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# 6-Amino-3,4-dimethyl-1,2,4-triazin-1-ium 2-anilinobenzoate–3-amino-5,6-dimethyl-1,2,4-triazine (1/1)

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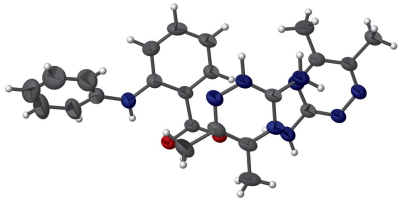
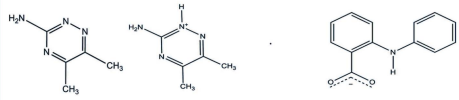
**Structural data**: full structural data are available from iucrdata.iucr.org

In the title molecular salt, $C_5H_9N_4^+\cdot C_{13}H_{10}NO_2^-\cdot C_5H_8N_4$, the asymmetric unit consists of a 6-amino-3,4-dimethyl-1,2,4-triazin-1-ium cation, a 2-anilinobenzoate anion and a neutral 3-amino-5,6-dimethyl-1,2,4-triazine molecule. The typical intramolecular N···H—O hydrogen bond is observed in the 2-anilinobenzoate anion. In the crystal, the protonated N atom and the 3-amino group are hydrogen bonded to the carboxylate oxygen atoms *via* a pair of N—H···O hydrogen bonds, forming an $R_2^2(8)$ ring motif. These motifs are further linked with adjacent neutral 3-amino-5,6-dimethyl-1,2,4-triazine molecules by N—H···O and N—H···N hydrogen bonds to produce centrosymmetric six-membered units, enclosing $R_2^2(8)$ and $R_4^3(9)$ ring motifs. They are reinforced by a C—H···N hydrogen bond and stack up the *b*-axis direction.

**3D view**



**Chemical scheme**



## Structure description

Heterocyclic compounds play a role in the design of new drugs and materials (García-Valverde & Torroba, 2005; Kumar *et al.*, 2014). 1,2,4-Triazines and their analogues occupy a important position in medicinal chemistry as a result of their pharmacological activities (Abdel-Monem, 2010). Drugs containing a 1,2,4-triazine ring are present in natural and many synthetic products, for example azaribine and lamotrigine, that have important biological activities. The crystal structure of 3-amino-5,6-dimethyl-1,2,4-triazine (Wu *et al.*, 2012) and 3-amino-5,6-dimethyl-1,2,4-triazin-2-ium nitrate (Bel Haj Salah *et al.*, 2013) have been reported. In order to study potential hydrogen-bonding interactions, the crystal structure determination of the title molecular salt was carried out.

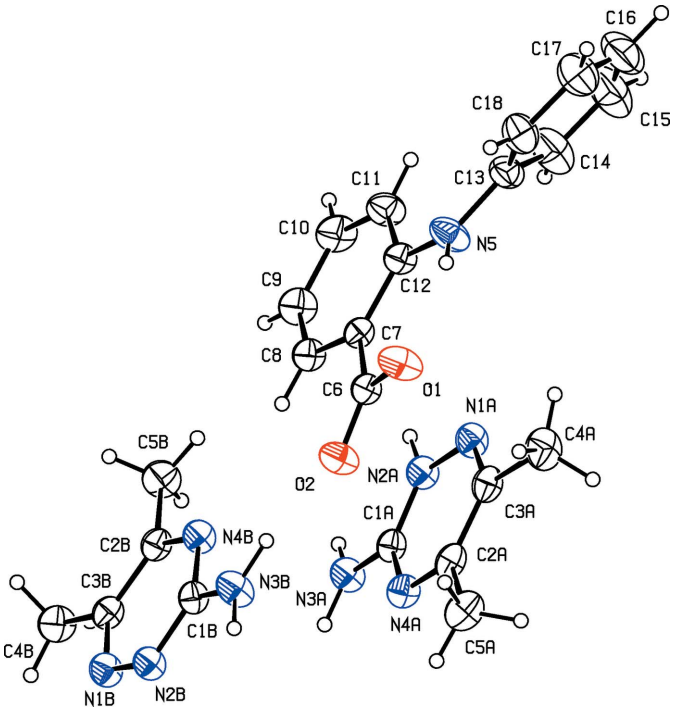



**Figure 1**
A view of the molecular structure of the title molecular salt, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

