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2-(3-Methoxyphenyl)butanedinitrile

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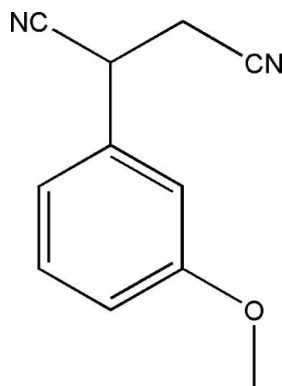
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.043; wR factor = 0.128; data-to-parameter ratio = 17.3.

In the title compound, $\text{C}_{11}\text{H}_{10}\text{N}_2\text{O}$, the dicyanoethylene portion has an *anti* conformation. The crystal structure features non-classical $\text{C}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

For the synthesis, see: Johnson *et al.* (1962). The title compound is an intermediate in the synthesis of drugs (Obniska *et al.*, 2005).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{10}\text{N}_2\text{O}$
 $M_r = 186.21$
 Monoclinic, $P2_1/c$

$a = 5.5263$ (8) Å
 $b = 16.105$ (2) Å
 $c = 11.0332$ (16) Å

$\beta = 97.179$ (2)°
 $V = 974.3$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.08$ mm⁻¹
 $T = 298$ K
 $0.4 \times 0.2 \times 0.1$ mm

Data collection

Bruker SMART area-detector diffractometer
 Absorption correction: none
 8042 measured reflections

2210 independent reflections
 1963 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.128$
 $S = 1.04$
 2210 reflections

128 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C8}-\text{H8}\cdots\text{N1}^{\text{i}}$	0.98	2.61	3.4864 (17)	150
$\text{C10}-\text{H10A}\cdots\text{O1}^{\text{ii}}$	0.97	2.38	3.2470 (15)	149
$\text{C10}-\text{H10B}\cdots\text{N2}^{\text{iii}}$	0.97	2.60	3.4823 (18)	151

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2569).

References

- Bruker (2000). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Johnson, F., Panella, J. P. & Carlson, A. A. (1962). *J. Org. Chem.* **28**, 2241–2243.
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 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

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2-(3-Methoxyphenyl)butanedinitrile

W.-X. Du, Y. Ye and X.-W. Wei

Comment

The title compound is an important intermediate in drugs synthesis (Obniska *et al.*, 2005). In this paper, we report the structure of the title compound (I). In (I), the succinonitrile moiety adopts an anti conformation. Six atoms of succinonitrile moiety, (N1/C9/C8/N2/C11/C10), almost lie on one plane, the maximum deviations from the mean plane of the succinonitrile moiety being 0.0275 (8) Å. This mean plane is almost perpendicular to the phenyl mean plane with a dihedral angle of 87.55 (6) Å. The crystal packing is stabilized by two intermolecular non-classic C—H \cdots N hydrogen bonds and one intermolecular non-classic C—H \cdots O hydrogen bond.

Experimental

The compound (I) was obtained by reaction of (*Z*)-ethyl-2-cyano-3-(4-methoxyphenyl)acrylate and NaCN in ethanol-water mixture according to the reported method (Johnson *et al.*, 1962). Single crystals suitable for X-ray diffraction were obtained by evaporation of an ethanol solution at room temperature.

Refinement

All non-hydrogen atoms were refined anisotropically. H atoms bonded to C atoms were introduced at calculated positions and refined using a riding model with C—H distances of 0.93–0.97 Å. In all cases, the H-atom $U_{\text{iso}}(\text{H})$ is 1.2 times U_{eq} of the parent atom.

Figures

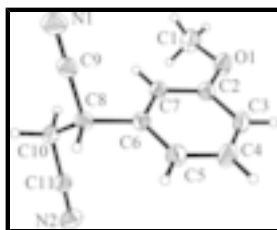


Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.



Fig. 2. A packing diagram of (I) viewed down the *a* axis. Dotted lines show the C—H \cdots N and C—H \cdots O hydrogen bonds.

2-(3-Methoxyphenyl)butanedinitrile

Crystal data

$C_{11}H_{10}N_2O$	$F_{000} = 392$
$M_r = 186.21$	$D_x = 1.270 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 5.5263 (8) \text{ \AA}$	Cell parameters from 5752 reflections
$b = 16.105 (2) \text{ \AA}$	$\theta = 2.3\text{--}27.4^\circ$
$c = 11.0332 (16) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$\beta = 97.179 (2)^\circ$	$T = 298 \text{ K}$
$V = 974.3 (2) \text{ \AA}^3$	Block, colorless
$Z = 4$	$0.4 \times 0.2 \times 0.1 \text{ mm}$

Data collection

Bruker SMART area-detector diffractometer	1963 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.022$
Monochromator: graphite	$\theta_{\text{max}} = 27.5^\circ$
$T = 298 \text{ K}$	$\theta_{\text{min}} = 2.3^\circ$
φ and ω scans	$h = -7 \rightarrow 7$
Absorption correction: None	$k = -20 \rightarrow 20$
8042 measured reflections	$l = -14 \rightarrow 14$
2210 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.128$	$w = 1/[\sigma^2(F_o^2) + (0.0729P)^2 + 0.1612P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
2210 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
128 parameters	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
	Extinction correction: none

