organic papers

1,3,5-Triiodobenzene

Dominik Margraf[a] **and Jan W. Bats**[b]*

[a]Institut für Physikalische und Theoretische Chemie, Universität Frankfurt, Marie-Curie-Strasse 11, D-60439 Frankfurt am Main, Germany, and [b]Institut für Organische Chemie, Universität Frankfurt, Marie-Curie-Strasse 11, D-60439 Frankfurt am Main, Germany

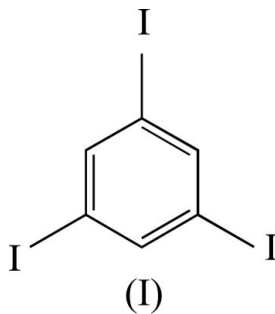Correspondence e-mail:
bats@chemie.uni-frankfurt.de

Key indicators

Single-crystal X-ray study
T = 161 K
Mean σ(C–C) = 0.004 Å
R factor = 0.022
wR factor = 0.049
Data-to-parameter ratio = 36.9

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

Molecules of the title compound, $C_6H_3I_3$, form stacks along the [100] direction. Neighbouring stacks are connected by weak intermolecular C—H···I interactions.

Comment

1,3,5-Triiodobenzene, (I), was prepared as a starting compound for Sonogashira coupling reactions in order to synthesize rigid rod-like bi- and triradical systems.

(I)

The unit-cell dimensions of (I) are rather similar to those reported for the structures of 1,3,5-trichlorobenzene and 1,3,5-tribromobenzene (Milledge & Pant, 1960). The space group is also identical. Thus, all three structures are probably isomorphous. Surprisingly, the fractional coordinates observed for (I) do not agree with those reported for the corresponding Cl and Br compounds. There is an approximate correspondence, however, if the coordinates reported for the Cl and Br compounds are shifted by a vector (1/4, 1/8, 1/4). The structures reported by Milledge & Pant (1960) are based on a limited number of film reflections, and so a systematic error in those structures cannot be excluded.

The molecular structure of (I) is shown in Fig. 1. The benzene ring is essentially planar, with a mean deviation of the C atoms from this plane of 0.004 Å. Atom I1 deviates by 0.104 (5) Å from the plane of the benzene ring. Atoms I2 and I3 show no significant deviation from this plane [0.006 (5) Å for I2 and 0.002 (5) Å for I3]. The C—I bond lengths of 2.090 (3), 2.091 (3) and 2.101 (3) Å are in good agreement with the values of 2.086 and 2.096 Å found for two triiodobenzene clathrates (Jetti *et al.*, 2001).

The molecules form stacks parallel to the *a* axis (Fig. 2) and the distance between the planes of neighbouring benzene rings in the stack is 3.58 Å. Neighbouring benzene rings show partially overlapping π systems. The shortest intermolecular C(π)···C(π) distance is 3.605 (5) Å, between atoms C1 and C4($x - 1, y, z$). Deviation of atom I1 from the benzene plane is required in order to avoid a short intermolecular contact
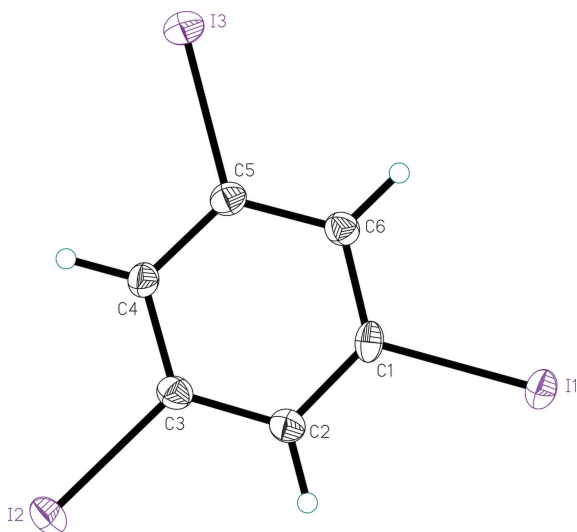
Figure 1
The molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.
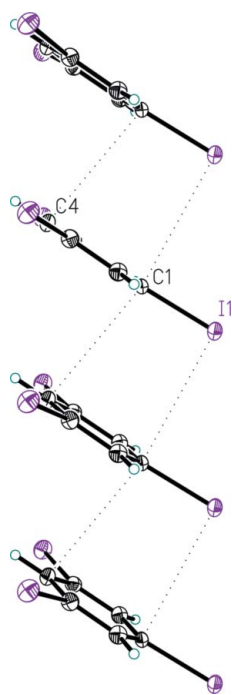

Figure 2
Molecules of (I), stacked along the *a* axis. Dotted lines represent the shortest intermolecular $C(\pi)\cdots C(\pi)$ contact of 3.605 (5) Å and the intermolecular steric $C\cdots I$ contact of 3.696 (3) Å.

