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# 2-[1-(3-Aminophenyl)ethylidene]-propanedinitrile

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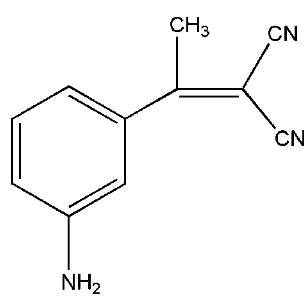
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.051; $wR$ factor = 0.124; data-to-parameter ratio = 13.3.

In the title compound, $C_{11}H_9N_3$, all bond lengths and angles are normal. The crystal packing is stabilized by intermolecular N—H···N hydrogen-bond interactions involving the H atoms of the amino groups and N atoms of the cyano groups.

## Related literature

For related literature, see: Bigi *et al.* (2000); Freeman (1980); Wardell *et al.* (2006).




## Experimental

### Crystal data

$C_{11}H_9N_3$
$M_r$ = 183.21
Monoclinic, $P2_1/n$
$a$ = 7.4654 (8) Å
$b$ = 13.7051 (18) Å
$c$ = 9.5361 (17) Å
$\beta$ = 99.961 (2)°
$V$ = 961.0 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 298 (2) K
0.40 × 0.35 × 0.11 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.969, $T_{max}$ = 0.991
4744 measured reflections
1687 independent reflections
944 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.053

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.124
$S$ = 1.00
1687 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H3$A$···N1$^{i}$ | 0.86 | 2.35 | 3.209 (3) | 173 |
| N3—H3$B$···N2$^{ii}$ | 0.86 | 2.33 | 3.182 (3) | 170 |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000) and *DIAMOND* (Brandenburg, 2004); 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: EZ2100).

## References

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o4316 [ **doi:10.1107/S1600536807047423** ]

## 2-[1-(3-Aminophenyl)ethylidene]propanedinitrile

**Q. Cheng, X. Xu, C. Zong and H. Feng**

### Comment

Knoevenagel condensation of carbonyl compounds with compounds containing an active methylene group is one of the most important methods of preparing substituted alkenes. As a part of our study of benzylidenemalonontriles, which are effective anti-fouling agents, fungicides, cytotoxic agents and insecticides (Freeman, 1980; Bigi *et al.*, 2000), we report here the structure of the title compound, (I), synthesized by Knoevenagel condensation of *m*-aminoacetophenone with malononitrile.

In (I) (Fig. 1), all bond lengths and angles agree well with those reported for related compounds (Wardell *et al.*, 2006). The amino and cyano groups are involved in intermolecular N—H···N hydrogen bonds (Table 1), which link the molecules into 26-membered rings (Fig. 2). The amino N atom acts as a hydrogen-bond donor to the cyano N atom in a neighbouring molecule, thus forming layers along the *bc*-plane.

### Experimental

A mixture of *m*-aminoacetophenone (15 mmol) and malononitrile (15 mmol) in distilled water (15 ml) was heated to 353 K for 2 h. Upon cooling to room temperature, a crude product crystallized. The precipitate was filtered off, washed with ethanol and recrystallized from ethanol to afford the desired product as a colourless solid. Colourless single crystals of (I) were obtained by slow evaporation of an aqueous ethanol (95%) solution at ambient temperatures after 10 d. Elemental analysis, calculated for C11 H9 N3: C 72.11, H 4.95, N 22.94%; found: C 72.04. H 4.91, N 22.98%.

### Refinement

All hydrogen atoms were geometrically fixed at calculated positions and allowed to ride on their parent atoms with C—H = 0.93–0.96 Å, N—H = 0.86 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(C,*N*).

### Figures

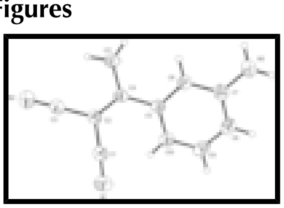

Fig. 1. Molecular structure of the title complex showing the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

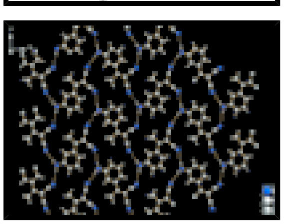

Fig. 2. Part of the crystal structure of (I), showing the 26-membered rings formed by N—H···N intermolecular hydrogen-bonds. Hydrogen bonds are shown as dashed lines.

