# organic compounds

## 4,4′,6,6′-Tetra-*tert*-butyl-2,2′-[butane-1,4-diylbis(nitrilomethanylylidene)]-diphenol

## Jia Ti Tee, Norbani Abdullah and Hamid Khaledi*

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The title compound, $C_{34}H_{52}N_2O_2$, is centrosymmetric, the midpoint of the central C—C bond being located on an inversion centre. Intramolecular O—H···N and weak C—H···O hydrogen bonds are observed, but no significant intermolecular interactions occur in the crystal structure.

## Related literature

For structures of some metal complexes of the title Schiff base, see: Doyle *et al.* (2007); Keizer *et al.* (2002*a*,*b*).
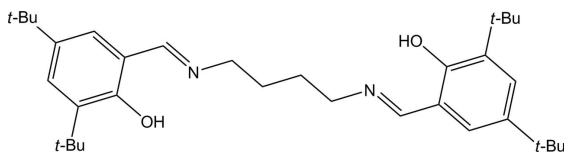


## Experimental

### Crystal data

$C_{34}H_{52}N_2O_2$
$M_r = 520.78$
Monoclinic, $P2_1/c$
$a = 19.1255 (4)$ Å
$b = 9.5702 (2)$ Å
$c = 8.6312 (1)$ Å
$\beta = 90.383 (1)°$

$V = 1579.78 (5)$ Å$^3$
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.07$ mm$^{-1}$
$T = 100$ K
$0.26 \times 0.15 \times 0.06$ mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.983$, $T_{max} = 0.996$

14602 measured reflections
3631 independent reflections
3039 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.114$
$S = 1.03$
3631 reflections
181 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.30$ e Å$^{-3}$
$\Delta\rho_{min} = -0.16$ e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···N1 | 0.927 (16) | 1.735 (17) | 2.5840 (13) | 150.8 (14) |
| C8—H8B···O1 | 0.98 | 2.29 | 2.9546 (16) | 125 |
| C9—H9A···O1 | 0.98 | 2.44 | 3.0720 (15) | 122 |

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97* and *publCIF* (Westrip, 2010).

## References

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Keizer, T. S., De Pue, L. J., Parkin, S. & Atwood, D. A. (2002*a*). *J. Cluster Sci.* **13**, 609–620.
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**supplementary materials**

## 4,4',6,6'-Tetra-*tert*-butyl-2,2'-[butane-1,4-diylbis(nitrilomethanylylidene)]diphenol

**J. T. Tee, N. Abdullah and H. Khaledi**

### Comment

The title Schiff base has been displayed ambidentate ligation behavior towards metal ions (Doyle *et al.*, 2007; Keizer *et al.*, 2002*a*,*b*). Herein, wish to report the crystal structure of the free ligand, obtained through the condensation reaction of 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde and 1,4-diaminobutane. The molecule lies across a crystallographic inversion centre. The imino group is almost coplanar with the phenyl ring [dihedral angle = 3.00 (13)] and adopts an *E* configuration. The hydroxyl group is engaged in an intramolecular O—H···N hydrogen bond with the imine group. Moreover, it acts as an acceptor in two intramolecular C—H···O hydrogen bonds (Table 1). The structure does not display any significant intermolecular interactions.

### Experimental

3,5-Di-*tert*-butyl-2-hydroxybenzaldehyde (5.86 g, 25 mmol) was dissolved in methanol (50 ml) in a round-bottomed flask fitted with a reflux condenser. The solution was heated, followed by portionwise addition of 1,4-diaminobutane (1.10 g; 12.5 mmol). The pale yellow solution formed was then gently refluxed for 3 h. The product obtained on cooling was recrystallized from ethanol at room temperature to give X-ray quality crystals of the title compound.

