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Crystal structures of two bis(iodomethyl)benzene derivatives: similarities and differences in the crystal packing

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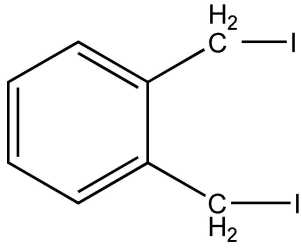
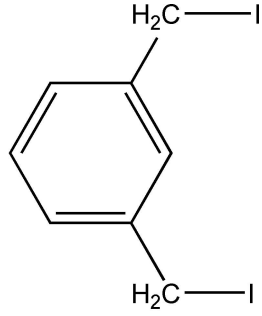
The isomeric derivatives 1,2-bis(iodomethyl)benzene, (I), and 1,3-bis(iodomethyl)benzene (II), both $C_8H_8I_2$, were prepared by metathesis from their dibromo analogues. The *ortho*-derivative, (I), lies about a crystallographic twofold axis that bisects the C—C bond between the two iodomethyl substituents. The packing in (I) relies solely on C—H···I hydrogen bonds supported by weak parallel slipped π–π stacking interactions [inter-centroid distance = 4.0569 (11) Å, inter-planar distance = 3.3789 (8) Å and slippage = 2.245 Å]. While C—H···I hydrogen bonds are also found in the packing of (II), type II, I···I halogen bonds [I···I = 3.8662 (2) Å] and C—H···π contacts feature prominently in stabilizing the three-dimensional structure.

1. Chemical context

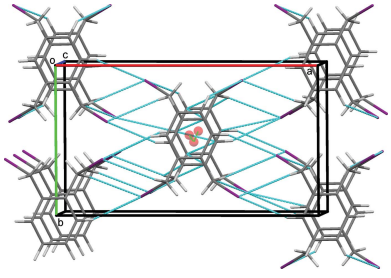
The isomeric xylene derivatives reported here, 1,2-bis(iodomethyl)benzene, (I), and 1,3-bis(iodomethyl)benzene (II), are useful synthons for the preparation of a range of organic compounds. (I) is used particularly in the synthesis of polycyclic aromatic systems (see for example: Takahashi *et al.* 2006; Abreu *et al.*, 2010; Wang *et al.*, 2012). Similarly (II) has been used in polymer formation (Pandya & Gibson, 1991), in the synthesis of metacyclophanes (Ramming & Gleiter, 1997) and to provide aromatic spacers in organic synthesis (Kida *et al.*, 2005). Our interest in such compounds is as components of ionene polymers. The compounds were readily prepared by metathesis from the bis(bromomethyl)benzene derivatives.



(I)



(II)



2. Structural commentary

The molecular structures of 1,2-bis(iodomethyl)benzene, (I), and 1,3-bis(iodomethyl)benzene, (II), are shown in Figs. 1 and 2 and are sufficiently similar to be discussed together. Each comprises a benzene ring with two iodomethyl substituents in the 1,2- and 1,3-positions for (I) and (II) respectively. The

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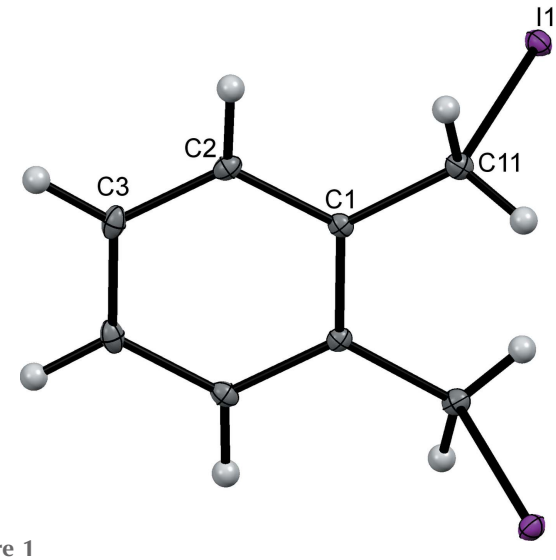

Figure 1
The molecular structure of compound (I), with displacement ellipsoids drawn at the 50% probability level. The unlabelled atoms are related to labelled atoms by the symmetry operation $(-x+1, y, -z+\frac{3}{2})$.

