Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Amino-4,6-dimethylpyrimidine–4-hydroxybenzoic acid (1/1)

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.130
Data-to-parameter ratio = 8.0

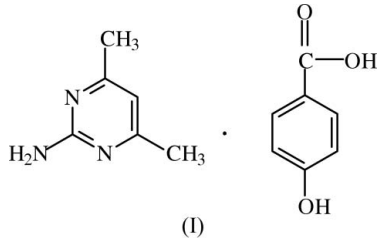
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 May 2006
Accepted 10 June 2006

In the title compound, $C_6H_9N_3{\cdot}C_7H_6O_3$, the 2-amino-4,6-dimethylpyrimidine and 4-hydroxybenzoic acid molecules link together *via* N—H···O and O—H···N hydrogen bonds to form an eight-membered $R_2^2(8)$ ring. Further hydrogen bonds and C—H···O interactions result in the formation of a three-dimensional network.

Comment

The crystal structures of various aminopyrimidine carboxylates (Hu *et al.*, 2002) and cocrystals (Chinnakali *et al.*, 1999) have been described. From our laboratory, the crystal structures of 2-amino-4,6-dimethylpyrimidinium bromide 2-amino-4,6-dimethylpyrimidine monohydrate (Panneerselvam *et al.*, 2004) and 2-amino-4,6-dimethylpyrimidine cinnamic acid (1/2) (Balasubramani *et al.*, 2005) have been reported. In this paper, the hydrogen-bonding patterns in the title compound, (I), are described.



(I)

The asymmetric unit of (I) contains a 2-amino-4,6-dimethylpyrimidine (AMPY) molecule and a 4-hydroxybenzoic (4-HBZ) acid molecule (Fig. 1). Both species are neutral, thus (I) is an adduct rather than a molecular salt. Atoms O2 and the –N2H_2 group act as hydrogen-bond donors to atoms N1 and O3, respectively, to form an eight-membered ring, which has the graph-set notation $R_2^2(8)$ (Etter, 1990; Bernstein *et al.*, 1995). This type of interaction has been observed in the crystal structures of other 2-aminopyrimidine–carboxylic acid adducts (Lynch & Jones, 2004).

The second H atom of the 2-amino group links to an O2 atom in an adjacent molecule *via* an N—H···O bond, and one of the C atoms (C11) of 4-HBZ is hydrogen bonded to O3 *via* a C—H···O interaction to form a ring having graph-set notation $R_2^3(8)$, leading to the supramolecular chain shown in Fig. 2. Hence, O3 acts as a bifurcated acceptor. The 4-HBZ hydroxy (O1) group is hydrogen bonded to pyrimidine atom N3 *via* an O—H···N interaction, to form a chain as shown in Fig. 3.

Aromatic π–π interactions between the pyrimidine ring of AMPY and the benzene ring of 4-HBZ are also observed in

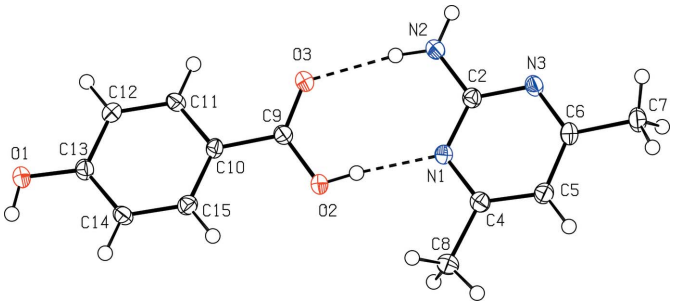


Figure 1
ORTEPII (Johnson, 1976) view of the asymmetric unit of (I), showing 50% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

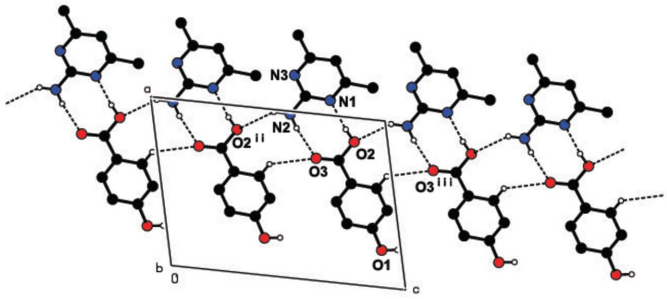


Figure 2
A view of the supramolecular chain in (I). Dashed lines indicate hydrogen bonds and H atoms not involved in hydrogen bonding have been omitted. [Symmetry codes: (ii) $x, -y, \frac{1}{2}+z$; (iii) $x, -y, z-\frac{1}{2}$.]

(I). The perpendicular separation is 3.552 Å, and the centroid-to-centroid distance is 3.660 (9) Å. The slip angle (the angle between the centroid-to-centroid vector and the normal to the plane) is 19.86°. These values are typical for aromatic π–π stacking interactions (Hunter, 1994).

