N1—C5—C6	108.4 (4)	C11—C12—C13	119.4 (6)
C4—C5—C6	121.6 (4)	C14—C13—C12	119.4 (6)
C5—C6—C7	119.2 (4)	C15—C14—C13	120.5 (6)
C5—C6—C8	106.4 (4)	C14—C15—C16	120.4 (6)
C7—C6—C8	134.2 (4)	C11—C16—C15	119.4 (6)
C2—C7—C6	119.8 (4)		
C2—S1—C1—C17	−65.2 (5)	C10—N2—C11—C12	91.4 (7)
C1—S1—C2—C3	147.6 (4)	C10—N2—C11—C16	−89.9 (7)
C1—S1—C2—C7	−36.6 (5)	N1—C9—C10—O2	2.9 (6)
C11—N2—C10—C9	1.8 (8)	C8—C9—C10—N2	−3.2 (9)

The integrated intensities were obtained by a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) peak-profile analysis procedure. All the H atoms were placed in calculated positions, riding on their attached C atoms, with atomic displacement coefficients 1.5 times the $U_{\rm eq}$ of the attached atom. The amide N atom shows a rather high anisotropy ratio, indicating disorder which propagates to the attached *n*-hexyl chain, as frequently happens for long hydrocarbon chains. A satisfactory model was obtained assuming this chain was disordered over two overlapping positions having site-occupation factors of 0.515 (12) (C18–C23) and 0.485 (12) (C24–C29). This disorder caused the refinement to be rather cumbersome, but did not significantly influence the results concerning the geometry of the rest of the molecule (see Table 2).

Data collection: local programs. Cell refinement: *LQPARM* (Nardelli & Mangia, 1984). Data reduction: local programs. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *PARST* (Nardelli, 1983) and *PARSTCIF* (Nardelli, 1991).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1000). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(*S*)-3-(1,1-Dimethyloxycarbonylamino)-1-phenylmethyloxypiperidine-2,6-dione,† $C_{17}H_{22}N_2O_5$

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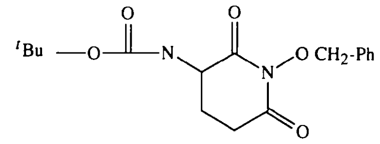
Abstract

The two molecules in the asymmetric unit differ in the orientation of the glutarimide ring. The glutarimide moiety adopts an envelope conformation in molecule *A* and a twisted conformation in molecule *B*. The other features are similar.

Comment

We have studied the preparation of biologically active compounds containing hydroxamate groups. The attempted condensation of the α-phenyl ester of BOC-glutamic acid with benzyloxyamine failed to provide the expected acyclic product but led to the cyclic derivative whose structure we now present.



(I)

The asymmetric unit consists of two independent molecules, *A* and *B*, whose structures differ essentially in the conformation of the glutarimide moiety. The glutarimide ring assumes an envelope conformation in molecule *A*. Atom C4 deviates by 0.70 (1) Å from the least-squares plane *P1* defined by N1, C2, C3, C5, C6, which has a maximum deviation of −0.013 (9) Å for C2 and C6. The O2, C2, N1, C6, O6 atoms define the least-squares plane *P2*; O1 deviates by +0.16 (2) Å from this plane. Such an envelope-like conformation has been observed in the crystal structures of *N*-(α-glutarimido)-4-bromophthalimide and of glutarimide (Petersen, 1969, 1971). C17 lies in the carbamate plane

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† Alternative name: (*S*)-*tert*-butyl 2,6-dioxo-1-benzyloxypiperidine-3-carbamate.

$P3$ (N14, C15, O15, O16) and C3 is $-0.28\,(2)$ Å from this plane. The glutarimide ring shows a twisted half-chair conformation in molecule B with C3′ and C4′ displaced by $-0.35\,(1)$ and $+0.35\,(1)$ Å, respectively, from the plane $P'1$ formed by N1′, C2′, C5′, C6′, which has a maximum deviation of 0.11 (8) Å for C6′. C3′ and C17′ lie in the carbamate $P'3$ plane (O15′, N14′, C15′, O16′), deviating by less than 0.04 (2) Å. Molecules A and B are linked together by weak hydrogen bonding [N14···O15′ 2.96 (1), H14···O15′ 2.21 (7) Å, N14—H14···O15′ 131 (7)°] and by van der Waals interactions.