### Refinement

The C-bound H atoms were placed at calculated positions and refined as riding on their parent atoms, with C–H = 0.95 (aryl), 0.98 (methyl) and 0.99 (methylene) Å. The O-bound H atom was located in a difference Fourier map and refined freely. For all H atoms $U_{iso}(H)$ were set to 1.2–1.5$U_{eq}$(carrier atom).

### Figures
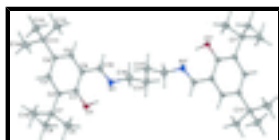


Fig. 1. Molecular structure of the title compound with displacement ellipsoids at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

**4,4',6,6'-Tetra-*tert*-butyl-2,2'-[butane-1,4- diylbis(nitrilomethanylylidene)]diphenol**

*Crystal data*

| | |
|---|---|
| $C_{34}H_{52}N_2O_2$ | $F(000) = 572$ |
| $M_r = 520.78$ | $D_x = 1.095$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 4857 reflections |

$a = 19.1255\ (4)$ Å

$b = 9.5702\ (2)$ Å

$c = 8.6312\ (1)$ Å

$\beta = 90.383\ (1)°$

$V = 1579.78\ (5)$ Å$^3$

$Z = 2$

$\theta = 2.4–30.3°$

$\mu = 0.07$ mm$^{-1}$

$T = 100$ K

Plate, yellow

$0.26 \times 0.15 \times 0.06$ mm

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 3631 independent reflections |
| Radiation source: fine-focus sealed tube | 3039 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.024$ |
| φ and ω scans | $\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | $h = -24 \rightarrow 24$ |
| $T_{min} = 0.983$, $T_{max} = 0.996$ | $k = -12 \rightarrow 12$ |
| 14602 measured reflections | $l = -11 \rightarrow 11$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.114$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.03$ | $w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 0.5079P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 3631 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 181 parameters | $\Delta\rho_{max} = 0.30$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.16$ 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 (Å²)*

