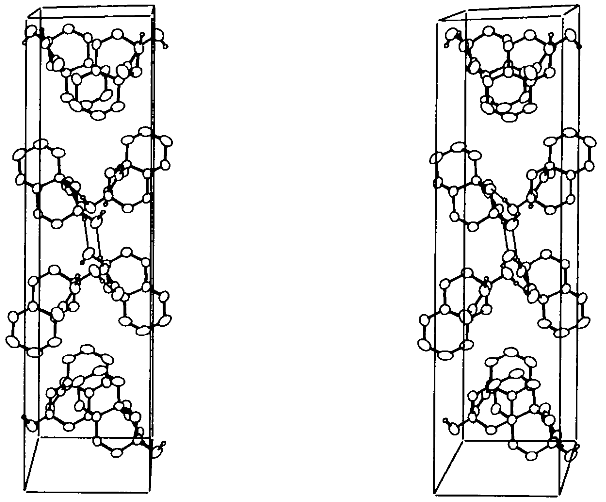


Fig. 3. Stereoscopic view of the crystal structure drawn by *ORTEP* (Johnson, 1965). The axial directions are **a**↑, **b**→ and **c** into the plane of the paper. Hydrogen bonds are indicated.

angles and torsion angles, and Fig. 3 the crystal packing.

Discussion. Of main interest in this analysis is the conformation of the α-hydroxylactam moiety. Although the ring torsion angle ω_1 about the amide bond N–C(11) is only 4°, the amide group as a whole shows considerable deviations from planarity, the out-of-plane parameters (Winkler & Dunitz, 1971; Dunitz & Winkler, 1975) being $\chi_N = -24°$, $\chi_C = -1·9°$, $\tau =$

−6·5°. The χ_N deformation is remarkably large, being exceeded only in molecules where the non-planarity of the amide group is enforced by severe ring-fusion constraints (van der Helm, Ealick & Washecheck, 1975) – which is certainly not the case here. The value of χ_N depends heavily on the amide H position; however, the observed displacement of this atom from the OC′N plane amounts to 0·24 Å or about 8 e.s.d.'s, so we believe the deformation to be real. In one experiment, the amide H was placed at a trial position in the mean plane of the other amide group atoms, but it moved back, close to the reported position, on least-squares refinement.

The out-of-plane displacement of the amide H may be required by the contingencies of optimal hydrogen bonding, but it may also be due to interaction between the filled *p* orbital of the N atom and the antibonding (σ^*) orbital of the adjacent C(3)–O(2) bond. Interaction between lone-pair and 'superjacent' σ^* orbitals has been invoked for the interpretation of the anomeric effect (Altona, 1964; David, Eisenstein, Hehre, Salem & Hoffmann, 1973). By analogy, the σ^* orbital of the electronegative C–O bond could be said to induce some degree of pyramidalization of the N atom in the interests of optimal overlap – a kind of 'reverse anomeric effect'. In this connexion, it should be noted that the H atom is displaced to the *opposite* side of the amide plane from the hydroxyl group, so the developing lone pair is on the *same* side as this group, leading to a more *syn* than *anti* orientation. The inverted displacement would tend to make the developing lone pair orthogonal to the C–O bond.

The remaining structural features merit no special comment. Analysis of the vibrational ellipsoids in terms of rigid-body motion (Schomaker & Trueblood, 1968) leads to quite satisfactory agreement between observed and calculated U_{ij} values $[\langle \Delta^2(U_{ij})\rangle^{1/2} = 0{\cdot}0021\ \text{Å}^2$, $\langle \sigma^2(U_{ij})\rangle^{1/2} = 0{\cdot}0016\ \text{Å}^2]$. Librational corrections to bond lengths amount to 0·006–0·012 Å (see Fig. 2); angle corrections are negligible.

The crystal packing (Fig. 3) is dominated by hydrogen bonding. Pairs of molecules related by inversion centres are linked into dimers by N–H···O(2) bonds of length 3·05 Å, and chains of molecules related by the *c* glide operation are linked by O(2)–H···O(1) bonds of length 2·69 Å.

This work was carried out with the financial support of the Swiss National Science Foundation. One of us (DJC) is indebted to the Royal Society of London for the award of a European Science Exchange Fellowship.

References

Altona, C. (1964). PhD Thesis, Univ. of Leiden.

Chadwick, D. J. & Dunitz, J. D. (1978). *Acta Cryst.* B**34**, 965–968.

David, S., Eisenstein, O., Hehre, W. J., Salem, L. & Hoffmann, R. (1973). *J. Am. Chem. Soc.* **95**, 3806–3807.

Dunitz, J. D. & Winkler, F. K. (1975). *Acta Cryst.* B**31**, 251–263.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Meyer, W. L. & Schnautz, N. G. (1962). *J. Org. Chem.* **27**, 2011–2015.

Schomaker, V. & Trueblood, K. N. (1968). *Acta Cryst.* B**24**, 63–76.

Van der Helm, D., Ealick, S. E. & Washecheck, D. (1975). *Acta Cryst.* A**31**, 104.

Winkler, F. K. & Dunitz, J. D. (1971). *J. Mol. Biol.* **59**, 169–182.

Acta Cryst. (1978). B**34**, 970–973

2′-Hydroxy-5′-[1-hydroxy-2-(3″-pentylamino)propyl]fluoromethanesulfonanilide Hydrochloride

By M. Mathew and Gus J. Palenik

Center for Molecular Structure, Department of Chemistry, University of Florida, Gainesville, Florida 32611, *USA*

(*Received* 18 *July* 1977; *accepted* 26 *October* 1977)

Abstract. $C_{15}H_{26}FN_2O_4ClS$, $M_r = 384{\cdot}90$, triclinic, $P\bar{1}$, $a = 8{\cdot}538\ (5)$, $b = 8{\cdot}810\ (5)$, $c = 13{\cdot}874\ (8)$ Å, $\alpha = 93{\cdot}76\ (5)$, $\beta = 108{\cdot}06\ (4)$, $\gamma = 103{\cdot}15\ (4)$, $V = 955{\cdot}6\ (9)$ Å^3, $Z = 2$, $D_x = 1{\cdot}338$, $D_m = 1{\cdot}334$ g cm^{-3}. The final R was 0·075 for the 1712 reflections used in the analysis. The OH of the side chain is *trans* to the amino N, a conformation not found in any of the active bronchodilators.