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2,6-Diaminopyrimidin-4(3*H*)-one–4-nitrobenzoic acid (1/1)

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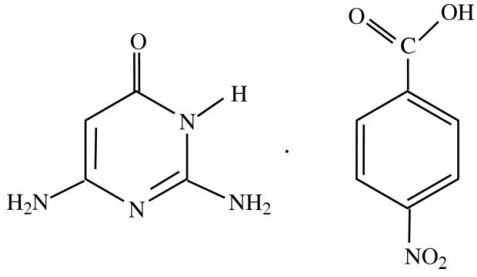
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.148; data-to-parameter ratio = 12.4.

In the title compound, $C_4H_6N_4O\cdot C_7H_5NO_4$, there are two types of base-pairing motifs, one involving a pair of N—H···N hydrogen bonds and the other involving a pair of N—H···O hydrogen bonds. These paired molecules are further linked by N—H···O hydrogen bonds to generate *DADA* and *DDAA* (*D* = donor and *A* = acceptor) arrays leading to a supramolecular sheet.

Related literature

For related literature, see: Baskar Raj *et al.* (2003); Bernstein *et al.* (1995); Lehn (1995); Panneerselvam *et al.* (2002); Stanley *et al.* (2005); Tavale & Pant (1971); Thanigaimani *et al.* (2006).



Experimental

Crystal data

$C_4H_6N_4O\cdot C_7H_5NO_4$
M_r = 293.25
Triclinic, $P\overline{1}$
a = 7.295 (3) Å
b = 9.781 (2) Å
c = 10.104 (3) Å
α = 102.27 (3)°
β = 107.76 (2)°
γ = 105.76 (3)°
V = 625.5 (4) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 1.08 mm^{-1}
T = 293 K
0.26 × 0.14 × 0.13 mm

Data collection

Siemens AED single-crystal diffractometer
Absorption correction: none
2381 measured reflections
2381 independent reflections
2115 reflections with $I > 2\sigma(I)$
1 standard reflection
every 100 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.148
S = 1.07
2381 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.82	1.75	2.550 (2)	166
N2—H2A···N1^{ii}	0.86	2.19	3.015 (2)	162
N2—H2B···O3^{iii}	0.86	2.17	2.875 (3)	138
N3—H3···O1^{iv}	0.86	1.99	2.846 (3)	178
N6—H6A···O3^{v}	0.86	2.43	3.270 (3)	165
N6—H6B···O4^{vi}	0.86	2.31	3.159 (3)	168

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

Data collection: local program; cell refinement: local program; data reduction: local program; 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: HB2563).

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supplementary materials

Acta Cryst. (2007). E**63**, o4244 [**doi:10.1107/S1600536807047861**]

2,6-Diaminopyrimidin-4(3*H*)-one-4-nitrobenzoic acid (1/1)

A. Subashini, P. T. Muthiah, G. Bocelli and A. Cantoni

Comment

Hydrogen bonds are used extensively as a tool to design the structure of molecular crystals, because of their strength, as well as their directional nature, compared to other intermolecular non-covalent interactions (Lehn, 1995). As part of our ougoing studies of hydrogen bonding in molecular arrays (Thanigaimani *et al.*, 2006), we now report the structure of the title 1:1 adduct. The crystal structure of 4-nitrobenzoic acid (Tavale & Pant, 1971) has been already reported in literature.

