N,N'-Bis(pyrimidin-2-yl)terephthalamide dihydrate[1]

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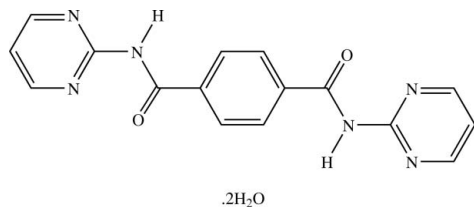Correspondence e-mail: hkfun@usm.my

The organic molecule of the title compound, $C_{16}H_{12}N_6O_2$·$2H_2O$, lies across a crystallographic inversion centre. The dihedral angle between the pyrimidine and benzene rings is 80.78 (6)°. The two pyrimidine rings are parallel by virtue of the centre of symmetry. The pyrimidine and benzene rings form dihedral angles of 41.41 (7) and 40.26 (7)°, respectively, with the amide plane. The molecules are linked by N—H···N and C—H···N hydrogen bonds into a two-dimensional network parallel to the $(1\bar{1}1)$ plane. O—H···O and C—H···O hydrogen bonds involving the water molecules link the adjacent layers into a three-dimensional network. In addition, a C—H···π interaction involving the benzene ring is observed.

Related literature

For bond-length data, see: Allen et al. (1987). For related literature on supramolecular chemistry, see: Desiraju (1989); Lehn (1995). For related structures, see: Goswami, Jana, Das et al. (2007); Goswami, Jana, Hazra et al. (2007). For related literature on the coordination chemistry and applications of aminopyrimidine derivatives, see: Aakeroy et al. (2006); Etter (1990); Fun et al. (2006); Gallagher et al. (2004); Goswami & Mahapatra (1999); Smith et al. (1998); Wang et al. (2006).

.2H$_2$O

Experimental

Crystal data

$C_{16}H_{12}N_6O_2$·$2H_2O$	γ = 82.333 (1)°
M_r = 356.35	V = 377.72 (1) Å^3
Triclinic, $P\bar{1}$	Z = 1
a = 5.0733 (1) Å	Mo $K\alpha$ radiation
b = 8.3233 (1) Å	μ = 0.12 mm^{-1}
c = 9.9622 (2) Å	T = 100.0 (1) K
α = 68.220 (1)°	0.33 × 0.22 × 0.08 mm
β = 75.441 (1)°	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	12751 measured reflections
	2202 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	1982 reflections with $I > 2\sigma(I)$
T_{min} = 0.963, T_{max} = 0.991	R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044	3 restraints
$wR(F^2)$ = 0.106	All H-atom parameters refined
S = 1.12	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
2202 reflections	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
154 parameters	

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

$Cg1$ is the centroid of the benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H2WA···O1W^i	0.80 (4)	1.99 (4)	2.791 (2)	171 (6)
O1W—H1W···O1	0.81 (2)	1.98 (2)	2.785 (2)	177 (3)
O1W—H2WB···O1W^{ii}	0.80 (6)	2.04 (5)	2.795 (2)	156 (6)
N3—H1N3···N1^{iii}	0.89 (2)	2.14 (2)	3.017 (2)	169 (2)
C1—H1···N2^{iv}	0.98 (2)	2.61 (2)	3.206 (2)	119 (1)
C2—H2···O1W^{iv}	0.95 (2)	2.58 (2)	3.508 (2)	164 (1)
C3—H3···$Cg1^v$	0.97 (2)	2.98 (2)	3.914 (2)	164 (2)
C3—H3···$Cg1^{iii}$	0.97 (2)	2.98 (2)	3.914 (2)	164 (2)

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

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); program(s) used to solve structure: SHELXTL (Sheldrick, 1998); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2534).

organic compounds

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supporting information

N,*N*′-Bis(pyrimidin-2-yl)terephthalamide dihydrate

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S1. Comment

Substituted 2-aminopyrimidines are very important compounds in molecular recognition and supramolecular chemistry (Desiraju, 1989; Lehn, 1995) for the presence of nice donor-acceptor arrays (Aakeroy *et al.*, 2006; Etter, 1990; Fun *et al.*, 2006; Gallagher, *et al.*, 2004; Goswami & Mahapatra, 1999). The donor-acceptor arrangement of the title molecule differs from the 2-aminopyrimidine based compounds (Goswami, Jana, Das *et al.*, 2007; Goswami, Jana, Hazra *et al.*, 2007). These types of compounds are also important for the metal coordination and related studies (Smith *et al.*, 1998; Wang *et al.*, 2006). The coordination chemistry of the title compound is under investigations.

