

Hydrogen-bonding patterns in the cocrystal 2-amino-4,6-dimethylpyrimidine–terephthalic acid (2/1)

Periasamy Devi and Packianathan Thomas Muthiah*

School of Chemistry, Bharathidasan University, Tiruchirappalli 620 024, Tamil Nadu, India

Correspondence e-mail: tommtrichy@yahoo.co.in

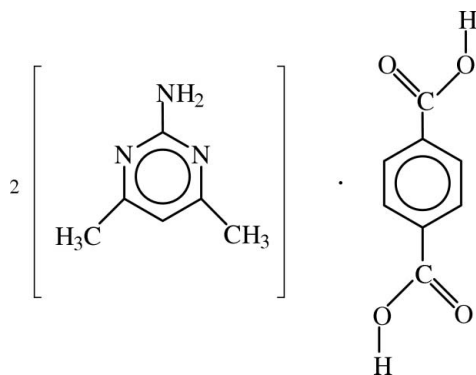
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.042; wR factor = 0.123; data-to-parameter ratio = 15.9.

In the crystal structure of the title compound, $2\text{C}_6\text{H}_9\text{N}_3 \cdot \text{C}_8\text{H}_6\text{O}_4$, terephthalic acid has a crystallographic inversion centre and the dimethylpyrimidine molecule shows approximate non-crystallographic mirror and twofold rotation symmetry. The inversion-related pyrimidine molecules form a base pair [$R_2^2(8)$ ring motif] via a pair of $\text{N}-\text{H} \cdots \text{N}$ hydrogen bonds. The carboxyl groups of terephthalic acid link the base pairs via $\text{O}-\text{H} \cdots \text{N}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds [$R_2^2(8)$ motif] to generate a supramolecular ribbon.

Related literature

For related literature, see: Baskar Raj *et al.* (2003); Lynch & Jones (2004); Du *et al.* (2005); Muthiah *et al.* (2006); Subashini *et al.* (2006); Thanigaimani *et al.* (2006); Bailey & Brown (1967); Baker & Santi (1965); Balasubramani *et al.* (2005, 2006); Chinnakali *et al.* (1999); Desiraju (1989); Etter & Adson (1990); Etter & Baures (1988); Goswami, Mahapatra, Ghosh, Nigam, Chinnakali & Fun (1999); Goswami, Mahapatra, Nigam, Chinnakali, Fun & Razak (1999); Hemamalini *et al.* (2003); Hu *et al.* (2002); Hunt *et al.* (1980); Hunter (1994); Johnson (1976); Scheinbeim & Schempp (1976); Schwalbe & Williams (1982).



Experimental

Crystal data

$2\text{C}_6\text{H}_9\text{N}_3 \cdot \text{C}_8\text{H}_6\text{O}_4$
 $M_r = 412.45$
 Monoclinic, $P2_1/n$
 $a = 3.9689$ (2) Å
 $b = 15.1778$ (8) Å
 $c = 16.5995$ (8) Å
 $\beta = 95.083$ (3)°

$V = 996.01$ (9) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293$ K
 $0.22 \times 0.20 \times 0.16$ mm

Data collection

Bruker Kappa APEXII diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004; Blessing, 1995)
 $T_{\min} = 0.969$, $T_{\max} = 0.978$

10926 measured reflections
 2198 independent reflections
 1685 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.123$
 $S = 1.08$
 2198 reflections

138 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1}-\text{H1} \cdots \text{N1}$	0.82	1.83	2.6332 (14)	167
$\text{N2}-\text{H2A} \cdots \text{N3}^i$	0.86	2.16	3.0225 (16)	177
$\text{N2}-\text{H2B} \cdots \text{O2}$	0.86	2.03	2.8817 (16)	173

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

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2/SAINT (Bruker, 2004); data reduction: SAINT/XPREF (Bruker, 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2041).

