feature in all the phenylethanolamines, is also observed here. The Cl^- ion is hydrogen bonded to $H2(N)_{554}$ (2·37 Å), $H3(N)_{555}$ (2·19 Å), and $H(O2)_{655}$ (2·10 Å). The most prominent new feature in the structure of this compound is the involvement of the alcohol H atom [H(O1)] in an intramolecular hydrogen-bonding interaction with the phenolic O(2) atom (2·24 Å). This interaction may account for the observed distortion in some of the dihedral angles.

The conformation of (I) in the crystal is at variance with results obtained using CNDO calculations (Katz *et al.*, 1974). According to these theoretical calculations, the most stable conformer in analogs of (I) is one in which one N proton is hydrogen bonded to the phenolic O. However, recent studies in our laboratories (Makriyannis & Knittel, 1978) using NMR techniques have provided evidence that the conformation observed in the crystal is also the most stable in solution. The discrepancy in the theoretical calculations may thus be due to an overestimation in the strength of the N—H···O(2) hydrogen bond.

It is tempting to suggest that the hydrogen-bonding interaction of the alcohol proton which is unique for *ortho*-substituted hydroxyphenylethanolamines may also be responsible for the lack of pharmacological activity in these compounds by making the alcohol proton unavailable for interaction with the adrenergic site.

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6,13-Pentacenequinone: Molecular Packing Analysis

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Abstract. $C_{22}H_{12}O_2$, monoclinic, $P2_1/b$, $a = 4.951$ (2), $b = 17.784$ (6), $c = 8.170$ (2) Å, $\gamma = 93.26$ (3)°, $V = 718.2$ (3) Å^3, $Z = 2$, $D_x = 1.42$ Mg m^{-3}. The structure was solved by a molecular-packing analysis and refined by least squares to $R = 0.041$. Apart from the carbonyl groups the molecule is strictly planar. However, the *p*-quinone ring adopts the chair-like form with planar >C=O groups inclined to the molecular plane at angles of 3·1°. The bond lengths and angles closely resemble those in unsubstituted *p*-benzoquinone and napththalene structures. The O atoms exhibit increased thermal anisotropy. This fact is discussed in connection with the observed nonplanarity of the central ring.

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Introduction. The X-ray study of 6,13-pentacenequinone (PCQ) has been undertaken in order to provide structural information which will help to relate its spectral-luminescent properties (Shcheglova & Lesnenko, 1971) with its molecular and crystal structure.

Single crystals of PCQ grown from $CHCl_3$ in the form of thin, reddish needles were kindly supplied by Dr N. A. Shcheglova. For the X-ray diffraction experiment a crystal of dimensions $0{\cdot}1 \times 0{\cdot}2 \times 0{\cdot}4$ mm was selected. The unit-cell parameters and the intensity data were measured on a Syntex $P\bar{1}$ automatic diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. The data collection was performed using the $\theta/2\theta$ scanning method up to $s_{max} = 0{\cdot}57$ Å^{-1}. Three standard reflections were checked after every 100 measurements; changes in their intensities were within 1% during the data collection. Of 1131 reflections with $s < 0{\cdot}57$ Å^{-1}, 603 were accepted as observed based on the criterion $I \geq 3\sigma_I$. No absorption corrections were made.

An attempt to solve the structure by direct methods was unsuccessful. Therefore a molecular-packing analysis was performed to determine the Eulerian angles φ, θ, and ψ which describe the orientation of the molecule occupying the symmetry position $\bar{1}$.

An approximate molecular model of PCQ was obtained considering its own symmetry D_{2h}, the values of bond lengths and angles being taken from literature data on quinones and aromatics (Kitaigorodskii, Zorkii & Belsky, 1979). The C—H bond length was taken as 1·08 Å.

The unit-cell parameter γ is very close to 90°, so the idealized Cheshire symmetry group *Pmmm* (Hirshfeld, 1968) was accepted. When combined with the molecular point group D_{2h} centered on a crystal center of symmetry, it produced the asymmetric unit $0 \leq \varphi$, $\theta \leq \pi/2$, $0 \leq \psi < \pi$.

