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2,6-Dibromo-4-chloroaniline

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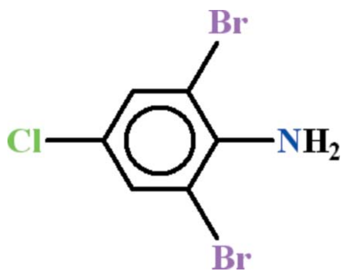
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.024; wR factor = 0.056; data-to-parameter ratio = 20.7.

The title compound, $C_6H_4Br_2ClN$, is almost planar (r.m.s. deviation = 0.024 Å) and two intramolecular N—H⋯Br hydrogen bonds generate $S(5)$ rings. In the crystal, N—H⋯Br hydrogen bonds link the molecules into chains propagating in [010].

Related literature

For related structures, see: Schlemper & Konnert (1967): Takazawa *et al.* (1989). For the synthesis, see: Harrison *et al.* (1981). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_6H_4Br_2ClN$
M_r = 285.37
Monoclinic, $P2_1/n$
a = 13.3132 (7) Å
b = 3.9387 (2) Å
c = 16.5476 (9) Å
β = 112.318 (2)°
V = 802.70 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 10.35 mm^{-1}
T = 296 K
0.35 × 0.15 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.170, T_{max} = 0.292
6640 measured reflections
1900 independent reflections
1429 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.056
S = 1.02
1900 reflections
92 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯Br2	0.86	2.64	3.067 (2)	112
N1—H1A⋯Br2^{i}	0.86	2.91	3.380 (3)	117
N1—H1B⋯Br1	0.86	2.67	3.099 (3)	112

Symmetry code: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6810).

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supplementary materials

Acta Cryst. (2012). E**68**, o1904 [doi:10.1107/S1600536812023331]

2,6-Dibromo-4-chloroaniline

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Comment

The title compound (I), (Fig. 1) has been synthesized as a pre-cursor. The crystal structures of 4-chloroaniline (Takazawa *et al.*, 1989), 2,4, 6-tribromoaniline (Schlemper & Konnert, 1967) have been published which are related to (I).

The molecule as a whole is almost planar with r. m. s. deviation of 0.0242 Å. In (I), there exist intramolecular H-bonding of N—H···Br type to form two S(5) rings (Bernstein *et al.*, 1995). The molecules are connected along the *b*-axis due to H-bondings of N—H···Br type (Table 1, Fig. 2). There does not exist any kind of π-interaction.

Experimental

The title compound has been synthesized from the 4-chloroaniline using the method of Harrison, *et al.*, 1981.

m. p. 352–354 K.

Refinement

The H-atoms were positioned geometrically at C—H = 0.93 and N—H = 0.86 Å, respectively and included in the refinement as riding with $U_{iso}(H) = xU_{eq}(C, N)$, where $x = 1.2$ for all H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

