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4-Iodo-3,3′-dimethoxybiphenyl

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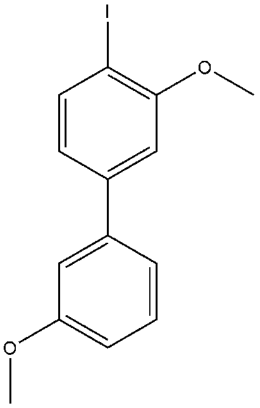
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.003 Å; R factor = 0.023; wR factor = 0.054; data-to-parameter ratio = 15.0.

Molecules of the title compound, $C_{14}H_{13}IO_2$, exhibit no π–π interactions. The dihedral angle between the two aromatic rings is 43.72 (9)°. The shortest intermolecular I$\cdots$O distance is 3.408 (2) Å, which is significantly less than the sum of the van der Waals radii for I and O (3.50 Å).

Related literature

For related literature, see: Litvinchuk *et al.* (2004); Baudry *et al.* (2006); Sisson *et al.* (2006); Ali *et al.* (2008); Ibad *et al.* (2008); Baumeister *et al.* (2001).



Experimental

Crystal data

$C_{14}H_{13}IO_2$	V = 1283.6 (4) Å^3
M_r = 340.14	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 11.932 (2) Å	μ = 2.48 mm^{-1}
b = 15.382 (3) Å	T = 153 (2) K
c = 6.9940 (14) Å	0.43 × 0.38 × 0.36 mm
β = 90.68 (3)°	

Data collection

Rigaku Mercury CCD diffractometer	9179 measured reflections
Absorption correction: multi-scan (Jacobson, 1998)	2337 independent reflections
T_{min} = 0.415, T_{max} = 0.469 (expected range = 0.362–0.409)	2206 reflections with $I > 2\sigma(I)$
	R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022	156 parameters
$wR(F^2)$ = 0.053	H-atom parameters constrained
S = 1.09	$\Delta\rho_{max}$ = 1.19 e Å^{-3}
2337 reflections	$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2006); cell refinement: *CrystalClear*; data reduction: *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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2736).

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4-Iodo-3,3′-dimethoxybiphenyl

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S1. Comment

Self-assembling molecules based on oligo(*p*-phenylene)s are receiving increased attention as building blocks for supramolecular structures, such as artficial ion channels (Litvinchuk *et al.*, 2004; Baudry *et al.*,2 006). One can envision that incorporation of a conjugated macrocycle such as porphyrin into an oligo(*p*-phenylene)s, extends the cylindrical supramolecular organization capabilities of the oligo(*p*phenylene)s (Sisson *et al.*, 2006) and can result in functionalized pores. The titled compound can be used as a precursor for the synthesis of oligo(*p*-phenylene)s (Baumeister *et al.*, 2001). The I1—O1 intermolecular distance is 3.408 (2) Å which is significantly less than 3.50 Å, the sum of the van der Waals radii for I and O. Reported data (Ali *et al.*, 2008) indicate that the oxygen atom of a methoxy group polarizes the electronic cloud surrounding the iodide causing a reduction in the I—O intermolecular distance. The phenyl rings are twisted by a dihedral angle of 43.72 (9)°, which is typical for biphenyl molecules (Ibad *et al.*, 2008). The presence of a iodo group at only one phenyl ring least to a twist between the two rings, whereas the rings are coplanar when both phenyl rings bore a iodo group (Ali *et al.*, 2008). The crystal packing diagram (Fig.2) shows that the molecules are interlinked by I—O interactions.

S2. Experimental

5 g (10.7 mmol) of 4,4`-diiodo-3`-methoxy[1,1`-biphenyl]-3-yl-methyl ether was dissolved in 30 ml of THF in a 250 ml round bottom flask. The reaction mixture was stirred until a clear solution formed. Then a *tert*-butyl lithium solution (8.2 ml, 1.7 *M* in pentane 13.9 mmol) was added at 0 C. The reaction was monitored after an interval of 5 minutes through TLC.The reaction was stirred for thirty five minutes until a spot of 4-iodo-3,3`-dimethoxy-1,1`-biphenyl appeared on the TLC and was then quenched with 10 ml (1 N HCl) and extracted with 30 ml of chloroform three times. The crude reaction mixture was concentrated using a rotary evaporator. A super saturated solution of crude reaction mixture was prepared in chlorofrom and then methanol was added to this super-saturated solution of reaction mixture, two layers were formed which were separated and analysed by TLC (Hexane:Chloroform 1:1). The methanol layer contained 4-iodo-3,3′-dimethoxybiphenyl as the major product. The slow evaporation of methanol at room temperature yielded colorless crystals.

