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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 128·4 (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→15, k 0→27, l 0→10), 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 *SHELX76* (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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refinement. Non-H atoms refined isotropically, then anisotropically; H atoms refined first with fixed, then with isotropic U's. $(\Delta/\sigma)_{\max} = 0{\cdot}101$ for U_{11} of O(61); $(\Delta/\sigma)_{\text{av}} = 0{\cdot}02$. Residual electron density in final difference Fourier synthesis 0·065 e Å^{-3}. Unit weights used, $R = 0{\cdot}051$. Goodness of fit = 0·89. Atomic scattering factors from *SHELX76*. *NRC* programs used (Ahmed, Hall, Pippy & Huber, 1966).*

Discussion. The final positional and thermal parameters are given in Table 2. Fig. 1 is a plot of the molecule by *PLUTO* (Motherwell, 1978) indicating the numbering scheme. Bond distances and angles are listed in Table 3.

Table 2. *Fractional coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2) *for non*-H *atoms*

$B_{\text{eq}} = \frac{8}{3}\pi^2$ trace $\tilde{\text{U}}$.

	x	y	z	B_{eq}
C(1)	3841 (1)	4861 (1)	1842 (2)	2·86 (5)
C(2)	3951 (2)	4281 (1)	2498 (2)	3·17 (5)
C(3)	3853 (2)	3775 (1)	1520 (3)	3·39 (5)
C(4)	3696 (2)	3837 (1)	−63 (2)	3·12 (5)
C(5)	3590 (2)	4426 (1)	−682 (2)	2·95 (5)
C(6)	3642 (1)	4941 (1)	264 (2)	2·71 (5)
C(21)	4235 (2)	4196 (1)	4172 (3)	4·02 (6)
C(41)	3709 (2)	3283 (1)	−1072 (3)	3·41 (5)
C(42)	3088 (2)	2775 (1)	−701 (4)	4·88 (8)
C(43)	3155 (3)	2243 (1)	−1592 (4)	5·90 (9)
C(44)	3829 (3)	2217 (1)	−2837 (4)	5·75 (9)
C(45)	4444 (3)	2717 (1)	−3233 (4)	5·28 (9)
C(46)	4376 (2)	3251 (1)	−2349 (3)	4·14 (7)
C(61)	3523 (2)	5586 (1)	−344 (2)	3·14 (5)
O(61)	3309 (1)	5619 (1)	−1967 (2)	3·45 (4)
O(1)	3887 (1)	5393 (1)	2707 (2)	3·39 (4)
O(21)	5310 (1)	4377 (1)	4482 (2)	4·74 (5)

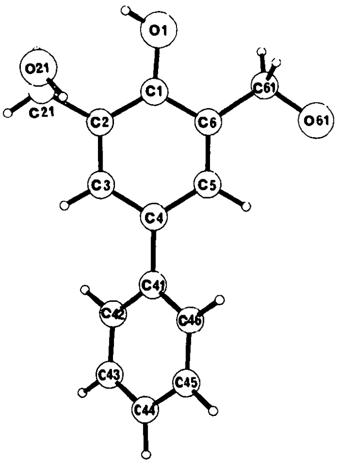

Fig. 1. View perpendicular to the phenolic ring with numbering scheme.

Table 3. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·393 (3)	C(6)–C(61)	1·508 (3)
C(2)–C(3)	1·395 (3)	C(61)–O(61)	1·427 (3)
C(3)–C(4)	1·385 (3)	C(4)–C(41)	1·490 (3)
C(4)–C(5)	1·399 (3)	C(41)–C(42)	1·391 (3)
C(5)–C(6)	1·391 (3)	C(42)–C(43)	1·395 (4)
C(6)–C(1)	1·394 (3)	C(43)–C(44)	1·366 (5)
C(1)–O(1)	1·383 (3)	C(44)–C(45)	1·378 (4)
C(2)–C(21)	1·497 (3)	C(45)–C(46)	1·396 (4)
C(21)–O(21)	1·426 (3)	C(46)–C(41)	1·382 (3)

