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Structure of (*R*,*R*)-(+)-3,6-Bis[1-hydroxy-1-(4-methylphenyl)ethyl]-1,2,4,5-tetrazine

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Abstract. $C_{20}H_{22}N_4O_2$, $M_r = 350\cdot4$, monoclinic, $P2_1$, $a = 11\cdot322\ (5)$, $b = 5\cdot655\ (3)$, $c = 14\cdot802\ (8)$ Å, $\beta = 96\cdot54\ (5)°$, $U = 941\cdot5$ Å^3, $Z = 2$, $D_x = 1\cdot24$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot048$ mm^{-1}, $F(000) = 372$, $T = 293$ K, $R = 0\cdot071$ for 893 unique reflections. Bond lengths and angles lie within normal ranges. The tetrazine and phenyl rings are planar. The structure contains one probable H bond, O(1*A*)$\cdots$N(4)(x, $1 + y$, z) 3·19 (1) Å.

Introduction. The title compound, m.p. 390–391 K, was prepared from (–)-4-methylatrolactamidinium chloride, $[\alpha]_{5461}$ $-60\cdot0°$ (in water) (Neilson, Mahmood & Watson, 1973). Its intense red colour precluded any measurement of its optical rotation. Some 1,2,4,5-tetrazines have proved of interest in the field of liquid crystal displays (Isenberg, Kruecke, Pelzl, Zashke & Demus, 1983) and our studies hope to show why some tetrazines show these properties and others do not.

Experimental. Crystals grown from water. Intense red prisms elongated along **b**. Crystal dimensions *ca* 0·3 × 0·7 × 0·3 mm. Cell dimensions from Weissenberg photographs and by diffractometry. Data collected in the range $k = 0$–5 from the b axis on a Stoe Stadi II two-circle diffractometer, $0 < \theta < 27°$, Mo $K\alpha$ radiation, range of indices: $-14 < h < 14$, $0 < k < 5$, $-18 < l < 18$. Standard reflections measured every 100 reflections, these varied by less than 5%. No absorption corrections were applied. 3916 reflections measured, 893 unique with $I > 3\sigma(I)$ regarded as observed, $R_{\rm int} = 0\cdot022$. Structure solved by direct methods with *MITHRIL* (Gilmore, 1984) using E values produced by *SHELX76* (Sheldrick, 1976). The resulting E map revealed all the atoms in the molecule. Refinement (on F) by blocked-matrix least squares with *SHELX76* (Sheldrick, 1976). Non-H atoms refined anisotropically; H atoms, attached to C atoms, included at calculated positions; hydroxyl H atoms fixed at positions approximately 0·9 Å from parent atom, these positions being based on significant peaks on difference map. H atoms were given isotropic temperature factors approximately 1·5 times those of their parent atoms. 235 parameters refined, $R = 0\cdot071$, $wR = 0\cdot084$, $w = 2\cdot85/[\sigma(F)^2 + 0\cdot0011F^2]$. Max. $\Delta/\sigma = 0\cdot076$; max. difference map peak 0·35, min. $-0\cdot25$ e Å^{-3}. The highest final difference map peaks lie at approximately 0·5 Å from atom O(1*A*) suggesting that the H atom connected to this atom has a certain amount of rotational disorder (peak used as H atom in the calculation was 50% larger than the other relevant peaks). Other programs used, *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO*