|  | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.38365 (4) | 0.98230 (9) | 0.47339 (10) | 0.0267 (2) |
| H1 | 0.4145 (8) | 0.9488 (17) | 0.3998 (19) | 0.040* |
| N1 | 0.43079 (5) | 0.86964 (11) | 0.22286 (12) | 0.0276 (2) |
| C1 | 0.31824 (6) | 0.96886 (11) | 0.41401 (12) | 0.0198 (2) |
| C2 | 0.26044 (6) | 1.01998 (11) | 0.49629 (12) | 0.0193 (2) |
| C3 | 0.19476 (6) | 1.00283 (11) | 0.42796 (12) | 0.0203 (2) |
| H3 | 0.1553 | 1.0373 | 0.4824 | 0.024* |
| C4 | 0.18292 (6) | 0.93820 (12) | 0.28429 (12) | 0.0197 (2) |
| C5 | 0.24129 (6) | 0.88907 (11) | 0.20702 (12) | 0.0196 (2) |
| H5 | 0.2353 | 0.8443 | 0.1096 | 0.024* |
| C6 | 0.30834 (6) | 0.90361 (11) | 0.26843 (12) | 0.0194 (2) |
| C7 | 0.26927 (7) | 1.09122 (12) | 0.65518 (12) | 0.0230 (3) |
| C8 | 0.31935 (8) | 1.21639 (13) | 0.64423 (15) | 0.0348 (3) |
| H8A | 0.3004 | 1.2849 | 0.5707 | 0.052* |
| H8B | 0.3651 | 1.1842 | 0.6085 | 0.052* |
| H8C | 0.3244 | 1.2599 | 0.7465 | 0.052* |
| C9 | 0.29756 (7) | 0.98400 (12) | 0.77258 (13) | 0.0254 (3) |
| H9A | 0.3430 | 0.9492 | 0.7377 | 0.038* |
| H9B | 0.2647 | 0.9058 | 0.7807 | 0.038* |
| H9C | 0.3030 | 1.0286 | 0.8741 | 0.038* |
| C10 | 0.19963 (7) | 1.14567 (14) | 0.71697 (14) | 0.0323 (3) |
| H10A | 0.2074 | 1.1915 | 0.8172 | 0.049* |
| H10B | 0.1672 | 1.0674 | 0.7300 | 0.049* |
| H10C | 0.1797 | 1.2130 | 0.6434 | 0.049* |
| C11 | 0.10988 (6) | 0.92199 (13) | 0.21236 (13) | 0.0246 (3) |
| C12 | 0.09358 (7) | 0.76642 (15) | 0.19055 (17) | 0.0357 (3) |
| H12A | 0.0932 | 0.7200 | 0.2918 | 0.053* |
| H12B | 0.1294 | 0.7237 | 0.1252 | 0.053* |
| H12C | 0.0477 | 0.7560 | 0.1407 | 0.053* |
| C13 | 0.10838 (7) | 0.99276 (15) | 0.05301 (15) | 0.0339 (3) |
| H13A | 0.0630 | 0.9757 | 0.0029 | 0.051* |
| H13B | 0.1457 | 0.9542 | −0.0114 | 0.051* |
| H13C | 0.1154 | 1.0936 | 0.0656 | 0.051* |
| C14 | 0.05296 (7) | 0.9870 (2) | 0.31222 (17) | 0.0449 (4) |
| H14A | 0.0526 | 0.9413 | 0.4138 | 0.067* |
| H14B | 0.0074 | 0.9745 | 0.2615 | 0.067* |
| H14C | 0.0623 | 1.0870 | 0.3255 | 0.067* |
| C15 | 0.36766 (6) | 0.85464 (12) | 0.17746 (13) | 0.0225 (2) |
| H15 | 0.3589 | 0.8100 | 0.0810 | 0.027* |
| C16 | 0.48707 (6) | 0.82786 (14) | 0.11850 (16) | 0.0312 (3) |
| H16A | 0.4676 | 0.7714 | 0.0324 | 0.037* |
| H16B | 0.5214 | 0.7697 | 0.1755 | 0.037* |
| C17 | 0.52336 (6) | 0.95676 (14) | 0.05349 (15) | 0.0294 (3) |
| H17A | 0.5653 | 0.9268 | −0.0045 | 0.035* |
| H17B | 0.5393 | 1.0163 | 0.1407 | 0.035* |

