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# Redetermination of 2,4′-methylenediphenol

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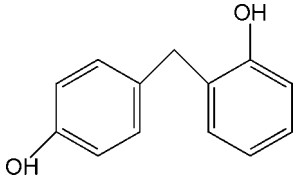
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.043; $wR$ factor = 0.144; data-to-parameter ratio = 13.8.

In the previous determination [Finn & Musti (1950). *J. Soc. Chem. Ind. (London)*, **69**, S849] of the title compound, $C_{13}H_{12}O_2$, the three-dimensional coordinates and displacement parameters were not reported. This redetermination at room temperature reveals that the dihedral angle between the benzene rings is 79.73 (6)°. In the crystal, intermolecular O—H···O hydrogen bonds between adjacent molecules result in two-dimensional wave-like supramolecular motifs parallel to the *ab* plane.

## Related literature

For the previous determination, see: Finn & Musti (1950). For the importance of bisphenol in industry, see: Patel & Patel (2009). For standard bond lengths, see: Allen *et al.* (1987).




## Experimental

### Crystal data

$C_{13}H_{12}O_2$ $M_r$ = 200.23
Monoclinic, $P2_1/c$
$a$ = 5.0923 (5) Å
$b$ = 15.3743 (14) Å
$c$ = 13.2321 (12) Å
$\beta$ = 96.660 (2)°
$V$ = 1028.96 (17) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 296 K
0.30 × 0.28 × 0.26 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min}$ = 0.975, $T_{max}$ = 0.978
5900 measured reflections
1904 independent reflections
1404 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.025

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.144
$S$ = 1.04
1904 reflections
138 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···O2$^{i}$ | 0.82 | 2.04 | 2.859 (2) | 175 |
| O2—H2$A$···O1$^{ii}$ | 0.82 | 2.00 | 2.811 (2) | 173 |

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2* and *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2130).

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# supporting information

*Acta Cryst.* (2011). E**67**, o3143 [https://doi.org/10.1107/S1600536811044989]

## Redetermination of 2,4′-methylenediphenol

**Wei-Dong Peng, Sheng-Chun Chen, Jie An, Fu-An Sun and Qun Chen**

### S1. Comment

2,4′-Dihydroxydiphenylmethane is one isomer of bisphenol F which is an important chemical and/or intermediate for the preparation of useful epoxy resins, phenolic resins, and polycarbonates in the plastic and rubber industries (Patel & Patel, 2009). This structure has been solved previously but with no available three-dimensional coordinates (Finn & Musti, 1950; CSD refcode: ZZZGWU). Herein, we present a redetermination at room temperature of the crystal structure of the title compound (Fig. 1). The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The dihedral angle between the benzene rings is 79.73 (6)°. Intermolecular O—H···O hydrogen bonds between adjacent molecules result in two-dimensional wave-like supramolecular motifs along the *ab* plane (Fig. 2).

### S2. Experimental

A 37% aqueous formaldehyde (20.31 g, 0.25 mol) solution was added to phenol (47.05 g, 0.50 mol) and oxalic acid (0.18 g, 1.40 mmol) at 70 °C with stirring for 4 h. Then the reaction mixture was condensed by vacuum distillation, affording a mixture of 4,4′-methylenebisphenol, 2,4′-methylenebisphenol and 2,2′-methylenebisphenol. By dissolving the resulting mixture (0.50 g) in the mixed solution of 2-propanol (20.0 ml) and water (10.0 ml), the needle colourless single crystals suitable for X-ray analysis were obtained after a slow evaporation of the solvents at room temperature for a period of about two weeks.

### S3. Refinement

All H atoms bound to C atoms were assigned to calculated positions, with C—H = 0.97 Å (methylene) and 0.93 Å (aromatic), and refined using a riding model, with $U_{iso}$(H) = 1.2$U_{eq}$(C). The H atoms of the hydroxyl groups were firstly located in a difference Fourier map and then refined with the distance restraint O—H = 0.820 (1) Å, and finally constrained to ride on the O atom with $U_{iso}$(H) = 1.5$U_{eq}$(O).