C(1)–C(2)–C(3)	117·7 (2)	C(3)–C(2)–C(21)	120·4 (2)
C(2)–C(3)–C(4)	122·0 (2)	C(5)–C(6)–C(61)	121·1 (2)
C(3)–C(4)–C(5)	118·6 (2)	C(3)–C(4)–C(41)	119·6 (2)
C(4)–C(5)–C(6)	121·0 (2)	C(5)–C(4)–C(41)	121·6 (2)
C(5)–C(6)–C(1)	118·6 (2)	C(4)–C(41)–C(46)	120·9 (2)
C(6)–C(1)–C(2)	121·9 (2)	C(4)–C(41)–C(42)	120·5 (2)
O(1)–C(1)–C(2)	122·8 (2)	C(41)–C(42)–C(43)	120·3 (3)
C(1)–C(2)–C(21)	121·8 (2)	C(42)–C(43)–C(44)	120·3 (3)
C(2)–C(21)–O(21)	111·7 (2)	C(43)–C(44)–C(45)	120·3 (3)
O(1)–C(1)–C(6)	115·3 (2)	C(44)–C(45)–C(46)	119·6 (3)
C(1)–C(6)–C(61)	118·3 (2)	C(45)–C(46)–C(41)	121·6 (3)
C(6)–C(61)–O(61)	114·0 (2)	C(46)–C(41)–C(42)	118·6 (2)

As usual the regular hexagonal geometry of the benzene ring is slightly modified by substituents; in the phenol ring the four internal angles at the substituted C atoms are less than 120° because of the σ-releasing character of the substituents (Domenicano, Vaciago & Coulson, 1975). On the other hand, the phenyl hexagon is regular except at C(41) probably due to the presence of the phenol group and at C(46) to keep the planarity.

As in other phenols the angles C(2)—C(1)—O(1) and C(6)—C(1)—O(1) have different values, 122·8 (2) and 115·3 (2)°, respectively, the angle larger than 120° being on the side of the phenolic H atom. All C—C and C—O distances are in good agreement with those found in similar products. Some torsional angles have been calculated; among them C(1)—C(6)—C(61)—O(61) and C(1)—C(2)—C(21)—O(21) have values −179·2 (2) and −69·4 (3)° respectively showing that hydroxymethyl C(61)—H_2—O(61) is near the phenol ring plane and that O(21) of the second hydroxymethyl group is out of the ring plane.

The phenol ring is not quite planar ($\chi^2 = 228$): two atoms show a small but significant displacement from the mean plane: C(3) [−0·018 (2)Å] and C(6) [−0·0161 (2) Å] giving a slight boat conformation to the ring. This effect is larger than for the isopropyl derivative ($\chi^2 = 46{\cdot}04$) (Oehler, Thozet & Perrin, 1985).

An important result is the value [128·4 (1)°] of the dihedral angle between the two ring planes corresponding to a geometry quite different from that found for 4-hydroxybiphenyl (Brock & Haller, 1984).

The packing of the molecules is shown in Fig. 2. As in the isopropyl derivative, hydrogen bonds are observed between molecules: they are given in Table 4.

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* Lists of structure factors, anisotropic vibration parameters, H-atom coordinates, torsional angles and deviations of atoms from selected mean planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43174 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

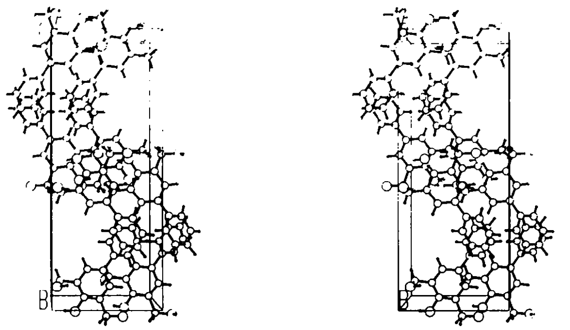

Fig. 2. Stereoscopic view of the structure seen along [001] drawn by *PLUTO*.