The asymmetric unit of (I) contains one 2,6-diamino-4-oxo pyrimidine (DAMPY) molecule and one 4-nitrobenzoic (4-NBA) acid molecule (Fig. 1). At the N3 position of the DAMPY ring, there is an increase in internal angle [121.93 (17)°] as compared with 116.75 (16)° at N1. This is due to the presence of a hydrogen atom covalently bonded to the ring nitrogen, N3. In the crystal, the DAMPY molecules form two types of pairing. Two inversion related DAMPY are paired *via* N—H···N hydrogen bonds involving the 2-amino group and the N1 atom, generating a $R_2^2(8)$ motif (Bernstein *et al.*, 1995). In addition to the base pairing, a hydrogen bonded acceptor (O3) bridges the 2-amino and 6-amino group on both side of the pairing, leading to a complementary linear DADA (D = donor in hydrogen bonds; A = acceptor in hydrogen bonds) array of quadruple hydrogen bonds. The resultant rings have the graph-set notation $R^2_3(8)$, $R^2_2(8)$ and $R^2_3(8)$ (Fig 2). This type of DADA array has been observed in trimethoprim (TMP) sulfonate salts (Baskar Raj *et al.*, 2003) and TMP-salicylate methanol solvate (Panneerselvam *et al.*, 2002). The two DAMPY molecules are also paired *via* a pair of N—H···O hydrogen bonds and the paired molecules are further bridged by the carboxyl group on either side, forming a DDAA array [graph set notation = ($R^2_3(10)$, $R^2_2(8)$ and $R^2_3(10)$]. In general, either one of the motifs (DADA or DDAA array) has been identified in diaminopyrimidine-carboxylate salts. In rare cases, the presence of both DADA and DDAA arrangement in a single-crystal structure has been reported (Stanley *et al.*, 2005). Interestingly, in the present study also both DADA and DDAA array motifs are arranged in an alternate manner (Fig. 3). These arrays are linked by N—H···O and O—H···O hydrogen bonds to form a supramolecular sheet with alternating $R^4_4(30)$ and $R^4_4(22)$ rings. The hydrogen bonds are listed in Table 1.

Experimental

Hot ethanol solutions of 2,6-diamino-4-oxopyrimidine (31 mg) and 4-nitrobenzoic acid (42 mg) were mixed in 1:1 molar ratio and warmed over a water bath for half an hour and kept at room temperature for crystallization. Yellow prisms of (I) were obtained after a week *via* slow evaporation.

Refinement

All hydrogen atoms were placed in idealized locations (C—H = 0.93 Å, N—H = 0.86 Å, O—H = 0.82 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}$(carrier).

Figures

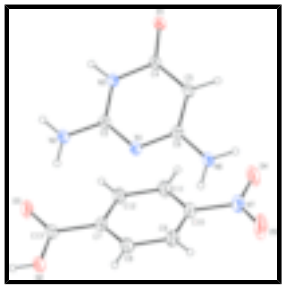

Fig. 1. View of the molecular structure of (I) showing 30% probability displacement ellipsoids for the non-hydrogen atoms.

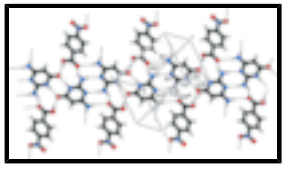

Fig. 2. DADA and DDAA arrays in compound (I). Symmetry Codes: (i) −*x* + 1, −*y* + 1, −*z* + 1; (ii) −*x*, −*y*, −*z* + 1; (iii) *x* + 1, *y* + 1, *z*.

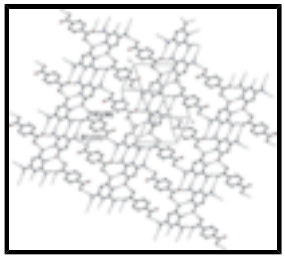

Fig. 3. A View of supramolecular sheet in compound (I).

2,6-Diaminopyrimidin-4(3*H*)-one–4-nitrobenzoic acid (1/1)

Crystal data

$C_4H_6N_4O·C_7H_5NO_4$	$Z = 2$
$M_r = 293.25$	$F_{000} = 304$
Triclinic, $P\overline{1}$	$D_x = 1.557$ Mg m^{-3}
Hall symbol: -P 1	Cu $K\alpha$ radiation $\lambda = 1.54178$ Å
$a = 7.295$ (3) Å	Cell parameters from 45 reflections
$b = 9.781$ (2) Å	$\theta = 4.8–70.1°$
$c = 10.104$ (3) Å	$\mu = 1.08$ mm^{-1}
$\alpha = 102.27$ (3)°	$T = 293$ K
$\beta = 107.76$ (2)°	Prism, yellow
$\gamma = 105.76$ (3)°	0.26 × 0.14 × 0.13 mm
$V = 625.5$ (4) Å^3	

Data collection

Siemens AED single-crystal diffractometer	$R_{int} = 0.0000$
Radiation source: fine-focus sealed tube	$\theta_{max} = 70.1°$
Monochromator: graphite	$\theta_{min} = 4.9°$
$T = 293$ K	$h = -8 \rightarrow 8$

