

The final atomic parameters are given in Table 1. Selected interatomic distances and angles are given in Table 2. Molecule *A* is depicted in Fig. 1. The greatest differences between the distances and angles of molecules *A* and *B* are 0.037 (4.4 $\sigma$ ) and 0.027 Å (2.8 $\sigma$ ) in C4—C5 and C72—O73, respectively, and 3.6 (9.0 $\sigma$ ) and 3.5° (7.0 $\sigma$ ) in C4—C7—O7 and C7—C4—C5, respectively. The shortest intermolecular contacts are O(103*A*)...H(94*B*<sup>i</sup>)—C(94*B*<sup>i</sup>) [2.54, 0.96 Å, 140°, (i) = *x*, *y*, 1+*z*], C(104*A*)—H(102*A*)...O(93*B*<sup>ii</sup>) [2.65, 0.96 Å, 128°, (ii) = 1+*x*, *y*, *z*−1] and O(93*A*)...H(104*B*<sup>i</sup>)—C(104*B*<sup>i</sup>) (2.67, 0.96 Å, 118°).

**Related literature.** The bond distances and angles in cyclohexene, acetoxybutyl and in the chain C4—C7—C8—C9—C10 are similar to those reported by Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987), Gieren & Kokkinidis (1986) and Jiménez-Garay, López-Castro & Márquez (1976), respectively.

We are grateful to Professor J. Pacák for providing the single crystals.

#### References

- ALLEN, F. H., KENNARD, O., WATSON, D. G., BRAMMER, L., ORPEN, A. G. & TAYLOR, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. 21–219.  
 DUAX, W. L., WEEKS, C. M. & ROHRER, D. C. (1976). *Top. Stereochem.* **9**, 271–273.  
 GIEREN, A. & KOKKINIDIS, M. (1986). *Z. Naturforsch. Teil C*, **41**, 618–626.  
 JIMÉNEZ-GARAY, R., LÓPEZ-CASTRO, A. & MÁRQUEZ, R. (1976). *Acta Cryst.* **B32**, 1367–1371.  
 KELLER, E. (1986). *SCHAKAL86. Chem. Unserer Zeit*, **20**, 178–181.  
 PACÁK, J. (1987). Private communication.  
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.  
 SHELDRIK, G. M. (1986). *SHELXS86*. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.

*Acta Cryst.* (1989). **C45**, 1084–1086

## Structure of *cyclo(-N-Hydroxyglycyl-L-alanyl-)*

BY YOSHINOBU YOKOMORI\*

*Department of Chemistry, The National Defense Academy, Hashirimizu, Yokosuka 239, Japan*

AND AKIRA KATOH AND MASAYASU AKIYAMA

*Department of Applied Chemistry for Resources, Tokyo University of Agriculture and Technology, Koganei, Tokyo 184, Japan*

(Received 24 June 1988; accepted 14 December 1988)

**Abstract.** C<sub>5</sub>H<sub>8</sub>N<sub>2</sub>O<sub>3</sub>, *M<sub>r</sub>* = 144.1, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 18.081 (3), *b* = 7.760 (1), *c* = 4.615 (1) Å, *V* = 647 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.478, *D<sub>m</sub>*(flotation in toluene/carbon tetrachloride) = 1.468 (6) Mg m<sup>−3</sup>, λ(Cu *Kα*) = 1.5418 Å, μ(Cu *Kα*) = 1.011 mm<sup>−1</sup>, *F*(000) = 304.0, *T* = 296 K, *R* = 0.042, *wR* = 0.061, for 590 observations. The present diketopiperazine ring adopts an almost flat conformation. There are two kinds of hydrogen bonds: O<sub>3</sub>—H...O<sub>2</sub>( $\frac{3}{2}$ −*x*, 1−*y*,  $\frac{1}{2}$ +*z*) 2.599 (1) Å; and N<sub>2</sub>—H...O<sub>1</sub>(2−*x*, − $\frac{1}{2}$ +*y*,  $\frac{3}{2}$ −*z*) 2.878 (1) Å.