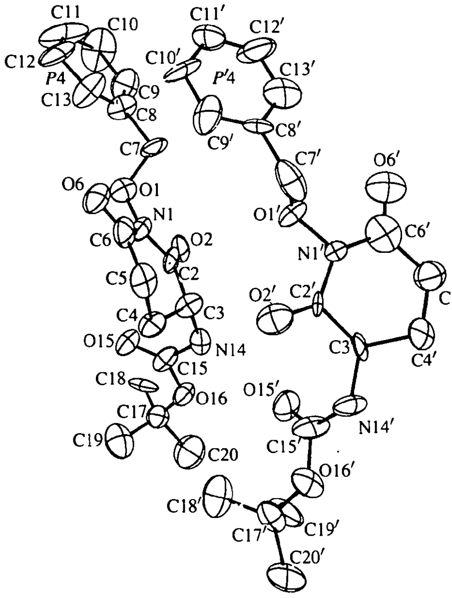

Fig. 1. A perspective view of the two independent molecules showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

Crystals of A and B were grown by recrystallization from methanol. The density D_m was measured by flotation in CH_2Cl_2/C_6H_{12}.

Crystal data

$C_{17}H_{22}N_2O_5$
$M_r = 334.37$
Monoclinic
$P2_1$
$a = 10.260\,(7)$ Å
$b = 10.819\,(5)$ Å
$c = 16.266\,(9)$ Å
$\beta = 106.07\,(7)°$
$V = 1735\,(1)$ Å^3
$Z = 4$
$D_x = 1.28$ Mg m^{-3}
$D_m = 1.28\,(2)$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 21 reflections
$\theta = 4.55$–15.7°
$\mu = 0.089$ mm^{-1}
Room temperature
Parallelepiped
0.425 × 0.425 × 0.075 mm
Brown

Data collection

Syntex $P2_1$ diffractometer
ω–2θ scans
Absorption correction: none
3107 measured reflections
3107 independent reflections
1384 observed reflections $[F > 3\sigma(F)]$

$\theta_{max} = 25°$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 20$
3 standard reflections monitored every 50 reflections
intensity decay: <0.02%

Refinement

Refinement on F
$R = 0.058$
$wR = 0.06$
$S = 1.01$
1384 reflections
286 parameters
H atoms refined isotropically
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = 0.07$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: none
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)*

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

	x	y	z	U_{eq}
O1	0.2064 (6)	0.9894 (6)	0.9469 (4)	0.0490 (7)
N1	0.3294 (8)	1.0299 (8)	1.0013 (5)	0.0460 (10)
O2	0.3485 (6)	0.8459 (7)	1.0674 (4)	0.0520 (8)
C2	0.3959 (8)	0.9468 (11)	1.0664 (6)	0.0430 (11)
C3	0.5243 (9)	0.9980 (9)	1.1249 (6)	0.0430 (11)
C4	0.5219 (11)	1.1387 (9)	1.1321 (6)	0.0490 (12)
C5	0.4979 (10)	1.1941 (9)	1.0431 (8)	0.0550 (13)
O6	0.3026 (8)	1.2088 (6)	0.9216 (5)	0.0610 (9)
C6	0.3704 (10)	1.1507 (11)	0.9824 (7)	0.0560 (14)
C7	0.2259 (10)	0.9271 (11)	0.8710 (6)	0.0510 (12)
C8	0.0973 (9)	0.9457 (11)	0.8034 (6)	0.0490 (12)
C9	0.0093 (13)	0.8489 (12)	0.7739 (9)	0.0800 (18)
C10	−0.1147 (13)	0.8551 (16)	0.7115 (12)	0.106 (2)
C11	−0.1479 (14)	0.976 (2)	0.6796 (8)	0.111 (2)
C12	−0.0592 (14)	1.0786 (14)	0.7052 (7)	0.0790 (17)
C13	0.0632 (11)	1.0559 (12)	0.7658 (8)	0.0710 (16)
N14	0.5538 (8)	0.9326 (8)	1.2064 (5)	0.0510 (10)
O15	0.3557 (7)	0.9871 (7)	1.2376 (4)	0.0620 (8)
C15	0.4692 (10)	0.9472 (9)	1.2583 (7)	0.0450 (12)
O16	0.5336 (6)	0.8983 (7)	1.3341 (4)	0.0500 (7)
C17	0.4713 (11)	0.8996 (11)	1.4025 (7)	0.0570 (12)
C18	0.3384 (15)	0.8185 (16)	1.3816 (8)	0.099 (2)
C19	0.4443 (16)	1.0217 (13)	1.4307 (9)	0.090 (2)
C20	0.5782 (17)	0.8383 (16)	1.4772 (10)	0.105 (2)
O1′	0.7007 (6)	0.9405 (7)	0.9371 (4)	0.0590 (8)
N1′	0.8253 (7)	0.9055 (9)	0.9901 (5)	0.0420 (9)
O2′	0.8085 (7)	1.0648 (7)	1.0817 (4)	0.0630 (9)
C2′	0.8556 (8)	0.9682 (10)	1.0671 (6)	0.0420 (12)
C3′	0.9680 (8)	0.8964 (9)	1.1378 (6)	0.0370 (10)
C4′	1.0750 (10)	0.8409 (11)	1.1047 (7)	0.0560 (13)
O15′	0.8529 (7)	0.9227 (7)	1.2717 (5)	0.0680 (9)
C5′	1.0137 (12)	0.7560 (12)	1.0302 (8)	0.0630 (15)
O6′	0.8245 (9)	0.7467 (8)	0.9030 (6)	0.0800 (11)
C6′	0.8793 (13)	0.7938 (11)	0.9698 (9)	0.0750 (18)
C7′	0.7098 (12)	1.0232 (10)	0.8683 (9)	0.0720 (17)
C8′	0.5768 (10)	1.0122 (10)	0.7956 (6)	0.0470 (12)
C9′	0.4925 (12)	1.1103 (10)	0.7753 (8)	0.0700 (17)
C10′	0.3728 (13)	1.0900 (14)	0.7136 (8)	0.0860 (19)
C11′	0.3458 (12)	0.9799 (13)	0.6668 (7)	0.0690 (16)
C12′	0.4336 (16)	0.8898 (13)	0.6887 (8)	0.0800 (18)
C13′	0.5472 (13)	0.8990 (12)	0.7531 (8)	0.0720 (16)
N14′	1.0238 (8)	0.9717 (8)	1.2141 (5)	0.0500 (9)
C15′	0.9564 (12)	0.9773 (10)	1.2723 (6)	0.0520 (13)
O16′	1.0272 (6)	1.0524 (6)	1.3380 (4)	0.0520 (8)

