

2,3,6-Trichloriodobenzene

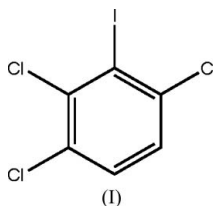
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Key indicators

Single-crystal X-ray study
T = 190 K
Mean $\sigma(\lambda)$ = 0.000 Å
Disorder in main residue
R factor = 0.034
wR factor = 0.089
Data-to-parameter ratio = 12.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, C₆H₂Cl₃I, is a building block of polychlorinated biphenyls (PCBs). In the crystal structure, the molecule is disordered over three orientations.Received 18 September 2006
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Comment

Trichloriodobenzenes are important starting materials for the synthesis of *ortho*-substituted polychlorinated biphenyls (PCBs) of environmental relevance (Kania-Korwel *et al.*, 2005). To date, the crystal structures of only two trichloriodobenzenes, namely 2,4,5-trichloriodobenzene (Kania-Korwel, Lehmler *et al.*, 2003) and 2,4,6-trichloriodobenzene (Kania-Korwel, Robertson *et al.*, 2003), have been published. We report here the crystal structure of 2,3,6-trichloriodobenzene, (I), to add to the available database of crystal structures of chlorinated iodobenzenes.The molecule of compound (I) is disordered over three orientations in the solid state; one orientation is shown in Fig. 1. The second orientation is obtained by an approximate twofold rotation about an axis passing through the mid-point of the C1–C2 and C4–C5 bonds of main orientation. The third orientation is approximated by a twofold rotation about the C1–I1 bond direction. As for 2,4,5-trichloriodobenzene (Kania-Korwel, Lehmler *et al.*, 2003), the disordered packing of (I) in the solid state is likely a result of the unsymmetrical chlorine substitution.

Experimental

The title compound, (I), was synthesized by chlorination of 2,6-dichloriodobenzene (Waller & Mash 1997). Colourless blades were obtained upon recrystallization from hot methanol.

Crystal data

C ₆ H ₂ Cl ₃ I	Z = 4
<i>M_r</i> = 307.33	<i>D_x</i> = 2.391 Mg m ⁻³
Orthorhombic, <i>Pca</i> 2 ₁	Mo <i>K</i> α radiation
<i>a</i> = 16.4873 (16) Å	<i>μ</i> = 4.61 mm ⁻¹
<i>b</i> = 4.0530 (4) Å	<i>T</i> = 190 (2) K
<i>c</i> = 12.7746 (13) Å	Blade, colourless
<i>V</i> = 853.64 (15) Å ³	0.24 × 0.09 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SCALEPACK; Otwinowski &
 Minor, 1997)
 $T_{\min} = 0.404$, $T_{\max} = 0.802$

20082 measured reflections
 1946 independent reflections
 1678 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.089$
 $S = 1.04$
 1946 reflections
 155 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.953P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.008$
 $\Delta\rho_{\text{max}} = 0.74 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.50 \text{ e } \text{Å}^{-3}$
 Absolute structure: Flack (1983),
 924 Friedel pairs
 Flack parameter: 0.30 (4)

Table 1

Selected geometric parameters (Å , $^\circ$).

C1—C6	1.392 (18)	C3—C4	1.414 (16)
C1—C2	1.405 (9)	C3—Cl3	1.718 (10)
C1—I1	2.131 (12)	C4—C5	1.346 (9)
C2—C3	1.370 (18)	C5—C6	1.387 (16)
C2—Cl2	1.710 (15)	C6—Cl6	1.719 (11)
C6—C1—C2	118.9 (13)	C2—C3—Cl3	122.0 (9)
C6—C1—I1	120.8 (8)	C4—C3—Cl3	117.3 (9)
C2—C1—I1	120.3 (12)	C5—C4—C3	119.2 (12)
C3—C2—C1	119.7 (13)	C4—C5—C6	121.2 (12)
C3—C2—Cl2	120.8 (10)	C5—C6—C1	120.3 (9)
C1—C2—Cl2	119.5 (13)	C5—C6—Cl6	119.8 (9)
C2—C3—C4	120.7 (9)	C1—C6—Cl6	119.8 (9)

The molecule is disordered over three orientations. The site occupancies refined to 0.625 (5):0.339 (3):0.036 (3) and were restrained to sum to 1.0. The molecular geometries were restrained to be the same. 'Partial' atoms occupying the same general site (e.g. I1, Cl2', and I1'') were constrained to have the same anisotropic displacement parameters, except Cl3'' [$U_{\text{iso}}(\text{Cl3}'') = 1.2U_{\text{eq}}(\text{C3}'')$]. The three orientations were restrained to be planar. An anti-bumping restraint was imposed on Cl3''...Cl6'' [$\text{symmetry code: } (i) \frac{1}{2} + x, -y, z$]. The value of the Flack parameter indicates partial inversion twinning. The riding model was used to position H atoms and to their isotropic thermal parameters.

Data collection: COLLECT (Nonius, 2000); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: SCALEPACK and DENZO (Otwinowski & Minor, 1997);

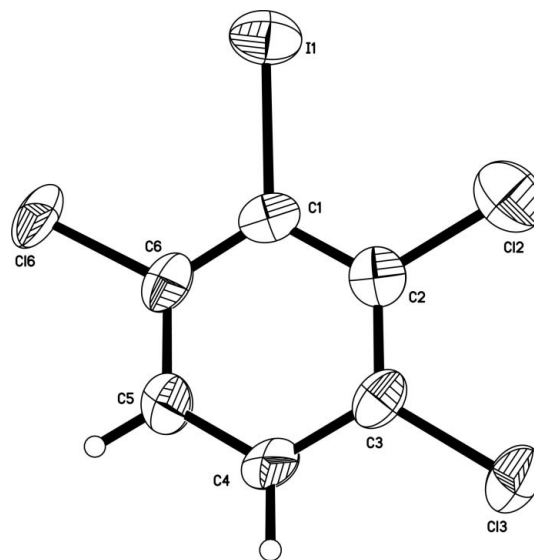


Figure 1

Displacement ellipsoid plot of (I) (50% probability level). Only the main orientation is shown.

program(s) used to solve structure: SHELXTL (Sheldrick, 2001); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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