ing the C—C—C angle; methyl and ethyl H atoms were placed in tetrahedral positions at 1.10 Å. Anisotropic displacement parameters were refined for all non-H atoms. H atoms were assigned the B_{eq} value of the C atom to which they were bonded. Programs used: *MULTAN76*; *ALLS* (Lapp & Jacobson, 1979); *FOUR* (Powell & Jacobson, 1980); *ORFFE* (Busing, Martin & Levy, 1964); *ORTEP*II (Johnson, 1971).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71606 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1051]

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A 2,5-Diketomorpholine

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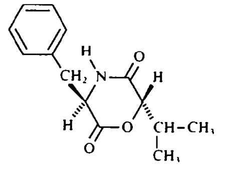
Abstract

We report the crystal structure of (3*R*,6*S*)-3-benzyl-6-isopropylmorpholine-2,5-dione, $C_{14}H_{17}NO_3$. The morpholine heterocycle adopts a boat conformation with the isopropyl group in an equatorial position and the benzyl substituent in an axial position. An intermolecular hydrogen bond connects symmetry-related amide groups to form an infinite chain.

Comment

The title compound (I) is a synthetic intermediate in the diastereoselective synthesis of amino acids (Maywald, 1987). The boat conformation of the morpholine heterocycle is clearly defined: O(1), C(2), N(4) and C(5) form the base [σ = 0.04 (1) Å] while C(3) and C(6) deviate from the base by 0.39 (1) and 0.46 (1) Å, respectively. The phenyl ring of the axial benzyl substituent is oriented towards the N atom.



(I)

The intermolecular packing is stabilized by a hydrogen bond from N(4) to O(51) [N(4)···O(51) 2.864 (2), H(4)···O(51) 1.93 (1) Å, N(4)—H(4)···O(51) 165 (2)°] with the two molecules related by a 2_1 axis ($1 - x$, $-\frac{1}{2} + y$, $1 - z$) thus forming an infinite chain parallel to **b**.

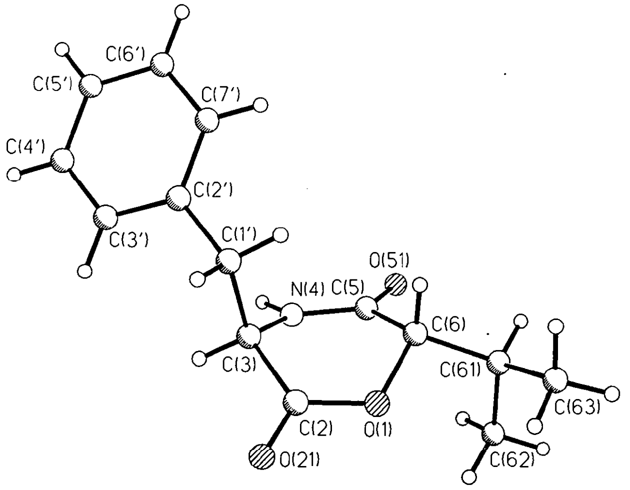


Fig. 1. A perspective view of the title compound showing the atom-numbering scheme.

Experimental

Crystal data

$C_{14}H_{17}NO_3$	Mo $K\alpha$ radiation
M_r = 247.3	λ = 0.71069 Å

Monoclinic
$P2_1$
$a = 9.873$ (1) Å
$b = 6.129$ (1) Å
$c = 11.594$ (1) Å
$\beta = 108.23$ (1)°
$V = 666.4$ (1) Å^3
$Z = 2$
$D_x = 1.232$ Mg m^{-3}

Cell parameters from 40 reflections
$\theta = 10$–12.5°
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Block
0.8 × 0.8 × 0.3 mm
Colourless, transparent
Crystal source: cyclohexane/ethyl acetate

Data collection

Stoe four-circle diffractometer
$\omega/2\theta$ scans with profile fitting (Clegg, 1981)
Absorption correction: none
1434 measured reflections
1364 independent reflections
1328 observed reflections [$F > 3\sigma(F)$]

$R_{\rm int} = 0.03$
$\theta_{\rm max} = 25°$
$h = -11 \rightarrow 11$
$k = -7 \rightarrow 1$
$l = 0 \rightarrow 13$
3 standard reflections monitored every 100 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.032$
$wR = 0.045$
$S = 1.79$
1328 reflections
172 parameters
Position of H(N) refined, positions of H(C) fixed
$w = 1/[\sigma^2(F) + 0.0005F^2]$
$(\Delta/\sigma)_{\rm max} = 0.03$

