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2-[(1-Methyl-1*H*-pyrrol-2-yl)methylidene]propanedinitrile

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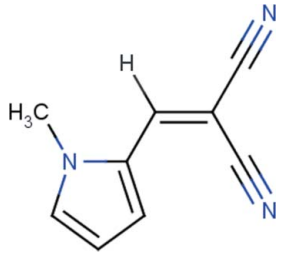
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.124; data-to-parameter ratio = 13.6.

In the title compound, $C_9H_7N_3$, the N-bound methyl group and vinyl H atom are *syn*. The 12 non-H atoms comprising the molecule are essentially coplanar (r.m.s. deviation = 0.071 Å). Supramolecular tapes feature in the crystal packing, orientated perpendicular to [10$\bar{1}$], and are formed by C—H···N interactions involving each cyano N atom. The tapes are connected into layers *via* π–π interactions occurring between translationally related pyrrole rings [ring centroid–centroid distance = 3.8754 (10) Å]; the layers stack along the *b* axis.

Related literature

For the anti-cancer effects of related compounds, see: Rostom *et al.* (2011). For structural studies of di-carbonitrile compounds, see: Asiri *et al.* (2011); Al-Youbi *et al.* (2012).



Experimental

Crystal data

$C_9H_7N_3$
M_r = 157.18
Triclinic, $P\bar{1}$
a = 3.8754 (2) Å
b = 8.7795 (5) Å
c = 12.1773 (7) Å
α = 97.517 (5)°
β = 90.962 (5)°
γ = 98.689 (5)°
V = 405.76 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 100 K
0.25 × 0.15 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.980, T_{max} = 0.996
5871 measured reflections
1866 independent reflections
1463 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.124
S = 1.01
1866 reflections
137 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···N3^{i}	0.976 (19)	2.612 (19)	3.579 (2)	170.8 (16)
C6—H6···N2^{ii}	0.969 (17)	2.515 (17)	3.469 (2)	167.8 (14)

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

Data collection: *CrysAlis PRO* (Agilent, 2011); 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: *X-SEED* (Barbour, 2001) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5851).

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supplementary materials

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Comment

Arylidenes are considered as key intermediates for the synthesis of a variety of heterocycles of biological importance, such as pyridine, pyridazine and quinoline derivatives. Previous studies have shown that the derived compounds exhibit a variety of biological activities, including anti-cancer effects (Rostom *et al.*, 2011). In continuation of structural studies of di-carbonitrile compounds (Asiri *et al.*, 2011; Al-Youbi *et al.*, 2012), the title compound, (I), was investigated.

In (I), Fig. 1, the N-bound methyl group and vinyl-H atom are *syn*. The 12 non-hydrogen atoms are co-planar having a r.m.s. deviation = 0.071 Å, with the maximum deviations being 0.118 (2) Å for the C1 atom and -0.084 (2) Å for the N2 atom.

In the crystal packing, each cyano-N atom participates in a C—H···N interaction, Table 1, with a centrosymmetrically related molecule to form a supramolecular tape. The tape is orientated along [10$\bar{1}$] and comprises alternating 10-membered $\{\cdots HC_3N\}_2$ and 16-membered $\{\cdots HC_6N\}_2$ synthons, Fig. 2. The tapes are connected into layers *via* π—π interactions occurring between translationally related pyrrazole rings [ring centroid..centroid distance = 3.8754 (10) Å for symmetry operation 1 + *x*, *y*, *z*]. The layers stack along the *b* axis, Fig. 3.

Experimental

A mixture of 1-methylpyrrole-2-carboxaldehyde (1.1 g, 0.01 mmol) and malononitrile (1.1 g, 0.01 mmol) in absolute ethanol (50 ml) was refluxed for 2 h. The reaction mixture was allowed to cool, and the formed precipitate was filtered, washed with water, dried and recrystallized from ethanol. Yield: 72%. *M*.pt: 427–229 K.

