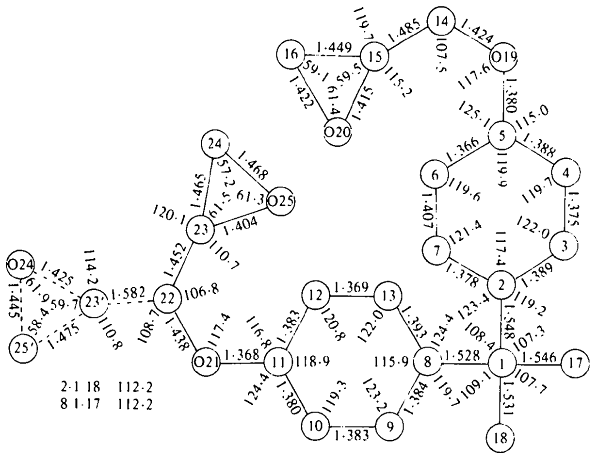

Fig. 2. Bond lengths (Å) and angles (°). For the ordered atoms standard deviations are of the order of 0·005 Å for bond lengths and 0·3° for bond angles. For disordered atoms these values are 0·008 Å and 0·6°.

Bond lengths and angles are illustrated in Fig. 2. Distances and angles in and around the epoxide rings are consistent with those found in other molecules (Foces-Foces, Cano & García-Blanco, 1977; Flippen & Karle, 1976). The CCO angle at the C which is not bonded to the remainder of the molecule is significantly smaller than both the other epoxide angles and the CCC external angle is larger than the external CCO angle. H atoms on the disordered end of the molecule were not very well determined. For the ordered H atoms the average C–H bond length was 0·93 (4) Å with a range from 0·87 (4)–1·13 (4) Å. The molecules are held together solely by van der Waals forces. Intermolecular approaches (excluding H atoms) less than van der Waals distances are C(4)–C(24) at 3·55, C(6)–C(25′) at 3·54 and C(13)–C(25′) at 3·53 Å.

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The Structure of 1,6-Dimethylphenazine: A Photoproduct of 2,2′-Diazido-6,6′-dimethylbiphenyl

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Abstract. $C_{14}H_{12}N_2$, $M_r = 208{\cdot}26$, monoclinic, $P2_1/n$, $a = 11{\cdot}627\,(4)$, $b = 4{\cdot}652\,(2)$, $c = 9{\cdot}826\,(3)$ Å, $\beta = 97{\cdot}95\,(4)°$, $V = 526{\cdot}4$ Å^3, $Z = 2$, $D_m = 1{\cdot}32$, $D_x = 1{\cdot}314$ Mg m^{-3}, μ(Mo $K\alpha$) = 0·085 mm^{-1}. The structure was solved by a direct method and refined by means of the least-squares procedure to a final R value of 0·085 for 1034 non-zero reflections collected by a four-circle diffractometer. The molecules, which have a center of symmetry, are held together by van der Waals interactions. All the atoms of the phenazine ring, together with the methyl C, lie in the same plane.

Introduction. In a study of the photolysis of organic azides, a low-temperature reaction of 2,2′-diazido-1,6-dimethylbiphenyl was undertaken (Yabe, 1980). The structures of the products were characterized by

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spectroscopic methods but were not established exclusively. One of the products was then subjected to X-ray analysis and the structure was determined as 1,6-dimethylphenazine.

The title compound was recrystallized from a benzene solution as yellow plates elongated in the **b** direction. Preliminary Weissenberg photographs showed the space group to be $P2_1/n$. The unit-cell parameters were refined by a least-squares procedure using the 2θ values of 15 reflections measured on a Rigaku four-circle diffractometer. A crystal having the dimensions 0·4 × 0·4 × 0·3 mm was used for the data collection. The intensities were recorded in the θ–2θ scan mode with a speed of $10°\ \mathrm{min}^{-1}$ and range of $(1{\cdot}6 + 0{\cdot}35 \tan\theta)°$ (2θ) (graphite-monochromatized Mo $K\alpha$ radiation). Of the 1205 independent reflections with $2\theta \le 55°$, 1034 were non-zero reflections. These were labelled observed and, after Lorentz and polarization corrections, were used in the refinement of the structure. No corrections for absorption were applied.