The structure of the title molecular salt is illustrated in Fig. 1. It is composed of a 6-amino-3,4-dimethyl-1,2,4-triazinium cation, a 2-anilinobenzoate anion and one neutral 3-amino-5,6-dimethyl-1,2,4-triazine molecule. Proton transfer from one of the carboxylate oxygen atoms (O1) to atom N2*A* of the cation results in widening of the C1*A*—N2*A*—N1*A* angle of the triazinium ring to 123.40 (13)°, compared to the corresponding angle of 117.9 (3)° in neutral 3-amino-5,6-dimethyl-1,2,4-triazine (Wu *et al.*, 2012), and that of the neutral 3-amino-5,6-dimethyl-1,2,4-triazine molecule present in the title molecular salt, where angle N1*B*—N2*B*—C1*B* is 117.46 (12) °. The dihedral angle between the rings in the anion is 44.55 (11)°. There is a typical intramolecular hydrogen bond N5—H1*N*5···O1 between the NH and carboxylate group of the 2-anilinobenzoate anion, forming an *S*(6) ring motif (Fig. 1).

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N5—H5*N*···O1 | 0.85 (2) | 1.92 (2) | 2.619 (2) | 138 (2) |
| N2*A*—H2*AA*···O1$^{i}$ | 1.02 (2) | 1.58 (2) | 2.593 (2) | 179 (2) |
| N3*A*—H3*AA*···O2$^{i}$ | 0.93 (2) | 1.90 (2) | 2.834 (2) | 175 (2) |
| N3*A*—H3*AB*···N1*B*$^{ii}$ | 0.92 (2) | 2.07 (2) | 2.986 (2) | 172 (2) |
| N3*B*—H3*BA*···N2*B*$^{iii}$ | 0.89 (2) | 2.20 (2) | 3.089 (2) | 179 (2) |
| N3*B*—H3*BB*···O2 | 0.97 (2) | 1.98 (2) | 2.915 (2) | 163 (2) |
| C4*B*—H4*BC*···N4*A*$^{ii}$ | 0.96 | 2.52 | 3.483 (2) | 177 |

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

In the crystal, the protonated N atom, N2*A*, and the 3-amino group (N3*A*) are hydrogen bonded to the carboxylate oxygen atoms (O1 and O2) *via* a pair of N2*A*—H1*N*2···O1$^{i}$ and N3*A*—H1*NA*···O2$^{i}$ hydrogen bonds, forming an $R_2^2$ (8) ring motif (Table 1 and Fig. 2). These motifs are further linked with adjacent neutral 3-amino-5,6-dimethyl-1,2,4-triazine molecules by N3*B*—H2*NB*···O2, N3*A*—H2*NA*···N1*B*$^{ii}$ and N3*B*—H1*NB*···N2*B*$^{iii}$ hydrogen bonds to produce a complementary *DDAA* (*D* = donor, *A* = acceptor) hydrogen-bonded array (Table 1 and Fig. 2). These centrosymmetric six-membered units enclose $R_2^2(8)$ and $R_4^3(9)$ ring motifs, are reinforced by a C—H···N hydrogen bond, and stack up the *b*-axis direction (Table 1, Fig. 3).

## Synthesis and crystallization

A hot methanol solution (20 ml) of 3-amino-5,6-dimethyl-1,2,4-triazine (62 mg, Aldrich) and 2-anilinobenzoic acid

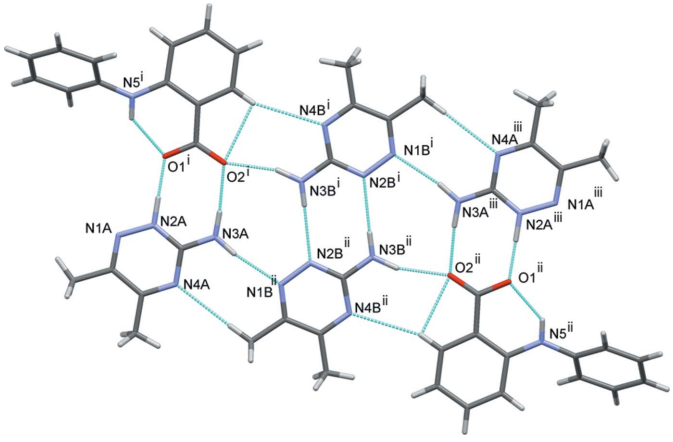



**Figure 2**
A view of the hydrogen bonded six-membered unit, involving the neutral triazine molecule, the triazinium cation and the benzoate anion. Hydrogen bonds are shown as dashed lines [symmetry codes for this figure are: (i) $x+1, y, z$; (ii) $-x+1, -y, z$; (iii) $-x+2, -y, -z$], and H atoms not involved in hydrogen bonding have been omitted for clarity.

