Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,4,5-Trichloro-1-iodobenzene

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.035
wR factor = 0.078
Data-to-parameter ratio = 16.7

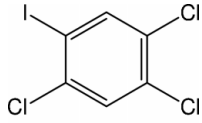
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 29 May 2003
Accepted 23 June 2003
Online 30 June 2003

The crystal structure of 2,4,5-trichloroiodobenzene, $C_6H_2Cl_3I$, a precursor of polychlorinated biphenyls (PCBs), is described. The molecule is disordered in two distinct ways. Despite its lack of inversion point symmetry, it straddles an inversion centre in space group $P2_1/n$. An additional minor disorder component occurs as a result of a 180° rotation of some molecules about [011].

Comment

PCBs are an important group of widespread environmental contaminants, known to cause a variety of toxic effects (Robertson & Hansen, 2001). During our attempts to synthesize pure PCB congeners, we obtained crystals of the title compound, (I), whose structure is reported here.



(I)

2,4,5-trichloro-1-iodobenzene is one compound in a series of chlorinated derivatives of iodo- or bromobenzenes, which are 'building blocks' to obtain polychlorinated biphenyls (PCBs). The 2,4,5-trichlorophenyl moiety is commonly found in PCB congeners and its three-dimensional structure determines their toxicity (Lehmler & Robertson, 2001). The availability of crystal structure data will therefore aid us in our understanding of toxicity of PCB congeners with a 2,4,5-substitution pattern.

Experimental

The 2,4,5-trichloroiodobenzene crystals were obtained while attempting to synthesize 2,4,5-trichlorobiphenyl in the Suzuki reaction (Lehmler *et al.*, 2001). Colorless block-shaped crystals formed from solution in methanol.

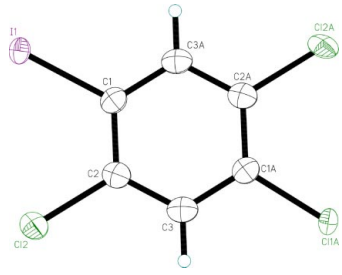


Figure 1
A view of 2,4,5-trichloroiodobenzene, with non-H atom displacement ellipsoids drawn at the 50% probability level. The suffix *A* denotes an atom generated by inversion symmetry.

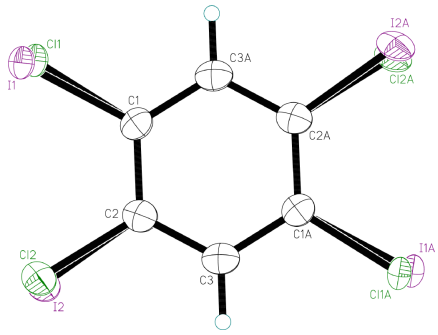


Figure 2
The nature of the disorder in 2,4,5-trichloroiodobenzene at 90 K.

Crystal data

$C_6H_2Cl_3I$	D_x = 2.471 Mg m^{-3}
M_r = 307.33	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 993 reflections
a = 3.9191 (8) Å	θ = 1.0–27.5°
b = 10.894 (2) Å	μ = 4.76 mm^{-1}
c = 9.852 (2) Å	T = 90.0 (2) K
β = 100.87 (3)°	Block, colorless
V = 413.07 (15) Å^3	0.20 × 0.10 × 0.10 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	884 reflections with $I > 2\sigma(I)$
ω scans at fixed χ = 55°	R_{int} = 0.035
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	θ_{max} = 27.5°
T_{min} = 0.570, T_{max} = 0.621	$h = -4 \rightarrow 5$
6306 measured reflections	$k = -13 \rightarrow 14$
936 independent reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2))^2 + 1.7326P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.035	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.078	$\Delta\rho_{max}$ = 0.64 e Å^{-3}
S = 1.35	$\Delta\rho_{min}$ = −0.72 e Å^{-3}
936 reflections	Extinction correction: *SHELXL*97
56 parameters	Extinction coefficient: 0.011 (2)
H-atom parameters constrained	

The structure is extensively disordered. The asymmetric unit contains nominally half a molecule, but it is disordered such that the Cl and I atoms are partially superimposed. Given the known chemical composition, a satisfactory model requires that the sum of the Cl atom occupancies should be 1.5 over the two halogen sites in the asymmetric unit, which was accomplished with the SUMP command in *SHELXL*97 (Sheldrick, 1997). This, in turn, allowed the I occupancy total over the two sites to be 0.5 while at the same time ensuring that the total occupancy of each site itself is unity. Bond distances for C—Cl and C—I are heavily correlated as a result of the disorder and it was not possible to independently refine both. The length of C—I bonds was restrained to be 1.15 times that of the corresponding C—Cl bonds. This treatment ensured that the C—Cl bonds were typical for this class of compound (*e.g.* Lehmler *et al.*, 2001). The C—I bond length reported here should thus be regarded as a compromise to improve the least-squares fit and no particular claims are made for the accuracy of this C—I distance. The anisotropic displacement parameters (ADPs) of each individual pair of overlapping Cl/I were constrained to be equal, while the ADPs of different pairs were restrained to be similar.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997) and local procedures.

This publication was made possible by a Fulbright Junior Research Grant and a Kosciuszko Foundation Grant to Izabela Kania-Korwel. The project was supported by grant number P42 ES 07380 from the National Institute of Environmental Health Sciences, NIH.

References

Lehmler, H.-J., Parkin, S. & Robertson, L. W. (2001). *Acta Cryst.* E**57**, o111–o112.
Lehmler, H.-J. & Robertson, L. W. (2001). *Chemosphere*, **45**, 137–143.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
Robertson, L. W. & Hansen, L. G. (2001). *Recent Advances in the Environmental Toxicology and Health Effects of PCBs*. Lexington: University Press of Kentucky.
Sheldrick, G. M. (1994). *SHELXTL/PC*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.