Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,5,13-Trimethyl-9-phenyl-8,10-dioxatetracyclo-[7.7.1.0^{2,7}.0^{11,16}]heptadeca-2,4,6,11,13,15-hexaene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.050
wR factor = 0.134
Data-to-parameter ratio = 13.3

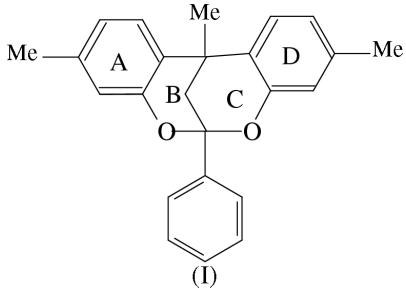
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 2 January 2001
Accepted 30 January 2001
Online 13 February 2001

In the title compound, $C_{24}H_{22}O_2$, both six-membered heterocyclic rings adopt a distorted envelope conformation. The mean planes passing through these rings are nearly perpendicular to each other [dihedral angle 88.39 (7)°].

Comment

The title compound, (I), was synthesized with a view to screening it for antimalarial activity. Before studying the activity, the X-ray structure determination was carried out. In (I), both the heterocyclic rings *B* and *C* adopt a distorted envelope conformation; the puckering parameters are: Q = 0.526 (2) Å, θ = 124.7° and φ = 309.47 (1)° for ring *B* and Q = 0.505 (2) Å, θ = 130.5 (2)° and φ = 65.54 (2)° for ring *C*, respectively (Cremer & Pople, 1975). Atom C17 deviates from the best plane passing through atoms C1/C2/C7/O8/C9 by 0.715 (2) Å and similarly the deviation of C17 from the best plane through C9/O10/C11/C16/C1 is −0.688 (2) Å. The mean planes through the two heterocyclic rings *B* and *C* are nearly perpendicular to each other [dihedral angle 88.39 (7)°]. The phenyl ring at C9 is equatorially attached to the rings *B* and *C* and with them it makes dihedral angles of 70.52 (8) and 19.04 (8)°, respectively. The methyl group at C1 is pseudo-equatorially disposed [C18—C1—C16—C11 −148.1 (1)° and C18—C1—C2—C7 152.9 (1)°]. In the crystal, C—H···π interactions involving rings *A* and *D* are observed [C18—H181···*CgA*(x, $-1+y$, z), with H···*Cg* 2.70, C18···*Cg* 3.612 (2) Å and *X*—H···*Cg* 158°; C18—H182···*CgD*(x, $-1+y$, z), with H···*Cg* 2.74, *X*···*Cg* 3.666 (2) Å and *X*—H···*Cg* 161°].



(I)

Experimental

4-Methylphenol (0.05 mol) and benzoylacetone (0.05 mol) were mixed throughly. 80% sulfuric acid (15 ml) was added gradually with constant stirring. The reaction mixture was cooled in an ice-bath during the addition. It was kept overnight at room temperature and then poured over crushed ice with constant stirring. The resulting solid was filtered, washed with water and then with a dilute sodium

hydroxide solution followed by water to remove unreacted substances. It was dried and crystallized from ethanol as colourless single crystals (yield 60%; m.p. 333 K).

Crystal data

$C_{24}H_{22}O_2$	D_x = 1.281 Mg m^{-3}
M_r = 342.42	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
a = 17.7903 (15) Å	θ = 2–25°
b = 5.5559 (5) Å	μ = 0.08 mm^{-1}
c = 18.0320 (17) Å	T = 293 (2) K
β = 94.815 (8)°	Needle, white
V = 1776.0 (3) Å^3	0.20 × 0.15 × 0.12 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.020
ω–2θ scans	θ_{max} = 25.3°
Absorption correction: ψ scan (North *et al.*, 1968	$h = 0 \rightarrow 21$
T_{min} = 0.982, T_{max} = 0.994	$k = 0 \rightarrow 6$
3351 measured reflections	$l = -21 \rightarrow 21$
3240 independent reflections	3 standard reflections every 100 reflections
2918 reflections with $I > 2\sigma(I)$	intensity decay: negligible

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.5073P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.134	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
S = 1.13	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
3219 reflections	Extinction correction: *SHELXL*97
242 parameters	Extinction coefficient: 0.019 (1)
H-atom parameters constrained	

All H atoms were fixed using geometrical considerations and their overall displacement parameters were refined.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *MolEN* (Fair, 1990); data reduction: *MolEN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL*97 and *PARST*95 (Nardelli, 1995).

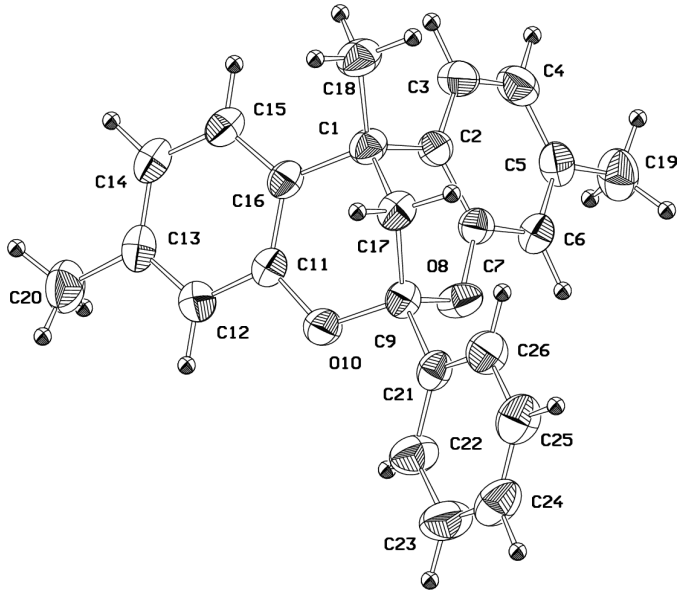


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids.

LV thanks the UGC, India, for the award of Minor Research Project during the period 1997–99. One of the authors (LV) thanks Dr Babu Varghese, RSIC, Indian Institute of Technology, Chennai, for his assistance in data collection.

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