*Atomic displacement parameters (Å²)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0244 (4) | 0.0311 (5) | 0.0246 (4) | −0.0036 (4) | −0.0030 (3) | −0.0032 (3) |
| N1 | 0.0240 (5) | 0.0289 (6) | 0.0299 (5) | −0.0012 (4) | 0.0047 (4) | −0.0003 (4) |
| C1 | 0.0244 (6) | 0.0152 (5) | 0.0196 (5) | −0.0026 (4) | −0.0019 (4) | 0.0027 (4) |
| C2 | 0.0299 (6) | 0.0126 (5) | 0.0155 (5) | 0.0009 (4) | −0.0011 (4) | 0.0015 (4) |
| C3 | 0.0273 (6) | 0.0173 (5) | 0.0163 (5) | 0.0051 (4) | 0.0019 (4) | 0.0010 (4) |
| C4 | 0.0241 (6) | 0.0183 (5) | 0.0167 (5) | 0.0011 (4) | −0.0012 (4) | 0.0022 (4) |
| C5 | 0.0271 (6) | 0.0169 (5) | 0.0149 (5) | −0.0014 (4) | 0.0005 (4) | −0.0011 (4) |
| C6 | 0.0245 (6) | 0.0150 (5) | 0.0188 (5) | −0.0013 (4) | 0.0023 (4) | 0.0014 (4) |
| C7 | 0.0365 (7) | 0.0163 (5) | 0.0163 (5) | 0.0007 (5) | −0.0025 (4) | −0.0011 (4) |
| C8 | 0.0597 (9) | 0.0205 (6) | 0.0241 (6) | −0.0090 (6) | −0.0021 (6) | −0.0033 (5) |
| C9 | 0.0379 (7) | 0.0204 (6) | 0.0178 (5) | 0.0002 (5) | −0.0058 (5) | −0.0004 (4) |
| C10 | 0.0474 (8) | 0.0301 (7) | 0.0195 (5) | 0.0118 (6) | −0.0011 (5) | −0.0064 (5) |
| C11 | 0.0242 (6) | 0.0301 (6) | 0.0194 (5) | 0.0025 (5) | −0.0021 (4) | −0.0005 (4) |
| C12 | 0.0289 (7) | 0.0346 (7) | 0.0434 (8) | −0.0081 (6) | −0.0053 (6) | 0.0044 (6) |
| C13 | 0.0378 (7) | 0.0374 (7) | 0.0263 (6) | −0.0042 (6) | −0.0118 (5) | 0.0057 (5) |
| C14 | 0.0272 (7) | 0.0746 (12) | 0.0327 (7) | 0.0171 (7) | −0.0063 (6) | −0.0116 (7) |
| C15 | 0.0276 (6) | 0.0184 (5) | 0.0215 (5) | −0.0020 (4) | 0.0035 (4) | −0.0001 (4) |
| C16 | 0.0245 (6) | 0.0314 (7) | 0.0377 (7) | 0.0021 (5) | 0.0075 (5) | −0.0009 (5) |
| C17 | 0.0192 (6) | 0.0355 (7) | 0.0334 (6) | −0.0014 (5) | 0.0037 (5) | −0.0032 (5) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.3549 (14) | C9—H9C | 0.9800 |
| O1—H1 | 0.927 (16) | C10—H10A | 0.9800 |
| N1—C15 | 1.2749 (15) | C10—H10B | 0.9800 |
| N1—C16 | 1.4638 (15) | C10—H10C | 0.9800 |
| C1—C2 | 1.4058 (16) | C11—C14 | 1.5257 (18) |
| C1—C6 | 1.4147 (15) | C11—C12 | 1.5325 (18) |
| C2—C3 | 1.3939 (16) | C11—C13 | 1.5332 (16) |
| C2—C7 | 1.5398 (14) | C12—H12A | 0.9800 |
| C3—C4 | 1.4028 (15) | C12—H12B | 0.9800 |
| C3—H3 | 0.9500 | C12—H12C | 0.9800 |
| C4—C5 | 1.3863 (15) | C13—H13A | 0.9800 |
| C4—C11 | 1.5328 (16) | C13—H13B | 0.9800 |
| C5—C6 | 1.3914 (16) | C13—H13C | 0.9800 |
| C5—H5 | 0.9500 | C14—H14A | 0.9800 |
| C6—C15 | 1.4613 (15) | C14—H14B | 0.9800 |
| C7—C10 | 1.5295 (17) | C14—H14C | 0.9800 |
| C7—C8 | 1.5371 (17) | C15—H15 | 0.9500 |
| C7—C9 | 1.5380 (15) | C16—C17 | 1.5244 (18) |
| C8—H8A | 0.9800 | C16—H16A | 0.9900 |
| C8—H8B | 0.9800 | C16—H16B | 0.9900 |
| C8—H8C | 0.9800 | C17—C17$^i$ | 1.525 (3) |
| C9—H9A | 0.9800 | C17—H17A | 0.9900 |

