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ISSN 2414-3146

Diaminomesitylene

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Received 24 January 2016
Accepted 29 February 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

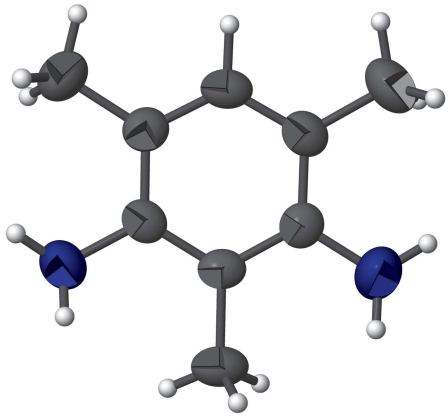
Keywords: crystal structure; N—H···N hydrogen bonds; C—H···π interactions.

CCDC reference: 1456540

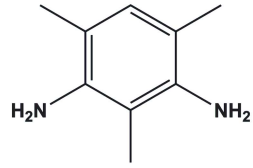
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_9H_{14}N_2$ (systematic name: 2,4,6-trimethylbenzene-1,3-diamine), is almost planar (r.m.s. deviation = 0.025 Å). In the crystal, molecules are linked *via* N—H···N hydrogen bonds, forming zigzag chains along the *b*-axis direction. Only one of the four N-bonded H atoms forms a hydrogen bond, perhaps due to steric crowding. The chains are linked by C—H···π interactions, forming sheets lying parallel to the *bc* plane

3D view



Chemical scheme



Structure description

Aromatic amines are a class of chemicals found in the plastic and chemical industries as byproducts of the manufacture of compounds such as polyurethane foams, dyes, pesticides, pharmaceuticals and semiconductors. They are also found in environmental pollution from diesel exhausts, the combustion of wood chips and rubber, tobacco smoke and substances in grilled meats and fish (DeBruin *et al.*, 1999; DeBruin & Josephy (2002).

The structure of dibromomesitylene (DBM) was resolved by neutron diffraction at 120 and 14 K. It crystallizes in the space group *P*21/*n* (Hernandez *et al.*, 2003). As part of our project which aims to study new substituted mesitylene or 1,3,5-trimethylbenzene compounds, for example 1,3,5-trimethyl-2,4-dinitrobenzene (Brihi *et al.*, 2015), we report herein on the synthesis and crystal structure of the title compound.

The molecular structure of the title compound, also know as diaminomesitylene (DAM), is illustrated in the Fig. 1. The non-H atoms are almost coplanar, r.m.s. deviation = 0.025 Å, with a maximum deviation of 0.044 (2) Å for atom C11, which lies between the amine groups. The crystal packing is illustrated in Fig. 2, which shows the zigzag N—H···N hydrogen-bonded chains along [010], which are linked *via* C—H···π interactions forming sheets parallel to the *bc* plane (Table 1).

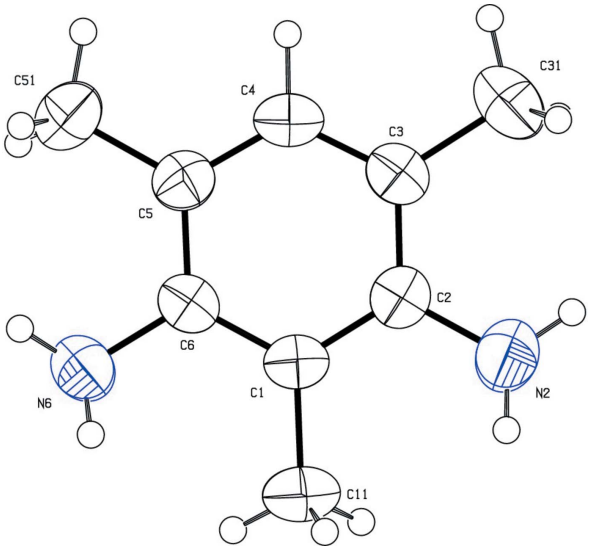


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

In a round-bottom flask were placed 1 mmol (210 mg) of 2,4-dinitromesitylene and 1.52 mmol (180 mg) of granulated tin. 10 ml of HCl was added in three equal parts to the mixture that was kept cool for 20–30 min. NaOH was added to the mixture until there was no further precipitation of tin hydroxide. The resulting amine was extracted with ether that was then evacuated by distillation. The title compound was obtained as colourless crystals on recrystallization from ethanol solution.

Refinement

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

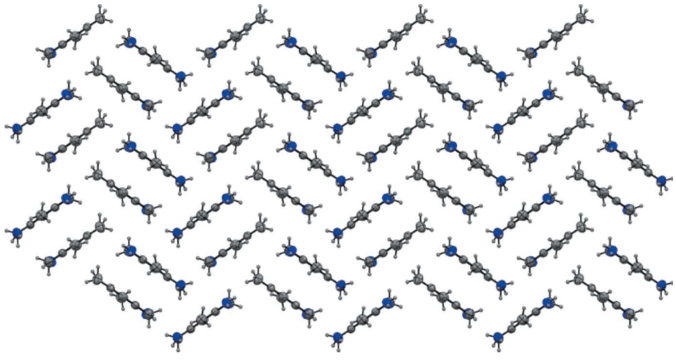


Figure 2
The crystal packing of the title compound viewed along the *a* axis.

