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ISSN 2414-3146

1-(2-Amino-4,5-dimethylphenyl)ethanone

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Received 21 February 2018
Accepted 22 February 2018

Edited by J. Simpson, University of Otago, New Zealand

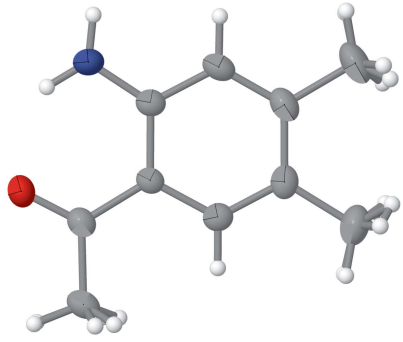
Keywords: crystal structure; 2-aminoacetophenone; hydrogen bonding.

CCDC reference: 1825318

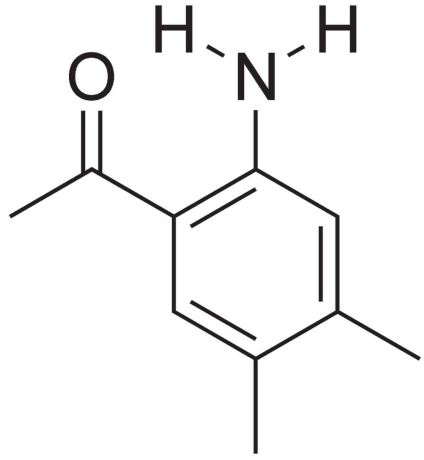
Structural data: full structural data are available from iucrdata.iucr.org

The molecule of the title compound, $C_{10}H_{13}NO$, also referred to as 2-amino-4,5-dimethylacetophenone, lies on a crystallographic mirror plane with four molecules in the orthorhombic unit cell and features an intramolecular N—H···O hydrogen bond. In the crystal, the molecules are linked by N—H···O hydrogen bonds, forming ribbons along the *a* axis that pack to form sheets lying in the (010) plane.

3D view



Chemical scheme



Structure description

The synthesis of 2-aminoacetophenone was accomplished in a one-pot reduction and hydration of 2-nitrophenacetylene with a variety of reagents generally used for the reduction of nitrobenzenes (Bosch & Jeffries, 2001). The asymmetric unit of the title compound comprises a single molecule that lies on a mirror plane with hydrogen atoms of the three methyl groups disordered over two positions. An intramolecular N1—H1*N*···O1 hydrogen bond (Fig. 1 and Table 1) supports the planar structure. A search of the Cambridge Structural Database (CSD, Version 5.39, November 2017, Groom *et al.*, 2016) using *Conquest* (Bruno *et al.*, 2002) for neutral uncomplexed molecules, including the 2-aminoacetophenone framework, yielded 99 hits. CSD entries not including atomic coordinates for H atoms were excluded. In 96 of these structures, an intramolecular N—H···O hydrogen bond was observed.

In the crystal, ribbons form along the *a*-axis direction through N1—H2*N*···O1 hydrogen bonds. Adjacent ribbons pack to form sheets lying in the (010) plane (Fig. 2). These sheets stack parallel to (101). There are possible extremely weak offset π–π stacking interactions (Fig. 3), with a centroid-to-centroid distance between the stacked benzene rings of 5.1075 (8) Å and with a slippage of 3.768 Å. In addition, the closest contact between stacked molecules is between the acetophenone methyl group and the centroid of the benzene ring, with a C8···*Cg*1 distance of 3.4531 (3) Å, suggesting that

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1	0.89 (2)	1.96 (2)	2.6603 (19)	135 (2)
N1—H2*N*···O1^{i}	0.86 (2)	2.09 (2)	2.9478 (18)	170 (2)

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

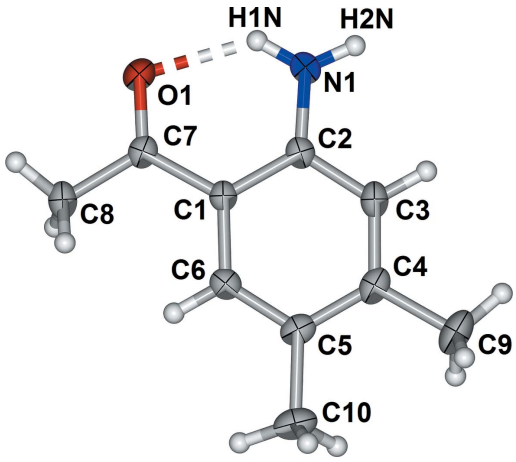


Figure 1
The asymmetric unit of the title compound, showing the atom-numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level and H atoms are shown as circles of arbitrary size. An intramolecular hydrogen bond is shown as a dashed line.