## 2-[1-(3-Aminophenyl)ethylidene]propanedinitrile

### *Crystal data*

| | |
|---|---|
| $C_{11}H_9N_3$ | $F_{000}$ = 384 |
| $M_r$ = 183.21 | $D_x$ = 1.266 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2yn | Cell parameters from 1277 reflections |
| $a$ = 7.4654 (8) Å | θ = 2.5–22.2º |
| $b$ = 13.7051 (18) Å | μ = 0.08 mm$^{-1}$ |
| $c$ = 9.5361 (17) Å | $T$ = 298 (2) K |
| β = 99.961 (2)º | Needle, colourless |
| $V$ = 961.0 (2) Å$^3$ | 0.40 × 0.35 × 0.11 mm |
| $Z$ = 4 | |

### *Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 1687 independent reflections |
| Radiation source: fine-focus sealed tube | 944 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.053 |
| $T$ = 298(2) K | $\theta_{max}$ = 25.0º |
| φ and ω scans | $\theta_{min}$ = 2.6º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -8 \rightarrow 7$ |
| $T_{min}$ = 0.969, $T_{max}$ = 0.991 | $k = -12 \rightarrow 16$ |
| 4744 measured reflections | $l = -11 \rightarrow 11$ |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.051 | H-atom parameters constrained |
| $wR(F^2)$ = 0.124 | $w = 1/[\sigma^2(F_o^2) + (0.0469P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.00 | $(\Delta/\sigma)_{max}$ < 0.001 |
| 1687 reflections | $\Delta\rho_{max}$ = 0.16 e Å$^{-3}$ |
| 127 parameters | $\Delta\rho_{min}$ = −0.17 e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | 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 $F^2 > 2\text{sigma}(F^2)$ 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 | 0.2598 (3) | 0.98666 (15) | 0.4529 (3) | 0.0732 (7) |
| N2 | 0.1402 (3) | 0.89557 (17) | 0.0215 (3) | 0.0784 (7) |
| N3 | 0.2430 (3) | 0.53195 (15) | 0.7174 (2) | 0.0798 (7) |
| H3A | 0.2525 | 0.5189 | 0.8066 | 0.096* |
| H3B | 0.2815 | 0.4908 | 0.6613 | 0.096* |
| C1 | 0.2043 (3) | 0.92393 (18) | 0.3799 (3) | 0.0536 (7) |
| C2 | 0.1377 (3) | 0.84685 (16) | 0.2806 (2) | 0.0461 (6) |
| C3 | 0.1375 (3) | 0.87283 (17) | 0.1360 (3) | 0.0553 (7) |
| C4 | 0.0880 (3) | 0.75772 (16) | 0.3203 (2) | 0.0444 (6) |
| C5 | 0.0877 (3) | 0.73249 (16) | 0.4696 (2) | 0.0435 (6) |
| C6 | 0.1566 (3) | 0.64313 (16) | 0.5219 (3) | 0.0499 (6) |
| H6 | 0.1970 | 0.5988 | 0.4604 | 0.060* |
| C7 | 0.1666 (3) | 0.61835 (18) | 0.6645 (3) | 0.0536 (7) |
| C8 | 0.1040 (3) | 0.68524 (19) | 0.7545 (3) | 0.0577 (7) |
| H8 | 0.1115 | 0.6709 | 0.8507 | 0.069* |
| C9 | 0.0308 (3) | 0.7728 (2) | 0.7010 (3) | 0.0592 (7) |
| H9 | −0.0139 | 0.8161 | 0.7616 | 0.071* |
| C10 | 0.0223 (3) | 0.79763 (17) | 0.5605 (3) | 0.0509 (6) |
| H10 | −0.0265 | 0.8573 | 0.5267 | 0.061* |
| C11 | 0.0328 (3) | 0.68186 (16) | 0.2086 (3) | 0.0583 (7) |
| H11A | 0.0460 | 0.7075 | 0.1172 | 0.088* |
| H11B | −0.0918 | 0.6640 | 0.2073 | 0.088* |
| H11C | 0.1088 | 0.6254 | 0.2294 | 0.088* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0955 (18) | 0.0534 (14) | 0.0705 (17) | −0.0135 (13) | 0.0140 (14) | −0.0121 (13) |
