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A neo-clerodane diterpene from *Teucrium tomentosum*. Corrigendum

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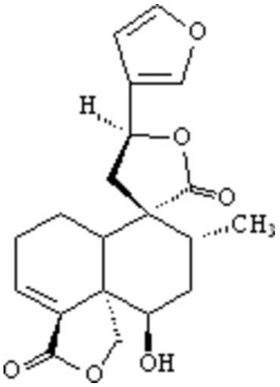
The chemical name of the title compound in the paper by Devi, Malathi, Rajan, Aravind, Krishnakumari & Ravikumar [*Acta Cryst.* (2004), E**60**, o117–o119] is corrected and the structural diagram is updated.

In the paper by Devi, Malathi, Rajan, Aravind, Krishnakumari & Ravikumar [*Acta Cryst.* (2004), E**60**, o117–o119], the chemical name given in the *Abstract* should be '15,16-epoxy-6β-hydroxyneo-cleroda-3,13(16),14-triene-18,19:20,12-diolide'. An updated structural diagram is shown below.



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A neo-clerodane diterpene from *Teucrium tomentosum*

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Key indicators

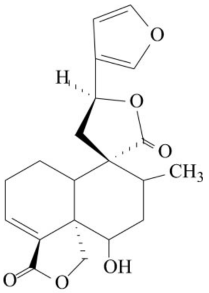
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.111
Data-to-parameter ratio = 8.9

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

A neo-clerodane diterpene, 15-hydroxy-15,16-epoxy-6α-neo-cleroda-3,13(16),14-triene-18,19:20,12-diolide, $C_{20}H_{22}O_6$, showing antifeedant activity against lepidopterous pests, has been isolated from *Teucrium tomentosum* and characterized by NMR and X-ray crystallographic methods. The crystallographic studies reveal that the cyclohexene and cyclohexane rings adopt a half-chair and a chair conformation, respectively. The furan ring exhibits rotational disorder. The molecules in the crystal structure are stabilized by a network of O—H···O and C—H···O hydrogen bonds.

Comment

As part of our ongoing research on terpenoid compounds with antifeedant activity, a neo-clerodane diterpene, an antifeedant against lepidopterous pests (De la Torre *et al.*, 1994; Merrit & Ley, 1992), was isolated from Teucrium tomentosum. The richest source of this kind of compound has been plants belonging to the genus *Teucrium* of the family *Labiatae*, from which a large number of clerodanes have been isolated in the past few years (Piozzi *et al.*, 1998; Merrit & Ley, 1992; Rodriguez *et al.*, 1994). The present study was carried out as part of structural studies of ecofriendly bioinsecticides. The chemical structure of (I) was obtained from ^{13}C and ^{1}H NMR data and the three-dimensional structure through the single-crystal X-ray diffraction study reported here.



(I)

The cyclohexene ring *A* and cyclohexane ring *B* exist in a half-chair and a chair conformation, respectively [Q_T = 0.518 (2) Å, $\varphi_2 = -136.2$ (3)°, $q_2 = 0.394$ (2) Å for ring *A*; Q_T = 0.570 (1) Å, $\varphi_2 = 17.6$ (8)°, $q_2 = 0.134$ (1) Å for ring *B* (Cremer & Pople, 1975)]. The γ-lactone rings *C* and *D* adopt envelope conformations [$\varphi_2 = -126.4$ (7)°, $q_2 = 0.202$ (2) Å for ring *C*; $\varphi_2 = -6.9$ (4)°, $q_2 = 0.371$ (2) Å for ring *D*], whereas the furan ring *E* is in a planar conformation (Nardelli, 1995) and is almost perpendicular to ring *C* [O12—C12—C13—C16 = −97.5 (3)°]. The rings *A*/*B* and *A*/*D* are *trans* fused. The methyl group at C8 and the hydroxy group at C6 are anti-

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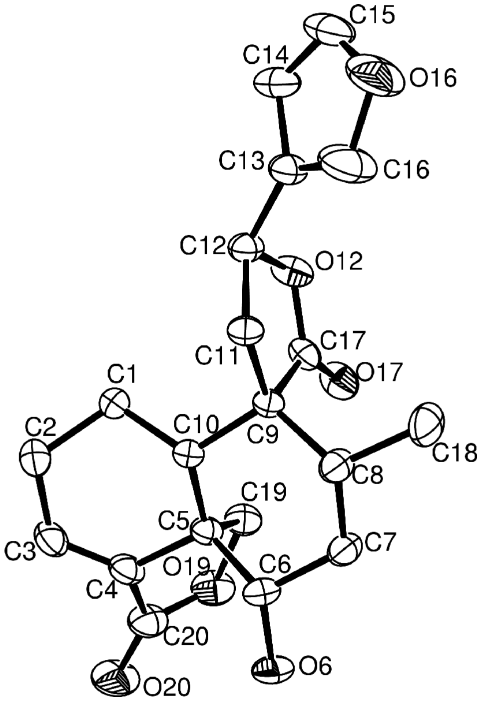

Figure 1
The molecular structure of compound (I), with 30% probability displacement ellipsoids and the atomic numbering scheme. H atoms have been omitted.

periplanar, with torsion angles C6—C7—C8—C18 = −176.8 (2)° and C10—C5—C6—O6 = 171.3 (1)°.

