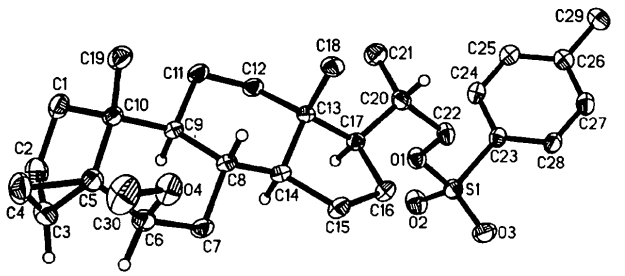


Fig. 1. The molecular structure of (I) showing the atom-numbering scheme. The H atoms of the tolyl ring and of the CH_3 and CH_2 groups have been omitted for clarity. Those remaining are shown as small spheres of arbitrary radii. Displacement ellipsoids are drawn at the 20% probability level.

Experimental

The title compound (m.p. 415–417 K) was synthesized according to Partridge, Faber & Uskokovic (1974).

Crystal data

$C_{30}H_{44}O_4S$
M_r = 500.71
Monoclinic
$P2_1$
a = 7.3890 (10) Å
b = 16.073 (3) Å
c = 12.161 (2) Å
β = 106.10 (3)°
V = 1387.6 (4) Å^3
Z = 2
D_x = 1.198 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 11.6–18.3°
μ = 0.149 mm^{-1}
T = 293 (2) K
Plate
0.30 × 0.30 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5301 measured reflections
4614 independent reflections
3058 reflections with $I > 2\sigma(I)$

R_{int} = 0.027
θ_{max} = 29.96°
$h = -10 \rightarrow 10$
$k = -22 \rightarrow 2$
$l = -2 \rightarrow 17$
2 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.119
S = 1.025
4608 reflections
491 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.06P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.008
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = 0.10 (8)

H atoms were located from the ΔF map; thereafter, they were freely refined with individual isotropic displacement parameters. Bond lengths and angles assume typical values, with uncertainties on C—C bonds in the range 0.003–0.007 Å.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994). Cell refinement: *CAD-4 EXPRESS*. Data reduction: *GX* (Mallinson & Muir, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Software used to prepare material for publication: *SHELXL93*.

DSY thanks the EPSRC for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1146). Services for accessing these data are described at the back of the journal.

References

Dasgupta, S. K., Crump, D. R. & Gut, M. (1974). *J. Org. Chem.* **39**, 1658–1662.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Mallinson, P. R. & Muir, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.
Partridge, J. J., Faber, S. & Uskokovic, M. R. (1974). *Helv. Chim. Acta*, **57**, 764–771.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Vanderah, D. J. & Djerassi, C. (1978). *J. Org. Chem.* **43**, 1442–1448.

Acta Cryst. (1997). C**53**, 982–984

6α,7β-Dihydroxyvouacapan-17β-oic Acid

Silvana Guilardi Ruggiero,[a] Bernardo Lages Rodrigues,[b] Nelson Goncalves Fernandes,[c] Guglielmo Marconi Stefani[c] and Dorila Pilo Veloso[c]

[a]*Departamento de Química, Universidade Federal de Uberlandia, Caixa Postal 593, 38400-902, Uberlandia MG, Brazil,* [b]*DQFM-IQSC-USP, Caixa Postal 780, 13560-970 São Carlos SP, Brazil, and* [c]*Departamento de Química, ICEX, Universidade Federal de Minas Gerais, Caixa Postal 702, 31270-901 Belo Horizonte MG, Brazil. E-mail: silvana@ufu.br*

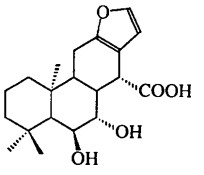
(*Received 29 January 1996; accepted 2 December 1996*)

Abstract

The title compound, 1,5,5a,6,7,7a,8,9,10,11,11a,11b-dodecahydro-6,7-dihydroxy-8,8,11a-trimethylphenanthro[3,2-*b*]furan-5-carboxylic acid, $C_{20}H_{28}O_5$, presents both anti-inflammatory and analgesic activities. Two of the six-membered rings adopt chair conformations, whereas the ring fused to furan is in a half-chair conformation. Crystal packing is established by three intermolecular hydrogen bonds.