S3. Refinement

All H atoms were geometrically positioned and allowed to ride on the corresponding parent atom with C—H = 0.96 Å and $U_{\rm iso}(H) = 1.5U_{\rm eq}(C_{\rm methyl})$ or $1.2U_{\rm eq}(C_{\rm aromatic})$, respectively. The methyl groups were allowed to rotate but not to tip.

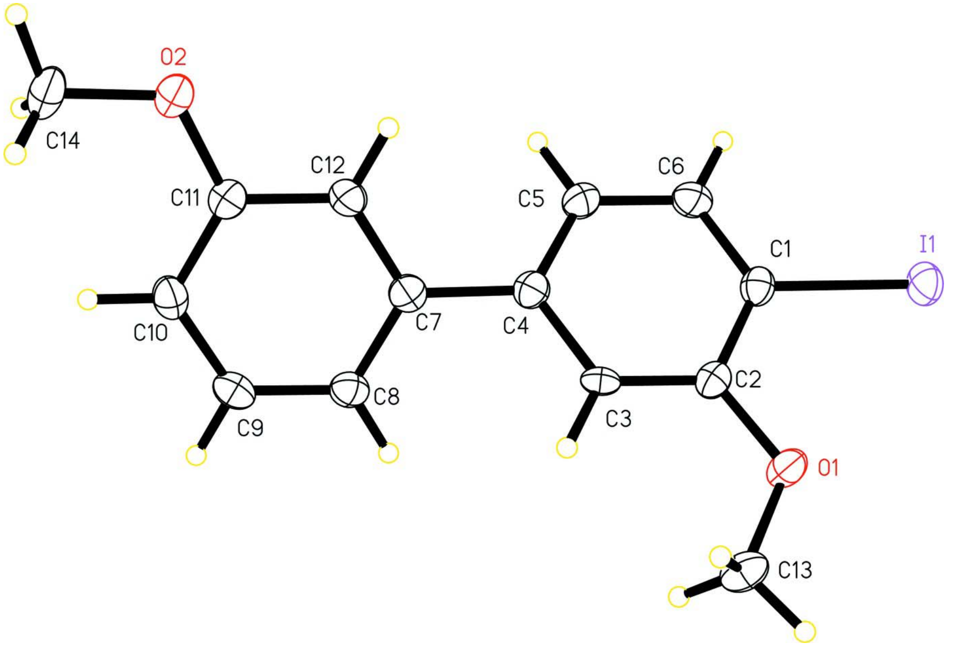

Figure 1

A perspective thermal elipsoid drawing (50% probability) of the molecule showing the atom labelling scheme.