Refinement

All H-atoms were located in a difference map and were refined freely, the range of C—H bond lengths = 0.952 (19) to 1.002 (19) Å.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

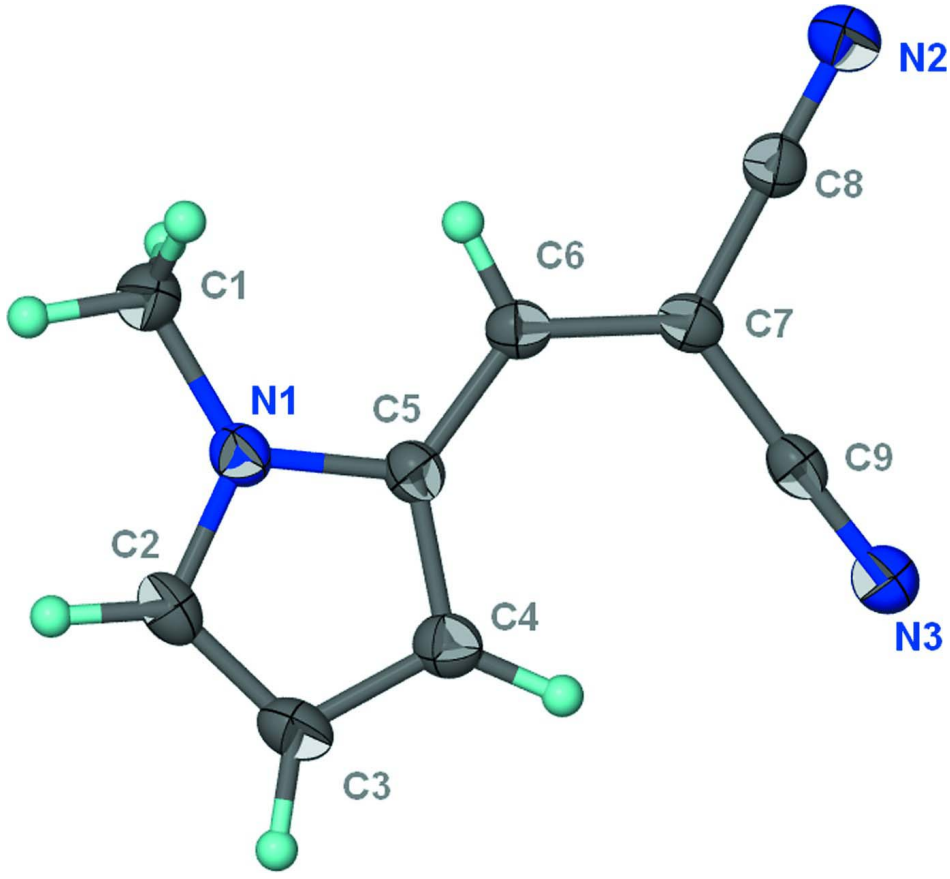

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 70% probability level.

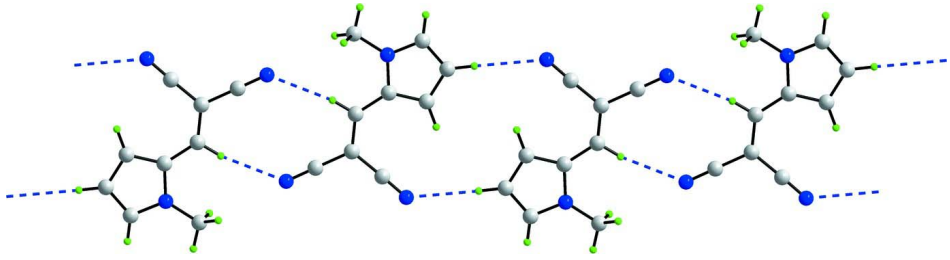

Figure 2

A view of the supramolecular tape in (I) with C—H···N interactions shown as blue dashed lines.

