Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Hydrogen-bonding patterns in the cocrystal 2-amino-4,6-dimethoxypyrimidine–anthranilic acid (1/1)

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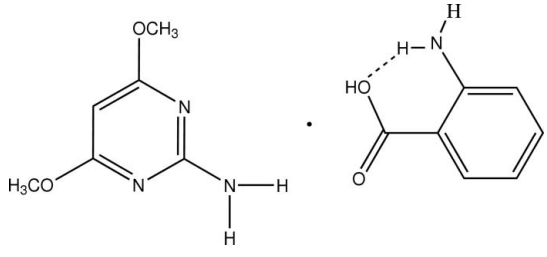
Received 21 November 2007; accepted 26 November 2007

Key indicators: single-crystal X-ray study; $T$ = 120 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.050; $wR$ factor = 0.134; data-to-parameter ratio = 16.1.

In the title cocrystal, $C_6H_9N_3O_2{\cdot}C_7H_7NO_2$, the asymmetric unit contains two crystallographically independent 2-amino-4,6-dimethoxy pyrimidine-anthranilic acid adducts. The 2-amino-4,6-dimethoxy pyrimidine molecules interact with the carboxylic group of the respective anthranilic acid molecules through N—H···O and O—H···N hydrogen bonds, forming a cyclic hydrogen-bonded motif $R_2^2(8)$. The pyrimidine molecules also form base pairs *via* a pair of N—H···N hydrogen bonds, forming another $R_2^2(8)$ motif. The typical intramolecular N—H···O hydrogen bond is observed in the anthranilic acid molecules. Furthermore, the crystal structure is stabilized by C—H···O hydrogen bonds.

## Related literature

For related literature, see: Baker & Santi (1965); Balasubramani *et al.* (2005, 2006); Bernstein *et al.* (1995); Boone *et al.* (1977); Chinnakali *et al.* (1999); Desiraju (1989); Etter (1990); Hunt *et al.* (1980); Hunter (1994); Low *et al.* (2002); Lynch & Jones (2004); Muthiah *et al.* (2006); Schwalbe & Williams (1982); Stanley *et al.* (2005); Takazawa *et al.* (1986); Thanigaimani *et al.* (2006, 2007*a*,*b*).




## Experimental

### Crystal data

$C_6H_9N_3O_2{\cdot}C_7H_7NO_2$
$M_r$ = 292.30
Triclinic, $P\overline{1}$
$a$ = 7.2802 (3) Å
$b$ = 7.4095 (2) Å
$c$ = 25.8035 (9) Å
$\alpha$ = 83.636 (2)°
$\beta$ = 83.1620 (10)°
$\gamma$ = 82.373 (2)°
$V$ = 1363.38 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 120 K
0.24 × 0.19 × 0.08 mm

### Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: none
24443 measured reflections
6286 independent reflections
4140 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.064

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.134
$S$ = 1.04
6286 reflections
390 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2$A$—H2$A$1···O4$A$ | 0.86 | 1.97 | 2.818 (2) | 169 |
| N2$A$—H2$A$2···N3$A^{i}$ | 0.86 | 2.35 | 3.166 (2) | 160 |
| O3$A$—H3$A$···N1$A$ | 0.82 | 1.86 | 2.6808 (19) | 175 |
| O4$B$—H4$B$···N1$B$ | 0.82 | 1.81 | 2.603 (2) | 164 |
| N4$A$—H7···O4$A$ | 0.91 (2) | 1.97 (2) | 2.650 (2) | 130 (2) |
| N2$B$—H2$B$1···O3$B$ | 0.86 | 2.22 | 3.075 (2) | 170 |
| N2$B$—H2$B$2···N3$B^{ii}$ | 0.86 | 2.33 | 3.165 (2) | 164 |
| N4$A$—H16···N4$A^{iii}$ | 0.87 (2) | 2.58 (2) | 3.100 (3) | 119.7 (18) |
| N4$B$—H4$B$1···O3$B$ | 0.86 | 2.12 | 2.749 (2) | 129 |
| N4$B$—H4$B$2···O3$A^{iv}$ | 0.86 | 2.59 | 3.450 (2) | 176 |
| C8$B$—H8$B$3···O3$B^{iv}$ | 0.96 | 2.47 | 3.339 (2) | 150 |
| C14$A$—H14$A$···O3$A$ | 0.93 | 2.42 | 2.747 (3) | 100 |
| C14$B$—H14$B$···O4$B$ | 0.93 | 2.30 | 2.655 (2) | 102 |

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $-x+3, -y+1, -z+1$; (iii) $-x+1, -y+3, -z$; (iv) $-x+2, -y+2, -z+1$.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; 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*.

KT thanks the UGC (New Delhi) for the UGC–Rajiv Gandhi Junior Fellowship [Reference No: F.16–12/2000 (SA-II)] DL thanks the EPSRC National Crystallography Service (Southampton, England) for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2644).

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# supplementary materials

*Acta Cryst.* (2008). E**64**, o107-o108 [ **doi:10.1107/S1600536807063271** ]

# Hydrogen-bonding patterns in the cocrystal 2-amino-4,6-dimethoxypyrimidine-anthranilic acid (1/1)

**K. Thanigaimani, P. T. Muthiah and D. E. Lynch**

## Comment

Aminopyrimidine-Carboxylate interactions are of fundamental importantance since they are involved in protein-nucleic acids recognition and protein-drug binding (Hunt *et al.*, 1980; Baker & Santi, 1965). Hydrogen bonding plays a key role in molecular recognition and crystal engineering research (Desiraju, 1989). The adducts of carboxylic acids with 2-aminoheterocylic ring system form a graph-set motif $R_2^2(8)$ (Lynch & Jones, 2004). This motif is very robust in aminopyrimidine-carboxylic acid/ carboxylates systems. The crystal structures of aminopyrimidine derivatives (Schwalbe & Williams, 1982), aminopyrimidine carboxylates (Stanley *et al.*, 2005) and co-crystals (Chinnakali *et al.*, 1999) have been reported. The crystal structure of 2-amino-4,6-dimethoxy pyrimidine has also been reported (Low *et al.*, 2002). The crystal structures of 2-amino-4,6-dimethoxy pyrimidine 4-aminobenzoic acid (1/1) (Thanigaimani *et al.*, 2006), 2-amino-4,6-dimethoxy pyrimidine phthalic acid (1/1) (Thanigaimani *et al.*, 2007*a*), 2-amino-4,6-dimethyl pyrimidine cinnamic acid (1/2) (Balasubramani *et al.*, 2005) and 2-amino-4,6-dimethyl pyrimidine 4-hydroxybenzoic acid (1/1) (Balasubramani *et al.*, 2006), have been recently reported from our laboratory. The crystal structure of anthranilic acid (Boone *et al.*, 1977; Takazawa *et al.*, 1986) is known. In the present study, the hydrogen-bonding patterns in the 2-amino-4,6-dimethoxypyrimidine anthranilic acid (1/1) cocrystal, (I), are investigated.

