organic compounds

2,2'-(Piperazine-1,4-diyl)diacetonitrile

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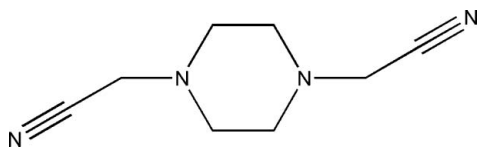
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The complete molecule of the title compound, $C_8H_{12}N_4$, is generated by a crystallographic inversion centre. The piperazine ring adopts a chair conformation with the N-bonded substituents in equatorial positions. In the crystal, molecules are linked by $C-H{\cdots}N_c$ (c = cyanide) hydrogen bonds.

Related literature

For related structures, see: Ma *et al.* (2007); Liu & Liu (2011); Luo & Weng (2011).

Experimental

Crystal data

$C_8H_{12}N_4$	b = 6.6731 (13) Å
M_r = 164.22	c = 11.077 (2) Å
Monoclinic, $P2_1/c$	β = 95.61 (3)°
a = 6.3452 (13) Å	V = 466.78 (16) Å^3

Z = 2	T = 293 K
Mo $K\alpha$ radiation	$0.20 \times 0.18 \times 0.10$ mm
μ = 0.08 mm^{-1}	

Data collection

Rigaku Saturn CCD diffractometer	3739 measured reflections
Absorption correction: multi-scan	1076 independent reflections
(*CrystalClear*; Rigaku/MSC, 2005)	835 reflections with $I > 2\sigma(I)$
T_{min} = 0.985, T_{max} = 0.992	R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046	55 parameters
$wR(F^2)$ = 0.150	H-atom parameters constrained
S = 1.05	$\Delta\rho_{max}$ = 0.10 e Å^{-3}
1076 reflections	$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

$D-H{\cdots}A$	$D-H$	$H{\cdots}A$	$D{\cdots}A$	$D-H{\cdots}A$
C3—H3$A{\cdots}$N2^i	0.97	2.57	3.427 (2)	147

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

References

Liu, X.-F. & Liu, X.-H. (2011). *Acta Cryst.* E**67**, o202.
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Ma, H.-F., Jia, H.-S., Qian, Y., Wen, F. & Chen, B.-L. (2007). *Acta Cryst.* E**63**, o311–o312.
Rigaku/MSC (2005). *CrystalClear*. Rigaku/MSC Inc. The Woodlands, Texas, USA.
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supplementary materials

2,2′-(Piperazine-1,4-diyl)diacetonitrile

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Experimental

Piperazine (25 mmol) and triethylamine (50 mmol), dissolved in 20 ml 95% of ethanol, was added dropwise to the stirred solution of chloroacetonitrile (50 mmol) at reflux. The mixture was stirred for 8 h at reflux. The mixture was stirred overnight at room temperature, evaporated in vacuum and the residue was purified by recrystallization from ethanol to give the title compound. Colourless blocks were grown from dichloromethane/ethanol solution.