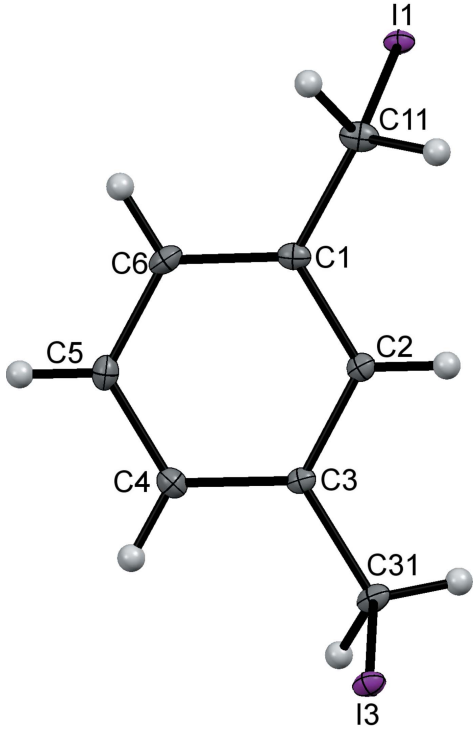

Figure 2
The molecular structure of compound (II), with displacement ellipsoids drawn at the 50% probability level.

molecule of (I) lies about a twofold axis that bisects the C—C bond between the two iodomethyl substituents. For each molecule the C—I bonds of the substituents point away from opposite faces of the benzene rings with the C—C—I planes almost orthogonal to the ring planes; dihedral angles = 87.99 (14)° for (I) and 82.23 (14) and 83.61 (15)° for (II). The C1—C11 and C11—I1 bond lengths in (I) and C1—C11, C11—I1, C3—C31 and C31—I3 in (II) are reasonably self-consistent and also compare well with those found in the isomeric 1,4-bis(iodomethyl)benzene (McAdam *et al.* 2009).

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···I1^{i}	0.95	3.38	4.046 (2)	129
C11—H112···I1^{ii}	0.99	3.33	4.179 (2)	145
C2—H2···I1^{ii}	0.95	3.36	4.257 (2)	158

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

3. Supramolecular features

3.1. Crystal packing for (I)

In the crystal of (I), weak parallel slipped π–π stacking interactions [inter-centroid distance = 4.0569 (11) Å, interplanar distance = 3.3789 (8) Å, slippage = 2.245 Å], between the benzene rings of inversion-related molecules are supported by C3—H3···I1 hydrogen bonds, Table 1, to link molecules in a head-to tail-fashion, stacking them along *c*, Fig. 3. In addition, the iodine atoms act as bifurcated acceptors, forming weak C2—H2···I1 and C11—H112···I1 hydrogen bonds generating $R_2^1(6)$ ring motifs (Bernstein *et al.*, 1995). These contacts link the molecules into zigzag chains along [101], Fig. 4. These contacts combine to link stacked columns of molecules through weak C—H···I hydrogen

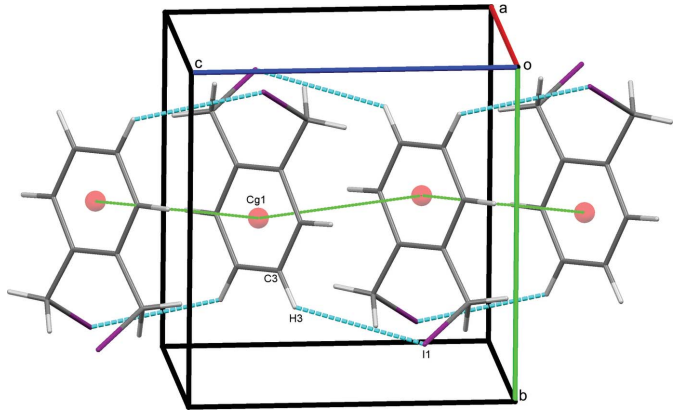

Figure 3
π–π stacking interactions (green dotted lines) supported by C—H···I hydrogen bonds for (I). Hydrogen bonds in this and subsequent figures are drawn as blue dashed lines.

