of the benzene ring around the C3—C4 single bond. The angle between least-squares planes calculated through the unsaturated chain and benzene ring is 3·96 (19) in (I) and 17·94 (7)° in (II).

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Structure of 2,3-Diphenacylquinoxaline

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Abstract. $C_{24}H_{18}N_2O_2$, $M_r = 366\cdot42$, orthorhombic, *Pbcn*, $a = 23\cdot254$ (6), $b = 11\cdot495$ (3), $c = 6\cdot944$ (2) Å, $V = 1856\cdot2$ (9) Å^3, $Z = 4$, $D_x = 1\cdot31$ g cm^{-3}, λ(Cu $K\alpha$) = $1\cdot54178$ Å, $\mu = 6\cdot356$ cm^{-1}, $F(000) = 768$, $T = 293$ K, $R = 0\cdot048$, $wR = 0\cdot056$ for 1042 unique reflections with $I \geq 2\sigma(I)$. The structure is built up of discrete molecules held together by van der Waals interactions only. The molecule is placed in the special position 4(*c*) possessing crystallographic C_2 symmetry. An intramolecular N—H···O hydrogen bond is present in the structure.

Introduction. The 3,4-dihydroxy-1,6-diphenyl-2,4-hexadiene-1,6-dione (II) is a product of the reaction between acetophenone and diethyl oxalate (Kaitner, Jovanovski & Janev, 1992). This substance reacts with *o*-phenylenediamine (I) and the resulting product is 2,3-diphenacylquinoxaline. It was not possible to distinguish between tautomers (III) and (IV) by infrared spectroscopy. An X-ray structure determination was therefore undertaken.

(III)   (IV)
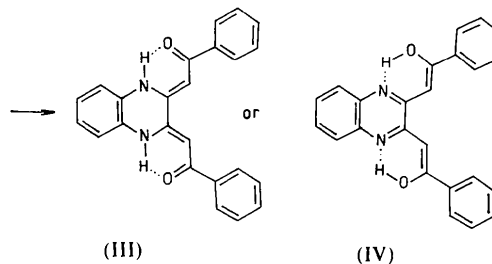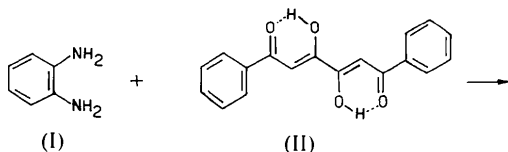
Experimental. The title compound was obtained upon mixing and refluxing ethanol solutions of *o*-phenylenediamine and 3,4-dihydroxy-1,6-diphenyl-2,4-hexadiene-1,6-dione in an equimolar ratio. The microcrystalline product was recrystallized from ethanol; red–orange needle-shaped crystals were used in the structure determination.

Accurate cell dimensions and the crystal orientation matrix were determined on a Philips PW1100 automatic four-circle diffractometer by a least-squares treatment of the setting angles of 16 reflections in the range $7 < \theta < 15°$. Crystal dimensions were $0\cdot15 \times 0\cdot16 \times 0\cdot30$ mm. The intensities of reflections $h\,0$ to 29, $k\,0$ to 15, $l\,0$ to 9 with $2 < \theta < 70°$ were measured using the θ–2θ scan mode; scan width $1\cdot0°$; scan speed $0\cdot04°$ s^{-1}; graphite-monochromatized Cu $K\alpha$ radiation. Intensities of

(I)   +   (II)   ⟶

Table 1. *Final atomic coordinates* ($\times 10^4$, $\times 10^3$ *for HN*) *for refined atoms and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^4$) *for non-hydrogen atoms*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O	1446 (1)	127 (2)	673 (2)	506 (6)
N	550 (1)	1274 (2)	1870 (3)	416 (6)
C1	286 (1)	4417 (2)	2195 (5)	662 (9)
C2	572 (1)	3383 (2)	1906 (4)	542 (9)
C3	286 (1)	2335 (2)	2186 (3)	429 (7)
C4	302 (1)	228 (2)	2147 (3)	372 (6)
C5	598 (1)	−780 (2)	1790 (3)	394 (7)
C6	1179 (1)	−789 (2)	1086 (3)	399 (6)
C7	1477 (1)	−1923 (2)	869 (3)	421 (8)
C8	1934 (1)	−2013 (2)	−443 (4)	532 (9)
C9	2240 (1)	−3038 (3)	−583 (5)	693 (12)
C10	2112 (1)	−3975 (3)	575 (5)	682 (12)
C11	1659 (1)	−3903 (2)	1870 (5)	649 (11)
C12	1345 (1)	−2887 (2)	2000 (4)	527 (9)
HN	89 (1)	124 (2)	120 (5)	—