The asymmetric unit (Fig. 1) contains pair of molecules of 2-amino-4,6-dimethoxypyrimidine (A&B) and anthranilic acid (A&B). The carboxyl group of each anthranilic acid interacts with the corresponding 2-amino-4,6-dimethoxy pyrimidine molecule *via* a pair of N—H···O and O—H···N hydrogen bonds generating $R_2^2(8)$ ring motif. (Etter, 1990; Bernstein *et al.*, 1995). In both the types (A & B) inversion related bases are paired *via* (2) N—H···N(3) hydrogen bonds forming another type of $R_2^2(8)$ motif. This type of base pairing has been reported in the crystal structures of 2-amino-4,6-dimethylpyrimidinium salicylate (Muthiah *et al.*, 2006) and 2-amino-4,6-dimethoxypyrimidinium 4-hydroxybenzoate (Thanigaimani *et al.*, 2007*b*). The carboxyl oxygen atom (O3A) of anthranilic acid (Molecule A) is linked to 4-amino group (N4B) of anthranilic acid (Molecule B) *via* N—H···O hydrogen bonds. In each of the anthranilic acid molecule, there is a typical intramolecular hydrogen bond between the amino $NH_2$ group and the carboxylic group, (graph-set notation S6). There is also a C—H···O hydrogen bond involving atom C8B of the pyrimidine moiety and O4B of the anthranilic acid molecule. The π-π stacking interactions between the aromatic ring are also observed. The pyrimidine ring of 2-amino-4,6-diemthoxy pyrimidine (molecule A) forms stacking interactions with the aryl rings of the anthranilic acid (molecule A), with interplanar and centroid-centroid distance of 3.430 Å and 3.5436 (11) Å, respectively, and a slip angle (angle between the centroid vector and the normal to the plane) is 13.85°. A similar type of stacking is also observed between two 2-amino-4,6-dimethoxy pyrimidine (molecule B) related by an inversion centre. The centroid-centroid distance and interplanar distance are 3.5411 (10) Å and 3.380 Å, respectively, the slip angle being 17.33°. These are typical aromatic stacking values (Hunter, 1994).

### Experimental

A hot methanol solution (20 ml) of 2-amino-4,6-dimethoxypyrimidine (38 mg, Aldrich) and anthranilic acid (34 mg, Loba Chemie) was warmed for half an hour over a water bath. The mixture was cooled slowly and kept at room temperature; after a few days, colourless plate-like crystals were obtained.

### Refinement

The hydrogen atoms of the N4A (H4A1, H4A2) were located in difference Fourier map and refined freely. The other hydrogen atoms were positioned geometrically and were refined using a riding model. The C—H and O—H bond lengths are 0.93–0.96 and 0.82Å respectively [$U_{iso}$(H)= 1.2Ueq (C, O)].

### Figures

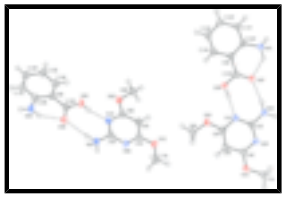


Fig. 1. The asymmetric unit of (I), showing 50% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

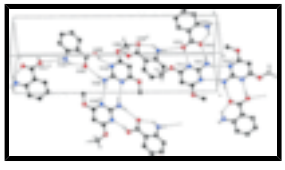


Fig. 2. The crystal structure of (I). Dashed lines indicate hydrogen bonds [symmetry code: (i) −*x* + 2, −*y* + 1, −*z*; (ii) −*x* + 3, −*y* + 1, −*z* + 1; (iv) −*x* + 2, −*y* + 2, −*z* + 1.

### 2-amino-4,6-dimethoxypyrimidine–anthranilic acid (1/1)

*Crystal data*

| | |
|---|---|
| $C_6H_9N_3O_2·C_7H_7NO_2$ | $Z$ = 4 |
| $M_r$ = 292.30 | $F_{000}$ = 616 |
| Triclinic, $P\overline{1}$ | $D_x$ = 1.424 Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 7.2802 (3) Å | Cell parameters from 25 reflections |
| $b$ = 7.4095 (2) Å | θ = 3.0–27.6º |
| $c$ = 25.8035 (9) Å | μ = 0.11 mm$^{-1}$ |
| α = 83.636 (2)º | $T$ = 120 K |
| β = 83.1620 (10)º | Plate-like, colourless |
| γ = 82.373 (2)º | 0.24 × 0.19 × 0.08 mm |
| $V$ = 1363.38 (8) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker–Nonius CCD | 6286 independent reflections |

diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Monochromator: graphite
Detector resolution: 9.091 pixels mm$^{-1}$
$T$ = 120 K
φ and ω scans
Absorption correction: none
24443 measured reflections
4140 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.064
$\theta_{max}$ = 27.6º
$\theta_{min}$ = 3.0º
$h$ = −9→9
$k$ = −9→9
$l$ = −33→33

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.134
$S$ = 1.04
6286 reflections
390 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$
Extinction correction: shelxl, $FC^*=KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Extinction coefficient: 0.022 (3)

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1A | 1.21498 (19) | 0.13688 (17) | 0.11198 (5) | 0.0321 (4) |
| O2A | 0.9788 (2) | 0.62312 (17) | 0.21411 (5) | 0.0325 (4) |
| N1A | 0.9714 (2) | 0.65251 (19) | 0.12796 (6) | 0.0245 (5) |
| N2A | 0.9553 (2) | 0.6942 (2) | 0.03985 (6) | 0.0299 (5) |
| N3A | 1.0875 (2) | 0.41201 (19) | 0.07324 (6) | 0.0242 (5) |
| C2A | 1.0060 (2) | 0.5827 (2) | 0.08148 (7) | 0.0240 (6) |

