3·456 (4) and 3·402 (4) Å for molecules *A* and *B* respectively.

A model of the desired reaction product, the diazasteroid (II), was generated by computer graphics (Badler *et al.*, 1979) using the coordinates for molecule *A* of the title compound as a starting point; the C(1)–C(1*A*) bonding constraint was removed and the C(1*A*)–C(4*A*)–C(4)–C(13) torsion angle was altered from approximately 0° in (I) to 180° in (II). Analysis of the geometry of this molecule (II) reveals steric interference between the indicated H atom and its neighboring phenyl group; in particular, the separation between the indicated H and phenyl C atoms [distinguished by asterisks in (II), the diazasteroid] is 1·8 Å while the sum of their van der Waals radii is 2·9 Å. This implies that severe buckling of the principal ring system would be required to relieve this close approach if the diazasteroid were formed. Thus the principal pathway of the reaction is determined by the lesser amount of strain or buckling in the observed major product. It is likely that the synthesis of the diazasteroid (II) is sterically inhibited for this reason.

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The Structure of 2,2-Dimethyl-3-ureido-6-phenoxyacetamidopenam*

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Abstract

$C_{16}H_{20}N_4O_4S$, M_r = 364·43, an active derivative, resistant to penicillinase, with a ureide moiety instead of a carboxy group, crystallizes in the orthorhombic space group $P2_12_12_1$ with a = 17·751 (6), b = 15·306 (4), c = 6·493 (2) Å, V = 1764 Å^3, Z = 4, D_m = 1·364, D_c = 1·371 Mg m^{-3}, $F(000)$ = 768. R = 0·040 for 1704 observed independent reflections. The thiazolidine ring exhibits an envelope conformation with C(3) 0·52 Å out of the plane through the remaining four atoms. The β-lactam ring is folded 9° from planarity. Each molecule is hydrogen bonded to four neighbouring molecules. The resistance to β-lactamase is caused by the changed chemical properties of the compound rather than by shielding of the β-lactam.

* {3,3-Dimethyl-7-oxo-6-[(phenoxyacetyl)amino]-4-thia-1-azabicyclo[3.2.0]hept-2-yl}urea.

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Introduction

One of the crucial problems in the chemical modifications of penicillin is the development of compounds with increased resistance to penicillinases. The problem has been partially solved by the introduction of voluminous substituents into the acyl moiety of penicillin. The steric hindrance shields the β-lactam from the attack of the enzyme and results in the high resistance of compounds like cloxacillin to the degradative action of penicillinase. Nevertheless compounds of that type exhibit activity only against Gram-positive bacteria. Extensive work has been done to develop other types of modified penicillins which, apart from not being the substrates of penicillinases, would exhibit activity against Gram-negative bacteria.

A novel type of active penicillin derivative, namely the ureido analogues in which the carboxyl has been replaced by an unsubstituted or substituted ureido group, has been recently developed (Domaradzki, Mikołajczyk, Kazimierczak, Rogowska & Sikora, 1977). These compounds exhibit high resistance to penicillinase, although they do not contain an acyl moiety like normal penicillin *G*.

The question arises whether the resistance to penicillinase of this type of compound is due to steric hindrance caused by the particular conformation of ureido analogues, or to the changed chemical properties of the compound and thus the changed susceptibility to enzyme action.

Our studies were aimed at the elucidation of this problem.

Experimental

The compound was synthesized by Dr Domaradzki of the Institute of Pharmaceutical Industry, Warsaw. Crystals were grown from methanol as colourless needles. The space group was determined from oscillation and Weissenberg photographs which showed orthorhombic symmetry and systematic absences for $h00$, $0k0$ and $00l$ with h, k and l odd respectively. The density was measured by flotation in water/KI solution. A crystal of dimensions 0·25 × 0·35 × 0·9 mm was used for data collection. Cell parameters were obtained by least squares from 2θ values for 31 high-order (>100°) reflections measured on a diffractometer. The intensities of 1735 reflections were collected on a Stoe automatic four-circle diffractometer up to an angle of θ = 130° with Ni-filtered Cu $K\alpha$ radiation (λ = 1·54179 Å), using the θ–2θ scan technique. Of these, 31 were considered as unobserved [$I < 2\sigma(I)$]. Two standard reflections were measured every 40 measurements with no significant deterioration in intensity. No absorption correction was applied ($\mu = 1{\cdot}85$ mm^{-1}).