References

- Bailey, M. & Brown, C. J. (1967). *Acta Cryst.* **22**, 387–391.
 Baker, B. R. & Santi, D. V. (1965). *J. Pharm. Sci.* **54**, 1252–1257.
 Balasubramani, K., Muthiah, P. T. & Lynch, D. E. (2006). *Acta Cryst.* **E62**, o2907–o2909.
 Balasubramani, K., Muthiah, P. T., RajaRam, R. K. & Sridhar, B. (2005). *Acta Cryst.* **E61**, o4203–o4205.
 Baskar Raj, S., Muthiah, P. T., Rychlewski, U. & Warzajtis, B. (2003). *CrystEngComm*, **5**, 48–53.
 Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
 Bruker (2004). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
 Chinnakali, K., Fun, H.-K., Goswami, S., Mahapatra, A. K. & Nigam, G. D. (1999). *Acta Cryst.* **C55**, 399–401.

- Desiraju, G. R. (1989). *Crystal Engineering – the Design of Organic Solids*, Materials Science Monographs, Vol. 54. Amsterdam: Elsevier.
- Du, M., Zhang, Z.-H. & Zhao, X.-J. (2005). *Cryst. Growth Des.* **5**, 1199–1208.
- Etter, M. C. & Adson, D. A. (1990). *J. Chem. Soc. Chem. Commun.* pp. 589–591.
- Etter, M. C. & Baures, P. W. (1988). *J. Am. Chem. Soc.* **110**, 639–640.
- Goswami, S., Mahapatra, A. K., Ghosh, K., Nigam, G. D., Chinnakali, K. & Fun, H.-K. (1999). *Acta Cryst.* **C55**, 87–89.
- Goswami, S., Mahapatra, A. K., Nigam, G. D., Chinnakali, K., Fun, H.-K. & Razak, I. A. (1999). *Acta Cryst.* **C55**, 583–585.
- Hemamalini, M., Muthiah, P. T., Bocelli, G. & Cantoni, A. (2003). *Acta Cryst.* **E59**, o14–o17.
- Hu, M.-L., Ye, M.-D., Zain, S. M. & Ng, S. W. (2002). *Acta Cryst.* **E58**, o1005–o1007.
- Hunt, W. E., Schwalbe, C. H., Bird, K. & Mallinson, P. D. (1980). *J. Biochem.* **187**, 533–536.
- Hunter, C. A. (1994). *Chem. Soc. Rev.* **23**, 101–109.
- Johnson, C. K. (1976). *ORTEP II*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Lynch, D. E. & Jones, G. D. (2004). *Acta Cryst.* **B60**, 748–754.
- Muthiah, P. T., Balasubramani, K., Rychlewska, U. & Plutecka, A. (2006). *Acta Cryst.* **C62**, o605–o607.
- Scheinbeim, J. & Schempp, E. (1976). *Acta Cryst.* **B32**, 607–609.
- Schwalbe, C. H. & Williams, G. J. B. (1982). *Acta Cryst.* **B38**, 1840–1843.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Gottingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Subashini, A., Muthiah, P. T., Bocelli, G. & Cantoni, A. (2006). *Acta Cryst.* **E62**, o3847–o3849.
- Thanigaimani, K., Muthiah, P. T. & Lynch, D. E. (2006). *Acta Cryst.* **E62**, o2976–o2978.