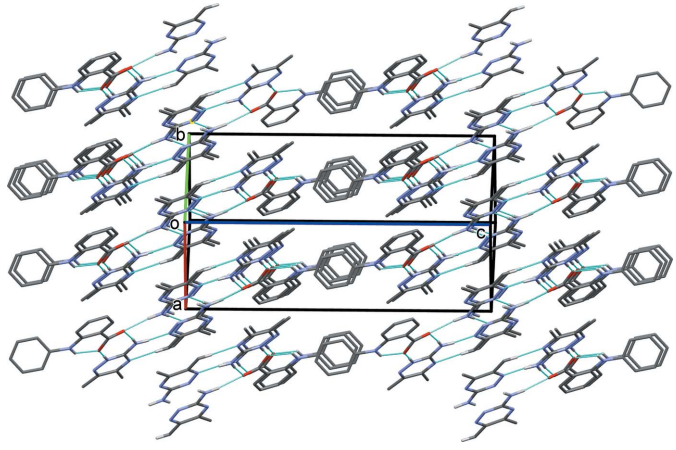



**Figure 3**
The crystal packing of the title molecular salt, viewed along the normal to (110). H atoms not involved in hydrogen bonding (dashed lines; see Table 1) have been omitted for clarity.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{13}H_{10}NO_2{\cdot}C_5H_9N_4{\cdot}C_5H_8N_4$ |
| $M_r$ | 461.53 |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 293 |
| $a, b, c$ (Å) | 7.5018 (3), 7.6661 (3), 21.4332 (9) |
| $\alpha, \beta, \gamma$ (°) | 93.743 (1), 94.776 (1), 106.197 (1) |
| $V$ (Å$^3$) | 1174.52 (8) |
| $Z$ | 2 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.09 |
| Crystal size (mm) | 0.61 × 0.49 × 0.18 |
| Data collection | |
| Diffractometer | Bruker SMART APEXII CCD area-detector diffractometer |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2009) |
| $T_{min}$, $T_{max}$ | 0.947, 0.984 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 35176, 4622, 3468 |
| $R_{int}$ | 0.049 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.617 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.045, 0.133, 1.04 |
| No. of reflections | 4622 |
| No. of parameters | 335 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.17, −0.15 |

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009).

(100 mg, Merk) were mixed and warmed over a heating magnetic stirrer hotplate for a few minutes. The resulting solution was allowed to cool slowly at room temperature and green plate-like crystals of the title compound appeared after a few days.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.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# full crystallographic data

*IUCrData* (2017). **2**, x170829 [https://doi.org/10.1107/S241431461700829X]

## 6-Amino-3,4-dimethyl-1,2,4-triazin-1-ium 2-anilinobenzoate–3-amino-5,6-dimethyl-1,2,4-triazine (1/1)

**Ramalingam Sangeetha, Kasthuri Balasubramani, Kaliyaperumal Thanigaimani and Ibrahim Abdul Razak**

**6-Amino-3,4-dimethyl-1,2,4-triazin-1-ium 2-anilinobenzoate; 3-amino-5,6-dimethyl-1,2,4-triazine**

*Crystal data*

$C_5H_9N_4^+·C_{13}H_{10}NO_2^-·C_5H_8N_4$
$M_r$ = 461.53
Triclinic, $P\overline{1}$
$a$ = 7.5018 (3) Å
$b$ = 7.6661 (3) Å
$c$ = 21.4332 (9) Å
$\alpha$ = 93.743 (1)°
$\beta$ = 94.776 (1)°
$\gamma$ = 106.197 (1)°
$V$ = 1174.52 (8) Å$^3$
$Z$ = 2
$F$(000) = 488
$D_x$ = 1.305 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2760 reflections
$\theta$ = 1.0–29.0°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
Plate, green
0.61 × 0.49 × 0.18 mm

*Data collection*

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.947, $T_{max}$ = 0.984
35176 measured reflections
4622 independent reflections
3468 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.049
$\theta_{max}$ = 26.0°, $\theta_{min}$ = 1.9°
$h$ = −9→9
$k$ = −9→9
$l$ = −26→26

