Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tricoccin R6

A. Abdul Ajees,[a] K. Sekar,[b] S. Parthasarathy,[a]* H. Schenk,[c] B. Epe[d] and A. Mondon[d]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Bioinformatics Centre, Raman Building, Indian Institute of Science, Bangalore 560 012, India, [c]Laboratory Voor Kristallografie, Nieuwe Achtergracht 166, 1018, WU, Amsterdam, The Netherlands, and [d]Institute of Organic Chemistry, University of Kiel, D-2300 Kiel, Olshausenstrasse Be 40-60, Germany

Correspondence e-mail: partha24@hotmail.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.092
Data-to-parameter ratio = 8.0

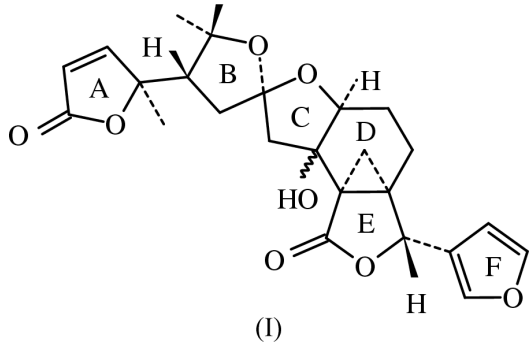
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 December 2000
Accepted 8 January 2001
Online 19 January 2001

The X-ray crystal structure of the title compound, $C_{25}H_{28}O_8$, has been determined. In the structure, both the terminal five-membered rings (*A* and *F*) are planar. The fused five-membered rings *C* and *E* are in envelope conformations, and ring *B* is in a slightly distorted half-chair conformation. The six-membered ring *D* is in a slightly distorted sofa conformation. The structure is stabilized by O—H···O hydrogen bonds and C—H···O inter- and intramolecular interactions.

Comment

The molecular structure of tricoccin R6, (I), is shown in Fig. 1. The bond geometry conforms to expectations. The puckering parameters evaluated using *PARST*97 (Nardelli, 1995) show that (i) both the terminal five-membered rings (*A* and *F*) are planar, (ii) the fused five-membered ring *B* is in a slightly distorted half-chair conformation, and rings *C* and *E* are in envelope conformations [q_2 = 0.334 (2) Å, φ_2 = −119.7 (4)° for ring *B*, q_2 = 0.440 (2) Å, φ_2 = −134.7 (3)° for ring *C*, and q_2 = 0.129 (2) Å, φ_2 = 151.2 (11)° for ring *E*]. The six-membered ring *D* has a slightly distorted sofa conformation [q_2 = 0.369 (3), q_3 = 0.364 (3), Q_T = 0.519 (2) Å, φ_2 = 63.0 (4)°]. Fig. 2 shows the packing diagram of the molecules. The structure is stabilized by intermolecular O—H···O hydrogen bonds and C—H···O inter- and intramolecular interactions (see Table 1).



(I)

Experimental

The title compound was isolated from *Cneorum tricoccin* L., a shrub native to coastal areas of the western Mediterranean with hairless leaves, yellow blossoms and red fruits (Herz *et al.*, 1983). It was crystallized from ethanol/acetone.

Crystal data

$C_{25}H_{28}O_8$
M_r = 456.47
Orthorhombic, $P2_12_12_1$
a = 7.224 (2) Å
b = 15.521 (2) Å
c = 20.404 (1) Å
V = 2287.8 (7) Å^3
Z = 4
D_x = 1.325 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 20–30°
μ = 0.82 mm^{-1}
T = 293 (2) K
Section from needle, colourless
0.25 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
2417 measured reflections
2417 independent reflections
2171 reflections with $I > 2\sigma(I)$
θ_{max} = 69.9°
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 24$
3 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.092
S = 1.04
2417 reflections
303 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.3026P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0078 (4)
Absolute structure: Flack (1983)
Flack parameter = 0.2 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C28—H28*C*···O32	0.96	2.41	2.894 (4)	111
O26—H26···O25^{i}	0.82	2.00	2.805 (2)	169
C23—H23···O31^{ii}	0.93	2.43	3.283 (3)	152

Symmetry codes: (i) $x-\frac{1}{2}, \frac{1}{2}-y, 2-z$; (ii) $2-x, y-\frac{1}{2}, \frac{5}{2}-z$.

The data set contains no Friedel pairs.

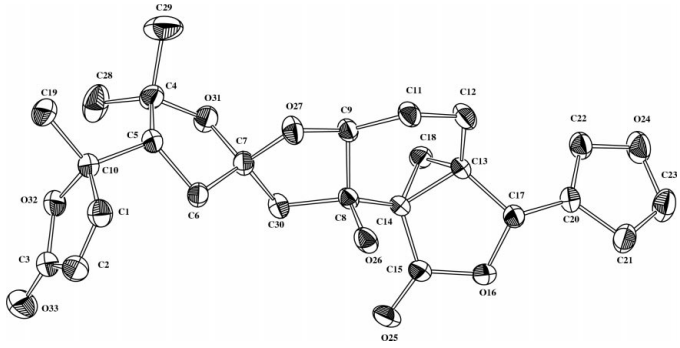


Figure 1
The molecular structure of the title compound with 30% probability displacement ellipsoids.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SDP* (Frenz, 1978); data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Johnson & Burnett, 1998); software used to prepare material for publication: *SHELXL*97 and *PARST*97 (Nardelli, 1995).

References

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
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.
Herz, W., Grisebach, H. & Kirby, G. W. (1983). *Progress in the Chemistry of Organic Natural Products*, Vol. 44, pp. 101–187. Wien, New York: Springer Verlag.
Johnson, C. K. & Burnett, M. N. (1998). *ORTEP*III. University of Glasgow, Scotland.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. Release 97–2. University of Göttingen, Germany.