ω/2θ scans	$k = -11 \rightarrow 10$
Absorption correction: none	$l = -6 \rightarrow 12$
2381 measured reflections	1 standard reflections
2381 independent reflections	every 100 reflections
2115 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.0884P)^2 + 0.1718P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.148$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
2381 reflections	$\Delta\rho_{min} = -0.26$ e Å^{-3}
192 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001Fc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0069 (16)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.2091 (2)	0.28643 (17)	0.68916 (14)	0.0582 (5)
O3	−0.0884 (2)	0.25985 (18)	0.51990 (14)	0.0643 (5)
O4	0.1804 (3)	−0.1464 (2)	0.00742 (17)	0.0831 (7)
O5	0.4695 (3)	−0.1014 (3)	0.1759 (2)	0.1094 (9)
N7	0.3027 (3)	−0.08897 (19)	0.13449 (18)	0.0583 (6)
C7	0.1363 (3)	0.15405 (18)	0.44513 (18)	0.0410 (5)
C8	0.3350 (3)	0.1534 (2)	0.4841 (2)	0.0495 (5)
C9	0.3921 (3)	0.0758 (2)	0.3817 (2)	0.0530 (6)
C10	0.2446 (3)	−0.0024 (2)	0.24225 (19)	0.0465 (5)
C11	0.0462 (3)	−0.0038 (2)	0.2001 (2)	0.0533 (6)
C12	−0.0072 (3)	0.0765 (2)	0.3035 (2)	0.0503 (6)

C13	0.0740 (3)	0.23944 (19)	0.55510 (18)	0.0425 (5)
O1	0.1138 (2)	0.40516 (16)	−0.10381 (12)	0.0554 (4)
N1	0.4530 (2)	0.40898 (17)	0.30142 (14)	0.0443 (5)
N2	0.2528 (2)	0.51813 (19)	0.38755 (15)	0.0517 (5)
N3	0.1934 (2)	0.46264 (16)	0.14123 (14)	0.0441 (5)
N6	0.6491 (3)	0.2979 (2)	0.21274 (17)	0.0605 (6)
C2	0.3024 (3)	0.46214 (19)	0.27595 (17)	0.0408 (5)
C4	0.2278 (3)	0.4014 (2)	0.01796 (17)	0.0439 (5)
C5	0.3826 (3)	0.3424 (2)	0.04147 (18)	0.0468 (5)
C6	0.4933 (3)	0.3499 (2)	0.18368 (18)	0.0438 (5)
H2	0.16380	0.32640	0.74510	0.0870*
H8	0.43080	0.20550	0.57950	0.0590*
H9	0.52620	0.07650	0.40650	0.0640*
H11	−0.04970	−0.05720	0.10490	0.0640*
H12	−0.14010	0.07830	0.27730	0.0600*
H2A	0.31890	0.51910	0.47450	0.0620*
H2B	0.15480	0.55310	0.37220	0.0620*
H3	0.10000	0.50200	0.13160	0.0530*
H5	0.41230	0.29830	−0.03660	0.0560*
H6A	0.71580	0.30340	0.30130	0.0730*
H6B	0.68200	0.25920	0.14280	0.0730*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0645 (8)	0.0846 (10)	0.0354 (7)	0.0463 (7)	0.0206 (6)	0.0113 (6)
O3	0.0624 (8)	0.0930 (11)	0.0450 (7)	0.0524 (8)	0.0199 (6)	0.0063 (7)
O4	0.0936 (12)	0.1154 (14)	0.0470 (9)	0.0599 (11)	0.0315 (8)	0.0041 (9)
O5	0.0892 (13)	0.158 (2)	0.0816 (13)	0.0853 (14)	0.0293 (10)	−0.0076 (12)
N7	0.0675 (11)	0.0693 (11)	0.0515 (10)	0.0401 (9)	0.0321 (8)	0.0120 (8)
C7	0.0483 (9)	0.0467 (8)	0.0373 (8)	0.0259 (7)	0.0211 (7)	0.0137 (7)
C8	0.0484 (9)	0.0610 (10)	0.0392 (9)	0.0271 (8)	0.0163 (7)	0.0078 (8)
C9	0.0490 (10)	0.0684 (11)	0.0494 (10)	0.0338 (9)	0.0224 (8)	0.0128 (9)
C10	0.0571 (10)	0.0515 (9)	0.0426 (9)	0.0298 (8)	0.0277 (8)	0.0123 (8)
C11	0.0523 (10)	0.0661 (11)	0.0386 (9)	0.0294 (9)	0.0156 (8)	0.0036 (8)
C12	0.0478 (9)	0.0655 (11)	0.0408 (9)	0.0320 (8)	0.0174 (7)	0.0078 (8)
C13	0.0505 (9)	0.0499 (9)	0.0359 (8)	0.0274 (7)	0.0204 (7)	0.0133 (7)
O1	0.0672 (8)	0.0866 (9)	0.0281 (6)	0.0536 (7)	0.0179 (5)	0.0158 (6)
N1	0.0562 (8)	0.0594 (9)	0.0301 (7)	0.0375 (7)	0.0193 (6)	0.0138 (6)
N2	0.0645 (9)	0.0764 (10)	0.0299 (7)	0.0496 (8)	0.0201 (6)	0.0132 (7)
N3	0.0525 (8)	0.0601 (9)	0.0300 (7)	0.0376 (7)	0.0167 (6)	0.0108 (6)
N6	0.0779 (11)	0.0956 (13)	0.0378 (8)	0.0662 (10)	0.0287 (8)	0.0242 (8)
C2	0.0500 (9)	0.0490 (9)	0.0303 (8)	0.0283 (7)	0.0168 (6)	0.0108 (6)
C4	0.0524 (9)	0.0578 (10)	0.0288 (8)	0.0317 (8)	0.0169 (7)	0.0108 (7)
C5	0.0576 (10)	0.0639 (10)	0.0319 (8)	0.0383 (8)	0.0221 (7)	0.0125 (7)
C6	0.0535 (9)	0.0538 (9)	0.0360 (8)	0.0330 (8)	0.0214 (7)	0.0139 (7)