Experimental

Hot methanol solutions (20 ml) of 2-amino-4,6-dimethylpyrimidine (30 mg, Aldrich) and 4-hydroxybenzoic acid (32 mg, LOBA Chemie, India) were mixed and warmed over a water bath for a few minutes. The resulting solution was allowed to cool slowly at room temperature. Crystals of (I) appeared from the mother liquor after a few days.

Crystal data

$C_6H_9N_3\cdot C_7H_6O_3$
$M_r = 261.28$
Monoclinic, Cc
$a = 9.0693\ (3)$ Å
$b = 11.1141\ (4)$ Å
$c = 12.6080\ (5)$ Å
$\beta = 102.916\ (2)°$
$V = 1238.70\ (8)$ Å^3
$Z = 4$
$D_x = 1.401$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 120\ (2)$ K
Cube, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.980$, $T_{max} = 0.980$
4983 measured reflections
1419 independent reflections
1360 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 27.5°$

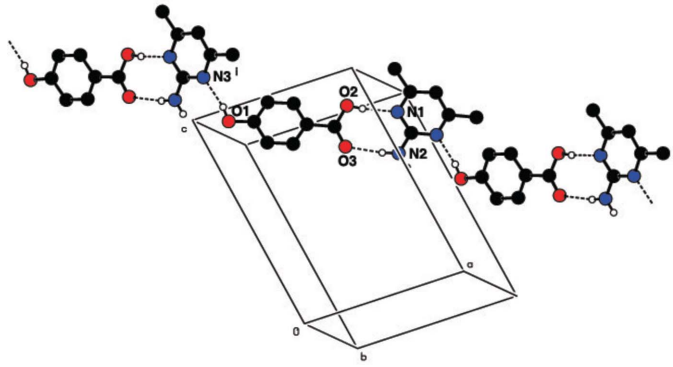


Figure 3
A view of the hydrogen-bonding patterns in (I). Dashed lines indicate hydrogen bonds and H atoms not involved in hydrogen bonding have been omitted. [Symmetry code: (i) $1+x, -y, z-\frac{1}{2}$.]

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.130$
$S = 1.34$
1419 reflections
177 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.97$ e Å^{-3}
$\Delta\rho_{min} = -0.93$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.171 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N3^{i}	0.82	1.94	2.742 (3)	167
O2—H2···N1	0.82	1.90	2.711 (3)	173
N2—H2A···O3	0.86	2.00	2.843 (3)	168
N2—H2B···O2^{ii}	0.86	2.56	3.229 (3)	135
C15—H15···O3^{iii}	0.93	2.55	3.181 (3)	126

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

In the absence of significant anomalous scattering effects, Friedel pairs were averaged. All the H atoms were positioned geometrically (C—H = 0.93–0.96 Å, N—H = 0.86 Å and O—H = 0.82 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}$(carrier).

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *PLATON*.

DL thanks the EPSRC National Crystallography Service (Southampton, England) for the X-ray data collection.

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supporting information

Acta Cryst. (2006). E**62**, o2907–o2909 [https://doi.org/10.1107/S1600536806022239]