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.133
$S$ = 1.04
4622 reflections
335 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.222P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | −0.06951 (14) | 0.41109 (18) | 0.27879 (6) | 0.0645 (4) |
| O2 | 0.04258 (14) | 0.29631 (16) | 0.19905 (5) | 0.0577 (3) |
| N5 | 0.1635 (2) | 0.6138 (2) | 0.37067 (7) | 0.0626 (4) |
| H5N | 0.052 (3) | 0.560 (3) | 0.3544 (9) | 0.067 (6)* |
| C6 | 0.06398 (19) | 0.4021 (2) | 0.24742 (7) | 0.0426 (3) |
| C7 | 0.25436 (19) | 0.52475 (19) | 0.27027 (7) | 0.0406 (3) |
| C8 | 0.3935 (2) | 0.5411 (2) | 0.23022 (8) | 0.0484 (4) |
| H8 | 0.3652 | 0.4748 | 0.1910 | 0.058* |
| C9 | 0.5720 (2) | 0.6525 (2) | 0.24700 (9) | 0.0596 (5) |
| H9 | 0.6636 | 0.6598 | 0.2198 | 0.071* |
| C10 | 0.6127 (2) | 0.7530 (3) | 0.30453 (10) | 0.0638 (5) |
| H10 | 0.7325 | 0.8299 | 0.3160 | 0.077* |
| C11 | 0.4795 (2) | 0.7416 (2) | 0.34506 (9) | 0.0602 (5) |
| H11 | 0.5100 | 0.8125 | 0.3834 | 0.072* |
| C12 | 0.2980 (2) | 0.6253 (2) | 0.33008 (7) | 0.0472 (4) |
| C13 | 0.1844 (3) | 0.6725 (2) | 0.43498 (8) | 0.0601 (5) |
| C14 | 0.3339 (3) | 0.6662 (4) | 0.47597 (10) | 0.0913 (7) |
| H14 | 0.4305 | 0.6270 | 0.4610 | 0.110* |
| C15 | 0.3400 (4) | 0.7182 (5) | 0.53918 (12) | 0.1181 (10) |
| H15 | 0.4412 | 0.7134 | 0.5665 | 0.142* |
| C16 | 0.2015 (5) | 0.7762 (4) | 0.56239 (12) | 0.1152 (10) |
| H16 | 0.2079 | 0.8122 | 0.6050 | 0.138* |
| C17 | 0.0526 (5) | 0.7806 (4) | 0.52206 (13) | 0.1038 (9) |
| H17 | −0.0440 | 0.8187 | 0.5375 | 0.125* |
| C18 | 0.0435 (3) | 0.7293 (3) | 0.45877 (10) | 0.0760 (6) |
| H18 | −0.0590 | 0.7332 | 0.4319 | 0.091* |
| N1A | 0.48051 (18) | 0.19326 (19) | 0.29896 (6) | 0.0516 (3) |
| N2A | 0.59717 (17) | 0.18820 (18) | 0.25468 (6) | 0.0474 (3) |
| H2AA | 0.727 (3) | 0.276 (3) | 0.2642 (9) | 0.072 (5)* |
| N3A | 0.67322 (19) | 0.0737 (2) | 0.16252 (7) | 0.0519 (4) |
| H3AA | 0.796 (3) | 0.147 (2) | 0.1719 (8) | 0.059 (5)* |
| H3AB | 0.633 (3) | 0.001 (3) | 0.1252 (9) | 0.063 (5)* |
| N4A | 0.36953 (16) | −0.02836 (18) | 0.18697 (6) | 0.0479 (3) |
| C1A | 0.54793 (19) | 0.0788 (2) | 0.20174 (7) | 0.0425 (3) |
| C2A | 0.2532 (2) | −0.0255 (2) | 0.22879 (8) | 0.0478 (4) |
| C3A | 0.3111 (2) | 0.0859 (2) | 0.28723 (8) | 0.0500 (4) |
| C4A | 0.1833 (3) | 0.0886 (3) | 0.33697 (10) | 0.0702 (5) |
| H4AA | 0.1544 | −0.0262 | 0.3550 | 0.105* |
| H4AC | 0.0703 | 0.1087 | 0.3187 | 0.105* |