C8—H8···*Cg*1 contacts may also consolidate the stacking interaction, *Cg*1 is the centroid of the C1–C6 benzene ring.

Synthesis and crystallization

The title compound was synthesized by a one-pot hydration and reduction of 1-ethynyl-4,5-dimethyl-2-nitrobenzene with Fe/HCl, $SnCl_2$ or nickel boride and the isolation and characterization have been reported previously (Bosch & Jeffries, 2001). Crystals suitable for X-ray data collection were obtained by slow evaporation of a dichloromethane solution.

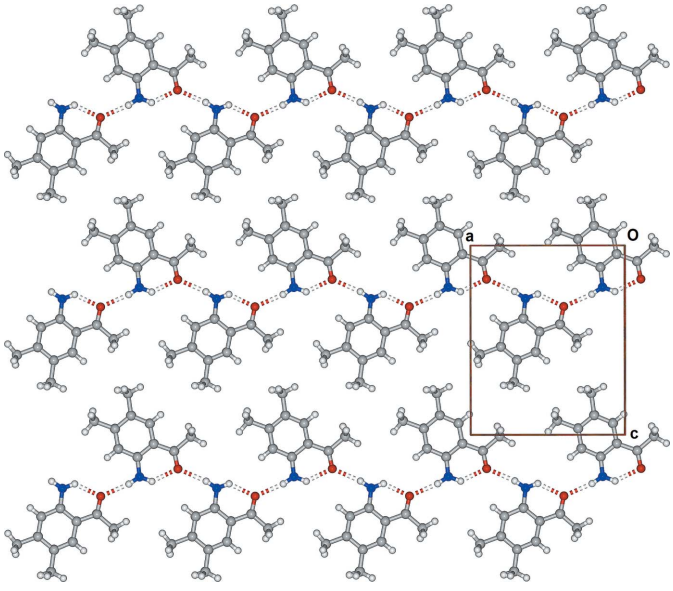


Figure 2
A view of a sheet comprising side-by-side packing of one-dimensional hydrogen-bonded ribbons of the title compound. Hydrogen bonds are shown as a dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{13}NO$
M_r	163.21
Crystal system, space group	Orthorhombic, *Pnma*
Temperature (K)	173
a, *b*, *c* (Å)	10.4838 (7), 6.8965 (5), 12.8538 (9)
V (Å^3)	929.35 (11)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.08
Crystal size (mm)	0.33 × 0.32 × 0.21
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.954, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11353, 1115, 883
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.641
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.116, 1.09
No. of reflections	1115
No. of parameters	80
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.19

Computer programs: *SMART* and *SAINT* (Bruker 2014), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2017* (Sheldrick, 2015*b*) and *X-SEED* (Barbour, 2001).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

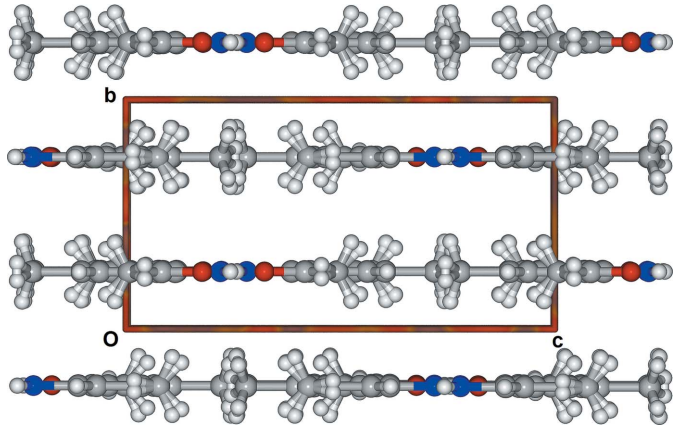


Figure 3
View along the *a* axis of the offset π-stacked planes shown in Fig. 2.

Acknowledgements

We thank the Missouri State University Provost Incentive Fund that funded the purchase of the X-ray diffractometer.