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Hydrogen-bonding patterns in the cocrystal 2-amino-4,6-dimethylpyrimidine-terephthalic acid (2/1)

P. Devi and P. T. Muthiah

Comment

Hydrogen bonding plays an important role in molecular recognition and crystal engineering (Desiraju, 1989). Pyrimidine and aminopyrimidine derivatives are components of nucleic acid. Some pyrimidine derivatives act as antifolate drugs (Hunt *et al.*, 1980; Baker & Santi, 1965). Etter and co workers (Etter & Baures, 1988; Etter & Adsmund, 1990) studied the hydrogen bonding motifs, packing patterns and intermolecular interactions of some of the cocrystals structures. The crystal structures of aminopyrimidine derivatives (Schwalbe & Williams, 1982), aminopyrimidine cocrystals (Chinnakali *et al.*, 1999) and aminopyrimidine carboxylates (Hu *et al.*, 2002) have been reported in literature. The crystal structure of trimethoprim terephthalate-terephthalic acid (2/1/1) (Hemamalini *et al.*, 2003) has also been reported from our laboratory. Terephthalic acid self assembles *via* the $R_2^2(8)$ motif and forms interesting supramolecular architectures in the form of tapes and sheets (Du *et al.*, 2005). The adducts of carboxylic acids with 2-aminopyrimidines form the familiar $R_2^2(8)$ ring motif (Lynch & Jones, 2004). These interactions are of significance in drug design strategies. In the present study, hydrogen bonding patterns involving 2-amino-4,6-dimethyl pyrimidine-terephthalic acid (2/1), are discussed.

An *ORTEP* (Johnson, 1976) view of the title compound is shown in Fig. 1. Terephthalic acid has crystallographic inversion symmetry in the middle of the benzene ring, and the dimethylpyrimidine has approximate non-crystallographic mirror or twofold rotation symmetry (*m* or 2 along N2, C2, C5). The asymmetric unit contains one 2-amino-4,6-dimethylpyrimidine and half of a molecule of terephthalic acid. The observed bond lengths and bond angles are in agreement with the reported crystal structures of 2-aminopyrimidine (Scheinbeim & Schempp, 1976) and terephthalic acid (Bailey & Brown, 1967). The N1 atom and the 2-amino group (N2—H2B) of the pyrimidine ring form an eight membered ring motif [graph set $R_2^2(8)$ (Lynch & Jones, 2004)] with the acid molecule *via* N—H \cdots O and O—H \cdots N hydrogen bonds (Table. 1). This motif has also been observed in crystal structures of 2-aminopyrimidine-terephthalic acid (Goswami, Mahapatra, Ghosh *et al.*, 1999) and 2-aminopyrimidine-fumaric acid (Goswami, Mahapatra, Nigam *et al.*, 1999). This motif has also been reported from our laboratory in the crystal structures of 2-amino-4,6-dimethylpyrimidine-4-hydroxy benzoic acid (1/1) (Balasubramani *et al.*, 2006), 2-amino-4,6-dimethoxy pyrimidine 4-aminobenzoic acid (1/1) (Thanigaimani *et al.*, 2006) and 2-amino-4,6-dimethyl pyrimidine cinnamic acid (1/2) (Balasubramani *et al.*, 2005). Alternatively, the inversion related pyrimidine molecules form a base pair [$R_2^2(8)$ ring motif] *via* a pair of N—H \cdots N hydrogen bonds involving the 2-amino group (N2—H2A) and the pyrimidine N3 atom. This type of base pairing has been reported in the crystal structures of trimethoprim *m*-chlorobenzoate dihydrate (Baskar Raj *et al.*, 2003), 2-amino-4,6-dimethylpyrimidinium salicylate (Muthiah *et al.*, 2006) and 2-amino-4,6-dimethylpyrimidinium picrate (Subashini *et al.*, 2006). Two such independent $R_2^2(8)$ ring motifs generate the supramolecular ribbons shown in the b,c plane of Fig. 2. Here aminopyrimidine is linked to both the heteromeric and homomeric eight membered $R_2^2(8)$ ring motifs. Similar hydrogen bonded patterns are also observed in the crystal structure of 2-aminopyrimidine terephthalic acid (Goswami, Mahapatra, Ghosh *et al.*, 1999) where heteromeric ring motifs are only observed. Further the presented crystal structure is stabilized by stacking interactions between the tereph-

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thalic acid molecules (Fig. 3) with centroid-centroid distances of 3.9689 (9) Å, slip angle (the angle between the centroid vector and normal to the plane) of 29.31° and perpendicular separation of 3.461 Å. The observed values are in agreement with the aromatic stacking interactions (Hunter, 1994).

