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Phthalic acid–4,6-dimethylpyrimidin-2-amine (1/1)

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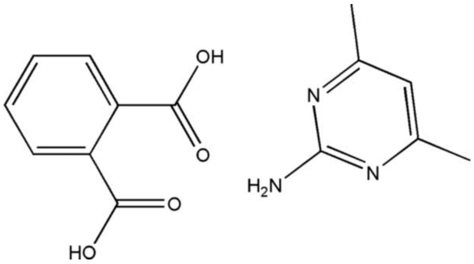
Key indicators: single-crystal X-ray study; *T* = 294 K; mean σ(C–C) = 0.004 Å; *R* factor = 0.047; *wR* factor = 0.135; data-to-parameter ratio = 13.9.

In the crystal structure of the title compound, $C_8H_6O_4 \cdot C_6H_9N_3$, the two molecules interact *via* N—H···O and O—H···N hydrogen bonds, resulting in centrosymmetric clusters.

Related literature

For related literature, see: Ren *et al.* (2005).



Experimental

Crystal data

$C_8H_6O_4 \cdot C_6H_9N_3$
M_r = 289.29
Monoclinic, $P2_1/n$
a = 14.472 (7) Å
b = 7.083 (4) Å
c = 14.563 (8) Å
β = 112.108 (8)°
V = 1383.1 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.976, T_{max} = 0.982
7676 measured reflections
2801 independent reflections
1550 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.135
S = 1.00
2801 reflections
202 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.81	2.613 (3)	168
O4—H4···N2^{i}	0.82	1.84	2.656 (3)	172
N3—H3*A*···O3^{i}	0.881 (9)	2.027 (9)	2.890 (3)	166.2 (18)
N3—H3*B*···O2	0.890 (19)	1.984 (19)	2.865 (3)	170.7 (19)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997; 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: HB2370).

References

Bruker (1997). *SADABS* (Version 2.01), *SMART* (Version 5.044), *SAINT* (Version 5.01) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Ren, Y.-H., Song, J.-R., Huang, J., Sun, X.-H. & Hu, H.-M. (2005). *Acta Cryst.* E**61**, o4013–o4015.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

Acta Cryst. (2007). E**63**, o2673 [**doi:10.1107/S1600536807019265**]

Phthalic acid-4,6-dimethylpyrimidin-2-amine (1/1)

J. Li, Z.-P. Liang and H.-M. Guo

Comment

In this paper, the structure of the title compound, (I), a 1:1 adduct of phthalic acid and 4,6-dimethylpyrimidin-2-amine is reported. (Fig. 1). The geometrical parameters of 4,6-dimethylpyrimidin-2-amine molecule in (I) are similar to those in methyl *N*-[4-(4,6-dimethylpyrimidin-2-ylamino)thiocarbonyl]carbamate (Ren *et al.*, 2005). The heterocycle is essentially planar, within 0.014 (4) Å, and the dihedral angle between this plane and the benzene ring of the phthalic acid molecule is 21.3 (2) °. The crystal structure is stabilized by N—H···O and O—H···N hydrogen bonds (Fig. 2 and Table 1) to result in tetrameric associations of molecules, generated by inversion.

Experimental

A mixture of phthalic acid (0.01 mol) and 4,6-dimethylpyrimidin-2-amine (0.01 mol) in ethanol (20 ml) was refluxed for 0.5 h, and cooled. The solution was kept at room temperature for 12 d. Natural evaporation gave yellow blocks of (I) suitable for X-ray analysis.

Refinement

The N-bound H atoms were located in a difference map and their positions and U_{iso} values were freely refined.

The other H atoms were positioned geometrically (C—H = 0.93 Å, O—H = 0.82 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C, O)$.

Figures

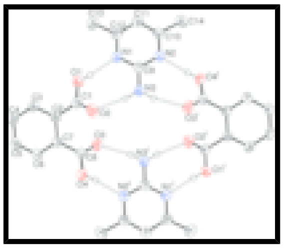

Fig. 1. A tetrameric cluster in (I), drawn with 30% probability ellipsoids (arbitrary spheres for the H atoms; C-bound H atoms omitted for clarity). Hydrogen bonds are indicated by dashed lines. Symmetry code: (i) -x, -y, 1-z.

