

|      |             |             |            |             |
|------|-------------|-------------|------------|-------------|
| 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

|         |           |         |           |
|---------|-----------|---------|-----------|
| P1—P2   | 3.0 (8)   | P'1—P'3 | 112.6 (5) |
| P'1—P'2 | 171.8 (9) | P2—P3   | 105.3 (7) |
| P1—P3   | 107.8 (4) | P4—P'4  | 149.5 (5) |

The structure was determined by direct methods using the program *SHELXS86* (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 *SHELX76* (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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*Acta Cryst.* (1996). **C52**, 201–203

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

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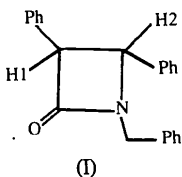
(Received 7 December 1994; accepted 30 March 1995)

## Abstract

The crystal structure of *N*-benzyl-3,4-diphenyl-2-azetidinone, C<sub>22</sub>H<sub>19</sub>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.

### Comment

The  $\beta$ -lactam ring plays a key role in biological activity and the substituents attached to the ring allow selective activity (Takasuka, Nishikawa & Tori, 1982; Boyd, 1983; Cohen, 1983). Since a complete knowledge of the stereochemistry of the  $\beta$ -lactam ring is required to determine antibiotic activity, the title compound (I) [which was synthesized by the reaction of an imine, an acid chloride and triethylamine in toluene under similar conditions to those used by Alcaide *et al.* (1982)] was structurally characterized.



The aim of the present X-ray structure analysis was to determine the relative configuration of the two chiral centres at C1 and C2. The crystal structure shows that atoms H1(C2) and H2(C3) are in a *trans* conformation, at distances of  $-0.640$  and  $0.656$  Å, respectively, from the plane of the  $\beta$ -lactam ring composed of atoms N1, C1, C2 and C3. The absolute configuration at C2 and C3 is *S*. The N1 atom is  $0.032$  (9) Å above the plane containing atoms C1 and C3 of the  $\beta$ -lactam ring and atom C16 of the benzyl group. The sum of the bond angles about N1 is exactly  $360^\circ$  [C1—N1—C3  $98$  (1), C1—N1—C16  $132$  (1) and C3—N1—C16  $130$  (1)°].

The bond length N1—C1 of  $1.33$  (1) Å is shorter than bond lengths N1—C3 and N1—C16 of  $1.41$  (1) and  $1.38$  (1) Å, respectively, which is in agreement with those observed in previous studies (Pain, Biswas, Bose

& Banerjee, 1992; Vinković & Herak, 1993) This is due to a delocalization of the lone pair on N1 over atoms N1 and C1, which are conjugated with the C=O bond. This is confirmed by the C=O bond length of  $1.26$  (1) Å, which is slightly longer than those found in the literature. The benzyl fragment is nearly planar and is orthogonal to the  $\beta$ -lactam ring, the dihedral angle being  $86.01^\circ$ .

### Experimental

The title compound was synthesized by the reaction of PhCH=N—CH<sub>2</sub>Ph, PhCH<sub>2</sub>C(O)Cl and Et<sub>3</sub>N (molar ratio 1:1:2) in toluene for 20 h at 383 K under a nitrogen atmosphere. Recrystallization was from a CH<sub>2</sub>Cl<sub>2</sub>/*p*-xylene solution.

#### Crystal data

C<sub>22</sub>H<sub>19</sub>NO  
 $M_r = 313.40$   
 Monoclinic  
 $P2_1$   
 $a = 6.798$  (4) Å  
 $b = 13.675$  (8) Å  
 $c = 9.722$  (7) Å  
 $\beta = 99.72$  (4)°  
 $V = 890.9$  (1) Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 1.168$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation  
 $\lambda = 1.54184$  Å  
 Cell parameters from 25 reflections  
 $\theta = 8-12^\circ$   
 $\mu = 0.5214$  mm<sup>-1</sup>  
 $T = 293$  K  
 Prism  
 $0.6 \times 0.4 \times 0.2$  mm  
 White

#### Data collection

Enraf-Nonius CAD-4 diffractometer  
 $\omega-2\theta$  scans  
 Absorption correction: none  
 1156 measured reflections  
 1077 independent reflections  
 795 observed reflections  
 $[I > 1.0\sigma(I)]$

$R_{int} = 0.045$   
 $\theta_{max} = 53.0^\circ$   
 $h = -7 \rightarrow 7$   
 $k = 0 \rightarrow 14$   
 $l = 0 \rightarrow 10$   
 3 standard reflections  
 frequency: 60 min  
 intensity decay: 3%

