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2-[(Dimethylamino)methylidene]-propanedinitrile

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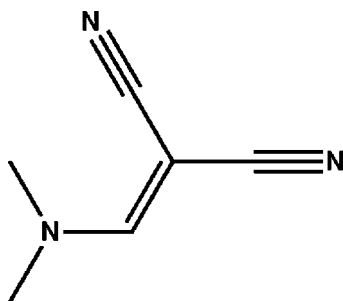
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.066; wR factor = 0.206; data-to-parameter ratio = 15.7.

In the title molecule, $\text{C}_6\text{H}_7\text{N}_3$, the mean plane of the dimethylamino group [maximum deviation = 0.006 (2) Å] forms a dihedral angle of 7.95 (18)° with the mean plane of the propanedinitrile fragment [maximum deviation = 0.008 (2) Å]. In the crystal, weak $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules into a three-dimensional network.

Related literature

For applications of enamines, see: Omran *et al.* (1997); Saleh *et al.* (1999). For related structures, see: Kant *et al.* (2012); Karlsen *et al.* (2002).



Experimental

Crystal data

$\text{C}_6\text{H}_7\text{N}_3$
 $M_r = 121.15$

Monoclinic, $P2_1/c$
 $a = 4.0368$ (3) Å

$b = 15.5642$ (10) Å
 $c = 10.8500$ (7) Å
 $\beta = 97.488$ (6)°
 $V = 675.89$ (8) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 293$ K
 $0.3 \times 0.2 \times 0.2$ mm

Data collection

Oxford Diffraction Xcalibur
 Sapphire3 diffractometer
 Absorption correction: multi-scan
 (*CrysAlis PRO*; Oxford Diffraction, 2010)
 $T_{\min} = 0.637$, $T_{\max} = 1.000$

15029 measured reflections
 1320 independent reflections
 875 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.067$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.206$
 $S = 1.05$
 1320 reflections

84 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C2}-\text{H2}\cdots\text{N8}^i$	0.93	2.51	3.399 (4)	161
$\text{C4}-\text{H4B}\cdots\text{N9}^{ii}$	0.96	2.62	3.569 (4)	170

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

RK acknowledges the Department of Science & Technology for access to the single-crystal X-ray diffractometer sanctioned as a National Facility under Project No. SR/S2/CMP-47/2003.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5587).

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supporting information

Acta Cryst. (2013). E69, o433 [doi:10.1107/S1600536813004960]

2-[(Dimethylamino)methylidene]propanedinitrile

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S1. Comment

[(Dimethylamino)methylidene]propanedinitrile (I) is a potentially versatile substance which can be used for the synthesis of number of heterocyclic compounds and drug intermediates (Omran *et al.*, 1997; Saleh *et al.*, 1999).

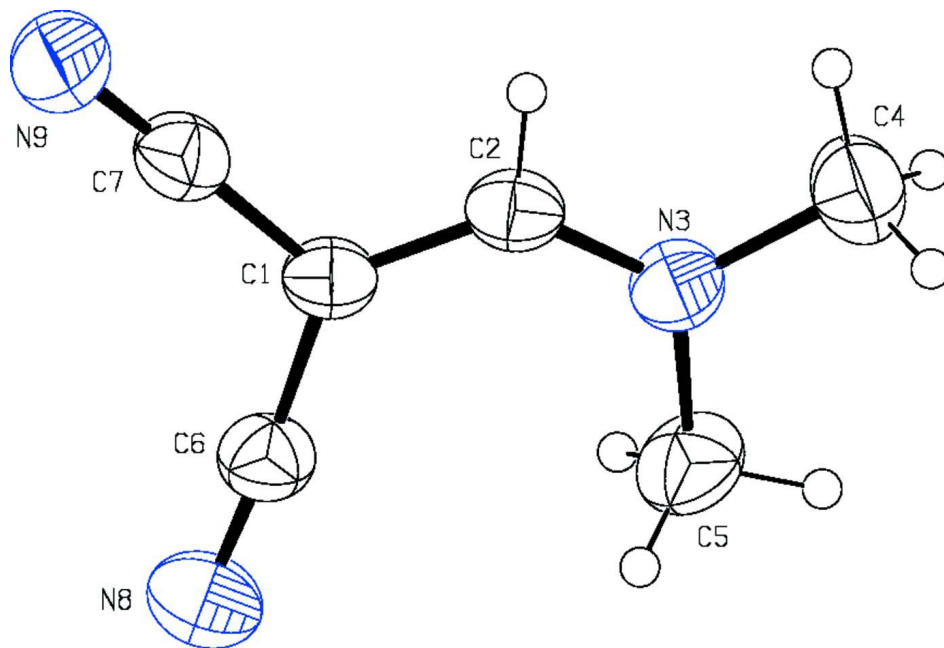
In (I)(Fig.1), all bond lengths and angles are normal and correspond to those observed in related structures (Kant *et al.*, 2012; Karlsen *et al.*, 2002). The dihedral angle between dimethylamino group (N3/C2/C4/C5 with a maximum deviation of 0.006 (2)Å for N3) and propanedinitrile fragment (C1/C6/C7/N8/N9 with a maximum deviation of 0.008 (2)Å for C6) is 7.95 (18)°. In the crystal, weak C2—H2···N8ⁱ and C4—H4B···N9ⁱⁱ hydrogen bonds link molecules to form a three-dimensional supramolecular structure (Fig. 2, Table 1.).