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

Cg is the centroid of the C1–C6 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H22···N6^{i}	0.89	2.37	3.170 (3)	150
N6—H61···*Cg*^{ii}	0.90	2.62	3.355 (2)	140
C11—H112···*Cg*^{iii}	0.92	2.82	3.665 (2)	152

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_{14}N_2$
M_r	150.22
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, *b*, *c* (Å)	8.1735 (7), 12.9313 (9), 8.7300 (8)
β (°)	105.803 (9)
V (Å^3)	887.83 (13)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.07
Crystal size (mm)	0.10 × 0.08 × 0.07
Data collection	
Diffractometer	Oxford Diffraction Xcalibur
No. of measured, independent and observed [$I > 3.0\sigma(I)$] reflections	3794, 1953, 1153
R_{int}	0.017
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.049, 0.103, 0.88
No. of reflections	968
No. of parameters	101
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.13, −0.12

Computer programs: *XCALIBUR* (Oxford Diffraction, 2002), *CrysAlis PRO* (Agilent, 2004), *SIR2002* (Burla *et al.*, 2005), *CRYSTALS* (Betteridge *et al.*, 2003), *CAMERON* (Watkin *et al.*, 1996).

Acknowledgements

This work was supported by the Laboratoire de Cristallographie Département de Physique, Université Fréres Mentouri-Constantine, Algeria. We would also like to thank Mr F. Saidi, Engineer at the Université Mentouri-Constantine, for assistance during the data collection on the Xcalibur X-ray diffractometer.

References

Agilent (2004). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Brihi, O., Hamdouni, N., Boudjada, A. & Meinnel, J. (2015). *Acta Cryst.* E**71**, o670–o671.
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
DeBruin, L. & Josephy, P. (2002). *Environ. Health Perspect.* **110**, 119–128.

DeBruin, L., Pawliszyn, J. & Josephy, P. (1999). *Chem. Res. Toxicol.* **12**, 78–82.
Hernandez, O., Cousson, A., Plazanet, M., Nierlich, M. & Meinnel, J. (2003). *Acta Cryst.* C**59**, o445–o450.
Oxford Diffraction (2002). *XCALIBUR*. Oxford Diffraction Ltd, Abingdon, England.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.

full crystallographic data

IUCrData (2016). **1**, x160351 [doi:10.1107/S2414314616003515]

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2,4,6-Trimethylbenzene-1,3-diamine

