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2-Aminopyrimidine–terephthalic acid (1:1) complex

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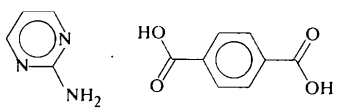
Abstract

Mutual recognition of 2-aminopyrimidine, $C_4H_5N_3$, and terephthalic acid, $C_8H_6O_4$, is possible through the formation of cyclic eight-membered hydrogen-bonded rings in the co-crystal. Each molecule can participate in two such hydrogen-bonded ring patterns, and these result in infinite hydrogen-bonded zigzag chains.

Comment

Hydrogen bonding plays a key role in molecular recognition (Goswami & Ghosh, 1997) and crystal engineering research (Goswami *et al.*, 1998). The design of highly specific solid-state structures is of considerable significance in organic chemistry due to their important applications in the development of new optical, magnetic and electronic systems (Lehn, 1990). The crystal structure of 2-aminopyrimidine itself has been reported (Scheinbeim & Schempp, 1976) where the pyrimidine ring nitrogen and amine groups are both involved to make an eight-membered hydrogen-bonded pattern. This self-assembling pattern can be replaced by a hetero-assembling pattern on co-crystallization of 2-aminopyrimidine with carboxylic acids (Etter & Adsmond, 1990; Etter *et al.*, 1990) where the best donors (acid OH) are paired with the best acceptors (ring N). This communication reports the hydrogen-bonded heterodimer of 2-aminopyrimidine with terephthalic acid, (I), where the two molecules are associated through a typical eight-membered hydrogen-bonded ring pattern.



(I)

The asymmetric unit contains a half molecule each of 2-aminopyrimidine and terephthalic acid. The 2-aminopyrimidine molecules have crystallographic *m* symmetry, with N2, C1 and C3 in the mirror plane. The terephthalic acid molecule lies on an inversion centre. The bond lengths and bond angles observed in the molecules of the co-crystal agree with those observed in their independent crystal structures (Scheinbeim & Schempp, 1976; Bailey & Brown, 1967). The acid molecule is planar within ±0.028 (1) Å and it forms a dihedral angle of 22.34 (6)° with the planar pyrimidine ring. In the co-crystal the 2-aminopyrimidine molecules are linked to the terephthalic acid molecules by N—H···O and O—H···N hydrogen bonds through the formation of eight-

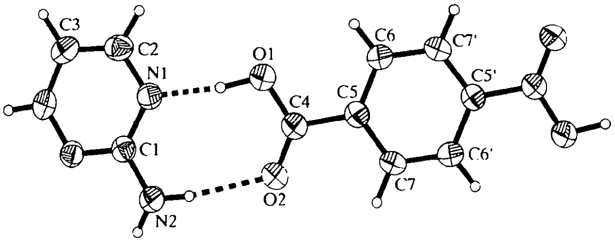


Fig. 1. The structure of the title complex showing 50% probability displacement ellipsoids and the atom-numbering scheme, where ′ signifies the symmetry operation 2−*x*, 1−*y*, −*z*. Also note that the pyrimidine molecule lies across a mirror plane and the terephthalic acid molecule on an inversion centre.

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membered hydrogen-bonded rings. In combination with the crystallographic symmetry this results in hydrogen-bonded zigzag chains extended along the *b* axis (Fig. 2). The pyrimidine and benzene rings are each stacked independently along the *c* direction with N1···C2(*x*, *y*, *z* + 1) [3.364 (2) Å] and C5···C6(*x*, *y*, *z* + 1) [3.513 (2) Å] being the shortest contacts between pyrimidine and benzene rings, respectively.

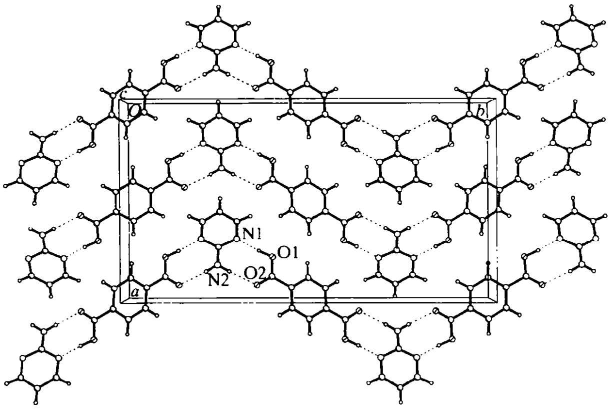

Fig. 2. Packing of the molecules viewed down the *c* axis.

Experimental

Single crystals were grown by slow evaporation of a solution of 2-aminopyrimidine and terephthalic acid (1:1) in a 5:1 mixture of acetone and methanol.

Crystal data

$C_4H_5N_3{\cdot}C_8H_6O_4$
$M_r = 261.24$
Orthorhombic
Pnma
$a = 13.0323$ (3) Å
$b = 23.9443$ (1) Å
$c = 3.7927$ (1) Å
$V = 1183.51$ (4) Å^3
$Z = 4$
$D_x = 1.466$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 3850 reflections
$\theta = 1.61$–$27.50°$
$\mu = 0.113$ mm^{-1}
$T = 293$ (2) K
Block
0.36 × 0.22 × 0.20 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
6863 measured reflections
1381 independent reflections

1090 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.50°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 30$
$l = 0 \rightarrow 4$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.106$

$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.3608P]$ where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.073$
1378 reflections
114 parameters
All H-atom parameters refined