**Experimental.** Colorless cubic crystals by slow evaporation of an aqueous methanol solution at room temperature. Crystal 0.3 × 0.3 × 0.05 mm. Rigaku-Denki RASA 5R-II automated diffractometer. Systematic absences *h*00 for *h* odd, 0*k*0 for *k* odd, 00*l* for *l* odd. Cell constants by least squares using 15 reflec-

tions with 40° ≤ 2θ(Cu) ≤ 80° measured on the diffractometer. Intensity data collected in ω−2θ scan mode. 603 independent reflections, 2θ ≤ 120.0°, 0 ≤ *h* ≤ 20, 0 ≤ *k* ≤ 8, 0 ≤ *l* ≤ 5. No systematic fluctuations in 11 $\bar{3}$ , 732, 91 $\bar{2}$ , monitored every 100 reflections.

Structure determined using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Block-diagonal least-squares refinement (*HBLISIV*: Ashida, 1981).  $\sum w(|F_o| - k|F_c|)^2$  minimized. Weighting scheme:  $w = [\sigma^2(F_o) + 0.0590|F_o| + 0.0013|F_o|^2]^{-1}$ . Subsequent difference Fourier maps revealed positions for all 8 H atoms; however, all H atoms were fixed at calculated positions (with C—H = 1.08 Å) with fixed isotropic temperature factors (*B* = 5.0 Å<sup>2</sup>). Final cycle of block-diagonal least squares gave *R* = 0.042, *wR* = 0.061, *S* = 0.69, using 590 observations with |*F<sub>o</sub>*| > 2σ(*F<sub>o</sub>*) and 92 variables. (*Δ*/σ)<sub>max</sub> = 0.033. Final difference map contained no peak higher than 0.4 e Å<sup>−3</sup>. Atomic scattering factors

\* Author to whom correspondence should be addressed.

from *International Tables for X-ray Crystallography* (1974). The molecule and numbering scheme are shown in Fig. 1 and a stereoscopic view in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for non-H atoms are given in Table 1.\* Bond lengths and angles are given in Table 2.

**Related literature.** The flat conformation has been assigned to this molecule in DMSO (Akiyama, Katoh & Tsuchiya, 1988). A number of related diketopiperazine compounds have been reported (Radding, Donzel, Ueyama & Goodman, 1980; Benedetti, Marsh & Goodman, 1976; Lieberek, Bednarek, Kitowska &

\* Lists of structure amplitudes, H-atom positional parameters and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51682 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

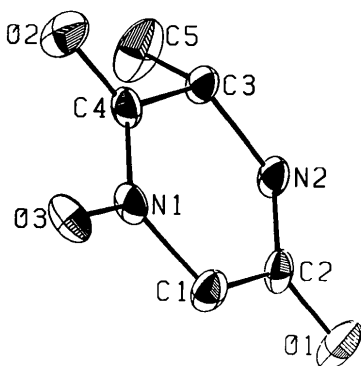


Fig. 1. View of the molecule. Thermal ellipsoids are at 50% probability (ORTEPII, Johnson, 1976).

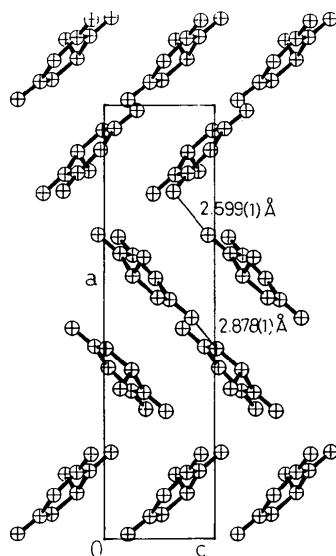


Fig. 2. The packing in the cell.