Molecules of the title compound lie across a crystallographic inversion centre (Fig. 1). All bond lengths and angles have normal values (Allen *et al.*, 1987). The *N*,*N*-di-pyrimidin-2-yl-terepthalamide molecule has a staggered conformation. The orientation of the pyrimidine ring (C1–C4/N1/N2) with respect to the benzene ring (C6–C8/C6A–C8A) can be described by the dihedral angle formed by these planes of 80.78 (6)° and the torsion angle C4–N3–C5–C6 of -169.50 (11)°. The two pyrimidine rings are parallel by virtue of the centre of symmetry. The pyrimidine and benzene rings form dihedral angles of 41.41 (7)° and 40.26 (7)°, respectively, with the amide plane.

The structure shows O—H···O and C—H···O hydrogen bond between water and *N*,*N*-di-pyrimidin-2-yl-terepthalamide molecule. In the crystal packing (Fig. 2), the molecules are linked by N—H···N and C—H···N hydrogen bonds into a two-dimensional network parallel to the (1 $\bar{1}$ 1) plane. The O—H···O and C—H···O hydrogen bonds (Table 1) involving the water molecules link the adjacent layers into a three-dimensional network. The crystal structure is further stablilized by C—H···π interactions involving the benzene ring (centroid *Cg*1).

S2. Experimental

A solution of terepthaloyl chloride (203 mg, 1 mmol), in dry CH_2Cl_2 (20 ml) was put in a round bottle flask under nitrogen atmosphere. 2-Aminopyrimidine (190 mg, 2 mmol) containing triethylamine (0.55 ml) in dry CH_2Cl_2 (15 ml) was added dropwise. The reaction mixture was stirred at room temperature for 10 h. The reaction mixture was dried, washed with sodium bicarbonate solution, and then extracted with CH_2Cl_2 (4 × 20 ml). The crude mixture was purified by column chromatography (silica gel, 100–200 mesh) using 20% ethyl acetate-petroleum ether solution as eluent to afford a white solid compound (188 mg, 65%). Single crystals of the title compound were grown by slow evaporation of a $CHCl_3$—CH_3OH (3:1 *v*/*v*) solution (m.p. 457–459 K).

S3. Refinement

All H atoms were located from the difference map and isotropically refined. One of the water hydrogen atoms is disordered over two positions with occupancies each 0.50. The O—H distances were restrained to be equal within 0.03 Å.
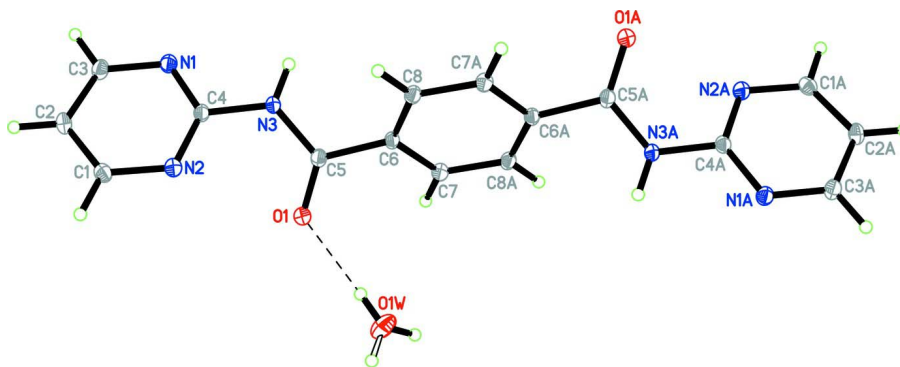
Figure 1