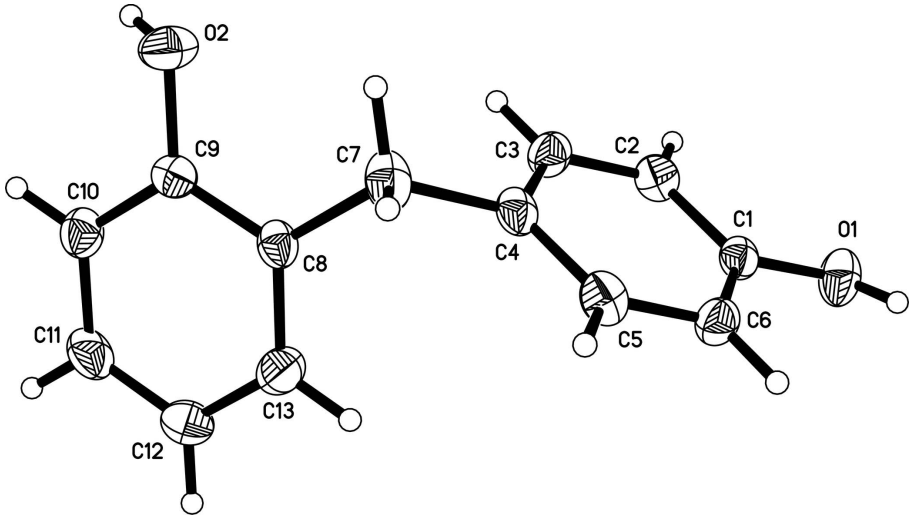


**Figure 1**

The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

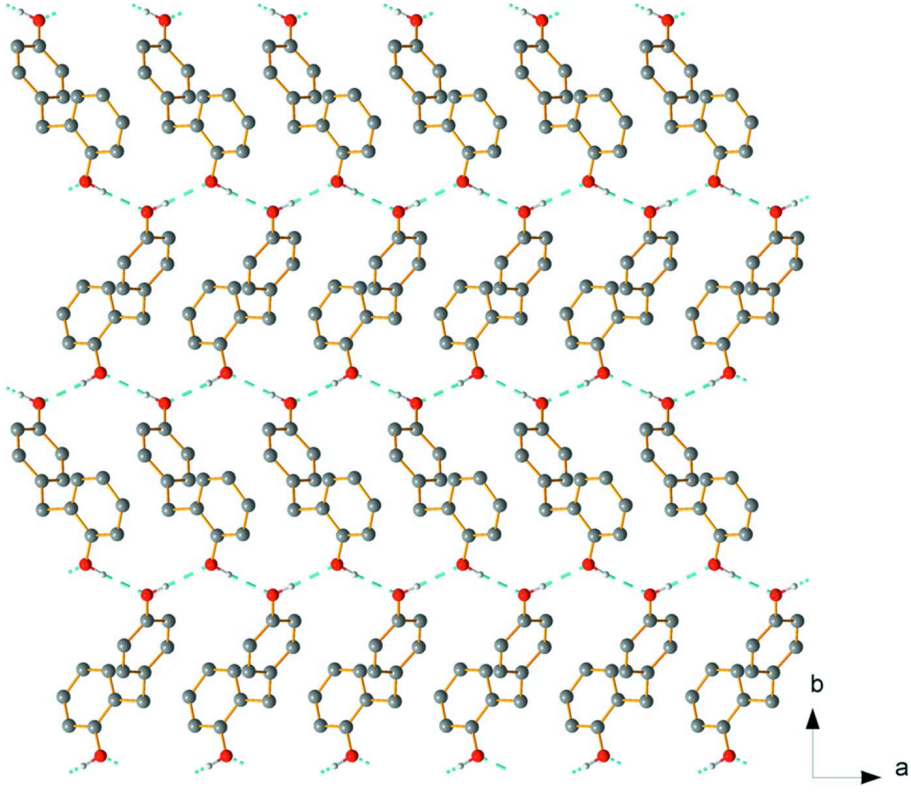


**Figure 2**

The two-dimensional structure of the title compound. Hydrogen atoms are omitted for clarity.

## 2,4′-methylenediphenol

*Crystal data*

$C_{13}H_{12}O_2$
$M_r = 200.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.0923$ (5) Å
$b = 15.3743$ (14) Å
$c = 13.2321$ (12) Å
$\beta = 96.660$ (2)°
$V = 1028.96$ (17) Å$^3$
$Z = 4$
$F(000) = 424$
$D_x = 1.293$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1692 reflections
$\theta = 3.1$–$24.9°$
$\mu = 0.09$ mm$^{-1}$
$T = 296$ K
Block, colourless
0.30 × 0.28 × 0.26 mm

*Data collection*

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min} = 0.975$, $T_{max} = 0.978$
5900 measured reflections
1904 independent reflections
1404 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.0°$
$h = -6 \rightarrow 6$
$k = -16 \rightarrow 18$
$l = -13 \rightarrow 16$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.144$
$S = 1.04$
1904 reflections
138 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.2818P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ e Å$^{-3}$

