

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2,2'-Diisopropoxy-5,5'-methylenedibenzaldehyde

 G. Suresh,^a V. Sabari,^a A. Devaraj,^b M. Bakthadoss^b and S. Aravindhan^{a*}
^aDepartment of Physics, Presidency College (Autonomous), Chennai 600 005, India, and ^bDepartment of Organic Chemistry, University of Madras, Chennai 600 025, India

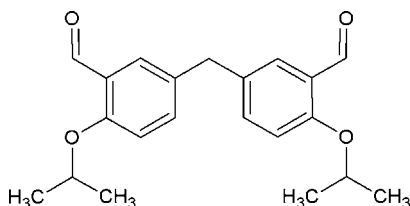
Correspondence e-mail: aravindhanpresidency@gmail.com

Received 19 June 2012; accepted 20 June 2012

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.040; wR factor = 0.114; data-to-parameter ratio = 9.0.

 Molecules of the title compound, $\text{C}_{21}\text{H}_{24}\text{O}_4$, are located on a twofold rotation axis running through the central methylene C atom. The aldehyde group is coplanar with the benzene ring [$\text{C}-\text{C}-\text{O} = 175.7$ (4) °].

Related literature

 For related salicylaldehyde compounds, see: Qiu *et al.* (2009); Yu *et al.* (2007); Wang *et al.* (2009).


Experimental

Crystal data

 $\text{C}_{21}\text{H}_{24}\text{O}_4$
 $M_r = 340.40$

 Orthorhombic, $Fdd2$
 $a = 26.337$ (9) Å
 $b = 28.349$ (10) Å
 $c = 4.990$ (2) Å
 $V = 3726$ (2) Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.08$ mm⁻¹
 $T = 293$ K

 $0.35 \times 0.25 \times 0.20$ mm

Data collection

 Bruker SMART APEXII area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2008)
 $T_{\min} = 0.972$, $T_{\max} = 0.984$

 4329 measured reflections
 1022 independent reflections
 886 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.114$
 $S = 1.06$
 1022 reflections
 114 parameters

 1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.13$ e Å⁻³

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

GS and SA thank Dr Babu Varghese, SAIF, IIT Madras, for the structure refinement clarifications and the Department of Bio Physics, University of Madras, for providing the single-crystal XRD facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5945).

References

- Bruker (2008). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Qiu, L., Lin, J. & Xu, Y. (2009). *Inorg. Chem. Commun.* **12**, 986–989.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Wang, H., Zhang, D., Ni, Z.-H., Li, X., Tian, L. & Jiang, J. (2009). *Inorg. Chem.* **48**, 5946–5956.
- Yu, T.-Z., Su, W.-M., Li, W.-L., Hong, Z.-R., Hua, R.-N. & Li, B. (2007). *Thin Solid Films*, **515**, 4080–4084.

supplementary materials

Acta Cryst. (2012). E68, o2199 [doi:10.1107/S1600536812028048]

2,2'-Diisopropoxy-5,5'-methylenedibenzaldehyde

G. Suresh, V. Sabari, A. Devaraj, M. Bakthadoss and S. Aravindhnan

Comment

Salicylaldehyde and its derivatives are widely used in the construction of metal complexes (Qiu *et al.*, 2009; Wang *et al.*, 2009; Yu *et al.*, 2007).

X-Ray analysis confirms the molecular structure and atom connectivity as illustrated in (Fig. 1). There is one half molecule in the asymmetric unit located on a two-fold rotation axis. The aldehyde group is coplanar with the benzene ring [C2—C1—C8—O2 = 175.7 (4) °].

The crystal packing is stabilized by weak C-H...O hydrogen bonds.