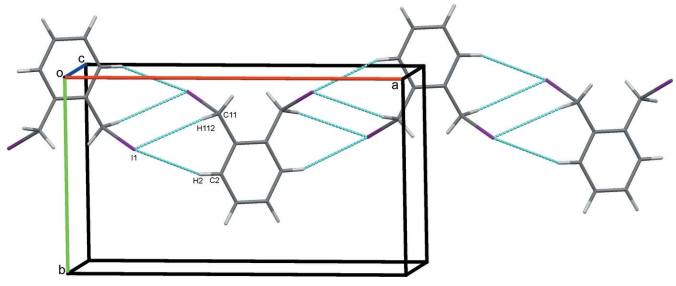

Figure 4
Chains of molecules of (I) in [101].

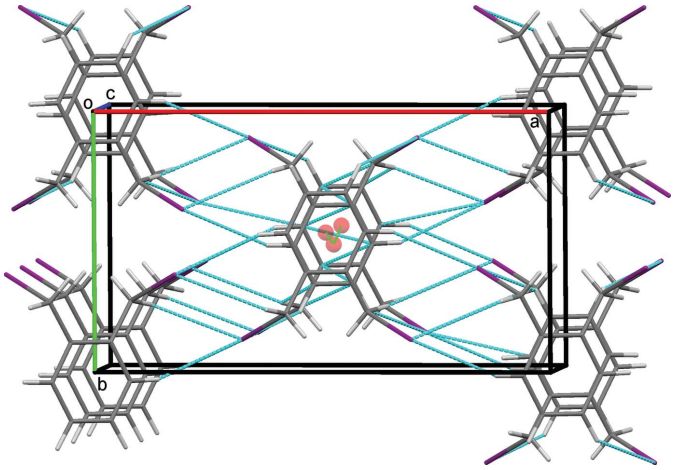

Figure 5
Overall packing for (I) viewed along the *c*-axis direction.

bonds and generate a three dimensional network structure, Fig. 5.

3.2. Crystal packing for (II)

In the crystal of (II), C11—H11*B*···I1 hydrogen bonds, Table 2, form a column supported by a series of C31—H31*B*···*Cg*1 contacts. C31—H31*A*···I3 hydrogen bonds link these in an obverse fashion, forming double chains along *b*,

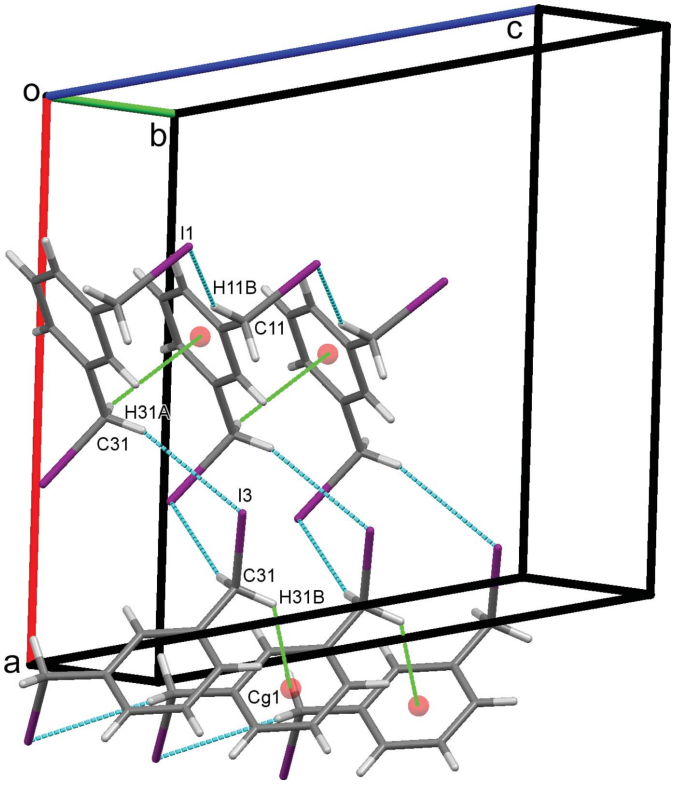

Figure 6
Double chains of molecules of (II) formed by a series of C31—H31*B*···*Cg*1 contacts (green dotted lines) linked by C—H···I hydrogen bonds.

Table 2
Hydrogen-bond geometry (Å, °) for (II).

