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# 1,1′-{2,2′-[1,4-Phenylenebis(methylene)]bis(oxy)bis(2,1-phenylene)}diethanone

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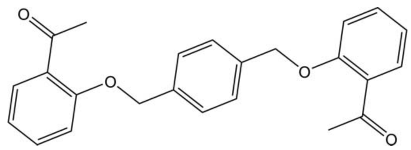
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.035; $wR$ factor = 0.100; data-to-parameter ratio = 15.7.

The asymmetric unit of the title compound, $C_{24}H_{22}O_4$, contains one half-molecule, the other half being generated by a crystallographic center of inversion. The central benzene ring makes a dihedral angle of 72.49 (5)° with the terminal benzene ring. In the crystal, adjacent molecules are linked through C—H⋯O interactions, forming a sheet structure parallel to the *bc* plane. The sheets are stacked along the *a* axis *via* $\pi$–$\pi$ interactions formed between the terminal benzene rings [centroid–centroid separation = 3.7276 (6) Å].

## Related literature

For related structures, see: Hu (2010); Tang *et al.* (2008).



## Experimental

### Crystal data

$C_{24}H_{22}O_4$
$M_r$ = 374.42
Orthorhombic, *Pbca*
$a$ = 6.8490 (1) Å
$b$ = 15.0815 (2) Å
$c$ = 17.8519 (3) Å
$V$ = 1843.98 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 100 K
0.35 × 0.21 × 0.16 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.969, $T_{\max}$ = 0.986
15454 measured reflections
2013 independent reflections
1728 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.029

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.100
$S$ = 1.06
2013 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{\min}$ = −0.21 e Å$^{-3}$

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| C7—H7⋯O1$^{\rm i}$ | 0.95 | 2.56 | 3.4649 (14) | 158 |

Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2792).

## References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hu, T. (2010). *Acta Cryst.* E**66**, o995.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tang, K.-Z., Tang, Y., Li, Y.-F., Liu, W.-S. & Tan, M.-Y. (2008). *Chin. J. Struct. Chem.* **27**, 451–454.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

# supporting information

*Acta Cryst.* (2011). E**67**, o3016 [doi:10.1107/S1600536811042528]

## 1,1′-{2,2′-[1,4-Phenylenebis(methylene)]bis(oxy)bis(2,1-phenylene)}diethanone

**Nassir N. N. Al-Mohammed, Yatimah Alias, Zanariah Abdullah and Hamid Khaledi**

### S1. Comment

The title compound was obtained through the condensation of *α*,*α*′-dibromo-*p*-xylene with two equivalents of 2′-hydroxyacetophenone. The compound has a centrosymmetric molecular structure, the centroid of the central aromatic ring being located on an inversion center. The central aromatic ring makes a dihedral angle of 72.49 (5)° with the terminal rings. This value is comparable to those observed in similar structures (Hu, 2010; Tang *et al.*, 2008). In the crystal, the adjacent molecules are linked through C—H···O interactions (Table 1) to form a sheet parallel to the *bc* plane (Fig. 2). The sheets are connected into a three-dimensional network *via* *π*–*π* interactions formed between the terminal rings in the *a* direction [centroid-centroid separation = 3.7276 (6) Å].

### S2. Experimental

To a suspension of *α*,*α*′-dibromo-*p*-xylene (1 g, 3.8 mmol) and potassium carbonate (1.05 g, 7.6 mmol) in dry acetone (25 ml), 2′-hydroxyacetophenone (1.03 g, 7.6 mmol) was added portionwise and the mixture was refluxed for 48 hr. The solvent was then evaporated under reduced pressure and the crude material was extracted by dichloromethane (3 × 25 ml). The combined organic layers was washed with water and brine and dried over anhydrous sodium sulfate. The solvent was evaporated under vacuum and the formed amorphous solid was re-crystallized from chloroform to obtain off-white crystals of the title compound (m.p. = 435–437 K).

### S3. Refinement

Hydrogen atoms were placed at calculated positions and refined as riding atoms, with C—H distances of 0.95 (aryl), 0.98 (methyl) and 0.99 (methylene) Å, and with $U_{\mathrm{iso}}$(H) set to 1.2 (1.5 for methyl) $U_{\mathrm{eq}}$(C).