| | | | | |
|---|---|---|---|---|
| C4A | 1.1324 (2) | 0.3082 (2) | 0.11596 (7) | 0.0251 (6) |
| C5A | 1.0999 (3) | 0.3628 (2) | 0.16628 (7) | 0.0282 (6) |
| C6A | 1.0193 (3) | 0.5400 (2) | 0.16970 (7) | 0.0258 (6) |
| C7A | 1.2546 (3) | 0.0744 (3) | 0.06061 (8) | 0.0364 (7) |
| C8A | 1.0194 (3) | 0.5193 (3) | 0.26230 (7) | 0.0425 (8) |
| O1B | 1.23977 (18) | 0.52942 (18) | 0.36928 (5) | 0.0318 (4) |
| O2B | 0.76374 (17) | 0.84403 (18) | 0.47739 (5) | 0.0303 (4) |
| N1B | 1.0400 (2) | 0.7436 (2) | 0.50440 (6) | 0.0250 (5) |
| N2B | 1.3186 (2) | 0.6512 (2) | 0.53585 (6) | 0.0290 (5) |
| N3B | 1.2895 (2) | 0.5829 (2) | 0.45247 (5) | 0.0244 (5) |
| C2B | 1.2140 (3) | 0.6598 (2) | 0.49642 (7) | 0.0244 (6) |
| C4B | 1.1779 (3) | 0.5992 (2) | 0.41485 (7) | 0.0243 (6) |
| C5B | 0.9958 (3) | 0.6842 (2) | 0.41835 (7) | 0.0268 (6) |
| C6B | 0.9336 (3) | 0.7556 (2) | 0.46478 (7) | 0.0251 (6) |
| C7B | 1.4261 (3) | 0.4377 (3) | 0.36371 (8) | 0.0355 (7) |
| C8B | 0.6364 (3) | 0.8702 (3) | 0.43854 (7) | 0.0309 (6) |
| O3A | 0.79403 (19) | 0.97701 (17) | 0.15298 (5) | 0.0306 (4) |
| O4A | 0.7717 (2) | 1.03505 (17) | 0.06735 (5) | 0.0372 (4) |
| N4A | 0.6470 (2) | 1.3700 (3) | 0.02930 (6) | 0.0298 (5) |
| C9A | 0.6511 (2) | 1.2676 (2) | 0.12255 (7) | 0.0240 (5) |
| C10A | 0.6112 (2) | 1.4017 (2) | 0.08103 (7) | 0.0251 (6) |
| C11A | 0.5288 (3) | 1.5756 (3) | 0.09344 (7) | 0.0290 (6) |
| C12A | 0.4867 (3) | 1.6146 (3) | 0.14425 (7) | 0.0315 (6) |
| C13A | 0.5262 (3) | 1.4836 (3) | 0.18544 (8) | 0.0321 (6) |
| C14A | 0.6066 (3) | 1.3120 (3) | 0.17417 (7) | 0.0279 (6) |
| C15A | 0.7426 (3) | 1.0846 (2) | 0.11184 (7) | 0.0259 (6) |
| O3B | 1.10809 (18) | 0.82786 (17) | 0.63163 (5) | 0.0302 (4) |
| O4B | 0.85924 (18) | 0.8829 (2) | 0.58677 (5) | 0.0333 (5) |
| N4B | 1.0645 (2) | 0.9650 (2) | 0.72764 (6) | 0.0355 (6) |
| C9B | 0.8187 (3) | 0.9691 (2) | 0.67164 (7) | 0.0243 (5) |
| C10B | 0.8820 (3) | 0.9998 (2) | 0.71877 (7) | 0.0251 (6) |
| C11B | 0.7504 (3) | 1.0696 (3) | 0.75827 (7) | 0.0293 (6) |
| C12B | 0.5670 (3) | 1.1126 (3) | 0.75049 (8) | 0.0313 (6) |
| C13B | 0.5050 (3) | 1.0903 (3) | 0.70327 (8) | 0.0315 (6) |
| C14B | 0.6303 (3) | 1.0186 (3) | 0.66481 (7) | 0.0300 (6) |
| C15B | 0.9429 (3) | 0.8870 (2) | 0.62876 (7) | 0.0258 (6) |
| H2A1 | 0.90370 | 0.80380 | 0.04380 | 0.0360* |
| H2A2 | 0.97430 | 0.65630 | 0.00910 | 0.0360* |
| H7A1 | 1.31410 | −0.04940 | 0.06330 | 0.0550* |
| H7A2 | 1.33590 | 0.15120 | 0.03920 | 0.0550* |
| H7A3 | 1.14050 | 0.07980 | 0.04500 | 0.0550* |
| H5A | 1.13060 | 0.28490 | 0.19560 | 0.0340* |
| H8A1 | 0.98330 | 0.59430 | 0.29070 | 0.0640* |
| H8A2 | 1.15070 | 0.47900 | 0.26080 | 0.0640* |
| H8A3 | 0.95170 | 0.41500 | 0.26780 | 0.0640* |
| H5B | 0.92150 | 0.69200 | 0.39110 | 0.0320* |
| H2B1 | 1.27320 | 0.69810 | 0.56440 | 0.0350* |
| H2B2 | 1.43160 | 0.59880 | 0.53280 | 0.0350* |
| H8B1 | 0.52000 | 0.93280 | 0.45240 | 0.0460* |