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···I1^{i}	0.99	3.22	4.060 (3)	144
C5—H5···I1^{ii}	0.95	3.25	4.078 (3)	147
C31—H31*A*···I3^{iii}	0.99	3.27	4.224 (3)	162
C31—H31*A*···*Cg*^{iv}	0.99	2.84	3.453 (3)	121

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x, y+1, z$.

Fig. 6. C5—H5···I1 hydrogen bonds, Fig. 7, link the double chains into sheets in the *ab* plane. An extensive series of I1···I3 halogen bonds Fig. 8, I1···I3^{v,vi} = 3.8662 (2) Å;

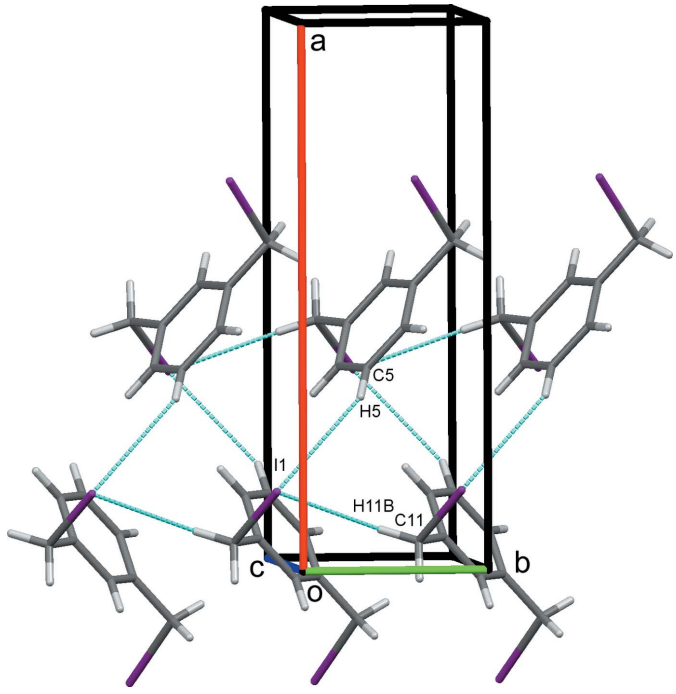

Figure 7
Sheets of molecules of (II) in the *ab* plane formed by C—H···I. hydrogen bonds.

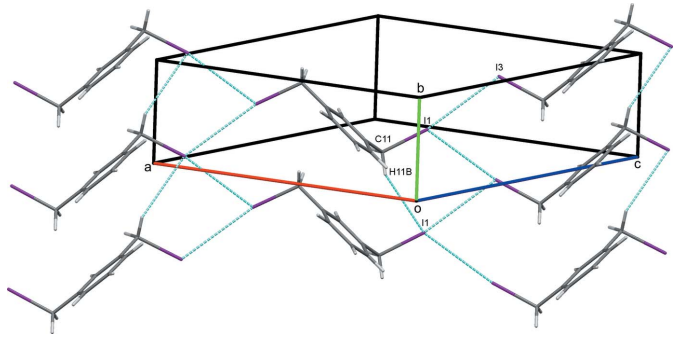

Figure 8
Sheets of molecules of (II) in the (101) plane formed by I···I halogen bonds, blue dashed lines, supported by C—H···I hydrogen bonds.

symmetry codes: (v) = $-\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (vi) = $\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$ (Desiraju *et al.*, 2013; Metrangolo *et al.*, 2008), extend the structure in the third dimension, Fig. 9. The angles C11—I1—I3 = 117° and C31—I3—I1 = 165° characterize this halogen bond as type II (Pedireddi *et al.*, 1994).