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.4293 (4) | 1.00937 (12) | 0.83972 (14) | 0.0379 (5) |
| C2 | 0.2466 (4) | 0.94424 (14) | 0.84369 (15) | 0.0441 (5) |
| H2 | 0.1297 | 0.9454 | 0.8927 | 0.053* |
| C3 | 0.2377 (4) | 0.87678 (13) | 0.77407 (15) | 0.0441 (5) |

| | | | | |
|---|---|---|---|---|
| H3 | 0.1138 | 0.8327 | 0.7769 | 0.053* |
| C4 | 0.4087 (4) | 0.87356 (12) | 0.70047 (14) | 0.0393 (5) |
| C5 | 0.5900 (4) | 0.94019 (14) | 0.69828 (15) | 0.0455 (5) |
| H5 | 0.7077 | 0.9392 | 0.6495 | 0.055* |
| C6 | 0.6006 (4) | 1.00814 (13) | 0.76676 (15) | 0.0438 (5) |
| H6 | 0.7227 | 1.0528 | 0.7636 | 0.053* |
| C7 | 0.3999 (4) | 0.79840 (14) | 0.62684 (17) | 0.0548 (6) |
| H7A | 0.3855 | 0.7447 | 0.6644 | 0.066* |
| H7B | 0.5656 | 0.7966 | 0.5974 | 0.066* |
| C8 | 0.1742 (4) | 0.80219 (12) | 0.54106 (14) | 0.0384 (5) |
| C9 | 0.0121 (4) | 0.73108 (12) | 0.51658 (14) | 0.0381 (5) |
| C10 | −0.1909 (4) | 0.73465 (14) | 0.43729 (15) | 0.0469 (5) |
| H10 | −0.2967 | 0.6861 | 0.4214 | 0.056* |
| C11 | −0.2353 (4) | 0.81017 (15) | 0.38226 (16) | 0.0538 (6) |
| H11 | −0.3720 | 0.8128 | 0.3292 | 0.065* |
| C12 | −0.0785 (5) | 0.88160 (15) | 0.40538 (16) | 0.0563 (6) |
| H12 | −0.1087 | 0.9327 | 0.3682 | 0.068* |
| C13 | 0.1238 (4) | 0.87742 (14) | 0.48385 (17) | 0.0514 (6) |
| H13 | 0.2294 | 0.9261 | 0.4990 | 0.062* |
| O1 | 0.4294 (3) | 1.07617 (10) | 0.90993 (12) | 0.0556 (4) |
| H1 | 0.5765 | 1.0985 | 0.9185 | 0.083* |
| O2 | 0.0601 (3) | 0.65582 (10) | 0.57267 (12) | 0.0551 (4) |
| H2A | −0.0787 | 0.6291 | 0.5742 | 0.083* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0381 (10) | 0.0335 (10) | 0.0397 (10) | 0.0055 (8) | −0.0058 (8) | −0.0021 (8) |
| C2 | 0.0392 (10) | 0.0521 (12) | 0.0416 (11) | −0.0007 (9) | 0.0075 (8) | −0.0011 (9) |
| C3 | 0.0379 (11) | 0.0413 (11) | 0.0516 (12) | −0.0055 (8) | −0.0012 (9) | 0.0010 (9) |
| C4 | 0.0345 (10) | 0.0409 (11) | 0.0396 (10) | 0.0086 (8) | −0.0083 (8) | −0.0030 (8) |
| C5 | 0.0349 (10) | 0.0595 (13) | 0.0421 (11) | 0.0011 (9) | 0.0046 (8) | −0.0029 (9) |
| C6 | 0.0379 (10) | 0.0425 (11) | 0.0498 (12) | −0.0069 (8) | 0.0008 (9) | 0.0003 (9) |
| C7 | 0.0519 (13) | 0.0518 (13) | 0.0559 (13) | 0.0186 (10) | −0.0146 (10) | −0.0147 (10) |
| C8 | 0.0382 (10) | 0.0401 (11) | 0.0358 (10) | 0.0070 (8) | −0.0005 (8) | −0.0064 (8) |
| C9 | 0.0383 (10) | 0.0390 (11) | 0.0376 (10) | 0.0056 (8) | 0.0071 (8) | 0.0017 (8) |
| C10 | 0.0421 (11) | 0.0485 (13) | 0.0480 (12) | −0.0038 (9) | −0.0031 (9) | −0.0053 (9) |
| C11 | 0.0525 (13) | 0.0639 (15) | 0.0409 (11) | 0.0062 (11) | −0.0118 (9) | 0.0020 (10) |
| C12 | 0.0654 (15) | 0.0503 (14) | 0.0503 (13) | 0.0064 (11) | −0.0054 (11) | 0.0125 (10) |
| C13 | 0.0530 (13) | 0.0403 (12) | 0.0584 (13) | −0.0035 (10) | −0.0038 (10) | 0.0025 (10) |
| O1 | 0.0550 (9) | 0.0489 (9) | 0.0616 (10) | 0.0010 (7) | 0.0004 (8) | −0.0201 (7) |
| O2 | 0.0504 (9) | 0.0460 (9) | 0.0671 (10) | −0.0005 (7) | −0.0008 (7) | 0.0169 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.372 (3) | C7—H7B | 0.9700 |
| C1—C6 | 1.374 (3) | C8—C9 | 1.386 (3) |
| C1—O1 | 1.385 (2) | C8—C13 | 1.390 (3) |