| | | | | |
|---|---|---|---|---|
| H8B2 | 0.61650 | 0.75350 | 0.42880 | 0.0460* |
| H8B3 | 0.68700 | 0.94200 | 0.40820 | 0.0460* |
| H7B1 | 1.45250 | 0.39360 | 0.32970 | 0.0530* |
| H7B2 | 1.43830 | 0.33640 | 0.39020 | 0.0530* |
| H7B3 | 1.51240 | 0.52150 | 0.36750 | 0.0530* |
| H3A | 0.84810 | 0.88000 | 0.14350 | 0.0460* |
| H7 | 0.722 (3) | 1.265 (3) | 0.0224 (9) | 0.057 (7)* |
| H11A | 0.50220 | 1.66620 | 0.06650 | 0.0350* |
| H12A | 0.43080 | 1.73080 | 0.15140 | 0.0380* |
| H13A | 0.49890 | 1.51140 | 0.22000 | 0.0390* |
| H14A | 0.63210 | 1.22300 | 0.20160 | 0.0330* |
| H16 | 0.653 (3) | 1.465 (3) | 0.0067 (9) | 0.046 (7)* |
| H4B | 0.93260 | 0.83400 | 0.56440 | 0.0500* |
| H11B | 0.78900 | 1.08660 | 0.79030 | 0.0350* |
| H12B | 0.48240 | 1.15760 | 0.77730 | 0.0380* |
| H13B | 0.38060 | 1.12330 | 0.69780 | 0.0380* |
| H14B | 0.58890 | 1.00230 | 0.63310 | 0.0360* |
| H4B1 | 1.14640 | 0.92230 | 0.70380 | 0.0430* |
| H4B2 | 1.09790 | 0.98560 | 0.75710 | 0.0430* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1A | 0.0400 (8) | 0.0249 (7) | 0.0303 (7) | 0.0042 (6) | −0.0089 (6) | −0.0023 (6) |
| O2A | 0.0483 (9) | 0.0293 (7) | 0.0203 (7) | −0.0006 (6) | −0.0080 (6) | −0.0041 (5) |
| N1A | 0.0279 (8) | 0.0238 (8) | 0.0213 (8) | −0.0010 (6) | −0.0036 (6) | −0.0018 (6) |
| N2A | 0.0427 (10) | 0.0238 (8) | 0.0205 (8) | 0.0073 (7) | −0.0042 (7) | −0.0035 (6) |
| N3A | 0.0249 (8) | 0.0212 (8) | 0.0255 (8) | 0.0018 (6) | −0.0041 (6) | −0.0016 (6) |
| C2A | 0.0226 (10) | 0.0262 (10) | 0.0230 (9) | −0.0018 (8) | −0.0023 (7) | −0.0027 (7) |
| C4A | 0.0241 (10) | 0.0220 (9) | 0.0292 (10) | −0.0020 (8) | −0.0055 (8) | −0.0005 (8) |
| C5A | 0.0337 (11) | 0.0274 (10) | 0.0234 (10) | −0.0018 (8) | −0.0092 (8) | 0.0024 (8) |
| C6A | 0.0273 (10) | 0.0277 (10) | 0.0238 (10) | −0.0052 (8) | −0.0055 (8) | −0.0037 (8) |
| C7A | 0.0461 (13) | 0.0269 (10) | 0.0337 (11) | 0.0092 (9) | −0.0060 (9) | −0.0063 (8) |
| C8A | 0.0650 (16) | 0.0404 (13) | 0.0218 (10) | −0.0009 (11) | −0.0123 (10) | 0.0003 (9) |
| O1B | 0.0296 (7) | 0.0409 (8) | 0.0255 (7) | 0.0022 (6) | −0.0056 (6) | −0.0106 (6) |
| O2B | 0.0225 (7) | 0.0416 (8) | 0.0264 (7) | 0.0017 (6) | −0.0062 (5) | −0.0037 (6) |
| N1B | 0.0224 (8) | 0.0311 (9) | 0.0214 (8) | −0.0019 (7) | −0.0042 (6) | −0.0022 (6) |
| N2B | 0.0230 (8) | 0.0428 (10) | 0.0212 (8) | 0.0038 (7) | −0.0053 (6) | −0.0088 (7) |
| N3B | 0.0252 (8) | 0.0281 (8) | 0.0202 (8) | −0.0037 (6) | −0.0026 (6) | −0.0028 (6) |
| C2B | 0.0236 (10) | 0.0272 (10) | 0.0224 (9) | −0.0053 (8) | −0.0017 (7) | −0.0008 (7) |
| C4B | 0.0282 (10) | 0.0265 (10) | 0.0194 (9) | −0.0078 (8) | −0.0020 (7) | −0.0030 (7) |
| C5B | 0.0256 (10) | 0.0333 (11) | 0.0226 (9) | −0.0037 (8) | −0.0062 (7) | −0.0037 (8) |
| C6B | 0.0228 (10) | 0.0278 (10) | 0.0243 (10) | −0.0032 (8) | −0.0036 (7) | 0.0011 (8) |
| C7B | 0.0308 (11) | 0.0460 (12) | 0.0298 (11) | 0.0033 (9) | −0.0038 (8) | −0.0136 (9) |
| C8B | 0.0242 (10) | 0.0394 (11) | 0.0296 (10) | −0.0029 (9) | −0.0080 (8) | −0.0007 (8) |
| O3A | 0.0376 (8) | 0.0260 (7) | 0.0255 (7) | 0.0048 (6) | −0.0033 (6) | −0.0013 (5) |
| O4A | 0.0541 (9) | 0.0290 (7) | 0.0252 (7) | 0.0094 (7) | −0.0050 (6) | −0.0049 (6) |
| N4A | 0.0387 (10) | 0.0255 (9) | 0.0225 (9) | 0.0059 (8) | −0.0037 (7) | −0.0023 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9A | 0.0210 (9) | 0.0260 (9) | 0.0248 (9) | −0.0010 (8) | −0.0025 (7) | −0.0040 (7) |
| C10A | 0.0217 (10) | 0.0297 (10) | 0.0241 (10) | 0.0003 (8) | −0.0034 (7) | −0.0064 (8) |
| C11A | 0.0292 (11) | 0.0270 (10) | 0.0299 (10) | 0.0039 (8) | −0.0072 (8) | −0.0029 (8) |
| C12A | 0.0324 (11) | 0.0279 (10) | 0.0336 (11) | 0.0043 (8) | −0.0047 (9) | −0.0085 (8) |
| C13A | 0.0332 (11) | 0.0363 (11) | 0.0263 (10) | 0.0013 (9) | −0.0014 (8) | −0.0092 (8) |
| C14A | 0.0266 (10) | 0.0301 (10) | 0.0260 (10) | −0.0017 (8) | −0.0016 (8) | −0.0016 (8) |
| C15A | 0.0254 (10) | 0.0278 (10) | 0.0240 (10) | −0.0017 (8) | −0.0029 (8) | −0.0019 (8) |
| O3B | 0.0248 (7) | 0.0375 (8) | 0.0274 (7) | −0.0005 (6) | −0.0022 (5) | −0.0034 (6) |
| O4B | 0.0282 (8) | 0.0494 (9) | 0.0217 (7) | 0.0018 (6) | −0.0026 (6) | −0.0090 (6) |
| N4B | 0.0276 (9) | 0.0528 (11) | 0.0283 (9) | −0.0019 (8) | −0.0064 (7) | −0.0134 (8) |
| C9B | 0.0260 (10) | 0.0242 (9) | 0.0216 (9) | −0.0034 (8) | −0.0003 (7) | 0.0004 (7) |
| C10B | 0.0257 (10) | 0.0244 (9) | 0.0251 (10) | −0.0044 (8) | −0.0022 (8) | −0.0006 (7) |
| C11B | 0.0323 (11) | 0.0319 (10) | 0.0241 (10) | −0.0029 (8) | −0.0014 (8) | −0.0069 (8) |
| C12B | 0.0318 (11) | 0.0295 (10) | 0.0316 (11) | −0.0041 (9) | 0.0054 (8) | −0.0070 (8) |
| C13B | 0.0241 (10) | 0.0343 (11) | 0.0355 (11) | −0.0009 (8) | −0.0003 (8) | −0.0066 (9) |
| C14B | 0.0267 (10) | 0.0347 (11) | 0.0287 (10) | −0.0010 (8) | −0.0045 (8) | −0.0049 (8) |
| C15B | 0.0257 (10) | 0.0293 (10) | 0.0218 (9) | −0.0038 (8) | −0.0021 (8) | 0.0002 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1A—C4A | 1.340 (2) | C7A—H7A2 | 0.9598 |
| O1A—C7A | 1.437 (2) | C7A—H7A1 | 0.9599 |
| O2A—C6A | 1.344 (2) | C7A—H7A3 | 0.9598 |
| O2A—C8A | 1.428 (2) | C8A—H8A2 | 0.9604 |
| O1B—C4B | 1.342 (2) | C8A—H8A1 | 0.9595 |
| O1B—C7B | 1.433 (3) | C8A—H8A3 | 0.9594 |
| O2B—C6B | 1.342 (2) | C4B—C5B | 1.387 (3) |
| O2B—C8B | 1.426 (2) | C5B—C6B | 1.367 (2) |
| O3A—C15A | 1.315 (2) | C5B—H5B | 0.9293 |
| O4A—C15A | 1.230 (2) | C7B—H7B1 | 0.9595 |
| O3A—H3A | 0.8200 | C7B—H7B3 | 0.9599 |
| O3B—C15B | 1.232 (3) | C7B—H7B2 | 0.9600 |
| O4B—C15B | 1.310 (2) | C8B—H8B3 | 0.9605 |
| O4B—H4B | 0.8193 | C8B—H8B2 | 0.9602 |
| N1A—C2A | 1.342 (2) | C8B—H8B1 | 0.9599 |
| N1A—C6A | 1.338 (2) | C9A—C15A | 1.469 (2) |
| N2A—C2A | 1.338 (2) | C9A—C10A | 1.407 (2) |
| N3A—C4A | 1.321 (2) | C9A—C14A | 1.398 (3) |
| N3A—C2A | 1.352 (2) | C10A—C11A | 1.401 (3) |
| N2A—H2A2 | 0.8605 | C11A—C12A | 1.364 (3) |
| N2A—H2A1 | 0.8598 | C12A—C13A | 1.387 (3) |
| N1B—C6B | 1.343 (2) | C13A—C14A | 1.374 (3) |
| N1B—C2B | 1.338 (3) | C11A—H11A | 0.9303 |
| N2B—C2B | 1.333 (2) | C12A—H12A | 0.9302 |
| N3B—C4B | 1.324 (2) | C13A—H13A | 0.9300 |
| N3B—C2B | 1.351 (2) | C14A—H14A | 0.9304 |
| N2B—H2B1 | 0.8608 | C9B—C15B | 1.475 (3) |
| N2B—H2B2 | 0.8604 | C9B—C10B | 1.402 (3) |
| N4A—C10A | 1.369 (2) | C9B—C14B | 1.399 (3) |