S2. Experimental

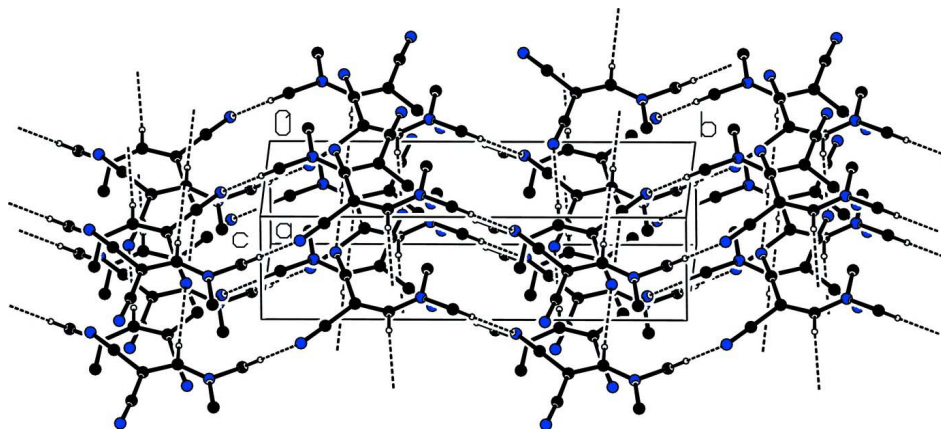
In a 50 ml round bottomed flask charged with 3 mmol of malononitrile and 3 mmol of dimethyl formamide dimethyl-acetal was stirred for 2 - 3hrs at room temp. The reaction was monitored by TLC. After completion of the reaction, a precipitate was formed. Finally, the product was filtered and washed with pet ether. Yield: 75%, m.p. 361–363 K. Diffraction quality single crystals were grown by slow evaporation of an ethanol solution of the title compound at room temperature

S3. Refinement

All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

**Figure 1**

The molecular structure of (I) with displacement ellipsoids drawn at the 40% probability level. H atoms are shown as small spheres of arbitrary radii.

**Figure 2**

Part of the crystal structure with dashed lines showing weak intermolecular C—H...N hydrogen bonds.

2-[(Dimethylamino)methylidene]propanedinitrile

Crystal data

$C_6H_7N_3$

$M_r = 121.15$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2_1/c$

$a = 4.0368 (3) \text{ \AA}$

$b = 15.5642 (10) \text{ \AA}$

$c = 10.8500 (7) \text{ \AA}$

$\beta = 97.488 (6)^\circ$

$V = 675.89 (8) \text{ \AA}^3$

$Z = 4$

$F(000) = 256$

$D_x = 1.191 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3950 reflections

$\theta = 3.8\text{--}29.2^\circ$

$\mu = 0.08 \text{ mm}^{-1}$

$T = 293$ K $0.3 \times 0.2 \times 0.2$ mm
 Block, colourless

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer	15029 measured reflections
Radiation source: fine-focus sealed tube	1320 independent reflections
Graphite monochromator	875 reflections with $I > 2\sigma(I)$
Detector resolution: 16.1049 pixels mm ⁻¹	$R_{\text{int}} = 0.067$
ω scans	$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 3.8^\circ$
Absorption correction: multi-scan	$h = -4 \rightarrow 4$
(<i>CrysAlis PRO</i> ; Oxford Diffraction, 2010)	$k = -19 \rightarrow 19$
$T_{\text{min}} = 0.637$, $T_{\text{max}} = 1.000$	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.066$	H-atom parameters constrained
$wR(F^2) = 0.206$	$w = 1/[\sigma^2(F_o^2) + (0.1075P)^2 + 0.0919P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
1320 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
84 parameters	$\Delta\rho_{\text{max}} = 0.23 \text{ e } \text{Å}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.16 \text{ e } \text{Å}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. CrysAlisPro, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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

