Table 3. *Hydrogen-bonding contacts* (Å) *and angles* (°) *in* 6,7-*dimethoxy*-9-*phenyl*-1,4-*phenanthrenequinone*

X—H···Y	X—H	H···Y	X···Y	X—H···Y	Symmetry operation
C(22*A*)—H(222*A*)···O(6*B*)	1·14 (4)	2·57 (4)	3·657 (5)	160 (3)	$1-x, 1-y, 1-z$
C(3*A*)—H(3*A*)···O(6*B*)	0·87 (3)	2·60 (3)	3·417 (4)	156 (4)	$x, y, 1+z$
C(2*B*)—H(2*B*)···O(1*A*)	0·92 (3)	2·48 (3)	3·233 (4)	138 (4)	$1+x, 1+y, z$
C(21*B*)—H(213*B*)···O(4*A*)	0·90 (4)	2·51 (4)	3·307 (4)	147 (4)	$x, y, -1+z$

ecules, the C(22) methyl group twists slightly [−0·118 (4) Å] out of the plane of the aromatic ring system. O(6*B*) and O(7*B*) deviate −0·007 (3) and 0·033 (2) Å from the ring C(5) to C(8), C(13) and C(14).

The phenyl rings are rotated from the phenanthrene rings by 52·7° in molecule *A* and 56·0° in molecule *B* (Fig. 3). Consequently, the packing of the molecules (Fig. 4) in this structure is less dense than in the PQ structures cited above; the calculated densities for those structures vary between 1·485 and 1·405 g cm^{-3}, compared to 1·356 g cm^{-3} for the title compound. The structure is stabilized by very weak C—H···O contacts (Table 3) and van der Waals forces.

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Structure of 2,2-Dimethyl-5-methylaminomethylene-1,3-dioxane-4,6-dione

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Abstract. $C_8H_{11}NO_4$, $M_r = 185·2$, triclinic, $P\bar{1}$, $a = 5·277 (2)$, $b = 8·872 (3)$, $c = 10·123 (3)$ Å, $\alpha = 102·86 (2)$, $\beta = 90·07 (3)$, $\gamma = 102·87 (3)°$, $V = 449·8 (3)$ Å^3, $Z = 2$, $D_m = 1·37$, $D_x = 1·38$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·7107$ Å, $\mu = 0·103$ mm^{-1}, $F(000) = 196$, $T = 300$ K, final $R = 0·080$ for 765 independent observed reflections. Owing to the push–pull effect, the C═C bond distance is as long as 1·399 (10) Å and the torsion angles around this bond are 176·8 (10) and 0·5 (17)°. The 1,3-dioxane ring is in a half-boat conformation due to the equatorial substituents on C(4) and C(6), and also to the resonance system. The molecule has an intramolecular hydrogen bond between the amine and one carbonyl group. The molecules are linked by van der Waals forces.

Introduction. Our studies on the structural chemistry of compounds with push–pull effects have shown that the amine H atom H(1) has a strong proclivity to intramolecular hydrogen bonding. The driving force for such hydrogen bonding can be attributed to six-membered ring formation resulting from a preferred directionality of the sp^2 lone pairs associated with one carbonyl O atom.

A large number of substituted ethylenes are known where the C—C bond is significantly longer than in ethylene and the deviation from planarity is quite

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Table 1. *Atomic and equivalent isotropic thermal parameters*

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

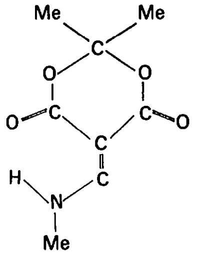
	x	y	z	U_{eq}(Å^2 × 10^3)
O(3)	0·9437 (12)	0·4086 (7)	0·7055 (6)	54 (3)
O(1)	1·1530 (10)	0·1979 (7)	0·6910 (6)	45 (3)
C(5)	0·8793 (17)	0·2517 (11)	0·8721 (8)	45 (4)
O(4)	0·7049 (13)	0·4768 (8)	0·8802 (7)	73 (4)
N	0·6193 (16)	0·2794 (10)	1·0655 (8)	66 (4)
C(6)	1·0589 (17)	0·1619 (11)	0·8065 (9)	49 (4)
O(6)	1·1333 (15)	0·0615 (9)	0·8469 (8)	85 (5)
C(4)	0·8286 (17)	0·3845 (11)	0·8202 (9)	51 (4)
C(2)	1·0058 (17)	0·2747 (11)	0·6148 (9)	48 (4)
C(22)	1·1908 (21)	0·3369 (14)	0·5183 (11)	80 (6)
C(7)	0·7718 (19)	0·2117 (13)	0·9894 (11)	67 (5)
C(21)	0·7649 (19)	0·1567 (12)	0·5483 (10)	62 (4)
C(8)	0·5102 (23)	0·2306 (14)	1·1872 (10)	77 (6)