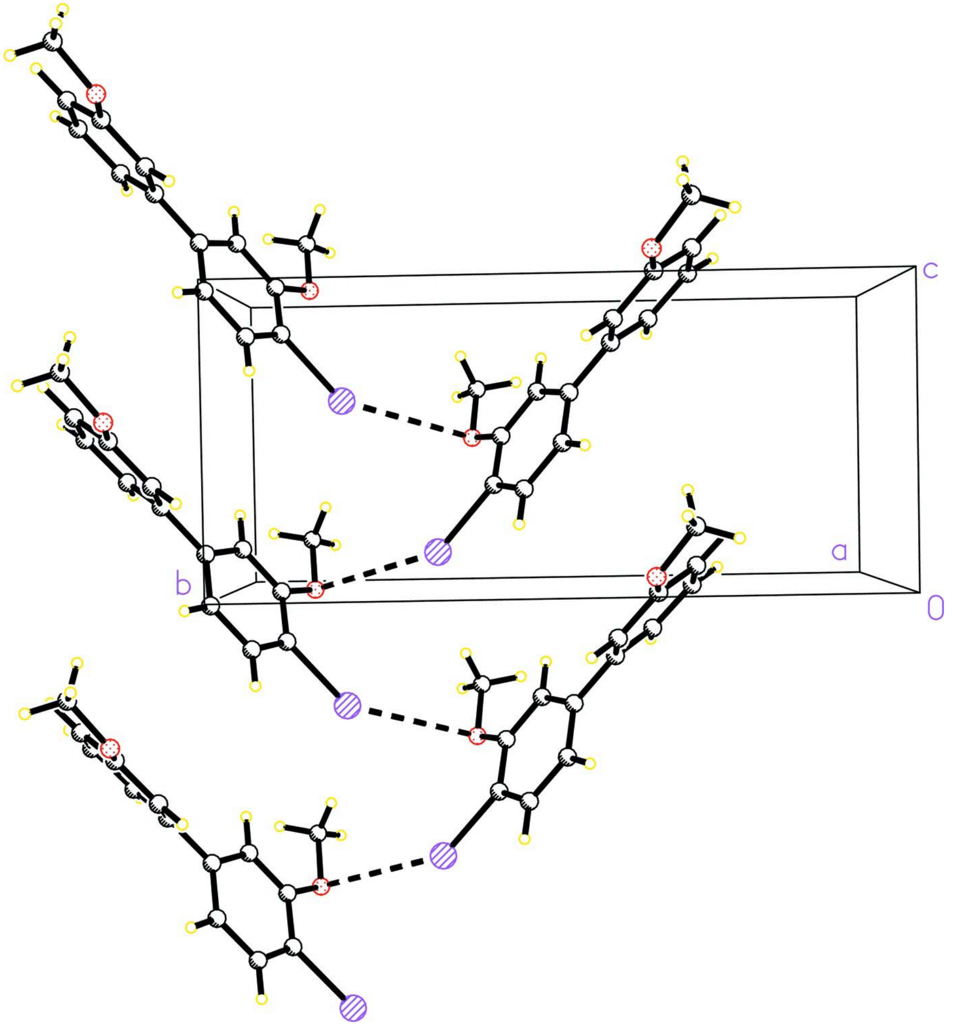

Figure 2

A packing diagram along the *a* axis showing the short I - O contacts with dashed lines.

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Crystal data

$C_{14}H_{13}IO_2$
$M_r = 340.14$
Monoclinic, $P2_1/c$
$a = 11.932$ (2) Å
$b = 15.382$ (3) Å
$c = 6.9940$ (14) Å
$\beta = 90.68$ (3)°
$V = 1283.6$ (4) Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.760$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4368 reflections
$\theta = 2.9$–26.4°
$\mu = 2.48$ mm^{-1}
$T = 153$ K
Chip, colorless
0.43 × 0.38 × 0.36 mm

Data collection

Rigaku Mercury CCD diffractometer
Radiation source: Sealed Tube
Graphite Monochromator monochromator
Detector resolution: 14.6306 pixels mm^{-1}
ω scans

Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.415, T_{max} = 0.469
9179 measured reflections
2337 independent reflections
2206 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.4°, θ_{min} = 3.2°
$h = -14 \rightarrow 14$
$k = -18 \rightarrow 18$
$l = -8 \rightarrow 6$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.054$
$S = 1.10$
2337 reflections
156 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
$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 1.6489P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 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}
I1	0.351251 (14)	0.680339 (10)	0.13178 (2)	0.02636 (8)
C1	0.3045 (2)	0.59482 (15)	0.3506 (3)	0.0207 (5)
C2	0.37606 (19)	0.58357 (15)	0.5087 (3)	0.0187 (5)
C3	0.34448 (17)	0.52906 (15)	0.6519 (3)	0.0158 (4)
H3A	0.3926	0.5222	0.7618	0.019*
C4	0.2424 (2)	0.48233 (15)	0.6425 (3)	0.0203 (5)
C5	0.1732 (2)	0.49371 (17)	0.4818 (4)	0.0251 (5)
H5A	0.1037	0.4625	0.4718	0.030*
C6	0.2044 (2)	0.55002 (17)	0.3361 (4)	0.0250 (5)
H6A	0.1566	0.5577	0.2261	0.030*
C7	0.2107 (2)	0.42139 (15)	0.7984 (3)	0.0203 (5)
C8	0.2903 (2)	0.36430 (16)	0.8774 (3)	0.0219 (5)
H8A	0.3663	0.3652	0.8341	0.026*
C9	0.2583 (2)	0.30671 (16)	1.0183 (4)	0.0233 (5)
H9A	0.3129	0.2672	1.0707	0.028*
C10	0.1492 (2)	0.30429 (16)	1.0864 (4)	0.0235 (5)
H10A	0.1285	0.2638	1.1844	0.028*
C11	0.0710 (2)	0.36164 (16)	1.0095 (4)	0.0226 (5)
C12	0.1018 (2)	0.41935 (16)	0.8648 (4)	0.0222 (5)