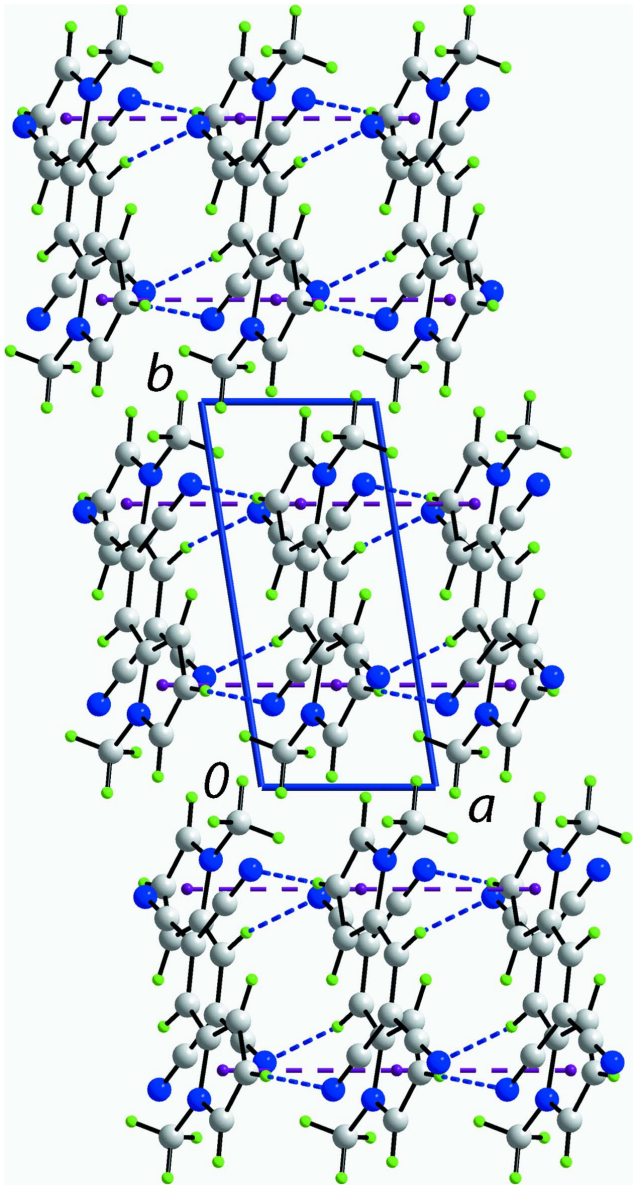

Figure 3

A view in projection down the *c* axis of the unit-cell contents of (I) showing the stacking of layers along the *b* axis. The C—H···N and π—π interactions are shown as blue and purple dashed lines, respectively.

2-[(1-Methyl-1*H*-pyrrol-2-yl)methylidene]propanedinitrile

Crystal data

$C_9H_7N_3$
M_r = 157.18
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 3.8754 (2) Å
b = 8.7795 (5) Å
c = 12.1773 (7) Å
α = 97.517 (5)°
β = 90.962 (5)°
γ = 98.689 (5)°
V = 405.76 (4) Å^3
Z = 2
$F(000)$ = 164
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1724 reflections
θ = 2.4–27.5°
μ = 0.08 mm^{-1}

T = 100 K
Prism, light-brown

0.25 × 0.15 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)

T_{min} = 0.980, T_{max} = 0.996
5871 measured reflections
1866 independent reflections
1463 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.6°, θ_{min} = 2.4°
$h = -5 \rightarrow 5$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.124
S = 1.01
1866 reflections
137 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
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 0.1622P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
N1	0.3583 (3)	0.18886 (15)	0.26433 (10)	0.0202 (3)
N2	0.2317 (4)	0.71488 (16)	0.02337 (11)	0.0267 (3)
N3	0.8617 (4)	0.78536 (16)	0.32981 (11)	0.0267 (3)
C1	0.1644 (5)	0.0922 (2)	0.16953 (14)	0.0250 (4)
C2	0.4697 (4)	0.13538 (19)	0.35574 (13)	0.0234 (4)
C3	0.6539 (4)	0.25848 (19)	0.42647 (13)	0.0246 (4)
C4	0.6548 (4)	0.39208 (19)	0.37650 (12)	0.0220 (4)
C5	0.4681 (4)	0.34899 (17)	0.27400 (12)	0.0188 (3)
C6	0.3763 (4)	0.43469 (17)	0.19062 (12)	0.0185 (3)
C7	0.4655 (4)	0.59152 (18)	0.18566 (12)	0.0190 (3)
C8	0.3370 (4)	0.65898 (17)	0.09541 (12)	0.0202 (3)
C9	0.6828 (4)	0.69787 (17)	0.26673 (12)	0.0196 (3)
H11	0.106 (5)	−0.015 (3)	0.1842 (17)	0.041 (6)*
H12	0.308 (5)	0.090 (2)	0.1053 (17)	0.038 (5)*
H13	−0.050 (5)	0.132 (2)	0.1524 (16)	0.033 (5)*
H2	0.412 (5)	0.027 (2)	0.3608 (15)	0.025 (4)*
H3	0.763 (5)	0.250 (2)	0.4978 (16)	0.031 (5)*
H4	0.769 (5)	0.500 (2)	0.4051 (14)	0.024 (4)*
H6	0.220 (4)	0.378 (2)	0.1311 (14)	0.020 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0225 (7)	0.0198 (7)	0.0189 (6)	0.0031 (5)	0.0018 (5)	0.0047 (5)