On comparison of the shortest cell dimension a = 4·95 Å with the overall dimensions of the PCQ molecule, it was concluded that the molecules are arranged in stacks along a. If δ is the angle of tilt of the molecular plane to the a axis and d is the 'thickness' of an aromatic molecular layer, then $\cos\delta = d/a$. An average d value of 3·4 Å may be accepted for aromatics (Kitaigorodskii, 1971), hence $\delta \sim 45°$. Thus only three trial models containing the tilt $\delta = 45°$ were chosen in the region $0 \leq \varphi$, $\theta \leq \pi/2$, $0 \leq \psi < \pi$. Their refinement to minimum energy with respect to φ, θ and ψ was then undertaken by the variable-metric method (Fletcher, 1970) using analytical first derivatives.

The quantity minimized was the 'repulsive lattice energy' (Williams, 1969) calculated as a sum of the quadratic functions

$$E_r = \tfrac{1}{2}\sum w(d_o - d_{ij})^2,\ d_{ij} < d_o,$$

where d_{ij} is the non-bonded interatomic distance and w and d_o are empirical parameters which depend on the nature of the ith and jth atoms.

The version of program *PMC* (packing of molecules in crystals) (Dzyabchenko, Belsky & Zorkii, 1979) modified for quadratic potential functions was used throughout the energy calculations using a BESM-6 computer.

As a result of energy calculations three distinct minima with E_r = 11·3, 30·9 and 89·5 kJ mol^{-1} were found. To take into account the correct monoclinic case three additional models were obtained from those found by their 'reflection' in the mirror plane $m \perp a^*$ followed by energy minimization. The latter gave minima with E_r = 26·8, 71·1 and 33·4 kJ mol^{-1}.

The set of non-hydrogen atomic coordinates of the model with the lowest E_r = 11·3 kJ mol^{-1} served as the starting set for a further refinement based on the diffraction data. The full-matrix least-squares program XRAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) was used. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, $w = \sigma_F^{-2}$. Initially, individual isotropic temperature factors were used in the calculations and R dropped to 0·082. A difference Fourier synthesis revealed all the H atoms. Anisotropic refinement was carried out and the final R was 0·041 (R_w = 0·043) for all 603 reflections. The final atomic coordinates and their standard deviations are listed in Table 1.†

Discussion. Interatomic bond distances, valence angles and their standard deviations are presented in Fig. 1.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34464 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates*

	x	y	z
O(1)	0·1084 (7)	−0·0682 (2)	0·2859 (4)
C(1)	0·0636 (8)	−0·0368 (2)	0·1574 (5)
C(2)	−0·1319 (8)	0·0236 (2)	0·1496 (5)
C(3)	−0·1959 (7)	0·0579 (2)	−0·0025 (5)
C(4)	−0·2540 (8)	0·0463 (2)	0·2892 (5)
C(5)	−0·3820 (8)	0·1127 (2)	−0·0055 (5)
C(6)	−0·4444 (8)	0·1023 (2)	0·2898 (6)
C(7)	−0·5096 (8)	0·1363 (2)	0·1380 (5)
C(8)	−0·5723 (9)	0·1256 (2)	0·4332 (5)
C(9)	−0·7009 (9)	0·1926 (2)	0·1364 (5)
C(10)	−0·7575 (10)	0·1795 (3)	0·4264 (6)
C(11)	−0·8201 (9)	0·2131 (2)	0·2771 (7)
H(4)	−0·201 (8)	0·028 (2)	0·387 (5)
H(5)	−0·405 (9)	0·140 (2)	−0·105 (5)
H(8)	−0·529 (9)	0·099 (2)	0·534 (5)
H(9)	−0·752 (9)	0·214 (2)	0·030 (5)
H(10)	−0·837 (9)	0·196 (2)	0·523 (6)
H(11)	−0·963 (8)	0·249 (2)	0·276 (5)