4. Database survey

A search of the Cambridge Structural Database (Version 5.36 with three updates; Groom & Allen, 2014) for molecules incorporating a C_6CH_2I fragment surprisingly generated only five hits for iodomethylbenzene derivatives. One of these is the isomeric 1,4-bis(iodomethyl)benzene reported by us previously (McAdam *et al.*, 2009), while two others are the organic compounds 2-(iodomethyl)-1,3,5-trimethylbenzene (Bats, 2014) and 3′-iodo-5′-(iodomethyl)biphenyl-4-carbonitrile (He *et al.*, 2013). The other two entries are metal complexes (Martínez-García *et al.*, 2010; Rivada-Wheelaghan *et al.*, 2012). In one of these, the iodine atom of the iodomethyl unit was found to act as a ligand to a platinum(II) nucleus (Rivada-Wheelaghan *et al.*, 2012). The structures of both the chloro- and bromo-analogues of 1,2-bis(iodomethyl)benzene (Basaran *et al.*, 1992; Jones & Kus, 2007) and 1,3-bis(iodomethyl)benzene (Sanders *et al.*, 2013; Li *et al.*, 2006; Jones & Kus, 2007) have also been reported. Interestingly, 1,3-bis(bromomethyl)benzene is isostructural with (II) and the packing features for the two compounds are identical, apart from somewhat increased distances for the iodo compound.

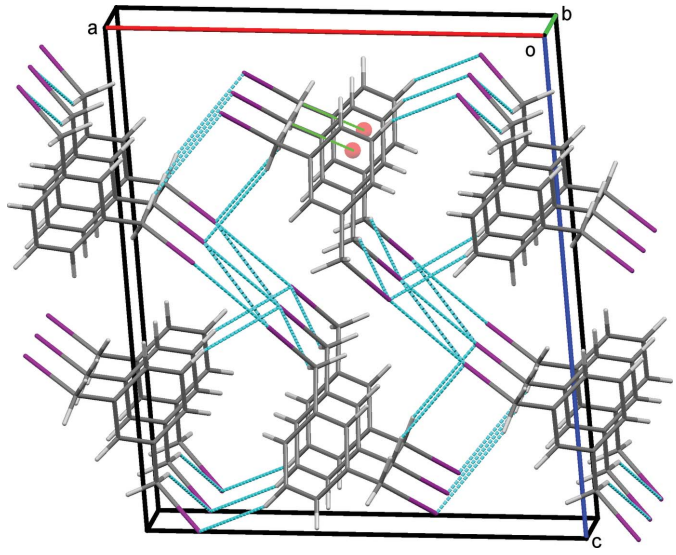

Figure 9
Overall packing for (II) viewed along the *b*-axis direction.

For example I1···I3 = 3.8662 (2) Å for (II) but the equivalent Br···Br distance is 3.6742 (3) Å for the *meta*-dibromo analogue (Jones & Kus, 2007). Similar isostructural behaviour is observed for *para*-bis(iodomethyl)benzene (McAdam *et al.*, 2009) and its dibromo analogue (Jones & Kus, 2007). However, in contrast, despite (I) and the *ortho*-dibromo analogue both displaying twofold symmetry, compound (I) crystallizes in the monoclinic space group *C*2/*c* while that for

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_8H_8I_2$	$C_8H_8I_2$
M_r	357.94	357.94
Crystal system, space group	Monoclinic, *C*2/*c*	Monoclinic, $P2_1/n$
Temperature (K)	90	90
a, *b*, *c* (Å)	14.5485 (5), 8.0461 (3), 8.0582 (3)	13.5323 (3), 4.5464 (1), 15.6269 (4)
β (°)	101.637 (2)	95.203 (1)
V (Å^3)	923.89 (6)	957.46 (4)
Z	4	4
Radiation type	Mo *K*α	Mo *K*α
μ (mm^{-1})	6.74	6.50
Crystal size (mm)	0.31 × 0.17 × 0.15	0.45 × 0.06 × 0.05
Data collection		
Diffractometer	Bruker APEXII CCD area detector	Bruker APEXII CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.534, 1.000	0.569, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8422, 1667, 1552	16804, 3435, 2826
R_{int}	0.030	0.033
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.775	0.775
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.018, 0.044, 1.15	0.024, 0.048, 1.06
No. of reflections	1667	3435
No. of parameters	46	91
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.52, −1.23	1.24, −0.77

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *TITAN2000* (Hunter & Simpson, 1999), *Mercury* (Macrae *et al.*, 2008), *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009), *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

the dibromo counterpart is found to be orthorhombic, *Fdd*2 (Jones & Kus, 2007).