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C6	0.6739 (2)	0.85144 (7)	0.67253 (10)	0.0379 (3)
C7	0.5211 (2)	0.80066 (7)	0.73097 (11)	0.0406 (3)
H7	0.4177	0.8237	0.7822	0.049*
C5	0.8292 (3)	0.81733 (9)	0.59747 (12)	0.0518 (3)
H5	0.9321	0.8512	0.5588	0.062*
C2	0.5237 (2)	0.71565 (7)	0.71258 (11)	0.0439 (3)
C3	0.6789 (3)	0.68150 (8)	0.63618 (14)	0.0551 (4)
H3	0.6799	0.6245	0.6231	0.066*
C4	0.8306 (3)	0.73211 (9)	0.58018 (15)	0.0622 (4)
H4	0.9358	0.7090	0.5299	0.075*
C8	0.6709 (2)	0.94492 (7)	0.69077 (10)	0.0384 (3)
H8	0.7891	0.9698	0.6423	0.046*
C10	0.7385 (2)	0.97151 (7)	0.82538 (11)	0.0414 (3)
H10A	0.7311	1.0315	0.8311	0.050*
H10B	0.6209	0.9485	0.8744	0.050*
C9	0.4279 (2)	0.97888 (7)	0.64751 (11)	0.0435 (3)
O1	0.37911 (19)	0.66090 (5)	0.76429 (10)	0.0605 (3)
C1	0.2421 (3)	0.69035 (10)	0.85616 (15)	0.0638 (4)
H1A	0.3505	0.7141	0.9218	0.096*
H1B	0.1545	0.6450	0.8867	0.096*
H1C	0.1286	0.7318	0.8221	0.096*
N1	0.2393 (2)	1.00418 (8)	0.61421 (12)	0.0604 (3)
C11	0.9826 (2)	0.94328 (7)	0.87350 (10)	0.0415 (3)
N2	1.1722 (2)	0.92210 (8)	0.91331 (11)	0.0581 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C6	0.0380 (6)	0.0381 (6)	0.0376 (5)	-0.0021 (4)	0.0054 (4)	-0.0030 (4)
C7	0.0415 (6)	0.0363 (6)	0.0462 (6)	-0.0003 (4)	0.0139 (5)	-0.0042 (4)
C5	0.0540 (8)	0.0522 (7)	0.0537 (7)	-0.0070 (6)	0.0239 (6)	-0.0074 (5)
C2	0.0443 (6)	0.0368 (6)	0.0518 (6)	-0.0028 (5)	0.0112 (5)	-0.0029 (5)
C3	0.0617 (8)	0.0384 (6)	0.0679 (8)	0.0001 (5)	0.0188 (6)	-0.0143 (6)
C4	0.0668 (9)	0.0565 (8)	0.0696 (9)	-0.0002 (7)	0.0339 (7)	-0.0175 (7)
C8	0.0364 (6)	0.0373 (6)	0.0417 (6)	-0.0028 (4)	0.0053 (4)	0.0024 (4)
C10	0.0399 (6)	0.0366 (5)	0.0467 (6)	0.0036 (4)	0.0019 (5)	-0.0048 (4)
C9	0.0436 (7)	0.0397 (6)	0.0460 (6)	-0.0049 (5)	0.0007 (5)	0.0055 (5)

supplementary materials

O1	0.0694 (7)	0.0352 (5)	0.0829 (7)	-0.0077 (4)	0.0339 (5)	-0.0025 (4)
C1	0.0727 (10)	0.0539 (8)	0.0708 (9)	-0.0093 (7)	0.0331 (8)	0.0033 (7)
N1	0.0479 (7)	0.0619 (7)	0.0679 (8)	0.0001 (5)	-0.0065 (5)	0.0126 (6)
C11	0.0429 (6)	0.0402 (6)	0.0413 (6)	0.0009 (5)	0.0050 (5)	-0.0025 (4)
N2	0.0469 (6)	0.0739 (8)	0.0525 (6)	0.0129 (5)	0.0023 (5)	-0.0037 (5)

Geometric parameters (Å, °)