| | | | |
|---|---|---|---|
| C9—H9B | 0.9800 | C17—H17B | 0.9900 |
| C1—O1—H1 | 107.4 (10) | H10A—C10—H10C | 109.5 |
| C15—N1—C16 | 118.61 (11) | H10B—C10—H10C | 109.5 |
| O1—C1—C2 | 120.20 (10) | C14—C11—C12 | 108.67 (12) |
| O1—C1—C6 | 119.71 (10) | C14—C11—C4 | 112.44 (10) |
| C2—C1—C6 | 120.09 (10) | C12—C11—C4 | 109.40 (10) |
| C3—C2—C1 | 117.05 (10) | C14—C11—C13 | 108.51 (11) |
| C3—C2—C7 | 121.48 (10) | C12—C11—C13 | 108.45 (10) |
| C1—C2—C7 | 121.47 (10) | C4—C11—C13 | 109.28 (10) |
| C2—C3—C4 | 124.44 (10) | C11—C12—H12A | 109.5 |
| C2—C3—H3 | 117.8 | C11—C12—H12B | 109.5 |
| C4—C3—H3 | 117.8 | H12A—C12—H12B | 109.5 |
| C5—C4—C3 | 116.69 (10) | C11—C12—H12C | 109.5 |
| C5—C4—C11 | 120.36 (10) | H12A—C12—H12C | 109.5 |
| C3—C4—C11 | 122.95 (10) | H12B—C12—H12C | 109.5 |
| C4—C5—C6 | 121.73 (10) | C11—C13—H13A | 109.5 |
| C4—C5—H5 | 119.1 | C11—C13—H13B | 109.5 |
| C6—C5—H5 | 119.1 | H13A—C13—H13B | 109.5 |
| C5—C6—C1 | 120.00 (10) | C11—C13—H13C | 109.5 |
| C5—C6—C15 | 118.70 (10) | H13A—C13—H13C | 109.5 |
| C1—C6—C15 | 121.27 (10) | H13B—C13—H13C | 109.5 |
| C10—C7—C8 | 107.48 (10) | C11—C14—H14A | 109.5 |
| C10—C7—C9 | 107.51 (10) | C11—C14—H14B | 109.5 |
| C8—C7—C9 | 110.11 (10) | H14A—C14—H14B | 109.5 |
| C10—C7—C2 | 111.77 (10) | C11—C14—H14C | 109.5 |
| C8—C7—C2 | 110.79 (9) | H14A—C14—H14C | 109.5 |
| C9—C7—C2 | 109.11 (9) | H14B—C14—H14C | 109.5 |
| C7—C8—H8A | 109.5 | N1—C15—C6 | 122.40 (10) |
| C7—C8—H8B | 109.5 | N1—C15—H15 | 118.8 |
| H8A—C8—H8B | 109.5 | C6—C15—H15 | 118.8 |
| C7—C8—H8C | 109.5 | N1—C16—C17 | 110.12 (11) |
| H8A—C8—H8C | 109.5 | N1—C16—H16A | 109.6 |
| H8B—C8—H8C | 109.5 | C17—C16—H16A | 109.6 |
| C7—C9—H9A | 109.5 | N1—C16—H16B | 109.6 |
| C7—C9—H9B | 109.5 | C17—C16—H16B | 109.6 |
| H9A—C9—H9B | 109.5 | H16A—C16—H16B | 108.1 |
| C7—C9—H9C | 109.5 | C16—C17—C17$^i$ | 113.31 (13) |
| H9A—C9—H9C | 109.5 | C16—C17—H17A | 108.9 |
| H9B—C9—H9C | 109.5 | C17$^i$—C17—H17A | 108.9 |
| C7—C10—H10A | 109.5 | C16—C17—H17B | 108.9 |
| C7—C10—H10B | 109.5 | C17$^i$—C17—H17B | 108.9 |
| H10A—C10—H10B | 109.5 | H17A—C17—H17B | 107.7 |
| C7—C10—H10C | 109.5 | | |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| O1—H1···N1 | 0.927 (16) | 1.735 (17) | 2.5840 (13) | 150.8 (14) |
| C8—H8B···O1 | 0.98 | 2.29 | 2.9546 (16) | 125. |
| C9—H9A···O1 | 0.98 | 2.44 | 3.0720 (15) | 122. |

**Fig. 1**