Refinement

All the H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

Computing details

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear* (Rigaku/MSC, 2005); data reduction: *CrystalClear* (Rigaku/MSC, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).
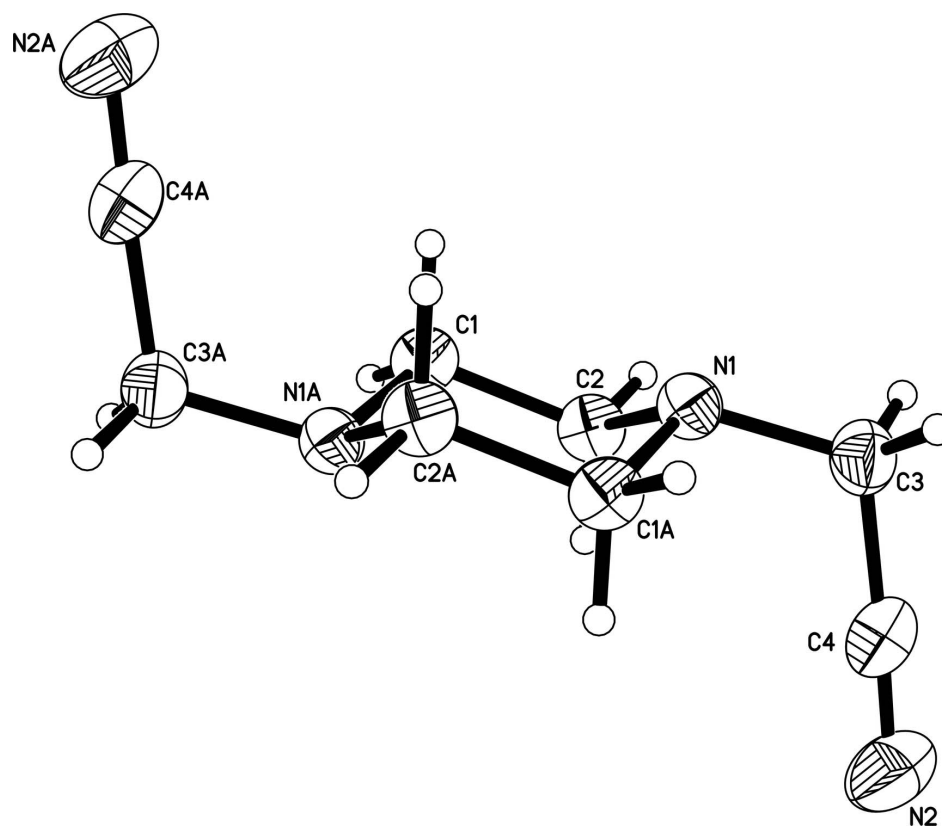
Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.
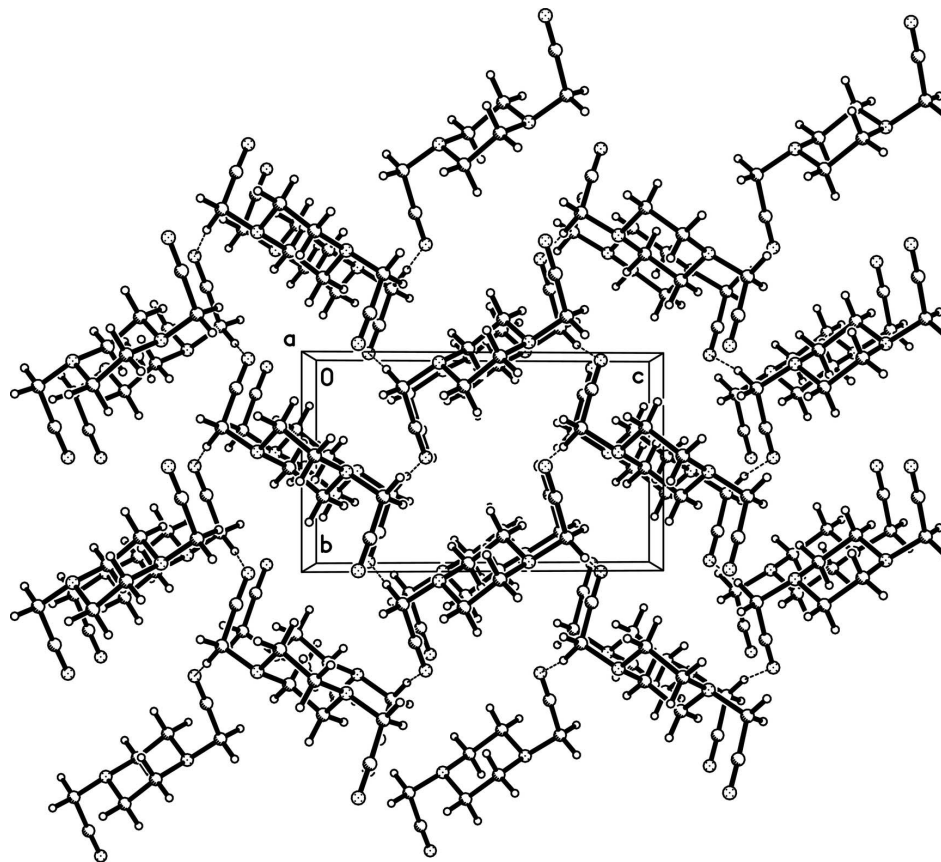
Figure 2

The crystal packing for (I).