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

O—C6	1·256 (3)	C4—C5	1·370 (3)
N—C3	1·383 (3)	C5—C6	1·437 (3)
N—C4	1·347 (3)	C6—C7	1·484 (3)
N—HN	0·93 (3)	C7—C8	1·404 (3)
C1—C1^i	1·396 (3)	C7—C12	1·392 (3)
C1—C2	1·377 (3)	C8—C9	1·380 (4)
C2—C3	1·390 (3)	C9—C10	1·377 (5)
C3—C3^i	1·400 (3)	C10—C11	1·387 (4)
C4—C4^i	1·488 (3)	C11—C12	1·380 (3)

C3—N—C4	125·1 (2)	C5—C6—C7	118·7 (2)
C1^i—C1—C2	120·3 (2)	O—C6—C7	118·8 (2)
C1—C2—C3	119·8 (2)	C6—C7—C12	122·6 (2)
N—C3—C2	122·0 (2)	C6—C7—C8	119·0 (2)
C2—C3—C3^i	119·9 (2)	C8—C7—C12	118·3 (2)
N—C3—C3^i	118·1 (2)	C7—C8—C9	119·9 (2)
N—C4—C5	120·9 (2)	C8—C9—C10	121·0 (2)
N—C4—C4^i	116·8 (2)	C9—C10—C11	119·7 (3)
C4^i—C4—C5	122·3 (2)	C10—C11—C12	119·6 (3)
C4—C5—C6	122·7 (2)	C7—C12—C11	121·3 (2)
O—C6—C5	122·5 (2)		

Symmetry code: (i) $-x, y, 0·5 - z$.

three standard reflections measured throughout the data collection showed no evidence of crystal decay. A total of 1049 unique reflections were measured, and 1042 of them with $F_o \geq 2\sigma(F_o)$ were used in structure solution and refinement. Data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved by direct methods. Refinement was by full-matrix least squares (on F) initially with isotropic and then with anisotropic thermal parameters for non-hydrogen atoms. At an intermediate stage of refinement, difference maps showed maxima in positions consistent with the locations of all the hydrogen atoms. The N-hydrogen atom was included in least-squares refinement. All other hydrogen atoms were placed in their geometrically expected positions (C—H 1·08 Å) and included as riding atoms in structure-factor calculations but were not refined. An overall isotropic thermal parameter was allowed and refined for all

hydrogen atoms ($U_{iso} = 0·078$ Å^2). The final cycle of refinement included 132 variable parameters, $R = 0·048$, $wR = 0·056$, goodness of fit $= 0·81$, $w = 1/(\sigma^2 F_o + 0·00588 F_o^2)$; the maximum shift/e.s.d. was $-0·016$ (x coordinate of HN). Largest peaks and valleys in final difference map were $+0·21$ and $-0·25$ e Å^{-3}; there were no chemically significant features. Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–101, 149–150). All calculations were performed on a UNIVAC 1110 computer of the Zagreb University Computing Centre and on an IBM PC/AT compatible microcomputer (microprocessor 80386/25 MHz and mathematical coprocessor 80387) using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreetti, 1987). The *ORTEP*II program (Johnson, 1976) was used to generate the illustration.