Table 2. *Bond distances* (Å) *and bond angles* (°)

O(3)—C(4)	1·348 (11)	O(4)—C(4)	1·217 (12)
O(3)—C(2)	1·431 (10)	N—C(7)	1·270 (13)
O(1)—C(6)	1·345 (11)	N—C(8)	1·471 (14)
O(1)—C(2)	1·459 (12)	C(6)—O(6)	1·197 (14)
C(5)—C(6)	1·440 (13)	C(2)—C(22)	1·488 (14)
C(5)—C(4)	1·466 (15)	C(2)—C(21)	1·502 (11)
C(5)—C(7)	1·398 (14)		

C(4)—O(3)—C(2)	117·5 (7)	O(3)—C(4)—O(4)	120·1 (8)
C(6)—O(1)—C(2)	118·7 (6)	O(3)—C(4)—C(5)	116·4 (7)
C(4)—C(5)—C(7)	122·9 (8)	O(3)—C(2)—O(1)	108·9 (6)
C(6)—C(5)—C(7)	116·9 (8)	O(1)—C(2)—C(21)	109·3 (7)
C(6)—C(5)—C(4)	120·0 (7)	O(1)—C(2)—C(22)	105·0 (7)
C(7)—N—C(8)	124·6 (9)	O(3)—C(2)—C(21)	111·5 (7)
O(1)—C(6)—C(5)	116·1 (8)	O(3)—C(2)—C(22)	107·3 (8)
C(5)—C(6)—O(6)	125·7 (8)	C(22)—C(2)—C(21)	114·3 (8)
O(1)—C(6)—O(6)	118·0 (8)	C(5)—C(7)—N	127·2 (10)
C(5)—C(4)—O(4)	123·1 (8)		

appreciable (Adhikesavalu & Venkatesan, 1981; Diánez, López-Castro & Márquez, 1985*a*,*b*, 1988), so the X-ray study of the title compound (1) was undertaken in order to establish unequivocally the molecular conformation and to perform a comparison with previously determined structures.



(1)

Experimental. Density measured by flotation (benzene/CCl$_4$). Colourless crystals with approximate dimensions 0·25 × 0·23 × 0·18 mm. Accurate cell dimensions and crystal orientation matrix were determined by a least-squares treatment of the setting angles of 25 reflections in the range $4 < \theta < 13°$. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, $2\theta_{max} = 42°$. 1251 independent reflections in the range $-5 < h < 5$, $-9 < k < 9$, $0 < l < 11$, of which 765 were considered observed [$I > 2\sigma(I)$]. Two standard reflections ($1\bar{2}\bar{1}$ and $\bar{1}21$) monitored every 100 reflections showed only statistical fluctuations. Intensity corrected for Lp, but absorption and extinction corrections ignored. Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), full-matrix least-squares refinement based on F of the positional and isotropic thermal parameters for non-H atoms, then positional and anisotropic thermal parameters. All H atoms were included at calculated positions and were not refined; further refinement with anisotropic thermal parameters for non-H atoms; and fixed H atoms with isotropic temperature factors corresponding to those of carrier atoms. Final $R = 0{\cdot}080$, $wR = 0{\cdot}080$ [$w = 1/\sigma^2(F)$], $S = 1{\cdot}89$. $\Delta/\sigma_{max} = 0{\cdot}006$. Residual electron density in final difference map showed that $\Delta\rho = \pm 0{\cdot}3$ e Å^{-3}.

The high R factor and e.s.d.'s seem to be due to the poor quality of the crystals.

