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3-Bromo-β-lapachone

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.114
Data-to-parameter ratio = 17.0

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

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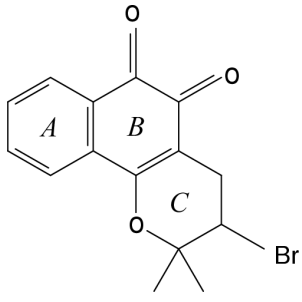
In the title compound, $C_{15}H_{13}BrO_3$, the benzo and quinone rings are planar, while the heterocycle is in a distorted half-chair conformation.

Comment

Naphthoquinone compounds are often found in nature and their biological activity has been associated with various medicinal applications. Their action ranges from antibiotic to antineoplastic activity, although some of them do not presently have a defined function (Pinto *et al.*, 1980). Among the naphthoquinone compounds, lapachol, lapachones and their derivatives have been of interest to the scientific communities of several countries for more than 100 years because of the large range of biological activities found for these compounds (Subramanian, 1996). Among these activities are: antiviral (Pinto, Pinto *et al.*, 1987), antimalarial (Fieser *et al.*, 1967), antitumor (Li *et al.*, 1999), and activity against trypanosoma cruzi, the protozoan of Chagas disease (Gonçalves *et al.*, 1980; Pinto, Ferreira *et al.*, 1987). As part of a search for compounds with therapeutic activity against a number of parasitic diseases endemic to Brazil, a series of derivatives has been prepared from lapachol (Cruz *et al.*, 1977).



(I)

The title compound, (I), is a derivative of lapachol. It was tested in two biological assays, against trypanosoma cruzi (Lopes *et al.*, 1978), and as a protection against the penetration of Schistosomiasis mansoni cercariae in tails of mice (Pinto *et al.*, 1977), and showed, in both tests, discrete biological activity.

The crystal structure of (I) (Fig. 1) shows that the atoms comprising rings *A* and *B* and the adjacent C and O atoms are essentially coplanar, with an r.m.s. deviation of 0.039 Å for the 14 atoms. Atoms C2 and C3 are 0.256 (2) and 0.519 (3) Å out of this plane, respectively. Therefore, the *C* ring assumes a

distorted half-chair conformation, the Cremer & Pople (1975) ring-puckering parameters being q_2 = 0.407 (2), q_3 = 0.314 (2) Å, Q = 0.514 (2) Å, θ = 52.4 (2)° and φ = 160.8 (3)°. The overall geometry of both the *B* and *C* rings is in good agreement with that found for this moiety in a similar compound (Pereira, 1989).

Experimental

The title compound, (I), was synthesized for the first time by Paternó (1882). However, the method used here was that of Hooker (1892). This substance is easily prepared, in chloroform solvent, by reaction of lapachol with bromine, followed by evaporation and crystallization from ethanol. It was recrystallized from acetone at room temperature.

Crystal data

$C_{15}H_{13}BrO_3$
M_r = 321.16
Monoclinic, $P2_1/c$
a = 11.823 (2) Å
b = 8.191 (2) Å
c = 13.894 (3) Å
β = 106.05 (1)°
V = 1293.1 (5) Å^3
Z = 4
D_x = 1.650 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3176 reflections
θ = 1.0–27.5°
μ = 3.18 mm^{-1}
T = 120 (2) K
Prism, orange
0.22 × 0.15 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.541, T_{max} = 0.702
5298 measured reflections
2971 independent reflections
2601 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 27.5°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 10$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.114
S = 1.09
2971 reflections
175 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.069P)^2 + 0.7539P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.05 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br—C3	1.958 (2)	C4*A*—C4	1.505 (3)
O1—C1	1.351 (3)	C3—C4	1.513 (3)
O1—C2	1.473 (3)	C3—C2	1.536 (4)
C4*A*—C1	1.363 (3)		
C1—O1—C2	119.39 (18)	O1—C2—C3	105.23 (18)
C4—C3—C2	112.2 (2)	C4*A*—C4—C3	107.7 (2)
C2—C3—Br	111.39 (16)		

H atoms were positioned geometrically and refined with a riding model, with isotropic displacement parameters equal to 1.5 (for methyl H atoms) or 1.2 times U_{eq} of the parent atom.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

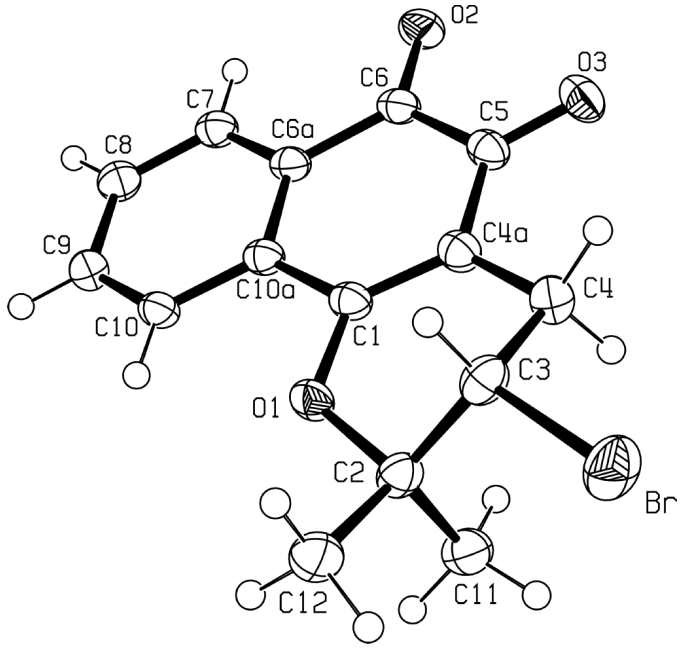


Figure 1
The molecular structure of (I), showing the atom labelling and 50% probability displacement ellipsoids.

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