Experimental

A hot methanol solution of 2-amino-4,6-dimethylpyrimidine (30 mg, Aldrich) and terephthalic acid (41 mg, Merck) were mixed in 1:1 molar ratio and warmed in a water bath for 30 minutes. On slow evaporation, plate-like crystals of compound the title compound were obtained.

Refinement

All the hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The C—H, O—H and N—H distances are 0.93 – 0.96 Å, 0.82 Å and 0.86 Å respectively.

Figures



Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds. Inversion related atoms are labelled with "a".

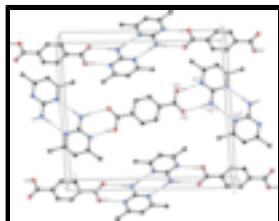


Fig. 2. View of a two-dimensional motif of supramolecular ribbons along the [1 0 0] direction. Hydrogen bonds are indicated by dashed lines. [Symmetry code: $-x, -y + 2, -z$]

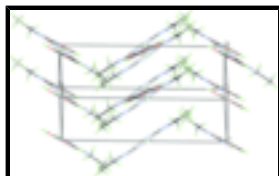


Fig. 3. View of the crystal packing along the [1 0 1] direction showing the herring bone motif and the stacking of neighbouring ribbons.

2-amino-4,6-dimethylpyrimidine–terephthalic acid (2/1)

Crystal data

$2\text{C}_6\text{H}_9\text{N}_3 \cdot \text{C}_8\text{H}_6\text{O}_4$

$M_r = 412.45$

Monoclinic, $P2_1/n$

Hall symbol: $-P 2_1n$

$a = 3.9689$ (2) Å

$b = 15.1778$ (8) Å

$c = 16.5995$ (8) Å

$F_{000} = 436$

$D_x = 1.375$ Mg m $^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3678 reflections

$\theta = 1.8$ – 27.2°

$\mu = 0.10$ mm $^{-1}$

$T = 293$ K

$\beta = 95.083 (3)^\circ$
 $V = 996.01 (9) \text{ \AA}^3$
 $Z = 2$

Plate-like, colourless
 $0.22 \times 0.20 \times 0.16 \text{ mm}$

Data collection

Bruker Kappa APEXII diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
 $T = 293 \text{ K}$
 ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004; Blessing, 1995)
 $T_{\min} = 0.969, T_{\max} = 0.978$
10926 measured reflections
2198 independent reflections
1685 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$
 $\theta_{\max} = 27.2^\circ$
 $\theta_{\min} = 1.8^\circ$
 $h = -5 \rightarrow 5$
 $k = -19 \rightarrow 19$
 $l = -21 \rightarrow 21$
1 standard reflections every 100 reflections
intensity decay: ?

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.123$
 $S = 1.08$
2198 reflections
138 parameters
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
 $w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.2062P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.24 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.18 \text{ e \AA}^{-3}$
Extinction correction: ?
Extinction coefficient: ?

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 $F^2 > 2\sigma(F^2)$ 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.

