Data collection: *XSCANS* (Siemens 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXTL-Plus*.

The author thanks the CWRU Chemistry Department for support of this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1198). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cicero, R. L., Zhao, D. & Protasiewicz, J. D. (1996). *Inorg. Chem.* **35**, 275–276.
Mishra, A. K., Olmstead, M. M., Ellison, J. J. & Power, P. P. (1995). *Inorg. Chem.* **34**, 3210–3214.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Sheldrick, G. M. (1994). *SHELXTL-Plus*. Release 5.01. PC version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994). XSCANS. *X-ray Single Crystal Analysis System*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Yamada, Y., Yamamoto, T. & Okawara, M. (1975). *Chem. Lett.* pp. 361–362.

Acta Cryst. (1996). C**52**, 1572–1574

Clemizole

MASOOD PARVEZ* AND AALIYA P. SABIR

Department of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4. E-mail: parvez@acs.ucalgary.ca

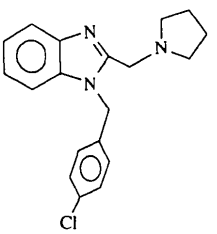
(Received 11 December 1995; accepted 16 January 1996)

Abstract

The crystal structure of clemizole [1-(4-chlorobenzyl)-2-(1-pyrrolidinylmethyl)-1*H*-benzimidazole, $C_{19}H_{20}ClN_3$] is composed of independent molecules of the free base having normal molecular dimensions and no unusual contacts shorter than van der Waals distances.

Comment

The crystal structure of clemizole hydrochloride has been reported previously (Parvez, 1996). We have now separated the free base from its hydrochloride salt and have grown crystals suitable for data collection by the X-ray diffraction method. In the present paper, we report the crystal structure of the free base, clemizole, (I), which is an anti-allergic drug effective on H1 receptors.



(I)

An *ORTEP*II (Johnson, 1976) drawing of clemizole with the atomic numbering scheme is shown in Fig. 1. The molecular dimensions are unexceptional with distances C_{sp^2}—Cl 1.755 (5), C=N 1.328 (5) and C_{sp^3}—C_{sp^2} 1.492 (6) Å, and mean N—C_{sp^3} 1.462 (12), N—C_{sp^2} 1.384 (12), C_{sp^3}—C_{sp^3} 1.517 (7) and C—$C_{aromatic}$ 1.380 (14) Å.

The benzimidazole system and phenyl ring are essentially individually planar with maximum deviations from the respective least-squares planes of 0.061 (5) and 0.012 (6) Å; these planes are inclined at an angle of

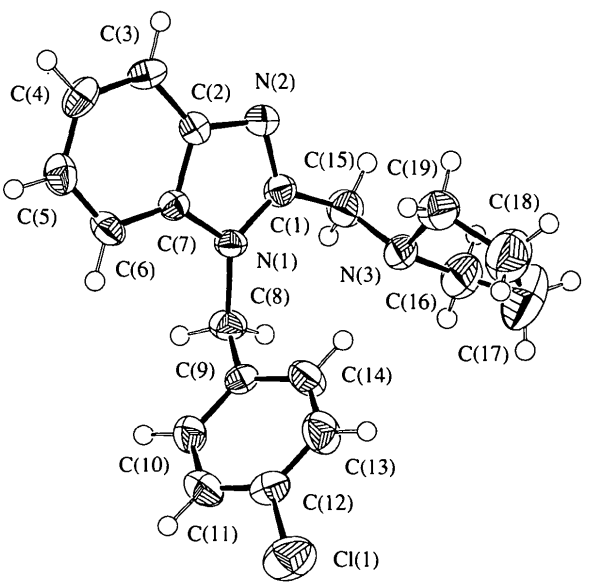


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms have been assigned arbitrary radii.

68.8 (6)° with respect to each other. The corresponding angle in the crystal structure of clemizole hydrochloride was 82.8 (9)° (Parvez, 1996). The lack of hydrochloride in the free base appears to be responsible for this drastic conformational difference between the two structures and results in the orientation of atom H(14) of the phenyl ring towards the lone pair of electrons of the pyrrolidinyl N(3) atom, with an N···H interaction distance of 2.67 Å. The pyrrolidinyl ring has an N(3)-envelope conformation, with atom N(3) 0.578 (7) Å out of the plane of the remaining four C atoms; a similar conformation of the five-membered ring was observed in the crystal structure of clemizole hydrochloride (Parvez, 1996).

Experimental

An aqueous solution of clemizole hydrochloride (Sigma Inc.) was treated with an aqueous solution of NaOH. The free base was extracted with *n*-hexane, dried over $MgSO_4$ and allowed to crystallize at room temperature.