Structure determination and refinement

The structure was solved with *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). The E map calculated from the set of phases with the highest combined figure of merit showed all 25 non-H atoms. Isotropic and anisotropic full-matrix least-squares refinement (*CRYLSQ* program) reduced R ($=\sum||F_o| - |F_c||/\sum|F_o|$) to 0·076. A difference synthesis at this stage clearly indicated all H atoms which were included in the refinement with isotropic temperature factors. One very strong reflection (031) presumed to be affected by extinction or measurement error was eliminated. The final R was 0·040. Unit weights were used for all reflections. No significant peaks were observed in the final difference map except for a few peaks of *ca* 0·4 e Å^{-3} in the vicinity of the S atom. None of the positional parameters of the non-H atoms shifted more than $0{\cdot}07\sigma$ in the last cycle. The final positional parameters are listed in Table 1.* Anomalous-dispersion corrections for S and O were

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, and positional and thermal parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35928 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final positional parameters and equivalent isotropic thermal parameters* (×10²) *for the non-*H *atoms with their e.s.d.'s in parentheses*

U_{eq} is defined according to Hamilton (1959).

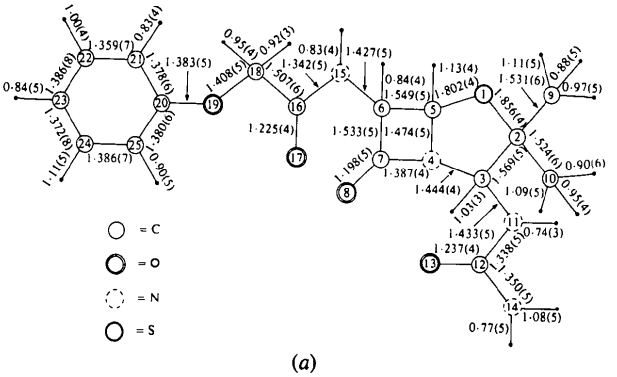
	x	y	z	U_{eq} (Å²)
S(1)	0·64989 (6)	0·52307 (8)	0·5495 (2)	6·41 (5)
C(2)	0·5529 (2)	0·5604 (3)	0·6095 (6)	4·7 (1)
C(3)	0·5517 (2)	0·5668 (2)	0·8507 (6)	3·7 (1)
N(4)	0·5995 (2)	0·4968 (2)	0·9223 (5)	3·7 (1)
C(5)	0·6686 (2)	0·4800 (2)	0·8026 (7)	4·5 (1)
C(6)	0·6593 (2)	0·3817 (2)	0·8530 (6)	4·2 (1)
C(7)	0·5844 (2)	0·4088 (2)	0·9514 (7)	4·3 (1)
O(8)	0·5318 (2)	0·3719 (2)	1·0280 (5)	6·5 (1)
C(9)	0·4970 (3)	0·4905 (3)	0·5388 (8)	7·1 (2)
C(10)	0·5395 (3)	0·6478 (3)	0·5031 (8)	7·9 (2)
N(11)	0·5783 (1)	0·6482 (2)	0·9321 (5)	4·0 (1)
C(12)	0·5322 (2)	0·7129 (2)	0·9893 (6)	4·3 (1)
O(13)	0·4633 (1)	0·7081 (2)	0·9630 (5)	6·0 (1)
N(14)	0·5657 (2)	0·7828 (2)	1·0765 (6)	5·7 (1)
N(15)	0·6524 (2)	0·3220 (2)	0·6854 (5)	4·3 (1)
C(16)	0·7131 (2)	0·3047 (2)	0·5691 (7)	4·5 (1)
O(17)	0·7743 (1)	0·3380 (2)	0·6073 (5)	6·5 (1)
C(18)	0·6981 (2)	0·2478 (3)	0·3842 (7)	5·0 (1)
O(19)	0·7684 (2)	0·2199 (2)	0·3085 (5)	6·4 (1)
C(20)	0·7674 (2)	0·1689 (2)	0·1327 (7)	4·9 (1)
C(21)	0·7051 (2)	0·1540 (3)	0·0112 (7)	5·6 (2)
C(22)	0·7112 (3)	0·1032 (3)	−0·1599 (8)	6·8 (2)
C(23)	0·7797 (4)	0·0667 (3)	−0·2154 (8)	7·3 (2)
C(24)	0·8416 (3)	0·0808 (3)	−0·0932 (9)	6·7 (2)
C(25)	0·8360 (2)	0·1322 (3)	0·0818 (8)	5·6 (1)