supplementary materials

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0917 (3)	0.60043 (7)	0.31830 (6)	0.0423 (4)
O2	0.3105 (3)	0.47269 (8)	0.28173 (6)	0.0494 (4)
C9	0.2581 (4)	0.52744 (10)	0.33266 (8)	0.0315 (5)
C10	0.3850 (4)	0.51463 (9)	0.41970 (8)	0.0279 (4)
C11	0.3215 (4)	0.57593 (10)	0.47848 (8)	0.0331 (5)
C12	0.4362 (4)	0.56113 (10)	0.55841 (8)	0.0327 (5)
N1	-0.1629 (3)	0.63231 (8)	0.16925 (6)	0.0277 (3)
N2	0.0773 (4)	0.51007 (8)	0.11590 (7)	0.0385 (4)
N3	-0.2311 (3)	0.60097 (8)	0.02749 (6)	0.0298 (4)
C2	-0.1084 (4)	0.58227 (9)	0.10406 (8)	0.0279 (4)
C4	-0.4185 (4)	0.67319 (9)	0.01642 (8)	0.0291 (4)
C5	-0.4849 (4)	0.72782 (10)	0.07997 (8)	0.0310 (4)
C6	-0.3484 (4)	0.70539 (9)	0.15686 (8)	0.0281 (4)
C7	-0.5565 (4)	0.69249 (11)	-0.06899 (9)	0.0392 (5)
C8	-0.3990 (4)	0.76140 (11)	0.22893 (9)	0.0384 (5)
H1	0.02920	0.60360	0.27000	0.0630*
H11	0.20170	0.62700	0.46410	0.0400*
H12	0.39290	0.60230	0.59770	0.0390*
H2A	0.11570	0.47710	0.07560	0.0460*
H2B	0.15940	0.49620	0.16390	0.0460*
H5	-0.61720	0.77810	0.07120	0.0370*
H7A	-0.37370	0.70710	-0.10080	0.0590*
H7B	-0.71100	0.74120	-0.06920	0.0590*
H7C	-0.67290	0.64150	-0.09150	0.0590*
H8A	-0.46280	0.72480	0.27220	0.0580*
H8B	-0.57440	0.80370	0.21500	0.0580*
H8C	-0.19220	0.79160	0.24580	0.0580*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0618 (8)	0.0386 (6)	0.0242 (5)	0.0146 (6)	-0.0083 (5)	0.0019 (4)
O2	0.0752 (9)	0.0448 (7)	0.0261 (5)	0.0172 (6)	-0.0075 (5)	-0.0038 (5)
C9	0.0373 (9)	0.0316 (8)	0.0252 (7)	-0.0003 (7)	-0.0002 (6)	0.0023 (6)
C10	0.0309 (8)	0.0277 (7)	0.0247 (6)	-0.0018 (6)	-0.0001 (5)	0.0021 (5)
C11	0.0399 (9)	0.0290 (8)	0.0297 (7)	0.0075 (7)	-0.0016 (6)	0.0025 (6)
C12	0.0416 (9)	0.0303 (8)	0.0258 (7)	0.0040 (7)	0.0015 (6)	-0.0023 (5)
N1	0.0332 (7)	0.0285 (6)	0.0209 (5)	-0.0012 (5)	-0.0004 (4)	-0.0006 (4)
N2	0.0585 (9)	0.0350 (7)	0.0208 (6)	0.0117 (6)	-0.0026 (5)	-0.0023 (5)
N3	0.0369 (7)	0.0314 (7)	0.0205 (5)	-0.0041 (5)	-0.0009 (5)	0.0004 (5)
C2	0.0339 (8)	0.0283 (7)	0.0213 (6)	-0.0052 (6)	0.0007 (5)	-0.0001 (5)
C4	0.0290 (8)	0.0323 (8)	0.0250 (7)	-0.0074 (6)	-0.0025 (5)	0.0040 (5)
C5	0.0333 (8)	0.0293 (8)	0.0299 (7)	-0.0002 (6)	-0.0008 (6)	0.0034 (6)
C6	0.0290 (8)	0.0291 (7)	0.0259 (7)	-0.0041 (6)	0.0010 (5)	0.0003 (5)

C7	0.0444 (10)	0.0436 (9)	0.0277 (7)	-0.0029 (7)	-0.0073 (6)	0.0056 (6)
C8	0.0445 (10)	0.0391 (9)	0.0314 (7)	0.0039 (7)	0.0019 (6)	-0.0047 (6)

Geometric parameters (Å, °)

