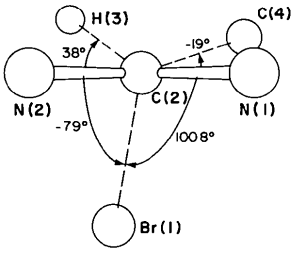


Fig. 2. Newman projection looking down the C(2)—C(3) bond.

References

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.

COBBLEDICK, R. E. & SMALL, R. W. H. (1973). *Acta Cryst.* B**29**, 1659–1666.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

LEVINE, B. (1963). *J. Chem. Phys.* **38**, 2326.

MASON, R. & ROBERTSON, G. B. (1966). *Advances in Structure Research by Diffraction Methods*, Vol. 2, edited by R. BRILL & R. MASON, p. 57. New York: Interscience.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

ONDRUS, T. A., KNAUS, E. E. & GIAM, C. S. (1979). *Can. J. Chem.* **57**, 2342–2349.

ONDRUS, T. A., PEDNEKAR, P. & KNAUS, E. E. (1985). *Can. J. Chem.* **63**, 2362–2368.

STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The *XRAY*70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

WARREN, B. K. & KNAUS, E. E. (1982). *J. Heterocycl. Chem.* **19**, 1259–1260.

Acta Cryst. (1987). C**43**, 565–567

β-Propiolactam (1-Aza-2-cyclobutanone) at 170 K

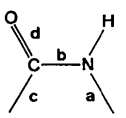
BY QING-CHUAN YANG,* PAUL SEILER AND JACK D. DUNITZ

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

(*Received* 17 *September* 1986; *accepted* 20 *October* 1986)

Abstract. C_3H_5NO, $M_r = 71{\cdot}08$, space group $P\bar{1}$, $a = 5{\cdot}011\ (2)$, $b = 5{\cdot}188\ (2)$, $c = 7{\cdot}395\ (4)$ Å, $\alpha = 107{\cdot}82\ (4)$, $\beta = 106{\cdot}04\ (4)$, $\gamma = 99{\cdot}23\ (3)°$, $V = 169{\cdot}48$ Å^3 at 170 K, $Z = 2$, $D_x = 1{\cdot}39$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}66$ cm^{-1}, $F(000) = 76$, $R = 0{\cdot}037$ for 595 observed reflections. The four-membered ring is exactly planar (to within 0·001 Å), and the carbonyl O and amide H atoms also lie almost exactly in this plane [displacements −0·010 (1) and 0·005 (20) Å, respectively]. The molecules form centrosymmetric dimers linked by N—H···O hydrogen bonds.

Introduction. The recent synthesis of β-propiolactam by Pfaendler & Hoppe (1985) provides an opportunity to extend our knowledge about the structural details of the *cis* amide group (1) in the direction of small ring size.



(1)

* On leave from Chemistry Department, Peking University, Beijing, China.

Experimental. A sample provided by Professor H. R. Pfaendler (University of Munich) was recrystallized from tetrachloromethane. Needles, crystal size 0·15 × 0·25 × 0·70 mm, handled in dry atmosphere and enclosed in capillary; Enraf–Nonius CAD-4 diffractometer with cooling device; Mo radiation, graphite monochromator; ω–θ scan; unit-cell dimensions by least-squares fit to setting angles of 14 automatically centred reflections with $6 < \theta < 15°$. No absorption correction. Max. $(\sin\theta)/\lambda$ in intensity measurements 0·64 Å^{-1}. Three data sets measured: at room temperature, 223 K and 170 K (see Table 1). All lead to similar results, and only the 170 K measurements and results are discussed here in detail as they are slightly more accurate than the others. Three standard reflections, *ca* 10% intensity loss during measurement period. 800 reflections measured, 741 unique, $R_{int} = 0{\cdot}028$, 595 counted as observed [$I > 3\sigma(I)$], index range h

Table 1. *Cell dimensions for β-propiolactam at three temperatures*

T (K)	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)
170	5·011 (2)	5·188 (2)	7·395 (4)	107·82 (4)	106·04 (4)	99·23 (3)
223	5·038 (1)	5·199 (1)	7·402 (3)	106·77 (3)	106·53 (3)	99·37 (3)
293 (RT)	5·077 (1)	5·220 (2)	7·458 (2)	107·00 (3)	106·07 (3)	99·54 (3)

0108-2701/87/030565-03$01.50

−6/5, *k* −6/6, *l* 0/9. Structure solved by direct methods using room-temperature data with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refined by full-matrix least-squares analysis on *F* values with *SHELX*76 (Sheldrick, 1976). All H atoms were located in difference maps and included in the least-squares refinement with isotropic Gaussian displacement parameters (other atoms anisotropic). For 170 K data, $R = 0.0373$, $wR = 0.0386$ with weights w assigned as $[\sigma^2(F) + 0.0003\,F^2]^{-1}$. Max. Δ/σ in final refinement cycle 0·20 for heavy-atom positional parameters, 0·17 for heavy-atom anisotropic displacement parameters, 0·48 for H positional parameters, 1·28 for H displacement parameters. Max. and min. residual electron density in final difference Fourier synthesis 0·20, −0·36 e Å^{-3}. Atomic scattering factors for C, N, O from Cromer & Mann (1986), for H from Stewart, Davidson & Simpson (1965).

