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Key indicators

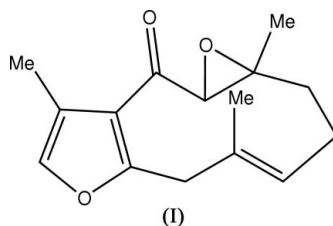
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.034
 wR factor = 0.097
Data-to-parameter ratio = 8.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.5,9,14-Trimethyl-4,12-dioxatricyclo[9.3.0.0^{3,5}]-tetradeca-1(11),8,13-trien-2-one (zederone)

In the title compound, $\text{C}_{15}\text{H}_{18}\text{O}_3$, the epoxy and furyl rings are planar and inclined to each other by $44.84(17)^\circ$. The α,β -carbonyl system has *S-cis* conformation and both methyl groups in the ten-membered ring are in axial positions. The molecule is stabilized by van der Waals interactions.

Received 13 March 2006
Accepted 20 March 2006

Comment

The title compound, (I), is a sesquiterpenoid keto-dioxide compound found in the rhizome of curcuma, a genus of the family Zingiberaceae herb that grows in tropical Asia. The detailed structure of zederone obtained from the rhizome of *Curcuma zedoaria* Roscoe used as an aromatic stomachic in the crude drug zedoary was established by NMR studies (Hikino *et al.*, 1968). In the present study, crystals of (I) were obtained from an extract of *Curcuma inodora* aff. Blatter collected from West Malaysia.



The molecular structure contains three fused rings, *viz.* the ten-membered ring C3–C12, the five-membered ring C1–C4/O1 and the epoxy ring C10/C11/O2 (Fig. 1). The epoxy ring is perfectly planar, while the furyl ring O1/C1–C4 has a maximum deviation of $0.010(2)\text{ \AA}$ for atoms C1 and O1. The β -furyl methyl atom C13 is displaced by $0.029(2)\text{ \AA}$ from the least-squares plane. As a result, the α,β -unsaturated carbonyl system has an *S-cis* conformation. The dihedral angle between the furyl and epoxy rings is $44.84(17)^\circ$. Both atoms C14 and C15 occupy axial positions of the somewhat chair-like conformation of the C6–C11 portion of the ten-membered ring. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987).

Experimental

The dried rhizomes of *Curcuma inodora* aff. Blatter were collected from MARDI, Teluk Intan, Perak, Malaysia. A voucher specimen (HI1336) has been deposited in the herbarium of the University of Malaya, Kuala Lumpur. Fresh *C. inodora* aff. Blatter rhizomes (1 kg) were dried for about 2 d in an oven and then ground to a fine powder for extraction. The powder was soaked in methanol for 3 d, and then the solution was filtered and the solvent was removed on a rotary

evaporator. The extraction was repeated once and the combined dried extract obtained was 15.0 g. The crude solid extract was further extracted using hexane. The hexane extract (2.30 g) was subjected to column chromatography using silica as stationary phase and eluted using hexane with an increasing ratio of ethyl acetate. Fractions 78–80 containing the compound [$R_f = 0.8$, solvent hexane-EA(7:3)] were combined and concentrated by evaporation to give a colourless crystalline solid (0.9 g, 0.10%). Suitable crystals for X-ray investigation were obtained by multiple recrystallization from a mixture of hexane–acetone (3:1 v/v). The melting point of 426.5–427.0 K and the NMR spectra of the product are in agreement with the reported values (Hikino *et al.*, 1968).

Crystal data

$C_{15}H_{18}O_3$	Mo $K\alpha$ radiation
$M_r = 246.29$	Cell parameters from 4030 reflections
Orthorhombic, $P2_12_12_1$	$\theta = 2.5\text{--}26.0^\circ$
$a = 9.672$ (2) Å	$\mu = 0.09$ mm $^{-1}$
$b = 11.414$ (3) Å	$T = 298$ (2) K
$c = 11.656$ (3) Å	Block, colourless
$V = 1286.7$ (5) Å 3	$0.45 \times 0.40 \times 0.40$ mm
$Z = 4$	
$D_x = 1.271$ Mg m $^{-3}$	

Data collection

Bruker SMART APEX area-detector diffractometer	1465 independent reflections
ω scan	1374 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$R_{int} = 0.019$
$T_{min} = 0.958$, $T_{max} = 0.971$	$\theta_{max} = 26.0^\circ$
7096 measured reflections	$h = -10 \rightarrow 11$
	$k = -14 \rightarrow 13$
	$l = -14 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 0.0797P]$
$R[F^2 > 2\sigma(F^2)] = 0.034$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.097$	$(\Delta/\sigma)_{max} = 0.006$
$S = 1.08$	$\Delta\rho_{max} = 0.13$ e Å $^{-3}$
1465 reflections	$\Delta\rho_{min} = -0.11$ e Å $^{-3}$
166 parameters	
H-atom parameters constrained	

All H atoms were located in a difference map, but they were repositioned geometrically, with C–H = 0.93–0.98 Å, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.5$ (for methyl H atoms) and 1.2 (other H atoms) times $U_{eq}(C)$. In the

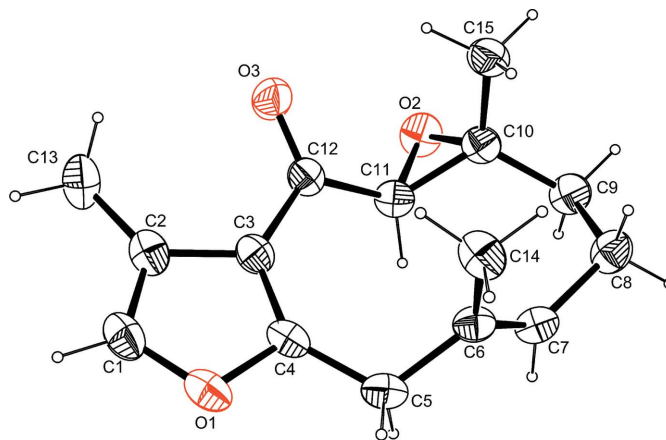


Figure 1

The molecular structure of the title compound, (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as spheres of arbitrary radii.

absence of any significant anomalous dispersion, Friedel pairs were merged before the final refinement.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997b); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank the Malaysian Government and Kebangsaan Malaysia for the research grants and facilities provided.

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