Comment

In Brazilian folk medicine, the seed infusion of the Pterodon genus, a tree widely distributed in savannah, is reputedly useful in the treatment of throat infections; the oil has been described as a protection against *S. mansoni* penetration of the skin (Mors, Fascio, Monteiro, Gilbert & Pellegrino, 1967). Phytochemical studies of different species of this genus show they have a large number of furanditerpenes, one of which is the title compound, (I), isolated from *P. Polygalaeflorus Benth* as reported previously (Fascio *et al.*, 1976; Mahajan & Monteiro, 1973). Some of its derivatives have been reported as possessing anti-inflammatory and analgesic properties (Rubinger, Piló-Veloso, Stefani & Alves, 1991).



(I)

An *ORTEP* (Johnson, 1965) drawing of the title molecule, which has three six-membered rings and a furan ring, is shown in Fig. 1. According to puckering parameters θ and φ (Cremer & Pople, 1975), the ring fused to furan (θ = 129 and φ = 217°) is in a half-chair conformation, while rings C5–C10 (θ = 169 and φ = 173°) and C1–C5, C10 (θ = 170 and φ = 206°) adopt chair conformations.

The mean value of the distance between C atoms with sp^3 hybridization is 1.534 (7) Å for both chair-shaped six-membered rings. The furan ring with sp^2-hybridized C atoms has a mean C—C distance of 1.37 (4) Å.

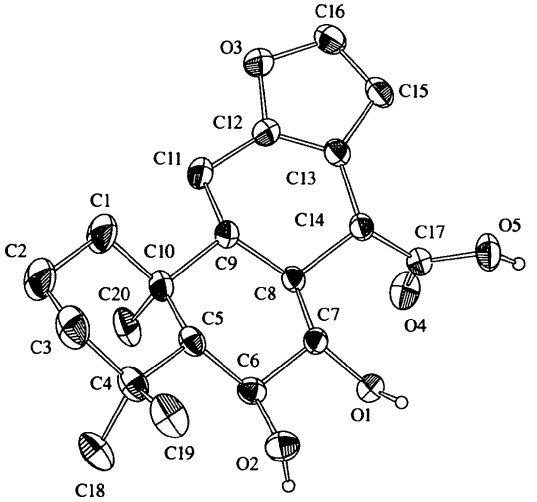


Fig. 1. View of the title compound with 50% probability ellipsoids and the atomic numbering scheme (only H atoms involved in hydrogen bonds are shown for clarity).

The C11—C12 and C13—C14 bonds are between sp^3- and sp^2-hybridized C atoms, and the average distance between them is 1.49 (2) Å.

As the refined Flack parameter and its standard deviation are of the same magnitude, only the relative stereochemistry of the molecule was determined.

The crystal packing (Table 1) involves three intermolecular hydrogen bonds which link the molecules in chains along the **a** and **b** directions.

Experimental

Suitable single crystals of the title compound were obtained by slow evaporation of an ethanol/water (1:1) solution.

Crystal data

$C_{20}H_{28}O_5$	Mo $K\alpha$ radiation
M_r = 348.42	λ = 0.71073 Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	θ = 2.32–26.32°
a = 8.293 (1) Å	μ = 0.091 mm^{-1}
b = 9.589 (2) Å	T = 293 (2) K
c = 22.698 (4) Å	Prism
V = 1805.0 (5) Å^3	0.4 × 0.3 × 0.2 mm
Z = 4	White
D_x = 1.282 Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 26.29°
$\omega/2\theta$ scans	$h = -10 \rightarrow 0$
Absorption correction: none	$k = 0 \rightarrow 11$
2112 measured reflections	$l = 0 \rightarrow 28$
2109 independent reflections	3 standard reflections
1643 reflections with $I > 2\sigma(I)$	frequency: 120 min
	intensity decay: −1.6%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = −0.001
$R(F)$ = 0.0492	$\Delta\rho_{max}$ = 0.200 e Å^{-3}
$wR(F^2)$ = 0.1357	$\Delta\rho_{min}$ = −0.195 e Å^{-3}
S = 1.215	Extinction correction: none
2101 reflections	Scattering factors from *International Tables for Crystallography* (Vol. C)
232 parameters	
H atoms refined as riding	Flack parameter for absolute configuration determination = 5 (3)
$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 1.0445P]$ where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D···A	D—H···A
O1—H10···O3^{i}	2.904 (4)	156 (6)
O5—H50···O1^{ii}	2.677 (4)	161 (2)
O2—H20···O4^{iii}	2.846 (5)	165 (1)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure:

SHELXL93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: MS-DOS 6.20 editor.

