# organic compounds

# 4,4′-Diiodo-3,3′-dimethoxybiphenyl

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The molecules of the title compound, $C_{14}H_{12}I_2O_2$, lie on inversion centers and are linked by I···O interactions with intermolecular distances of 3.324 (3) Å. The aromatic rings display no significant intercalation or stacking interactions.

## Related literature

For related literature see: Sakai & Matile (2003); Sakai et al. (1997); Anelli et al. (2001); Baumeister et al. (2001); Fidzinski et al. (2003); Mullen & Wegner (1998); Schwab & Levin (1999); Sisson et al. (2006).
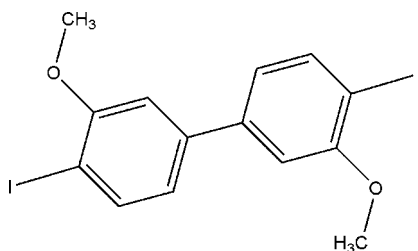


## Experimental

### Crystal data

| | |
|---|---|
| $C_{14}H_{12}I_2O_2$ | Monoclinic, $P2_1/n$ |
| $M_r = 466.04$ | $a = 6.8616$ (14) Å |
| $b = 7.7386$ (15) Å | Mo $K\alpha$ radiation |
| $c = 13.435$ (3) Å | $\mu = 4.50$ mm$^{-1}$ |
| $\beta = 102.43$ (3)° | $T = 153$ (2) K |
| $V = 696.7$ (2) Å$^3$ | $0.36 \times 0.26 \times 0.24$ mm |
| $Z = 2$ | |

### Data collection

| | |
|---|---|
| Rigaku Mercury CCD diffractometer | 5295 measured reflections |
| Absorption correction: multi-scan (REQAB; Jacobson, 1998) | 1417 independent reflections |
| $T_{min} = 0.241$, $T_{max} = 0.337$ | 1381 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.039$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.024$ | 84 parameters |
| $wR(F^2) = 0.062$ | H-atom parameters constrained |
| $S = 1.13$ | $\Delta\rho_{max} = 1.08$ e Å$^{-3}$ |
| 1417 reflections | $\Delta\rho_{min} = -0.90$ e Å$^{-3}$ |

Data collection: *CrystalClear* (Rigaku/MSC, 2006); cell refinement: *CrystalClear*; data reduction: *REQAB* (Jacobson, 1998) and *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2077).

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**supplementary materials**

## 4,4'-Diiodo-3,3'-dimethoxybiphenyl

## Q. Ali, M. R. Shah and D. VanDerveer

### Comment

Over the last 25 years, much attention has been focused on the synthesis of artificial ion channels due to their potential applications in biomedical and material sciences (Schwab & Levin, 1999; Mullen & Wegner, 1998; Fidzinski *et al.*, 2003). The title compound has been used as a precursor for the synthesis of oligo(*p*-phenylene)s (Baumeister *et al.*, 2001) and as a source of hydrophobicity and rigidity (Sakai *et al.*, 1997; Sakai & Matile, 2003) in artificial ion channels. When a macrocycles like porphyrin is attached to the oligo(*p*-phenylene)s, the π-π stacking of porphyrin and the antiperiplanar arrangement of the oligo(*p*-phenylene)s should result in cylindrical self-assembly process and ultimately lead to the formation of functionalized pores (Sisson *et al.*, 2006). Furthermore, iodinated biphenyl has a bright prospect as X-ray contrast media (Anelli *et al.*, 2001).

In this paper, we report the sysnthesis and crystal structure of the title compound, (I). The molecules of (I) lie on crystallographic inversion centers. The I1—O1 intermolecular distance is 3.324 (3) Å which is significantly shorter than 3.50 Å, the sum of the van der Waals radii for I and O, supporting the idea that oxygen atom of methoxy disturbs the electronic cloud surrounding the iodide, hence creating polarization over iodide and subsequently causes reduction in the I1—O1 intermolecular distance. The crystal structures of three hexaiododerivatives of biphenyl have been reported (Anelli *et al.*, (2001).