Table 1. Final atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^2$ ), with e.s.d.'s in parentheses

$$B_{eq} = \frac{1}{3} \sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$B_{eq}$
O1	9881 (1)	4215 (3)	12912 (6)	382 (9)
O2	7978 (1)	2648 (3)	4414 (5)	384 (9)
O3	8027 (1)	5872 (3)	6249 (4)	323 (8)
N1	8443 (1)	4591 (3)	7556 (5)	248 (8)
N2	9418 (1)	2261 (3)	9831 (6)	258 (9)
C1	8976 (2)	5216 (4)	9624 (7)	301 (11)
C2	9461 (1)	3836 (4)	10897 (6)	252 (10)
C3	8924 (1)	1657 (4)	7538 (6)	267 (11)
C4	8410 (1)	3030 (4)	6406 (6)	257 (10)
C5	8486 (2)	95 (4)	8555 (9)	440 (15)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

O1—C2	1.236 (3)	O3—N1—C1	114.3 (2)
O2—C4	1.242 (3)	O3—N1—C4	117.2 (2)
O3—N1	1.384 (3)	C1—N1—C4	127.2 (2)
N1—C1	1.440 (4)	C2—N2—C3	127.1 (2)
N1—C4	1.323 (3)	N1—C1—C2	114.2 (2)
N2—C2	1.319 (3)	O1—C2—N2	122.5 (2)
N2—C3	1.461 (3)	O1—C2—C1	118.9 (2)
C1—C2	1.502 (4)	N2—C2—C1	118.6 (2)
C3—C4	1.506 (4)	N2—C3—C4	113.7 (2)
C3—C5	1.521 (4)	N2—C3—C5	110.4 (2)
		C4—C3—C5	110.4 (2)
		O2—C4—N1	123.0 (2)
		O2—C4—C3	118.4 (2)
		N1—C4—C3	118.6 (2)

Macikowska, 1977; Calcagni, Mazza, Pochetti, Rossi & Lucente, 1985; Groth, 1969; Ramani, Sasisekharan & Venkatesan, 1977; Degenh & Marsh, 1959; Benedetti, Corradini & Pedone, 1969; Sletten, 1970; Suguna, Ramakumar, Shamala, Venkataramprasad & Balaram, 1982).

#### References

- AKIYAMA, M., KATOH, A. & TSUCHIYA, Y. (1988). *J. Chem. Soc. Perkin Trans. 1*. In the press.
- ASHIDA, T. (1981). *HBL SIV. The Universal Computing System—Nagoya*, pp. 65–70. Nagoya Univ., Japan.
- BENEDETTI, E., CORRADINI, P. & PEDONE, C. (1969). *J. Phys. Chem.* **73**, 2891–2895.
- BENEDETTI, E., MARSH, R. E. & GOODMAN, M. (1976). *J. Am. Chem. Soc.* **98**, 6676–6684.
- CALCAGNI, A., MAZZA, F., POCHETTI, G., ROSSI, D. & LUCENTE, G. (1985). *Int. J. Pept. Protein Res.* **26**, 166–173.
- DEGEILH, R. & MARSH, R. E. (1959). *Acta Cryst.* **12**, 1007–1014.
- GROTH, P. (1969). *Acta Chem. Scand.* **23**, 3155–3162.
- International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 202–207. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- LIEBEREK, B., BEDNAREK, M., KITOWSKA, A. & MACIKOWSKA, A. (1977). *Rocz. Chem.* **51**, 189–191.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

RADDING, W., DONZEL, B., UHEYAMA, N. & GOODMAN, M. (1980). *J. Am. Chem. Soc.* **102**, 5999–6005.  
 RAMANI, R., SASISEKHARAN, V. & VENKATESAN, K. (1977). *Int. J. Pept. Protein Res.* **9**, 277–292.

SLETTEN, E. (1970). *J. Am. Chem. Soc.* **92**, 172–177.  
 SUGUNA, K., RAMAKUMAR, S., SHAMALA, N., VENKATARAM-PRASAD, B. V. & BALARAM, P. (1982). *Biopolymers*, **21**, 1847–1855.