5. Synthesis and crystallization

Preparation of the title compounds was based on literature methods (Moore & Stupp, 1986; Kida *et al.*, 2005). The appropriate bis(bromomethyl)benzene (1.32 g, 5 mmol) was refluxed for 7 h with sodium iodide (2.25 g, 15 mmol) in acetone (25 ml). The solution was allowed to cool overnight, the crystals that developed were rinsed gently with water to remove sodium bromide and air dried. The product was recrystallized a second time from acetone to give X-ray quality crystals. Confirmation of the metathesised (iodo) product was by microanalysis and mass spectroscopy. ^{13}C NMR spectra of the diiodo compounds are distinct from those of their dibromo precursors.

Compound (I): Analysis calculated for $C_8H_8I_2$: C, 26.84; H, 2.25%. Found: C, 26.86; H, 2.14%. ^{13}C NMR (δ p.p.m.): 137.4, 130.8, 129.0, 1.8.

Compound (II): Analysis calculated for $C_8H_8I_2$: C, 26.84; H, 2.25%. Found: C, 26.63; H, 2.19%. ^{13}C NMR (δ p.p.m.): 140.0, 129.4, 129.0, 128.4, 4.9.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were refined using a riding model with d(C—H) = 0.95 Å, U_{iso} = 1.2U_{eq}(C) for aromatic and 0.99 Å, U_{iso} = 1.2U_{eq}(C) for CH_2 H atoms. For (I), a low-angle reflection with $F_o << F_c$, that may have been affected by the beam-stop, was omitted from the final refinement cycles.

Acknowledgements

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supporting information

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2013); cell refinement: *APEX2* and *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b) and *TITAN2000* (Hunter & Simpson, 1999); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b), *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009), *publCIF* (Westrip, 2010) and *WinGX* (Farrugia, 2012).

(I) 1,2-Bis(iodomethyl)benzene

Crystal data

$C_8H_8I_2$
M_r = 357.94
Monoclinic, $C2/c$
a = 14.5485 (5) Å
b = 8.0461 (3) Å
c = 8.0582 (3) Å
β = 101.637 (2)°
V = 923.89 (6) Å^3
Z = 4
$F(000)$ = 648
D_x = 2.573 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5091 reflections
θ = 2.6–32.9°
μ = 6.74 mm^{-1}
T = 90 K
Block, colourless
0.31 × 0.17 × 0.15 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.534, T_{max} = 1.000
8422 measured reflections
1667 independent reflections
1552 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 33.4°, θ_{min} = 2.9°
$h = -21 \rightarrow 21$
$k = -11 \rightarrow 12$
$l = -12 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.044
S = 1.15
1667 reflections
46 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0175P)^2 + 1.2212P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −1.23 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. One low angle reflection with F_o << Fc was omitted from the final refinement cycles.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.31503 (2)	0.11885 (2)	0.75250 (2)	0.01529 (5)
C11	0.41526 (13)	0.2215 (2)	0.6102 (3)	0.0142 (3)
H111	0.4651	0.1386	0.6070	0.017*
H112	0.3826	0.2433	0.4921	0.017*
C1	0.45886 (13)	0.3782 (2)	0.6864 (2)	0.0111 (3)
C2	0.41839 (13)	0.5301 (2)	0.6268 (3)	0.0136 (3)
H2	0.3623	0.5307	0.5427	0.016*
C3	0.45882 (14)	0.6802 (2)	0.6886 (3)	0.0156 (4)
H3	0.4304	0.7823	0.6470	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.01370 (7)	0.01474 (7)	0.01747 (8)	−0.00335 (4)	0.00325 (5)	0.00078 (4)
C11	0.0139 (8)	0.0163 (8)	0.0132 (9)	−0.0016 (6)	0.0044 (7)	−0.0027 (7)
C1	0.0115 (8)	0.0123 (8)	0.0102 (8)	−0.0008 (5)	0.0039 (6)	−0.0001 (6)
C2	0.0131 (8)	0.0158 (8)	0.0122 (9)	0.0026 (6)	0.0035 (7)	0.0011 (7)
C3	0.0212 (9)	0.0122 (8)	0.0153 (9)	0.0026 (7)	0.0085 (7)	0.0031 (7)

Geometric parameters (Å, º)