used and R for the inverted structure was 0·042. Scattering factors for C, N, O and S were taken from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965). All computations were performed on a CDC Cyber 175 computer at the Wissenschaftliche Rechenzentrum Berlin with the XRAY system (Stewart, 1976) and *MULTAN* (Main *et al.*, 1977).

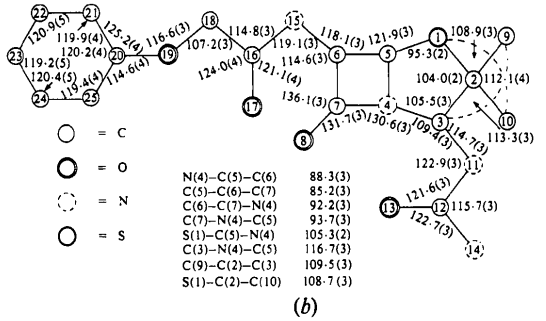
Results and discussion

The bond lengths and angles are given in Fig. 1 with the atom numbering. Torsion angles are summarized in Table 2. A stereoscopic drawing of the molecule is given in Fig. 2 and unit-cell contents in Fig. 3.

The thiazolidine ring, S(1)–C(2)–C(3)–N(4)–C(5), exhibits an envelope conformation with C(3) 0·52 Å out of the plane of the remaining four ring atoms (Table 3). The C–S–C angle is 95·3°; the C–S bonds differ slightly in length, S–C(2) being 1·856 and S–C(5) 1·802 Å; this is a common effect for all penicillin derivatives (Sweet, 1972; Domiano, Nardelli, Balsamo, Macchia & Macchia, 1979; Csöregh & Palm, 1977; Palm & Csöregh, 1978). N(4) is pyramidal and has the S configuration; it lies 0·36 Å out of the plane defined by its three substituents. The sum of the valency angles around N(4) is 341°.



(*a*)



(*b*)

Fig. 1. Atom numbering and (*a*) bond lengths (Å) and (*b*) angles (°).

Table 2. *Selected torsion angles* (°)