Geometric parameters (Å, °)

O2—C13	1.301 (2)	N6—H6B	0.8599
O3—C13	1.213 (3)	N6—H6A	0.8603
O4—N7	1.218 (2)	C7—C12	1.383 (3)
O5—N7	1.209 (3)	C7—C13	1.503 (3)
O2—H2	0.8196	C7—C8	1.383 (3)
O1—C4	1.272 (2)	C8—C9	1.386 (3)
N7—C10	1.472 (3)	C9—C10	1.378 (3)
N1—C6	1.360 (2)	C10—C11	1.374 (3)
N1—C2	1.318 (3)	C11—C12	1.388 (3)
N2—C2	1.340 (2)	C8—H8	0.9295
N3—C2	1.353 (2)	C9—H9	0.9305
N3—C4	1.389 (2)	C11—H11	0.9300
N6—C6	1.346 (3)	C12—H12	0.9301
N2—H2A	0.8602	C4—C5	1.383 (3)
N2—H2B	0.8602	C5—C6	1.392 (2)
N3—H3	0.8602	C5—H5	0.9304
C13—O2—H2	109.45	O3—C13—C7	122.20 (16)
O4—N7—O5	122.5 (2)	O2—C13—C7	113.48 (19)
O5—N7—C10	118.68 (18)	O2—C13—O3	124.31 (18)
O4—N7—C10	118.8 (2)	C9—C8—H8	119.84
C2—N1—C6	116.75 (15)	C7—C8—H8	119.76
C2—N3—C4	121.93 (17)	C10—C9—H9	121.00
H2A—N2—H2B	119.99	C8—C9—H9	120.92
C2—N2—H2B	119.97	C10—C11—H11	120.89
C2—N2—H2A	120.04	C12—C11—H11	120.94
C4—N3—H3	119.02	C11—C12—H12	119.80
C2—N3—H3	119.06	C7—C12—H12	119.87
C6—N6—H6B	120.00	N1—C2—N2	119.17 (15)
C6—N6—H6A	119.99	N1—C2—N3	123.04 (17)
H6A—N6—H6B	120.01	N2—C2—N3	117.79 (19)
C8—C7—C13	120.69 (16)	O1—C4—C5	127.16 (18)
C8—C7—C12	120.09 (19)	N3—C4—C5	116.08 (15)
C12—C7—C13	119.2 (2)	O1—C4—N3	116.75 (19)
C7—C8—C9	120.40 (18)	C4—C5—C6	119.02 (18)
C8—C9—C10	118.1 (2)	N1—C6—C5	123.1 (2)
N7—C10—C9	118.3 (2)	N6—C6—C5	121.25 (18)
C9—C10—C11	122.9 (2)	N1—C6—N6	115.62 (16)
N7—C10—C11	118.82 (17)	C4—C5—H5	120.52
C10—C11—C12	118.18 (18)	C6—C5—H5	120.46
C7—C12—C11	120.3 (2)		
O5—N7—C10—C9	7.7 (3)	C13—C7—C8—C9	−179.27 (18)
O4—N7—C10—C9	−172.9 (2)	C8—C7—C12—C11	0.8 (3)
O4—N7—C10—C11	7.5 (3)	C13—C7—C12—C11	−179.69 (18)
O5—N7—C10—C11	−171.8 (2)	C8—C7—C13—O2	−11.3 (3)
C2—N1—C6—C5	−0.8 (3)	C12—C7—C8—C9	0.3 (3)
C6—N1—C2—N2	178.30 (18)	C7—C8—C9—C10	−1.4 (3)

