

Table 2.\* The molecular structure is shown in Fig. 1, drawn using *PLUTO* (Motherwell & Clegg, 1978).

**Related literature.** There appear to have been no previous crystal structure determinations of molecules having the pyrrolo[1,2-*b*]isoquinoline-5,10-dione ring nucleus. The bond lengths and angles

\* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55268 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0528]

involving the disordered atoms are unreliable. Other molecular dimensions show no abnormal features.

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## Structure of 1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]-1-propanone

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**Abstract.**  $C_{14}H_{20}O_2$ ,  $M_r = 220.3$ , monoclinic,  $C2/m$ ,  $a = 16.200$  (2),  $b = 7.182$  (2),  $c = 13.758$  (2) Å,  $\beta = 126.99$  (1)°,  $V = 1278.6$  (5) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.14$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 0.70$  cm<sup>-1</sup>,  $F(000) = 480$ ,  $T = 294$  K,  $R = 0.056$  and  $wR = 0.051$  for 701 significant reflections. An intramolecular hydrogen bond exists between the carbonyl and hydroxyl groups.

**Experimental.** A colourless crystal  $0.70 \times 0.60 \times 0.20$  mm [obtained as described by Martin, Lafrance & Demerseman (1991) and recrystallized from ethanol], was used for data collection on an Enraf-Nonius diffractometer, with graphite-monochromated Mo  $K\alpha$  radiation. Intensities were measured using  $\theta$ - $2\theta$  scans at room temperature. Unit-cell dimensions were determined by least-squares treatment of 25 reflections ( $17.5 < \theta < 18^\circ$ ). No absorption correction was applied to the data, which were collected to  $(\sin\theta/\lambda)_{\max} = 0.59$  Å<sup>-1</sup>. Two standard reflections measured every 2 h showed no significant variation. 1229 unique reflections were measured, of

which 701 were used in calculations [ $I \geq 3\sigma(I)$ ];  $h - 15 \rightarrow 15$ ,  $k 0 \rightarrow 8$ ,  $l 0 \rightarrow 16$ . Calculations were performed using the program *CRYSTALS* (Carruthers & Watkin, 1985) on a VAX 11/725 computer; the structure was solved by direct methods (Sheldrick, 1985). The molecule lies in a crystallographic mirror plane, except C(12) and C(12') of the *tert*-butyl group and some H atoms which are in general position in the space group  $C2/m$ . Refinements in  $C2$  did not significantly improve the  $R$  factor. H atoms were localized on a difference Fourier synthesis and refined with an overall isotropic thermal parameter. Refinement of 129 least-squares parameters [(number of reflections)/(number of varied parameters) = 5.4 (refined by full-matrix least squares); no extinction correction] converged at  $R(F) = 0.056$ ,  $wR = 0.051$  ( $w = 1$ ); average shift/e.s.d. = 0.17, maximum shift/e.s.d. = 1.0 in the last cycle; maximum  $\Delta\rho = 0.20$ , minimum  $\Delta\rho = -0.15$  e Å<sup>-3</sup> in the final difference Fourier synthesis. Scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 shows atomic parameters

Table 1. *Positional parameters and equivalent isotropic thermal parameters (Å<sup>2</sup>)*

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j.$$

	x	y	z	<i>U</i> <sub>eq</sub>
O(1)	0.0395 (3)	0	-0.0684 (4)	0.0616
O(2)	0.2009 (3)	0	0.1359 (4)	0.0813
C(1)	0.2037 (4)	0	-0.0338 (5)	0.0474
C(2)	0.0941 (4)	0	-0.1126 (5)	0.0468
C(3)	0.0362 (4)	0	-0.2407 (5)	0.0487
C(4)	0.0932 (5)	0	-0.2850 (5)	0.0562
C(5)	0.1997 (5)	0	-0.2107 (6)	0.0597
C(6)	0.2573 (4)	0	-0.0859 (5)	0.0504
C(7)	0.2547 (4)	0	0.1001 (5)	0.0545
C(8)	0.3691 (5)	0	0.1954 (6)	0.0613
C(9)	0.4006 (6)	0	0.3253 (6)	0.0816
C(10)	-0.0817 (4)	0	-0.3238 (5)	0.0603
C(11)	-0.1260 (6)	0	-0.4601 (7)	0.0843
C(12)	-0.1242 (4)	0.1767 (8)	-0.3029 (5)	0.0752
C(13)	0.3728 (5)	0	-0.0185 (7)	0.0715