C17′	0.9818 (11)	1.0737 (9)	1.4142 (7)	0.0530 (12)
C18′	0.8519 (13)	1.1342 (14)	1.3929 (9)	0.0840 (17)
C19′	0.9665 (14)	0.9470 (12)	1.4586 (7)	0.0780 (17)
C20′	1.0954 (12)	1.1406 (12)	1.4713 (6)	0.0650 (15)

Table 2. *Selected bond lengths (Å) and angles (°)*

O1—N1	1.397 (11)	O1′—N1′	1.382 (10)
O1—C7	1.468 (12)	O1′—C7′	1.456 (14)
N1—C2	1.413 (13)	N1′—C2′	1.381 (13)
N1—C6	1.432 (14)	N1′—C6′	1.407 (16)
O2—C2	1.196 (13)	O2′—C2′	1.203 (13)
C2—C3	1.501 (14)	C2′—C3′	1.589 (13)
C3—C4	1.528 (14)	C3′—C4′	1.478 (14)
C3—N14	1.458 (13)	C3′—N14′	1.462 (12)
C4—C5	1.523 (15)	C4′—C5′	1.511 (17)
C5—C6	1.481 (16)	C5′—C6′	1.511 (19)
O6—C6	1.214 (14)	O6′—C6′	1.190 (17)
C7—C8	1.479 (14)	C7′—C8′	1.543 (16)
C8—C9	1.379 (17)	C8′—C9′	1.351 (16)
C8—C13	1.341 (17)	C8′—C13′	1.398 (17)
C9—C10	1.39 (2)	C9′—C10′	1.372 (18)
C10—C11	1.41 (3)	C10′—C11′	1.40 (2)
C11—C12	1.42 (2)	C11′—C12′	1.31 (2)
C12—C13	1.387 (18)	C12′—C13′	1.338 (19)
N14—C15	1.378 (14)	N14′—C15′	1.320 (13)
O15—C15	1.200 (12)	O15′—C15′	1.213 (14)
C15—O16	1.335 (12)	C15′—O16′	1.378 (12)
O16—C17	1.429 (13)	O16′—C17′	1.459 (12)
C17—C18	1.58 (2)	C17′—C18′	1.439 (18)
C17—C19	1.451 (18)	C17′—C19′	1.577 (16)
C17—C20	1.54 (2)	C17′—C20′	1.464 (16)
N1—O1—C7	111.5 (7)	N1′—O1′—C7′	113.7 (7)
O1—N1—C2	116.1 (8)	O1′—N1′—C2′	111.6 (8)
O1—N1—C6	114.7 (8)	O1′—N1′—C6′	116.4 (8)
C2—N1—C6	129.2 (9)	C2′—N1′—C6′	129.1 (9)
N1—C2—O2	118.5 (9)	N1′—C2′—O2′	127.2 (9)
N1—C2—C3	113.4 (8)	N1′—C2′—C3′	110.7 (8)
O2—C2—C3	128.1 (9)	O2′—C2′—C3′	122.1 (9)
C2—C3—C4	112.8 (8)	C2′—C3′—C4′	113.4 (8)
C2—C3—N14	108.8 (8)	C2′—C3′—N14′	112.2 (7)
C4—C3—N14	114.7 (8)	C4′—C3′—N14′	112.3 (8)
C3—C4—C5	108.8 (8)	C3′—C4′—C5′	110.7 (9)
C4—C5—C6	112.9 (9)	C4′—C5′—C6′	117.3 (10)
N1—C6—C5	113.8 (9)	N1′—C6′—C5′	115.3 (11)
N1—C6—O6	120.7 (10)	N1′—C6′—O6′	117.7 (12)
C5—C6—O6	125.4 (10)	C5′—C6′—O6′	126.4 (12)
O1—C7—C8	105.1 (8)	O1′—C7′—C8′	108.1 (9)
C7—C8—C9	121.2 (10)	C7′—C8′—C9′	119.8 (10)
C7—C8—C13	121.8 (10)	C7′—C8′—C13′	118.1 (10)
C9—C8—C13	116.9 (11)	C9′—C8′—C13′	122.1 (11)
C8—C9—C10	126.5 (13)	C8′—C9′—C10′	115.8 (11)
C9—C10—C11	113.0 (15)	C9′—C10′—C11′	122.4 (12)
C10—C11—C12	123.1 (15)	C10′—C11′—C12′	117.9 (13)
C11—C12—C13	116.7 (13)	C11′—C12′—C13′	123.0 (14)
C8—C13—C12	123.5 (12)	C8′—C13′—C12′	118.3 (12)