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 72–80). Calculations performed with the *XRAY* system of crystallographic programs (Stewart, Kundell & Baldwin, 1970) and *PARST* (Nardelli, 1983).

Discussion. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.* Bond distances and bond angles are listed in Table 2. The identification of the atoms and the configuration of the molecule are shown in the *ORTEP*II (Johnson, 1971) drawing of Fig. 1.

As we can see reflected in the molecular dimensions, there is extensive electron delocalization involving the donor N and the acceptor carbonyl

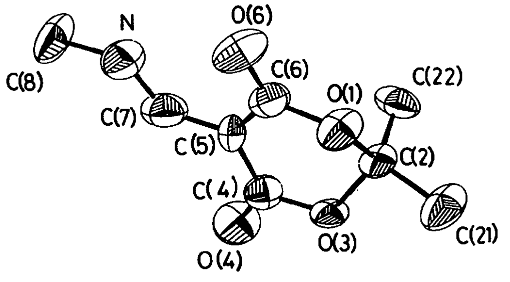


Fig. 1. An *ORTEP* view of the molecule with the atom labelling.

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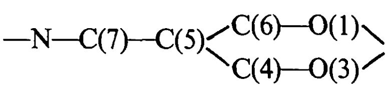
* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53543 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

groups. Bond lengths and angles agree well with those reported for analogous compounds (Vickery, Willey & Drew, 1985) and with those containing analogous resonance systems (Diánez, López-Castro & Márquez, 1987). The ethylene group is planar, the C═C bond distance is as long as 1·399 (10) Å and the torsion angles around this bond are 176·8 (10) and 0·5 (17)°. Angles at C(2) are in the range 105·0 (8)–114·4 (8)°, consistent with the expected high degree of tetrahedral character corresponding to the (sp^3) carbon. The asymmetry (Nardelli, 1983) and puckering parameters (Cremer & Pople, 1975) for the atomic sequence O(1), C(2), O(3), C(4), C(5) and C(6), $\theta = 119\,(1)°$, $\varphi = -117\,(1)°$, $Q = 0{\cdot}42\,(1)$ Å and $\Delta C_s[C(2)] = 0{\cdot}013\,(4)$, show that the molecular conformation of the dioxane ring approximates a 'half-boat' with a mirror plane through the vertex C(2). The substituents O(4), O(2) and C(7) are in equatorial positions. The half-boat conformation of the 1,3-dioxane ring is consistent with the quasi-planarity of resonance systems. The group is



$$\text{—N—C(7)—C(5)}\begin{array}{l}\diagup\text{C(6)—O(1)}\diagdown\\ \diagdown\text{C(4)—O(3)}\diagup\end{array}$$

strictly planar [maximum deviation from the least-squares plane −0·040 (9) Å] and the O(4) and O(6) atoms are at −0·137 (7) and −0·148 (8) Å. The C(8) and C(2) atoms are at 0·016 (12) and 0·597 (9) Å from the same plane.

In addition, the distortion of the dioxane ring from the chair conformation is also due to the O(4) and O(6) substituents in equatorial positions, which agrees with the results of Kok & Romers (1970) and Nader (1975) which showed that equatorial substituents at C(4) and C(6) have a considerable effect on the torsion angles and hence the heterocyclic conformation.

As expected there is an intramolecular H bond between the N atom and a carbonyl oxygen O(4) [N—H···O(4) = 2·811 (12) Å and N—H···O(4) = 127·8 (5)°], showing a chelate structure. There are no intermolecular distances less than the sum of van der Waals radii.

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Structure of 2,6-Dimethoxy-1,4-oxathiane 4,4-Dioxide

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Abstract. $C_6H_{12}O_5S$, $M_r = 196{\cdot}2$, monoclinic, $P2_1/c$, $a = 8{\cdot}473\,(4)$, $b = 7{\cdot}437\,(3)$, $c = 13{\cdot}952\,(2)$ Å, $\beta = 94{\cdot}51\,(2)°$, $V = 876{\cdot}3\,(6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}49$, $D_m = 1{\cdot}48$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}03$ mm^{-1}, $F(000) = 416$, $T = 293$ K, $R = 0{\cdot}038$ for 1307 independent reflections. The oxathiane ring is in

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