N2	0.0320 (8)	0.0269 (7)	0.0215 (7)	0.0040 (6)	−0.0007 (6)	0.0056 (6)
N3	0.0310 (8)	0.0241 (7)	0.0245 (7)	0.0008 (6)	−0.0021 (6)	0.0062 (6)
C1	0.0282 (9)	0.0221 (8)	0.0235 (8)	0.0002 (7)	−0.0013 (7)	0.0037 (6)
C2	0.0255 (8)	0.0227 (8)	0.0244 (8)	0.0054 (6)	0.0050 (6)	0.0097 (6)
C3	0.0254 (8)	0.0301 (9)	0.0201 (8)	0.0054 (7)	0.0008 (6)	0.0089 (6)
C4	0.0209 (8)	0.0249 (8)	0.0204 (8)	0.0024 (6)	0.0018 (6)	0.0046 (6)
C5	0.0188 (7)	0.0196 (7)	0.0182 (7)	0.0022 (6)	0.0029 (6)	0.0045 (6)
C6	0.0179 (7)	0.0213 (8)	0.0163 (7)	0.0034 (6)	0.0018 (6)	0.0023 (6)
C7	0.0195 (7)	0.0221 (8)	0.0162 (7)	0.0042 (6)	0.0023 (6)	0.0041 (6)
C8	0.0214 (8)	0.0201 (7)	0.0191 (8)	0.0028 (6)	0.0022 (6)	0.0023 (6)
C9	0.0208 (7)	0.0201 (7)	0.0198 (8)	0.0041 (6)	0.0037 (6)	0.0079 (6)

Geometric parameters (Å, º)

N1—C2	1.352 (2)	C3—C4	1.390 (2)
N1—C5	1.3949 (19)	C3—H3	0.977 (19)
N1—C1	1.463 (2)	C4—C5	1.410 (2)
N2—C8	1.157 (2)	C4—H4	1.002 (19)
N3—C9	1.152 (2)	C5—C6	1.412 (2)
C1—H11	0.97 (2)	C6—C7	1.378 (2)
C1—H12	0.97 (2)	C6—H6	0.969 (17)
C1—H13	0.98 (2)	C7—C8	1.431 (2)
C2—C3	1.386 (2)	C7—C9	1.431 (2)
C2—H2	0.952 (19)		
C2—N1—C5	108.97 (13)	C3—C4—C5	107.69 (14)
C2—N1—C1	124.98 (13)	C3—C4—H4	127.8 (10)
C5—N1—C1	126.02 (13)	C5—C4—H4	124.5 (10)
N1—C1—H11	110.6 (12)	N1—C5—C4	106.64 (13)
N1—C1—H12	109.7 (11)	N1—C5—C6	120.42 (13)
H11—C1—H12	106.9 (17)	C4—C5—C6	132.91 (14)
N1—C1—H13	111.0 (11)	C7—C6—C5	128.23 (14)
H11—C1—H13	109.4 (17)	C7—C6—H6	115.2 (10)
H12—C1—H13	109.2 (16)	C5—C6—H6	116.5 (10)
N1—C2—C3	109.17 (14)	C6—C7—C8	120.18 (13)
N1—C2—H2	118.3 (11)	C6—C7—C9	124.54 (13)
C3—C2—H2	132.5 (11)	C8—C7—C9	115.29 (13)
C2—C3—C4	107.53 (14)	N2—C8—C7	179.15 (16)
C2—C3—H3	125.1 (11)	N3—C9—C7	178.23 (16)
C4—C3—H3	127.3 (11)		
C5—N1—C2—C3	−0.11 (17)	C1—N1—C5—C6	3.9 (2)
C1—N1—C2—C3	177.99 (14)	C3—C4—C5—N1	−0.10 (17)
N1—C2—C3—C4	0.05 (18)	C3—C4—C5—C6	177.75 (16)
C2—C3—C4—C5	0.04 (18)	N1—C5—C6—C7	−178.89 (14)
C2—N1—C5—C4	0.13 (17)	C4—C5—C6—C7	3.5 (3)
C1—N1—C5—C4	−177.94 (14)	C5—C6—C7—C8	−177.93 (14)
C2—N1—C5—C6	−178.05 (13)	C5—C6—C7—C9	1.6 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···N3^{i}	0.976 (19)	2.612 (19)	3.579 (2)	170.8 (16)
C6—H6···N2^{ii}	0.969 (17)	2.515 (17)	3.469 (2)	167.8 (14)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) −*x*, −*y*+1, −*z*.