to refine structure: *LSFM* in *SDP-Plus*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF VAX* in *MolEN* (Fair, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1007). Services for accessing these data are described at the back of the journal.

References

Benharref, A., Chekroun, A. & Lavergne, J. P. (1991). *Bull. Soc. Chim. Fr.* **128**, 738–741.

Benharref, A., El Jamili, H., Lassaba, E., Giorgi, M. & Pierrot, P. (1995). *Acta Cryst.* C**51**, 2658–2661.

Chiaroni, A., Pais, M., Riche, C., Benharref, A., Chekroun, A. & Lavergne, J. P. (1991). *Acta Cryst.* C**47**, 1945–1948.

Chiaroni, A., Riche, C., Benharref, A., Chekroun, A. & Lavergne, J. P. (1992). *Acta Cryst.* C**48**, 1720–1722.

Chiaroni, A., Riche, C., Benharref, A., Lassaba, E. & Baouid, A. (1996). *Acta Cryst.* C**52**, 2504-2507.

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.

Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Joseph, T. C. & Dev, S. (1968*a*). *Tetrahedron*, **24**, 3809–3827.

Joseph, T. C. & Dev, S. (1968*b*). *Tetrahedron*, **24**, 3841–3852.

Joseph, T. C. & Dev, S. (1968*c*). *Tetrahedron*, **24**, 3853–3859.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.

Narula, A. P. S. & Dev, S. (1977). *Tetrahedron*, **33**, 813–816.

Plattier, A. & Teisseire, P. (1974). *Recherches*, **19**, 131–144.

Teisseire, P. & Plattier, M. (1974). *Recherches*, **19**, 167–172.

Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1997). C**53**, 1945–1947

3,3-Dichloro-4-(4-chlorophenyl)-1-phenylazetidin-2-one

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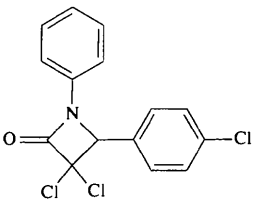
Abstract

In molecules of $C_{15}H_{10}Cl_3NO$, the four-membered lactam ring is nearly planar, with a C—N bond length of 1.367 (4) Å in the amide group and C—C distances of 1.535 (5) and 1.566 (5) Å. The displacement of the amide N atom from the plane through the atoms attached to it is 0.056 (3) Å, indicating some pyramidal character which is correlated to the biological activity in related compounds. The phenyl rings are nearly perpendicular to one another [dihedral angle 79.4 (1)°]. There are intramolecular and intermolecular hydrogen bonds in the structure.

Comment

As a result of their reported antibiotic and antifungal activity (Chambers & Doedens, 1980), structural studies of monocyclic β-lactams have been of interest to this laboratory (Ercan, Ülkü & Güner, 1996*a*,*b*). The four lactam ring atoms are coplanar to within experimental error in other monocyclic β-lactams (Paulus, Kobelt & Jensen, 1969; Kartha & Ambady, 1973; Colens, Declercq, Germain, Putzeys & Van Meerssche, 1974). However, in the title compound, (I), the lactam ring N atom is displaced by 0.056 (3) Å from the C2/C4/C11 plane which indicates that it adopts a flat pyramidal coordination. Sweet & Dahl (1970) reported a correlation between the pyramidal character of the amide N atom, which possibly arises from intramolecular repulsions and the amide C—N bond lengths. It has been suggested that the antibiotic activity of β-lactams depends on the pyramidal character of the lactam N atoms. There also seems to be a correlation between increased activity and decreased electron delocalization in the amide group as shown by a lengthening of the C—N bond and a shortening of the C=O bond (Lee *et al*, 1990; Lee *et al*, 1990).



(I)

A comparison of the activity-related structural parameters of (I) with those reported for biologically active and inactive compounds, indicates that its parameters are closer to those of the active compounds. For instance, the C—N bond length of 1.367 (4) Å is closer to the corresponding C—N bond length [1.392 (4) Å; Domiano, Nardelli, Blasmo, Macchia & Macchia, 1979] reported for an active penicillin derivative, than to the corresponding C—N bond lengths observed in some structurally related but biologically inactive compounds, such as azetidone derivatives [1.342 (4)–1.337 (4) Å; Lee *et al*., 1990; Lee *et al*, 1990] and Δ^2-cephalosporin (1.339 Å; Sweet & Dahl, 1970). The displacement of the lactam ring N atom is also notably larger than in the inactive compound mentioned above. Whether these

structural differences result in a biologically active compound remains to be seen. The C2—C3 bond length [1.535 (5) Å] is longer than most comparable values, but is slightly shorter than the value [1.561 (6) Å] observed in $C_{16}H_{17}ClN_2OS$ (Chambers & Doedens, 1980). The longest bond length in the lactam ring is C3—C4 [1.566 (5) Å], which fits the previously observed trend. The dihedral angle between the lactam ring and the phenyl ring is 10.5 (7)°, while the corresponding angle for the chlorophenyl ring is 68.9 (1)°. Van der Waals interactions seem to be dominant in the molecular packing. There is one intramolecular (C16—H16···O) and one intermolecular [C4—H4···O($x-1$, y, z)] hydrogen bond (Table 2).

