The molecular structure of BN 52021 provides a rigid model which can serve as reference for comparison of PAF–acether and specific antagonist structures.

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Structure of an Intermediate in the Synthesis of C_{20}-Gibberellins

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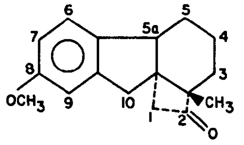
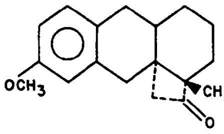
Abstract. 8-Methoxy-2aβ-methyl-2a,3,4,5,5aβ,10-hexahydro-1*H*-cyclobuta[*j*]fluoren-2-one, $C_{17}H_{20}O_2$, $M_r = 256{\cdot}34$, orthorhombic, *Pbca*, $a = 7{\cdot}757\ (2)$, $b = 17{\cdot}005\ (3)$, $c = 21{\cdot}321\ (7)$ Å, $V = 2812{\cdot}40\ (13)$ Å^3, $Z = 8$, $D_x = 1{\cdot}210$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 6{\cdot}21$ cm^{-1}, $F(000) = 1104$, $T = 293$ K, $R = 0{\cdot}041$ for 1669 reflections. The aromatic ring is planar and the cyclohexane ring adopts a distorted chair conformation. The five-membered ring is in an envelope conformation and the cyclobutanone ring has an angle of 157·0° between the planes through C(9), C(17), C(16) and C(9), C(14), C(16). Both the ring junctions *B–C* and *C–D* are *cis*-fused.

Introduction. The title compound (I), an important intermediate in the total synthesis of C_{20}-gibberellins (Ghatak, Ghosh & Sanyal, 1980), was obtained by stereoselective catalytic hydrogenation of unsaturated hydrocyclobutafluorenone *via* an acid-induced decomposition of a rigid polycyclic $\beta\gamma$-unsaturated diazomethyl ketone. The structure determination of this highly rigid polycyclic ketone (I) was undertaken in order to confirm its structure and stereochemistry which had been assigned by analogy with the corresponding hydrophenanthrene (II) of established stereochemistry.



(I)



(II)

Experimental. Transparent colourless plate-like crystal obtained by crystallization from petroleum ether solution, crystal dimensions: 0·2 × 0·3 × 0·3 mm. Syntex $P2_1$ four-circle diffractometer, unit-cell parameters by least-squares calculations from angular setting of 15 independent reflections, $9 < \theta < 16°$. 1885 unique reflections measured, $2\theta \le 114°$, $8 \ge h \ge 0$, $18 \ge k \ge 0$, $23 \ge l \ge 0$, graphite-monochromatized Cu $K\alpha$ radiation, ω-scan technique, no

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absorption correction, 1669 observed reflections ($F_o \geq 4\sigma F_o$), observed systematic absences indicated *Pbca*. Intensity of one standard reflection measured after every 50 reflections, no significant variation of intensity. Structure solved with *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), some H atoms located from difference Fourier map and the rest generated, isotropic and anisotropic refinement of H and non-H atoms, respectively, by block-diagonal least-squares method gave final $R = 0{\cdot}041$ and $wR = 0{\cdot}045$, function $\sum w||F_o| - |F_c||^2$ minimized with $w = 1/\sigma^2(F)$, $\sigma(F)$ standard deviation in the observed amplitude derived from counting statistics, $S = 2{\cdot}52$, maximum shift/e.s.d. = 0·8, maximum height in final difference Fourier map 0·27 and largest hole $-0{\cdot}030$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974), programmes used from *XRAY ARC* (Vickery, Bright & Mallinson, 1973), modified for the B6700 computer.

Table 1. *Final atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic thermal parameters for non-*H *atoms*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
O(1)	0·5893 (2)	0·1700 (1)	0·1640 (1)	6·16
O(2)	0·9188 (2)	0·1349 (1)	−0·2488 (1)	6·45
C(3)	0·7240 (2)	0·1230 (1)	0·0065 (1)	4·01
C(4)	0·8817 (2)	0·1611 (1)	0·0013 (1)	4·29
C(5)	0·9422 (3)	0·2044 (1)	0·0517 (1)	5·08
C(6)	0·8471 (3)	0·2090 (1)	0·1067 (1)	5·01
C(7)	0·6915 (3)	0·1697 (1)	0·1113 (1)	4·59
C(8)	0·6290 (2)	0·1276 (1)	0·0608 (1)	4·50
C(9)	0·8789 (3)	0·0500 (1)	−0·1542 (1)	4·73
C(10)	1·0566 (3)	0·0125 (1)	−0·1492 (1)	6·13
C(11)	1·1895 (3)	0·0642 (1)	−0·1175 (1)	6·12
C(12)	1·1253 (3)	0·0909 (1)	−0·0544 (1)	5·64
C(13)	0·9669 (2)	0·1439 (1)	−0·0608 (1)	4·45
C(14)	0·8186 (2)	0·1069 (1)	−0·1003 (1)	4·18
C(15)	0·6863 (2)	0·0777 (1)	−0·0520 (1)	4·58
C(16)	0·7534 (3)	0·1657 (1)	−0·1512 (1)	4·78
C(17)	0·8604 (3)	0·1202 (1)	−0·1980 (1)	4·84
C(18)	0·7417 (3)	−0·0107 (1)	−0·1719 (1)	6·47
C(19)	0·6597 (3)	0·1992 (2)	0·2203 (1)	6·56