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Table 1. *Coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\text{Å}^2 \times 10^3$) *for non-*H *atoms with e.s.d.'s in parentheses*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N(1)	722 (7)	1468 (16)	1260 (5)	56 (3)
N(2)	−266 (7)	2038 (17)	1624 (5)	56 (3)
N(4)	−829 (8)	−1973	1674 (6)	68 (3)
N(5)	168 (8)	−2554 (17)	1330 (6)	66 (3)
C(3)	−987 (9)	352 (20)	1830 (7)	51 (3)
C(6)	921 (10)	−886 (21)	1138 (7)	54 (3)
C(8*A*)	−2080 (9)	1024 (21)	2282 (6)	58 (3)
C(9*A*)	−3149 (9)	−306 (21)	1867 (6)	70 (3)
O(1*A*)	−2333 (7)	3452 (13)	2080 (5)	23 (2)
C(8*B*)	2077 (9)	−1652 (20)	842 (7)	63 (3)
C(9*B*)	2583 (8)	50 (20)	190 (6)	61 (3)
O(1*B*)	1887 (6)	−3835 (14)	375 (4)	67 (2)
C(1*A*)	−1855 (8)	693 (19)	3344 (7)	56 (3)
C(2*A*)	−2206 (9)	2311 (23)	3928 (7)	69 (4)
C(3*A*)	−2042 (10)	1939 (25)	4889 (7)	77 (4)
C(4*A*)	−1550 (9)	−78 (23)	5247 (7)	63 (4)
C(5*A*)	−1209 (9)	−1748 (25)	4667 (7)	75 (4)
C(6*A*)	−1352 (10)	−1348 (22)	3714 (7)	72 (4)
C(7*A*)	−1392 (10)	−547 (27)	6277 (6)	88 (4)
C(1*B*)	2968 (8)	−2015 (21)	1721 (6)	57 (3)
C(2*B*)	3098 (9)	−261 (22)	2399 (6)	68 (3)
C(3*B*)	3904 (8)	−551 (21)	3183 (7)	65 (3)
C(4*B*)	4592 (9)	−2460 (24)	3325 (7)	63 (3)
C(5*B*)	4510 (8)	−4145 (22)	2640 (9)	78 (4)
C(6*B*)	3711 (9)	−3907 (20)	1841 (7)	68 (4)
C(7*B*)	5454 (8)	−2790 (26)	4175 (7)	88 (4)

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

N(2)–N(1)	1·335 (12)	C(2*A*)–C(1*A*)	1·349 (16)
C(6)–N(1)	1·365 (15)	C(6*A*)–C(1*A*)	1·373 (15)
C(3)–N(2)	1·314 (14)	C(3*A*)–C(2*A*)	1·429 (15)
N(5)–N(4)	1·331 (13)	C(4*A*)–C(3*A*)	1·351 (18)
C(3)–N(4)	1·351 (11)	C(5*A*)–C(4*A*)	1·362 (17)
C(6)–N(5)	1·324 (15)	C(7*A*)–C(4*A*)	1·538 (14)
C(8*A*)–C(3)	1·520 (14)	C(6*A*)–C(5*A*)	1·419 (15)
C(8*B*)–C(6)	1·491 (15)	C(2*B*)–C(1*B*)	1·407 (15)
C(9*A*)–C(8*A*)	1·497 (14)	C(6*B*)–C(1*B*)	1·360 (15)
O(1*A*)–C(8*A*)	1·428 (14)	C(3*B*)–C(2*B*)	1·402 (13)
C(1*A*)–C(8*A*)	1·575 (13)	C(4*B*)–C(3*B*)	1·334 (17)
C(9*B*)–C(8*B*)	1·521 (15)	C(5*B*)–C(4*B*)	1·387 (17)
O(1*B*)–C(8*B*)	1·419 (13)	C(7*B*)–C(4*B*)	1·514 (13)
C(1*B*)–C(8*B*)	1·566 (13)	C(6*B*)–C(5*B*)	1·412 (15)
C(6)–N(1)–N(2)	116·6 (9)	C(1*B*)–C(8*B*)–O(1*B*)	109·7 (8)
C(3)–N(2)–N(1)	119·4 (9)	C(2*A*)–C(1*A*)–C(8*A*)	122·2 (9)
C(3)–N(4)–N(5)	116·2 (8)	C(6*A*)–C(1*A*)–C(8*A*)	120·7 (9)
C(6)–N(5)–N(4)	120·0 (9)	C(6*A*)–C(1*A*)–C(2*A*)	117·0 (10)
N(4)–C(3)–N(2)	124·6 (10)	C(3*A*)–C(2*A*)–C(1*A*)	121·6 (11)
C(8*A*)–C(3)–N(2)	118·7 (10)	C(4*A*)–C(3*A*)–C(2*A*)	121·1 (11)
C(8*A*)–C(3)–N(4)	116·7 (9)	C(5*A*)–C(4*A*)–C(3*A*)	118·2 (10)
N(5)–C(6)–N(1)	123·1 (10)	C(7*A*)–C(4*A*)–C(3*A*)	122·1 (11)
C(8*B*)–C(6)–N(1)	119·1 (10)	C(7*A*)–C(4*A*)–C(5*A*)	119·8 (11)
C(8*B*)–C(6)–N(5)	117·7 (10)	C(6*A*)–C(5*A*)–C(4*A*)	120·5 (12)
C(9*A*)–C(8*A*)–C(3)	110·8 (8)	C(5*A*)–C(6*A*)–C(1*A*)	121·7 (11)
O(1*A*)–C(8*A*)–C(3)	107·6 (8)	C(2*B*)–C(1*B*)–C(8*B*)	120·4 (9)
O(1*A*)–C(8*A*)–C(9*A*)	105·3 (8)	C(6*B*)–C(1*B*)–C(8*B*)	122·8 (9)
C(1*A*)–C(8*A*)–C(3)	111·3 (8)	C(6*B*)–C(1*B*)–C(2*B*)	116·6 (9)
C(1*A*)–C(8*A*)–C(9*A*)	112·4 (8)	C(3*B*)–C(2*B*)–C(1*B*)	120·9 (10)
C(1*A*)–C(8*A*)–O(1*A*)	109·2 (8)	C(4*B*)–C(3*B*)–C(2*B*)	122·6 (10)
C(9*B*)–C(8*B*)–C(6)	114·2 (9)	C(5*B*)–C(4*B*)–C(3*B*)	116·8 (9)
O(1*B*)–C(8*B*)–C(6)	107·9 (8)	C(7*B*)–C(4*B*)–C(3*B*)	122·7 (11)
O(1*B*)–C(8*B*)–C(9*B*)	106·8 (8)	C(7*B*)–C(4*B*)–C(5*B*)	120·4 (11)
C(1*B*)–C(8*B*)–C(6)	107·3 (8)	C(6*B*)–C(5*B*)–C(4*B*)	122·0 (10)
C(1*B*)–C(8*B*)–C(9*B*)	110·9 (8)	C(5*B*)–C(6*B*)–C(1*B*)	120·8 (10)