They are in close agreement with values reported for a number of quinones and aromatic structures. The bond-length and -angle pattern of the central *p*-quinone fragment, except for C(2)—C(3), is very similar to that of unsubstituted *p*-benzoquinone (van Bolhuis & Kiers, 1978) as well as those of terminal naphthalene nuclei and naphthalene molecules (Ponomarev, Filipenko & Atovmyan, 1976). Except for the carbonyl groups, the molecule is strictly planar with an r.m.s. deviation of 0·005 Å from the plane through the C atoms of the two naphthalene nuclei. The carbonyl-atom deviations from the plane are 0·030 and 0·095 Å for C(1) and O(1) respectively. The O(1)=C(1)$<^{C(2)}_{C(3)}$ fragment is planar within experimental error; this plane is inclined to the molecular plane at an angle of 3·1°. Thus the *p*-quinone ring has a flattened-chair form. Although of much smaller magnitude, similar molecular-plane distortions have been reported in *p*-benzoquinone itself (van Bolhuis & Kiers, 1978). In two *n*-substituted derivatives of acridone (*N*-methyl and *N*-ethyl), analogous inclinations of the C=O group to the molecular plane have also been found (Dzyabchenko, Zavodnik & Belsky, 1979; Zavodnik, Chetkina & Valkova, 1979).

An *ORTEP* view (Johnson, 1976) of the PCQ molecule is presented in Fig. 2. The thermal ellipsoid of O(1) has an anomalously aspherical form with the longest half-axis of length 0·53 Å, directed almost perpendicularly (86°) to the molecular plane, and two shorter in-plane axes of lengths 0·35 and 0·26 Å [compare with the half-axes of the terminal C(10) and C(11) atoms: 0·29, 0·38 and 0·42 and 0·29, 0·37 and 0·40 Å respectively].

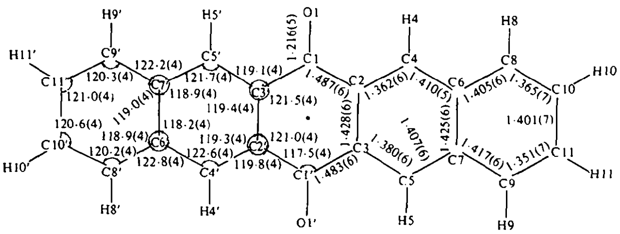

Fig. 1. Bond lengths (Å) and angles (°) with estimated standard deviations in parentheses.

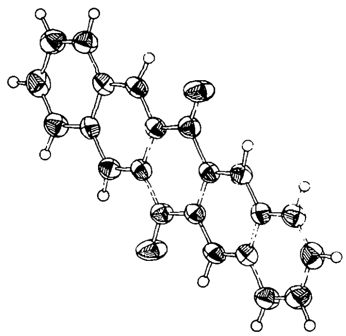
Fig. 2. An *ORTEP* drawing of the PCQ molecule. The ellipsoids are scaled to enclose 50% probability.

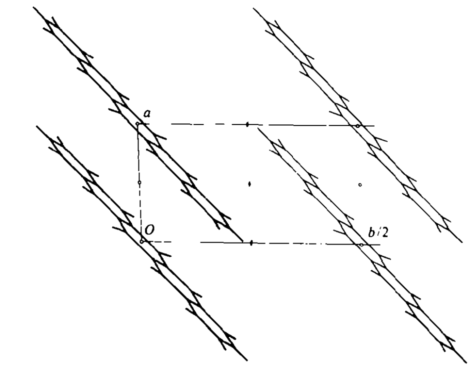

Fig. 3. A view of the structure along [001].

