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3,3-Dichloro-1,4-diphenylazetid-2-one

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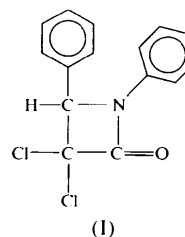
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Abstract

In the crystal structure of the title compound, C₁₅H₁₁Cl₂NO, the Cl—C—Cl plane is nearly perpendicular to the four-membered β -lactam ring [89.0(2)°] and the C—C bond distances in this group are 1.571(6) and 1.543(7) Å. The most out-of-plane atom from the best plane of the lactam ring is the carbonyl C atom [−0.029(5) Å]. The dihedral angle between the best planes of the phenyl rings is 77.4(2)°.

Comment

Recent developments in the field of β -lactams have shown that the essential feature of the antibacterial activity of these compounds is the presence of the β -lactam ring (azetid-2-one) (Brady & Gu, 1989; Takasuka *et al.*, 1982; Manhas *et al.*, 1988). The selectivity or activity can be decisively influenced by novel ring substituents (Sharma *et al.*, 1994; Kumar *et al.*, 1993). The biological activity of β -lactams has been studied by Chambers & Doedens (1980) and structural studies have been performed by Ercan *et al.* (1996a,b), Ülkü *et al.* (1997) and Paulus *et al.* (1969). The four atoms of the β -lactam ring are coplanar within experimental error. This paper describes the structure of (I), a molecule containing a β -lactam ring.



The four-membered β -lactam ring of (I) is nearly planar and the maximum deviation [−0.029(5) Å] is assumed by atom C7. The bond lengths in the lactam ring in (I) are comparable with the previous work on monocyclic azetid-2-ones (Ercan *et al.*, 1996a, b and references therein). The N1—C7 bond length of 1.362(6) Å, conjugated with the carbonyl group, is shorter than the N1—C9 and N1—C6 bond lengths [1.469(5) and 1.417(5) Å, respectively] and these results show a fair agreement with those of the previous studies (Ercan *et al.*, 1996a,b; Ülkü *et al.*, 1997). However, the O1=C7 bond length of 1.186(6) Å is slightly shorter than those found in the literature [1.198(12) Å; Allen *et al.*, 1987].

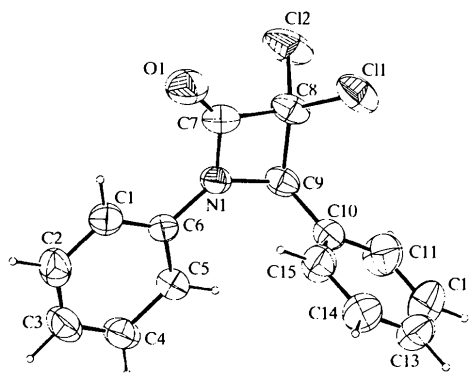


Fig. 1. PLATON (Spek, 1996) drawing of the title molecule, showing the molecular structure and atomic labelling scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms are shown as spheres of an arbitrary radius.

In the present work, the C7—C8 and C8—C9 bond lengths [1.543(7) and 1.571(6) Å, respectively] deviate slightly from those reported by Ercan *et al.* (1996a,b) and Ülkü *et al.* (1997) [1.536(5), 1.55(2), 1.535(5), 1.558(4), 1.60(2) and 1.566(5) Å]. The bond angle at C8 (C7—C8—C9) is 86.1(3)°, which is almost equal to those reported in previous work [86.3(2), 87.0(3) and 87.1(7)°; Ercan *et al.* (1996a,b); Ülkü *et al.* (1997)]. The dihedral angle formed by the phenyl rings is 77.4(2)°. The C1—C6 phenyl ring is nearly coplanar with the β -lactam moiety [6.0(2)°] and atom C7 deviates from the plane of the aromatic ring by 0.090(5) Å. In contrast, the second phenyl ring, C10—C15, makes a dihedral angle of 71.9(2)° with the best

plane of the β -lactam moiety. Atom N1 was found to be 0.015 (5) Å above the C6/C7/C9 plane, which may be due to the intramolecular interactions between O1...N1, O1...C8, N1...C1 and N1...C8 [2.352 (5), 2.521 (7), 2.422 (6) and 2.068 (5) Å, respectively]. The sum of the bond angles about N1 is exactly 360°.