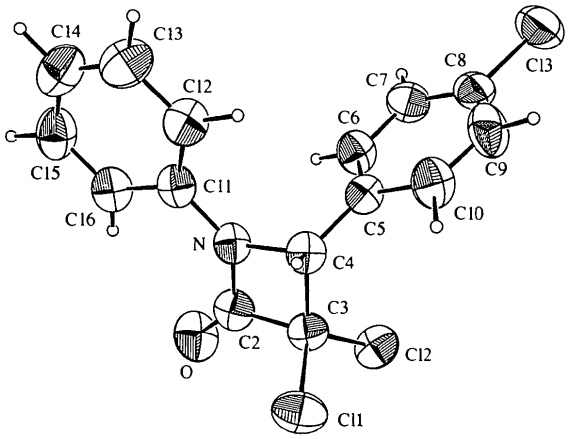


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

Experimental

p-Cholorobenzylideneaniline (0.01 mol, 2.75 g) and triethylamine (0.02 mol, 2.78 ml) in benzene (50 ml) were stirred for 15 min. Dichloroacetyl chloride (0.02 mol, 1.92 ml) was added dropwise to the solution. The mixture was stirred at room temperature for one day. Triethylamine salts were filtered off and the product was recrystallized from ethanol.

Crystal data

$C_{15}H_{10}Cl_3NO$
M_r = 326.61
Monoclinic
$P2_1/n$
a = 5.8656 (2) Å
b = 14.0699 (2) Å
c = 17.8671 (2) Å
β = 92.335 (4)°
V = 1473.4 (1) Å^3
Z = 4
D_x = 1.47 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–18°
μ = 0.62 mm^{-1}
T = 295 K
Prismatic
0.60 × 0.26 × 0.18 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*MolEN*; Fair, 1990)
T_{min} = 0.726, T_{max} = 0.894
3832 measured reflections
2573 independent reflections
2083 reflections with $I > \sigma(I)$
R_{int} = 0.012
θ_{max} = 25.0°
$h = -1 \rightarrow 6$
$k = -1 \rightarrow 16$
$l = -21 \rightarrow 21$
3 standard reflections
frequency: 120 min
intensity decay: 1.4%

Refinement

Refinement on F
R = 0.055
wR = 0.064
S = 1.10
2083 reflections
185 parameters
H atoms: see below
$w = 1/[\sigma F^2 + (0.02F)^2 + 0.40]$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Cl1—C3	1.770 (4)	N—C4	1.483 (4)
Cl2—C3	1.758 (4)	N—C11	1.408 (5)
Cl3—C8	1.731 (4)	C2—C3	1.535 (5)
O—C2	1.198 (4)	C3—C4	1.566 (5)
N—C2	1.367 (4)	C4—C5	1.487 (5)
C2—N—C4	96.9 (3)	C2—C3—C4	87.0 (3)
C2—N—C11	133.9 (3)	N—C4—C3	85.4 (2)
C4—N—C11	128.7 (3)	N—C4—C5	116.0 (3)
O—C2—N	133.6 (4)	C3—C4—C5	119.1 (3)
O—C2—C3	135.7 (3)	C4—C5—C6	122.9 (3)
N—C2—C3	90.7 (3)	C4—C5—C10	119.2 (3)
Cl1—C3—Cl2	109.5 (2)	C6—C5—C10	117.8 (3)
Cl1—C3—C2	112.1 (3)	Cl3—C8—C7	119.4 (3)
Cl1—C3—C4	113.0 (2)	Cl3—C8—C9	120.6 (3)
Cl2—C3—C2	115.9 (2)	N—C11—C12	119.3 (3)
Cl2—C3—C4	117.9 (2)	N—C11—C16	120.8 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O	0.950	2.587	3.186 (5)	121.5
C4—H4···O^{i}	0.982 (3)	2.493 (3)	3.475 (4)	177.9 (3)

Symmetry code: (i) $x-1$, y, z.

All non-H atoms were refined with anisotropic displacement parameters. All H atoms except H4 were placed geometrically 0.95 Å from their corresponding C atoms with U_{iso}(H) = 1.3U_{eq}(C) and a riding model was used. H4 was located in a difference Fourier map and refined isotropically.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *MolEN* version of *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1315). Services for accessing these data are described at the back of the journal.