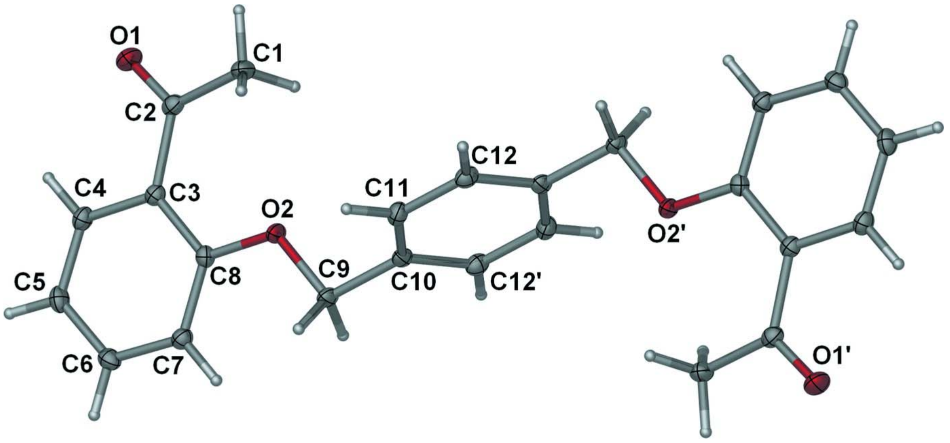

**Figure 1**

Molecular structure of the title compound with displacement ellipsoids drawn at 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. [Symmetry code: ′ = -*x*, -*y*, -*z*.]

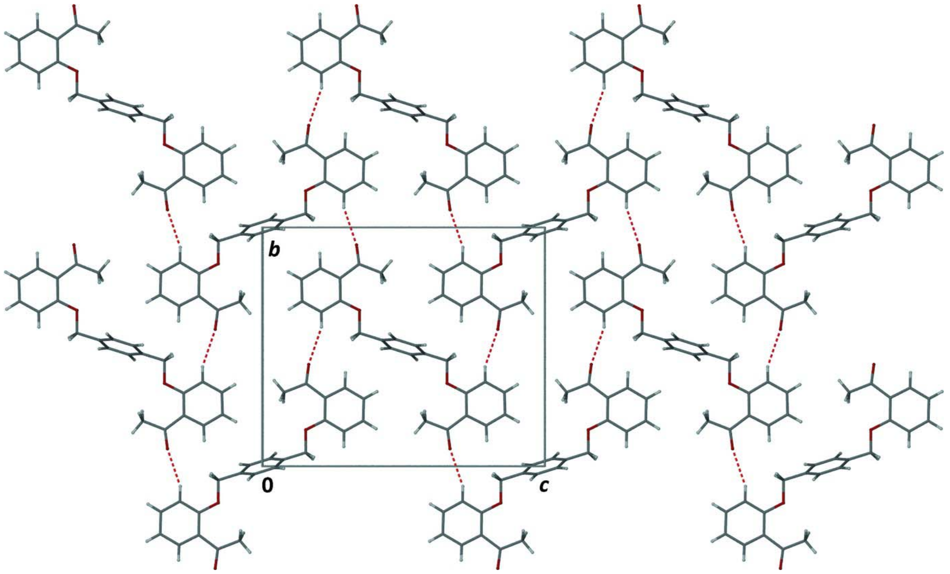

**Figure 2**

The two-dimensional network in the *bc* plane formed by C—H···O interactions (dashed lines).

### 1-(2-{4-[(2-Acetylphenoxy)methyl]benzyloxy}phenyl)ethanone

*Crystal data*

$C_{24}H_{22}O_4$
$M_r$ = 374.42
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a$ = 6.8490 (1) Å
$b$ = 15.0815 (2) Å
$c$ = 17.8519 (3) Å
$V$ = 1843.98 (5) Å$^3$
$Z$ = 4

$F(000)$ = 792
$D_x$ = 1.349 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 4755 reflections
$\theta$ = 2.7–30.5°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 100 K
Block, colorless
0.35 × 0.21 × 0.16 mm