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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3330 (6)	0.77364 (16)	0.7562 (2)	0.0501 (7)
C2	0.2937 (6)	0.69295 (15)	0.7036 (2)	0.0510 (7)
H2	0.1698	0.6914	0.6251	0.061*
N3	0.4033 (5)	0.61815 (13)	0.74689 (19)	0.0565 (6)
C4	0.3449 (7)	0.54179 (18)	0.6690 (3)	0.0752 (9)
H4A	0.2019	0.5562	0.5941	0.113*
H4B	0.2401	0.4983	0.7132	0.113*
H4C	0.5542	0.5206	0.6483	0.113*
C5	0.5891 (7)	0.6056 (2)	0.8695 (3)	0.0726 (9)
H5A	0.7939	0.6377	0.8759	0.109*
H5B	0.6386	0.5457	0.8821	0.109*

H5C	0.4577	0.6253	0.9316	0.109*
C6	0.5281 (6)	0.79802 (16)	0.8689 (2)	0.0568 (7)
C7	0.1665 (6)	0.84244 (17)	0.6873 (2)	0.0578 (7)
N8	0.6838 (6)	0.82284 (18)	0.9572 (2)	0.0805 (8)
N9	0.0355 (6)	0.89864 (16)	0.6341 (2)	0.0789 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0514 (13)	0.0581 (15)	0.0392 (13)	-0.0003 (11)	0.0003 (10)	0.0009 (11)
C2	0.0508 (13)	0.0622 (17)	0.0391 (13)	-0.0035 (11)	0.0017 (10)	0.0040 (11)
N3	0.0647 (13)	0.0565 (13)	0.0466 (13)	0.0020 (10)	0.0009 (10)	0.0064 (10)
C4	0.090 (2)	0.0557 (17)	0.077 (2)	-0.0048 (14)	0.0003 (16)	-0.0040 (15)
C5	0.0853 (19)	0.0757 (19)	0.0537 (18)	0.0151 (15)	-0.0032 (14)	0.0126 (15)
C6	0.0573 (15)	0.0640 (17)	0.0477 (15)	0.0020 (12)	0.0016 (12)	-0.0023 (13)
C7	0.0643 (16)	0.0599 (16)	0.0469 (16)	-0.0022 (13)	-0.0018 (12)	-0.0053 (13)
N8	0.0883 (17)	0.0896 (19)	0.0576 (16)	-0.0012 (14)	-0.0125 (13)	-0.0126 (14)
N9	0.0944 (18)	0.0635 (15)	0.0717 (18)	0.0074 (13)	-0.0155 (14)	0.0030 (14)

Geometric parameters (Å, °)

C1—C2	1.380 (3)	C4—H4B	0.9600
C1—C6	1.417 (3)	C4—H4C	0.9600
C1—C7	1.424 (3)	C5—H5A	0.9600
C2—N3	1.311 (3)	C5—H5B	0.9600
C2—H2	0.9300	C5—H5C	0.9600
N3—C5	1.453 (3)	C6—N8	1.143 (3)
N3—C4	1.460 (3)	C7—N9	1.139 (3)
C4—H4A	0.9600		
C2—C1—C6	128.4 (2)	N3—C4—H4C	109.5
C2—C1—C7	116.5 (2)	H4A—C4—H4C	109.5
C6—C1—C7	115.0 (2)	H4B—C4—H4C	109.5
N3—C2—C1	130.2 (2)	N3—C5—H5A	109.5
N3—C2—H2	114.9	N3—C5—H5B	109.5
C1—C2—H2	114.9	H5A—C5—H5B	109.5
C2—N3—C5	123.9 (2)	N3—C5—H5C	109.5
C2—N3—C4	119.6 (2)	H5A—C5—H5C	109.5
C5—N3—C4	116.5 (2)	H5B—C5—H5C	109.5
N3—C4—H4A	109.5	N8—C6—C1	175.8 (3)
N3—C4—H4B	109.5	N9—C7—C1	178.6 (3)
H4A—C4—H4B	109.5		
C6—C1—C2—N3	5.6 (4)	C1—C2—N3—C5	2.7 (4)
C7—C1—C2—N3	-176.8 (2)	C1—C2—N3—C4	-176.2 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C2—H2···N8 ⁱ	0.93	2.51	3.399 (4)	161
C4—H4B···N9 ⁱⁱ	0.96	2.62	3.569 (4)	170

Symmetry codes: (i) $x-1, -y+3/2, z-1/2$; (ii) $-x, y-1/2, -z+3/2$.