Crystal data

$C_{19}H_{20}ClN_3$
M_r = 325.84
Monoclinic
$P2_1/a$
a = 11.105 (2) Å
b = 8.978 (2) Å
c = 17.219 (1) Å
β = 90.13 (1)°
V = 1716.7 (4) Å^3
Z = 4
D_x = 1.261 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 10–18°
μ = 0.225 mm^{-1}
T = 296 K
Prism
0.54 × 0.53 × 0.26 mm
Colourless

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction:
ψ scan of 3 reflections (North, Phillips & Mathews, 1968)
T_{min} = 0.95, T_{max} = 1.00
3436 measured reflections
3259 independent reflections

1481 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.041
θ_{max} = 25°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 10$
$l = -19 \rightarrow 19$
3 standard reflections monitored every 200 reflections
intensity decay: 0.64%

Refinement

Refinement on F
R = 0.0473
wR = 0.0472
S = 2.825
1481 reflections
208 parameters
H atoms geometrically idealized with C—H 0.95 Å

$w = 1/[\sigma^2(F_o) + 0.008(F_o^2)]$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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)*

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

	x	y	z	U_{eq}
Cl(1)	0.1667 (1)	0.3700 (2)	0.9342 (1)	0.079 (1)
N(1)	0.4470 (3)	−0.0469 (4)	0.6547 (2)	0.033 (1)
N(2)	0.6277 (3)	−0.1480 (5)	0.6285 (2)	0.039 (1)
N(3)	0.4665 (4)	−0.2927 (5)	0.7827 (3)	0.043 (1)
C(1)	0.5211 (5)	−0.1678 (5)	0.6618 (3)	0.037 (2)
C(2)	0.6214 (4)	−0.0068 (5)	0.5949 (3)	0.032 (2)
C(3)	0.7028 (4)	0.0699 (6)	0.5480 (3)	0.038 (2)
C(4)	0.6687 (5)	0.2031 (6)	0.5169 (3)	0.045 (2)
C(5)	0.5558 (5)	0.2661 (6)	0.5326 (3)	0.046 (2)
C(6)	0.4748 (4)	0.1943 (6)	0.5802 (3)	0.041 (2)
C(7)	0.5077 (4)	0.0577 (5)	0.6100 (3)	0.031 (2)
C(8)	0.3185 (4)	−0.0359 (6)	0.6758 (3)	0.042 (2)
C(9)	0.2895 (4)	0.0720 (6)	0.7389 (3)	0.033 (2)
C(10)	0.2055 (4)	0.1815 (6)	0.7262 (3)	0.047 (2)
C(11)	0.1683 (5)	0.2733 (6)	0.7851 (4)	0.056 (2)
C(12)	0.2158 (5)	0.2559 (6)	0.8578 (3)	0.046 (2)
C(13)	0.3026 (5)	0.1497 (7)	0.8713 (3)	0.058 (2)
C(14)	0.3379 (4)	0.0575 (6)	0.8119 (3)	0.049 (2)
C(15)	0.4840 (4)	−0.3096 (6)	0.7001 (3)	0.047 (2)
C(16)	0.4190 (5)	−0.4288 (7)	0.8180 (4)	0.070 (2)
C(17)	0.4450 (7)	−0.4095 (9)	0.9035 (4)	0.110 (3)
C(18)	0.5472 (6)	−0.2965 (8)	0.9092 (4)	0.092 (3)
C(19)	0.5780 (5)	−0.2622 (7)	0.8253 (3)	0.059 (2)

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

Cl(1)—C(12)	1.755 (5)	N(3)—C(16)	1.464 (6)
N(1)—C(1)	1.367 (5)	N(3)—C(19)	1.464 (6)
N(1)—C(7)	1.389 (5)	C(1)—C(15)	1.492 (6)
N(1)—C(8)	1.477 (5)	C(8)—C(9)	1.492 (6)
N(2)—C(1)	1.328 (5)	C(16)—C(17)	1.509 (8)
N(2)—C(2)	1.395 (6)	C(17)—C(18)	1.526 (8)
N(3)—C(15)	1.443 (6)	C(18)—C(19)	1.516 (7)
C(1)—N(1)—C(7)	107.1 (4)	N(2)—C(2)—C(3)	131.0 (5)
C(1)—N(1)—C(8)	127.8 (4)	N(2)—C(2)—C(7)	109.9 (4)
C(7)—N(1)—C(8)	124.1 (4)	N(1)—C(7)—C(2)	105.0 (4)
C(1)—N(2)—C(2)	104.9 (4)	N(1)—C(7)—C(6)	132.8 (4)
C(15)—N(3)—C(16)	111.8 (5)	N(1)—C(8)—C(9)	115.6 (4)
C(15)—N(3)—C(19)	113.4 (4)	N(3)—C(15)—C(1)	112.6 (4)
C(16)—N(3)—C(19)	104.6 (4)	N(3)—C(16)—C(17)	104.0 (5)
N(1)—C(1)—N(2)	113.1 (4)	C(16)—C(17)—C(18)	106.2 (5)
N(1)—C(1)—C(15)	123.4 (4)	C(17)—C(18)—C(19)	104.1 (5)
N(2)—C(1)—C(15)	123.5 (5)	N(3)—C(19)—C(18)	104.3 (5)

The space group ($P2_1/a$) was determined uniquely from the systematic absences: $h0l$, $h = 2n + 1$; $0k0$, $k = 2n + 1$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

The authors thank the Natural Sciences and Engineering Research Council, Canada, for providing the diffractometer through an equipment grant to the University of Calgary, and the University of Calgary for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1153). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Fan, H.-F. (1991). *SAPI91. Structure Analysis Programs with Intelligent Control.* Rigaku Corporation, Tokyo, Japan.