2,2′-(Piperazine-1,4-diyl)diacetonitrile

Crystal data

$C_8H_{12}N_4$	$F(000) = 176$
$M_r = 164.22$	$D_x = 1.168$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.3452$ (13) Å	Cell parameters from 1156 reflections
$b = 6.6731$ (13) Å	$\theta = 3.1–27.8°$
$c = 11.077$ (2) Å	$\mu = 0.08$ mm^{-1}
$\beta = 95.61$ (3)°	$T = 293$ K
$V = 466.78$ (16) Å^3	Block, colorless
$Z = 2$	$0.20 \times 0.18 \times 0.10$ mm

Data collection

Rigaku Saturn CCD diffractometer	3739 measured reflections
Radiation source: rotating anode	1076 independent reflections
Confocal monochromator	835 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.032$
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)	$\theta_{max} = 27.9°$, $\theta_{min} = 6.4°$
	$h = -8 \rightarrow 5$
	$k = -8 \rightarrow 8$
$T_{min} = 0.985$, $T_{max} = 0.992$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.046$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.150$	
$S = 1.05$	H-atom parameters constrained
1076 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0937P)^2 + 0.0176P]$ where $P = (F_o^2 + 2F_c^2)/3$
55 parameters	
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.10$ e Å^{-3}
	$\Delta\rho_{min} = -0.15$ 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.

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}
N1	0.06045 (14)	0.54542 (14)	0.12377 (8)	0.0464 (4)
N2	0.3536 (2)	0.9796 (2)	0.16532 (15)	0.0904 (6)
C1	−0.21559 (17)	0.52728 (19)	−0.04400 (11)	0.0497 (4)
H1A	−0.2823	0.4143	−0.0078	0.060*
H1B	−0.3249	0.6036	−0.0910	0.060*
C2	−0.10710 (17)	0.65762 (18)	0.05439 (11)	0.0499 (4)
H2A	−0.0474	0.7746	0.0184	0.060*
H2B	−0.2095	0.7030	0.1079	0.060*
C3	0.1569 (2)	0.6568 (2)	0.22683 (12)	0.0584 (4)
H3A	0.2562	0.5701	0.2741	0.070*
H3B	0.0476	0.6945	0.2779	0.070*
C4	0.2700 (2)	0.8402 (2)	0.19376 (13)	0.0647 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0471 (6)	0.0463 (6)	0.0461 (5)	0.0023 (4)	0.0062 (4)	−0.0010 (4)
N2	0.0816 (10)	0.0743 (10)	0.1129 (13)	−0.0215 (7)	−0.0029 (9)	−0.0028 (8)
C1	0.0410 (6)	0.0516 (7)	0.0570 (7)	0.0054 (4)	0.0062 (5)	−0.0018 (5)
C2	0.0465 (6)	0.0479 (7)	0.0561 (7)	0.0087 (4)	0.0092 (5)	−0.0032 (5)
C3	0.0657 (8)	0.0575 (8)	0.0512 (7)	−0.0009 (6)	0.0013 (5)	−0.0049 (5)
C4	0.0592 (8)	0.0618 (9)	0.0707 (9)	−0.0035 (6)	−0.0057 (6)	−0.0112 (7)

Geometric parameters (Å, º)

N1—C3	1.4471 (16)	C1—H1B	0.9700
N1—C2	1.4567 (15)	C2—H2A	0.9700
N1—C1ⁱ	1.4680 (14)	C2—H2B	0.9700
N2—C4	1.1305 (19)	C3—C4	1.4824 (19)
C1—N1ⁱ	1.4681 (14)	C3—H3A	0.9700
C1—C2	1.5071 (17)	C3—H3B	0.9700
C1—H1A	0.9700		
C3—N1—C2	112.51 (10)	C1—C2—H2A	109.6
C3—N1—C1ⁱ	112.82 (9)	N1—C2—H2B	109.6
C2—N1—C1ⁱ	110.49 (9)	C1—C2—H2B	109.6
N1ⁱ—C1—C2	109.89 (9)	H2A—C2—H2B	108.2
N1ⁱ—C1—H1A	109.7	N1—C3—C4	114.00 (10)
C2—C1—H1A	109.7	N1—C3—H3A	108.8
N1ⁱ—C1—H1B	109.7	C4—C3—H3A	108.8
C2—C1—H1B	109.7	N1—C3—H3B	108.8
H1A—C1—H1B	108.2	C4—C3—H3B	108.8
N1—C2—C1	110.06 (9)	H3A—C3—H3B	107.6
N1—C2—H2A	109.6	N2—C4—C3	178.07 (16)
C3—N1—C2—C1	−174.46 (8)	C2—N1—C3—C4	−64.42 (14)
C1ⁱ—N1—C2—C1	58.45 (13)	C1ⁱ—N1—C3—C4	61.41 (14)
N1ⁱ—C1—C2—N1	−58.10 (13)	N1—C3—C4—N2	13 (5)

Symmetry code: (i) $-x, -y+1, -z$.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···N2ⁱⁱ	0.97	2.57	3.427 (2)	147

Symmetry code: (ii) $-x+1, y-1/2, -z+1/2$.