C3—N14—C15	119.6 (8)	C3′—N14′—C15′	118.3 (8)
N14—C15—O15	126.6 (10)	O15′—C15′—N14′	127.1 (10)
N14—C15—O16	106.6 (8)	O15′—C15′—O16′	124.5 (10)
O15—C15—O16	126.6 (9)	N14′—C15′—O16′	108.4 (9)
C15—O16—C17	120.1 (8)	C15′—O16′—C17′	121.9 (8)
O16—C17—C18	111.9 (9)	O16′—C17′—C18′	111.0 (9)
O16—C17—C19	114.9 (10)	O16′—C17′—C19′	110.3 (9)
O16—C17—C20	103.6 (10)	O16′—C17′—C20′	103.5 (8)
C18—C17—C19	110.2 (11)	C18′—C17′—C19′	107.6 (10)
C18—C17—C20	108.3 (11)	C18′—C17′—C20′	116.7 (10)
C19—C17—C20	107.4 (11)	C19′—C17′—C20′	107.6 (9)

Table 3. *Angles (°) between planes in A and B*

*P*1—*P*2	3.0 (8)	*P*′1—*P*′3	112.6 (5)
P′1—*P*′2	171.8 (9)	*P*2—*P*3	105.3 (7)
*P*1—*P*3	107.8 (4)	*P*4—*P*′4	149.5 (5)

The structure was determined by direct methods using the program *SHELXS*86 (Sheldrick, 1985). Full-matrix least-squares refinement on *F* magnitudes with anisotropic displacement parameters for non-H atoms and isotropic parameters for H atoms was performed with *SHELX*76 (Sheldrick, 1976). On account of the strong correlations existing between molecules *A* and *B*, we have refined the positions and displacement factors of the molecules in different cycles. H atoms were located from difference Fourier maps and refined as riding groups. Illustrations were generated with the program *MACORTEP* (Michalowicz & André, 1991). Dihedral angles were calculated with *Best Planes Program* (Ito & Sugawara, 1983). H-atom positions were calculated.

Lists of structure factors, least-squares-planes data, torsion angles, anisotropic displacement parameters, van der Waals contacts, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1119). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Benzyl-3,4-diphenyl-2-azetidinone

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Abstract

The crystal structure of *N*-benzyl-3,4-diphenyl-2-azetidinone, $C_{22}H_{19}NO$, shows that the relative configuration of the two chiral centres, C2 and C3, is *trans*. The N—C and C═O bond lengths in the β-lactam amide group are 1.33 (1) and 1.26 (1) Å, respectively. The N atom is displaced 0.032 (9) Å from the plane of its three attached atoms, *i.e.* C1 and C3 of the β-lactam ring, and C16 of the benzyl group.