Crystal data

$C_9H_{14}N_2$
M_r = 150.22
Monoclinic, $P2_1/c$
a = 8.1735 (7) Å
b = 12.9313 (9) Å
c = 8.7300 (8) Å
β = 105.803 (9)°
V = 887.83 (13) Å^3
Z = 4
$F(000)$ = 328
D_x = 1.124 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2457 reflections
θ = 4.0–27.9°
μ = 0.07 mm^{-1}
T = 293 K
Needle, colourless
0.10 × 0.08 × 0.07 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Graphite monochromator
$\omega/2\theta$ scans
3794 measured reflections
1953 independent reflections
1153 reflections with $I > 3.0\sigma(I)$
R_{int} = 0.017
θ_{max} = 28.7°, θ_{min} = 3.4°
h = −10→11
k = −16→15
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.103
S = 0.88
968 reflections
101 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
Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed belo*w* and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 0.191E + 04 0.570E + 04 0.179E + 04 0.189E + 04
$(\Delta/\sigma)_{max}$ = 0.0002
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N2	−0.0520 (3)	−0.04877 (17)	0.7012 (2)	0.0722
N6	0.0592 (3)	0.25972 (15)	1.0272 (2)	0.0605
C1	0.0030 (2)	0.10506 (16)	0.8621 (2)	0.0432
C2	0.0612 (3)	0.02612 (16)	0.7813 (2)	0.0461
C3	0.2327 (3)	0.02171 (16)	0.7811 (2)	0.0492
C4	0.3405 (2)	0.09757 (18)	0.8639 (3)	0.0516
C5	0.2888 (3)	0.17661 (17)	0.9470 (2)	0.0490
C6	0.1174 (2)	0.17840 (16)	0.9474 (2)	0.0443
C11	−0.1830 (3)	0.1110 (2)	0.8552 (3)	0.0628
C31	0.2977 (4)	−0.0630 (2)	0.6941 (3)	0.0751
C51	0.4127 (3)	0.2583 (2)	1.0309 (3)	0.0725
H41	0.4600	0.0935	0.8640	0.0617*
H111	−0.2178	0.1723	0.8870	0.0980*
H112	−0.2227	0.0616	0.9125	0.0981*
H113	−0.2567	0.0986	0.7456	0.0980*
H311	0.4211	−0.0544	0.7091	0.1203*
H312	0.2793	−0.1332	0.7340	0.1205*
H313	0.2428	−0.0622	0.5781	0.1206*
H511	0.3719	0.3262	1.0041	0.1103*
H512	0.4348	0.2551	1.1498	0.1097*
H513	0.5238	0.2444	1.0147	0.1102*
H21	−0.1497	−0.0530	0.7222	0.0861*
H22	−0.0121	−0.0969	0.6483	0.0861*
H61	0.1413	0.2892	1.1051	0.0743*
H62	−0.0261	0.2420	1.0560	0.0743*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0724 (14)	0.0698 (14)	0.0755 (14)	−0.0162 (11)	0.0223 (11)	−0.0161 (11)
N6	0.0629 (12)	0.0598 (12)	0.0616 (11)	0.0055 (9)	0.0215 (10)	−0.0052 (9)
C1	0.0396 (10)	0.0477 (12)	0.0420 (10)	0.0041 (9)	0.0106 (9)	0.0138 (9)
C2	0.0513 (11)	0.0441 (11)	0.0413 (10)	−0.0009 (10)	0.0102 (9)	0.0076 (10)
C3	0.0545 (12)	0.0477 (12)	0.0475 (11)	0.0072 (11)	0.0171 (10)	0.0068 (10)
C4	0.0399 (10)	0.0604 (14)	0.0557 (13)	0.0049 (10)	0.0150 (9)	0.0093 (11)
C5	0.0467 (11)	0.0495 (12)	0.0481 (12)	0.0017 (10)	0.0087 (9)	0.0056 (10)
C6	0.0490 (11)	0.0440 (12)	0.0410 (10)	0.0079 (9)	0.0140 (9)	0.0086 (9)
C11	0.0424 (11)	0.0740 (16)	0.0732 (16)	0.0037 (11)	0.0177 (11)	0.0107 (13)
C31	0.0810 (18)	0.0716 (17)	0.0778 (19)	0.0186 (14)	0.0302 (15)	−0.0083 (14)

C51	0.0580 (14)	0.0710 (16)	0.0834 (18)	−0.0109 (13)	0.0104 (13)	−0.0121 (14)

Geometric parameters (Å, º)

N2—C2	1.390 (3)	C4—H41	0.978
N2—H21	0.868	C5—C6	1.401 (3)
N2—H22	0.888	C5—C51	1.508 (3)
N6—C6	1.414 (3)	C11—H111	0.911
N6—H61	0.899	C11—H112	0.924
N6—H62	0.835	C11—H113	0.996
C1—C2	1.396 (3)	C31—H311	0.987
C1—C6	1.396 (3)	C31—H312	0.998
C1—C11	1.507 (3)	C31—H313	0.989
C2—C3	1.403 (3)	C51—H511	0.947
C3—C4	1.384 (3)	C51—H512	1.005
C3—C31	1.510 (3)	C51—H513	0.973
C4—C5	1.384 (3)		
C2—N2—H21	117.6	N6—C6—C5	119.0 (2)
C2—N2—H22	117.5	N6—C6—C1	120.11 (18)
H21—N2—H22	123.7	C5—C6—C1	120.81 (19)
C6—N6—H61	113.9	C1—C11—H111	115.4
C6—N6—H62	111.4	C1—C11—H112	116.3
H61—N6—H62	113.6	H111—C11—H112	104.6
C2—C1—C6	119.69 (17)	C1—C11—H113	111.7
C2—C1—C11	119.8 (2)	H111—C11—H113	106.0
C6—C1—C11	120.6 (2)	H112—C11—H113	101.5
C1—C2—N2	119.47 (18)	C3—C31—H311	109.8
C1—C2—C3	120.54 (19)	C3—C31—H312	112.1
N2—C2—C3	120.0 (2)	H311—C31—H312	107.6
C2—C3—C4	117.71 (19)	C3—C31—H313	112.5
C2—C3—C31	121.2 (2)	H311—C31—H313	107.3
C4—C3—C31	121.1 (2)	H312—C31—H313	107.3
C3—C4—C5	123.71 (19)	C5—C51—H511	112.6
C3—C4—H41	117.1	C5—C51—H512	112.4
C5—C4—H41	119.1	H511—C51—H512	104.2
C4—C5—C6	117.5 (2)	C5—C51—H513	109.3
C4—C5—C51	120.7 (2)	H511—C51—H513	114.6
C6—C5—C51	121.8 (2)	H512—C51—H513	103.4

Hydrogen-bond geometry (Å, º)

Cg is the centroid of benzene ring C1–C6.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H22···N6^{i}	0.89	2.37	3.170 (3)	150

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