C2—N1—C6—N6	179.30 (18)	C8—C9—C10—N7	−178.05 (18)
C6—N1—C2—N3	−1.4 (3)	C8—C9—C10—C11	1.5 (3)
C4—N3—C2—N1	2.5 (3)	N7—C10—C11—C12	179.05 (18)
C4—N3—C2—N2	−177.28 (18)	C9—C10—C11—C12	−0.5 (3)
C2—N3—C4—O1	178.64 (18)	C10—C11—C12—C7	−0.7 (3)
C2—N3—C4—C5	−1.2 (3)	O1—C4—C5—C6	179.3 (2)
C8—C7—C13—O3	169.4 (2)	N3—C4—C5—C6	−0.9 (3)
C12—C7—C13—O2	169.13 (18)	C4—C5—C6—N6	−178.1 (2)
C12—C7—C13—O3	−10.1 (3)	C4—C5—C6—N1	1.9 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.82	1.75	2.550 (2)	166
N2—H2A···N1^{ii}	0.86	2.19	3.015 (2)	162
N2—H2B···O3^{iii}	0.86	2.17	2.875 (3)	138
N3—H3···O1^{iv}	0.86	1.99	2.846 (3)	178
N6—H6A···O3^{v}	0.86	2.43	3.270 (3)	165
N6—H6B···O4^{vi}	0.86	2.31	3.159 (3)	168

