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2,6-Dibromo-4-methylaniline

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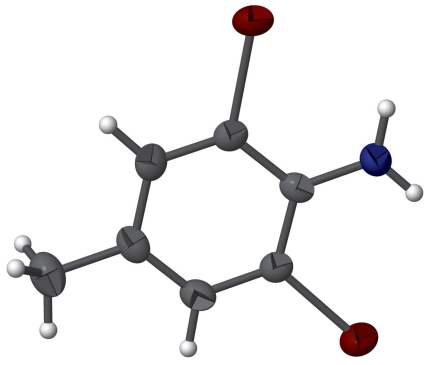
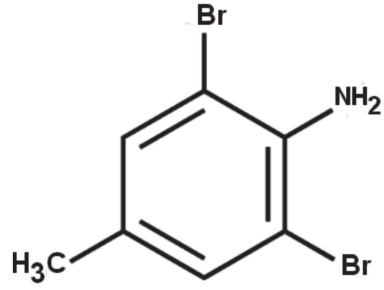
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_7H_7Br_2N$, the C—C—C bond angles of the benzene ring are notably distorted and two short intamolecular N—H···Br contacts occur. In the crystal, the molecules are linked by N—H···N hydrogen bonds to generate $C(2)$ chains propagating in the [100] direction.

3D view



Chemical scheme



Structure description

The solid-state structure of the title compound, $C_7H_7Br_2N$, was established by single-crystal X-ray diffraction analysis at 200 K and the molecular structure is illustrated in Fig. 1. The bromine atoms are slightly displaced from the mean plane of C1–C4/C6/C7 benzene ring, by 0.032 (1) and 0.065 (1) Å for Br1 and Br2, respectively. This can also be quantified by the C4—C3—C2—Br1 and C4—C6—C7—Br2 torsion angles, which are 179.7 (3) and −178.5 (3)°, respectively. The bond angles in the benzene ring are notably distorted from the ideal value of 120° with C7—C1—C2 = 115.1 (4), C1—C2—C3 = 122.8 (4) and C1—C7—C6 = 123.0 (4)°. The amine group lying between the bromine atoms results in two short intramolecular N—H···Br contacts (Table 1).

In the crystal, the molecules are linked by weak N1—H1*B*···N1 hydrogen bonds (Table 1) with N···N = 3.120 (7) Å to generate [100] $C(2)$ chains with adjacent molecules related by the 2_1 screw axis. A similar hydrogen bond was observed in diamino-mesithylene (Brihi *et al.*, 2016). The packing is illustrated in Fig. 2, which shows the topology of the chain is a zigzag, with an angle of inclination of the benzene ring to the *a* axis of 53.73 (14)°.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Br1	0.86	2.65	3.077 (4)	112
N1—H1B···Br2	0.86	2.64	3.072 (4)	113
N1—H1B···N1^{i}	0.86	2.38	3.120 (7)	144

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

Synthesis and crystallization

The title compound is commercially available (Lancaster Synthesis). It was purified by recrystallization from a solution of 80% ethanol and 20% distilled water. The colorless single crystals obtained are in the form of needles, which grow along the *a* axis.

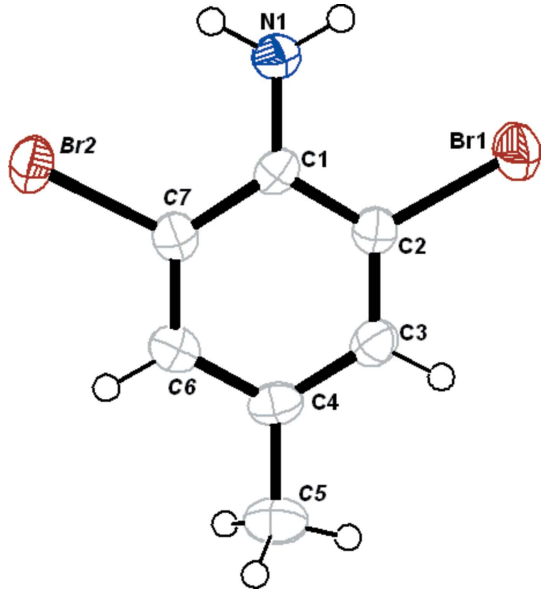


Figure 1
The molecular structure of the title compound showing displacement ellipsoids at the 50% probability level.

