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N-{(1*R**,5*S**,7*R**)-6-Oxobicyclo[3.2.0]hept-2-en-7-yl}phthalimide, $C_{15}H_{11}NO_3$

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Abstract. $M_r = 253\cdot25$, orthorhombic, *Pcab*, $a = 7\cdot434$ (1), $b = 13\cdot163$ (2), $c = 24\cdot684$ (4) Å, $Z = 8$, $V = 2415\cdot3$ Å^3, $D_x = 1\cdot39$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1\cdot5418$ Å, $\mu = 0\cdot716$ mm^{-1}, $F(000) = 1056$, $T = 293$ K, $R = 0\cdot082$ for 1300 unique observed reflections $[F_o \geq 3\sigma(F_o)]$. The cyclobutanone and cyclopentene rings of the bicyclohept-2-en-6-one moiety are *cis* fused; the phthalimide system is essentially planar. Bond lengths and angles are normal.

Introduction. The β-lactam antibiotics such as penicillin are thought to owe their action to their ability to inhibit the *trans*-peptidase enzyme necessary for bacterial wall synthesis (Ghuysen, Frère & Leyh-Boille, 1981). Bacterial resistance to β-lactam antibiotics is primarily due to the ability of the bacteria to make the enzyme β-lactamase which catalyses the hydrolysis of the β-lactam ring (Charnas & Knowles, 1981; Orlek, Sammes, Knott-Hunziker & Waley, 1979).

We have suggested (Gensmantel, McLellan, Morris, Page, Proctor & Randhawa, 1981; Proctor, Gensmantel & Page, 1982; Page, 1984) that the β-lactam ring is not an essential feature required for the drug to exhibit antibacterial activity. A potential class of new inhibitors of both classes of enzymes are therefore the carbocyclic analogues of penicillin in which the β-lactam ring is replaced by a cyclobutanone. We report here details of the structure of such a carbocyclic analogue of penicillin.

Experimental. The material was prepared by M. I. Page. Tabular crystal $0\cdot44 \times 0\cdot18 \times 0\cdot09$ mm. Enraf–Nonius CAD-4F diffractometer. Non-standard setting of space group *Pbca* used [equivalent positions $\pm(x, y, z; \frac{1}{2}-x, y, \frac{1}{2}+z; \frac{1}{2}+x, \frac{1}{2}-y, z; x, \frac{1}{2}+y, \frac{1}{2}-z)$]. No correction for absorption. $2\theta_{\max} = 120°$, h 0 to 8, k −14 to 14, l −27 to 27; 7804 reflections measured. Check reflection: average count 238, calculated σ (of the distribution) = 17·6. Cell dimensions from θ measurements of 48 reflections. Data merged using *SHELX* (Sheldrick, 1976) giving 1673 unique reflections, 1300 considered observed $[F_o \geq 3\sigma(F_o)]$, merging $R_{\text{int}} = 0\cdot0532$. *MULTAN*80 (Main *et al.*, 1980) used to solve the structure. Least-squares refinement with *SHELX*76; positional parameters of all atoms and anisotropic thermal parameters for non-H atoms refined; $\sum w(\Delta F)^2$ minimized; unit weights. H atoms from difference Fourier syntheses. In the final cycle max. Δ/σ 0·450 (H) and 0·142 (non-H), average 0·035. $\Delta\rho$ final difference Fourier map within +0·46 and −0·53 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Anisotropic scaling factor (Shakked & Rabinovich, 1977) applied. $R = 0\cdot082$ for 1300 observed reflections. [*]

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Discussion. The molecule and numbering scheme are shown in Fig. 1, and a stereoscopic view of the cell packing is shown in Fig. 2. Atom coordinates and equivalent isotropic temperature factors are in Table 1* and bond lengths and angles in Table 2.

The five-membered ring containing N(1) is quite planar, and the two C—C bonds connected to the phenyl ring show considerable double-bond character [average C—C bond length 1·475 (11) Å].

The arrangement of the bonds of the C(9)—C(15) link is *cis*.

We thank the University of Leeds Computing Service for the provision of computing facilities.

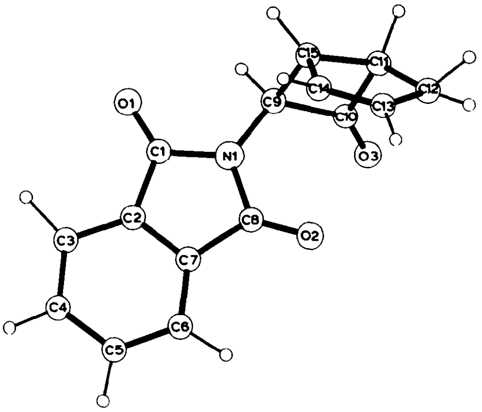

Fig. 1. View of the title compound with atom labels, drawn by the program *PLUTO*78 (Motherwell & Clegg, 1978).

