

## 2,4,6-Trichloriodobenzene

Izabela Kania-Korwel,<sup>a</sup> Larry W. Robertson,<sup>a</sup> Hans-Joachim Lehmler<sup>a</sup> and Sean Parkin<sup>b\*</sup><sup>a</sup>Department of Occupational and Environmental Health, College of Public Health, 100 Oakdale Campus #124 IREH, Iowa City, Iowa 52242-5000, USA, and <sup>b</sup>Department of Chemistry, University of Kentucky, Lexington, KY 40506-0055, USA

Correspondence e-mail: spark2@uky.edu

## Key indicators

Single-crystal X-ray study  
 $T = 90$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.021  
 $wR$  factor = 0.052  
Data-to-parameter ratio = 20.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The crystal structure of 2,4,6-trichloriodobenzene,  $\text{C}_6\text{H}_2\text{Cl}_3\text{I}$ , a precursor of polychlorinated biphenyls (PCBs), is described.

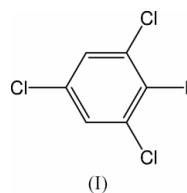
Received 23 September 2003

Accepted 8 October 2003

Online 23 October 2003

## Comment

Chlorinated iodo- and bromobenzenes, such as 2,4,6-trichloriodobenzene, are precursors of polychlorinated biphenyls (PCBs), a group of important and widespread environmental pollutants (Lehmler *et al.*, 2001). During attempts to develop a novel synthesis of tetra-*ortho*-substituted PCBs, we obtained crystals of the title compound, (I). In spite of its relative simplicity, the crystal structure has not been reported in the literature, but that of an isomer, 2,4,5-trichloriodobenzene, was recently published (Kania-Korwel *et al.*, 2003). Notwithstanding the outward similarity of the two isomers, their crystal structures show dramatic differences. While the 2,4,5-trichloriodobenzene isomer was extensively disordered in the crystalline state, the title compound is well ordered.



## Experimental

The 2,4,6-trichloriodobenzene crystals were obtained while attempting to synthesize 2,2',4,4',6,6'-hexachlorobiphenyl using a Suzuki coupling reaction (Lehmler & Robertson, 2001). White needles formed upon recrystallization from methanol.

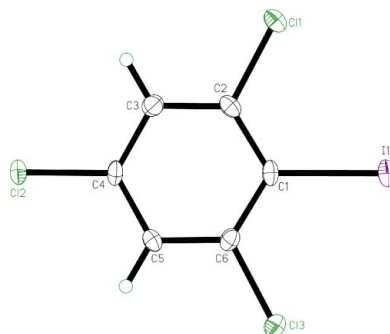


Figure 1

An ellipsoid plot of 2,4,6-trichloro-1-iodobenzene, with non-H atoms drawn at the 50% probability level.

*Crystal data*

C<sub>6</sub>H<sub>2</sub>Cl<sub>3</sub>I  
*M<sub>r</sub>* = 307.33  
 Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 3.9970 (1) Å  
*b* = 21.5840 (4) Å  
*c* = 9.7510 (2) Å  
 $\beta$  = 100.994 (1)°  
*V* = 825.79 (3) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 2.472 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 1946  
 reflections  
 $\theta$  = 1.0–27.5°  
 $\mu$  = 4.76 mm<sup>-1</sup>  
*T* = 90.0 (2) K  
 Block, colourless  
 0.15 × 0.10 × 0.10 mm

*Data collection*

Nonius KappaCCD diffractometer  
 $\omega$  scans at fixed  $\chi$  = 55°  
 Absorption correction: multi-scan  
 (SCALEPACK; Otwinowski &  
 Minor, 1997)  
*T*<sub>min</sub> = 0.514, *T*<sub>max</sub> = 0.621  
 3722 measured reflections

1891 independent reflections  
 1689 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.019  
 $\theta$ <sub>max</sub> = 27.5°  
*h* = -5 → 5  
*k* = -27 → 27  
*l* = -12 → 12

*Refinement*

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.021  
*wR* (*F*<sup>2</sup>) = 0.052  
*S* = 1.10  
 1891 reflections  
 91 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0246P)^2 + 0.3565P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 (Δ/σ)<sub>max</sub> = 0.001  
 Δρ<sub>max</sub> = 0.88 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.84 e Å<sup>-3</sup>

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1994); software used to prepare material for publication: *SHELX97-2* (Sheldrick, 1997) and local programs.

This publication was made possible by a Fulbright Junior Research Grant and a Kosciuszko Foundation Grant to IKK. The project was supported by grant No. P42 ES 07380 from the National Institute of Environmental Health Sciences, NIH.

**References**

- Kania-Korwel, I., Robertson, L. W., Lehmler, H.-J. & Parkin, S. (2003) *Acta Cryst.* **E59**, o1048–o1049.  
 Lehmler, H.-J., Parkin, S. & Robertson, L. W. (2001). *Acta Cryst.* **E57**, 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.  
 Sheldrick, G. M. (1994). *SHELXTL/PC*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (1997). *SHELXL97*, *SHELXS97* and *SHELX97-2*. University of Göttingen, Germany.