### Experimental

Fast Blue B salt (*o*-dianisidine bisdiazotated zinc double salt, 10.00 g, 21 mmol), was added to a solution of KI (28 g, 0.17 mmol) in water (200 ml). The mixture was stirred at room temperature for 14 h. After dilution with dichloromethane, the crude reaction mixture was concentrated *in vacuo*. Purification of the crude product on silica gel (dichloromethane:hexane 1:4) followed by evaporation of the solvent under *in vacuo* gave the pure desired product as a pale yellow solid in 70% yield. Single crystals suitable for X-ray crystallography were obtained by slow evaporation of a solution of pale yellow solid in ethanol at room temperature.

### Refinement

All H atoms were geometrically positioned and were allowed to ride on the corresponding C-atoms with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ of the attached C atom for methyl H atoms and $1.2U_{eq}(C)$ for other H atoms. The highest peak in the final difference Fourier map corresponding to a residual electron density of 1.08 e/Å$^3$ was located at 1.2 Å from H5 and was deemed meaningless.
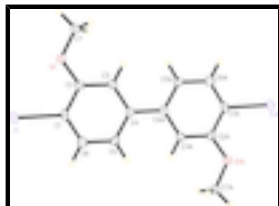
**Figures**



Fig. 1. A thermal ellipsoid plot of the title compound drawn at 50% probability level. The symmetry related atoms have been identified by the letter A in atomic labels.
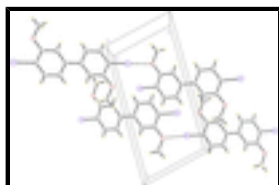


Fig. 2. Packing diagram of the structure viewed down the *b* axis. Hydrogen bonds have been indicated with dashed lines.

## 4,4'-diiodo-3,3'-dimethoxybiphenyl

*Crystal data*

| | |
|---|---|
| $C_{14}H_{12}I_2O_2$ | $F(000) = 436$ |
| $M_r = 466.04$ | $D_x = 2.222$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2yn | Cell parameters from 2340 reflections |
| $a = 6.8616$ (14) Å | $\theta = 3.0–26.4°$ |
| $b = 7.7386$ (15) Å | $\mu = 4.51$ mm$^{-1}$ |
| $c = 13.435$ (3) Å | $T = 153$ K |
| $\beta = 102.43$ (3)° | Chip, colorless |
| $V = 696.7$ (2) Å$^3$ | $0.36 \times 0.26 \times 0.24$ mm |
| $Z = 2$ | |

*Data collection*

| | |
|---|---|
| Rigaku Mercury CCD diffractometer | 1417 independent reflections |
| Radiation source: Sealed Tube | 1381 reflections with $I > 2\sigma(I)$ |
| Graphite Monochromator | $R_{int} = 0.039$ |
| Detector resolution: 14.6306 pixels mm$^{-1}$ | $\theta_{max} = 26.4°$, $\theta_{min} = 3.1°$ |
| ω scans | $h = -8 \rightarrow 8$ |
| Absorption correction: multi-scan (REQAB; Jacobson, 1998) | $k = -9 \rightarrow 9$ |
| $T_{min} = 0.241$, $T_{max} = 0.337$ | $l = -16 \rightarrow 11$ |
| 5295 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |

$R[F^2 > 2\sigma(F^2)] = 0.024$

$wR(F^2) = 0.062$

$S = 1.13$

1417 reflections

84 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0318P)^2 + 1.081P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 1.08$ e Å$^{-3}$

$\Delta\rho_{min} = -0.90$ e Å$^{-3}$

Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0437 (19)

*Special details*

**Experimental**. 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$^2$ > 2sigma(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.

**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.

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

|  | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| I1 | 0.28040 (3) | 0.14667 (2) | 0.340534 (14) | 0.01974 (14) |
| O1 | 0.5702 (3) | 0.3479 (3) | 0.22987 (17) | 0.0195 (5) |
| C1 | 0.5402 (4) | 0.2888 (3) | 0.3985 (2) | 0.0152 (5) |
| C2 | 0.6482 (4) | 0.3641 (3) | 0.3325 (2) | 0.0129 (6) |
| C3 | 0.8273 (4) | 0.4479 (4) | 0.3731 (2) | 0.0151 (5) |
| H3 | 0.9008 | 0.5005 | 0.3278 | 0.018* |
| C4 | 0.9026 (4) | 0.4569 (3) | 0.4787 (2) | 0.0139 (5) |
| C5 | 0.7896 (4) | 0.3821 (4) | 0.5427 (2) | 0.0183 (6) |
| H5 | 0.8366 | 0.3884 | 0.6153 | 0.022* |
| C6 | 0.6107 (4) | 0.2990 (4) | 0.5026 (2) | 0.0203 (6) |
| H6 | 0.5353 | 0.2483 | 0.5475 | 0.024* |
| C7 | 0.6900 (5) | 0.4047 (5) | 0.1613 (2) | 0.0230 (7) |
| H7A | 0.7072 | 0.5277 | 0.1668 | 0.035* |
| H7B | 0.6251 | 0.3752 | 0.0927 | 0.035* |
| H7C | 0.8180 | 0.3493 | 0.1783 | 0.035* |

