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4-Bromo-2,6-dichloroaniline

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.013 Å
R factor = 0.058
wR factor = 0.198
Data-to-parameter ratio = 15.4

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

The title compound, $C_6H_4BrCl_2N$, crystallizes in the space group $P2_1/c$ with two molecules in the asymmetric unit. Molecules related by an a-axis translation are stacked over each other, bound by π–π interactions. Molecules in adjacent stacks are linked to each other through weak N—H···N hydrogen bonds.

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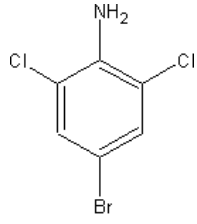
Comment

The title compound, (I), was synthesized for a study of the electrochemical reductive cleavage of carbon–halogen bonds (Arun Prasad & Sangaranarayanan, 2004). It crystallizes with two molecules in the asymmetric unit. The average C—Br and C—Cl bond lengths of 1.895 and 1.743 Å are in excellent agreement with the standard values of 1.899 (12) and 1.739 (10) Å, respectively (Allen *et al.*, 1987).



(I)

Molecules related by an a-axis translation are stacked over each other and the perpendicular distance between the stacking planes is 3.596 Å (Fig. 2). It is, therefore, inferred that the stacked molecules are bound to each other by π–π interactions (Hunter & Sanders, 1990). The stacked columns are weakly linked by N—H···N hydrogen bonds (Table 1).

Experimental

2,6-Dichloroaniline (21 g) was added to 6 *M* hydrochloric acid (120 ml) with continuous stirring. After complete dissolution of 2,6-

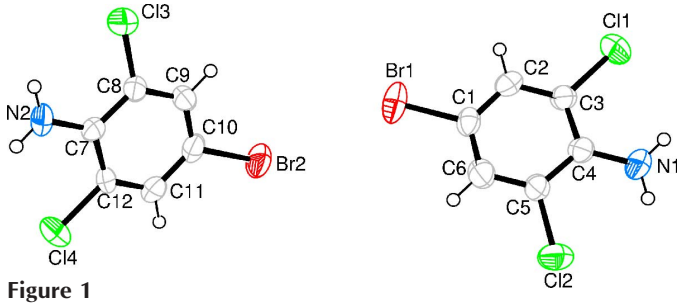


Figure 1
The molecular structure of the asymmetric unit of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

dichloroaniline, bromine vapour was passed into the solution, during which the reaction flask was cooled with ice. When the solution attained a yellow colour, the passage of bromine vapour was stopped and the final product was a dark pasty mass. Crude 4-bromo-2,6-dichloroaniline was filtered off, washed well with water and thoroughly dried. It was then purified by column chromatography (silica gel) using hexane as eluent; slow solvent evaporation produced colourless crystals of (I).

Crystal data

$C_6H_4BrCl_2N$
M_r = 240.91
Monoclinic, $P2_1/c$
a = 4.015 (3) Å
b = 15.41 (1) Å
c = 25.730 (5) Å
β = 91.22 (7)°
V = 1591.6 (16) Å^3
Z = 8
D_x = 2.011 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–15°
μ = 5.75 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} = 0.187, T_{max} = 0.316
3225 measured reflections
2796 independent reflections
1287 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 25.0°
$h = 0 \rightarrow 4$
$k = 0 \rightarrow 18$
$l = -30 \rightarrow 30$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.198
S = 1.00
2796 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1152P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0043 (11)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N2^{i}	0.86	2.39	3.148 (11)	147
N2—H2A···N1^{ii}	0.86	2.41	3.199 (11)	152

Symmetry codes: (i) $1+x, y-1, z$; (ii) $x, 1+y, z$.

All H atoms were positioned geometrically and were refined using the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(carrier atom) and C—H = 0.93 Å or N—H = 0.86 Å.

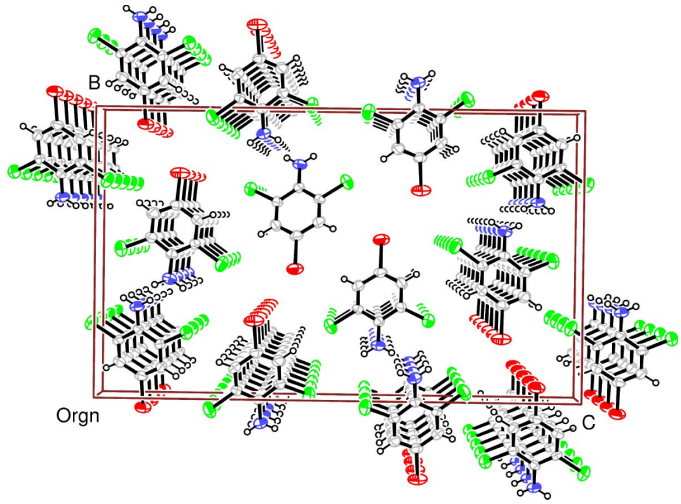


Figure 2
A packing diagram of the title compound, viewed down [100].

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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