C(5)–S(1)–C(2)–C(3)	20·1 (3)	C(3)–N(4)–C(7)–O(8)	−44·0 (7)
C(5)–S(1)–C(2)–C(9)	−96·5 (3)	C(5)–N(4)–C(7)–C(6)	6·4 (3)
C(5)–S(1)–C(2)–C(10)	141·1 (3)	C(5)–N(4)–C(7)–O(8)	174·3 (5)
C(2)–S(1)–C(5)–N(4)	−0·3 (3)	S(1)–C(5)–C(6)–C(7)	−101·3 (3)
C(2)–S(1)–C(5)–C(6)	97·3 (3)	S(1)–C(5)–C(6)–N(15)	14·2 (4)
S(1)–C(2)–C(3)–N(4)	−34·5 (3)	N(4)–C(5)–C(6)–C(7)	5·7 (3)
S(1)–C(2)–C(3)–N(11)	86·0 (3)	N(4)–C(5)–C(6)–N(15)	121·2 (3)
C(9)–C(2)–C(3)–N(4)	81·7 (4)	C(5)–C(6)–C(7)–N(4)	−6·1 (3)
C(9)–C(2)–C(3)–N(11)	−157·8 (3)	C(5)–C(6)–C(7)–O(8)	174·7 (5)
C(10)–C(2)–C(3)–N(4)	−152·3 (3)	N(15)–C(6)–C(7)–N(4)	−124·9 (3)
C(10)–C(2)–C(3)–N(11)	−31·8 (4)	N(15)–C(6)–C(7)–O(8)	55·9 (7)
C(2)–C(3)–N(4)–C(5)	39·1 (4)	C(2)–C(3)–N(11)–C(12)	97·9 (4)
C(2)–C(3)–N(4)–C(7)	−82·5 (4)	N(4)–C(3)–N(11)–C(12)	−143·8 (4)
N(11)–C(3)–N(4)–C(5)	−84·7 (4)	C(5)–C(6)–N(15)–C(16)	71·8 (4)
N(11)–C(3)–N(4)–C(7)	153·6 (4)	C(7)–C(6)–N(15)–C(16)	169·9 (3)
C(3)–N(4)–C(5)–S(1)	−23·3 (3)	N(15)–C(16)–C(18)–O(19)	−166·8 (3)
C(3)–N(4)–C(5)–C(6)	−146·0 (3)	O(17)–C(16)–C(18)–O(19)	17·7 (5)
C(7)–N(4)–C(5)–S(1)	116·4 (3)	C(16)–C(18)–O(19)–C(20)	−177·7 (3)
C(7)–N(4)–C(5)–C(6)	−6·3 (3)	C(18)–O(19)–C(20)–C(21)	9·8 (6)
C(3)–N(4)–C(7)–C(6)	136·7 (4)		

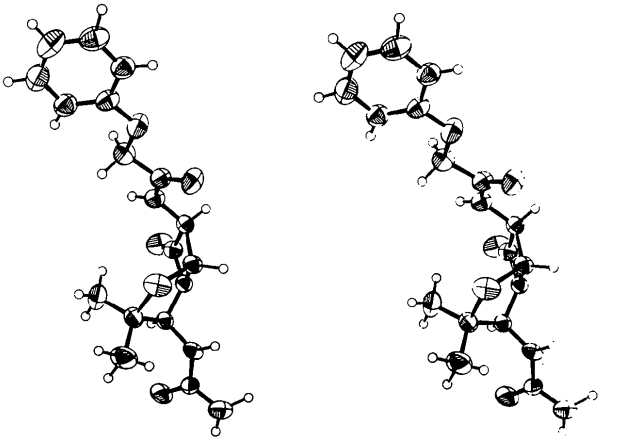


Fig. 2. A stereoscopic pair of the molecule (*ORTEP* II, Johnson, 1976).

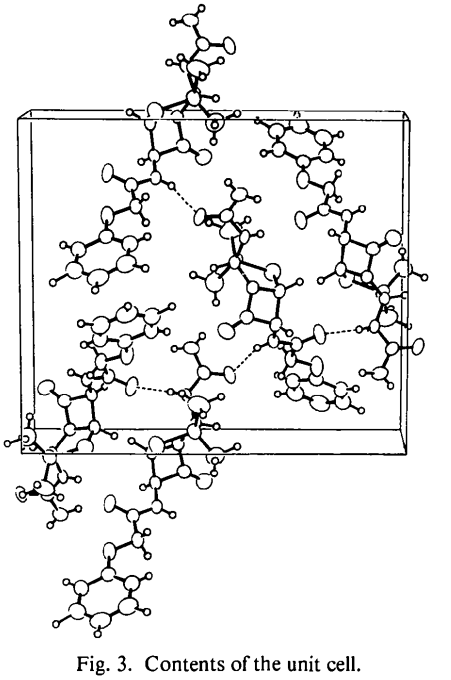


Fig. 3. Contents of the unit cell.

Table 3. *Mean-plane calculations*

(*a*) Deviations (Å) of atoms from least-squares planes. Atoms not included in the calculation of the planes are starred. E.s.d.'s are <0·01 Å for non-H and <0·08 Å for H atoms.