| N2 | 0.1029 (19) | 0.0718 (16) | 0.0607 (16) | 0.0024 (14) | 0.0152 (14) | 0.0081 (14) |
| N3 | 0.119 (2) | 0.0599 (14) | 0.0606 (15) | 0.0067 (14) | 0.0162 (14) | 0.0154 (13) |
| C1 | 0.0626 (18) | 0.0447 (15) | 0.0532 (16) | 0.0020 (13) | 0.0090 (13) | 0.0022 (14) |
| C2 | 0.0488 (15) | 0.0424 (14) | 0.0467 (15) | 0.0022 (11) | 0.0072 (12) | 0.0003 (12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3 | 0.0638 (17) | 0.0450 (15) | 0.0570 (17) | −0.0001 (12) | 0.0107 (14) | 0.0013 (14) |
| C4 | 0.0399 (13) | 0.0436 (13) | 0.0496 (15) | 0.0053 (11) | 0.0073 (11) | −0.0013 (12) |
| C5 | 0.0424 (14) | 0.0403 (13) | 0.0476 (15) | −0.0037 (11) | 0.0074 (11) | −0.0027 (12) |
| C6 | 0.0580 (16) | 0.0438 (14) | 0.0487 (15) | −0.0029 (12) | 0.0115 (12) | −0.0002 (13) |
| C7 | 0.0541 (16) | 0.0506 (15) | 0.0548 (17) | −0.0081 (13) | 0.0062 (13) | 0.0078 (15) |
| C8 | 0.0611 (17) | 0.0674 (18) | 0.0452 (15) | −0.0068 (15) | 0.0113 (13) | 0.0015 (15) |
| C9 | 0.0524 (16) | 0.0719 (19) | 0.0561 (18) | −0.0034 (14) | 0.0175 (13) | −0.0129 (15) |
| C10 | 0.0471 (15) | 0.0494 (14) | 0.0572 (16) | −0.0003 (12) | 0.0119 (12) | −0.0032 (13) |
| C11 | 0.0678 (17) | 0.0506 (15) | 0.0560 (17) | −0.0070 (13) | 0.0089 (13) | −0.0077 (13) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| N1—C1 | 1.138 (3) | C6—C7 | 1.391 (3) |
| N2—C3 | 1.139 (3) | C6—H6 | 0.9300 |
| N3—C7 | 1.372 (3) | C7—C8 | 1.390 (3) |
| N3—H3A | 0.8600 | C8—C9 | 1.379 (3) |
| N3—H3B | 0.8600 | C8—H8 | 0.9300 |
| C1—C2 | 1.448 (3) | C9—C10 | 1.373 (3) |
| C2—C4 | 1.350 (3) | C9—H9 | 0.9300 |
| C2—C3 | 1.424 (3) | C10—H10 | 0.9300 |
| C4—C5 | 1.466 (3) | C11—H11A | 0.9600 |
| C4—C11 | 1.494 (3) | C11—H11B | 0.9600 |
| C5—C6 | 1.387 (3) | C11—H11C | 0.9600 |
| C5—C10 | 1.391 (3) | | |
| C7—N3—H3A | 120.0 | N3—C7—C6 | 121.2 (2) |
| C7—N3—H3B | 120.0 | C8—C7—C6 | 118.4 (2) |
| H3A—N3—H3B | 120.0 | C9—C8—C7 | 119.9 (2) |
| N1—C1—C2 | 176.8 (3) | C9—C8—H8 | 120.0 |
| C4—C2—C3 | 122.9 (2) | C7—C8—H8 | 120.0 |
| C4—C2—C1 | 123.8 (2) | C10—C9—C8 | 121.7 (2) |
| C3—C2—C1 | 113.2 (2) | C10—C9—H9 | 119.2 |
| N2—C3—C2 | 178.2 (3) | C8—C9—H9 | 119.2 |
| C2—C4—C5 | 122.2 (2) | C9—C10—C5 | 119.2 (2) |
| C2—C4—C11 | 119.0 (2) | C9—C10—H10 | 120.4 |
| C5—C4—C11 | 118.8 (2) | C5—C10—H10 | 120.4 |
| C6—C5—C10 | 119.4 (2) | C4—C11—H11A | 109.5 |
| C6—C5—C4 | 119.7 (2) | C4—C11—H11B | 109.5 |
| C10—C5—C4 | 120.9 (2) | H11A—C11—H11B | 109.5 |
| C5—C6—C7 | 121.4 (2) | C4—C11—H11C | 109.5 |
| C5—C6—H6 | 119.3 | H11A—C11—H11C | 109.5 |
| C7—C6—H6 | 119.3 | H11B—C11—H11C | 109.5 |
| N3—C7—C8 | 120.3 (2) | | |
| C3—C2—C4—C5 | −179.5 (2) | C4—C5—C6—C7 | 177.0 (2) |
| C1—C2—C4—C5 | 3.5 (3) | C5—C6—C7—N3 | −176.7 (2) |
| C3—C2—C4—C11 | 1.0 (3) | C5—C6—C7—C8 | 0.6 (3) |
| C1—C2—C4—C11 | −176.0 (2) | N3—C7—C8—C9 | 178.6 (2) |
| C2—C4—C5—C6 | −136.5 (2) | C6—C7—C8—C9 | 1.3 (4) |
| C11—C4—C5—C6 | 43.0 (3) | C7—C8—C9—C10 | −1.9 (4) |
| C2—C4—C5—C10 | 42.3 (3) | C8—C9—C10—C5 | 0.6 (4) |