| | | | |
|---|---|---|---|
| N4A—H7 | 0.91 (2) | C10B—C11B | 1.406 (3) |
| N4A—H16 | 0.87 (2) | C11B—C12B | 1.364 (3) |
| N4B—C10B | 1.361 (3) | C12B—C13B | 1.382 (3) |
| N4B—H4B2 | 0.8602 | C13B—C14B | 1.368 (3) |
| N4B—H4B1 | 0.8601 | C11B—H11B | 0.9304 |
| C4A—C5A | 1.387 (2) | C12B—H12B | 0.9297 |
| C5A—C6A | 1.373 (2) | C13B—H13B | 0.9295 |
| C5A—H5A | 0.9299 | C14B—H14B | 0.9302 |
| O1B···C8A | 3.366 (2) | C7B···H2B2$^{viii}$ | 2.9499 |
| O2A···O3A | 3.1345 (18) | C8A···H5A | 2.5674 |
| O2A···C11B$^{i}$ | 3.386 (3) | C8B···H5B | 2.5643 |
| O2B···O4B | 3.0386 (18) | C8B···H14B$^{iii}$ | 2.6313 |
| O3A···O2A | 3.1345 (18) | C10B···H5A$^{ii}$ | 2.8828 |
| O3A···N1A | 2.6808 (19) | C11A···H7A1$^{xv}$ | 3.0702 |
| O3B···C8B$^{i}$ | 3.339 (2) | C11B···H5A$^{ii}$ | 2.8311 |
| O3B···N4B | 2.749 (2) | C11B···H12A$^{xiii}$ | 2.9790 |
| O3B···N2B | 3.075 (2) | C12A···H12B$^{xiii}$ | 2.8221 |
| O4A···N4A | 2.650 (2) | C12B···H13A$^{xiii}$ | 2.9326 |
| O4A···N2A | 2.818 (2) | C12B···H12A$^{xiii}$ | 2.9020 |
| O4B···C7B$^{ii}$ | 3.405 (3) | C13A···H12B$^{xiii}$ | 2.9162 |
| O4B···O2B | 3.0386 (18) | C13B···H4B1$^{xiv}$ | 3.0357 |
| O4B···C6B$^{i}$ | 3.320 (2) | C15A···H2A1 | 2.9083 |
| O4B···C6B | 3.348 (2) | C15A···H7 | 2.55 (2) |
| O4B···N1B | 2.603 (2) | C15B···H4B1 | 2.6288 |
| O1A···H11B$^{ii}$ | 2.8584 | C15B···H2B1 | 3.0282 |
| O1B···H8A1 | 2.8768 | H2A1···C7A$^{vi}$ | 3.0537 |
| O2A···H3A | 2.6478 | H2A1···C15A | 2.9083 |
| O2A···H11B$^{i}$ | 2.8917 | H2A1···H7A3$^{vi}$ | 2.4003 |
| O2B···H4B | 2.6746 | H2A1···O4A | 1.9706 |
| O3A···H4B2$^{i}$ | 2.5912 | H2A2···H7A3$^{vi}$ | 2.3895 |
| O3A···H12B$^{iii}$ | 2.7485 | H2A2···H2A2$^{vi}$ | 2.3863 |
| O3A···H14A | 2.4228 | H2A2···C7A$^{vi}$ | 3.0049 |
| O3B···H8B3$^{i}$ | 2.4721 | H2A2···H7$^{vii}$ | 2.3871 |
| O3B···H2B1 | 2.2238 | H2A2···N4A$^{vii}$ | 2.8055 |
| O3B···H4B1 | 2.1213 | H2A2···N3A$^{vi}$ | 2.3451 |
| O4A···H7 | 1.97 (2) | H2A2···C2A$^{vi}$ | 3.0643 |
| O4A···H7A3$^{iv}$ | 2.7343 | H7A1···C11A$^{xvi}$ | 3.0702 |
| O4A···H2A1 | 1.9706 | H7A1···H11A$^{xvi}$ | 2.3548 |
| O4B···H7B2$^{ii}$ | 2.8459 | H3A···C6A | 2.7105 |
| O4B···H14B | 2.3016 | H3A···C2A | 2.8994 |
| N1A···C15A | 3.413 (2) | H3A···N1A | 1.8629 |
| N1A···O3A | 2.6808 (19) | H3A···O2A | 2.6478 |
| N1B···O4B | 2.603 (2) | H7A2···N3A | 2.6166 |

| | | | |
|---|---|---|---|
| N2A···C11A$^{v}$ | 3.429 (3) | H7A2···N4A$^{xii}$ | 2.9272 |
| N2A···O4A | 2.818 (2) | H4B···C2B | 2.8079 |
| N2A···N3A$^{vi}$ | 3.166 (2) | H4B···N1B | 1.8054 |
| N2A···N4A$^{vii}$ | 3.223 (2) | H4B···C6B | 2.6964 |
| N2A···C7A$^{vi}$ | 3.355 (3) | H4B···O2B | 2.6746 |
| N2B···O3B | 3.075 (2) | H4B···H2B1 | 2.5495 |
| N2B···C7B$^{viii}$ | 3.324 (3) | H7A3···H2A2$^{vi}$ | 2.3895 |
| N2B···N3B$^{viii}$ | 3.165 (2) | H7A3···H2A1$^{vi}$ | 2.4003 |
| N3A···N2A$^{vi}$ | 3.166 (2) | H7A3···O4A$^{v}$ | 2.7343 |
| N3B···N2B$^{viii}$ | 3.165 (2) | H7A3···N2A$^{vi}$ | 2.7065 |
| N4A···N4A$^{ix}$ | 3.100 (3) | H7A3···N3A | 2.6110 |
| N4A···O4A | 2.650 (2) | H5A···C11B$^{ii}$ | 2.8311 |
| N4A···N2A$^{vii}$ | 3.223 (2) | H5A···H8A2 | 2.3603 |
| N4B···C13B$^{x}$ | 3.424 (3) | H5A···C10B$^{ii}$ | 2.8828 |
| N4B···O3B | 2.749 (2) | H5A···N4B$^{ii}$ | 2.9299 |
| N1A···H3A | 1.8629 | H5A···C8A | 2.5674 |
| N1B···H4B | 1.8054 | H5A···H8A3 | 2.3629 |
| N2A···H7$^{vii}$ | 2.72 (2) | H5B···H8B3 | 2.3881 |
| N2A···H7A3$^{vi}$ | 2.7065 | H5B···C8B | 2.5643 |
| N2B···H7B2$^{viii}$ | 2.7691 | H5B···H8B2 | 2.3237 |
| N3A···H16$^{vii}$ | 2.79 (2) | H8A1···O1B | 2.8768 |
| N3A···H7A3 | 2.6110 | H7···H2A2$^{vii}$ | 2.3871 |
| N3A···H2A2$^{vi}$ | 2.3450 | H7···O4A | 1.97 (2) |
| N3A···H7A2 | 2.6166 | H7···N2A$^{vii}$ | 2.72 (2) |
| N3B···H8B2$^{x}$ | 2.8142 | H7···C15A | 2.55 (2) |
| N3B···H7B3 | 2.6125 | H8A2···C5A | 2.7552 |
| N3B···H7B2 | 2.6081 | H8A2···H5A | 2.3603 |
| N3B···H2B2$^{viii}$ | 2.3290 | H8A3···H5A | 2.3629 |
| N4A···H7A2$^{xi}$ | 2.9272 | H8A3···N4B$^{ii}$ | 2.8226 |
| N4A···H16$^{ix}$ | 2.58 (2) | H8A3···C5A | 2.7598 |
| N4A···H11A$^{ix}$ | 2.8681 | H2B1···H4B | 2.5495 |
| N4A···H2A2$^{vii}$ | 2.8055 | H2B1···C7B$^{viii}$ | 3.0355 |
| N4B···H5A$^{ii}$ | 2.9299 | H2B1···H7B2$^{viii}$ | 2.4952 |
| N4B···H8A3$^{ii}$ | 2.8226 | H2B1···C15B | 3.0282 |
| N4B···H13B$^{x}$ | 2.7127 | H2B1···O3B | 2.2238 |
| C2A···C10A$^{v}$ | 3.331 (2) | H2B2···N3B$^{viii}$ | 2.3290 |
| C2A···C11A$^{v}$ | 3.458 (3) | H2B2···C2B$^{viii}$ | 3.0727 |
| C2B···C5B$^{ii}$ | 3.558 (2) | H2B2···C7B$^{viii}$ | 2.9499 |
| C2B···C6B$^{ii}$ | 3.401 (2) | H2B2···H2B2$^{viii}$ | 2.4004 |
| C4A···C9A$^{v}$ | 3.539 (2) | H2B2···H7B2$^{viii}$ | 2.4198 |
| C4A···C15A$^{v}$ | 3.488 (3) | H8B1···H14B$^{iii}$ | 2.4098 |
| C5A···C12A$^{xii}$ | 3.537 (3) | H11A···H16 | 2.3411 |