O1—C9	1.3009 (19)	C11—H11	0.9294
O2—C9	1.2164 (18)	C12—H12	0.9302
O1—H1	0.8194	C4—C5	1.385 (2)
N1—C2	1.3550 (17)	C4—C7	1.503 (2)
N1—C6	1.3371 (19)	C5—C6	1.3842 (19)
N2—C2	1.326 (2)	C6—C8	1.496 (2)
N3—C2	1.3504 (17)	C5—H5	0.9305
N3—C4	1.3283 (19)	C7—H7A	0.9603
N2—H2B	0.8598	C7—H7B	0.9603
N2—H2A	0.8598	C7—H7C	0.9599
C9—C10	1.5000 (19)	C8—H8A	0.9596
C10—C12 ⁱ	1.383 (2)	C8—H8B	0.9601
C10—C11	1.387 (2)	C8—H8C	0.9599
C11—C12	1.3824 (19)		
C9—O1—H1	109.51	C5—C4—C7	121.58 (13)
C2—N1—C6	117.83 (11)	N3—C4—C5	122.04 (12)
C2—N3—C4	117.03 (11)	N3—C4—C7	116.37 (12)
H2A—N2—H2B	119.98	C4—C5—C6	118.01 (14)
C2—N2—H2B	119.99	N1—C6—C5	120.73 (12)
C2—N2—H2A	120.02	N1—C6—C8	117.36 (12)
O2—C9—C10	121.16 (14)	C5—C6—C8	121.90 (13)
O1—C9—O2	124.73 (13)	C4—C5—H5	120.97
O1—C9—C10	114.11 (12)	C6—C5—H5	121.02
C11—C10—C12 ⁱ	119.59 (13)	C4—C7—H7A	109.50
C9—C10—C11	121.61 (13)	C4—C7—H7B	109.43
C9—C10—C12 ⁱ	118.79 (12)	C4—C7—H7C	109.43
C10—C11—C12	120.08 (14)	H7A—C7—H7B	109.48
C10 ⁱ —C12—C11	120.33 (13)	H7A—C7—H7C	109.46
C10—C11—H11	119.96	H7B—C7—H7C	109.52
C12—C11—H11	119.97	C6—C8—H8A	109.43
C11—C12—H12	119.88	C6—C8—H8B	109.47
C10 ⁱ —C12—H12	119.79	C6—C8—H8C	109.44
N1—C2—N2	118.10 (12)	H8A—C8—H8B	109.49
N1—C2—N3	124.34 (13)	H8A—C8—H8C	109.49
N2—C2—N3	117.56 (12)	H8B—C8—H8C	109.51
C6—N1—C2—N3	-0.5 (2)	O1—C9—C10—C12 ⁱ	179.68 (14)
C2—N1—C6—C8	-178.37 (13)	C12 ⁱ —C10—C11—C12	-0.1 (2)
C6—N1—C2—N2	179.54 (14)	C9—C10—C12 ⁱ —C11 ⁱ	-178.67 (14)
C2—N1—C6—C5	1.2 (2)	C9—C10—C11—C12	178.64 (14)
C4—N3—C2—N1	-0.3 (2)	C11—C10—C12 ⁱ —C11 ⁱ	0.1 (2)
C4—N3—C2—N2	179.61 (14)	C10—C11—C12—C10 ⁱ	0.1 (2)
C2—N3—C4—C7	-179.30 (13)	C7—C4—C5—C6	179.98 (14)

supplementary materials

C2—N3—C4—C5	0.5 (2)	N3—C4—C5—C6	0.2 (2)
O1—C9—C10—C11	0.9 (2)	C4—C5—C6—N1	-1.1 (2)
O2—C9—C10—C11	-178.85 (15)	C4—C5—C6—C8	178.48 (14)
O2—C9—C10—C12 ⁱ	-0.1 (2)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	0.82	1.83	2.6332 (14)	167
N2—H2A \cdots N3 ⁱⁱ	0.86	2.16	3.0225 (16)	177
N2—H2B \cdots O2	0.86	2.03	2.8817 (16)	173

Symmetry codes: (ii) $-x, -y+1, -z$.