Discussion. The final positional parameters are given in Table 2* together with isotropic displacement parameters *U* (contracted from **U** for C, N, O). Bond distances and angles are given in Table 3. See Fig. 1 for atom labelling.

The exact planarity of the amide group in β-propiolactam itself contrasts with the extreme pyramidality of the amide N atom (Δ_N ~0·54 Å) in strained β-lactam antibiotics (Pfaendler, Gosteli, Woodward & Rihs, 1981). The bond lengths and angles in the amide group also show considerable differences from those in strained β-lactam structures and may serve as reference values for the unstrained system in energy calculations.

Dimensions of the *cis* amide group in accurately determined lactam structures are listed in Table 3. Some trends are evident; the changes in bond angle are obviously heavily dependent on geometrical factors, and the decrease of ~0·02 Å in the C—O bond on going from the eight- to the four-membered ring has the concomitant rehybridization at the trigonal C atom as its mostly likely origin.

In the crystal structure the molecules are paired into centrosymmetric dimers linked by NH···O hydrogen bonds [N···O 2·967 (2) Å; angle N—H···O 153 (2)°; angle H···O=C 120 (1)°]. The planes of the two molecules are parallel but not quite coplanar, being

Table 2. *Atomic coordinates and isotropic Gaussian displacement parameters U (all × 10⁴) with e.s.d.'s in parentheses from analysis of the* 170 K *data*

For C, N, O atoms the *U* values are estimated as tr **U**/3 after transformation of **U** to an orthogonal coordinate system.

	x	*y*	*z*	*U*
O(1)	557 (2)	−3444 (2)	−1516 (1)	277 (3)
N(2)	1870 (2)	1016 (2)	−1648 (2)	252 (3)
C(3)	1737 (3)	−1697 (3)	−2050 (2)	201 (3)
C(4)	3545 (3)	−1689 (3)	−3398 (2)	242 (4)
C(5)	3622 (3)	1435 (3)	−2880 (2)	257 (4)
H(21)	1188 (41)	2138 (44)	−920 (31)	431 (50)
H(41)	2486 (39)	−2865 (39)	−4802 (26)	336 (42)
H(42)	5317 (39)	−2024 (39)	−2868 (25)	397 (45)
H(51)	5561 (35)	2751 (35)	−2070 (22)	267 (37)
H(52)	2553 (41)	1930 (40)	−4014 (27)	320 (42)

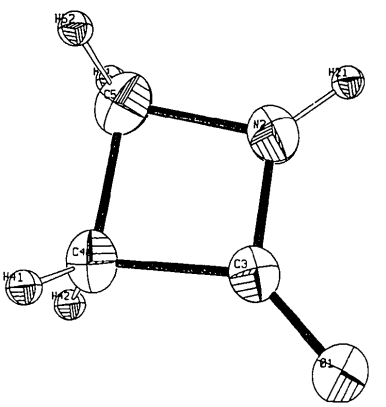


Fig. 1. β-Propiolactam molecule showing atomic displacement ellipsoids (50%) and atomic labelling.

* Lists of structure factors, anisotropic thermal parameters and geometrical parameters involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43501 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond lengths* (Å) *and angles* (°) *in the cis amide grouping of lactams* (*n* = *ring size*)

For explanation of symbols see (1).

Compound	*n*	*a*	*b*	*c*	*d*	*ab*	*bc*	*bd*	*cd*	Reference
Enantholactam (98 K)	8	1·462	1·334	1·510	1·246	127·8	119·0	120·0	121·0	(*a*)
Caprolactam (RT)	7	1·470	1·327	1·501	1·242	125·5	118·5	120·9	120·6	(*b*)
Valerolactam (138 K)	6	1·462	1·333	1·512	1·243	126·4	117·9	122·0	120·1	(*c*)
Various	5	1·455	1·335	1·514	1·232	114·9	108·4	125·8	125·7	(*d*)
Propiolactam (170 K)	4	1·467 (2)	1·333 (2)	1·522 (2)	1·226 (1)	96·2 (1)	91·7 (1)	132·4 (1)	135·9 (1)	(*e*)

Values for *n* = 5 are averages over 41 variously substituted γ-lactams and have e.s.d.'s < 0·01 Å for distances and < 1·0° for angles. For the other ring sizes the values are taken from a single relatively accurate structure (uncorrected for thermal motion) and have e.s.d.'s of ~ 0·002 Å for distances and ~ 0·2° for angles.
References: (*a*) Winkler & Seiler (1979); (*b*) Winkler & Dunitz (1975); (*c*) van der Helm & Ekstrand (1979); (*d*) Norskov-Lauritsen, Bürgi, Hofmann & Schmidt (1985); (*e*) this work.
Other bond lengths and angles in propiolactam (atom numbering as in Fig. 1).