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bosch, E. & Jeffries, L. (2001). *Tetrahedron Lett.* **42**, 8141–8142.
Bruker (2014). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* B**58**, 389–397.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2018). **3**, x180313 [https://doi.org/10.1107/S2414314618003139]

1-(2-Amino-4,5-dimethylphenyl)ethanone

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Crystal data

$C_{10}H_{13}NO$
M_r = 163.21
Orthorhombic, *Pnma*
a = 10.4838 (7) Å
b = 6.8965 (5) Å
c = 12.8538 (9) Å
V = 929.35 (11) Å^3
Z = 4
F(000) = 352
D_x = 1.167 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2665 reflections
θ = 2.5–26.4°
μ = 0.08 mm^{-1}
T = 173 K
Cut block, yellow
0.33 × 0.32 × 0.21 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3660 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.954, T_{max} = 1.000
11353 measured reflections
1115 independent reflections
883 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.1°, θ_{min} = 2.5°
h = −13→13
k = −8→8
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.116
S = 1.09
1115 reflections
80 parameters
2 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.1911P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.39680 (11)	0.250000	0.32229 (9)	0.0446 (4)	
N1	0.64524 (14)	0.250000	0.28016 (11)	0.0448 (4)	
H1N	0.5657 (16)	0.250000	0.2566 (16)	0.054*	
H2N	0.7141 (16)	0.250000	0.2431 (15)	0.054*	
C1	0.55346 (14)	0.250000	0.45470 (11)	0.0281 (4)	
C2	0.65892 (14)	0.250000	0.38534 (12)	0.0308 (4)	
C7	0.42132 (14)	0.250000	0.41648 (12)	0.0314 (4)	
C6	0.57905 (15)	0.250000	0.56227 (12)	0.0320 (4)	
H6	0.510103	0.250000	0.607801	0.038*	
C5	0.69982 (16)	0.250000	0.60405 (12)	0.0352 (4)	
C8	0.31262 (15)	0.250000	0.49238 (13)	0.0374 (4)	
H8A	0.326942	0.347668	0.544295	0.056*	0.5
H8B	0.306872	0.125247	0.525175	0.056*	0.5
H8C	0.234499	0.277084	0.456228	0.056*	0.5
C3	0.78212 (15)	0.250000	0.42849 (13)	0.0363 (4)	
H3	0.851834	0.250000	0.383748	0.044*	
C4	0.80412 (15)	0.250000	0.53421 (14)	0.0369 (4)	
C10	0.7192 (2)	0.250000	0.72029 (14)	0.0511 (5)	
H10A	0.743203	0.377630	0.742750	0.077*	0.5
H10B	0.785360	0.159710	0.738081	0.077*	0.5
H10C	0.641263	0.212661	0.754040	0.077*	0.5
C9	0.93866 (18)	0.250000	0.57495 (18)	0.0587 (6)	
H9A	0.956895	0.373156	0.606419	0.088*	0.5
H9B	0.996882	0.227695	0.518555	0.088*	0.5
H9C	0.948051	0.149149	0.625851	0.088*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0299 (6)	0.0696 (9)	0.0342 (7)	0.000	−0.0056 (5)	0.000
N1	0.0298 (8)	0.0746 (11)	0.0300 (8)	0.000	0.0048 (6)	0.000
C1	0.0234 (7)	0.0315 (8)	0.0294 (8)	0.000	0.0014 (6)	0.000
C2	0.0273 (8)	0.0345 (8)	0.0307 (8)	0.000	0.0029 (6)	0.000
C7	0.0261 (8)	0.0331 (8)	0.0349 (8)	0.000	−0.0018 (6)	0.000
C6	0.0310 (8)	0.0350 (8)	0.0300 (8)	0.000	0.0024 (6)	0.000
C5	0.0368 (9)	0.0364 (9)	0.0323 (8)	0.000	−0.0066 (7)	0.000
C8	0.0252 (8)	0.0433 (10)	0.0437 (10)	0.000	0.0032 (7)	0.000
C3	0.0237 (8)	0.0439 (9)	0.0414 (9)	0.000	0.0041 (7)	0.000
C4	0.0267 (8)	0.0390 (9)	0.0449 (9)	0.000	−0.0064 (7)	0.000
C10	0.0549 (12)	0.0631 (12)	0.0355 (10)	0.000	−0.0120 (8)	0.000
C9	0.0315 (10)	0.0784 (15)	0.0661 (14)	0.000	−0.0156 (9)	0.000

Geometric parameters (Å, º)