The molecular structure of the title compound, showing 60% probability displacement ellipsoids and the atomic numbering. One of the H atoms of the water molecule is disordered over two positions. The dashed line indicates a hydrogen bond. Atoms labelled with the suffix A are generated by the symmetry operation (-*x*, 1 - *y*, 1 - *z*).
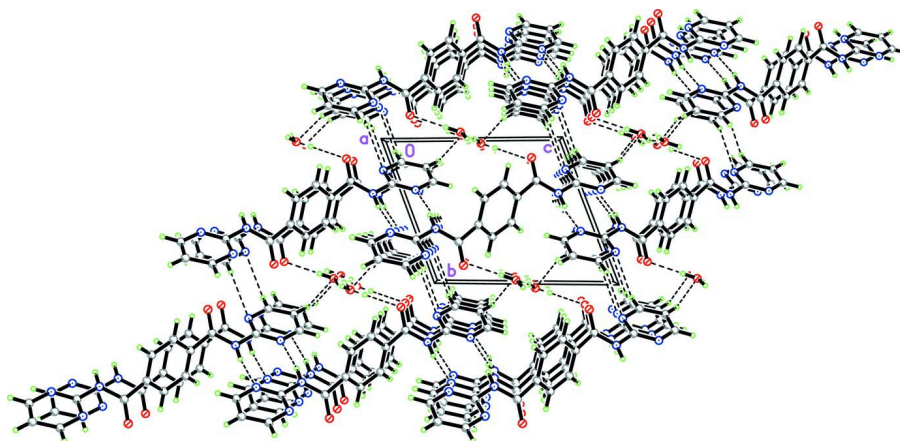
Figure 2

The crystal packing of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_{16}H_{12}N_6O_2 \cdot 2H_2O$	$Z = 1$
$M_r = 356.35$	$F(000) = 186$
Triclinic, $P\overline{1}$	$D_x = 1.567$ Mg m^{-3}
Hall symbol: -P 1	Melting point = 457–459 K
$a = 5.0733$ (1) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 8.3233$ (1) Å	Cell parameters from 2202 reflections
$c = 9.9622$ (2) Å	$\theta = 2.3$–30.0°
$\alpha = 68.220$ (1)°	$\mu = 0.12$ mm^{-1}
$\beta = 75.441$ (1)°	$T = 100$ K
$\gamma = 82.333$ (1)°	Block, colourless
$V = 377.72$ (1) Å^3	0.33 × 0.22 × 0.08 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	12751 measured reflections
Radiation source: fine-focus sealed tube	2202 independent reflections
Graphite monochromator	1982 reflections with $I > 2\sigma(I)$
Detector resolution: 8.33 pixels mm^{-1}	$R_{int} = 0.035$
ω scans	$\theta_{max} = 30.0°$, $\theta_{min} = 2.3°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -7 \rightarrow 7$
	$k = -11 \rightarrow 11$
$T_{min} = 0.963$, $T_{max} = 0.991$	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.044$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.106$	
$S = 1.12$	All H-atom parameters refined
2202 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 0.2638P]$ where $P = (F_o^2 + 2F_c^2)/3$
154 parameters	
3 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.39$ e Å^{-3}
	$\Delta\rho_{min} = -0.26$ e Å^{-3}

