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3,3-Dichloro-4-(*p*-methoxyphenyl)-1-phenyl-2-azetidinone

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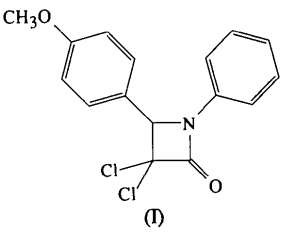
Abstract

The structure of the title compound consists of discrete $C_{16}H_{13}Cl_2NO_2$ molecules with a closest intermolecular contact of 2.54 (3) Å between the carbonyl O atom and a methyl H atom. The four-membered ring is nearly planar, with long C—C distances of 1.564 (5) and 1.537 (5) Å, similar to the distances observed in other substituted monocyclic β-lactams. The dihedral angle between the planes of the phenyl rings is 78.6 (1)°.

Comment

Many monocyclic β-lactams are reported to show antibiotic as well as antifungal activity (Chambers & Doedens, 1980). Structural information may provide some explanation for such behaviour. The molecular structure of 3,3-dichloro-4-(*p*-methoxyphenyl)-1-phenyl-2-azetidinone, (I), has been determined and the results are presented here.



(I)

The four-membered ring of (I) is nearly planar; deviations from the mean plane are C2 −0.018 (4), C3 0.016 (4), C4 −0.016 (3) and N1 0.019 (3) Å (Fig. 1). While the distances within the four-membered ring are in the range of previously observed minimum (1.342 Å) and maximum (1.602 Å) values for other substituted monocyclic 2-azetidinones (Paulus, Kobelt & Jensen, 1969; Parthasarathy, 1970; Kartha & Ambady, 1973; Colens, Declercq, Germain, Putzeys & Van Meerssche, 1974; Chambers & Doedens, 1980), the ring angle at C3, with a value of 86.3 (2)°, is slightly outside the range of 85.4–85.6° observed previously. The long C3—C4 distance [1.564 (5) Å] reported here seems to be in agreement with those found in similar molecules. The phenyl rings have unexceptional geometry. Their least-squares planes are almost perpendicular to one another [dihedral angle 78.6 (1)°]. The shortest intermolecular distance of 2.54 (3) Å is between the carbonyl O atom and a methyl H atom.

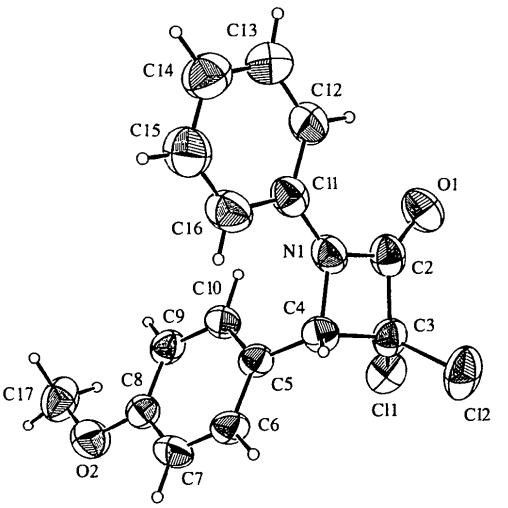


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title molecule with the atom-numbering scheme. Displacement ellipsoids are shown at 50% probability levels.

Experimental

A solution of *p*-methoxybenzylideneaniline (0.01 mol, 2.11 g) and triethylamine (0.02 mol, 2.78 ml) in 35 ml benzene was stirred for 15 min. Dichloroacetyl chloride (0.02 mol, 1.92 ml) was added dropwise to the solution and the mixture stirred at

room temperature for one day. The triethylamine salts were filtered off and the title compound was recrystallized from ethanol.