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Crystal data

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Monoclinic, *Cc*
Hall symbol: C -2yc
$a = 9.0693$ (3) Å
$b = 11.1141$ (4) Å
$c = 12.6080$ (5) Å
$\beta = 102.916$ (2)°
$V = 1238.70$ (8) Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.401$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 2.9$–$26.0°$
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Cube, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Bruker Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
$T_{min} = 0.980$, $T_{max} = 0.980$
4983 measured reflections
1419 independent reflections
1360 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$
$h = -11 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -13 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.130$
$S = 1.34$
1419 reflections
177 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.97$ e Å^{-3}
$\Delta\rho_{min} = -0.93$ e Å^{-3}
Extinction correction: SHELXL97, $FC^*=KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Extinction coefficient: 0.171 (13)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All e.s.d.'s are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.8055 (2)	0.11658 (19)	0.07773 (16)	0.0171 (5)
N2	0.9130 (2)	0.0797 (2)	0.25815 (18)	0.0236 (6)
N3	0.6766 (2)	0.1679 (2)	0.21803 (17)	0.0187 (6)
C2	0.7952 (3)	0.1219 (2)	0.1826 (2)	0.0176 (7)
C4	0.6897 (3)	0.1626 (2)	0.0032 (2)	0.0183 (7)
C5	0.5650 (3)	0.2122 (2)	0.0327 (2)	0.0198 (7)
C6	0.5633 (3)	0.2143 (2)	0.1422 (2)	0.0183 (7)
C7	0.4343 (3)	0.2693 (2)	0.1815 (2)	0.0232 (7)
C8	0.7040 (3)	0.1597 (2)	−0.1131 (2)	0.0219 (7)
O1	1.67254 (19)	−0.16175 (18)	−0.06525 (15)	0.0243 (5)
O2	1.0396 (2)	0.03521 (19)	−0.00365 (15)	0.0224 (5)
O3	1.1621 (2)	0.0095 (2)	0.16966 (16)	0.0278 (6)
C9	1.1602 (3)	0.0057 (2)	0.0723 (2)	0.0183 (7)
C10	1.2931 (3)	−0.0348 (2)	0.0317 (2)	0.0174 (7)
C11	1.4103 (3)	−0.0928 (2)	0.1046 (2)	0.0193 (7)
C12	1.5354 (3)	−0.1350 (2)	0.0711 (2)	0.0208 (7)
C13	1.5476 (3)	−0.1186 (2)	−0.0367 (2)	0.0189 (7)
C14	1.4314 (3)	−0.0591 (2)	−0.1096 (2)	0.0193 (6)
C15	1.3049 (3)	−0.0179 (2)	−0.0759 (2)	0.0188 (7)
H2A	0.99090	0.05080	0.23860	0.0280*
H2B	0.91050	0.08170	0.32590	0.0280*
H5	0.48480	0.24330	−0.01930	0.0240*
H7A	0.36070	0.20840	0.18560	0.0350*
H7B	0.38820	0.33100	0.13170	0.0350*
H7C	0.47120	0.30370	0.25230	0.0350*
H8A	0.79730	0.19730	−0.11870	0.0330*
H8B	0.62070	0.20230	−0.15770	0.0330*
H8C	0.70320	0.07770	−0.13720	0.0330*
H1	1.66190	−0.15700	−0.13140	0.0360*
H2	0.97110	0.05580	0.02520	0.0340*
H11	1.40370	−0.10310	0.17660	0.0230*
H12	1.61190	−0.17450	0.12020	0.0250*
H14	1.43910	−0.04720	−0.18120	0.0230*
H15	1.22790	0.02100	−0.12500	0.0230*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0146 (9)	0.0201 (9)	0.0169 (10)	−0.0011 (7)	0.0043 (7)	−0.0004 (7)

N2	0.0208 (10)	0.0349 (12)	0.0161 (10)	0.0077 (9)	0.0061 (8)	0.0016 (9)
N3	0.0174 (9)	0.0214 (11)	0.0190 (10)	−0.0002 (8)	0.0075 (8)	−0.0005 (8)
C2	0.0163 (11)	0.0185 (11)	0.0186 (12)	−0.0017 (9)	0.0050 (9)	−0.0008 (9)
C4	0.0184 (11)	0.0181 (11)	0.0192 (12)	−0.0037 (9)	0.0056 (9)	−0.0012 (8)
C5	0.0168 (11)	0.0223 (12)	0.0201 (12)	0.0003 (9)	0.0038 (8)	0.0018 (9)
C6	0.0156 (11)	0.0178 (11)	0.0229 (12)	−0.0003 (9)	0.0071 (9)	0.0005 (9)
C7	0.0182 (11)	0.0271 (13)	0.0264 (13)	0.0044 (10)	0.0094 (10)	0.0015 (10)
C8	0.0228 (12)	0.0248 (12)	0.0185 (12)	0.0005 (10)	0.0055 (9)	0.0018 (9)
O1	0.0176 (9)	0.0367 (10)	0.0198 (9)	0.0058 (7)	0.0068 (7)	0.0003 (8)
O2	0.0161 (8)	0.0334 (10)	0.0181 (9)	0.0063 (7)	0.0049 (7)	−0.0015 (7)
O3	0.0190 (8)	0.0474 (12)	0.0180 (9)	0.0063 (8)	0.0062 (7)	−0.0025 (8)
C9	0.0171 (11)	0.0209 (11)	0.0174 (12)	−0.0014 (9)	0.0051 (9)	−0.0023 (9)
C10	0.0128 (11)	0.0207 (12)	0.0189 (12)	−0.0012 (9)	0.0042 (9)	−0.0020 (9)
C11	0.0191 (11)	0.0252 (12)	0.0145 (11)	−0.0013 (10)	0.0059 (9)	−0.0008 (9)
C12	0.0163 (11)	0.0264 (12)	0.0185 (12)	0.0023 (9)	0.0016 (9)	0.0004 (10)
C13	0.0162 (12)	0.0212 (12)	0.0203 (12)	−0.0008 (9)	0.0060 (9)	−0.0011 (9)
C14	0.0202 (11)	0.0234 (11)	0.0154 (11)	−0.0017 (9)	0.0065 (9)	−0.0019 (9)
C15	0.0167 (11)	0.0205 (11)	0.0184 (13)	−0.0013 (9)	0.0025 (9)	0.0002 (9)

Geometric parameters (Å, º)