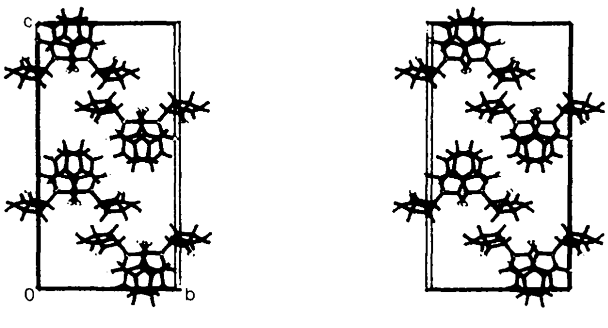

Fig. 2. Stereoscopic view of the crystal structure along **a**.

Table 1. *Atom coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2 ×10^4)

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	4803 (13)	−2276 (6)	1349 (3)	311
C(2)	5471 (12)	−2678 (6)	831 (3)	279
C(3)	5734 (14)	−3674 (7)	662 (4)	406
C(4)	6378 (15)	−3803 (8)	137 (4)	507
C(5)	6757 (14)	−2984 (8)	−199 (4)	477
C(6)	6501 (14)	−1997 (8)	−26 (3)	385
C(7)	5870 (12)	−1860 (6)	497 (3)	273
C(8)	5508 (13)	−909 (6)	798 (3)	308
C(9)	4355 (14)	−539 (6)	1743 (3)	300
C(10)	3635 (14)	532 (7)	1610 (3)	376
C(11)	4853 (14)	1036 (6)	2025 (3)	346
C(12)	6256 (19)	1768 (8)	1810 (5)	618
C(13)	7872 (15)	1094 (8)	1691 (4)	483
C(14)	7624 (15)	140 (7)	1841 (3)	392
C(15)	5798 (14)	−18 (6)	2095 (3)	318
O(1)	4287 (11)	−2718 (4)	1753 (2)	487
O(2)	5695 (9)	−32 (4)	664 (2)	406
O(3)	2511 (11)	848 (5)	1297 (3)	581
N(1)	4863 (10)	−1216 (5)	1305 (2)	273

Table 2. *Bond distances* (Å) *and angles* (°)

C(1)—C(2)	1·470 (11)	C(8)—N(1)	1·401 (10)
C(1)—N(1)	1·400 (10)	N(1)—C(9)	1·450 (10)
C(1)—O(1)	1·216 (9)	C(9)—C(10)	1·544 (12)
C(2)—C(3)	1·390 (12)	C(9)—C(15)	1·542 (12)
C(2)—C(7)	1·388 (11)	C(10)—O(3)	1·210 (11)
C(3)—C(4)	1·391 (13)	C(10)—C(11)	1·519 (13)
C(4)—C(5)	1·389 (15)	C(11)—C(12)	1·516 (14)
C(5)—C(6)	1·381 (13)	C(11)—C(15)	1·565 (12)
C(6)—C(7)	1·386 (11)	C(12)—C(13)	1·522 (16)
C(7)—C(8)	1·480 (11)	C(13)—C(14)	1·323 (13)
C(8)—O(2)	1·209 (10)	C(14)—C(15)	1·510 (14)
N(1)—C(1)—C(2)	106·4 (6)	C(1)—N(1)—C(9)	123·2 (6)
N(1)—C(1)—O(1)	123·3 (7)	C(8)—N(1)—C(9)	125·3 (6)
O(1)—C(1)—C(2)	130·4 (7)	N(1)—C(9)—C(10)	119·6 (6)
C(1)—C(2)—C(3)	130·4 (7)	N(1)—C(9)—C(15)	120·9 (8)
C(1)—C(2)—C(7)	108·0 (7)	C(10)—C(9)—C(15)	87·4 (6)
C(3)—C(2)—C(7)	121·6 (8)	C(9)—C(10)—C(11)	92·8 (7)
C(2)—C(3)—C(4)	116·2 (9)	C(9)—C(10)—O(3)	133·5 (8)
C(3)—C(4)—C(5)	122·1 (9)	O(3)—C(10)—C(11)	133·7 (8)
C(4)—C(5)—C(6)	121·2 (8)	C(10)—C(11)—C(12)	116·9 (8)
C(5)—C(6)—C(7)	117·2 (9)	C(10)—C(11)—C(15)	87·5 (6)
C(6)—C(7)—C(8)	129·8 (8)	C(12)—C(11)—C(15)	107·1 (8)
C(6)—C(7)—C(2)	121·6 (8)	C(11)—C(12)—C(13)	103·9 (8)
C(2)—C(7)—C(8)	108·6 (6)	C(12)—C(13)—C(14)	112·9 (10)
C(7)—C(8)—N(1)	105·5 (6)	C(13)—C(14)—C(15)	111·9 (9)
C(7)—C(8)—O(2)	130·5 (8)	C(14)—C(15)—C(9)	116·9 (7)
N(1)—C(8)—O(2)	124·0 (7)	C(14)—C(15)—C(11)	103·6 (7)
C(1)—N(1)—C(8)	111·5 (6)	C(9)—C(15)—C(11)	91·1 (7)