| | | | |
|---|---|---|---|
| C5A···C13A$^{xii}$ | 3.447 (3) | H11A···H7A1$^{xv}$ | 2.3548 |
| C5B···C15B$^{i}$ | 3.341 (2) | H11A···N4A$^{ix}$ | 2.8681 |
| C5B···C2B$^{ii}$ | 3.558 (2) | H11B···H4B2 | 2.3639 |
| C5B···C9B$^{i}$ | 3.559 (2) | H11B···H12A$^{xiii}$ | 2.4408 |
| C6A···C12A$^{xii}$ | 3.493 (3) | H11B···O1A$^{ii}$ | 2.8584 |
| C6B···O4B | 3.348 (2) | H11B···O2A$^{i}$ | 2.8917 |
| C6B···O4B$^{i}$ | 3.320 (2) | H8B2···H5B | 2.3237 |
| C6B···C2B$^{ii}$ | 3.401 (2) | H8B2···N3B$^{xiv}$ | 2.8142 |
| C6B···C15B$^{i}$ | 3.514 (2) | H8B2···C5B | 2.7274 |
| C7A···N2A$^{vi}$ | 3.355 (3) | H12A···C12B$^{xiii}$ | 2.9019 |
| C7B···O4B$^{ii}$ | 3.405 (3) | H12A···C11B$^{xiii}$ | 2.9790 |
| C7B···N2B$^{viii}$ | 3.324 (3) | H12A···H11B$^{xiii}$ | 2.4408 |
| C8A···O1B | 3.366 (2) | H12A···H12B$^{xiii}$ | 2.2849 |
| C8B···O3B$^{i}$ | 3.339 (2) | H12B···O3A$^{iii}$ | 2.7485 |
| C8B···C14B$^{iii}$ | 3.447 (3) | H12B···C12A$^{xiii}$ | 2.8220 |
| C9A···C4A$^{iv}$ | 3.539 (2) | H12B···C13A$^{xiii}$ | 2.9162 |
| C9B···C5B$^{i}$ | 3.559 (2) | H12B···H12A$^{xiii}$ | 2.2849 |
| C10A···C2A$^{iv}$ | 3.331 (2) | H12B···H13A$^{xiii}$ | 2.4834 |
| C11A···N2A$^{iv}$ | 3.429 (3) | H8B3···H5B | 2.3881 |
| C11A···C2A$^{iv}$ | 3.458 (3) | H8B3···O3B$^{i}$ | 2.4721 |
| C11B···O2A$^{i}$ | 3.386 (3) | H8B3···C5B | 2.7678 |
| C12A···C5A$^{xi}$ | 3.537 (3) | H8B3···H14B$^{iii}$ | 2.3522 |
| C12A···C6A$^{xi}$ | 3.493 (3) | H13A···H12B$^{xiii}$ | 2.4834 |
| C12A···C12B$^{xiii}$ | 3.517 (3) | H13A···C12B$^{xiii}$ | 2.9326 |
| C12B···C13A$^{xiii}$ | 3.526 (3) | H13B···N4B$^{xiv}$ | 2.7127 |
| C12B···C12A$^{xiii}$ | 3.517 (3) | H13B···H4B1$^{xiv}$ | 2.3894 |
| C13A···C5A$^{xi}$ | 3.447 (3) | H14A···H4B2$^{i}$ | 2.5915 |
| C13A···C12B$^{xiii}$ | 3.526 (3) | H14A···O3A | 2.4228 |
| C13B···N4B$^{xiv}$ | 3.424 (3) | H14B···C8B$^{iii}$ | 2.6313 |
| C14B···C8B$^{iii}$ | 3.447 (3) | H14B···O4B | 2.3016 |
| C15A···N1A | 3.413 (2) | H14B···H8B1$^{iii}$ | 2.4098 |
| C15A···C4A$^{iv}$ | 3.488 (3) | H14B···H8B3$^{iii}$ | 2.3522 |
| C15B···C5B$^{i}$ | 3.341 (2) | H7B2···N2B$^{viii}$ | 2.7691 |
| C15B···C6B$^{i}$ | 3.514 (2) | H7B2···O4B$^{ii}$ | 2.8459 |
| C2A···H3A | 2.8994 | H7B2···H2B1$^{viii}$ | 2.4952 |
| C2A···H2A2$^{vi}$ | 3.0643 | H7B2···H2B2$^{viii}$ | 2.4198 |
| C2B···H2B2$^{viii}$ | 3.0727 | H7B2···N3B | 2.6081 |
| C2B···H4B | 2.8079 | H16···H16$^{ix}$ | 2.28 (3) |
| C5A···H8A3 | 2.7598 | H16···H11A | 2.3411 |
| C5A···H8A2 | 2.7552 | H16···N4A$^{ix}$ | 2.58 (2) |
| C5B···H8B3 | 2.7678 | H16···N3A$^{vii}$ | 2.79 (2) |