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 15454 measured reflections |
| Radiation source: fine-focus sealed tube | 2013 independent reflections |
| Graphite monochromator | 1728 reflections with $I > 2\sigma(I)$ |
| $\varphi$ and $\omega$ scans | $R_{\text{int}} = 0.029$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | $\theta_{\text{max}} = 27.0°$, $\theta_{\text{min}} = 2.7°$ |
| $T_{\text{min}} = 0.969$, $T_{\text{max}} = 0.986$ | $h = -8\rightarrow 8$ |
| | $k = -19\rightarrow 19$ |
| | $l = -22\rightarrow 22$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.035$ | H-atom parameters constrained |
| $wR(F^2) = 0.100$ | $w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.5451P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.06$ | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 2013 reflections | $\Delta\rho_{\text{max}} = 0.30$ e Å$^{-3}$ |
| 128 parameters | $\Delta\rho_{\text{min}} = -0.21$ e Å$^{-3}$ |
| 0 restraints | |
| 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{\text{iso}}$*/$U_{\text{eq}}$ |
|---|---|---|---|---|
| O1 | 0.04377 (13) | 0.42646 (5) | 0.16533 (5) | 0.0229 (2) |
| O2 | 0.04118 (12) | 0.15272 (5) | 0.15468 (4) | 0.0159 (2) |
| C1 | 0.07382 (18) | 0.31010 (8) | 0.07811 (6) | 0.0203 (3) |
| H1A | −0.0462 | 0.2780 | 0.0654 | 0.031* |
| H1B | 0.1854 | 0.2696 | 0.0751 | 0.031* |
| H1C | 0.0925 | 0.3591 | 0.0428 | 0.031* |
| C2 | 0.05802 (16) | 0.34623 (7) | 0.15636 (6) | 0.0165 (3) |
| C3 | 0.05993 (16) | 0.28692 (7) | 0.22360 (6) | 0.0146 (2) |
| C4 | 0.06897 (16) | 0.32882 (8) | 0.29338 (6) | 0.0171 (3) |
| H4 | 0.0753 | 0.3917 | 0.2950 | 0.020* |
| C5 | 0.06905 (17) | 0.28253 (8) | 0.35995 (7) | 0.0187 (3) |
| H5 | 0.0761 | 0.3130 | 0.4065 | 0.022* |
| C6 | 0.05866 (16) | 0.19058 (8) | 0.35781 (6) | 0.0177 (3) |
| H6 | 0.0586 | 0.1579 | 0.4033 | 0.021* |
| C7 | 0.04836 (16) | 0.14611 (7) | 0.29006 (6) | 0.0160 (2) |
| H7 | 0.0401 | 0.0832 | 0.2893 | 0.019* |

| | | | | |
|---|---|---|---|---|
| C8 | 0.05005 (15) | 0.19348 (7) | 0.22261 (6) | 0.0140 (2) |
| C9 | 0.02559 (17) | 0.05749 (7) | 0.15472 (6) | 0.0162 (2) |
| H9A | −0.0921 | 0.0387 | 0.1828 | 0.019* |
| H9B | 0.1417 | 0.0308 | 0.1789 | 0.019* |
| C10 | 0.01168 (16) | 0.02795 (7) | 0.07449 (6) | 0.0152 (2) |
| C11 | 0.17455 (17) | 0.03233 (7) | 0.02807 (6) | 0.0177 (3) |
| H11 | 0.2946 | 0.0543 | 0.0472 | 0.021* |
| C12 | 0.16313 (17) | 0.00478 (7) | −0.04609 (6) | 0.0172 (3) |
| H12 | 0.2750 | 0.0083 | −0.0774 | 0.021* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0305 (5) | 0.0127 (4) | 0.0254 (4) | 0.0002 (3) | −0.0012 (4) | 0.0010 (3) |
| O2 | 0.0239 (4) | 0.0099 (4) | 0.0138 (4) | −0.0011 (3) | −0.0001 (3) | −0.0015 (3) |
| C1 | 0.0278 (6) | 0.0157 (5) | 0.0176 (6) | −0.0019 (4) | −0.0012 (5) | 0.0030 (4) |
| C2 | 0.0145 (5) | 0.0141 (5) | 0.0210 (6) | −0.0010 (4) | −0.0013 (4) | 0.0016 (4) |
| C3 | 0.0131 (5) | 0.0136 (5) | 0.0171 (6) | 0.0001 (4) | −0.0001 (4) | −0.0005 (4) |
| C4 | 0.0172 (5) | 0.0133 (5) | 0.0207 (6) | 0.0000 (4) | −0.0002 (4) | −0.0032 (4) |
| C5 | 0.0186 (6) | 0.0213 (6) | 0.0162 (6) | −0.0008 (4) | −0.0001 (4) | −0.0056 (4) |
| C6 | 0.0174 (5) | 0.0208 (6) | 0.0150 (6) | −0.0007 (4) | 0.0002 (4) | 0.0019 (4) |
| C7 | 0.0165 (5) | 0.0134 (5) | 0.0182 (6) | −0.0001 (4) | −0.0005 (4) | 0.0000 (4) |
| C8 | 0.0127 (5) | 0.0147 (5) | 0.0147 (5) | 0.0003 (4) | −0.0004 (4) | −0.0022 (4) |
| C9 | 0.0229 (6) | 0.0097 (5) | 0.0159 (5) | 0.0000 (4) | −0.0001 (4) | −0.0002 (4) |
| C10 | 0.0220 (6) | 0.0082 (5) | 0.0153 (5) | 0.0018 (4) | −0.0010 (4) | −0.0005 (4) |
| C11 | 0.0177 (6) | 0.0150 (5) | 0.0204 (6) | −0.0019 (4) | −0.0020 (4) | −0.0019 (4) |
| C12 | 0.0191 (6) | 0.0134 (5) | 0.0193 (6) | −0.0001 (4) | 0.0028 (4) | −0.0008 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.2246 (13) | C6—C7 | 1.3846 (15) |
| O2—C8 | 1.3609 (13) | C6—H6 | 0.9500 |
| O2—C9 | 1.4401 (12) | C7—C8 | 1.4002 (15) |
| C1—C2 | 1.5033 (15) | C7—H7 | 0.9500 |
| C1—H1A | 0.9800 | C9—C10 | 1.5029 (15) |
| C1—H1B | 0.9800 | C9—H9A | 0.9900 |
| C1—H1C | 0.9800 | C9—H9B | 0.9900 |
| C2—C3 | 1.4970 (15) | C10—C12$^{i}$ | 1.3907 (16) |
| C3—C4 | 1.3982 (15) | C10—C11 | 1.3912 (16) |
| C3—C8 | 1.4109 (15) | C11—C12 | 1.3897 (16) |
| C4—C5 | 1.3783 (16) | C11—H11 | 0.9500 |
| C4—H4 | 0.9500 | C12—C10$^{i}$ | 1.3908 (16) |
| C5—C6 | 1.3892 (16) | C12—H12 | 0.9500 |
| C5—H5 | 0.9500 | | |
| | | | |
| C8—O2—C9 | 116.96 (8) | C6—C7—C8 | 120.24 (10) |
| C2—C1—H1A | 109.5 | C6—C7—H7 | 119.9 |
| C2—C1—H1B | 109.5 | C8—C7—H7 | 119.9 |