The arrangement of molecules as viewed along [001] is shown in Fig. 3. All the intermolecular distances correspond to normal van der Waals interactions: the shortest O···H distance, 2·63 (4) Å, is observed from atom O(1) to H(8′) of a molecule related by a *c* translation. Hence there is no direct evidence of crystal forces influencing the molecular conformation. However, we believe that the observed non-planarity of the *p*-quinone ring is related to the high thermal anisotropy of O(1) and is caused by the asymmetry of the out-of-plane displacements of C=O groups due to crystal packing. The additional model calculation shows that for the observed packing arrangement the small rotation (less than 15°) of the CO group about the C(2)···C(2′) vector from the planar conformation decreases (~0·105 kJ mol^{-1} deg^{-1}) the intermolecular repulsion E_r when the sign of rotation is consistent with the real case; in contrast, rotation in the opposite direction increases E_r.

We are grateful to Dr A. N. Sobolev for preparation of the *ORTEP* drawings of the PCQ molecule.

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cis,trans-Tetrahydromitchelladione

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Abstract. $C_{30}H_{44}O_2$, M_r = 436·68, orthorhombic, $P2_12_12_1$, a = 18·723 (9), b = 12·334 (6), c = 11·300 (6) Å, Z = 4, D_m = 1·100 (2), D_x = 1·112 Mg m^{-3}. The structure was solved by direct methods with diffractometer data. Refinement of the structure gave a final value of R = 0·073 on F for 1325 non-zero reflections. The sesquiterpene dimer from the wood oil of *Eremophila mitchelli* exhibits a conformation in which the centres of the two halves of the molecule are as close as possible, most probably due to a dipolar interaction between them.

Introduction. From the wood oil of *Eremophila mitchelli*, two related sesquiterpene dimers have been isolated, and their gross structures have been determined by a combination of chemical and spectroscopic techniques (Lewis, 1978). The stereochemistry of the C ring could not, however, be determined, and consequently the structure determination of *cis,trans*-tetrahydromitchelladione [THM(I),† Fig. 1] was undertaken.

A mixture of (I) and (II) was obtained by reduction of the corresponding dihydro compound, and from this mixture suitable crystals of THM(I) were obtained by slow fractional crystallization from petroleum ether. THM(I) crystallized as colourless prisms elongated along c. Accurate cell parameters were obtained from the ω scans of the h00, 0k0 and 00l reflections. The space group $P2_12_12_1$ was assigned from the absence of odd reflections from these classes. Integrated reflection intensities for the hk0–7 layers were collected out to $2\theta = 40°$ using a Stoe automatic Weissenberg diffractometer, fitted with a graphite monochromator, and Mo $K\alpha$ radiation (λ = 0·7107 Å) and ω scans. A standard reflection chosen for each layer was monitored after every 30 measurements and showed no significant change. Of the 1397 independent reflections collected, 72 were designated unobserved by the criterion $I_{\mathbf{h}} < 2\sigma I_{\mathbf{h}}$. The crystal used for data collection measured approximately 0·3 × 0·3 × 0·5 mm. Lorentz and polarization corrections were applied, but absorption corrections were not applied [$\mu_{(\mathrm{Mo}\,K\alpha)}$ = 0·035 mm^{-1}]. The structure was solved using the program *MULTAN* (Germain, Main & Woolfson, 1971). An E map computed from the set of phases with the highest absolute figure of merit (1·19) using 480 reflections with $E \geq 1·0$ revealed 31 of the 32 non-hydrogen atoms. The remaining carbon atom, C(29), was located from a difference map.

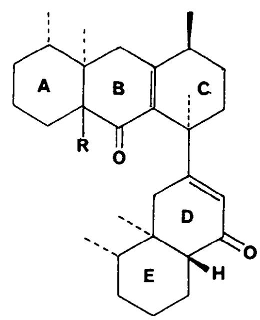

Fig. 1. *cis,trans*-Tetrahydromitchelladione isomers [(I) R = ···H, (II) R = ◀H].

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† The full IUPAC name for THM is (1S,4S,5S,8aS,10aR)-1-[(4′aR,8′S,8′aR)-8′,8′a-dimethyl-4′-oxo-1′,4′,4′a,5′,6′,7′,8′,8′a-octahydronaphthalen-2′-yl]-1,4,5,10a-tetramethyl-1,2,3,4,5,6,7,8,-8a,9,10,10a-dodecahydroanthracen-9-one.

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