| | | | |
|---|---|---|---|
| C5B···H8B2 | 2.7274 | H7B3···N3B | 2.6125 |
| C6A···H3A | 2.7105 | H4B1···O3B | 2.1213 |
| C6B···H4B | 2.6964 | H4B1···C13B$^{x}$ | 3.0357 |
| C7A···H2A2$^{vi}$ | 3.0049 | H4B1···C15B | 2.6288 |
| C7A···H2A1$^{vi}$ | 3.0537 | H4B1···H13B$^{x}$ | 2.3894 |
| C7B···H2B1$^{viii}$ | 3.0355 | H4B2···H11B | 2.3639 |
| C4A—O1A—C7A | 118.05 (15) | C4B—C5B—H5B | 122.42 |
| C6A—O2A—C8A | 118.02 (14) | C6B—C5B—H5B | 122.43 |
| C4B—O1B—C7B | 117.81 (15) | O1B—C7B—H7B2 | 109.47 |
| C6B—O2B—C8B | 117.41 (15) | O1B—C7B—H7B3 | 109.50 |
| C15A—O3A—H3A | 109.45 | O1B—C7B—H7B1 | 109.50 |
| C15B—O4B—H4B | 109.42 | H7B2—C7B—H7B3 | 109.50 |
| C2A—N1A—C6A | 116.21 (14) | H7B1—C7B—H7B2 | 109.40 |
| C2A—N3A—C4A | 114.84 (15) | H7B1—C7B—H7B3 | 109.45 |
| C2A—N2A—H2A1 | 120.01 | H8B1—C8B—H8B2 | 109.51 |
| C2A—N2A—H2A2 | 120.03 | O2B—C8B—H8B3 | 109.49 |
| H2A1—N2A—H2A2 | 119.95 | O2B—C8B—H8B2 | 109.50 |
| C2B—N1B—C6B | 116.94 (16) | O2B—C8B—H8B1 | 109.46 |
| C2B—N3B—C4B | 114.73 (16) | H8B1—C8B—H8B3 | 109.45 |
| H2B1—N2B—H2B2 | 119.91 | H8B2—C8B—H8B3 | 109.41 |
| C2B—N2B—H2B1 | 120.04 | C10A—C9A—C15A | 120.50 (15) |
| C2B—N2B—H2B2 | 120.05 | C14A—C9A—C15A | 120.32 (16) |
| H7—N4A—H16 | 118 (2) | C10A—C9A—C14A | 119.16 (15) |
| C10A—N4A—H7 | 116.0 (15) | N4A—C10A—C11A | 118.55 (16) |
| C10A—N4A—H16 | 116.8 (15) | C9A—C10A—C11A | 118.15 (16) |
| C10B—N4B—H4B1 | 120.00 | N4A—C10A—C9A | 123.30 (15) |
| C10B—N4B—H4B2 | 120.00 | C10A—C11A—C12A | 121.29 (19) |
| H4B1—N4B—H4B2 | 120.01 | C11A—C12A—C13A | 121.0 (2) |
| N1A—C2A—N2A | 116.23 (14) | C12A—C13A—C14A | 118.69 (19) |
| N1A—C2A—N3A | 125.95 (16) | C9A—C14A—C13A | 121.71 (18) |
| N2A—C2A—N3A | 117.82 (16) | O4A—C15A—C9A | 122.52 (16) |
| N3A—C4A—C5A | 124.69 (14) | O3A—C15A—C9A | 115.63 (15) |
| O1A—C4A—N3A | 119.51 (15) | O3A—C15A—O4A | 121.84 (15) |
| O1A—C4A—C5A | 115.79 (15) | C10A—C11A—H11A | 119.34 |
| C4A—C5A—C6A | 115.23 (16) | C12A—C11A—H11A | 119.36 |
| O2A—C6A—C5A | 125.55 (16) | C13A—C12A—H12A | 119.48 |
| O2A—C6A—N1A | 111.41 (14) | C11A—C12A—H12A | 119.53 |
| N1A—C6A—C5A | 123.04 (16) | C12A—C13A—H13A | 120.66 |
| C6A—C5A—H5A | 122.42 | C14A—C13A—H13A | 120.65 |
| C4A—C5A—H5A | 122.35 | C9A—C14A—H14A | 119.12 |
| O1A—C7A—H7A2 | 109.37 | C13A—C14A—H14A | 119.18 |
| H7A1—C7A—H7A3 | 109.49 | C14B—C9B—C15B | 118.51 (17) |
| O1A—C7A—H7A3 | 109.51 | C10B—C9B—C14B | 118.82 (17) |
| H7A1—C7A—H7A2 | 109.42 | C10B—C9B—C15B | 122.68 (19) |
| O1A—C7A—H7A1 | 109.53 | N4B—C10B—C11B | 119.07 (17) |
| H7A2—C7A—H7A3 | 109.50 | C9B—C10B—C11B | 118.18 (19) |
| H8A2—C8A—H8A3 | 109.48 | N4B—C10B—C9B | 122.75 (17) |
| H8A1—C8A—H8A3 | 109.47 | C10B—C11B—C12B | 121.16 (18) |

| | | | |
|---|---|---|---|
| O2A—C8A—H8A3 | 109.55 | C11B—C12B—C13B | 121.0 (2) |
| H8A1—C8A—H8A2 | 109.44 | C12B—C13B—C14B | 118.7 (2) |
| O2A—C8A—H8A2 | 109.46 | C9B—C14B—C13B | 122.10 (18) |
| O2A—C8A—H8A1 | 109.42 | O3B—C15B—C9B | 124.16 (17) |
| N2B—C2B—N3B | 118.31 (18) | O4B—C15B—C9B | 112.92 (18) |
| N1B—C2B—N2B | 116.21 (16) | O3B—C15B—O4B | 122.92 (16) |
| N1B—C2B—N3B | 125.48 (17) | C10B—C11B—H11B | 119.44 |
| N3B—C4B—C5B | 125.03 (16) | C12B—C11B—H11B | 119.40 |
| O1B—C4B—N3B | 119.61 (17) | C11B—C12B—H12B | 119.59 |
| O1B—C4B—C5B | 115.36 (17) | C13B—C12B—H12B | 119.46 |
| C4B—C5B—C6B | 115.15 (18) | C12B—C13B—H13B | 120.60 |
| O2B—C6B—N1B | 111.18 (15) | C14B—C13B—H13B | 120.71 |
| O2B—C6B—C5B | 126.17 (18) | C9B—C14B—H14B | 118.94 |
| N1B—C6B—C5B | 122.65 (19) | C13B—C14B—H14B | 118.95 |
| C7A—O1A—C4A—N3A | 1.2 (2) | C4B—C5B—C6B—O2B | 179.87 (16) |
| C7A—O1A—C4A—C5A | −179.01 (17) | C14A—C9A—C15A—O4A | 174.43 (19) |
| C8A—O2A—C6A—N1A | 179.08 (17) | C15A—C9A—C10A—N4A | 2.6 (2) |
| C8A—O2A—C6A—C5A | −1.1 (3) | C15A—C9A—C10A—C11A | −178.15 (17) |
| C7B—O1B—C4B—N3B | 0.6 (2) | C14A—C9A—C10A—N4A | −178.92 (17) |
| C7B—O1B—C4B—C5B | −179.14 (16) | C14A—C9A—C10A—C11A | 0.3 (2) |
| C8B—O2B—C6B—C5B | −1.5 (3) | C10A—C9A—C14A—C13A | −0.5 (3) |
| C8B—O2B—C6B—N1B | 178.82 (15) | C15A—C9A—C14A—C13A | 177.98 (19) |
| C2A—N1A—C6A—O2A | −179.97 (17) | C10A—C9A—C15A—O4A | −7.1 (3) |
| C6A—N1A—C2A—N3A | −1.4 (2) | C14A—C9A—C15A—O3A | −6.4 (3) |
| C6A—N1A—C2A—N2A | 178.93 (16) | C10A—C9A—C15A—O3A | 172.05 (16) |
| C2A—N1A—C6A—C5A | 0.2 (3) | C9A—C10A—C11A—C12A | −0.5 (3) |
| C4A—N3A—C2A—N1A | 1.1 (2) | N4A—C10A—C11A—C12A | 178.82 (19) |
| C4A—N3A—C2A—N2A | −179.32 (14) | C10A—C11A—C12A—C13A | 0.8 (3) |
| C2A—N3A—C4A—O1A | −179.60 (14) | C11A—C12A—C13A—C14A | −0.9 (3) |
| C2A—N3A—C4A—C5A | 0.6 (2) | C12A—C13A—C14A—C9A | 0.8 (3) |
| C6B—N1B—C2B—N3B | −1.7 (3) | C10B—C9B—C15B—O3B | 4.9 (3) |
| C2B—N1B—C6B—C5B | 1.0 (2) | C10B—C9B—C15B—O4B | −175.17 (15) |
| C2B—N1B—C6B—O2B | −179.28 (14) | C14B—C9B—C15B—O3B | −175.29 (17) |
| C6B—N1B—C2B—N2B | 179.12 (15) | C14B—C9B—C15B—O4B | 4.7 (2) |
| C4B—N3B—C2B—N2B | −179.14 (15) | C15B—C9B—C10B—N4B | 3.4 (2) |
| C4B—N3B—C2B—N1B | 1.7 (2) | C15B—C9B—C10B—C11B | −176.60 (16) |
| C2B—N3B—C4B—C5B | −1.0 (2) | C10B—C9B—C14B—C13B | −2.2 (3) |
| C2B—N3B—C4B—O1B | 179.26 (15) | C15B—C9B—C14B—C13B | 177.97 (18) |
| O1A—C4A—C5A—C6A | 178.51 (17) | C14B—C9B—C10B—N4B | −176.38 (17) |
| N3A—C4A—C5A—C6A | −1.7 (3) | C14B—C9B—C10B—C11B | 3.6 (2) |
| C4A—C5A—C6A—N1A | 1.3 (3) | N4B—C10B—C11B—C12B | 177.68 (18) |
| C4A—C5A—C6A—O2A | −178.56 (18) | C9B—C10B—C11B—C12B | −2.3 (3) |
| N3B—C4B—C5B—C6B | 0.5 (3) | C10B—C11B—C12B—C13B | −0.6 (3) |
| O1B—C4B—C5B—C6B | −179.79 (15) | C11B—C12B—C13B—C14B | 2.0 (3) |
| C4B—C5B—C6B—N1B | −0.4 (2) | C12B—C13B—C14B—C9B | −0.6 (3) |