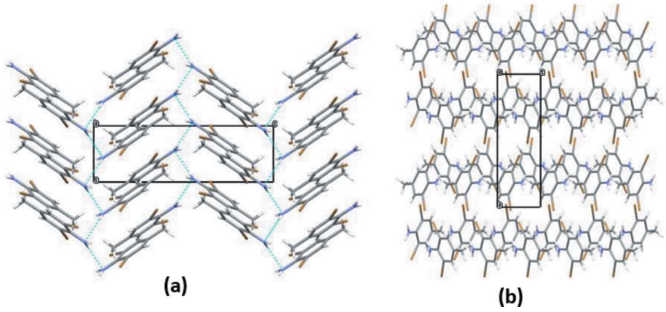


Figure 2
Views along the (*a*) *b* and (*b*) *c* axes of the crystal packing of the title compound with hydrogen bonds shown as dotted lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_7H_7Br_2N$
M_r	264.96
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	200
a, b, c (Å)	4.3773 (7), 13.585 (2), 14.057 (3)
V (Å^3)	835.9 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	9.62
Crystal size (mm)	0.12 × 0.05 × 0.04
Data collection	
Diffractometer	Bruker APEXII QUAZAR CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.396, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7550, 1715, 1422
R_{int}	0.061
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.626
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.072, 0.91
No. of reflections	1715
No. of parameters	92
H-atom treatment	H-atom parameters not refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.38
Absolute structure	Flack (1983)
Absolute structure parameter	0.02 (2)

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SIR92* (Altomare *et al.*, 1994), *SHELXL2013* (Sheldrick, 2015), *ORTEP* for Windows and *WinGX* publication routines (Farrugia, 2012).

Refinement

Crystal data, data collection and structure refinement details of the compound are summarized in Table 2.

Acknowledgements

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full crystallographic data

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Crystal data

$C_7H_7Br_2N$
M_r = 264.96
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 4.3773 (7) Å
b = 13.585 (2) Å
c = 14.057 (3) Å
V = 835.9 (2) Å^3
Z = 4
$F(000)$ = 504
D_x = 2.105 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7750 reflections
θ = 2.1–26.4°
μ = 9.62 mm^{-1}
T = 200 K
Needle, colorless
0.12 × 0.05 × 0.04 mm

Data collection

Bruker APEXII QUAZAR CCD diffractometer
Radiation source: ImuS
Graphite monochromator
Detector resolution: 8.02 pixels mm^{-1}
f\ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.396, T_{max} = 0.746
7550 measured reflections
1715 independent reflections
1422 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 26.4°, θ_{min} = 2.1°
h = −5→5
k = −15→16
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.072
S = 0.91
1715 reflections
92 parameters
0 restraints
0 constraints
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 not refined
$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack (1983)
Absolute structure parameter: 0.02 (2)