between atoms I1 and C1($x - 1$, y, z). The observed intermolecular $I\cdots C$ distance is 3.696 (3) Å. Neighbouring stacks are connected by weak intermolecular $C-H\cdots I$ interactions (Fig. 3). There are three symmetry-independent $C-H\cdots I$ interactions, with $H\cdots I$ distances of 3.27, 2.37 and 3.38 Å, and $C-H-I$ angles of 156, 160 and 164°, respectively.
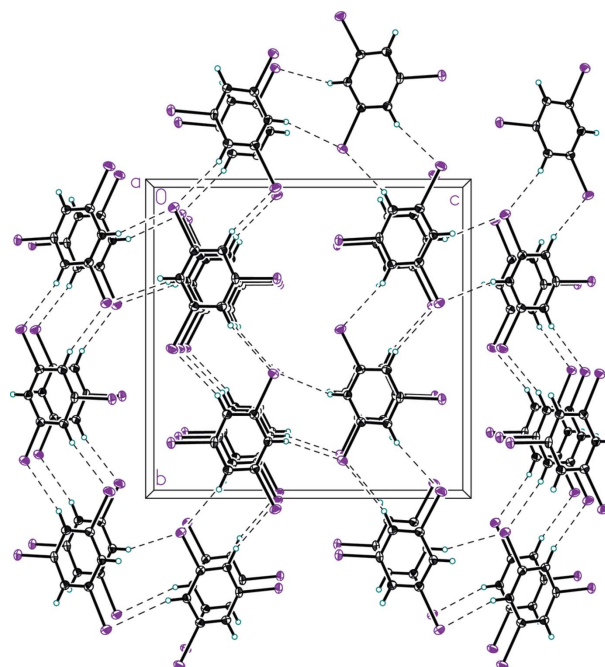

Figure 3
The crystal packing of (I), viewed down the *a* axis. Weak intermolecular $C-H\cdots I$ interactions are represented as broken lines. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

1,3,5-Triiodobenzene was prepared from 1,3,5-tribromobenzene, following the procedure described by Schöberl *et al.* (1997). Single crystals were obtained by evaporation of a solution of (I) in $CDCl_3$.

Crystal data

$C_6H_3I_3$	Mo $K\alpha$ radiation
$M_r = 455.78$	Cell parameters from 249
Orthorhombic, $P2_12_12_1$	reflections
$a = 4.3287$ (6) Å	$\theta = 3–23°$
$b = 14.224$ (2) Å	$\mu = 10.41$ mm^{-1}
$c = 14.515$ (2) Å	$T = 161$ (2) K
$V = 893.7$ (2) Å^3	Rod, colourless
$Z = 4$	$0.46 \times 0.08 \times 0.07$ mm
$D_x = 3.387$ Mg m^{-3}	

Data collection

Siemens SMART 1K CCD area-detector diffractometer	3064 independent reflections
	2925 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.051$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)	$\theta_{max} = 32.4°$
	$h = -6 \rightarrow 6$
$T_{min} = 0.176$, $T_{max} = 0.483$	$k = -20 \rightarrow 20$
15783 measured reflections	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = 0.004$
$R[F^2 > 2\sigma(F^2)] = 0.022$	$\Delta\rho_{max} = 1.07$ e Å^{-3}
$wR(F^2) = 0.049$	$\Delta\rho_{min} = -0.58$ e Å^{-3}
$S = 1.07$	Extinction correction: *SHELXL97*
3064 reflections	(Sheldrick, 1997)
83 parameters	Extinction coefficient: 0.00248 (18)
H-atom parameters constrained	Absolute structure: Flack (1983),
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$	with 1223 Friedel pairs
where $P = (F_o^2 + 2F_c^2)/3$	Flack parameter: 0.39 (4)

organic papers

Equivalent reflections were merged and Friedel opposites were not merged. There are 1223 unique Friedel pairs. The value of the Flack (1983) parameter indicates that the crystal was probably a twin consisting of domains with opposite chirality. H atoms were positioned geometrically and refined as riding, with Csp^2—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXL97*.

References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Jetti, R. K. R., Nangia, A., Xue, F. & Mak, T. C. W. (2001). *Chem. Commun.* pp. 919–920.
Milledge, H. J. & Pant, L. M. (1960). *Acta Cryst.* **13**, 285–290.
Schöberl, U., Magnera, T. F., Harrison, R. M., Fleischer, F., Pflug, J. L., Schwab, P. F. H., Meng, X., Lipiak, D., Noll, B. C., Allured, V. S., Rudalevige, T., Lee, S. & Michl, J. (1997). *J. Am. Chem. Soc.* **119**, 3907–3917.
Sheldrick, G. M. (1996). *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2000). *SADABS*. University of Göttingen, Germany.
Siemens (1995). *SMART* (Version 4.05) and *SAINT* (Version 4.05). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.