Crystal data

$C_{16}H_{13}Cl_2NO_2$
M_r = 322.19
Monoclinic
$P2_1/n$
a = 5.739 (3) Å
b = 14.237 (2) Å
c = 18.644 (2) Å
β = 93.65 (1)°
V = 1520.2 (8) Å^3
Z = 4
D_x = 1.41 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–16°
μ = 0.43 mm^{-1}
T = 296 K
Needle
0.70 × 0.32 × 0.25 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.918, T_{max} = 0.999
3089 measured reflections
2666 independent reflections

1950 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.015
θ_{max} = 25.0°
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 16$
$l = -22 \rightarrow 22$
3 standard reflections
frequency: 120 min
intensity decay: 1.1%

Refinement

Refinement on F
R = 0.042
wR = 0.040
S = 0.64
1950 reflections
229 parameters
H atoms refined with U(H) = 1.3U_{eq}(C)

$w = (84.32/F)^2$ if $F \geq 84.32$, otherwise w = 1.0
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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)*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Cl1	0.8047 (2)	0.26871 (6)	0.28527 (5)	5.04 (2)
Cl2	1.0826 (2)	0.20606 (7)	0.17131 (6)	6.11 (2)
O1	0.6125 (4)	0.3435 (2)	0.1184 (1)	5.73 (6)
O2	1.2007 (4)	0.5869 (2)	0.4783 (1)	5.09 (5)
C2	0.7945 (5)	0.3590 (2)	0.1530 (2)	4.22 (7)
C3	0.9478 (5)	0.3037 (2)	0.2088 (2)	3.95 (7)
C4	1.1051 (5)	0.3929 (2)	0.2147 (2)	3.67 (6)
N1	0.9443 (5)	0.4328 (2)	0.1572 (1)	3.92 (6)
C5	1.1239 (5)	0.4469 (2)	0.2832 (2)	3.40 (6)
C6	1.3194 (5)	0.4356 (2)	0.3310 (2)	3.91 (7)
C7	1.3401 (5)	0.4825 (2)	0.3948 (2)	4.15 (7)
C8	1.1660 (5)	0.5431 (2)	0.4140 (2)	3.62 (6)
C9	0.9693 (5)	0.5562 (2)	0.3675 (2)	3.80 (7)
C10	0.9509 (5)	0.5081 (2)	0.3030 (2)	3.79 (6)
C11	0.9597 (5)	0.5165 (2)	0.1182 (2)	3.96 (7)
C12	0.7902 (6)	0.5397 (3)	0.0646 (2)	5.09 (8)
C13	0.8094 (7)	0.6211 (3)	0.0255 (2)	5.93 (9)
C14	0.9951 (7)	0.6804 (3)	0.0395 (2)	6.1 (1)
C15	1.1605 (7)	0.6574 (3)	0.0928 (2)	6.2 (1)
C16	1.1456 (6)	0.5770 (3)	0.1318 (2)	5.22 (8)
C17	1.0263 (8)	0.6514 (3)	0.4980 (2)	5.81 (9)

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

Cl1—C3	1.762 (3)	C2—N1	1.357 (4)
Cl2—C3	1.757 (3)	C3—C4	1.558 (4)
O1—C2	1.213 (4)	C4—N1	1.482 (4)
O2—C8	1.355 (4)	C4—C5	1.487 (4)
O2—C17	1.423 (5)	N1—C11	1.403 (4)
C2—C3	1.536 (5)		
C8—O2—C17	117.4 (3)	Cl2—C3—C4	113.9 (2)
O1—C2—C3	134.8 (3)	C2—C3—C4	86.3 (2)
O1—C2—N1	133.9 (3)	C3—C4—N1	85.9 (2)
C3—C2—N1	91.3 (2)	C3—C4—C5	119.5 (3)
Cl1—C3—Cl2	109.7 (2)	N1—C4—C5	115.5 (3)
Cl1—C3—C2	114.6 (2)	C2—N1—C4	96.3 (3)
Cl1—C3—C4	118.1 (2)	C2—N1—C11	133.5 (3)
Cl2—C3—C2	112.6 (2)	C4—N1—C11	130.1 (3)
C17—O2—C8—C7	178.3 (3)	C2—C3—C4—C5	114.2 (3)
C3—C2—N1—C4	−3.4 (2)	C3—C4—C5—C6	101.0 (3)
C3—C2—N1—C11	172.4 (3)	C2—N1—C11—C12	3.9 (5)

All H atoms except H4 were bonded geometrically at a distance of 0.95 Å from their corresponding C atoms. Atom H4 was obtained from a difference Fourier map and refined isotropically.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN SIMPEL*. Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEP* (Johnson, 1965) in *MolEN*. Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: CF1057). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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