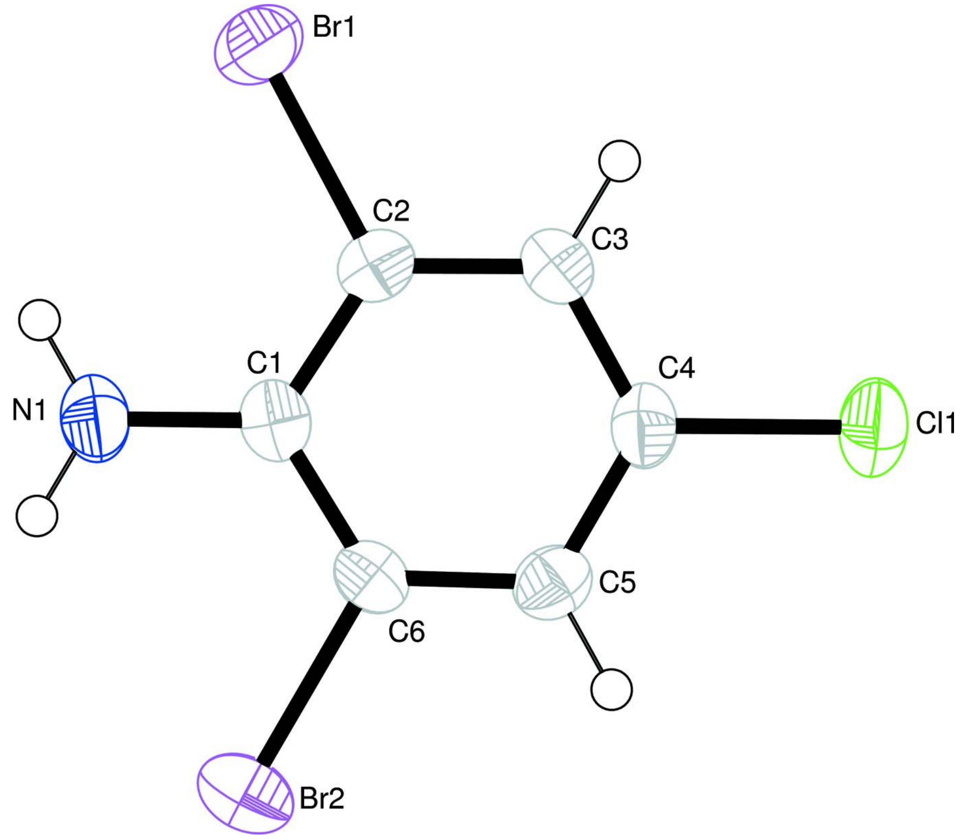

Figure 1

View of the title compound with displacement ellipsoids drawn at the 50% probability level.

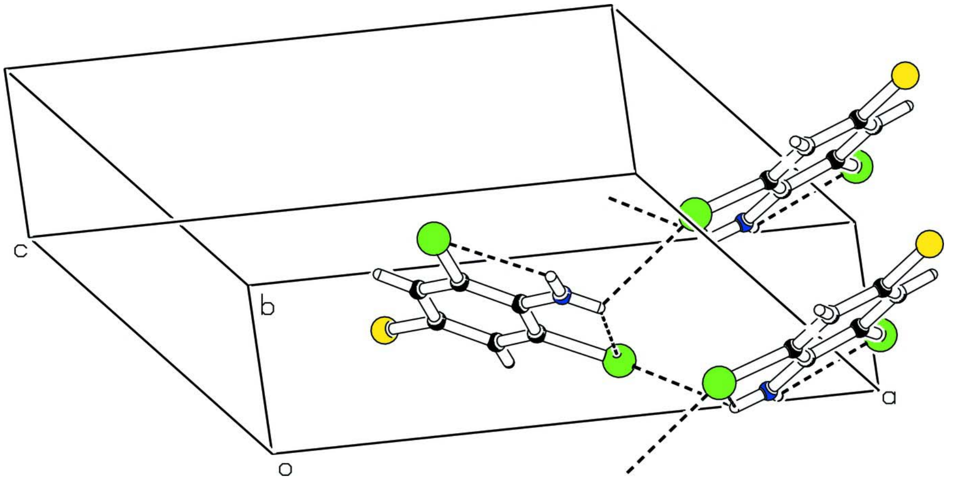

Figure 2

The partial packing (*PLATON*; Spek, 2009), which shows that molecules form C(2) chains extending along [0 1 0] direction.

2,6-Dibromo-4-chloroaniline

Crystal data

$C_6H_4Br_2ClN$
$M_r = 285.37$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 13.3132\ (7)$ Å
$b = 3.9387\ (2)$ Å
$c = 16.5476\ (9)$ Å
$\beta = 112.318\ (2)°$
$V = 802.70\ (7)$ Å^3
$Z = 4$

$F(000) = 536$
$D_x = 2.361$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1429 reflections
$\theta = 2.5–27.9°$
$\mu = 10.35$ mm^{-1}
$T = 296$ K
Needle, dark brown
0.35 × 0.15 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.60 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.170$, $T_{max} = 0.292$

6640 measured reflections
1900 independent reflections
1429 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.5°$
$h = -17 \rightarrow 17$
$k = -3 \rightarrow 5$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.056$
$S = 1.02$
1900 reflections
92 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0225P)^2 + 0.3103P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0048 (5)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Br1	0.43969 (2)	0.63712 (8)	0.36884 (2)	0.0452 (1)
Br2	0.63382 (2)	0.13268 (8)	0.14100 (2)	0.0465 (1)
Cl1	0.21601 (6)	0.6350 (2)	0.01323 (5)	0.0528 (3)