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

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| I1 | 0.01475 (17) | 0.02317 (18) | 0.02110 (17) | −0.00579 (6) | 0.00339 (9) | −0.00124 (6) |
| O1 | 0.0194 (10) | 0.0253 (12) | 0.0136 (10) | −0.0088 (8) | 0.0029 (8) | −0.0017 (7) |

| | | | | | |
|---|---|---|---|---|---|
| C1 | 0.0090 (11) | 0.0127 (12) | 0.0230 (13) | −0.0005 (10) | 0.0019 (10) | −0.0010 (10) |
| C2 | 0.0140 (13) | 0.0108 (13) | 0.0137 (14) | 0.0005 (9) | 0.0024 (10) | −0.0009 (9) |
| C3 | 0.0156 (12) | 0.0141 (12) | 0.0161 (13) | −0.0002 (10) | 0.0042 (10) | 0.0009 (10) |
| C4 | 0.0119 (12) | 0.0111 (11) | 0.0180 (13) | 0.0021 (10) | 0.0019 (10) | 0.0018 (10) |
| C5 | 0.0143 (13) | 0.0267 (14) | 0.0121 (13) | −0.0008 (12) | −0.0014 (10) | 0.0032 (11) |
| C6 | 0.0148 (13) | 0.0258 (15) | 0.0200 (14) | 0.0004 (12) | 0.0028 (10) | 0.0021 (12) |
| C7 | 0.0239 (14) | 0.0306 (17) | 0.0145 (14) | −0.0100 (14) | 0.0038 (11) | −0.0008 (12) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| I1—C1 | 2.097 (3) | C4—C5 | 1.401 (4) |
| O1—C2 | 1.373 (4) | C4—C4$^i$ | 1.493 (5) |
| O1—C7 | 1.429 (4) | C5—C6 | 1.388 (4) |
| C1—C6 | 1.380 (4) | C5—H5 | 0.9600 |
| C1—C2 | 1.399 (4) | C6—H6 | 0.9600 |
| C2—C3 | 1.392 (4) | C7—H7A | 0.9599 |
| C3—C4 | 1.404 (4) | C7—H7B | 0.9599 |
| C3—H3 | 0.9600 | C7—H7C | 0.9599 |
| C2—O1—C7 | 117.8 (2) | C6—C5—C4 | 120.8 (3) |
| C6—C1—C2 | 120.0 (3) | C6—C5—H5 | 119.6 |
| C6—C1—I1 | 119.4 (2) | C4—C5—H5 | 119.6 |
| C2—C1—I1 | 120.5 (2) | C1—C6—C5 | 120.5 (3) |
| O1—C2—C3 | 123.8 (3) | C1—C6—H6 | 119.7 |
| O1—C2—C1 | 117.0 (2) | C5—C6—H6 | 119.7 |
| C3—C2—C1 | 119.3 (3) | O1—C7—H7A | 109.5 |
| C2—C3—C4 | 121.4 (3) | O1—C7—H7B | 109.5 |
| C2—C3—H3 | 119.3 | H7A—C7—H7B | 109.5 |
| C4—C3—H3 | 119.3 | O1—C7—H7C | 109.5 |
| C5—C4—C3 | 117.9 (2) | H7A—C7—H7C | 109.5 |
| C5—C4—C4$^i$ | 121.2 (3) | H7B—C7—H7C | 109.5 |
| C3—C4—C4$^i$ | 120.9 (3) | | |

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

**Fig. 1**

**Fig. 2**