Table 4. *Hydrogen bonds* (Å, °)

	D···*A*	*D*—H	H···*A*	O—H···*A*
O(1)—H(11)···O(21)	3·228 (3)	0·90 (4)	2·67 (4)	122 (3)
O(1)—H(11)···O(21^{i})	2·670 (3)	0·90 (3)	1·83 (4)	155 (4)
O(21)—H(21)···O(61^{ii})	2·770 (3)	1·01 (4)	1·79 (4)	163 (4)
O(61)—H(61)···O(1^{iii})	2·801 (3)	1·00 (3)	1·81 (4)	171 (2)

Symmetry code: (i) $1-x$, $1-y$, $1-z$; (ii) $1-x$, $1-y$, $\bar{z}$; (iii) $\frac{1}{2}-x$, y, $z-\frac{1}{2}$.

Atom O(1) is a donor to atom O(21) of the same molecule and atom O(21) of a neighbouring one; the geometry around it suggests an asymmetric bifurcated hydrogen bond. Sheets of molecules are held together by van der Waals interactions.

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Die Struktur von Trimesylhydroxylamin, $(CH_3SO_2)_2NOSO_2CH_3$*

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Abstract. $C_3H_9NO_7S_3$, $M_r = 267{\cdot}30$, monoclinic, $P2_1/c$, $a = 12{\cdot}81$ (1), $b = 7{\cdot}550$ (5), $c = 10{\cdot}251$ (9) Å, $\beta = 92{\cdot}68$ (7)°, $V = 990{\cdot}3$ Å^3, $Z = 4$, $D_m = 1{\cdot}75$, $D_x = 1{\cdot}79$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 7{\cdot}4$ cm^{-1}, $F(000) = 552$, $T = 120$ K, $R = 0{\cdot}032$ for 1922 independent observed reflections. The N atom is pyramidally coordinated. The N—S bond distances are 1·715 (2) and 1·753 (2) Å, respectively. Their difference can be attributed to different conformations of the CH_3SO_2 group with respect to the lone pair on the N atom.

Einleitung. Im Rahmen von Arbeiten zur Struktur und Reaktivität von Mesylhydroxylaminen (Brink & Mattes, 1986) sind wir besonders an den Strukturen vollständig mesylierter Stickstoffverbindungen interessiert, um zu prüfen, wie sich die Mesylierung auf die Koordination des Stickstoffs und auf die Länge der N—S-Bindung auswirkt. Aus diesem Grund haben wir bereits früher die Struktur von Tetramesylhydrazin aufgeklärt (Sucker, Bliefert, Brink & Mattes, 1983). Die Strukturen der vollständig sulfonierten, ionischen Derivate des Ammoniaks und Hydroxylamins sind ebenfalls bekannt: $K_3[(SO_3)_3N].2H_2O$ (Tillack & Kennard, 1970), $K_3[(SO_3)_2NOSO_3].1{,}5H_2O$ (Brown & Strydom, 1977). Die analogen mesylierten Derivate des Ammoniaks bzw. Hydroxylamins $(CH_3SO_2)_3N$ (Blaschette & Wieland, 1983) und $(CH_3SO_2)_2$-$NOSO_2CH_3$ (Boldhaus, Bliefert, Brink & Mattes, 1981) wurden vor kurzem synthetisiert. Wir berichten hier über die Struktur von Trimesylhydroxylamin.

Experimentelles. Darstellung von Trimesylhydroxylamin nach Literaturangeben (Boldhaus, Bliefert, Brink & Mattes, 1981). Dichte mit Schwebemethode. Kristallgröße 0,3 × 0,3 × 0,5 mm. Kristall in Lindemannkapillare; Syntex $P2_1$-Diffraktometer, Graphitmonochromator, Mo $K\alpha$-Strahlung. Gitterkonstanten aus 15 Reflexen ($22 < 2\theta < 32°$); empirische Absorptionskorrektur (ψ scan). 2347 Reflexe gemessen ($2\theta_{\max}$

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