Data were corrected for Lorentz and polarization factors and reduced to F_o^2 and $\sigma(F_o^2)$ using *XCAD*4 (Harms, 1993). The structure was determined by direct methods. After several cycles of refinement, all of the non-H atoms were refined anisotropically. An additional difference Fourier map revealed nearly all of the H-atom positions. Since a few were missing, all H atoms bonded to C atoms were initially placed in calculated positions. The H atoms during the final stages of refinement were allowed to refine freely. A final difference Fourier map was essentially featureless with the largest peaks, $|\Delta\rho| < 0.18$ e Å^{-3}.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *XCAD*4 (Harms, 1993), *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1020). Services for accessing these data are described at the back of the journal.

References

Cadet, J., Anselmino, C., Douki, T. & Voituriez, L. (1992). *J. Photochem. Photobiol. B*, **15**, 277–298.

Clivio, P., Fourrey, J. L., Gasche, J. & Farve, A. (1992). *Tetrahedron Lett.* **33**, 1615–1618.

Enraf–Nonius (1994). *CAD*-4 *EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.

Friedberg, E. C. (1985). In *DNA Repair*. New York: W. H. Freeman.

Gangamani, B. P., Cheravakkattu, G. S. & Ganesh, K. H. (1994). *J. Chem. Soc. Chem. Commun.* pp. 2275–2276.

Harms, K. (1993). *XCAD*4. *Data Reduction Program for the PC*. University of Marburg, Germany.

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

Khan, N., Morris, T. H., Smith, E. H. & Walsh, R. (1991). *J. Chem. Soc. Perkin Trans.* 1, pp. 865–870.

Kim, S.-T., Malhotra, K., Smith, C. A., Taylor, J.-S. & Sancar, A. (1994). *J. Biol. Chem.* **269**, 8535–8540.

Liu, J. & Taylor, J.-S. (1996). *J. Am. Chem. Soc.* **118**, 3287–3288.

Morris, T. H., Smith, E. H. & Walsh, R. (1987). *J. Chem. Soc. Chem. Commun.* pp. 964–965.

Prakash, G. & Falvey, D. E. (1995). *J. Am. Chem. Soc.* **117**, 11375–11376.

Ragini, A. (1965). *Tetrahedron Lett.* **42**, 3713–3717.

Rahn, R. O. & Hosszu, J. L. (1969). *Photochem. Photobiol.* **10**, 131–137.

Sancar, A. (1995). *Biochemistry*, **33**, 2–9.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Todo, T., Takemori, H., Ryo, H., Ihara, M., Matsunaga, T., Nikaido, O., Sato, K. & Nomura, T. (1993). *Nature (London)*, **361**, 371–374.

Varghese, A. J. & Wang, S. Y. (1968). *Science*, **160**, 186–187.

Wulff, D. L. & Fraenkel, G. (1961). *Biochem. Biophys. Acta*, **51**, 332–339.

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(20*S*)-6β-Methoxy-20-(*p*-toluenesulfonyloxymethyl)-3α,5-cyclo-5α-pregnane

KAMAL A. KETULY,† DMITRII S. YUFIT, CHARLES J. W. BROOKS AND ANDREW A. FREER

Chemistry Department, University of Glasgow, Glasgow G12 8QQ, Scotland. E-mail: dima@chem.gla.ac.uk

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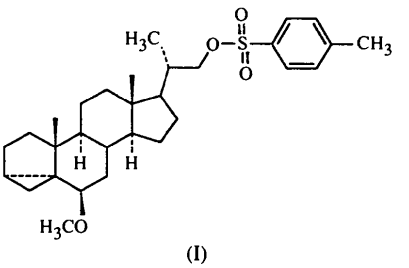
Abstract

The crystal and molecular structure of the title compound, $C_{30}H_{44}O_4S$, has been determined to confirm the molecular conformation. The fused cyclopropane moiety corresponding to part of ring *A* has a β-configuration and the associated cyclopentane ring has an envelope conformation.

Comment

(20*S*)-20-Hydroxymethyl-6β-methoxy-3α,5-cyclo-5α-pregnane is a key intermediate in the synthesis of sterol side chains from simple pregnane-type compounds. The crystal structure analysis of its *p*-toluenesulfonate derivative, (I) (Fig. 1), established the 20*S* configuration previously assigned on the basis of chemical and spectroscopic data (Partridge, Faber & Uskokovic, 1974; Dasgupta, Crump & Gut, 1974; Vanderah & Djerassi, 1978). The cyclopentane ring, fused to the cyclopropane moiety, adopts an envelope conformation, with C1 0.512 Å out of the plane defined by atoms C2, C3, C5 and C10.



(I)

† Present address: Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia.

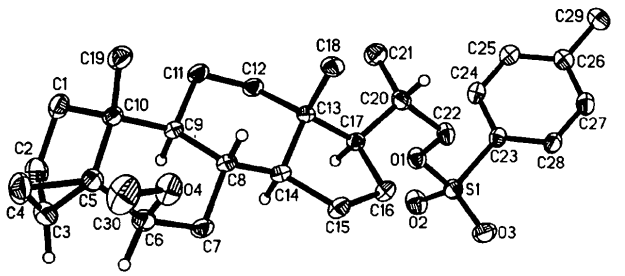


Fig. 1. The molecular structure of (I) showing the atom-numbering scheme. The H atoms of the tolyl ring and of the CH_3 and CH_2 groups have been omitted for clarity. Those remaining are shown as small spheres of arbitrary radii. Displacement ellipsoids are drawn at the 20% probability level.

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*.

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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.

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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*

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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.