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.175$ e Å^{-3}
$\Delta\rho_{min} = -0.140$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
O1	0.79037 (8)	0.39949 (5)	0.0107 (4)	0.0532 (4)
O2	0.92701 (9)	0.35764 (4)	0.2425 (4)	0.0598 (4)
N1	0.69648 (9)	0.29998 (5)	0.0088 (3)	0.0366 (3)
N2	0.82564 (13)	1/4	0.2775 (5)	0.0411 (4)
C1	0.73824 (14)	1/4	0.0938 (5)	0.0332 (4)
C2	0.60664 (11)	0.29883 (6)	−0.1596 (4)	0.0409 (4)
C3	0.5570 (2)	1/4	−0.2456 (6)	0.0438 (5)
C4	0.88676 (11)	0.39825 (6)	0.1118 (4)	0.0397 (4)
C5	0.94398 (11)	0.45135 (6)	0.0523 (4)	0.0364 (3)
C6	0.89653 (11)	0.49862 (6)	−0.0863 (4)	0.0395 (4)
C7	1.04779 (11)	0.45312 (6)	0.1378 (4)	0.0399 (4)

Table 2. *Selected geometric parameters (Å, °)*

O1—C4	1.314 (2)	C4—C5	1.491 (2)
O2—C4	1.211 (2)	C5—C7	1.392 (2)
N1—C2	1.334 (2)	C5—C6	1.393 (2)
N1—C1	1.3536 (14)	C6—C7^i	1.378 (2)
N2—C1	1.335 (3)	N2···O2	2.899 (1)
C2—C3	1.375 (2)	O1···N1	2.678 (2)
O2—C4—C5—C7	2.6 (2)	O1—C4—C5—C6	2.5 (2)

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

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating thirty initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *SMART* (Siemens, 1996*a*). Cell refinement: *SAINT* (Siemens, 1996*b*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1229). Services for accessing these data are described at the back of the journal.

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o-Chlorobenzaldehyde nicotinoylhydrazone dihydrate

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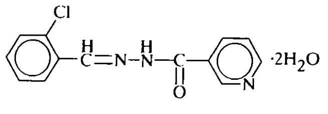
Abstract

The title compound, $C_{13}H_{10}ClN_3O{\cdot}2H_2O$, adopts the keto tautomeric form and the azomethine C═N double bond is in the *E* configuration. In the solid state, the molecules form a column-like supramolecular structure in which hydrogen bonds link the molecules to each other through water molecules.

Comment

In recent years, there has been considerable interest in the chemistry of aroylhydrazone compounds owing to their chelating ability with transition and lanthanide metal ions (Dutta & Hossain, 1985; Fun, Lu, Duan, Tian, You, Gong & Guo, 1997; Lu *et al.*, 1997; Ma *et al.*, 1994) and their medicinal properties (Constable & Holmes, 1987). In this paper, we report the structure of *o*-chlorobenzaldehyde nicotinoylhydrazone dihydrate, (I).



(I)

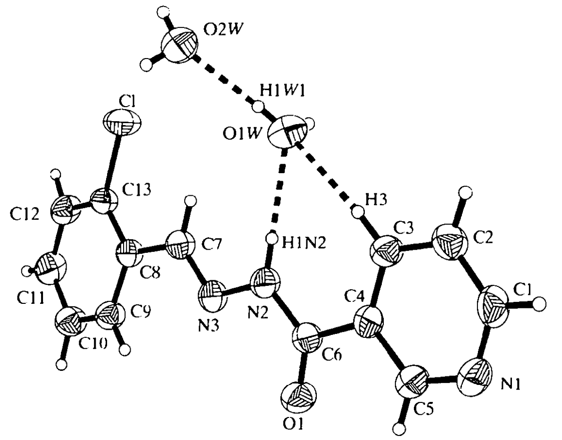


Fig. 1. The structure of title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

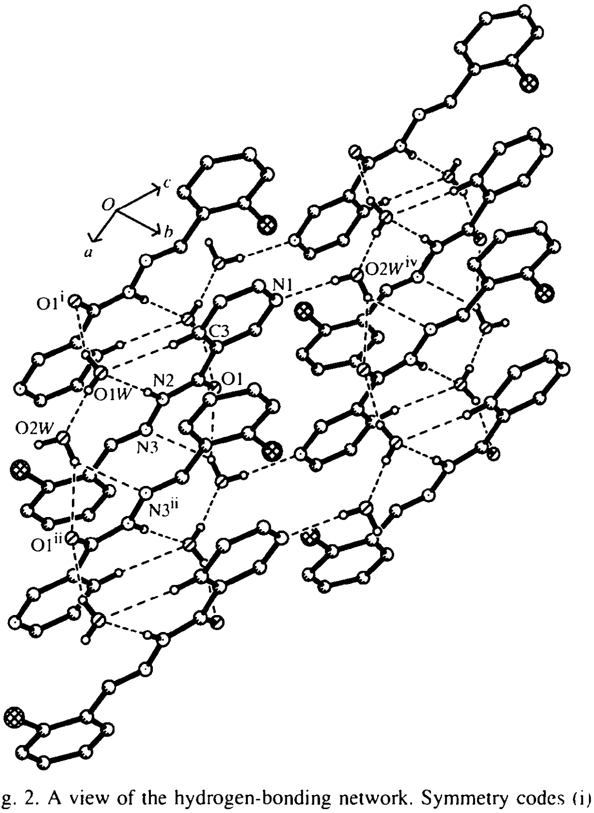


Fig. 2. A view of the hydrogen-bonding network. Symmetry codes (i) and (ii) are as given in Table 2; (iv) = $x-1, 1+y, z$.

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