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5-Bromo-1,3-dichloro-2-iodobenzene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.015 Å
R factor = 0.057
wR factor = 0.147
Data-to-parameter ratio = 17.3

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

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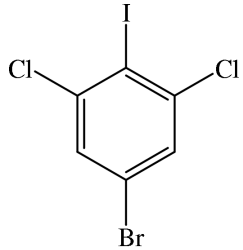
The title compound, $C_6H_2BrCl_2I$, crystallizes in space group $P2_1/c$ with two molecules in the asymmetric unit. The molecules stack in two different directions, with their plane normals approximately parallel to [110] and [1$\bar{1}$0]. The molecules of the asymmetric unit are held together by π–π interactions.

Comment

The title compound, (I), was synthesized for a study of the electrochemical reductive cleavage of carbon–halogen bonds (Arun Prasad & Sagaranarayanan, 2004). The average C—Cl bond length is 1.735 Å, which is in excellent agreement with the normal value of 1.739 Å (Allen *et al.*, 1987). Likewise, the average C—Br and C—I bond lengths of 1.879 and 2.086 Å are in good agreement with the normal values of 1.899 and 2.095 Å, respectively (Allen *et al.*, 1987). The halogen atoms are very slightly displaced from the benzene ring plane, as a result of steric repulsion; the largest deviation is 0.089 (3) Å for atom I2. Such steric interactions between the halogens in polyhalobenzenes are well known (Solenova *et al.*, 1960).



(I)

The asymmetric unit of (I) contains two molecules that are nearly parallel to each other, the angle between their plane normals being 10.82 (5)°. The approximate perpendicular distance between the planes, 3.42 Å, is indicative of π–π interactions. The asymmetric unit pairs stack along planes normal to [110] and [1$\bar{1}$0]. The perpendicular distance of 3.87 Å between the stacked pairs suggests that there is no extended π–π interaction in the stacking direction.

Experimental

2,6-Dichloroaniline was brominated by passing bromine vapour into a solution of 2,6-dichloroaniline (21 g) in hydrochloric acid (6 *M*, 120 ml). Solid 4-bromo-2,6-dichloroaniline was filtered off and purified by column chromatography (silica gel). 4-Bromo-2,6-dichloroaniline (30 g) was then diazotized in hydrochloric acid (6 *M*, 150 ml) using aqueous sodium nitrite (15 g/36 ml water) and the resulting solution was slowly added to aqueous potassium iodide (36 g/45 ml

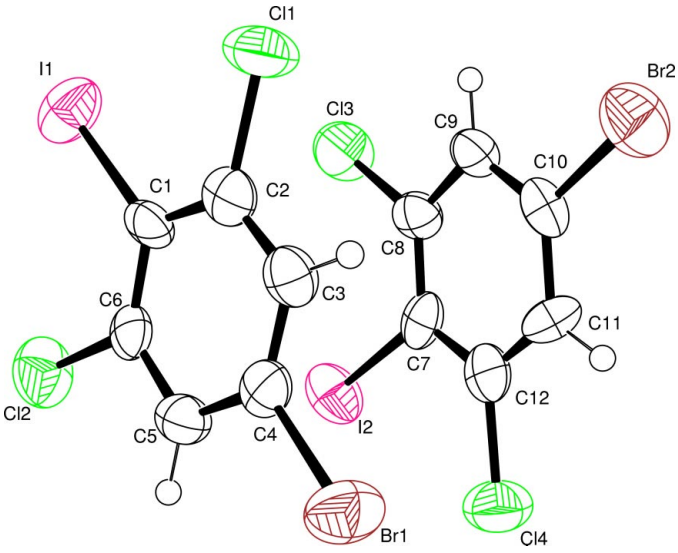


Figure 1
The asymmetric unit of the title compound, with the atomic numbering scheme. Displacement parameter ellipsoids are drawn at the 50% probability level.

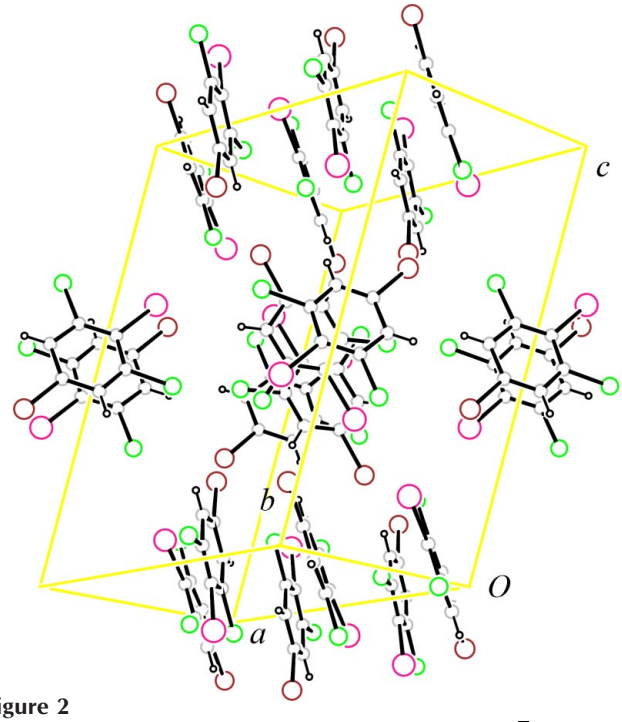


Figure 2
A packing diagram of the title compound, viewed down [$\bar{1}$10].

water). When no further gas was evolved, the crude product was filtered off, and washed first with aqueous sodium hydroxide, subsequently with sodium metabisulphite and finally with water. 5-Bromo-1,3-dichloro-2-iodobenzene was separated and purified by column chromatography (silica gel) using hexane as the eluant; slow solvent evaporation produced X-ray quality crystals of (I).

Crystal data

$C_6H_2BrCl_2I$
M_r = 351.79
Monoclinic, $P2_1/c$
a = 11.630 (6) Å
b = 10.020 (4) Å
c = 16.30 (1) Å
β = 109.84 (4)°
V = 1786.6 (15) Å^3
Z = 8
D_x = 2.616 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–15°
μ = 8.58 mm^{-1}
T = 293 (2) K
Block, colourless
0.3 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.361, T_{max} = 1.000
3287 measured reflections
3125 independent reflections
1730 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 11$
$l = -19 \rightarrow 18$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.147
S = 0.92
3125 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0856P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.16 e Å^{-3}
$\Delta\rho_{min}$ = −1.71 e Å^{-3}

All H atoms were placed in calculated positions, with C—H = 0.93 Å, and were refined using the riding-model approximation, with U_{iso} values constrained to 1.2U_{eq} of the carrier atom. The largest residual electron-density peak is located 1.13 Å from atom I1 and the deepest hole is located 1.20 Å from atom I1.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 in *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SIR*92 in *WinGX*; program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*3 in *WinGX*; software used to prepare material for publication: *SHELXL*97.

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