I1—C11	2.1902 (19)	C1—C1^{i}	1.410 (4)
C11—C1	1.487 (3)	C2—C3	1.391 (3)
C11—H111	0.9900	C2—H2	0.9500
C11—H112	0.9900	C3—C3^{i}	1.392 (4)
C1—C2	1.399 (3)	C3—H3	0.9500
C1—C11—I1	112.15 (13)	C1^{i}—C1—C11	121.93 (11)
C1—C11—H111	109.2	C3—C2—C1	121.16 (18)
I1—C11—H111	109.2	C3—C2—H2	119.4
C1—C11—H112	109.2	C1—C2—H2	119.4
I1—C11—H112	109.2	C2—C3—C3^{i}	119.72 (12)
H111—C11—H112	107.9	C2—C3—H3	120.1
C2—C1—C1^{i}	119.10 (11)	C3^{i}—C3—H3	120.1
C2—C1—C11	118.94 (18)		
I1—C11—C1—C2	−93.41 (19)	C11—C1—C2—C3	−177.12 (17)

I1—C11—C1—C1^{i}	88.3 (2)	C1—C2—C3—C3^{i}	0.2 (3)
C1^{i}—C1—C2—C3	1.2 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···I1^{ii}	0.95	3.38	4.046 (2)	129
C11—H112···I1^{iii}	0.99	3.33	4.179 (2)	145
C2—H2···I1^{iii}	0.95	3.36	4.257 (2)	158

Symmetry codes: (ii) $x, -y+1, z-1/2$; (iii) $-x+1/2, -y+1/2, -z+1$.

(II) 1,3-Bis(iodomethyl)benzene

Crystal data

$C_8H_8I_2$
M_r = 357.94
Monoclinic, $P2_1/n$
a = 13.5323 (3) Å
b = 4.5464 (1) Å
c = 15.6269 (4) Å
β = 95.203 (1)°
V = 957.46 (4) Å^3
Z = 4
$F(000)$ = 648
D_x = 2.483 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
θ = 2.6–33.0°
μ = 6.50 mm^{-1}
T = 90 K
Needle, colourless
0.45 × 0.06 × 0.05 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.569, T_{max} = 1.000
16804 measured reflections
3435 independent reflections
2826 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 33.4°, θ_{min} = 3.0°
$h = -20 \rightarrow 20$
$k = -6 \rightarrow 5$
$l = -23 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.048
S = 1.06
3435 reflections
91 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0109P)^2 + 1.4343P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 1.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
I1	0.35561 (2)	0.36401 (4)	0.46514 (2)	0.01445 (4)
C11	0.4498 (2)	0.1469 (6)	0.37746 (17)	0.0197 (5)
H11A	0.5193	0.1461	0.4034	0.024*
H11B	0.4282	−0.0599	0.3690	0.024*
C1	0.44471 (19)	0.2984 (6)	0.29275 (16)	0.0152 (5)
C2	0.51858 (18)	0.4993 (5)	0.27547 (16)	0.0138 (5)
H2	0.5707	0.5419	0.3185	0.017*
C3	0.51650 (18)	0.6379 (5)	0.19574 (16)	0.0128 (4)
C31	0.59593 (19)	0.8514 (6)	0.17788 (17)	0.0175 (5)
H31A	0.6261	0.9352	0.2326	0.021*
H31B	0.5667	1.0148	0.1421	0.021*
I3	0.71036 (2)	0.63230 (4)	0.11079 (2)	0.01692 (5)
C4	0.43920 (19)	0.5755 (6)	0.13244 (16)	0.0171 (5)
H4	0.4369	0.6696	0.0780	0.021*
C5	0.36591 (19)	0.3753 (6)	0.14961 (17)	0.0179 (5)
H5	0.3138	0.3322	0.1066	0.022*
C6	0.36832 (19)	0.2379 (6)	0.22919 (18)	0.0178 (5)
H6	0.3177	0.1021	0.2404	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.01484 (7)	0.01475 (9)	0.01451 (8)	0.00033 (6)	0.00534 (5)	0.00083 (6)
C11	0.0223 (12)	0.0167 (13)	0.0216 (12)	0.0064 (11)	0.0091 (10)	0.0020 (11)
C1	0.0174 (11)	0.0125 (12)	0.0167 (11)	0.0032 (10)	0.0065 (9)	0.0003 (9)
C2	0.0149 (11)	0.0112 (12)	0.0155 (11)	0.0011 (9)	0.0035 (9)	−0.0025 (9)
C3	0.0139 (10)	0.0097 (11)	0.0153 (10)	0.0010 (9)	0.0044 (8)	−0.0013 (9)
C31	0.0191 (12)	0.0131 (13)	0.0215 (12)	−0.0020 (10)	0.0083 (10)	−0.0037 (10)
I3	0.01582 (8)	0.01765 (9)	0.01833 (8)	−0.00134 (6)	0.00728 (6)	−0.00027 (6)
C4	0.0184 (11)	0.0182 (13)	0.0148 (11)	0.0024 (10)	0.0016 (9)	0.0005 (10)
C5	0.0152 (11)	0.0191 (13)	0.0190 (12)	0.0004 (10)	−0.0012 (9)	−0.0044 (10)
C6	0.0153 (11)	0.0147 (13)	0.0243 (13)	−0.0029 (10)	0.0057 (10)	−0.0010 (11)