Plane 1	N(4)	0·027	Plane 7	N(11)	0·0001
	C(5)	−0·044		C(12)	0·0004
	C(6)	0·041		O(13)	0·0001
	C(7)	−0·048		N(14)	0·0001
	O(8)*	−0·133		C(3)*	0·096
				H(111)*	−0·139
Plane 2	N(4)	0·0		H(141)*	0·593
	C(5)	0·0		H(142)*	−0·089
	C(6)	0·0			
			Plane 8	N(15)	0·005
Plane 3	N(4)	0·0		C(16)	−0·024
	C(6)	0·0		O(17)	0·005
	C(7)	0·0		C(18)	0·008
	O(8)*	0·012		C(6)*	0·101
				H(151)*	−0·020
Plane 4	C(3)	0·0			
	C(5)	0·0	Plane 9	C(20)	0·003
	C(7)	0·0		C(21)	−0·001
	N(4)*	0·355		C(22)	−0·005
				C(23)	0·008
Plane 5	S(1)	0·009		C(24)	−0·002
	C(2)	−0·234		C(25)	−0·003
	C(3)	0·250		O(19)*	0·021
	N(4)	−0·118		H(211)*	−0·012
	C(5)	0·049		H(221)*	−0·033
				H(231)*	0·050
Plane 6	S(1)	0·0		H(241)*	−0·011
	C(2)	−0·002		H(251)*	−0·017
	N(4)	0·002			
	C(5)	−0·004			
	C(3)*	0·518			

(*b*) Angles between planes (°)

Planes 1 and 5	58 (6)
Planes 1 and 8	58 (3)
Planes 2 and 3	9 (2)

Table 4. *Short contacts* (Å)

(*a*) Intramolecular		(*b*) Intermolecular	
C(2)···C(6)	3·682 (5)	C(3)···O(17^{i})	3·799 (4)
C(2)···C(7)	3·260 (6)	C(5)···S(1^{i})	3·600 (4)
C(3)···O(8)	3·217 (5)	N(11)···O(19^{i})	3·482 (4)
O(8)···N(15)	3·180 (4)	N(14)···O(17^{i})	3·396 (4)
C(7)···C(9)	3·339 (7)	N(14)···O(19^{i})	3·421 (5)
C(10)···N(11)	2·870 (6)	C(9)···N(14^{ii})	3·449 (6)
C(10)···C(12)	3·313 (7)	C(18)···O(13^{ii})	3·091 (4)
C(10)···O(13)	3·406 (6)	N(14)···C(24^{iii})	3·415 (6)
N(11)···S(1)	3·384 (3)	O(8)···N(14^{iv})	3·384 (5)
N(11)···C(5)	3·146 (5)	O(8)···C(24^{v})	3·479 (6)
C(6)···O(17)	2·675 (5)	O(8)···C(25^{v})	3·550 (5)
O(17)···S(1)	3·611 (3)	C(22)···N(15^{vi})	3·648 (6)
O(17)···C(5)	3·139 (5)	C(10)···N(14^{vi})	3·487 (6)
O(17)···C(19)	2·654 (5)		
C(18)···C(21)	2·819 (6)		

Symmetry code: (i) $\frac{3}{2}-x, 1-y, \frac{1}{2}+z$; (ii) $1-x, -\frac{1}{2}+y, \frac{3}{2}-z$; (iii) $\frac{3}{2}-x, 1-y, \frac{3}{2}+z$; (iv) $1-x, -\frac{1}{2}+y, \frac{5}{2}-z$; (v) $-\frac{1}{2}+x, \frac{1}{2}-y, 1-z$; (vi) $x, y, -1+z$.

Table 5. *Hydrogen bonds*

N(11)···O(17^{i})	2·861 (4) Å	N(15)···O(13^{ii})	2·860 (4) Å
N(11)–H(111)	0·74 (3)	N(15)–H(151)	0·83 (4)
H(111)···O(17^{i})	2·21 (4)	H(151)···O(13^{ii})	2·08 (4)
Angle	147 (4)°	Angle	157 (4)°

Symmetry code: (i) $\frac{3}{2}-x, 1-y, \frac{1}{2}+z$; (ii) $1-x, -\frac{1}{2}+y, \frac{3}{2}-z$.

The β-lactam ring is not planar, the angle between the N(4)–C(5)–C(6) and N(4)–C(6)–C(7) planes being 9°. O(8) lies in the latter plane. The angle between the mean planes through the thiazolidine and β-lactam rings is 58°. The configuration of C(5) is *R*.