Experimental

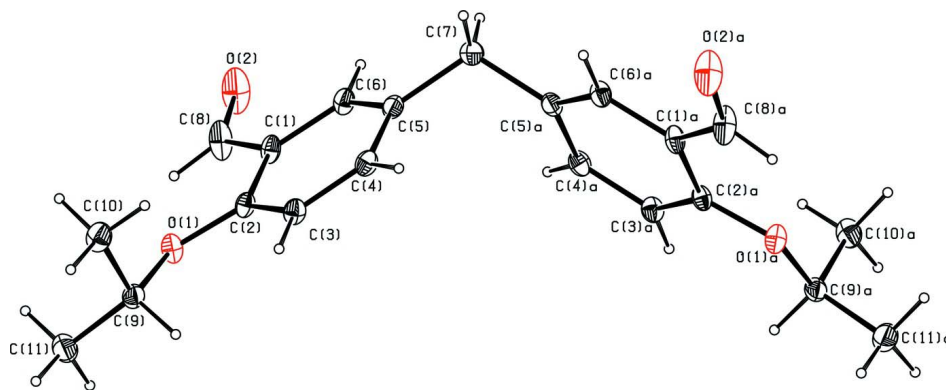
To a stirred solution of 5,5'-methylenebis(2-hydroxybenzaldehyde) (0.50 g, 1.95 mmol) in CH₃CN (20 ml) was added K₂CO₃ (1.35 g, 9.76 mmol) and stirred for 15 min. To this, isopropylbromide (0.40 ml, 4.29 mmol) was added and stirred for 24 h at rt. After completion of the reaction as indicated by TLC, the reaction mixture was concentrated and the resulting crude mass was diluted with water (15 ml) and extracted with ethyl acetate (3 × 15 ml). The combined organic layer was washed with brine (2 × 10 ml) and dried over anhydrous Na₂SO₄. The organic layer was concentrated and the solid thus obtained was washed with ethylacetate-hexanes (1:9) to afford the compound 5,5'-Methylenebis(2-isopropoxybenzaldehyde) as a colourless solid in 95% yield.

Refinement

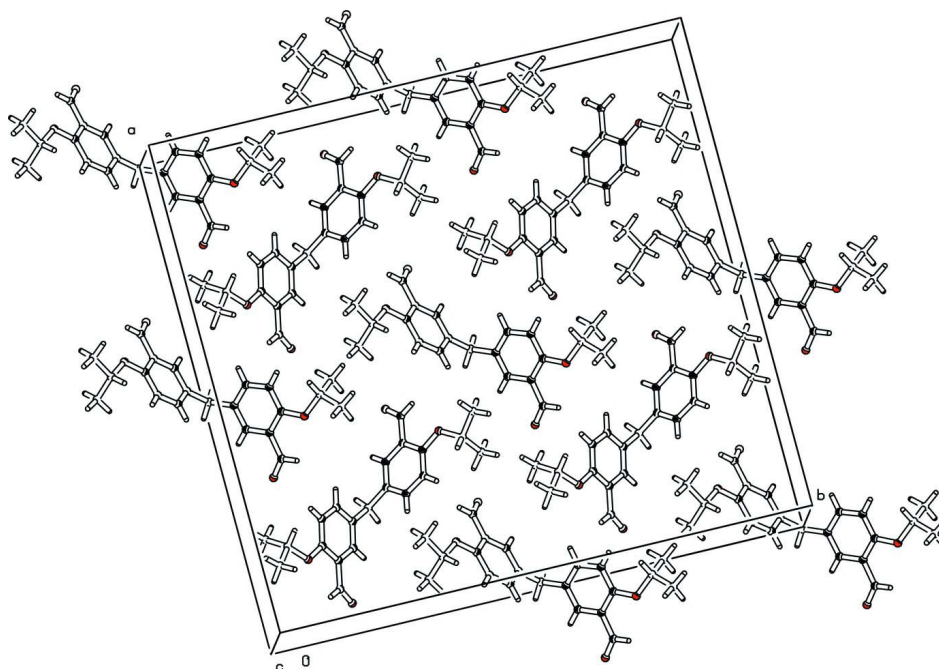
Hydrogen atoms were found in a difference electron density map and subsequently treated as riding atoms with distances of 0.96 Å (CH₃), 0.97 Å (CH₂), 0.98 Å (tertiary CH) or 0.93 Å (aromatic CH). $U_{\text{iso}}(\text{H})$ was set to 1.2 $U_{\text{eq}}(\text{C})$ or 1.5 $U_{\text{eq}}(\text{C}_{\text{methyl}})$, respectively. Due to the absence of anomalous scatterers, Friedel pairs were merged.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINTE* (Bruker, 2008); data reduction: *SAINTE* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 10% probability level.


Figure 2

A view of the crystal packing.