*Acta Cryst.* (1989). **C45**, 1086–1087

### 3-(2-Methoxyphenyl)-1H-2-benzopyran-1-one

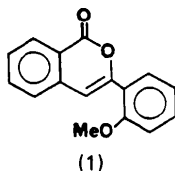
BY PHILIPPE PRINCE, JINA A. MILLER, FRANK R. FRONCZEK AND RICHARD D. GANDOUR\*

*Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA*

(Received 11 October 1988; accepted 16 December 1988)

**Abstract.**  $C_{16}H_{12}O_3$ ,  $M_r = 252.3$ , orthorhombic,  $Pna2_1$ ,  $a = 6.633$  (3),  $b = 13.367$  (2),  $c = 14.056$  (2) Å,  $V = 1246.3$  (9) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.344$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54184$  Å,  $\mu = 7.17$  cm<sup>-1</sup>,  $F(000) = 528$ ,  $T = 296$  K,  $R = 0.027$  for 2337 observations (of 2508 unique data). The average deviation from planarity is 0.013 (1) Å with a maximum of 0.028 (1) Å for the fused-rings system, and 0.003 (1) Å with a maximum of 0.005 (1) Å for the methoxyphenyl ring. The dihedral angle between the two systems is 4.7 (3)°. The methoxyphenyl ring is pushed away from the vinyl proton and towards the endocyclic O atom; the bond angles are 130.13 (9) and 110.13 (8)°, respectively.

**Experimental.** Colorless crystals of (1), m.p. 398–399 K, were isolated by two successive recrystallizations in methanol from the crude product obtained from the coupling of 2-iodoanisole and copper(I) methyl (2-benzoate)ethynide in pyridine at reflux temperature (Stephens & Castro, 1963). Crystal size



0.10 × 0.25 × 0.68 mm, space group from systematic absences  $0kl$  with  $k+l$  odd,  $h0l$  with  $h$  odd and successful refinement of a noncentrosymmetric model, cell dimensions from setting angles of 25 reflections having  $20 < \theta < 25^\circ$ . Data collection on Enraf–Nonius CAD-4 diffractometer, Cu  $K\alpha$  radiation, graphite monochromator,  $\omega$ - $2\theta$  scans designed for  $I = 50\sigma(I)$ , subject to max. scan time = 90 s, scan rates varied 0.61–3.30° min<sup>-1</sup>. One quadrant of data having  $2 < \theta < 75^\circ$ ,  $0 \leq h \leq 8$ ,  $0 \leq k \leq 16$ ,  $-17 \leq l \leq 17$  measured. Data corrected for background, Lorentz, polarization and absorption effects. Absorption correc-

tions were based on  $\psi$  scans, with a minimum relative transmission coefficient of 89.86%. Standard reflections 200 and 020 displayed no decline in intensity during the experiment. Equivalent data merged,  $R_{\text{int}} = 0.016$ , to yield 2508 unique data, 2337 observed with  $I > 3\sigma(I)$ . Structure solved by direct methods, using a data set previously obtained from a lower-quality crystal with Mo  $K\alpha$  radiation, employing MULTAN82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The structure was refined using the Cu  $K\alpha$  data by full-matrix least squares based upon  $F$  with weights  $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$  using Enraf–Nonius SDP (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located by  $\Delta F$  and refined isotropically. One reflection (171) was given zero weight on the basis of extremely poor agreement with  $F_c$  and with  $F_o$  for the 171 reflection. An attempt to determine the direction of the polar axis by refinement of the structure inverted through the origin was inconclusive. Final  $R = 0.027$  (0.031 for all data),  $wR = 0.035$ ,  $S = 1.918$  for 220 variables. Maximum shift 0.03 $\sigma$  in the final cycle, max. residual density 0.24 e Å<sup>-3</sup>, min. -0.17 e Å<sup>-3</sup>, extinction coefficient  $g = 5.2(2) \times 10^{-6}$  where the correction factor  $(1 + gI_c)^{-1}$  was applied to  $F_c$ . Coordinates are given in Table 1; bond distances, angles and torsion angles are given in Table 2.† The molecule is illustrated in Fig. 1, and a packing diagram is shown in Fig. 2.

**Related literature.** Structure of the dimethyl derivative of lateropyrone: Bushnell, Li & Poulton (1984). The title compound shows the same distorted bond angles

† Tables of H-atom coordinates, distances and angles involving H atoms, anisotropic thermal parameters, structure-factor amplitudes and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51686 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

\* To whom correspondence should be addressed.