H12A	0.0468	0.4580	0.8107	0.027*
C13	0.5475 (2)	0.62115 (17)	0.6703 (4)	0.0259 (5)
H13A	0.6153	0.6529	0.6478	0.039*
H13B	0.5651	0.5611	0.6924	0.039*
H13C	0.5108	0.6446	0.7803	0.039*
C14	−0.0733 (3)	0.31310 (18)	1.2189 (4)	0.0340 (7)
H14A	−0.1489	0.3273	1.2525	0.051*
H14B	−0.0248	0.3222	1.3275	0.051*
H14C	−0.0696	0.2533	1.1802	0.051*
O1	0.47461 (14)	0.62845 (11)	0.5063 (2)	0.0238 (4)
O2	−0.03855 (15)	0.36749 (13)	1.0649 (3)	0.0334 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02789 (11)	0.02800 (11)	0.02336 (11)	0.00115 (6)	0.00705 (7)	0.00536 (6)
C1	0.0225 (12)	0.0197 (11)	0.0200 (12)	−0.0003 (9)	0.0043 (9)	0.0001 (9)
C2	0.0185 (11)	0.0174 (11)	0.0205 (11)	−0.0006 (9)	0.0036 (9)	−0.0022 (9)
C3	0.0133 (10)	0.0184 (11)	0.0156 (11)	0.0032 (8)	−0.0027 (8)	−0.0051 (8)
C4	0.0206 (11)	0.0204 (11)	0.0200 (12)	−0.0002 (9)	0.0016 (9)	−0.0006 (9)
C5	0.0201 (12)	0.0284 (13)	0.0267 (13)	−0.0055 (10)	−0.0031 (10)	0.0035 (10)
C6	0.0220 (12)	0.0302 (13)	0.0226 (12)	−0.0008 (10)	−0.0047 (10)	0.0019 (10)
C7	0.0224 (11)	0.0176 (11)	0.0209 (12)	−0.0035 (9)	−0.0037 (9)	−0.0022 (9)
C8	0.0206 (12)	0.0229 (12)	0.0222 (12)	−0.0008 (9)	−0.0026 (9)	−0.0011 (10)
C9	0.0248 (13)	0.0243 (12)	0.0208 (12)	0.0030 (10)	−0.0053 (10)	−0.0015 (10)
C10	0.0290 (13)	0.0228 (12)	0.0188 (12)	−0.0031 (10)	−0.0017 (10)	0.0026 (10)
C11	0.0213 (12)	0.0226 (12)	0.0239 (12)	−0.0018 (9)	0.0008 (10)	0.0013 (10)
C12	0.0211 (12)	0.0197 (11)	0.0259 (13)	0.0017 (9)	−0.0010 (9)	0.0030 (10)
C13	0.0215 (12)	0.0306 (13)	0.0257 (13)	−0.0047 (10)	0.0006 (10)	−0.0071 (11)
C14	0.0318 (15)	0.0372 (16)	0.0331 (16)	−0.0057 (11)	0.0109 (12)	0.0092 (12)
O1	0.0195 (8)	0.0284 (9)	0.0236 (9)	−0.0066 (7)	0.0013 (7)	−0.0004 (7)
O2	0.0239 (9)	0.0370 (11)	0.0396 (11)	0.0024 (8)	0.0080 (8)	0.0157 (9)

Geometric parameters (Å, º)