| | | | |
|---|---|---|---|
| C11—C4—C5—C10 | −138.2 (2) | C6—C5—C10—C9 | 1.3 (3) |
| C10—C5—C6—C7 | −1.9 (3) | C4—C5—C10—C9 | −177.5 (2) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3A···N1$^{i}$ | 0.86 | 2.35 | 3.209 (3) | 173 |
| N3—H3B···N2$^{ii}$ | 0.86 | 2.33 | 3.182 (3) | 170 |

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

**Fig. 1**

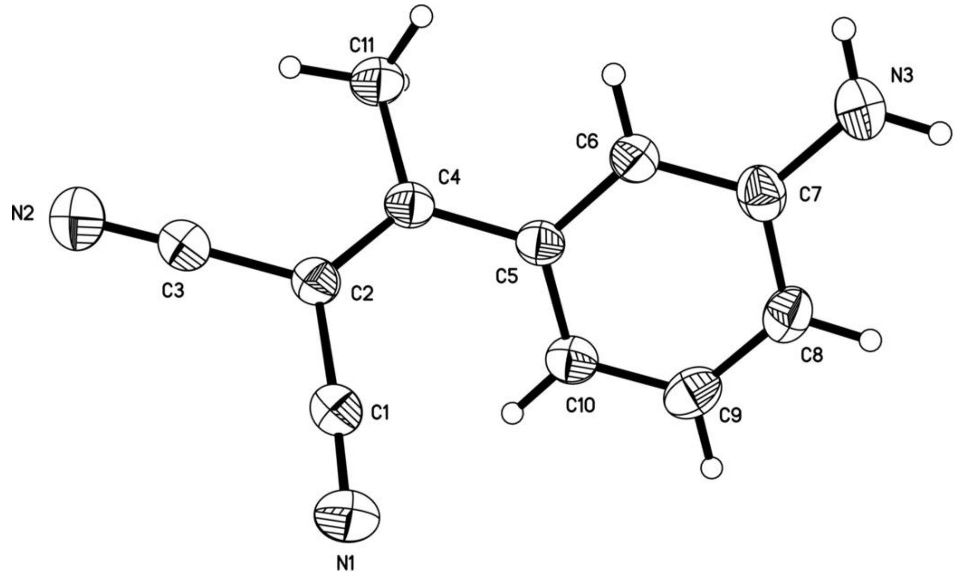

Fig. 2