An *ORTEP*III (Burnett & Johnson, 1996) diagram of the molecule (Fig. 1) shows that the displacement parameters are normal except for those of the furan ring. However, this ring is highly distorted, which can be attributed to rotational disorder about the C12—C13 bond, as demonstrated by the steady increase in the values of the displacement parameters from C13 to C16. Similar features were observed in the structures of limonoids extracted from other plants (Kabaleeswaran *et al.*, 2000; Malathi *et al.*, 2002; Narayanan *et al.*, 1980).

Molecules in the crystal structure are connected by intermolecular O—H···O and C—H···O hydrogen bonds (Table 1). The crystal structure is stabilized by a two-dimensional network of hydrogen bonds (Fig. 2). The O6—H6···O20 hydrogen bond forms an infinite chain, with a graph-set motif (Bernstein *et al.*, 1995) of *C*(7), and the C15—H15···O6 hydrogen bond forms a graph-set motif of *C*(11). In addition, the O—H···O and C—H···O hydrogen bonds in the structure together form an $R_4^4(35)$ ring motif.

Experimental

The shade-dried powdered aerial parts (8.4 kg) of *Teucrium tomentosum* were exhaustively percolated with acetone (3 × 35 l). The extract was then dried in a rotary evaporator to yield a residue (146 g). 55 g of the residue was subjected to column chromatography using silica gel (70–325) and eluted with chloroform–methanol (1–100%), to yield 69 fractions. Fractions 28–38 were further chromatographed over silica gel (70–325) and eluted with *n*-hexane–ethyl acetate (1–100%) to yield Teuscordin (75 mg). Rectangular block-shaped crystals of the compound were obtained from a mixture of methanol and chloroform (1:3) at room temperature (293 K).

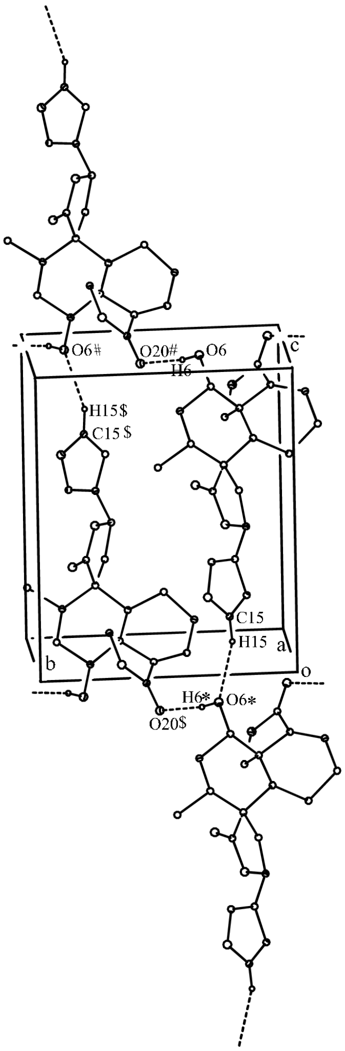

Figure 2
The crystal packing of (I), showing the $R_4^4(35)$ rings. Molecules labelled with an asterisk (*), dollar sign ($) and hash (#) correspond to symmetry positions ($x - 1, y, z - 1$), ($-x, y + \frac{1}{2}, -1 - z$), and ($1 - x, y + \frac{1}{2}, 2 - z$), respectively.

Crystal data

$C_{20}H_{22}O_6$
$M_r = 358.38$
Monoclinic, $P2_1$
$a = 7.7363$ (7) Å
$b = 9.5260$ (8) Å
$c = 11.8422$ (10) Å
$\beta = 96.134$ (2)°
$V = 867.73$ (13) Å^3
$Z = 2$
$D_x = 1.372$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2673 reflections
$\theta = 2.6$–27.7°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Rectangular block, colourless
0.30 × 0.24 × 0.15 mm

Data collection

Bruker SMART APEX CCD area-detector
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.979$, $T_{\max} = 0.982$
5505 measured reflections
2103 independent reflections
1912 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 28.0$°
$h = -9 \rightarrow 9$
$k = -7 \rightarrow 12$
$l = -15 \rightarrow 13$

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.111$
$S = 1.04$
2103 reflections
236 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0732P)^2 + 0.0489P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O6—H6⋯O20$^{\text{i}}$	0.82	1.94	2.754 (3)	171
C15—H15⋯O6$^{\text{ii}}$	0.93	2.43	3.292 (3)	155
C2—H2*A*⋯O6$^{\text{iii}}$	0.97	2.48	3.424 (3)	164
C12—H12⋯O17$^{\text{iv}}$	0.98	2.57	3.488 (3)	156

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

In the absence of significant anomalous scattering, Friedel pairs were merged before the final refinement. The enantiomer employed in the refined model was chosen to agree with the accepted configuration of diterpenes (Rogers *et al.*, 1979; Rodriguez *et al.*, 1994; De la Torre *et al.*, 1994). H atoms were constrained to an ideal geometry (C—H = 0.97–0.98 Å and O—H = 0.82 Å), with U_{iso}(H) = 1.2 (1.5 for methyl and OH) times U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL*97 and *PARST*97 (Nardelli, 1995).

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