N(11), C(12), O(13) and N(14) belonging to the ureide group are coplanar. C(3), of *R* configuration, with which this group is axially connected is 0·096 Å out of the plane. The torsion angle N(4)–C(3)–N(11)–C(12) (−144°) is larger than C(2)–C(3)–N(11)–C(12) (98°) and the distances between the α-methyl C(10) and N(11), C(12) and O(13) of the ureide group are relatively short (Table 4). Such a conformation of the ureide group is due to intermolecular hydrogen bonds formed by N(11) and O(13).

N(15), C(16), O(17) and C(18) forming the exocyclic amide group are coplanar within 0·03 Å, and C(6) (*R* configuration) lies 0·10 Å out of this plane. The angle between the best planes through the β-lactam ring and the exocyclic amide is 58°, so that O(17) lies close to C(5) (3·14 Å) and S(1) (3·61 Å). This angle is slightly less than the corresponding angle in other C(3)-type penicillin derivatives (Domiano *et al.*, 1979) due to the intermolecular hydrogen bonds formed by N(15) and O(17).

In contrast to other phenoxypenicillins (Domiano *et al.*, 1979; Abrahamsson, Hodgkin & Maslen, 1963; Simon, Morin & Dahl, 1972; Chaney & Jones, 1973; Domiano, Nardelli, Balsamo, Macchia, Macchia & Meinardi, 1978; Sweet & Dahl, 1970), the conformation about C(16)–C(18) is almost synperiplanar and not anticlinal; the torsion angle O(17)–C(16)–C(18)–O(19) is 18°. However, such a difference in conformation of the side chain does not seem to be responsible for changing the activity against β-lactamase, because in penicillin *G* and procaine penicillin (both non-resistant) similar differences in conformation occur. The H atom at N(15) is engaged in an intermolecular hydrogen bond and not an intramolecular one with O(19) as in most phenoxypenicillin derivatives. The whole phenoxyacetamido substituent is almost planar, the torsion angles about the four successive bonds from N(15) to C(20) differing from 180° by less than 13°.

There are two types of N–H···O hydrogen bonds forming a three-dimensional net in the crystal (Table 5). One hydrogen bond exists between N(11)–H(111) and O(17) of the molecule related by the 2_1^z axis, the second

between N(15)—H(151) and O(14) of the molecule related by the 2_1^y axis. Thus, each molecule is hydrogen bonded to four neighbouring molecules, two related by 2_1^z (part of the helix along **z**) and two others related by 2_1^y (part of the helix along **y**). The shortest non-bonded intra- and intermolecular contacts are listed in Table 4.

The results described above indicate that the conformation of the investigated compound is rather typical for normal penicillins. The high resistance of the ureidopenicillin to β-lactamase does not seem to be due to steric hindrance in the vicinity of the β-lactam ring but is probably caused by the modification of chemical properties of the compound and its affinity with the enzyme. The introduction of the ureido in place of the carboxy group makes the molecule a substrate for transpeptidase but no longer a substrate for β-lactamase.

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A Disordered Ring in the Molecular Structure of Cyclopentanecarboxamide (CYCLAM)

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Abstract

$C_6H_{11}NO$, $M_r = 113{\cdot}2$, is monoclinic, space group $C2/c$ with $a = 22{\cdot}13\,(1)$, $b = 6{\cdot}392\,(5)$, $c = 9{\cdot}474\,(5)$ Å, $\beta = 100{\cdot}12\,(1)°$, $V = 1319\,(1)$ Å^3, $D_c = 1{\cdot}137\,(1)$, $D_x = 1{\cdot}10\,(5)$ Mg m^{-3}, $Z = 8$. Final $R = 0{\cdot}089$ for 925 observed reflections. The cyclopentane ring is disordered; one of the C atoms exists in two alternative positions leading to two possible conformations, both of which are approximately of the envelope type.

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Introduction

Several authors have compared the ring conformation of proline with the stable conformations of cyclopentane. A better model for proline would be cyclo-