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*+1; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+2, −*z*+1; (iv) *x*, *y*+1, *z*; (v) *x*, *y*−1, *z*; (vi) −*x*+2, −*y*+1, −*z*; (vii) −*x*+2, −*y*+2, −*z*; (viii) −*x*+3, −*y*+1, −*z*+1; (ix) −*x*+1, −*y*+3, −*z*; (x) *x*+1, *y*, *z*; (xi) *x*−1, *y*+1, *z*; (xii) *x*+1, *y*−1, *z*; (xiii) −*x*+1, −*y*+3, −*z*+1; (xiv) *x*−1, *y*, *z*; (xv) *x*−1, *y*+2, *z*; (xvi) *x*+1, *y*−2, *z*.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2A—H2A1···O4A | 0.86 | 1.97 | 2.818 (2) | 169 |
| N2A—H2A2···N3A$^{vi}$ | 0.86 | 2.35 | 3.166 (2) | 160 |
| O3A—H3A···N1A | 0.82 | 1.86 | 2.6808 (19) | 175 |
| O4B—H4B···N1B | 0.82 | 1.81 | 2.603 (2) | 164 |
| N4A—H7···O4A | 0.91 (2) | 1.97 (2) | 2.650 (2) | 130 (2) |
| N2B—H2B1···O3B | 0.86 | 2.22 | 3.075 (2) | 170 |
| N2B—H2B2···N3B$^{viii}$ | 0.86 | 2.33 | 3.165 (2) | 164 |
| N4A—H16···N4A$^{ix}$ | 0.87 (2) | 2.58 (2) | 3.100 (3) | 119.7 (18) |
| N4B—H4B1···O3B | 0.86 | 2.12 | 2.749 (2) | 129 |
| N4B—H4B2···O3A$^{i}$ | 0.86 | 2.59 | 3.450 (2) | 176 |
| C8B—H8B3···O3B$^{i}$ | 0.96 | 2.47 | 3.339 (2) | 150 |
| C14A—H14A···O3A | 0.93 | 2.42 | 2.747 (3) | 100 |
| C14B—H14B···O4B | 0.93 | 2.30 | 2.655 (2) | 102 |

Symmetry codes: (vi) −*x*+2, −*y*+1, −*z*; (viii) −*x*+3, −*y*+1, −*z*+1; (ix) −*x*+1, −*y*+3, −*z*; (i) −*x*+2, −*y*+2, −*z*+1.

**Fig. 1**

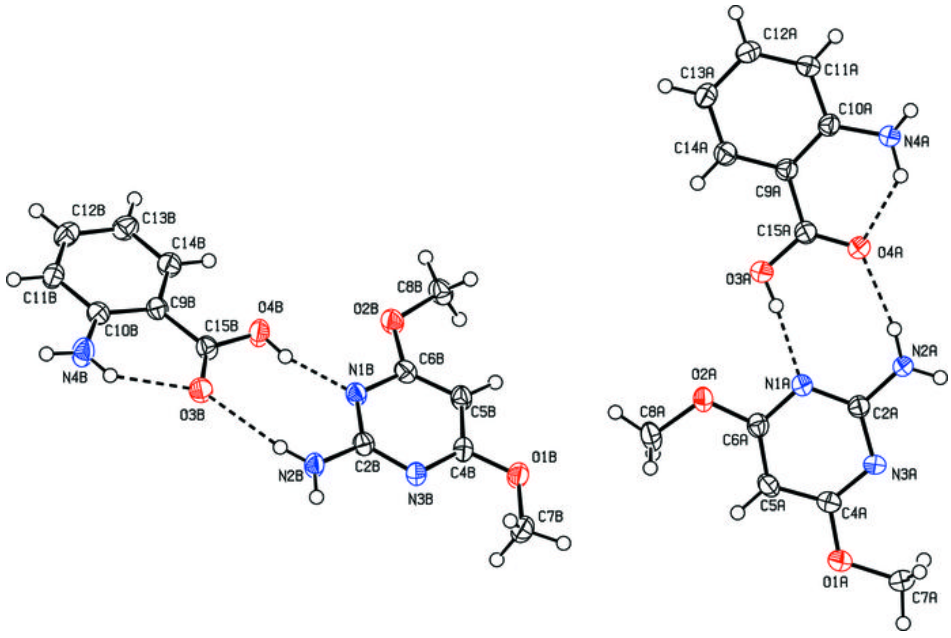
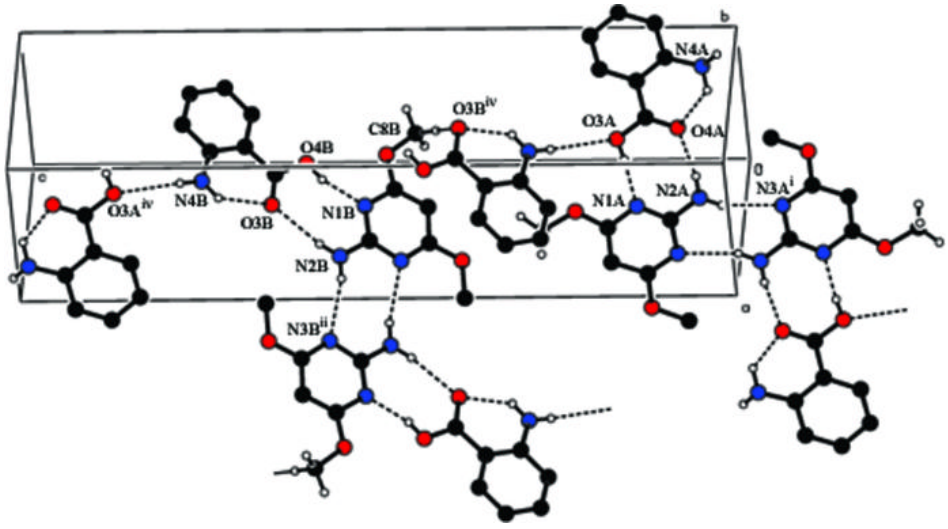

C12B
C13B
C14B
C11B
C9B
C10B
N4B
C15B
O4B
O3B
O2B
C8B
N1B
C6B
C5B
N2B
C2B
C4B
N3B
O1B
C7B
C12A
C13A
C11A
C10A
N4A
C14A
C9A
C15A
O4A
O3A
N2A
O2A
N1A
C8A
C2A
C6A
N3A
C5A
C4A
O1A
C7A

**Fig. 2**


b
N4A
O3B$^{iv}$
C8B
O3A
O4A
O4B
c
O3A$^{iv}$
N4B
O3B
N1B
N2B
N3B$^{ii}$
N1A
N2A
0
N3A$^{i}$
a