(Motherwell & Clegg, 1978). Scattering factors from *International Tables for X-ray Crystallography* (1974). No correction for secondary extinction. The rather high *R* value is attributed to the intensity measurement method used.

Discussion. Atomic coordinates are given in Table 1,* with bond lengths and angles in Table 2. The atomic numbering is given in Fig. 1. The bond lengths and angles show no unusual features. The tetrazine ring and the phenyl rings are planar within experimental error. The maximum deviations are tetrazine, 0·02 (2), phenyl *A*, 0·01 (2) and phenyl *B*, 0·02 (2) Å. There is one probable H bond in the structure, O(1*A*)···N(4)(x, $1 + y$, z), 3·19 (1) Å, with O(1*A*)–H = 0·87 (1), H···N(4) = 2·32 (1) Å, angle at H = 179 (1)°. Fig. 2 shows the molecular packing.

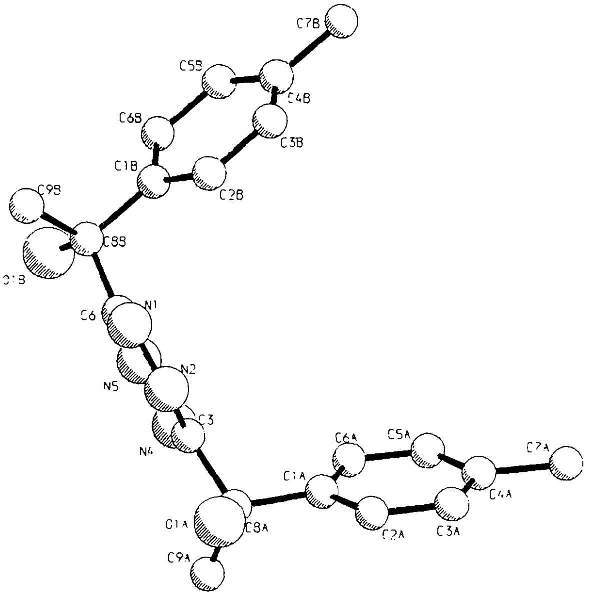

Fig. 1. View of the molecule along **b** showing the atomic numbering.