Table 2. *Intramolecular bond lengths* (Å) *and angles* (°) *with their e.s.d.'s in parentheses*

O(1)–C(7)	1·376 (3)	O(1)–C(19)	1·409 (3)
O(2)–C(17)	1·201 (3)	C(3)–C(4)	1·388 (3)
C(3)–C(8)	1·374 (3)	C(3)–C(15)	1·495 (3)
C(4)–C(5)	1·385 (3)	C(4)–C(13)	1·508 (3)
C(5)–C(6)	1·388 (3)	C(6)–C(7)	1·383 (3)
C(7)–C(8)	1·382 (3)	C(9)–C(10)	1·522 (3)
C(9)–C(14)	1·574 (3)	C(9)–C(17)	1·521 (3)
C(9)–C(18)	1·530 (3)	C(10)–C(11)	1·514 (3)
C(11)–C(12)	1·505 (3)	C(12)–C(13)	1·531 (3)
C(13)–C(14)	1·559 (3)	C(14)–C(15)	1·535 (3)
C(14)–C(16)	1·560 (3)	C(16)–C(17)	1·511 (3)
C(7)–O(1)–C(19)	118·3 (2)	C(4)–C(3)–C(8)	120·9 (2)
C(4)–C(3)–C(15)	110·3 (2)	C(8)–C(3)–C(15)	128·8 (2)
C(3)–C(4)–C(5)	119·0 (2)	C(3)–C(4)–C(13)	111·5 (2)
C(5)–C(4)–C(13)	129·4 (2)	C(4)–C(5)–C(6)	120·4 (2)
C(5)–C(6)–C(7)	119·8 (2)	O(1)–C(7)–C(6)	124·0 (2)
O(1)–C(7)–C(8)	115·9 (2)	C(6)–C(7)–C(8)	120·1 (2)
C(3)–C(8)–C(7)	119·9 (2)	C(10)–C(9)–C(14)	118·4 (2)
C(10)–C(9)–C(17)	117·2 (2)	C(10)–C(9)–C(18)	111·4 (2)
C(14)–C(9)–C(17)	86·5 (1)	C(14)–C(9)–C(18)	112·8 (2)
C(17)–C(9)–C(18)	108·2 (2)	C(9)–C(10)–C(11)	113·9 (2)
C(10)–C(11)–C(12)	110·3 (2)	C(11)–C(12)–C(13)	111·3 (2)
C(4)–C(13)–C(12)	112·8 (2)	C(4)–C(13)–C(14)	103·2 (1)
C(12)–C(13)–C(14)	113·7 (3)	C(9)–C(14)–C(13)	115·1 (2)
C(9)–C(14)–C(15)	119·3 (2)	C(9)–C(14)–C(16)	88·9 (1)
C(13)–C(14)–C(15)	105·2 (1)	C(13)–C(14)–C(16)	110·9 (2)
C(15)–C(14)–C(16)	117·2 (2)	C(3)–C(15)–C(14)	105·2 (2)
C(14)–C(16)–C(17)	87·3 (1)	O(2)–C(17)–C(9)	132·9 (2)
O(2)–C(17)–C(16)	134·1 (2)	C(9)–C(17)–C(16)	99·8 (2)

Discussion. Atomic parameters are given in Table 1, bond lengths and angles in Table 2.* The molecular structure projected normal to the *b* axis showing the atomic numbering is given in Fig. 1. The aromatic ring (*A*) adopts a planar conformation whereas the cyclohexane ring (*C*) has a distorted chair conformation as atoms C(12) and C(9) are displaced to opposite sides of the least-squares plane, through the remaining four atoms, by −0·65 and 0·31 Å, respectively. The five-membered ring (*B*) is in an envelope conformation with the flap at C(14), as this is 0·13 Å out of the least-squares plane through all five atoms of the ring. The cyclobutanone ring is not planar but has a dihedral angle of 157·0° between the planes through C(9), C(17), C(16) and C(9), C(14), C(16). Both the cyclobutanone (*D*) and cyclopentane (*B*) rings are *cis*-fused to the cyclohexane ring (*C*) by C(9)–C(14) and C(13)–C(14). All the bond lengths and angles in this molecule are normal and no unusual intermolecular contacts exist.