Geometric parameters (Å, º)

I1—C11	2.189 (3)	C3—C31	1.493 (3)
I1—I3^{i}	3.8662 (2)	C31—I3	2.187 (2)
C11—C1	1.488 (4)	C31—H31A	0.9900
C11—H11A	0.9900	C31—H31B	0.9900
C11—H11B	0.9900	C4—C5	1.390 (4)
C1—C6	1.394 (4)	C4—H4	0.9500
C1—C2	1.399 (3)	C5—C6	1.390 (4)
C2—C3	1.394 (3)	C5—H5	0.9500
C2—H2	0.9500	C6—H6	0.9500
C3—C4	1.402 (3)		

C11—I1—I3^{i}	117.47 (7)	C3—C31—I3	110.27 (16)
C1—C11—I1	111.45 (17)	C3—C31—H31A	109.6
C1—C11—H11A	109.3	I3—C31—H31A	109.6
I1—C11—H11A	109.3	C3—C31—H31B	109.6
C1—C11—H11B	109.3	I3—C31—H31B	109.6
I1—C11—H11B	109.3	H31A—C31—H31B	108.1
H11A—C11—H11B	108.0	C5—C4—C3	119.7 (2)
C6—C1—C2	119.2 (2)	C5—C4—H4	120.1
C6—C1—C11	120.9 (2)	C3—C4—H4	120.1
C2—C1—C11	119.9 (2)	C6—C5—C4	120.5 (2)
C3—C2—C1	120.7 (2)	C6—C5—H5	119.7
C3—C2—H2	119.6	C4—C5—H5	119.7
C1—C2—H2	119.6	C5—C6—C1	120.3 (2)
C2—C3—C4	119.5 (2)	C5—C6—H6	119.9
C2—C3—C31	120.3 (2)	C1—C6—H6	119.9
C4—C3—C31	120.2 (2)		

I1—C11—C1—C6	−83.6 (3)	C4—C3—C31—I3	−83.7 (3)
I1—C11—C1—C2	97.9 (2)	C2—C3—C4—C5	−0.3 (4)
C6—C1—C2—C3	−0.1 (4)	C31—C3—C4—C5	179.7 (2)
C11—C1—C2—C3	178.4 (2)	C3—C4—C5—C6	0.4 (4)
C1—C2—C3—C4	0.2 (4)	C4—C5—C6—C1	−0.3 (4)
C1—C2—C3—C31	−179.8 (2)	C2—C1—C6—C5	0.2 (4)
C2—C3—C31—I3	96.4 (2)	C11—C1—C6—C5	−178.4 (2)

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

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···I1^{ii}	0.99	3.22	4.060 (3)	144
C5—H5···I1^{iii}	0.95	3.25	4.078 (3)	147
C31—H31*A*···I3^{iv}	0.99	3.27	4.224 (3)	162
C31—H31*A*···*Cg*^{v}	0.99	2.84	3.453 (3)	121

Symmetry codes: (ii) $x, y-1, z$; (iii) $-x+1/2, y-1/2, -z+1/2$; (iv) $-x+3/2, y+1/2, -z+1/2$; (v) $x, y+1, z$.