I1—C1	2.098 (2)	C9—C10	1.393 (4)
C1—C6	1.382 (3)	C9—H9A	0.9600
C1—C2	1.400 (3)	C10—C11	1.388 (4)
C2—C3	1.363 (3)	C10—H10A	0.9600
C2—O1	1.364 (3)	C11—O2	1.370 (3)
C3—C4	1.416 (3)	C11—C12	1.399 (3)
C3—H3A	0.9600	C12—H12A	0.9600
C4—C5	1.397 (3)	C13—O1	1.436 (3)
C4—C7	1.490 (3)	C13—H13A	0.9599
C5—C6	1.391 (4)	C13—H13B	0.9599
C5—H5A	0.9600	C13—H13C	0.9599
C6—H6A	0.9600	C14—O2	1.429 (3)
C7—C12	1.386 (3)	C14—H14A	0.9599

C7—C8	1.402 (3)	C14—H14B	0.9599
C8—C9	1.382 (4)	C14—H14C	0.9599
C8—H8A	0.9600		

I1···O1^{i}	3.408 (2)

C6—C1—C2	121.0 (2)	C8—C9—H9A	119.1
C6—C1—I1	119.76 (18)	C10—C9—H9A	119.1
C2—C1—I1	119.25 (17)	C11—C10—C9	118.5 (2)
C3—C2—O1	124.5 (2)	C11—C10—H10A	120.7
C3—C2—C1	119.0 (2)	C9—C10—H10A	120.7
O1—C2—C1	116.5 (2)	O2—C11—C10	124.8 (2)
C2—C3—C4	121.6 (2)	O2—C11—C12	115.0 (2)
C2—C3—H3A	119.2	C10—C11—C12	120.1 (2)
C4—C3—H3A	119.2	C7—C12—C11	120.8 (2)
C5—C4—C3	118.3 (2)	C7—C12—H12A	119.6
C5—C4—C7	121.0 (2)	C11—C12—H12A	119.6
C3—C4—C7	120.7 (2)	O1—C13—H13A	109.5
C6—C5—C4	120.4 (2)	O1—C13—H13B	109.5
C6—C5—H5A	119.8	H13A—C13—H13B	109.5
C4—C5—H5A	119.8	O1—C13—H13C	109.5
C1—C6—C5	119.7 (2)	H13A—C13—H13C	109.5
C1—C6—H6A	120.1	H13B—C13—H13C	109.5
C5—C6—H6A	120.1	O2—C14—H14A	109.5
C12—C7—C8	119.1 (2)	O2—C14—H14B	109.5
C12—C7—C4	120.4 (2)	H14A—C14—H14B	109.5
C8—C7—C4	120.4 (2)	O2—C14—H14C	109.5
C9—C8—C7	119.4 (2)	H14A—C14—H14C	109.5
C9—C8—H8A	120.3	H14B—C14—H14C	109.5
C7—C8—H8A	120.3	C2—O1—C13	117.71 (19)
C8—C9—C10	121.9 (2)	C11—O2—C14	117.4 (2)

C6—C1—C2—C3	−1.6 (4)	C3—C4—C7—C8	−43.5 (3)
I1—C1—C2—C3	178.93 (16)	C12—C7—C8—C9	0.6 (4)
C6—C1—C2—O1	177.8 (2)	C4—C7—C8—C9	−178.3 (2)
I1—C1—C2—O1	−1.7 (3)	C7—C8—C9—C10	−0.8 (4)
O1—C2—C3—C4	−178.0 (2)	C8—C9—C10—C11	0.0 (4)
C1—C2—C3—C4	1.3 (3)	C9—C10—C11—O2	−178.8 (2)
C2—C3—C4—C5	−0.4 (3)	C9—C10—C11—C12	1.0 (4)
C2—C3—C4—C7	178.5 (2)	C8—C7—C12—C11	0.4 (4)
C3—C4—C5—C6	−0.4 (4)	C4—C7—C12—C11	179.2 (2)
C7—C4—C5—C6	−179.2 (2)	O2—C11—C12—C7	178.7 (2)
C2—C1—C6—C5	0.9 (4)	C10—C11—C12—C7	−1.2 (4)
I1—C1—C6—C5	−179.64 (19)	C3—C2—O1—C13	−3.3 (3)
C4—C5—C6—C1	0.1 (4)	C1—C2—O1—C13	177.3 (2)
C5—C4—C7—C12	−43.6 (3)	C10—C11—O2—C14	2.9 (4)

C3—C4—C7—C12	137.6 (2)	C12—C11—O2—C14	−176.9 (2)
C5—C4—C7—C8	135.3 (3)		

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