The authors offer sincere thanks to Professor U. R. Ghatak of IACS, Calcutta, for his helpful discussion on the conformation of the title compound which was also synthesized in his laboratory and to Dr S. Ray of the same institute for his help in solving some of the computational problems.

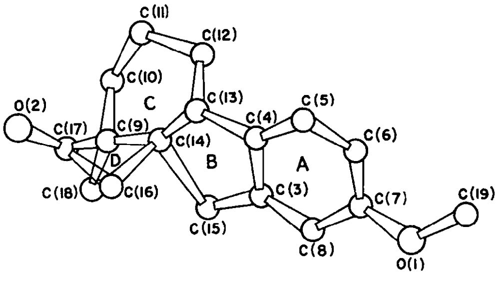

Fig. 1. View of the molecule projected along the *b* axis.

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* Lists of structure factors, anisotropic thermal parameters of non-H atoms, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43143 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Benzyl-D-gluconamide

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(*Reçu le* 6 *décembre* 1985, *accepté le* 12 *mai* 1986)

Abstract. $C_{13}H_{19}NO_6$, $M_r = 285{\cdot}3$, triclinic, $P1$, $a = 4{\cdot}820\,(2)$, $b = 5{\cdot}083\,(3)$, $c = 14{\cdot}334\,(6)$ Å, $\alpha = 96{\cdot}91\,(4)$, $\beta = 83{\cdot}10\,(3)$, $\gamma = 97{\cdot}12\,(4)°$, $V = 344{\cdot}0\,(8)$ Å^3, $Z = 1$, $D_x = 1{\cdot}378\,(3)$, $D_m = 1{\cdot}38\,(1)$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·7107 Å, $\mu = 0{\cdot}069$ mm^{-1}, $F(000) = 152$, $T = 293$ K, final $R = 0{\cdot}049$ for 1270 independent observed reflections. Six hydrogen bonds link the molecules to form the crystal. In common with related compounds, the gluconamide part of the molecule has a linear conformation with its long axis parallel to [100]. Comparison with related molecules supports the hypothesis that the length of the hydrophobic chain is related to the sweetness or bitterness of the compound.

Introduction. Ce travail s'inscrit le cadre d'un ensemble de recherches concernant la synthèse chimique, les propriétés conformationnelles et le pouvoir sucrant ou amer d'un certain nombre de gluconamides.

La structure du *N*-benzyl-D-gluconamide montre l'existence de nombreuses liaisons hydrogène. Une étude comparative succincte est faite avec le *N*-cyclohexyl-D-gluconamide (Darbon, Oddon, Lacombe, Decoster, Pavia & Reboul, 1984) ainsi qu'avec les *N*-isopropyl-D-gluconamide et *N*,*N*'-diéthyl-D-gluconamide (Darbon-Meyssonnier, Oddon, Decoster, Pavia, Pepe & Reboul, 1985) et le *N*-(chloro-2 éthyl)-D-gluconamide (Satzke & Mackay, 1975; Sindt & Mackay, 1977).

Partie expérimentale. Préparation suivant la méthode décrite par Ishikawa (1964). Recristallisation dans l'eau par évaporation lente, cristaux incolores sous forme de plaquettes. Masse volumique mesurée par flottaison du cristal dans un mélange hexane/CCl_4. Cristal taillé approximativement sous forme de cube de 0,18 mm. Mesures sur diffractomètre Nonius CAD-4 avec θ compris entre 4 et 33°, radiation Mo $K\bar{\alpha}$, monochromateur de graphite, enregistrement des réflexions avec $(\sin\theta)_{\max}/\lambda = 0{,}766$ Å^{-1} en balayage $\omega/2\theta$, une vitesse en ω de 2° min^{-1} et une amplitude $\Delta\theta = (1{,}6 + 0{,}5\ \mathrm{tg}\ \theta)°$. Paramètres des mailles affinés sur le diffractomètre à l'aide de 25 réflexions indépendantes. 2672 réflexions enregistrées avec $h = -7$ à 7, $k = -7$ à 7 et $l = 0$ à 21. Trois réflexions de référence (variation $\leq 3\%$) contrôlées toutes les 75 réflexions. 1045 réflexions indépendantes non observées, 1270 réflexions indépendantes avec $I > 2{,}5\sigma(I)$. Correction de Lorentz

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