Phthalic acid–4,6-dimethylpyrimidin-2-amine (1/1)

Crystal data

$C_8H_6O_4 \cdot C_6H_9N_3$	$F_{000} = 608$
$M_r = 289.29$	$D_x = 1.389$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo *K*α radiation

	$\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1525 reflections
$a = 14.472$ (7) Å	$\theta = 2.5–24.9°$
$b = 7.083$ (4) Å	$\mu = 0.10$ mm^{-1}
$c = 14.563$ (8) Å	$T = 294$ (2) K
$\beta = 112.108$ (8)°	Block, yellow
$V = 1383.1$ (12) Å^3	0.24 × 0.22 × 0.18 mm
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	2801 independent reflections
Radiation source: fine-focus sealed tube	1550 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.047$
$T = 294(2)$ K	$\theta_{\text{max}} = 26.4°$
ω scans	$\theta_{\text{min}} = 1.7°$
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -18 \rightarrow 17$
$T_{\text{min}} = 0.976$, $T_{\text{max}} = 0.982$	$k = -7 \rightarrow 8$
7676 measured reflections	$l = -18 \rightarrow 15$

Refinement

Refinement on F^2	Hydrogen site location: difmap and geom
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.0663P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.135$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.19$ e Å^{-3}
2801 reflections	$\Delta\rho_{\text{min}} = -0.22$ e Å^{-3}
202 parameters	Extinction correction: none
3 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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.

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}
O1	0.32059 (11)	0.0730 (2)	0.57966 (11)	0.0495 (5)
H1	0.2975	0.1014	0.6213	0.074*
O2	0.17859 (12)	0.1937 (2)	0.47642 (12)	0.0536 (5)
O3	0.10073 (11)	−0.1554 (3)	0.37463 (12)	0.0545 (5)
O4	0.04800 (11)	−0.0115 (2)	0.22987 (12)	0.0491 (5)
H4	−0.0064	−0.0495	0.2275	0.074*
N1	0.26384 (13)	0.1281 (3)	0.72801 (14)	0.0374 (5)
N2	0.13632 (13)	0.1101 (2)	0.79262 (13)	0.0382 (5)
N3	0.09888 (15)	0.1281 (3)	0.62636 (15)	0.0518 (6)
C1	0.25648 (17)	0.1130 (3)	0.49214 (17)	0.0380 (5)
C2	0.28825 (15)	0.0563 (3)	0.41096 (16)	0.0343 (5)
C3	0.38602 (16)	0.0813 (3)	0.42031 (17)	0.0408 (6)
H3	0.4329	0.1256	0.4797	0.049*
C4	0.41430 (17)	0.0416 (3)	0.34297 (18)	0.0448 (6)
H4A	0.4804	0.0580	0.3501	0.054*
C5	0.34617 (17)	−0.0222 (3)	0.25536 (19)	0.0452 (6)
H5	0.3658	−0.0489	0.2029	0.054*
C6	0.24853 (17)	−0.0469 (3)	0.24448 (18)	0.0408 (6)
H6	0.2020	−0.0879	0.1841	0.049*
C7	0.21890 (15)	−0.0116 (3)	0.32215 (16)	0.0344 (5)
C8	0.11693 (16)	−0.0646 (3)	0.31263 (17)	0.0377 (5)
C9	0.16713 (16)	0.1230 (3)	0.71695 (17)	0.0370 (5)
C10	0.20528 (16)	0.1091 (3)	0.88369 (16)	0.0377 (5)
C11	0.30461 (17)	0.1199 (3)	0.89960 (17)	0.0444 (6)
H11	0.3525	0.1231	0.9637	0.053*
C12	0.33204 (16)	0.1260 (3)	0.81947 (17)	0.0393 (6)
C13	0.43803 (16)	0.1279 (4)	0.8298 (2)	0.0547 (7)
H13A	0.4579	0.0026	0.8200	0.082*
H13B	0.4790	0.1710	0.8950	0.082*
H13C	0.4458	0.2112	0.7812	0.082*
C14	0.16923 (18)	0.0961 (4)	0.96640 (17)	0.0509 (7)
H14A	0.1267	0.2017	0.9636	0.076*
H14B	0.2252	0.0973	1.0284	0.076*
H14C	0.1325	−0.0191	0.9606	0.076*
H3A	0.0349 (8)	0.138 (3)	0.6152 (15)	0.050 (7)*
H3B	0.1167 (14)	0.147 (3)	0.5749 (12)	0.051 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0374 (10)	0.0815 (13)	0.0298 (10)	0.0072 (8)	0.0131 (8)	0.0016 (8)
O2	0.0453 (10)	0.0756 (12)	0.0422 (11)	0.0223 (9)	0.0192 (9)	0.0043 (8)