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

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1994). *TEXSAN. Single Crystal Structure Analysis Software.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Parvez, M. (1996). *Acta Cryst.* C**52**, 904–905.

Acta Cryst. (1996). C**52**, 1574–1576

The New Pentacyclic Saponine Ecdysantherin [3β-Hydroxy-20-methylpregn-5,14-dien-16-one-(18–20)-lactone] from *Ecdysanthera rosea Hook. et Arn.* (*Apocynaceae*) of Vietnam

PETER LUGER,[a] MANUELA WEBER,[a] NGUYEN XUAN DUNG,[b] PHAM THANH KY[c] AND CHINH LE THE[c]

[a]*Institut für Kristallographie, Fachbereich Chemie der Freien Universität, Takustrasse 6, 14195 Berlin, Germany,* [b]*Centre for Education and Development of Chromatography, 3 Giai Phong Street, Hai Ba District, 10000 Hanoi, Vietnam, and* [c]*Hanoi College of Pharmacy, 13–15 Le Thanh, Hanoi, Vietnam. E-mail: luger@chemie.fu-berlin.de*

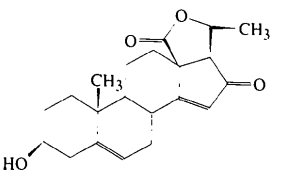
(*Received 28 November 1995; accepted 18 December 1995*)

Abstract

Ecdysantherin, 3β-hydroxy-20-methylpregn-5,14-dien-16-one-(18–20)-lactone, $C_{21}H_{26}O_4$, a new pentacyclic saponine, was isolated from the powder of *Ecdysanthera rosea Hook. et Arn.* (*Apocynaceae*) of Vietnam, and its structure was elucidated. It was found that ecdysantherin is a pentacyclic saponine molecule with three six-membered and two five-membered rings. An intermolecular OH···O hydrogen bond generates infinite chains of molecules in the z direction.

Comment

Ecdysanthera rosea Hook. et Arn. is a large climbing shrub scattered in forests of Vietnam, Taiwan and other countries (Ho, 1993; Chuyen, 1975; Ly, 1986; Huang, Sy & Lai, 1990). In Vietnamese folk-medicine, this plant is used as an anti-inflammatory and anti-hepatitic drug (Hsu & Chin, 1980). The fluid extract and the saponine extracted from the powder of the *Ecdysanthera rosea* plant have an anti-inflammatory (inhibiting bacteria) and a diuretic (to treat urinary stones) activity. We have not yet found any toxicity. Only malic acid, tartaric acid together with phytosterols and a new pentacyclic triterpene (Huang, Sy & Lai, 1990) have been isolated previously from this species, and their structures were elucidated on the basis of spectral data. In addition, the new pentacyclic saponine ecdysantherin, (I), has now been isolated from *Ecdysanthera rosea Hook. et Arn.* in Vietnam by column chromatography.



(I)

The sample was recrystallized from chloroform–ethanol, and needle-shaped colourless crystals were obtained. An X-ray analysis was carried out to establish its chemical identity and spatial geometry.

The molecular structure is shown in Fig. 1 along with the atom-numbering scheme. In the molecule, consisting mainly of the pentacyclic ring system *A–E*, two endocyclic double bonds, C5=C6 and C14=C15, were identified, indicated by the short bond lengths of 1.331 (7) and 1.340 (6) Å, respectively.

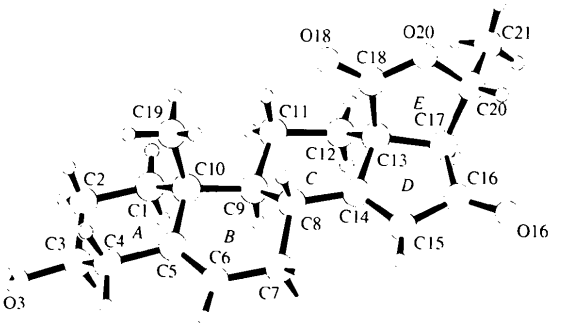


Fig. 1. *SCHAKAL88* (Keller, 1988) drawing of the molecular structure of ecdysantherin showing the numbering scheme.

The ring system is very similar to that of 20-epi-kibataline (Kutschabsky, Pfeiffer, Kretschmer & Adam, 1985; Ngoc, Kutschabsky, Phuong & Adam, 1984), a steroidal alkaloid also isolated from a Vietnamese plant; however, 20-epi-kibataline lacks the C16—O16 keto group and has a C14—C15 single bond. For this compound an *R* configuration was assigned to C20 on the basis of spectroscopic data (Ngoc *et al.*, 1984). It therefore seemed sensible for ecdysantherin, of which the absolute configuration was neither previously known