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

Fig. 1

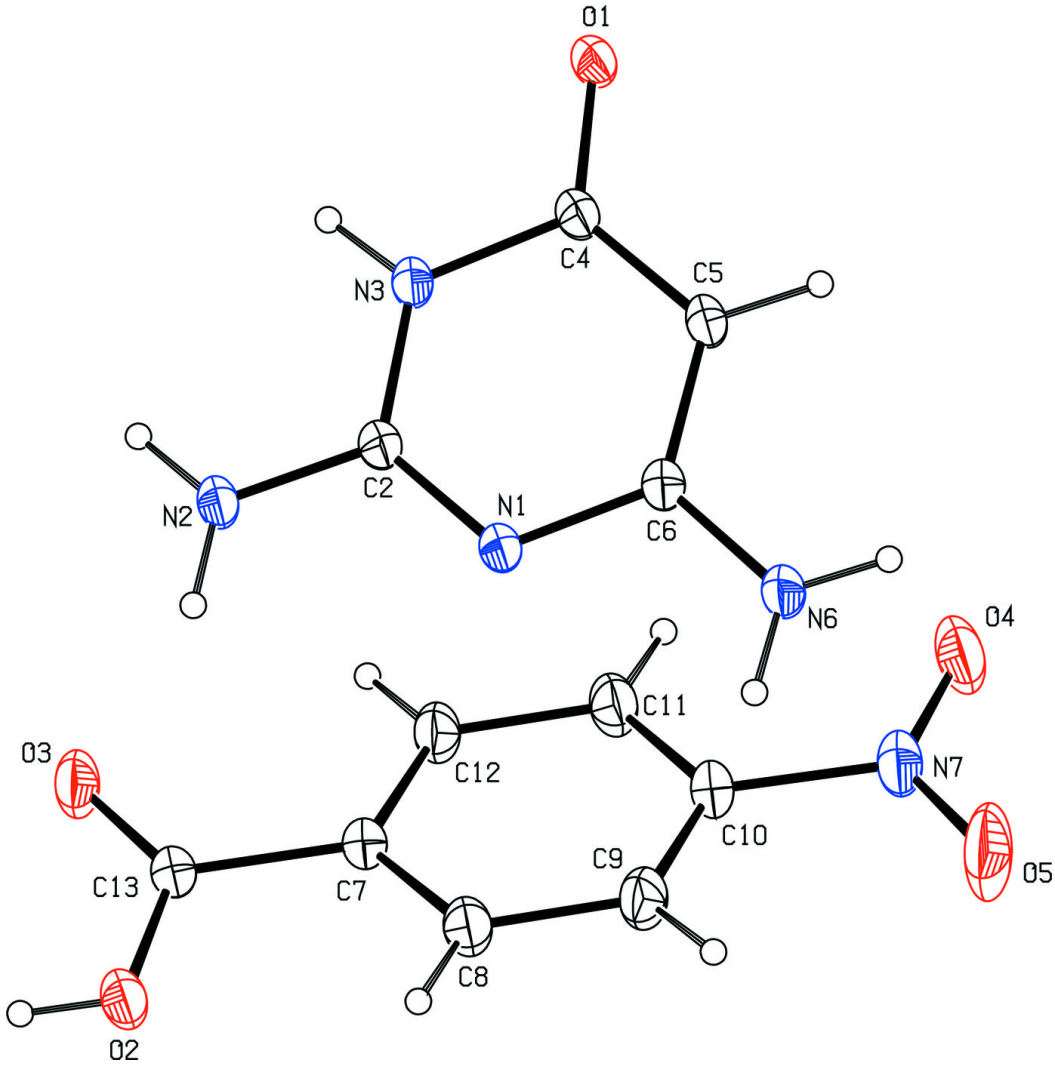

Fig. 2

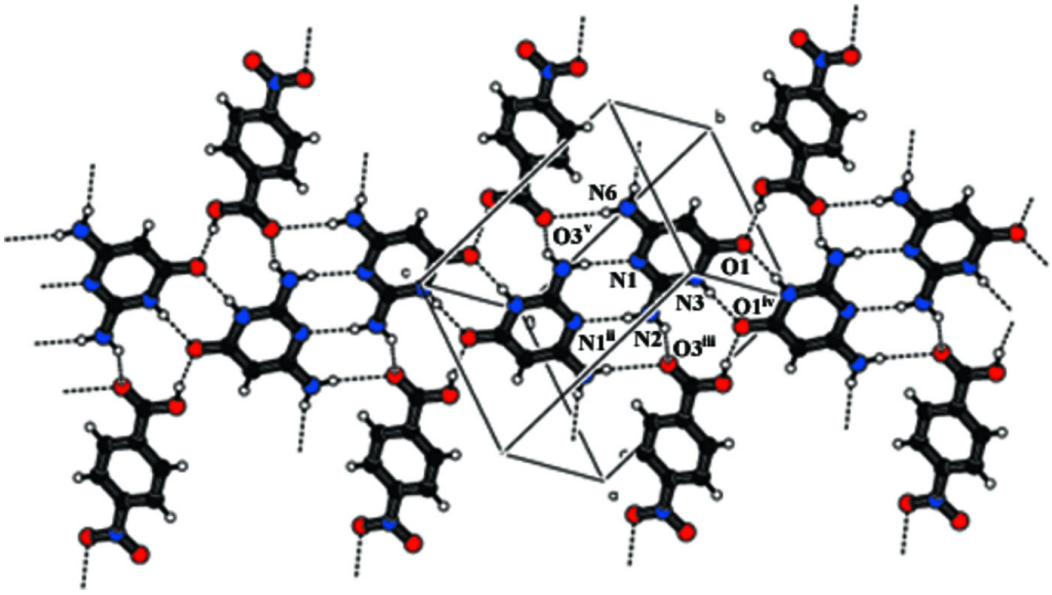

Fig. 3