Related literature. The structures of dimethylfulvene at 248 K (Norman & Post, 1961) and 2-(2,4-cyclopentadien-1-ylidene)adamantane or adamantylidenefulvene (Garcia, McLaughlin & Fronczek, 1989) exhibit the expected single–double bond alternation within the fulvene ring system as does the title compound. The bond angle C7—C6—C11 = 110·95 (9)° of the title compound is similar to the analogous bond angle in adamantylidenefulvene, 111·5 (2)°, and smaller than that of dimethylfulvene, 114·0 (6)°, indicative of ring strain.

The bicyclo[3.3.1]nonane ring system of the title molecule exhibits a close intramolecular contact, 1·95 (2) Å, between the axial C9 and C13 H atoms in the twin chair conformation similar to those observed by Watson, Grossie & Taylor (1982), Sim (1983), and Narasimhan & Chacko (1984). The true internuclear H···H distance is even shorter, since the X-ray experiment underestimates C—H distances, and the C—H bonds are convergent.

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Hydroquinone–1,4-Dioxane (1/1)

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Abstract. $C_6H_6O_2.C_4H_8O_2$, M_r = 198·22, monoclinic, $P2_1/a$, a = 7·388 (2), b = 7·684 (1), c = 9·264 (2) Å, β = 78·83 (2)°, V = 515·95 (19) Å^3, Z = 2, D_x = 1·37 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·09 mm^{-1}, F(000) = 212, T = 293 K, R = 0·048 for 601 reflections. The complex occurs as chains parallel to **c** with O···H—O bonding [2·722 (4) Å]. O···H 1·830 (5) Å, with O···H—O 168·8 (6)°. The torsion angles H···O—C—C average 25·7 (15)°. The short C—C distance observed in the dioxan molecule [1·489 (2) Å] may indicate some disorder.

Experimental. This previously unreported complex was prepared by allowing a solution of hydroquinone in 1,4-dioxane to evaporate at room temperature. Transparent colourless crystals up to 2 mm along their edges are stable indefinitely when stored under mother liquor but become opaque, losing dioxane within minutes when exposed to air at room temperature. Even specimens mounted dry in Lindemann-glass capillaries decomposed completely overnight. The crystal (dimensions 0·20 × 0·35 × 0·43 mm) used for data collection was mounted in a capillary partially filled with mother liquor not in contact with the crystal. This crystal showed no significant change in the intensities of three standard reflections checked hourly during data collection. Unit-cell dimensions were refined from 20 accurately centred reflections with $\theta \simeq 12°$ on an Enraf–Nonius CAD-4 diffractometer. Data were collected for a hemisphere of reciprocal space to $\theta = 25°$. 1367 measured reflections gave 764 unique data of which 601 with $F_o \geq 2\sigma(F)$ were used in the final refinement. $R_{\rm int}$ = 0·015. Ranges of indices $-8 \leq h \leq$

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8, $0 \leq k \leq 9$, $0 \leq l \leq 11$. The orientation of the crystal was verified every 200 reflections. Data were corrected for the Lorentz and polarization terms but not for absorption. A few low order reflections gave $|F_o| < |F_c|$ by up to 5σ. This may be secondary extinction but no correction was attempted in view of the fugacity of the crystals.

Programs used included *SHELXS*86 (Sheldrick, 1986), *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *SHELX*76.

The direct-methods routine *TREF* in *SHELXS*86 showed all non-H atoms. Routine refinement led to convergence with anisotropic temperature parameters for all non-H atoms. All H atoms were located on difference syntheses and refined with grouped isotropic temperature parameters.

Final atomic coordinates are given in Table 1 with bond lengths and angles in Table 2.* The structure is shown in Fig. 1. The hydroquinone molecule shows no unusual features and has small temperature parameters but the dioxane molecule has extended thermal ellipsoids and a short C—C bond [1·488 (2) Å]. These features could be explained by some disorder in the location of the dioxane molecules about the centre of symmetry at (0,0·5,0·5). Final refinement minimizing $\sum w|F_o - |F_c||^2$, 89 refined parameters, $R = 0{\cdot}048$, $wR = 0{\cdot}071$, $w = 1{\cdot}000/[\sigma(F)^2 + 0{\cdot}030710F^2]$, mean shift/e.s.d. = 0·011, max. shift/e.s.d. = 0·119, max. diff. peak = 0·204 e Å^{-3}, max. negative peak = 0·339 e Å^{-3}.

Related literature. The hydroquinone molecule has a geometry identical to that found in hydroquinone itself (Wallwork & Powell, 1980) and in hydroquinone complexes. The structure of hydroquinonebis(1,8-epoxymenthane) was reported recently (Barnes, 1988) with very similar hydrogen bonding [2·742 (2) Å]. There are numerous examples of 1,4-dioxane (DX) forming hydrogen bonds, including H_2SO_4.DX (Hassel & Hvoslev, 1954). In $NiBr_2.4H_2O.2DX$ (Barnes & Weakley, 1976) and $SmBr_3.9H_2O.2DX$ (Barnes & Nicholl, 1985) coordinated water molecules are interlinked by hydrogen bonds to dioxane. The thermal decomposition of hydroquinone–1,4-dioxane and hydroquinonebis(1,8-epoxymenthane) will be reported separately.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

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

	x	y	z	U_{eq}(Å^2)
O1	0·0956 (2)	0·9600 (2)	0·2715 (1)	0·059 (1)
C2	0·0473 (2)	0·9764 (2)	0·1359 (2)	0·042 (1)
C3	0·1158 (2)	1·1170 (2)	0·0492 (2)	0·046 (1)
C4	−0·0704 (2)	0·8601 (2)	0·0870 (2)	0·045 (1)
O11	0·0169 (2)	0·6485 (2)	0·4098 (2)	0·056 (1)
C12	0·0316 (3)	0·4768 (3)	0·3480 (2)	0·057 (1)
C13	0·0799 (3)	0·6474 (2)	0·5467 (2)	0·057 (1)

Table 2. *Interatomic distances (Å) and angles (°)*

C2—O1	1·379 (2)	C12—O11	1·434 (2)
C3—C2	1·380 (3)	C12—C13″	1·489 (2)
C4—C2	1·385 (2)	C13—O11	1·436 (2)
C3—C4′	1·381 (1)		
C3—C2—O1	118·0 (1)	C2—C3—C4′	120·2 (1)
C4—C2—O1	122·3 (1)	C13—O11—C12	109·6 (1)
C4—C2—C3	119·7 (2)	O11—C12—C13″	109·0 (1)

Symmetry operation: C4′ refers to atom at $-x, 2-y, -z$; C13″ refers to atom at $-x, 1-y, 1-z$.

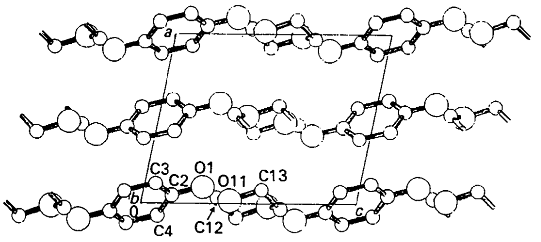

Fig. 1. Hydroquinone–1,4-dioxane complex, showing hydrogen-bonded chains parallel to **c**.

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* 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 52696 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.