The structure was solved by the direct method using the program *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and refined by a block-diagonal least-squares procedure with the program *HBLS* V (Ashida, 1973) with anisotropic temperature factors for the non-H atoms and isotropic temperature factors for the H atoms. In the refinement the function minimized was $\sum w(|F_o| - |F_c|)^2$ with the weighting scheme $w = [\sigma^2(F_o) + a|F_o| + b|F_o|^2]^{-1}$ where $\sigma(F_o)$ is the standard deviation based on the counting statistics. The final refinement ($a = -0{\cdot}074$, $b = 0{\cdot}001$) led to $R = \sum ||F_o| - |F_c|| / \sum |F_o| = 0{\cdot}085$. All scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are shown in Table 1.*

Discussion. The bond lengths and angles of the non-H atoms are shown in Fig. 1, together with the atomic numbering system. The estimated standard deviations of the bond lengths are 0·003–0·004 Å, while those of the bond angles are 0·2–0·3°. The lengths and angles involving the H atoms are shown in Table 2. All the bond lengths and angles are very close to the expected values (*International Tables for X-ray Crystallography*, 1962) and compare well with the corresponding values found in phenazine molecules (Herbstein & Schmidt, 1955*a*,*b*).

The least-squares plane is shown in Table 3. All the atoms of the phenazine ring are coplanar; the average deviation of the ring atoms from the plane is 0·003 Å,

Table 1. *Positional coordinates* ($\times 10^4$; *for* H $\times 10^3$), B_{eq} *values for non-*H *atoms* ($\times 10$) *and isotropic thermal parameters for* H *atoms* ($\times 10$) *with their e.s.d.'s in parentheses*

	x	y	z	B_{eq}*/B (Å^2)
C(1)	429 (1)	1905 (5)	−845 (2)	27 (1)
C(2)	931 (2)	3956 (5)	−1679 (2)	30 (1)
C(3)	1943 (2)	5234 (6)	−1163 (2)	35 (1)
C(4)	2520 (2)	4616 (6)	181 (2)	36 (2)
C(5)	2073 (2)	2675 (5)	985 (2)	33 (1)
C(6)	1007 (1)	1282 (5)	511 (2)	27 (1)
C(7)	331 (2)	4579 (6)	−3105 (2)	41 (2)
N	−573 (1)	623 (4)	−1334 (1)	28 (1)
H(C3)	226 (2)	685 (6)	−174 (3)	21 (6)
H(C4)	325 (2)	555 (7)	53 (3)	24 (6)
H(C5)	245 (2)	212 (7)	189 (3)	18 (5)
H1(C7)	27 (3)	263 (7)	−358 (3)	38 (8)
H2(C7)	−45 (3)	527 (7)	−311 (3)	36 (8)
H3(C7)	71 (3)	609 (9)	−348 (4)	42 (8)

* Defined according to Hamilton (1959).

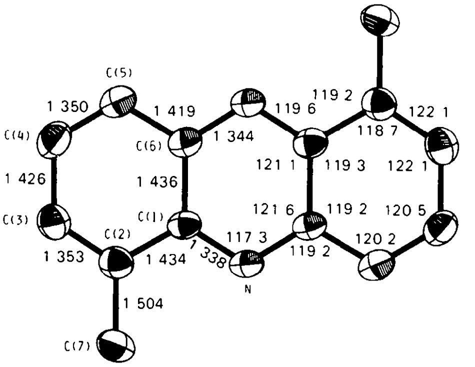

Fig. 1. Molecular structure viewed along the molecular plane normal, with the atomic numbering, bond lengths (Å) and bond angles (°). Thermal ellipsoids indicate 50% probability levels.

Table 2. *Bond lengths* (Å) *and angles* (°) *involving the* H *atoms*

C(3)-H(C3)	1·04 (3)	C(4)-H(C4)	0·98 (3)
C(5)-H(C5)	0·97 (3)	C(7)-H1(C7)	1·02 (4)
C(7)-H2(C7)	0·96 (4)	C(7)-H3(C7)	0·93 (4)
C(2)-C(3)-H(C3)	118 (2)	C(4)-C(3)-H(C3)	120 (2)
C(3)-C(4)-H(C4)	121 (2)	C(5)-C(4)-H(C4)	119 (2)
C(4)-C(5)-H(C5)	123 (2)	C(6)-C(5)-H(C5)	117 (2)
C(2)-C(7)-H1(C7)	104 (2)	C(2)-C(7)-H2(C7)	113 (2)
C(2)-C(7)-H3(C7)	109 (3)	H1(C7)-C(7)-H2(C7)	107 (3)
H2(C7)-C(7)-H3(C7)	104 (4)	H1(C7)-C(7)-H3(C7)	120 (4)