2,2'-Diisopropoxy-5,5'-methylenedibenzaldehyde

Crystal data

$C_{21}H_{24}O_4$

$M_r = 340.40$

Orthorhombic, $Fdd2$

Hall symbol: $F\ 2\ -2d$

$a = 26.337\ (9)\ \text{\AA}$

$b = 28.349\ (10)\ \text{\AA}$

$c = 4.990\ (2)\ \text{\AA}$

$V = 3726\ (2)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1456$

$D_x = 1.214\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1022 reflections

$\theta = 3.1\text{--}26.0^\circ$

$\mu = 0.08\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Orthorhombic, colourless

$0.35 \times 0.25 \times 0.20\ \text{mm}$

Data collection

Bruker SMART APEXII area-detector diffractometer	4329 measured reflections
Radiation source: fine-focus sealed tube	1022 independent reflections
Graphite monochromator	886 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{\text{int}} = 0.035$
Absorption correction: multi-scan (SADABS; Bruker, 2008)	$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 3.1^\circ$
$T_{\text{min}} = 0.972$, $T_{\text{max}} = 0.984$	$h = -32 \rightarrow 32$
	$k = -31 \rightarrow 34$
	$l = -6 \rightarrow 4$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.114$	$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 2.1412P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
1022 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
114 parameters	$\Delta\rho_{\text{max}} = 0.17 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.13 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.15629 (6)	0.10388 (6)	0.7445 (5)	0.0632 (5)	
C4	0.25293 (9)	0.18516 (9)	0.9981 (6)	0.0580 (6)	
H4	0.2873	0.1919	0.9767	0.070*	
C2	0.18085 (8)	0.13881 (8)	0.8776 (6)	0.0499 (6)	
C7	0.2500	0.2500	1.3451 (9)	0.0711 (11)	
H7A	0.2245	0.2642	1.4598	0.085*	0.50
H7B	0.2755	0.2358	1.4598	0.085*	0.50
C1	0.15181 (8)	0.16511 (8)	1.0577 (6)	0.0552 (7)	
C3	0.23209 (8)	0.14955 (8)	0.8487 (6)	0.0572 (7)	
H3	0.2521	0.1326	0.7288	0.069*	
C5	0.22521 (9)	0.21160 (8)	1.1795 (5)	0.0546 (6)	
C6	0.17461 (9)	0.20091 (8)	1.2046 (6)	0.0578 (7)	
H6	0.1549	0.2182	1.3240	0.069*	
C9	0.18476 (10)	0.06998 (9)	0.5895 (6)	0.0621 (7)	
H9	0.2076	0.0865	0.4661	0.075*	
O2	0.07084 (7)	0.17443 (8)	1.2544 (9)	0.1226 (13)	
C10	0.21526 (11)	0.03853 (10)	0.7716 (8)	0.0760 (9)	

H10A	0.2397	0.0571	0.8674	0.114*
H10B	0.2325	0.0151	0.6667	0.114*
H10C	0.1930	0.0233	0.8968	0.114*
C11	0.14556 (13)	0.04302 (12)	0.4324 (8)	0.0882 (11)
H11A	0.1274	0.0643	0.3176	0.132*
H11B	0.1222	0.0281	0.5537	0.132*
H11C	0.1620	0.0194	0.3255	0.132*
C8	0.09748 (9)	0.15523 (10)	1.0940 (10)	0.0888 (13)
H8	0.0828	0.1325	0.9845	0.107*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0503 (9)	0.0492 (9)	0.0902 (13)	-0.0024 (7)	0.0055 (10)	-0.0102 (10)
C4	0.0436 (11)	0.0618 (14)	0.0686 (15)	-0.0084 (10)	0.0086 (12)	0.0086 (14)
C2	0.0445 (11)	0.0388 (11)	0.0663 (15)	0.0025 (9)	0.0052 (12)	0.0066 (11)
C7	0.079 (2)	0.074 (3)	0.060 (2)	-0.0213 (19)	0.000	0.000
C1	0.0430 (11)	0.0383 (11)	0.0844 (19)	0.0028 (9)	0.0120 (13)	0.0036 (13)
C3	0.0477 (12)	0.0544 (14)	0.0695 (16)	-0.0004 (10)	0.0158 (12)	-0.0006 (14)
C5	0.0576 (12)	0.0495 (13)	0.0568 (15)	-0.0065 (10)	0.0027 (12)	0.0081 (12)
C6	0.0560 (12)	0.0444 (12)	0.0730 (18)	0.0018 (10)	0.0181 (13)	0.0005 (14)
C9	0.0704 (15)	0.0549 (14)	0.0609 (15)	-0.0082 (12)	0.0218 (14)	-0.0046 (13)
O2	0.0639 (12)	0.0834 (15)	0.221 (4)	-0.0002 (10)	0.0623 (19)	-0.031 (2)
C10	0.0793 (17)	0.0574 (15)	0.091 (2)	0.0121 (13)	0.0235 (18)	-0.0004 (17)
C11	0.098 (2)	0.082 (2)	0.084 (2)	-0.0235 (17)	0.011 (2)	-0.016 (2)
C8	0.0464 (13)	0.0554 (15)	0.164 (4)	0.0008 (12)	0.024 (2)	-0.018 (2)