O3	0.0366 (10)	0.0792 (13)	0.0503 (12)	−0.0038 (8)	0.0192 (9)	0.0189 (9)
O4	0.0318 (9)	0.0744 (12)	0.0411 (10)	−0.0030 (8)	0.0139 (9)	0.0074 (8)
N1	0.0288 (10)	0.0498 (12)	0.0334 (11)	−0.0021 (8)	0.0114 (9)	−0.0012 (8)
N2	0.0342 (11)	0.0511 (12)	0.0306 (11)	−0.0025 (9)	0.0135 (9)	−0.0026 (8)
N3	0.0286 (12)	0.0970 (17)	0.0292 (12)	0.0010 (11)	0.0104 (10)	0.0023 (11)
C1	0.0330 (13)	0.0462 (14)	0.0366 (14)	−0.0003 (11)	0.0153 (12)	0.0009 (11)
C2	0.0329 (12)	0.0393 (13)	0.0318 (13)	0.0028 (10)	0.0133 (11)	0.0045 (9)
C3	0.0279 (12)	0.0564 (16)	0.0369 (14)	0.0034 (11)	0.0110 (11)	0.0051 (11)
C4	0.0301 (13)	0.0609 (16)	0.0474 (16)	0.0045 (11)	0.0192 (13)	0.0072 (12)
C5	0.0435 (14)	0.0560 (16)	0.0457 (16)	0.0021 (12)	0.0279 (13)	−0.0014 (12)
C6	0.0398 (14)	0.0502 (15)	0.0342 (13)	−0.0019 (11)	0.0161 (11)	−0.0051 (10)
C7	0.0314 (12)	0.0396 (13)	0.0352 (13)	0.0012 (9)	0.0159 (11)	0.0026 (10)
C8	0.0328 (13)	0.0463 (14)	0.0347 (14)	0.0008 (10)	0.0135 (12)	−0.0020 (11)
C9	0.0331 (13)	0.0443 (14)	0.0339 (14)	−0.0036 (10)	0.0127 (12)	−0.0035 (10)
C10	0.0403 (14)	0.0416 (14)	0.0312 (13)	−0.0020 (10)	0.0132 (12)	−0.0024 (10)
C11	0.0368 (14)	0.0584 (16)	0.0314 (14)	−0.0009 (11)	0.0053 (11)	−0.0030 (11)
C12	0.0324 (12)	0.0424 (14)	0.0401 (15)	−0.0038 (10)	0.0104 (12)	−0.0051 (10)
C13	0.0347 (14)	0.0735 (19)	0.0535 (18)	−0.0026 (12)	0.0139 (13)	0.0005 (13)
C14	0.0509 (16)	0.0693 (18)	0.0356 (15)	0.0022 (13)	0.0196 (13)	0.0005 (12)

Geometric parameters (Å, °)