| | | | |
|---|---|---|---|
| H1A—C1—H1B | 109.5 | O2—C8—C7 | 122.38 (9) |
| C2—C1—H1C | 109.5 | O2—C8—C3 | 117.68 (9) |
| H1A—C1—H1C | 109.5 | C7—C8—C3 | 119.95 (9) |
| H1B—C1—H1C | 109.5 | O2—C9—C10 | 107.45 (8) |
| O1—C2—C3 | 119.09 (10) | O2—C9—H9A | 110.2 |
| O1—C2—C1 | 119.05 (10) | C10—C9—H9A | 110.2 |
| C3—C2—C1 | 121.86 (9) | O2—C9—H9B | 110.2 |
| C4—C3—C8 | 117.69 (10) | C10—C9—H9B | 110.2 |
| C4—C3—C2 | 116.40 (10) | H9A—C9—H9B | 108.5 |
| C8—C3—C2 | 125.90 (10) | C12$^{\rm i}$—C10—C11 | 119.34 (10) |
| C5—C4—C3 | 122.64 (10) | C12$^{\rm i}$—C10—C9 | 120.48 (10) |
| C5—C4—H4 | 118.7 | C11—C10—C9 | 120.18 (10) |
| C3—C4—H4 | 118.7 | C12—C11—C10 | 120.54 (11) |
| C4—C5—C6 | 118.80 (10) | C12—C11—H11 | 119.7 |
| C4—C5—H5 | 120.6 | C10—C11—H11 | 119.7 |
| C6—C5—H5 | 120.6 | C11—C12—C10$^{\rm i}$ | 120.12 (11) |
| C7—C6—C5 | 120.68 (10) | C11—C12—H12 | 119.9 |
| C7—C6—H6 | 119.7 | C10$^{\rm i}$—C12—H12 | 119.9 |
| C5—C6—H6 | 119.7 | | |

Symmetry code: (i) −*x*, −*y*, −*z*.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7···O1$^{\rm ii}$ | 0.95 | 2.56 | 3.4649 (14) | 158 |

Symmetry code: (ii) −*x*, *y*−1/2, −*z*+1/2.