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.40513 (19)	0.85138 (12)	0.21674 (10)	0.0152 (2)	
N1	0.7704 (2)	0.64103 (14)	−0.09155 (12)	0.0131 (2)	
N2	0.9113 (2)	0.77862 (14)	0.05021 (12)	0.0134 (2)	
N3	0.5215 (2)	0.61544 (14)	0.14450 (12)	0.0124 (2)	
H1N3	0.441 (4)	0.530 (2)	0.1395 (19)	0.013 (4)*	
C1	1.1248 (3)	0.83918 (16)	−0.06205 (14)	0.0135 (2)	
H1	1.248 (4)	0.908 (2)	−0.0464 (19)	0.016 (4)*	
C2	1.1741 (3)	0.80284 (17)	−0.19128 (14)	0.0144 (2)	
H2	1.328 (4)	0.846 (2)	−0.270 (2)	0.018 (4)*	
C3	0.9898 (3)	0.70138 (17)	−0.20052 (14)	0.0144 (2)	
H3	1.021 (4)	0.669 (2)	−0.287 (2)	0.015 (4)*	
C4	0.7440 (2)	0.68481 (15)	0.02820 (13)	0.0113 (2)	
C5	0.3775 (2)	0.69938 (16)	0.23761 (13)	0.0121 (2)	
C6	0.1803 (2)	0.59197 (16)	0.37078 (13)	0.0114 (2)	
C7	−0.0689 (3)	0.67033 (16)	0.41794 (14)	0.0131 (2)	

H7	−0.118 (4)	0.789 (2)	0.3599 (19)	0.015 (4)*	
C8	0.2492 (2)	0.42173 (16)	0.45293 (14)	0.0126 (2)	
H8	0.419 (4)	0.368 (2)	0.4211 (19)	0.014 (4)*	
O1W	0.2790 (3)	0.96683 (17)	0.45548 (13)	0.0296 (3)	
H1W	0.309 (5)	0.934 (3)	0.386 (2)	0.034 (6)*	
H2WA	0.123 (6)	0.994 (7)	0.484 (6)	0.050 (15)*	0.50
H2WB	0.380 (10)	1.013 (7)	0.479 (7)	0.064 (18)*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0159 (4)	0.0143 (4)	0.0149 (4)	−0.0037 (3)	0.0011 (3)	−0.0063 (3)
N1	0.0123 (5)	0.0147 (5)	0.0122 (5)	−0.0017 (4)	−0.0013 (4)	−0.0054 (4)
N2	0.0125 (5)	0.0142 (5)	0.0132 (5)	−0.0022 (4)	−0.0009 (4)	−0.0053 (4)
N3	0.0117 (5)	0.0136 (5)	0.0116 (5)	−0.0040 (4)	0.0014 (4)	−0.0057 (4)
C1	0.0118 (5)	0.0128 (5)	0.0151 (6)	−0.0018 (4)	−0.0026 (4)	−0.0040 (4)
C2	0.0110 (5)	0.0168 (6)	0.0128 (5)	−0.0028 (4)	0.0002 (4)	−0.0035 (4)
C3	0.0134 (5)	0.0179 (6)	0.0116 (5)	−0.0016 (4)	−0.0009 (4)	−0.0057 (5)
C4	0.0104 (5)	0.0112 (5)	0.0111 (5)	−0.0006 (4)	−0.0008 (4)	−0.0033 (4)
C5	0.0108 (5)	0.0148 (5)	0.0105 (5)	−0.0018 (4)	−0.0013 (4)	−0.0047 (4)
C6	0.0106 (5)	0.0145 (5)	0.0098 (5)	−0.0035 (4)	−0.0006 (4)	−0.0052 (4)
C7	0.0127 (5)	0.0140 (5)	0.0123 (5)	−0.0012 (4)	−0.0014 (4)	−0.0049 (4)
C8	0.0104 (5)	0.0154 (5)	0.0124 (5)	−0.0014 (4)	−0.0006 (4)	−0.0064 (4)
O1W	0.0388 (7)	0.0353 (7)	0.0186 (5)	−0.0039 (5)	−0.0004 (5)	−0.0170 (5)

Geometric parameters (Å, º)