O1—C1	1.294 (3)	C4—C5	1.363 (3)
O1—H1	0.8200	C4—H4A	0.9300
O2—C1	1.206 (2)	C5—C6	1.373 (3)
O3—C8	1.201 (3)	C5—H5	0.9300
O4—C8	1.298 (3)	C6—C7	1.375 (3)
O4—H4	0.8200	C6—H6	0.9300
N1—C12	1.326 (3)	C7—C8	1.478 (3)
N1—C9	1.348 (3)	C10—C11	1.369 (3)
N2—C10	1.326 (3)	C10—C14	1.485 (3)
N2—C9	1.338 (3)	C11—C12	1.367 (3)
N3—C9	1.318 (3)	C11—H11	0.9300
N3—H3A	0.881 (9)	C12—C13	1.484 (3)
N3—H3B	0.890 (19)	C13—H13A	0.9600
C1—C2	1.476 (3)	C13—H13B	0.9600
C2—C3	1.381 (3)	C13—H13C	0.9600
C2—C7	1.391 (3)	C14—H14A	0.9600
C3—C4	1.365 (3)	C14—H14B	0.9600
C3—H3	0.9300	C14—H14C	0.9600
C1—O1—H1	109.5	O3—C8—O4	123.6 (2)
C8—O4—H4	109.5	O3—C8—C7	122.4 (2)
C12—N1—C9	117.81 (19)	O4—C8—C7	113.88 (19)
C10—N2—C9	117.72 (19)	N3—C9—N2	118.0 (2)
C9—N3—H3A	121.8 (13)	N3—C9—N1	118.2 (2)
C9—N3—H3B	120.1 (13)	N2—C9—N1	123.8 (2)
H3A—N3—H3B	116.8 (15)	N2—C10—C11	121.1 (2)
O2—C1—O1	124.3 (2)	N2—C10—C14	116.7 (2)
O2—C1—C2	121.8 (2)	C11—C10—C14	122.2 (2)

O1—C1—C2	113.89 (19)	C12—C11—C10	118.7 (2)
C3—C2—C7	119.3 (2)	C12—C11—H11	120.6
C3—C2—C1	120.2 (2)	C10—C11—H11	120.6
C7—C2—C1	120.37 (19)	N1—C12—C11	120.8 (2)
C4—C3—C2	120.4 (2)	N1—C12—C13	116.9 (2)
C4—C3—H3	119.8	C11—C12—C13	122.3 (2)
C2—C3—H3	119.8	C12—C13—H13A	109.5
C5—C4—C3	120.3 (2)	C12—C13—H13B	109.5
C5—C4—H4A	119.8	H13A—C13—H13B	109.5
C3—C4—H4A	119.8	C12—C13—H13C	109.5
C4—C5—C6	120.1 (2)	H13A—C13—H13C	109.5
C4—C5—H5	120.0	H13B—C13—H13C	109.5
C6—C5—H5	120.0	C10—C14—H14A	109.5
C5—C6—C7	120.5 (2)	C10—C14—H14B	109.5
C5—C6—H6	119.7	H14A—C14—H14B	109.5
C7—C6—H6	119.7	C10—C14—H14C	109.5
C6—C7—C2	119.2 (2)	H14A—C14—H14C	109.5
C6—C7—C8	119.1 (2)	H14B—C14—H14C	109.5
C2—C7—C8	121.39 (19)		