Experimental

Solutions of benzylideneaniline (0.01 mol; 1.81 g) and triethylamine (0.02 mol; 2.78 ml) in benzene (50 ml) were added together and stirred for 15 min. Dichloroacetyl chloride (0.02 mol; 1.92 ml) was added dropwise to the stirred solution. The mixture was then stirred for 1 h at room temperature. The triethylamine salts were filtered off and the title compound was recrystallized from ethanol.

Crystal data

C₁₅H₁₁Cl₂NO $M_r = 292.16$

Monoclinic

C2/c

 $a = 19.305 (2) \text{ \AA}$ $b = 5.958 (1) \text{ \AA}$ $c = 23.847 (2) \text{ \AA}$ $\beta = 91.989 (8)^\circ$ $V = 2741.0 (5) \text{ \AA}^3$ $Z = 8$ $D_x = 1.416 \text{ Mg m}^{-3}$ D_m not measuredMo $K\alpha$ radiation $\lambda = 0.7107 \text{ \AA}$

Cell parameters from 22 reflections

 $\theta = 10.1\text{--}14.3^\circ$ $\mu = 0.463 \text{ mm}^{-1}$ $T = 293.2 \text{ K}$

Hexagonal

 $0.80 \times 0.35 \times 0.20 \text{ mm}$

Colourless

Data collection

Rigaku AFC-7S diffractometer

 $\omega/2\theta$ scans

Absorption correction:

 ψ scan (North *et al.*, 1968) $T_{\min} = 0.836$, $T_{\max} = 0.912$

4488 measured reflections

4360 independent reflections

1764 reflections with

 $I > 3\sigma(I)$ $R_{\text{int}} = 0.081$ $\theta_{\text{max}} = 30.01^\circ$ $h = 0 \rightarrow 26$ $k = 0 \rightarrow 8$ $l = -33 \rightarrow 33$

3 standard reflections

every 150 reflections

intensity decay: 0.11%

Refinement

Refinement on F $R = 0.057$ $wR = 0.057$ $S = 0.950$

1764 reflections

172 parameters

H-atom parameters not refined

Unit weights applied

 $(\Delta/\sigma)_{\text{max}} < 0.001$ $\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.55 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

C6—N1—C7	133.2 (4)	C11—C8—C9	117.1 (3)
C6—N1—C9	129.4 (4)	C12—C8—C7	111.5 (3)
C7—N1—C9	97.4 (3)	C12—C8—C9	114.0 (3)
N1—C6—C1	120.5 (4)	C7—C8—C9	86.1 (3)
N1—C6—C5	118.2 (4)	N1—C9—C8	85.7 (3)
O1—C7—N1	134.8 (5)	N1—C9—C10	115.5 (3)
O1—C7—C8	134.6 (5)	C8—C9—C10	118.3 (4)
N1—C7—C8	90.6 (4)	C9—C10—C11	119.8 (5)
C11—C8—C12	110.1 (2)	C9—C10—C15	121.3 (4)
C11—C8—C7	116.3 (3)		

The structure was solved by direct methods (Altomare *et al.*, 1993) and expanded using Fourier techniques (Beurskens *et al.*, 1994). The non-H atoms were refined anisotropically; H atoms were included but not refined. All H atoms were placed geometrically on the corresponding C atoms, except atom H9 which was located from the difference Fourier map.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN for Windows*. Software used to prepare material for publication: *TEXSAN for Windows*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1319). Services for accessing these data are described at the back of the journal.

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Table 1. Selected geometric parameters (Å, °)

C11—C8	1.756 (5)	N1—C9	1.469 (5)
C12—C8	1.759 (4)	C7—C8	1.543 (7)
O1=C7	1.186 (6)	C8—C9	1.571 (6)
N1—C6	1.417 (5)	C9—C10	1.505 (6)
N1—C7	1.362 (6)		