O1—C7	1.2377 (19)	C8—H8B^{i}	0.960 (10)
N1—C2	1.360 (2)	C8—H8C^{i}	0.960 (7)
N1—H1N	0.887 (15)	C3—C4	1.378 (2)
N1—H2N	0.864 (15)	C3—H3	0.9300
C1—C6	1.408 (2)	C4—C9	1.505 (2)
C1—C2	1.420 (2)	C10—H10A	0.9600
C1—C7	1.470 (2)	C10—H10B	0.9600
C2—C3	1.406 (2)	C10—H10C	0.9600
C7—C8	1.500 (2)	C10—H10A^{i}	0.960 (8)
C6—C5	1.375 (2)	C10—H10B^{i}	0.960 (17)
C6—H6	0.9300	C10—H10C^{i}	0.960 (9)
C5—C4	1.415 (2)	C9—H9A	0.9600
C5—C10	1.508 (2)	C9—H9B	0.9600
C8—H8A	0.9600	C9—H9C	0.9600
C8—H8B	0.9600	C9—H9A^{i}	0.960 (19)
C8—H8C	0.9600	C9—H9B^{i}	0.960 (10)
C8—H8A^{i}	0.960 (17)	C9—H9C^{i}	0.96 (3)
C2—N1—H1N	116.0 (14)	C5—C4—C9	120.25 (17)
C2—N1—H2N	117.4 (14)	C5—C10—H10A	109.5
H1N—N1—H2N	127 (2)	C5—C10—H10B	109.5
C6—C1—C2	117.90 (13)	H10A—C10—H10B	109.5
C6—C1—C7	120.50 (13)	C5—C10—H10C	109.5
C2—C1—C7	121.59 (14)	H10A—C10—H10C	109.5
N1—C2—C3	119.30 (14)	H10B—C10—H10C	109.5
N1—C2—C1	122.82 (14)	C5—C10—H10A^{i}	109.47 (16)
C3—C2—C1	117.88 (14)	H10A—C10—H10A^{i}	132.9
O1—C7—C1	121.51 (14)	H10B—C10—H10A^{i}	31.1
O1—C7—C8	118.58 (14)	H10C—C10—H10A^{i}	80.9
C1—C7—C8	119.91 (14)	C5—C10—H10B^{i}	109.5 (4)
C5—C6—C1	123.96 (14)	H10A—C10—H10B^{i}	31.1
C5—C6—H6	118.0	H10B—C10—H10B^{i}	80.9
C1—C6—H6	118.0	H10C—C10—H10B^{i}	132.9
C6—C5—C4	117.63 (15)	H10A^{i}—C10—H10B^{i}	109.5
C6—C5—C10	120.72 (15)	C5—C10—H10C^{i}	109.5 (2)
C4—C5—C10	121.65 (15)	H10A—C10—H10C^{i}	80.9
C7—C8—H8A	109.5	H10B—C10—H10C^{i}	132.9
C7—C8—H8B	109.5	H10C—C10—H10C^{i}	31.1
H8A—C8—H8B	109.5	H10A^{i}—C10—H10C^{i}	109.5
C7—C8—H8C	109.5	H10B^{i}—C10—H10C^{i}	109.5
H8A—C8—H8C	109.5	C4—C9—H9A	109.5
H8B—C8—H8C	109.5	C4—C9—H9B	109.5
C7—C8—H8A^{i}	109.5 (4)	H9A—C9—H9B	109.5
H8A—C8—H8A^{i}	89.1	C4—C9—H9C	109.5
H8B—C8—H8A^{i}	22.4	H9A—C9—H9C	109.5
H8C—C8—H8A^{i}	127.3	H9B—C9—H9C	109.5

C7—C8—H8B^{i}	109.5 (2)	C4—C9—H9A^{i}	109.5 (4)
H8A—C8—H8B^{i}	22.4	H9A—C9—H9A^{i}	124.4
H8B—C8—H8B^{i}	127.3	H9B—C9—H9A^{i}	92.9
H8C—C8—H8B^{i}	89.1	H9C—C9—H9A^{i}	18.4
H8A^{i}—C8—H8B^{i}	109.5	C4—C9—H9B^{i}	109.5 (2)
C7—C8—H8C^{i}	109.47 (17)	H9A—C9—H9B^{i}	92.9
H8A—C8—H8C^{i}	127.3	H9B—C9—H9B^{i}	18.4
H8B—C8—H8C^{i}	89.1	H9C—C9—H9B^{i}	124.4
H8C—C8—H8C^{i}	22.4	H9A^{i}—C9—H9B^{i}	109.5
H8A^{i}—C8—H8C^{i}	109.5	C4—C9—H9C^{i}	109.5 (6)
H8B^{i}—C8—H8C^{i}	109.5	H9A—C9—H9C^{i}	18.4
C4—C3—C2	122.87 (14)	H9B—C9—H9C^{i}	124.4
C4—C3—H3	118.6	H9C—C9—H9C^{i}	92.9
C2—C3—H3	118.6	H9A^{i}—C9—H9C^{i}	109.5
C3—C4—C5	119.75 (14)	H9B^{i}—C9—H9C^{i}	109.5
C3—C4—C9	120.00 (16)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1	0.89 (2)	1.96 (2)	2.6603 (19)	135 (2)
N1—H2*N*···O1^{ii}	0.86 (2)	2.09 (2)	2.9478 (18)	170 (2)

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