Fig. 1

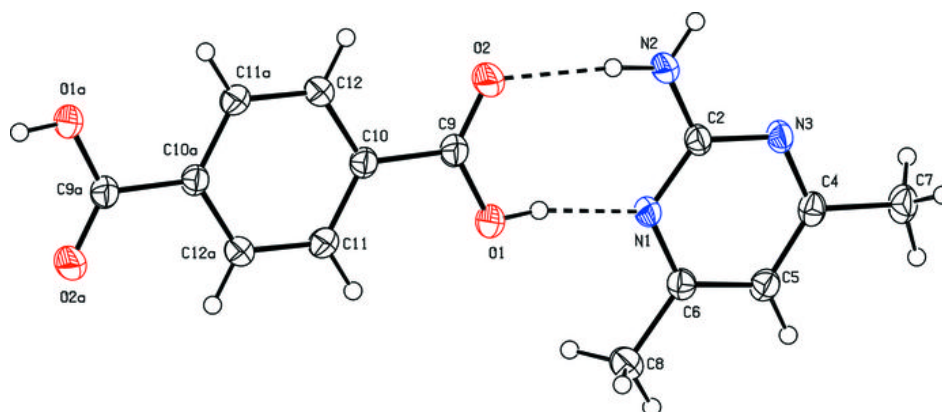


Fig. 2

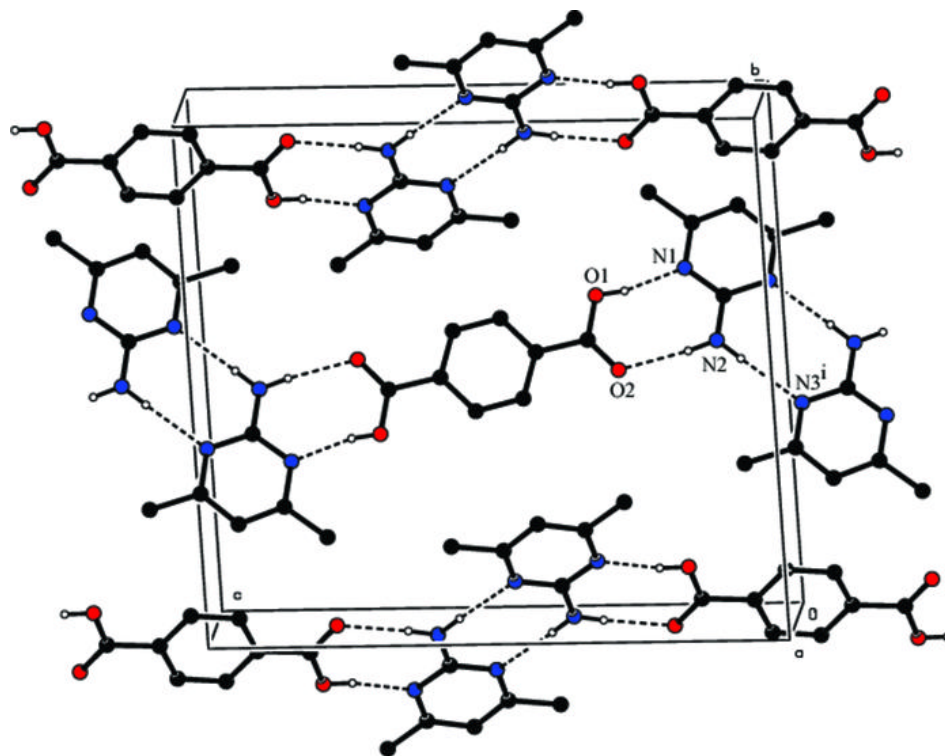


Fig. 3