C(4)—C(5)	1·538 (2)	C(3)—C(4)—C(5)	85·9 (1)	H(21)—N(2)—C(3)	132·3 (13)
N(2)—H(21)	0·85 (2)	C(4)—C(5)—N(2)	86·2 (1)	H(21)—N(2)—C(5)	131·5 (13)

mutually displaced by 0·36 Å. In keeping with the relatively short C=O bond, the N···O distance is 0·10–0·15 Å longer than the corresponding hydrogen-bonded distance in the other lactam structures mentioned in Table 3.

References

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

NORSKOV-LAURITSEN, L., BÜRGI, H.-B., HOFMANN, P. & SCHMIDT, H. R. (1985). *Helv. Chim. Acta*, **68**, 76–82.

PFAENDLER, H. R., GOSTELI, J., WOODWARD, R. B. & RIHS, G. (1981). *J. Am. Chem. Soc.* **103**, 4526–4531.

PFAENDLER, H. R. & HOPPE, H. (1985). *Heterocycles*, **23**, 265–272.

SHELDRICK, G. M. (1976). *SHELX*76. *Program for crystal structure determination.* Univ. of Cambridge, England.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

VAN DER HELM, D. & EKSTRAND, J. D. (1979). *Acta Cryst.* B**35**, 3101–3103.

WINKLER, F. K. & DUNITZ, J. D. (1975). *Acta Cryst.* B**31**, 268–269.

WINKLER, F. K. & SEILER, P. (1979). *Acta Cryst.* B**35**, 1920–1922.

Acta Cryst. (1987). C**43**, 567–570

Structures of Salsoline Hydrochloride Hydrate and Salsolidine Hydrochloride Dihydrate

BY HERMAN L. AMMON, SATYA M. PRASAD,* DAVID M. BARNHART† AND V. K. SYAL‡

Department of Chemistry, University of Maryland, College Park, Maryland 20742, *USA*

KARIMAT EL-SAYED

Physics Department, Faculty of Science, Ain Shams University, Cairo, Egypt

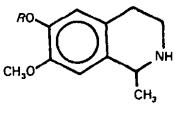
AND GAMILA M. WASSEL

Pharmaceutical Sciences Laboratory, National Research Center, Tahrir Street, Dokki Cairo, Egypt

(*Received* 16 *May* 1986; *accepted* 24 *September* 1986)

Abstract. $C_{11}H_{16}NO_2^+.Cl^-.H_2O$: $M_r = 247{\cdot}72$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}856$ (2), $b = 23{\cdot}246$ (6), $c = 6{\cdot}854$ (2) Å, $V = 1252$ (1) Å^3, $D_x = 1{\cdot}314$ Mg m^{-3} for $Z = 4$, $\mu = 2{\cdot}67$ mm^{-1}, $F(000) = 528$, diffractometer data λ(Cu $K\alpha$) = 1·5418 Å, $T = 293$ K, 1185 reflections, 1086 with $I > 3\sigma(I)$, $R = 0{\cdot}042$. $C_{12}H_{18}NO_2^+.Cl^-.2H_2O$: $M_r = 279{\cdot}76$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}293$ (3), $b = 12{\cdot}605$ (7), $c = 15{\cdot}708$ (8) Å, $V = 1444$ (2) Å^3, $D_x = 1{\cdot}287$ Mg m^{-3} for $Z = 4$, $\mu = 2{\cdot}42$ mm^{-1}, $F(000) = 600$, 1373 reflections, 1251 with $I > 3\sigma(I)$, $R = 0{\cdot}053$. There is extensive hydrogen bonding involving the phenolic OH, methoxy oxygens, water molecules, ammonium NH_2's and chloride anions.

Introduction. Salsoline [1,2,3,4-tetrahydro-7-methoxy-1-methylisoquinolin-6-ol (I)] and its methyl ether salsolidine [1,2,3,4-tetrahydro-6,7-dimethoxy-1-methylisoquinoline (II)] are found in the upper part of the west Mediterranean plant *Salsola kali* L. and *S. longifolia* Forsk (Chenopodiaceae). They have been isolated also from some European species of the genus *Salsola* (Orechoff & Proskurnina, 1934; Khalimatov & Rustamov, 1963; Rizaev, Rustamov, Muslimov & Khalmatov, 1967). These alkaloids have been found effective in the treatment of hypertension by reducing blood pressure and stimulating respiration (Gvishiani, 1939; Wastl, 1946; Krylov, Nauchn & Ryzausk, 1962). The crystal structures of the hydrochlorides of salsoline (III) and salsolidine (IV) are reported. Both crystal forms are hydrated.



(I): $R = H$
(II): $R = CH_3$
(III): (I) + HCl + H_2O
(IV): (II) + HCl + $2H_2O$

* Permanent address: Physics Department, Ranchi University, Ranchi 834008, India.

† Permanent address: Department of Physics, Eastern Montana College, Billings, Montana 59101, USA.

‡ Permanent address: Chemistry Department, Himachal Pradesh University, Simla-171005, India.

0108-2701/87/030567-04$01.50