References

Chambers, R. & Doedens, R. J. (1980). *Acta Cryst.* B**36**, 1507–1510.
Colens, A., Declercq, J.-P., Germain, G., Putzeys, J. P. & Van Meerssche, M. (1974). *Cryst. Struct. Commun.* **3**, 119–122.
Domiano, P., Nardelli, M., Balsamo, A., Macchia, B. & Macchia, F. (1979). *Acta Cryst.* B**35**, 1363–1372.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.
Ercan, F., Ülkü, D. & Güner, V. (1996*a*). *Acta Cryst.* C**52**, 1779–1780.
Ercan, F., Ülkü, D. & Güner, V. (1996*b*). *Z. Kristallogr.* **211**, 735–736.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kartha, G. & Ambady, G. (1973). *J. Chem. Soc. Perkin Trans.* 2, pp. 2042–2044.
Lee, C. H., Cho, S., Kim, E., Shin, H., Ruble, J. R. & Craven, B. M. (1990). *Acta Cryst.* C**46**, 1450–1452.
Lee, Y. H., Cho, S., Kim, E., Shin, H., Ruble, J. R. & Craven, B. M. (1990). *Acta Cryst.* C**46**, 120–122.
Paulus, E. F., Kobelt, D. & Jensen, H. (1969). *Angew. Chem. Int. Ed. Engl.* **8**, 990–991.
Sweet, R. M. & Dahl, L. F. (1970). *J. Am. Chem. Soc.* **92**, 5489–5507.

Acta Cryst. (1997). C**53**, 1947–1949

Dimethyl (±)-(1*S**,2*R**,3*S**)-[3-Phenyl-1-(*N*-phenylcarbamoyloxy)-2,3-epoxypropyl]-phosphonate

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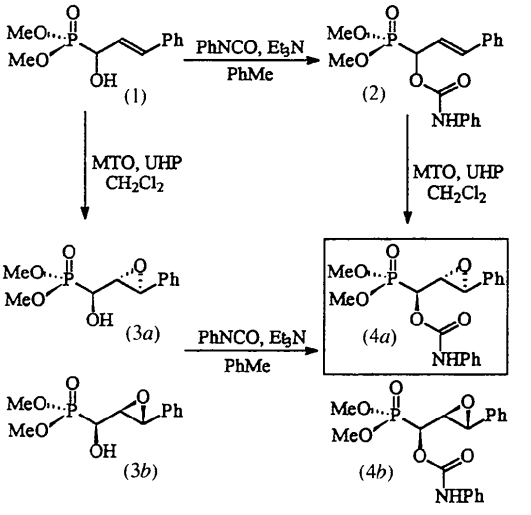
Abstract

The crystal structure of the racemic title compound, $C_{18}H_{20}NO_6P$ (m.p. 428–431 K), has been determined by X-ray diffraction. The title compound consists of a tetrahedral P atom bonded to two methoxy groups, and an alkyl chain. The alkyl chain is substituted at position 1 with a carbamate and with an epoxide at positions 2 and 3. The relative configuration of the 1-carbamate and 2,3-epoxide substituents was confirmed as *anti* (1*S*,2*R*,3*S*). The crystal structure contains an enantiomeric pair with two intermolecular hydrogen bonds in a 14-membered ring. The hydrogen bonds are formed between the P=O of one enantiomer and the N—H of the other.

Comment

Methyl trioxorhenium (MTO) when combined with aqueous hydrogen peroxide forms peroxy adducts capable of the epoxidation of alkenes (Herrmann, Fischer & Marz, 1991; Herrmann, Fischer, Scherer & Wauch, 1993; Herrmann, Fischer, Rauch & Scherer, 1994; Al-Ajlouni & Espenson, 1995). However, one of the potential shortcomings of this reagent combination is the need for a protic solvent which may lead to the destruction of sensitive products (Herrmann, Fischer, Rauch & Scherer, 1994) or a reduction in the stereoselectivity due to competitive hydrogen bonding by the solvent (Murray, Singh, Williams & Moncrieff, 1995). Realizing the potential need for a non-protic variant of this reagent system, we initiated a study (Boehlow & Spilling, 1997) to examine urea hydrogen peroxide (UHP) (Heaney, 1993) as a reoxidant of MTO in non-protic solvents for the catalytic epoxidation of alkenes.

During this study, we oxidized the allylic hydroxyphosphonate (1) and its carbamate derivative (2) to give diastereoisomeric mixtures of epoxides (3) (3.5:1) and (4) (1:3.8), respectively. The epoxide diastereoisomers were correlated by converting the epoxyalcohol (3) into the epoxycarbamate (4) with phenyl isocyanate. Interestingly, the allylic hydroxyphosphonate (1) and the carbamate (2) showed a preference for the opposite epoxide diastereoisomers. However, the relative stereochemistry of the epoxide diastereoisomers remained unconfirmed.



In an earlier experiment, the carbamate (2) was oxidized with dimethyl dioxirane (DMD) to give the epoxide isomers (4) in a 1:1 ratio. The epoxide isomer (4*a*) [major isomer from (2) with MTO/UHP] was isolated