Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.123$
$S = 1.096$
3262 reflections
263 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.112$ e Å^{-3}
$\Delta\rho_{\min} = -0.107$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
Extinction coefficient: 0.024 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—N2	1.431 (3)	N1—C1	1.473 (3)
O1—C1	1.439 (4)	N2—C8	1.287 (3)
O2—C23	1.321 (4)	C1—C2	1.491 (4)
O2—C24	1.450 (4)	C8—C9	1.470 (4)
O3—C23	1.187 (3)	C15—C23	1.513 (4)
N1—C8	1.389 (4)	C15—C16	1.535 (4)
N1—C15	1.455 (3)	C16—C17	1.507 (4)
N2—O1—C1	109.71 (19)	N2—C8—N1	115.6 (2)
C23—O2—C24	117.3 (3)	N1—C15—C23	111.5 (2)
C8—N1—C1	105.2 (2)	N1—C15—C16	111.9 (2)
C8—N2—O1	105.8 (2)	C17—C16—C15	111.5 (2)
O1—C1—N1	103.1 (2)	O3—C23—O2	122.4 (3)
O1—C1—C2	110.8 (2)	O3—C23—C15	125.2 (3)
N1—C1—C2	114.0 (2)	O2—C23—C15	112.4 (2)

The data were collected using radiation from a rotating-anode generator operating at 50 kV and 90 mA by taking 23 oscillation photos in the range of 0–184° with exposing time of 10 min per frame. Crystal-to-detector distance was 78.1 mm. The structure was solved by direct methods. All the non-H atoms were refined anisotropically. All H atoms were included with the riding model.

Data collection: R-AXIS II*C* diffractometer software (Rigaku, 1995). Cell refinement: R-AXIS II*C* diffractometer software. Data reduction: R-AXIS II*C* data processing software. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*). Molecular graphics: *SHELXTL* (Sheldrick, 1995). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1227). Services for accessing these data are described at the back of the journal.

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3,5-Di-*tert*-butyl-1,2-benzoquinone

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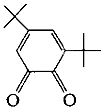
Abstract

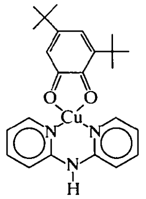
The molecular structure of the title compound, $C_{14}H_{20}O_2$, (I), has been investigated using X-ray diffraction. When compared with the semiquinone (DTBSQ) molecule in a coordinated state, *e.g.* [Cu{NH(py)$_2$}-(DTBSQ)]ClO$_4$·0.5C$_4$H$_8$O [(II); Thompson & Calabrese (1985). *Inorg. Chem.* **24**, 3167–3171], the different C—O distances [1.214 (3) and 1.217 (3) Å in (I); 1.293 (7) and 1.304 (7) Å in (II)] reveal information about the electron distributions of the carbonyl group in different environments.

Comment

When we studied the auto-oxidation of 3,5-di-*tert*-butylcatechol (DTBC) by copper complexes (Chyn & Urbach, 1991), it was revealed that the intermediates have various possible structural states, and that the oxidated compound exists as a moiety of 3,5-di-*tert*-butyl-1,2-benzoquinone (DTBQ) in copper complexes. To investigate the coordinating character of the carbonyl groups, the crystal structure of DTBQ, (I), has been established. Comparison with the cation [Cu{NH(py)$_2$}-

(DTBSQ)]$ClO_4\cdot0.5C_4H_8O$, (II), (Thompson & Calabrese, 1985) provides relevant information about the electron distribution of carbonyl groups in different environments.


(I)


(II)

An *ORTEP*II (Johnson, 1976) drawing of (I), with the atomic labelling scheme, is shown in Fig. 1. The bond lengths of C—O in uncoordinated carbonyl groups [C1—O1 = 1.217 (3) and C2—O2 = 1.214 (3) Å] are obviously shorter than those in (II) [1.293 (7) and 1.304 (7) Å]. This fact revealed that the bond electron densities of C—O in (II) are less than those in (I), owing to the coordination of the carbonyl groups in (II). For the same reason, the C1—C2—C3 and C2—C1—C6 bond angles in (I) [118.3 (2) and 118.7 (2)°, respectively] are also less than those in (II) [120.2 (6) and 120.5 (6)°, respectively]. Within the six-membered ring of compounds (I) and (II), the C3—C4 and C5—C6 distances [1.340 (3) and 1.344 (4) Å, respectively, in (I); 1.362 (8) and 1.361 (9) Å, respectively, in (II)] are always shorter than those of any other C—C bonds [1.439 (4)–1.554 (5) in (I) and 1.407 (9)–1.446 (9) Å in (II)], on account of double-bond character.

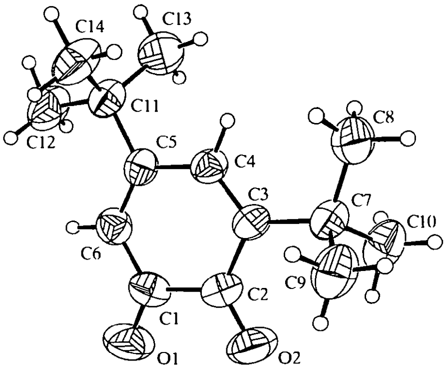


Fig. 1. The molecular structure of (I), showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of an arbitrary radius.

Experimental

The title compound was obtained by oxidation of 3,5-di-*tert*-butylcatechol with lead peroxide (PbO_2), following the literature procedure of Cuntze *et al.* (1969). Suitable crystals for the present study were obtained from an ether solution.

Crystal data

$C_{14}H_{20}O_2$
$M_r = 220.31$
Monoclinic
$P2_1/n$
$a = 9.285\,(1)$ Å
$b = 11.860\,(3)$ Å
$c = 12.530\,(2)$ Å
$\beta = 102.94\,(1)°$
$V = 1344.7\,(4)$ Å^3
$Z = 4$
$D_x = 1.088$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.28–16.63°$
$\mu = 0.07$ mm^{-1}
$T = 293$ K
Prismatic
0.56 × 0.31 × 0.25 mm
Red–brown

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2494 measured reflections
2362 independent reflections
1434 reflections with $I_{net} > 2\sigma(I_{net})$

$R_{int} = 0.033$
$\theta_{max} = 25°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 14$
3 standard reflections frequency: 60 min intensity decay: 3%

Refinement

Refinement on F
$R = 0.044$
$wR = 0.052$
$S = 1.90$
1434 reflections
146 parameters
H atoms not refined
$w = 1/[\sigma^2(F) + 0.0001F^2]$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: $3.8\,(2) \times 10^4$
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Data reduction: *DATRD*2 in *NRCVAX* (Gabe *et al.*, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*. Program(s) used to refine structure: *LSTSQ* in *NRCVAX*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TABLES* in *NRCVAX*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1163). Services for accessing these data are described at the back of the journal.

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