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 esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.69454 (12)	0.51851 (3)	0.40790 (4)	0.0449 (2)
Br2	0.49325 (11)	0.11063 (3)	0.35184 (3)	0.0374 (2)
N1	0.4343 (8)	0.3124 (3)	0.4488 (2)	0.0340 (14)
C1	0.6072 (9)	0.3165 (3)	0.3674 (3)	0.0255 (14)
C2	0.7481 (9)	0.4015 (3)	0.3360 (3)	0.0277 (14)
C3	0.9295 (10)	0.4045 (3)	0.2553 (3)	0.0323 (17)
C4	0.9781 (10)	0.3217 (3)	0.2004 (3)	0.0313 (14)
C5	1.1658 (11)	0.3259 (3)	0.1108 (3)	0.0447 (17)
C6	0.8409 (10)	0.2336 (3)	0.2315 (3)	0.0317 (14)
C7	0.6636 (10)	0.2322 (3)	0.3118 (3)	0.0280 (12)
H1	1.33755	0.28238	0.11654	0.0669*
H1A	0.40957	0.36443	0.48284	0.0407*
H1B	0.35064	0.25790	0.46576	0.0407*
H2	1.02080	0.46361	0.23780	0.0388*
H3	0.87095	0.17585	0.19722	0.0378*
H4	1.23699	0.39194	0.10090	0.0669*
H5	1.04240	0.30603	0.05765	0.0669*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0576 (3)	0.0274 (2)	0.0497 (3)	−0.0005 (2)	0.0057 (3)	−0.0016 (2)
Br2	0.0373 (3)	0.0279 (2)	0.0471 (3)	−0.0053 (2)	−0.0027 (3)	0.0002 (2)
N1	0.038 (3)	0.031 (2)	0.033 (2)	−0.0013 (18)	0.0070 (18)	0.0005 (17)
C1	0.0174 (19)	0.028 (2)	0.031 (3)	0.0015 (17)	−0.0052 (19)	0.007 (2)
C2	0.023 (2)	0.027 (2)	0.033 (3)	0.0023 (18)	−0.0039 (19)	0.0002 (19)
C3	0.027 (3)	0.029 (3)	0.041 (3)	0.0011 (19)	0.001 (2)	0.006 (2)
C4	0.021 (2)	0.041 (3)	0.032 (2)	0.005 (2)	0.000 (2)	0.007 (2)
C5	0.036 (3)	0.057 (3)	0.041 (3)	0.009 (3)	0.005 (3)	0.008 (3)
C6	0.030 (2)	0.038 (3)	0.027 (2)	0.004 (2)	−0.004 (2)	−0.002 (2)
C7	0.024 (2)	0.028 (2)	0.032 (2)	0.001 (2)	−0.006 (2)	−0.0010 (19)

Geometric parameters (Å, º)

Br1—C2	1.898 (4)	C4—C5	1.505 (6)
Br2—C7	1.898 (4)	C4—C6	1.409 (6)

N1—C1	1.373 (5)	C6—C7	1.370 (6)
N1—H1A	0.8600	C3—H2	0.9300
N1—H1B	0.8600	C5—H1	0.9600
C1—C7	1.408 (6)	C5—H4	0.9600
C1—C2	1.382 (6)	C5—H5	0.9600
C2—C3	1.385 (6)	C6—H3	0.9300
C3—C4	1.381 (6)		

C1—N1—H1B	120.00	Br2—C7—C1	118.3 (3)
H1A—N1—H1B	120.00	Br2—C7—C6	118.6 (3)
C1—N1—H1A	120.00	C1—C7—C6	123.0 (4)
C2—C1—C7	115.1 (4)	C2—C3—H2	119.00
N1—C1—C7	121.8 (4)	C4—C3—H2	119.00
N1—C1—C2	123.1 (4)	C4—C5—H1	110.00
Br1—C2—C1	118.3 (3)	C4—C5—H4	110.00
Br1—C2—C3	118.8 (3)	C4—C5—H5	109.00
C1—C2—C3	122.8 (4)	H1—C5—H4	109.00
C2—C3—C4	121.5 (4)	H1—C5—H5	109.00
C5—C4—C6	121.7 (4)	H4—C5—H5	109.00
C3—C4—C5	121.4 (4)	C4—C6—H3	120.00
C3—C4—C6	116.9 (4)	C7—C6—H3	120.00
C4—C6—C7	120.6 (4)		

N1—C1—C2—Br1	−1.0 (5)	Br1—C2—C3—C4	179.7 (3)
N1—C1—C2—C3	178.1 (4)	C1—C2—C3—C4	0.6 (7)
C7—C1—C2—Br1	−178.5 (3)	C2—C3—C4—C5	177.7 (4)
C7—C1—C2—C3	0.6 (6)	C2—C3—C4—C6	−1.5 (6)
N1—C1—C7—Br2	0.1 (6)	C3—C4—C6—C7	1.2 (6)
N1—C1—C7—C6	−178.5 (4)	C5—C4—C6—C7	−178.1 (4)
C2—C1—C7—Br2	177.6 (3)	C4—C6—C7—Br2	−178.5 (3)
C2—C1—C7—C6	−1.0 (6)	C4—C6—C7—C1	0.1 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Br1	0.86	2.65	3.077 (4)	112
N1—H1*B*···Br2	0.86	2.64	3.072 (4)	113
N1—H1*B*···N1^{i}	0.86	2.38	3.120 (7)	144

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