C6—C5	1.3791 (16)	C8—C9	1.4730 (16)
C6—C7	1.3903 (15)	C8—C10	1.5460 (16)
C6—C8	1.5193 (15)	C8—H8	0.9800
C7—C2	1.3843 (16)	C10—C11	1.4593 (16)
C7—H7	0.9300	C10—H10A	0.9700
C5—C4	1.386 (2)	C10—H10B	0.9700
C5—H5	0.9300	C9—N1	1.1365 (16)
C2—O1	1.3623 (15)	O1—C1	1.4205 (17)
C2—C3	1.3890 (17)	C1—H1A	0.9600
C3—C4	1.371 (2)	C1—H1B	0.9600
C3—H3	0.9300	C1—H1C	0.9600
C4—H4	0.9300	C11—N2	1.1368 (16)
C5—C6—C7	120.25 (11)	C6—C8—C10	113.36 (9)
C5—C6—C8	119.48 (10)	C9—C8—H8	108.3
C7—C6—C8	120.28 (9)	C6—C8—H8	108.3
C2—C7—C6	119.71 (10)	C10—C8—H8	108.3
C2—C7—H7	120.1	C11—C10—C8	111.38 (9)
C6—C7—H7	120.1	C11—C10—H10A	109.4
C6—C5—C4	119.46 (12)	C8—C10—H10A	109.4
C6—C5—H5	120.3	C11—C10—H10B	109.4
C4—C5—H5	120.3	C8—C10—H10B	109.4
O1—C2—C7	124.16 (11)	H10A—C10—H10B	108.0
O1—C2—C3	115.90 (11)	N1—C9—C8	179.20 (13)
C7—C2—C3	119.94 (11)	C2—O1—C1	118.40 (10)
C4—C3—C2	119.79 (11)	O1—C1—H1A	109.5
C4—C3—H3	120.1	O1—C1—H1B	109.5
C2—C3—H3	120.1	H1A—C1—H1B	109.5
C3—C4—C5	120.84 (12)	O1—C1—H1C	109.5
C3—C4—H4	119.6	H1A—C1—H1C	109.5
C5—C4—H4	119.6	H1B—C1—H1C	109.5
C9—C8—C6	110.45 (9)	N2—C11—C10	178.53 (13)
C9—C8—C10	108.02 (9)		
C5—C6—C7—C2	-0.64 (18)	C6—C5—C4—C3	0.3 (2)
C8—C6—C7—C2	179.32 (11)	C5—C6—C8—C9	118.72 (12)
C7—C6—C5—C4	0.4 (2)	C7—C6—C8—C9	-61.25 (14)
C8—C6—C5—C4	-179.55 (13)	C5—C6—C8—C10	-119.90 (12)
C6—C7—C2—O1	-179.11 (11)	C7—C6—C8—C10	60.14 (14)
C6—C7—C2—C3	0.13 (19)	C9—C8—C10—C11	-177.45 (10)
O1—C2—C3—C4	179.91 (14)	C6—C8—C10—C11	59.81 (13)
C7—C2—C3—C4	0.6 (2)	C7—C2—O1—C1	-9.4 (2)
C2—C3—C4—C5	-0.8 (2)	C3—C2—O1—C1	171.30 (14)

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8—H8 \cdots N1 ⁱ	0.98	2.61	3.4864 (17)	150
C10—H10A \cdots O1 ⁱⁱ	0.97	2.38	3.2470 (15)	149
C10—H10B \cdots N2 ⁱⁱⁱ	0.97	2.60	3.4823 (18)	151

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

Fig. 1

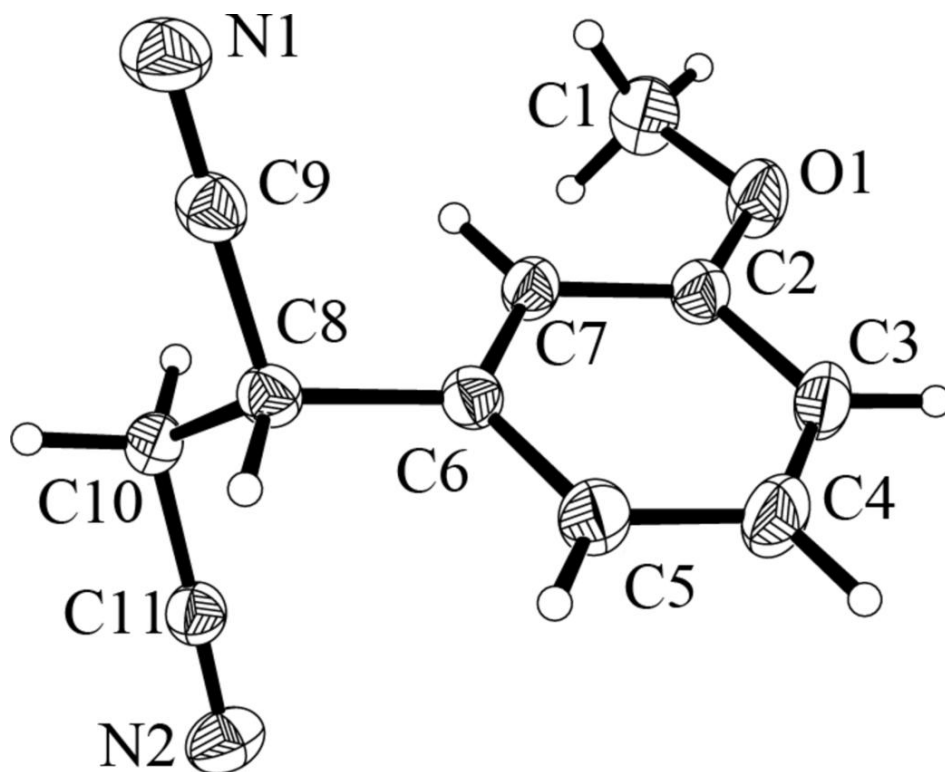


Fig. 2