Geometric parameters (\AA , $^\circ$)

O1—C2	1.357 (3)	C5—C6	1.372 (3)
O1—C9	1.444 (3)	C6—H6	0.9300
C4—C3	1.370 (4)	C9—C10	1.505 (4)
C4—C5	1.384 (4)	C9—C11	1.505 (4)
C4—H4	0.9300	C9—H9	0.9800
C2—C3	1.391 (3)	O2—C8	1.196 (5)
C2—C1	1.396 (3)	C10—H10A	0.9600
C7—C5 ⁱ	1.514 (4)	C10—H10B	0.9600
C7—C5	1.515 (4)	C10—H10C	0.9600
C7—H7A	0.9700	C11—H11A	0.9600
C7—H7B	0.9700	C11—H11B	0.9600
C1—C6	1.389 (3)	C11—H11C	0.9600
C1—C8	1.469 (3)	C8—H8	0.9300
C3—H3	0.9300		
C2—O1—C9	120.03 (18)	C5—C6—H6	118.9
C3—C4—C5	122.9 (2)	C1—C6—H6	118.9
C3—C4—H4	118.5	O1—C9—C10	110.4 (3)
C5—C4—H4	118.5	O1—C9—C11	105.1 (2)
O1—C2—C3	124.9 (2)	C10—C9—C11	112.3 (2)
O1—C2—C1	116.35 (18)	O1—C9—H9	109.6

C3—C2—C1	118.8 (2)	C10—C9—H9	109.6
C5 ⁱ —C7—C5	113.9 (4)	C11—C9—H9	109.6
C5 ⁱ —C7—H7A	108.8	C9—C10—H10A	109.5
C5—C7—H7A	108.8	C9—C10—H10B	109.5
C5 ⁱ —C7—H7B	108.8	H10A—C10—H10B	109.5
C5—C7—H7B	108.8	C9—C10—H10C	109.5
H7A—C7—H7B	107.7	H10A—C10—H10C	109.5
C6—C1—C2	119.6 (2)	H10B—C10—H10C	109.5
C6—C1—C8	119.7 (3)	C9—C11—H11A	109.5
C2—C1—C8	120.8 (2)	C9—C11—H11B	109.5
C4—C3—C2	119.6 (2)	H11A—C11—H11B	109.5
C4—C3—H3	120.2	C9—C11—H11C	109.5
C2—C3—H3	120.2	H11A—C11—H11C	109.5
C6—C5—C4	116.9 (2)	H11B—C11—H11C	109.5
C6—C5—C7	121.8 (2)	O2—C8—C1	124.6 (3)
C4—C5—C7	121.2 (2)	O2—C8—H8	117.7
C5—C6—C1	122.2 (2)	C1—C8—H8	117.7
C9—O1—C2—C3	9.8 (4)	C5 ⁱ —C7—C5—C6	-122.2 (3)
C9—O1—C2—C1	-169.6 (2)	C5 ⁱ —C7—C5—C4	58.8 (2)
O1—C2—C1—C6	178.9 (2)	C4—C5—C6—C1	0.4 (4)
C3—C2—C1—C6	-0.5 (4)	C7—C5—C6—C1	-178.6 (3)
O1—C2—C1—C8	-0.9 (4)	C2—C1—C6—C5	0.0 (4)
C3—C2—C1—C8	179.7 (3)	C8—C1—C6—C5	179.8 (3)
C5—C4—C3—C2	-0.1 (4)	C2—O1—C9—C10	68.9 (3)
O1—C2—C3—C4	-178.8 (2)	C2—O1—C9—C11	-169.7 (2)
C1—C2—C3—C4	0.6 (4)	C6—C1—C8—O2	-4.1 (6)
C3—C4—C5—C6	-0.4 (4)	C2—C1—C8—O2	175.7 (4)
C3—C4—C5—C7	178.7 (3)		

Symmetry code: (i) $-x+1/2, -y+1/2, z$.

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C8—H8 \cdots O1	0.93	2.42	2.749 (3)	101