#### Refinement

Refinement on  $F$   
 $R = 0.068$   
 $wR = 0.078$   
 $S = 1.448$   
 795 reflections  
 217 parameters  
 H-atom parameters not refined  
 $w = 4/[\sigma^2(I) + (PI)^2/I]$   
 $(\Delta/\sigma)_{max} = 0.01$

$\Delta\rho_{max} = 0.177$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = 0.142$  e Å<sup>-3</sup>  
 Extinction correction:  
 $F_c = |F_c|^2/(1 + gF_cLp)$   
 Extinction coefficient:  
 $g = 2.7142 \times 10^{-6}$   
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

|    | $B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$ |            |             |          |
|----|--|------------|-------------|----------|
|    | <i>x</i>   | <i>y</i>   | <i>z</i>    | $B_{eq}$ |
| O1 | -0.3762 (9)  | 0.421      | -0.0596 (6) | 8.1 (2)  |
| N1 | -0.086 (1)   | 0.4004 (7) | -0.1572 (8) | 8.1 (2)  |
| C1 | -0.278 (1)   | 0.3907 (7) | -0.1502 (9) | 7.4 (2)  |

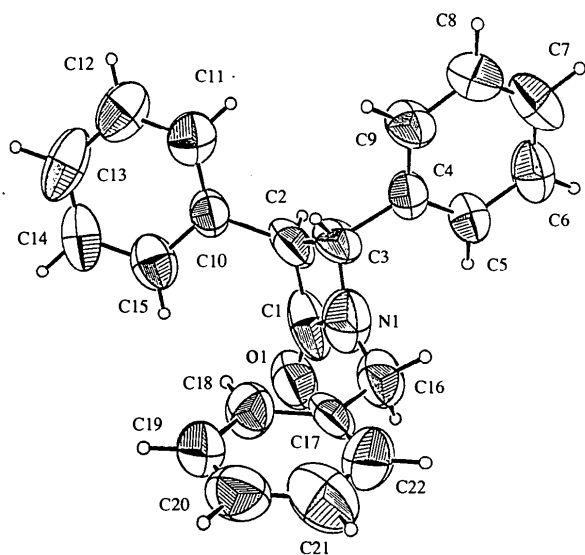


Fig. 1. The molecular structure of the title compound showing 50% probability displacement ellipsoids.

|     |            |            |             |         |
|-----|------------|------------|-------------|---------|
| C2  | -0.328 (1) | 0.3392 (7) | -0.2883 (9) | 7.2 (2) |
| C3  | -0.090 (1) | 0.3561 (7) | -0.2888 (8) | 5.6 (2) |
| C4  | 0.036 (1)  | 0.2648 (6) | -0.2883 (8) | 5.2 (2) |
| C5  | 0.072 (1)  | 0.2035 (6) | -0.1733 (8) | 5.4 (2) |
| C6  | 0.183 (1)  | 0.1180 (7) | -0.182 (1)  | 7.1 (3) |
| C7  | 0.248 (1)  | 0.0938 (7) | -0.301 (1)  | 7.3 (3) |
| C8  | 0.211 (2)  | 0.1551 (8) | -0.413 (1)  | 7.8 (3) |
| C9  | 0.107 (1)  | 0.2410 (8) | -0.4085 (8) | 5.9 (2) |
| C10 | -0.478 (1) | 0.3843 (7) | -0.4160 (8) | 5.7 (2) |
| C11 | -0.484 (1) | 0.3511 (7) | -0.5536 (9) | 6.3 (2) |
| C12 | -0.596 (1) | 0.3934 (9) | -0.6645 (9) | 7.5 (3) |
| C13 | -0.717 (1) | 0.4735 (9) | -0.639 (1)  | 8.4 (3) |
| C14 | -0.707 (1) | 0.5110 (8) | -0.509 (1)  | 7.4 (3) |
| C15 | -0.587 (1) | 0.4663 (7) | -0.3946 (9) | 6.8 (3) |
| C16 | 0.068 (1)  | 0.4457 (8) | -0.0700 (9) | 7.0 (3) |
| C17 | 0.102 (1)  | 0.5496 (7) | -0.1035 (8) | 6.0 (2) |
| C18 | -0.042 (1) | 0.6063 (7) | -0.1834 (9) | 6.3 (2) |
| C19 | -0.003 (2) | 0.7049 (8) | -0.2100 (9) | 7.8 (3) |
| C20 | 0.187 (2)  | 0.7431 (8) | -0.157 (1)  | 8.1 (3) |
| C21 | 0.331 (2)  | 0.6878 (9) | -0.081 (1)  | 9.7 (4) |
| C22 | 0.289 (1)  | 0.5926 (9) | -0.054 (1)  | 8.6 (3) |