Discussion. Final positional and equivalent isotropic thermal parameters are given in Table 1.* Interatomic distances and bond angles are in Table 2. A view of the molecule perpendicular to the plane of the quinoxaline ring showing the atom-numbering

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, least-squares planes and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54370 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
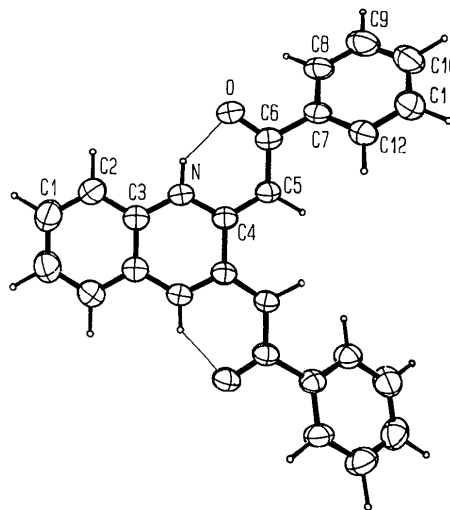

Fig. 1. A general view of the molecule showing the crystallographic numbering scheme. Thermal vibration ellipsoids scaled to 50% probability. Hydrogen atoms are shown as spheres of arbitrary radius. Hydrogen bonds (H···O) are indicated by thin lines.

scheme is given in Fig. 1. The molecules are not linked together by hydrogen bonds. The shortest intermolecular distance N⋯C6^i is 3·320 (3) Å [(i) = x, −y, z − 0·5]. The molecule is in the special position 4(c) of the space group Pbcn possessing crystallographic symmetry C_2. The maximum deviation [0·071 (3) Å] of an atom from the least-squares plane through C1, C2, C3, N, C4, C5, C6, O is that of C2. The dihedral angle between this plane and the least-squares plane through the phenyl ring is 28·89 (5)°. The phenyl group geometries are normal.

There is an intramolecular N—H⋯O hydrogen bond with carbonyl oxygen [O⋯HN 1·85 (3), N⋯O 2·602 (3) Å]. The C=O bond distance of 1·256 (3) Å corresponds to the expected value according to the molecular formula (III) (Stephen & Trotter, 1988; Corkern, Fronczek, Gandour, Guo, Oliver & Watkins, 1988; Kaitner, Jovanovski & Janev, 1992). At the same time the N—C4 bond length of 1·347 (3) Å is very close to the value expected according to the formula (IV) (Wheatley, 1957; Morrow & Huddle, 1972). Some of the bond distances do not strictly belong to either formula (III) or (IV); C4—C5 1·370 (3), C5—C6 1·437 (3) and

N—C3 1·383 (3) Å are midway between limiting values defining the above formulae.

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Structure of 3,5-Di-tert-butyl-10-iodoprotoadamantane

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Abstract. 6,7a-Di-tert-butyl-8-iodooctahydro-2,5-methano-1H-indene, $C_{18}H_{31}I$, $M_r = 374.35$, monoclinic, $P2_1$, $a = 9.095$ (1), $b = 7.087$ (1), $c = 14.149$ (2) Å, $\beta = 107.87$ (3)°, $V = 867.9$ (2) Å^3, $Z = 2$, $D_x = 1.432$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 18.1$ cm^{-1}, $F(000) = 384$, $T = 293$ K, $R = 0.025$ for 1673 observed reflections $[F_o \geq 4\sigma(F_o)]$. The protoadamantane cage has a five-membered ring in the envelope conformation, one six-membered ring (with the I substituent) in a distorted chair conformation and the other six-membered ring (with two tert-butyl groups) in a boat conformation. The I atom is in an axial position. Molecules are joined by van der Waals contacts.

Introduction. Protoadamantanes have been accepted as intermediates in acid-catalyzed multiple rearrangements of diamondoid hydrocarbons (McKervey, 1980). The title compound was obtained by hydriodic-acid-catalyzed rearrangement of di-tert-butyladamantol (Duddeck, McKervey & Rosenbaum, 1990). The starting material and products were characterized by one- and two-dimensional NMR experiments including NOE difference spectra. The X-ray analysis of one of the products, reported in this paper, was undertaken in order to confirm the structure. The results obtained are in agreement with NMR data.

Experimental. Suitable crystals for X-ray structure analysis were grown from ethanol at 277 K over a

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