O1—C13	1.351 (3)	C7—H7A	0.9601
O2—C9	1.324 (3)	C7—H7C	0.9599
O3—C9	1.225 (3)	C7—H7B	0.9602
O1—H1	0.8196	C8—H8B	0.9598
O2—H2	0.8195	C8—H8C	0.9602
N1—C4	1.344 (3)	C8—H8A	0.9604
N1—C2	1.347 (3)	C9—C10	1.481 (4)
N2—C2	1.347 (3)	C10—C15	1.397 (4)
N3—C2	1.354 (3)	C10—C11	1.398 (4)
N3—C6	1.340 (3)	C11—C12	1.378 (4)
N2—H2B	0.8598	C12—C13	1.400 (4)
N2—H2A	0.8608	C13—C14	1.400 (4)
C4—C5	1.382 (4)	C14—C15	1.387 (4)
C4—C8	1.501 (4)	C11—H11	0.9301
C5—C6	1.384 (4)	C12—H12	0.9302
C6—C7	1.499 (4)	C14—H14	0.9303
C5—H5	0.9301	C15—H15	0.9298
C13—O1—H1	109.46	H8A—C8—H8B	109.47
C9—O2—H2	109.50	C4—C8—H8B	109.46
C2—N1—C4	117.0 (2)	C4—C8—H8C	109.47
C2—N3—C6	116.7 (2)	H8B—C8—H8C	109.48
C2—N2—H2B	120.05	H8A—C8—H8C	109.52
C2—N2—H2A	119.97	O2—C9—C10	115.5 (2)
H2A—N2—H2B	119.98	O3—C9—C10	121.8 (2)
N1—C2—N3	125.1 (2)	O2—C9—O3	122.7 (2)
N1—C2—N2	117.4 (2)	C11—C10—C15	119.2 (2)

N2—C2—N3	117.5 (2)	C9—C10—C11	118.1 (2)
N1—C4—C5	121.5 (2)	C9—C10—C15	122.7 (2)
N1—C4—C8	116.8 (2)	C10—C11—C12	120.8 (2)
C5—C4—C8	121.7 (2)	C11—C12—C13	120.2 (2)
C4—C5—C6	117.9 (2)	O1—C13—C14	123.1 (2)
N3—C6—C7	116.8 (2)	O1—C13—C12	117.7 (2)
N3—C6—C5	121.8 (2)	C12—C13—C14	119.2 (2)
C5—C6—C7	121.3 (2)	C13—C14—C15	120.5 (2)
C6—C5—H5	121.03	C10—C15—C14	120.1 (2)
C4—C5—H5	121.11	C10—C11—H11	119.55
C6—C7—H7C	109.52	C12—C11—H11	119.61
C6—C7—H7A	109.49	C11—C12—H12	119.95
C6—C7—H7B	109.45	C13—C12—H12	119.90
H7B—C7—H7C	109.51	C13—C14—H14	119.70
H7A—C7—H7B	109.41	C15—C14—H14	119.79
H7A—C7—H7C	109.44	C10—C15—H15	119.99
C4—C8—H8A	109.42	C14—C15—H15	119.94
C2—N1—C4—C8	178.2 (2)	O2—C9—C10—C11	−165.6 (2)
C4—N1—C2—N2	−177.8 (2)	O2—C9—C10—C15	14.0 (3)
C4—N1—C2—N3	1.2 (4)	O3—C9—C10—C11	13.3 (4)
C2—N1—C4—C5	−0.6 (3)	C15—C10—C11—C12	−1.1 (4)
C6—N3—C2—N2	177.2 (2)	C9—C10—C15—C14	−179.2 (2)
C2—N3—C6—C5	1.9 (3)	C9—C10—C11—C12	178.5 (2)
C2—N3—C6—C7	−177.9 (2)	C11—C10—C15—C14	0.4 (3)
C6—N3—C2—N1	−1.9 (4)	C10—C11—C12—C13	0.9 (4)
N1—C4—C5—C6	0.6 (4)	C11—C12—C13—O1	−180.0 (2)
C8—C4—C5—C6	−178.1 (2)	C11—C12—C13—C14	−0.1 (4)
C4—C5—C6—C7	178.4 (2)	C12—C13—C14—C15	−0.6 (4)
C4—C5—C6—N3	−1.3 (4)	O1—C13—C14—C15	179.3 (2)
O3—C9—C10—C15	−167.1 (2)	C13—C14—C15—C10	0.5 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3^{i}	0.82	1.94	2.742 (3)	167
O2—H2···N1	0.82	1.90	2.711 (3)	173
N2—H2*A*···O3	0.86	2.00	2.843 (3)	168
N2—H2*B*···O2^{ii}	0.86	2.56	3.229 (3)	135
C15—H15···O3^{iii}	0.93	2.55	3.181 (3)	126

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