N1	0.61671 (18)	0.3035 (7)	0.31614 (15)	0.0447 (8)
C1	0.5233 (2)	0.3766 (6)	0.24594 (17)	0.0310 (8)
C2	0.4327 (2)	0.5295 (7)	0.25508 (16)	0.0312 (8)
C3	0.3397 (2)	0.6082 (7)	0.18522 (17)	0.0333 (8)
C4	0.3335 (2)	0.5335 (7)	0.10201 (18)	0.0351 (8)
C5	0.4205 (2)	0.3896 (7)	0.08856 (17)	0.0355 (8)
C6	0.5129 (2)	0.3157 (6)	0.15999 (17)	0.0301 (8)
H1A	0.67145	0.21719	0.30798	0.0537*
H1B	0.62055	0.34404	0.36830	0.0537*
H3	0.28153	0.71053	0.19381	0.0399*
H5	0.41666	0.34358	0.03233	0.0426*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0471 (2)	0.0548 (2)	0.0351 (2)	−0.0064 (1)	0.0173 (1)	−0.0081 (1)
Br2	0.0434 (2)	0.0423 (2)	0.0610 (2)	0.0059 (1)	0.0281 (2)	−0.0024 (2)
Cl1	0.0439 (4)	0.0637 (6)	0.0373 (4)	0.0116 (4)	0.0002 (3)	0.0030 (4)
N1	0.0297 (12)	0.0628 (18)	0.0369 (13)	0.0035 (12)	0.0072 (10)	0.0046 (12)
C1	0.0284 (13)	0.0255 (14)	0.0371 (13)	−0.0050 (12)	0.0103 (11)	0.0035 (12)
C2	0.0352 (14)	0.0284 (15)	0.0312 (13)	−0.0071 (12)	0.0141 (12)	−0.0026 (11)
C3	0.0294 (13)	0.0317 (16)	0.0384 (14)	0.0017 (12)	0.0125 (12)	0.0000 (12)
C4	0.0332 (14)	0.0315 (16)	0.0333 (14)	0.0024 (12)	0.0043 (12)	0.0020 (12)
C5	0.0412 (15)	0.0334 (16)	0.0320 (13)	−0.0017 (13)	0.0141 (12)	−0.0033 (12)
C6	0.0299 (13)	0.0229 (15)	0.0391 (14)	0.0004 (11)	0.0150 (11)	0.0009 (12)

Geometric parameters (Å, º)

Br1—C2	1.897 (3)	C1—C2	1.407 (4)
Br2—C6	1.896 (3)	C2—C3	1.371 (4)
Cl1—C4	1.738 (3)	C3—C4	1.380 (4)
N1—C1	1.372 (4)	C4—C5	1.381 (4)
N1—H1B	0.8600	C5—C6	1.375 (4)
N1—H1A	0.8600	C3—H3	0.9300
C1—C6	1.396 (4)	C5—H5	0.9300
C1—N1—H1B	120.00	Cl1—C4—C3	119.2 (2)
H1A—N1—H1B	120.00	Cl1—C4—C5	119.9 (2)
C1—N1—H1A	120.00	C4—C5—C6	118.7 (2)
C2—C1—C6	115.2 (2)	Br2—C6—C1	118.2 (2)
N1—C1—C6	122.3 (3)	Br2—C6—C5	118.5 (2)
N1—C1—C2	122.5 (2)	C1—C6—C5	123.3 (3)
Br1—C2—C1	118.78 (19)	C2—C3—H3	120.00
Br1—C2—C3	118.3 (2)	C4—C3—H3	120.00
C1—C2—C3	122.9 (2)	C4—C5—H5	121.00
C2—C3—C4	119.1 (3)	C6—C5—H5	121.00
C3—C4—C5	120.8 (3)		
N1—C1—C2—Br1	−0.3 (4)	Br1—C2—C3—C4	179.6 (2)
N1—C1—C2—C3	179.0 (3)	C1—C2—C3—C4	0.4 (4)

C6—C1—C2—Br1	−178.01 (19)	C2—C3—C4—Cl1	−179.7 (2)
C6—C1—C2—C3	1.2 (4)	C2—C3—C4—C5	−1.7 (4)
N1—C1—C6—Br2	−1.1 (3)	Cl1—C4—C5—C6	179.2 (2)
N1—C1—C6—C5	−179.4 (3)	C3—C4—C5—C6	1.3 (4)
C2—C1—C6—Br2	176.68 (19)	C4—C5—C6—Br2	−177.9 (2)
C2—C1—C6—C5	−1.6 (4)	C4—C5—C6—C1	0.5 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Br2	0.86	2.64	3.067 (2)	112
N1—H1*A*···Br2^{i}	0.86	2.91	3.380 (3)	117
N1—H1*B*···Br1	0.86	2.67	3.099 (3)	112

Symmetry code: (i) −*x*+3/2, *y*+1/2, −*z*+1/2.