| | | | |
|---|---|---|---|
| C2—C3 | 1.384 (3) | C9—O2 | 1.381 (2) |
| C2—H2 | 0.9300 | C9—C10 | 1.386 (3) |
| C3—C4 | 1.381 (3) | C10—C11 | 1.375 (3) |
| C3—H3 | 0.9300 | C10—H10 | 0.9300 |
| C4—C5 | 1.382 (3) | C11—C12 | 1.371 (3) |
| C4—C7 | 1.509 (3) | C11—H11 | 0.9300 |
| C5—C6 | 1.380 (3) | C12—C13 | 1.377 (3) |
| C5—H5 | 0.9300 | C12—H12 | 0.9300 |
| C6—H6 | 0.9300 | C13—H13 | 0.9300 |
| C7—C8 | 1.520 (3) | O1—H1 | 0.8200 |
| C7—H7A | 0.9700 | O2—H2A | 0.8200 |

| | | | |
|---|---|---|---|
| C2—C1—C6 | 120.35 (18) | C8—C7—H7B | 108.6 |
| C2—C1—O1 | 117.60 (18) | H7A—C7—H7B | 107.6 |
| C6—C1—O1 | 122.04 (18) | C9—C8—C13 | 117.51 (17) |
| C1—C2—C3 | 119.42 (19) | C9—C8—C7 | 121.52 (18) |
| C1—C2—H2 | 120.3 | C13—C8—C7 | 120.97 (18) |
| C3—C2—H2 | 120.3 | O2—C9—C8 | 118.16 (17) |
| C4—C3—C2 | 121.45 (19) | O2—C9—C10 | 120.67 (18) |
| C4—C3—H3 | 119.3 | C8—C9—C10 | 121.17 (18) |
| C2—C3—H3 | 119.3 | C11—C10—C9 | 119.79 (19) |
| C3—C4—C5 | 117.77 (18) | C11—C10—H10 | 120.1 |
| C3—C4—C7 | 120.60 (19) | C9—C10—H10 | 120.1 |
| C5—C4—C7 | 121.62 (19) | C12—C11—C10 | 120.15 (19) |
| C6—C5—C4 | 121.51 (19) | C12—C11—H11 | 119.9 |
| C6—C5—H5 | 119.2 | C10—C11—H11 | 119.9 |
| C4—C5—H5 | 119.2 | C11—C12—C13 | 119.8 (2) |
| C1—C6—C5 | 119.49 (19) | C11—C12—H12 | 120.1 |
| C1—C6—H6 | 120.3 | C13—C12—H12 | 120.1 |
| C5—C6—H6 | 120.3 | C12—C13—C8 | 121.6 (2) |
| C4—C7—C8 | 114.59 (16) | C12—C13—H13 | 119.2 |
| C4—C7—H7A | 108.6 | C8—C13—H13 | 119.2 |
| C8—C7—H7A | 108.6 | C1—O1—H1 | 109.5 |
| C4—C7—H7B | 108.6 | C9—O2—H2A | 109.5 |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | 0.6 (3) | C4—C7—C8—C13 | 49.1 (3) |
| O1—C1—C2—C3 | 179.23 (16) | C13—C8—C9—O2 | 179.89 (18) |
| C1—C2—C3—C4 | −0.1 (3) | C7—C8—C9—O2 | 0.2 (3) |
| C2—C3—C4—C5 | −0.1 (3) | C13—C8—C9—C10 | 0.7 (3) |
| C2—C3—C4—C7 | 178.59 (17) | C7—C8—C9—C10 | −179.05 (18) |
| C3—C4—C5—C6 | −0.3 (3) | O2—C9—C10—C11 | −179.92 (19) |
| C7—C4—C5—C6 | −178.93 (18) | C8—C9—C10—C11 | −0.7 (3) |
| C2—C1—C6—C5 | −0.9 (3) | C9—C10—C11—C12 | 0.3 (3) |
| O1—C1—C6—C5 | −179.51 (17) | C10—C11—C12—C13 | 0.1 (4) |
| C4—C5—C6—C1 | 0.8 (3) | C11—C12—C13—C8 | −0.1 (4) |
| C3—C4—C7—C8 | 77.4 (3) | C9—C8—C13—C12 | −0.3 (3) |
| C5—C4—C7—C8 | −104.0 (2) | C7—C8—C13—C12 | 179.5 (2) |
| C4—C7—C8—C9 | −131.1 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O2$^{i}$ | 0.82 | 2.04 | 2.859 (2) | 175 |
| O2—H2*A*···O1$^{ii}$ | 0.82 | 2.00 | 2.811 (2) | 173 |

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) −*x*, *y*−1/2, −*z*+3/2.