Table 3. *Best plane of the molecule and deviations of atoms* (Å) *from the plane*

Equation: $-0{\cdot}5644X + 0{\cdot}7346Y + 0{\cdot}3766Z + 0{\cdot}0025 = 0$, where X, Y and Z are coordinates referred to the orthogonal axes expressed by $X = ax + cz \cos\beta$, $Y = by$ and $Z = cz \cos\beta$.

C(1)	−0·003 (3)	C(2)	−0·001 (3)	C(3)	0·001 (3)
C(4)	0·006 (3)	C(5)	−0·007 (3)	C(6)	0·006 (3)
N	−0·000 (3)	C(7)*	−0·026 (3)		

* Atom not included in the calculation of the plane.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35963 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

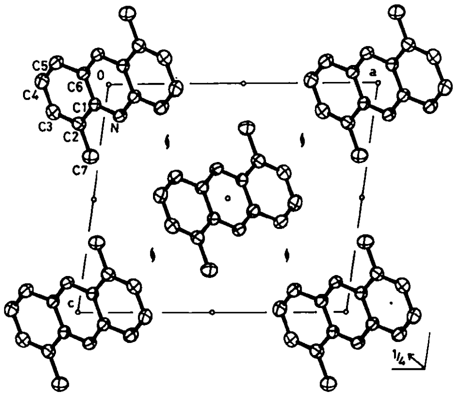

Fig. 2. The molecular arrangements in the crystal viewed along **b**.

with a maximum deviation of 0·007 Å for the C(5) atom. The methyl C atom C(7) attached to the phenazine ring also lies in the same plane with a deviation of 0·026 Å.

The molecular arrangements in the crystal viewed along the *b* axis are shown in Fig. 2. The crystal is built up of individual molecules, with adjacent molecules held together by van der Waals forces.

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(4*R*,5*R*)-1,2-Dithiane-4,5-diol

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Abstract. $C_4H_8O_2S_2$, M_r = 152·24, trigonal, $P3_121$, a = 10·322 (3), c = 10·630 (1) Å, Z = 6, V = 980·2 Å^3, D_c = 1·60, D_m = 1·60 Mg m^{-3}, $F(000)$ = 480, Cu $K\alpha$ radiation. The structure was solved by direct methods and refined by full-matrix least squares to a final R = 0·036 for 73 parameters and 754 observed reflexions. The ring has a chair conformation with a C–S–S–C angle of +58·8°. All molecules are connected by hydrogen bonds.

Introduction. Cyclic disulfides have been extensively used to correlate the chiroptical properties of the disulfide group in the absorption region between 250 and 330 nm with the torsion angle about the S–S bond. 1,2-Dithiane rings, with a fixed asymmetric center near the disulfide group, are sufficiently rigid to allow a direct comparison of the absorption wavelengths and the sign of the CD Cotton effect measured (Carmack & Neubert, 1967; Linderberg & Michl, 1970) in solution with the molecular conformation found in the solid state. In this paper we report the crystal structure of (4*R*,5*R*)-1,2-dithiane-4,5-diol. The compound was prepared by air-oxidation in aqueous solution at pH 8·5 (tris.HCl buffer) and extracted by chloroform. The UV and CD spectra showed the expected absorption band, in the range 280–290 nm, associated with a positive Cotton effect (Carmack & Neubert, 1967; Cleland, 1964).

The crystals were grown from chloroform as transparent prisms elongated along **c**. Systematic absences do not define the space group uniquely and $P3_121$ was established on the basis of the structure determination.

The intensity data were collected on an Enraf–Nonius CAD-4F automatic diffractometer with a crystal approximately 0·10 × 0·11 × 0·30 mm. Unit-cell parameters were refined by the least-squares fit of 18 high-angle reflexions. A $\theta/2\theta$ scanning mode with Ni-filtered Cu $K\alpha$ radiation was used to measure 816 independent reflexions with 2θ values below 135°, 754 of which were considered as observed by the criterion $I > 2{\cdot}5\sigma(I)$, where $\sigma(I)$ was determined from

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