O2—C1—C2—C3	137.6 (2)	C2—C7—C8—O3	−44.3 (3)
O1—C1—C2—C3	−40.5 (3)	C6—C7—C8—O4	−47.6 (3)
O2—C1—C2—C7	−38.4 (3)	C2—C7—C8—O4	138.6 (2)
O1—C1—C2—C7	143.5 (2)	C10—N2—C9—N3	−178.6 (2)
C7—C2—C3—C4	0.6 (3)	C10—N2—C9—N1	2.5 (3)
C1—C2—C3—C4	−175.4 (2)	C12—N1—C9—N3	178.9 (2)
C2—C3—C4—C5	0.4 (4)	C12—N1—C9—N2	−2.1 (3)
C3—C4—C5—C6	−0.1 (4)	C9—N2—C10—C11	−0.3 (3)
C4—C5—C6—C7	−1.3 (3)	C9—N2—C10—C14	179.6 (2)
C5—C6—C7—C2	2.4 (3)	N2—C10—C11—C12	−2.0 (3)
C5—C6—C7—C8	−171.6 (2)	C14—C10—C11—C12	178.1 (2)
C3—C2—C7—C6	−2.0 (3)	C9—N1—C12—C11	−0.4 (3)
C1—C2—C7—C6	174.0 (2)	C9—N1—C12—C13	178.8 (2)
C3—C2—C7—C8	171.8 (2)	C10—C11—C12—N1	2.3 (3)
C1—C2—C7—C8	−12.2 (3)	C10—C11—C12—C13	−176.8 (2)
C6—C7—C8—O3	129.6 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.81	2.613 (3)	168
O4—H4···N2^{i}	0.82	1.84	2.656 (3)	172
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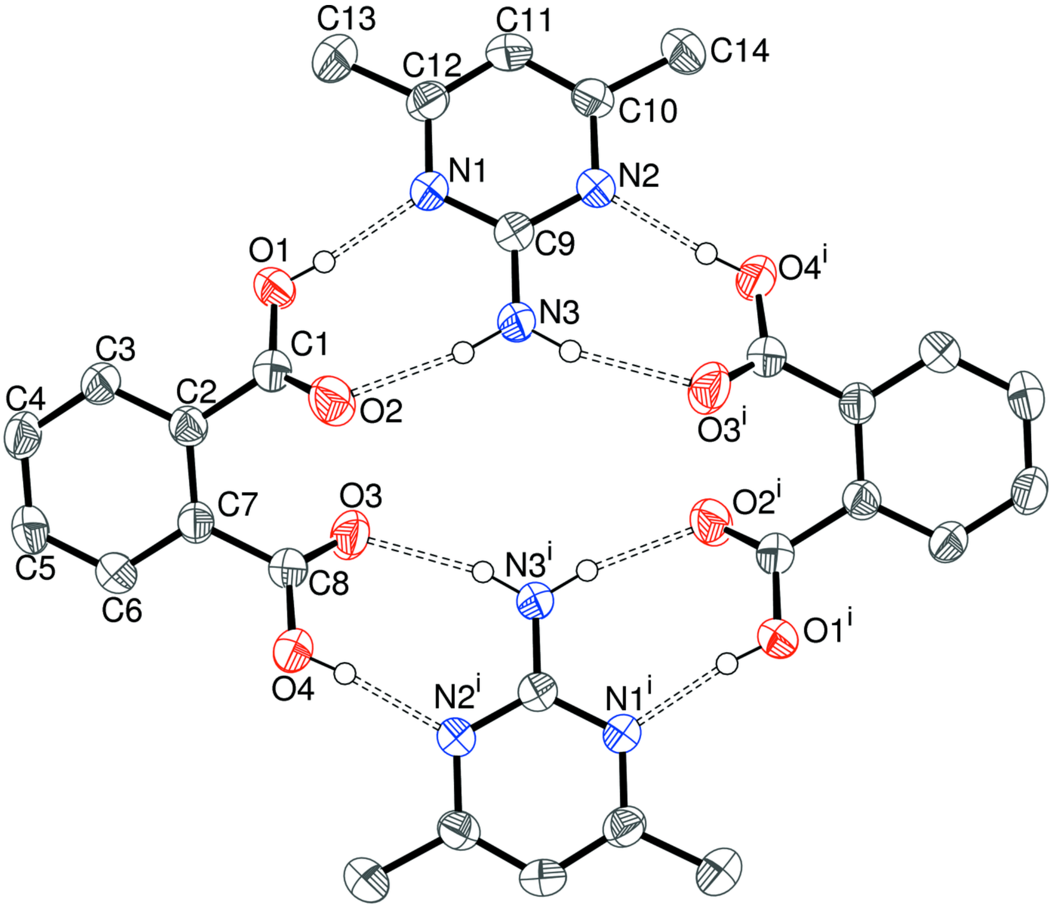
N3—H3B···O2	0.890 (19)	1.984 (19)	2.865 (3)	170.7 (19)

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

Fig. 1


C13
C11
C14
C12
C10
N1
N2
O1
C9
O4^i
N3
C3
C1
C4
C2
O2
O3^i
C7
O3
O2^i
C5
N3^i
C8
C6
O1^i
O4
N2^i
N1^i