$\Delta\rho_{\rm max} = 0.12$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.14$ e Å^{-3}
Extinction correction: $F^* = F[1 + (0.002\chi \times F^2/\sin 2\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.06$ (2)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O(1)	0.7943 (1)	0.5299†	0.3704 (1)	0.046 (1)
C(2)	0.8848 (2)	0.4104 (4)	0.4567 (2)	0.040 (1)
C(3)	0.8346 (2)	0.3288 (4)	0.5593 (2)	0.041 (1)
N(4)	0.6795 (1)	0.3046 (3)	0.5190 (1)	0.041 (1)
C(5)	0.5920 (2)	0.4421 (4)	0.4433 (1)	0.040 (1)
C(6)	0.6641 (2)	0.6148 (4)	0.3879 (2)	0.042 (1)
O(21)	1.0006 (1)	0.3696 (3)	0.4496 (1)	0.052 (1)
O(51)	0.4612 (1)	0.4351 (4)	0.4190 (1)	0.051 (1)
C(61)	0.5720 (2)	0.6963 (5)	0.2644 (2)	0.055 (1)
C(62)	0.5353 (2)	0.5156 (6)	0.1702 (2)	0.070 (1)
C(63)	0.6456 (3)	0.8885 (6)	0.2243 (3)	0.082 (1)
C(1′)	0.8902 (2)	0.4804 (5)	0.6695 (2)	0.050 (1)
C(2′)	0.8540 (2)	0.4041 (5)	0.7804 (2)	0.046 (1)
C(3′)	0.8989 (2)	0.2054 (5)	0.8338 (2)	0.056 (1)
C(4′)	0.8727 (2)	0.1453 (5)	0.9402 (2)	0.068 (1)
C(5′)	0.8000 (3)	0.2826 (6)	0.9929 (2)	0.071 (1)
C(6′)	0.7529 (2)	0.4813 (6)	0.9397 (2)	0.069 (1)
C(7′)	0.7787 (2)	0.5400 (5)	0.8333 (2)	0.056 (1)

† Coordinate fixed to define origin.

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(2)	1.333 (2)	O(1)—C(6)	1.459 (2)
C(2)—C(3)	1.510 (2)	C(2)—O(21)	1.197 (2)
C(3)—N(4)	1.461 (2)	C(3)—C(1′)	1.535 (3)
N(4)—C(5)	1.323 (2)	C(5)—C(6)	1.526 (2)
C(5)—O(51)	1.233 (2)	C(6)—C(61)	1.520 (3)
C(61)—C(62)	1.518 (4)	C(61)—C(63)	1.531 (4)
C(1′)—C(2′)	1.512 (2)	C(2′)—C(3′)	1.376 (3)
C(2′)—C(7′)	1.381 (3)	C(3′)—C(4′)	1.387 (3)
C(4′)—C(5′)	1.368 (4)	C(5′)—C(6′)	1.379 (5)
C(6′)—C(7′)	1.384 (3)		
C(6)—O(1)—C(2)	120.3 (1)	C(3)—C(2)—O(1)	117.7 (1)
O(21)—C(2)—O(1)	119.5 (1)	O(21)—C(2)—C(3)	122.8 (2)
N(4)—C(3)—C(2)	110.5 (1)	C(1′)—C(3)—C(2)	109.6 (1)
C(1′)—C(3)—N(4)	113.2 (1)	C(5)—N(4)—C(3)	123.3 (1)
C(6)—C(5)—N(4)	115.3 (1)	O(51)—C(5)—N(4)	123.3 (2)
O(51)—C(5)—C(6)	121.4 (2)	C(5)—C(6)—O(1)	111.0 (1)
C(61)—C(6)—O(1)	106.2 (1)	C(61)—C(6)—C(5)	113.8 (1)
C(62)—C(61)—C(6)	112.1 (2)	C(63)—C(61)—C(6)	109.7 (2)
C(63)—C(61)—C(62)	111.8 (2)	C(2′)—C(1′)—C(3)	113.7 (2)
C(3′)—C(2′)—C(1′)	121.9 (2)	C(7′)—C(2′)—C(1′)	119.6 (2)
C(7′)—C(2′)—C(3′)	118.4 (2)	C(4′)—C(3′)—C(2′)	120.8 (2)
C(5′)—C(4′)—C(3′)	120.2 (2)	C(6′)—C(5′)—C(4′)	119.6 (2)
C(7′)—C(6′)—C(5′)	119.9 (2)	C(6′)—C(7′)—C(2′)	121.0 (2)

The structure was solved by direct methods. All H atoms were located using a difference Fourier synthesis and were refined with fixed individual displacement parameters [U(H) = $1.2 \times U_{\rm eq}$(C, N)] using a riding model with C—H = 0.96 Å. The coordinates of the amide H atom were refined with the N—H distance restrained to 0.96 Å. Anisotropic full-matrix refinement was carried out using *XLS* (Sheldrick, 1987).

We thank Professor U. Schöllkopf (University of Göttingen) for providing the sample.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: SH1078). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Four *N*-Saccharinaliphatic Acids

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Abstract

The crystal structures of *N*-saccharinacetic acid (3-oxo-2*H*-1,2-benzisothiazol-2-acetic acid 1,1-dioxide),

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