O1—C5	1.2252 (15)	C3—H3	0.970 (18)
N1—C4	1.3429 (15)	C5—C6	1.5006 (16)
N1—C3	1.3441 (16)	C6—C8	1.3967 (17)
N2—C4	1.3325 (16)	C6—C7	1.3976 (17)
N2—C1	1.3403 (16)	C7—C8[i]	1.3937 (17)
N3—C5	1.3734 (15)	C7—H7	0.980 (18)
N3—C4	1.4037 (15)	C8—C7[i]	1.3937 (17)
N3—H1N3	0.886 (18)	C8—H8	0.951 (18)
C1—C2	1.3850 (17)	O1W—H1W	0.807 (19)
C1—H1	0.976 (18)	O1W—H2WA	0.80 (2)
C2—C3	1.3810 (17)	O1W—H2WB	0.80 (2)
C2—H2	0.956 (18)		
C4—N1—C3	115.24 (10)	N1—C4—N3	115.16 (10)
C4—N2—C1	115.74 (11)	O1—C5—N3	123.59 (11)
C5—N3—C4	123.80 (10)	O1—C5—C6	120.82 (11)
C5—N3—H1N3	117.2 (11)	N3—C5—C6	115.59 (10)
C4—N3—H1N3	116.3 (11)	C8—C6—C7	120.14 (11)
N2—C1—C2	122.42 (11)	C8—C6—C5	121.39 (11)
N2—C1—H1	116.0 (10)	C7—C6—C5	118.30 (11)
C2—C1—H1	121.5 (10)	C8[i]—C7—C6	119.91 (11)

C3—C2—C1	116.73 (11)	C8^i—C7—H7	119.9 (10)
C3—C2—H2	121.6 (11)	C6—C7—H7	120.2 (10)
C1—C2—H2	121.7 (11)	C7^i—C8—C6	119.95 (11)
N1—C3—C2	122.62 (11)	C7^i—C8—H8	119.9 (11)
N1—C3—H3	118.1 (11)	C6—C8—H8	120.2 (11)
C2—C3—H3	119.3 (11)	H1W—O1W—H2WA	116 (4)
N2—C4—N1	127.22 (11)	H1W—O1W—H2WB	128 (5)
N2—C4—N3	117.53 (11)	H2WA—O1W—H2WB	111 (6)
C4—N2—C1—C2	−1.83 (18)	C4—N3—C5—O1	10.1 (2)
N2—C1—C2—C3	0.61 (19)	C4—N3—C5—C6	−169.50 (11)
C4—N1—C3—C2	−0.68 (19)	O1—C5—C6—C8	−137.21 (13)
C1—C2—C3—N1	0.72 (19)	N3—C5—C6—C8	42.36 (16)
C1—N2—C4—N1	1.96 (19)	O1—C5—C6—C7	38.15 (17)
C1—N2—C4—N3	178.50 (11)	N3—C5—C6—C7	−142.27 (12)
C3—N1—C4—N2	−0.73 (19)	C8—C6—C7—C8^i	−0.1 (2)
C3—N1—C4—N3	−177.34 (11)	C5—C6—C7—C8^i	−175.54 (11)
C5—N3—C4—N2	36.36 (18)	C7—C6—C8—C7^i	0.1 (2)
C5—N3—C4—N1	−146.68 (12)	C5—C6—C8—C7^i	175.39 (11)

Symmetry code: (i) −x, −y+1, −z+1.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H2WA···O1W^{ii}	0.80 (4)	1.99 (4)	2.791 (2)	171 (6)
O1W—H1W···O1	0.81 (2)	1.98 (2)	2.785 (2)	177 (3)
O1W—H2WB···O1W^{iii}	0.80 (6)	2.04 (5)	2.795 (2)	156 (6)
N3—H1N3···N1^{iv}	0.89 (2)	2.14 (2)	3.017 (2)	169 (2)
C1—H1···N2^v	0.98 (2)	2.61 (2)	3.206 (2)	119 (1)
C2—H2···O1W^v	0.95 (2)	2.58 (2)	3.508 (2)	164 (1)
C3—H3···Cg1^{vi}	0.97 (2)	2.98 (2)	3.914 (2)	164 (2)
C3—H3···Cg1^{iv}	0.97 (2)	2.98 (2)	3.914 (2)	164 (2)

Symmetry codes: (ii) −x, −y+2, −z+1; (iii) −x+1, −y+2, −z+1; (iv) −x+1, −y+1, −z; (v) −x+2, −y+2, −z; (vi) x+1, y, z−1.