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39303 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Configuration Absolue du *p*-Bromobenzoyl-1 Perhydroindolecarboxylate-2 d'Ethyle-(2*S*,3a*S*,7a*S*) Hydraté, $C_{18}H_{22}BrNO_3.H_2O$

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Abstract. $M_r = 398\cdot3$, tetragonal, $P4_3$, $a = b = 14\cdot067\,(6)$, $c = 9\cdot654\,(3)$ Å, $V = 1910\,(2)$ Å^3, $Z = 4$, $D_x = 1\cdot385$ g cm^{-3}, Mo $K\alpha$, $\bar{\lambda} = 0\cdot71069$ Å, $\mu = 21\cdot16$ cm^{-1}, $F(000) = 824$, room temperature, final $R = 0\cdot068$ for 896 observed reflexions. The absolute configuration is 2*S*,3a*S*,7a*S*. Each water molecule is linked by three hydrogen bonds; two with other water molecules and one with the oxygen of the benzoyl group. The chain formed by the water molecules is wrapped around the fourfold axis. The five-membered ring has an envelope conformation.

Introduction. De nouveaux dérivés du perhydroindole présentant un fort pouvoir inhibiteur vis à vis de l'enzyme de conversion de l'angiotensine ont été synthétisés par Vincent, Remond, Portevin, Serkiz & Laubie (1982). Il s'est avéré nécessaire d'effectuer une étude structurale aux rayons X afin de confirmer la configuration absolue de ces composés. Dans ce but Vincent a préparé le dérivé bromobenzoylé du perhydroindolecarboxylate-2 d'éthyle.

Partie expérimentale. Parallélépipèdes translucides 0,60 × 0,20 × 0,15 mm, CAD-4 Enraf–Nonius, Mo $K\alpha$ avec monochromateur, $\theta < 23°$. 9 réflexions pour déterminer les paramètres de maille. Pas de variation des 3 étalons, 1413 réflexions indépendantes dont 896 avec $I > 3\sigma(I)$, corrections de Lorentz et de polarisation, pas de correction de l'absorption. $-9 \le h \le 10$, $0 \le k \le 15$, $0 \le l \le 10$. Section de Patterson pour localiser l'atome de brome puis Fourier successifs. Affinements des paramètres en matrice complète à partir des F_o avec des facteurs d'agitation thermique anisotrope (*SHELX*; Sheldrick, 1976). Pour les hydrogènes distances C—H fixées à 1,08 Å, et même B isotrope affiné à la valeur de 6,9 Å^2. La molécule d'eau a été trouvée sur une série différence. En fin d'affinement $R = 0{,}068$, $R_w = 0{,}066$ avec $w = 1\cdot0$. La précision médiocre sur les paramètres atomiques est due aux coefficients d'agitation thermiques élevés. Max. $\Delta/\sigma = 0{,}09$; série différence finale: $\Delta\rho \le |0{,}3|$ e Å^{-3}.* Origine des facteurs de diffusion: *SHELX*. Ordinateur IRIS 80 de l'Université Pierre et Marie Curie.

Discussion. Les coordonées atomiques sont données dans le Tableau 1 et les longueurs de liaisons et angles de valence dans le Tableau 2.

Grâce à la présence du brome la configuration absolue a été déterminée d'une part à partir de R (0,072 en $P4_1$ et 0,068 en $P4_3$) et d'autre part en comparant 19 facteurs de structure de paires de Bijvoet: 13 sont en faveur de $P4_3$. Les trois atomes H(2), H(3a), H(7a) liés aux trois carbones asymétriques C(2), C(3a), C(7a) sont du même côté du cycle à cinq. Il en résulte une configuration 2*S*,3a*S*,7a*S* comme l'étude des dérivés non substitués par RMN l'avait suggéré (Fig. 1). Le cycle à cinq atomes a une forme enveloppe avec N,

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* Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope et des coordonnées des atomes d'hydrogène ont été déposées au dépôt d'archives de la British Library Lending Division (Supplementary Publication No. SUP 39316: 7 pp.). On peut en obtenir des copies en s'adressant à: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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