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

|           |           |             |          |
|-----------|-----------|-------------|----------|
| O1—C1     | 1.26 (1)  | C10—C11     | 1.41 (1) |
| N1—C1     | 1.33 (1)  | C10—C15     | 1.38 (1) |
| N1—C3     | 1.41 (1)  | C11—C12     | 1.34 (2) |
| N1—C16    | 1.38 (1)  | C12—C13     | 1.42 (2) |
| C1—C2     | 1.50 (2)  | C13—C14     | 1.36 (2) |
| C2—C3     | 1.64 (2)  | C14—C15     | 1.40 (2) |
| C2—C10    | 1.59 (1)  | C16—C17     | 1.48 (2) |
| C3—C4     | 1.51 (1)  | C17—C18     | 1.38 (2) |
| C4—C5     | 1.39 (1)  | C17—C22     | 1.41 (2) |
| C4—C9     | 1.38 (1)  | C18—C19     | 1.41 (2) |
| C5—C6     | 1.40 (1)  | C19—C20     | 1.41 (2) |
| C6—C7     | 1.35 (2)  | C20—C21     | 1.35 (2) |
| C7—C8     | 1.36 (2)  | C21—C22     | 1.37 (2) |
| C8—C9     | 1.38 (2)  |             |          |
| C1—N1—C3  | 98 (1)    | C4—C9—C8    | 119 (1)  |
| C1—N1—C16 | 132 (1)   | C2—C10—C11  | 122 (1)  |
| C3—N1—C16 | 130 (1)   | C2—C10—C15  | 119 (1)  |
| O1—C1—N1  | 130 (1)   | C11—C10—C15 | 118 (1)  |
| O1—C1—C2  | 136 (1)   | C10—C11—C12 | 123 (1)  |
| N1—C1—C2  | 94 (1)    | C11—C12—C13 | 117 (1)  |
| C1—C2—C3  | 82.3 (9)  | C12—C13—C14 | 121 (1)  |
| C1—C2—C10 | 122.1 (9) | C13—C14—C15 | 120 (1)  |
| C3—C2—C10 | 116.6 (8) | C10—C15—C14 | 119 (1)  |
| N1—C3—C2  | 85.7 (8)  | N1—C16—C17  | 115 (1)  |
| N1—C3—C4  | 115 (1)   | C16—C17—C18 | 122 (1)  |
| C2—C3—C4  | 116.2 (9) | C16—C17—C22 | 119 (1)  |
| C3—C4—C5  | 121.7 (9) | C18—C17—C22 | 118 (1)  |
| C3—C4—C9  | 118 (1)   | C17—C18—C19 | 120 (1)  |
| C5—C4—C9  | 120 (1)   | C18—C19—C20 | 119 (1)  |
| C4—C5—C6  | 119 (1)   | C19—C20—C21 | 122 (1)  |
| C5—C6—C7  | 121 (1)   | C20—C21—C22 | 119 (2)  |
| C6—C7—C8  | 120 (1)   | C17—C22—C21 | 122 (1)  |
| C7—C8—C9  | 122 (1)   |             |          |

H atoms were included in the refinement at calculated positions with C—H = 0.95  $\text{\AA}$ .

Data collection and cell refinement: *CAD-4 Software* (Enraf-Nonius, 1989). The structure was solved by direct methods using *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Full-matrix least-squares refinement was carried out using *MolEN* (Fair, 1990). Program used to prepare figure: *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: PA1165). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1996). **C52**, 203–205

## 2-Cyano-*N*-furfuryl-3-(2-furyl)acrylamide

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(Received 28 September 1993; accepted 26 June 1995)

## Abstract

In the title compound, C<sub>13</sub>H<sub>10</sub>N<sub>2</sub>O<sub>3</sub>, the two equivalent molecules in the asymmetric unit form dimers by means of an intermolecular N—H···N hydrogen bond with an N···H distance of 2.42 (2)  $\text{\AA}$ . There are weak intramolecular and intermolecular interactions between the C—H···N and C—H···O atoms. There are no unusual intramolecular distances or angles. The furan rings are rotated 81.1 (2) $^\circ$  relative to each other.

## Comment

It is not surprising that many natural products exhibit some bioactivity. In most cases, however, they are not sufficiently active or are too toxic to be of immediate practical use. In such cases, the structures have to be modified for activity enhancement or toxicity reduction, either empirically or more logically, *e.g.* by quan-