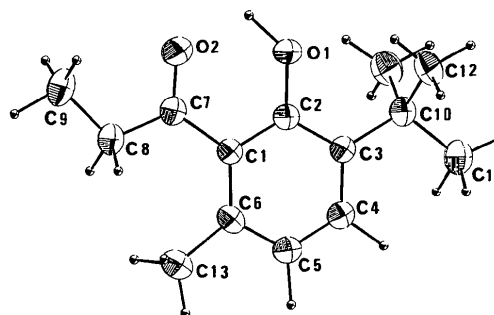


Fig. 1. ORTEP view (Johnson, 1976) showing the atom-numbering scheme. Ellipsoids are drawn at the 30% probability level; H atoms are given arbitrary radii.

Table 2. *Interatomic distances (Å) and angles (°) and hydrogen-bond geometry (Å, °)*

O(1)—C(2)	1.342 (6)	O(2)—C(7)	1.233 (6)
C(1)—C(2)	1.418 (7)	C(1)—C(6)	1.421 (7)
C(1)—C(7)	1.498 (7)	C(2)—C(3)	1.412 (7)
C(3)—C(4)	1.379 (7)	C(3)—C(10)	1.526 (7)
C(4)—C(5)	1.378 (8)	C(5)—C(6)	1.375 (8)
C(6)—C(13)	1.507 (8)	C(7)—C(8)	1.493 (8)
C(8)—C(9)	1.536 (9)	C(10)—C(11)	1.552 (9)
C(10)—C(12)	1.551 (6)		
C(6)—C(1)—C(2)	118.6 (5)	C(7)—C(1)—C(2)	116.8 (5)
C(7)—C(1)—C(6)	124.6 (5)	C(1)—C(2)—O(1)	121.1 (5)
C(3)—C(2)—O(1)	116.2 (5)	C(3)—C(2)—C(1)	122.7 (5)
C(4)—C(3)—C(2)	115.7 (5)	C(10)—C(3)—C(2)	121.8 (5)
C(10)—C(3)—C(4)	122.5 (5)	C(5)—C(4)—C(3)	123.0 (6)
C(6)—C(5)—C(4)	122.2 (6)	C(5)—C(6)—C(1)	117.9 (5)
C(13)—C(6)—C(1)	126.8 (6)	C(13)—C(6)—C(5)	115.3 (5)
C(1)—C(7)—O(2)	119.5 (5)	C(8)—C(7)—O(2)	116.8 (5)
C(8)—C(7)—C(1)	123.7 (5)	C(9)—C(8)—C(7)	112.9 (6)
C(11)—C(10)—C(3)	111.5 (5)	C(12)—C(10)—C(3)	110.8 (3)
C(12)—C(10)—C(11)	106.8 (4)	C(12)—C(10)—C(12')	109.8 (5)

Hydrogen bond

O(1)···O(2)	2.428 (6)	O(2)···H(1)	1.77 (7)	O(1)—H(1)···O(2)	129 (6)
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Symmetry code: (') x, -y, z.

and Table 2 interatomic distances and bond angles.\* Fig. 1 shows a view of the molecule with the numbering scheme.

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55324 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU0326]

**Related literature.** After examination of <sup>1</sup>H NMR spectra, the title compound was formulated as 1-[3-(1,1-dimethylethyl)-6-hydroxy-2-methylphenyl]-1-propanone (Martin, 1973). This contradicted our results with respect to the mobility of the *tert*-butyl group (Martin & Betoux, 1969, 1970, 1971). So, a structural analysis from X-ray data has been realized and has led to the formula 1-[3-(1,1-dimethylethyl)-2-hydroxy-6-methylphenyl]-1-propanone (Martin, Lafrance & Demerseman, 1991), for this essential intermediate in the synthesis of the vicinal *o*-hydroxypropiophenone, used in the pharmaceutical industry.

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