The authors thank the Instituto de Química de São Carlos for the data collection and CNPq, FINEP and FAPEMIG(Brazil) for financial support, and Dr Dalton L. F. Alves for helpful advice on the isolation of the title compound.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1165). Services for accessing these data are described at the back of the journal.

References

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Fascio, M., Mors, W. B., Gilbert, B., Mahajan, J. R., Monteiro, M. B., Santos Filho, D. & Vichnewski, W. (1976). *Phytochemistry*, **15**, 201–203.
Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft University Press.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Mahajan, J. R. & Monteiro, M. B. (1973). *J. Chem Soc. Perkin Trans.* pp. 520–524.
Mors, W. B., Fascio, M., Monteiro, H. J., Gilbert, B. & Pellegrino, J. (1967). *Science*, **157**, 950–951.
Rubinger, M. M. M., Piló-Veloso, D., Stefani, G. M. & Alves, D. L. F. (1991). *J. Brazil. Chem. Soc.* **2**, 124–128.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1997). C**53**, 984–985

N-Cyano-*N'*-(4-methoxyphenyl)guanidine

IAN D. CUNNINGHAM,[a] NAN CHI WAN,[a] DAVID C. POVEY,[a] GALLIENUS W. SMITH[a] AND BRIAN G. COX[b]

[a]*Department of Chemistry, University of Surrey, Guildford GU2 5XH, England, and* [b]*Zeneca Fine Chemicals Manufacturing Organization, Huddersfield HD2 1FF, England*

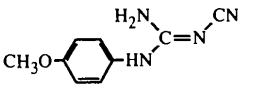
(*Received 14 September 1995; accepted 21 February 1997*)

Abstract

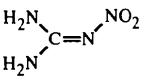
An X-ray structural analysis of the title compound, $C_9H_{10}N_4O$, shows a planar guanidine portion, the *N*-aryl group tilted *ca* 57° relative to the guanidine plane and *E* geometry about the formal 'imino' bond.

Comment

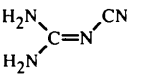
Monosubstituted guanidines such as nitroguanidine, (2), cyanoguanidine, (3), sulfaguanidine, (4), L-canavanine, (5), and *N*-methyl-*N'*-nitro-*N*-nitrosoguanidine, (6), which bear strongly electron-withdrawing groups, exist as neutral organic molecules preferring the 'imino' tautomeric form [*e.g.* $(NH_2)_2C{=}N{-}NO_2$] in solution and in the solid state (Patai & Rappoport, 1991). In a previous paper, we showed that disubstituted *N*-aryl-*N'*-cyanoguanidines in solution in Me_2SO also have the 'imino' structure seen for the title compound, (1) (Cunningham, Wan & Cox, 1994). While crystal structures have been reported for (3) (Hughes, 1940; Begley, Hubberstey & Moore, 1985; Hirshfield & Hope, 1980) and for the other guanidines listed above (Patai & Rappoport, 1991), none are known for *N*-cyano-*N'*-substituted guanidines. In this paper, we report the crystal structure of *N*-cyano-*N'*-(4-methoxyphenyl)-guanidine, (1).



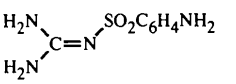
(1)



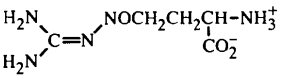
(2)



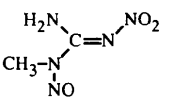
(3)



(4)



(5)



(6)

The X-ray crystal structure is shown in Fig. 1 (arbitrary numbering), where it can be seen that the imino tautomer is preferred in the solid state. The cyanoguanidine portion is essentially planar, as shown by the bond angles about C1, N1 and N2 (Table 1) and the C2—N3—C1—N1 and C2—N3—C1—N2 torsion angles. The N1—C1, N2—C1 and N3—C1 bonds which comprise the guanidine system are all of length 1.33 (1) Å; these lengths are almost identical to those seen for unsubstituted cyanoguanidine (Begley, Hubberstey & Moore, 1985) and are between the values for C—N and C=N (Allen *et al.*, 1987), indicating that the N-atom lone pairs and the C=N bond shown formally in (1) are completely delocalized over the guanidine system. The bond length (N4—C2) for the cyano group at 1.142 (3) Å is typical, suggesting that the cyano group is not involved in resonance with the guanidine system. Interestingly, the aryl group is twisted out of the guanidine plane by about 57° showing that resonance