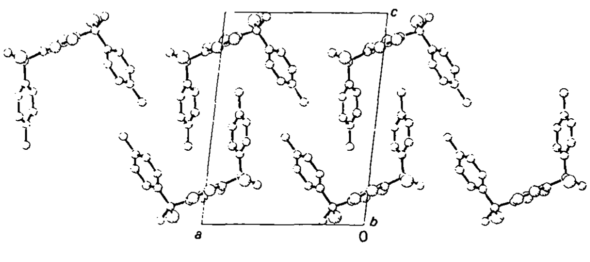

Fig. 2. View of the molecular packing along **b**.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43091 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 2,6-Bis(hydroxymethyl)-4-phenylphenol

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Abstract. $C_{14}H_{14}O_3$, $M_r = 230{\cdot}3$, orthorhombic, *Pcab*, $a = 12{\cdot}489\,(2)$, $b = 21{\cdot}835\,(3)$, $c = 8{\cdot}621\,(1)$ Å, $V = 2350{\cdot}9\,(6)$ Å^3, $Z = 8$, $D_x = 1{\cdot}301$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 5{\cdot}56$ cm^{-1}, $F(000) = 976$, $T = 293$ K, final $R = 0{\cdot}051$ for 2007 observed independent diffractometer-measured intensity data. The two hydroxymethyl groups have different orientations about the ring plane: one O atom is nearly coplanar and the other is tilted out of this plane. The dihedral angle between the two ring planes is $128{\cdot}4\,(3)°$. The molecules are held together by hydrogen bonds forming sheets inter-related by van der Waals bonds as found in 2,6-bis(hydroxymethyl)-4-isopropylphenol.

Introduction. Previous papers reported the crystal and molecular structures of precursors of phenolic resins (Perrin, Lamartine, Vicens, Perrin, Thozet, Hanton & Fugier, 1986; Oehler, Thozet & Perrin, 1985). Of the possible series we have now studied 2,6-bis(hydroxymethyl)phenol substituted in the *para* position; substituents are isopropyl, *tert*-butyl and phenyl groups. These three compounds have been synthesized by chemists and single crystals are available; only the *tert*-butyl derivative gives poor-quality single crystals; however, it was possible to determine the lattice parameters and space groups by film methods. The three compounds are isomorphous as seen in Table 1.

Table 1. *Cell parameters* (Å) *and space groups for* 2,6-*bis*(*hydroxymethyl*)-4-*M*-*phenols*

M = isopropyl	M = *tert*-butyl	M = phenyl
$a = 12{\cdot}897\,(2)$	$a = 13{\cdot}08$	$a = 12{\cdot}489\,(2)$
$b = 18{\cdot}978\,(3)$	$b = 24{\cdot}16$	$b = 21{\cdot}835\,(3)$
$c = 8{\cdot}497\,(3)$	$c = 8{\cdot}84$	$c = 8{\cdot}621\,(1)$
Pcab	*Pcab*	*Pcab*

The structure of the title compound is reported here to ascertain its molecular geometry particularly with reference to the positions of the hydroxymethyl and phenyl groups relative to the phenol ring plane.

Experimental. Suitable crystals obtained from acetone. Plates approximately 0·3 × 0·3 × 0·4 mm used first for film studies of space group (*Pcab*) then for data collection with automatic Nonius CAD-4 four-circle diffractometer; unit-cell parameters refined by a least-squares fit of 25 reflections, $11 < \theta < 48°$; ω–2θ scan technique used to collect intensities of 2753 independent reflections with $\theta > 73°$ (range of *hkl*: $h\,0\rightarrow15$, $k\,0\rightarrow27$, $l\,0\rightarrow10$), 2007 of which considered as observed $[I > 3\sigma(I)]$. One standard reflection measured every 60 min to control the intensity and the same at 100-reflection intervals to control the orientation of the crystal. Lorentz and polarization corrections applied but no absorption correction made. Structure determined by direct methods and refined on F by *SHELX*76 (Sheldrick, 1976). 210 independent parameters; E map showed all non-H atoms of the molecule. H determined by difference Fourier synthesis and introduced into the

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