| | | | | |
|---|---|---|---|---|
| H4AB | 0.2433 | 0.1850 | 0.3692 | 0.105* |
| C5A | 0.0571 (2) | −0.1422 (3) | 0.21318 (10) | 0.0635 (5) |
| H5AB | 0.0453 | −0.2096 | 0.1730 | 0.095* |
| H5AA | −0.0256 | −0.0666 | 0.2118 | 0.095* |
| H5AC | 0.0253 | −0.2257 | 0.2447 | 0.095* |
| N1B | 0.41507 (18) | 0.15155 (19) | −0.03886 (6) | 0.0511 (3) |
| N2B | 0.25699 (17) | 0.11190 (18) | −0.01023 (6) | 0.0485 (3) |
| N3B | 0.1073 (2) | 0.1583 (2) | 0.07556 (7) | 0.0583 (4) |
| H3BA | 0.003 (3) | 0.080 (3) | 0.0562 (9) | 0.063 (5)* |
| H3BB | 0.112 (3) | 0.218 (3) | 0.1170 (10) | 0.067 (5)* |
| N4B | 0.41980 (17) | 0.30830 (18) | 0.07997 (6) | 0.0496 (3) |
| C1B | 0.2648 (2) | 0.1919 (2) | 0.04733 (7) | 0.0442 (4) |
| C2B | 0.5721 (2) | 0.3445 (2) | 0.05112 (7) | 0.0458 (4) |
| C3B | 0.5703 (2) | 0.2650 (2) | −0.01015 (8) | 0.0462 (4) |
| C4B | 0.7392 (2) | 0.3046 (3) | −0.04550 (9) | 0.0610 (5) |
| H4BC | 0.7099 | 0.2325 | −0.0853 | 0.092* |
| H4BB | 0.8390 | 0.2750 | −0.0216 | 0.092* |
| H4BA | 0.7769 | 0.4317 | −0.0523 | 0.092* |
| C5B | 0.7458 (2) | 0.4714 (3) | 0.08573 (9) | 0.0647 (5) |
| H5BA | 0.7891 | 0.5753 | 0.0625 | 0.097* |
| H5BC | 0.8403 | 0.4093 | 0.0904 | 0.097* |
| H5BB | 0.7197 | 0.5111 | 0.1265 | 0.097* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0336 (6) | 0.0799 (8) | 0.0667 (7) | −0.0027 (5) | 0.0164 (5) | −0.0197 (6) |
| O2 | 0.0390 (6) | 0.0671 (7) | 0.0558 (7) | −0.0016 (5) | 0.0124 (5) | −0.0122 (6) |
| N5 | 0.0453 (8) | 0.0807 (11) | 0.0498 (8) | 0.0014 (7) | 0.0105 (7) | −0.0138 (7) |
| C6 | 0.0343 (7) | 0.0445 (8) | 0.0463 (8) | 0.0055 (6) | 0.0089 (6) | 0.0049 (7) |
| C7 | 0.0318 (7) | 0.0410 (8) | 0.0479 (8) | 0.0072 (6) | 0.0080 (6) | 0.0061 (6) |
| C8 | 0.0389 (8) | 0.0501 (9) | 0.0542 (9) | 0.0073 (7) | 0.0124 (7) | 0.0035 (7) |
| C9 | 0.0355 (8) | 0.0656 (11) | 0.0727 (12) | 0.0035 (7) | 0.0179 (8) | 0.0042 (9) |
| C10 | 0.0344 (8) | 0.0642 (11) | 0.0804 (13) | −0.0047 (7) | 0.0055 (8) | 0.0002 (9) |
| C11 | 0.0458 (9) | 0.0623 (11) | 0.0613 (10) | 0.0005 (8) | 0.0029 (8) | −0.0065 (8) |
| C12 | 0.0391 (8) | 0.0489 (9) | 0.0502 (9) | 0.0073 (6) | 0.0071 (7) | 0.0014 (7) |
| C13 | 0.0631 (11) | 0.0580 (10) | 0.0490 (9) | 0.0003 (8) | 0.0144 (8) | −0.0030 (8) |
| C14 | 0.0760 (14) | 0.127 (2) | 0.0610 (13) | 0.0152 (13) | 0.0033 (11) | −0.0005 (13) |
| C15 | 0.106 (2) | 0.161 (3) | 0.0582 (14) | −0.0041 (19) | −0.0043 (14) | 0.0025 (16) |
| C16 | 0.148 (3) | 0.118 (2) | 0.0536 (14) | −0.005 (2) | 0.0274 (17) | −0.0105 (14) |
| C17 | 0.146 (3) | 0.0996 (19) | 0.0746 (16) | 0.0367 (18) | 0.0564 (18) | 0.0055 (14) |
| C18 | 0.0891 (15) | 0.0833 (14) | 0.0600 (11) | 0.0244 (12) | 0.0320 (11) | 0.0092 (10) |
| N1A | 0.0436 (7) | 0.0580 (8) | 0.0523 (8) | 0.0101 (6) | 0.0131 (6) | 0.0076 (6) |
| N2A | 0.0345 (7) | 0.0522 (7) | 0.0510 (8) | 0.0033 (6) | 0.0099 (6) | 0.0052 (6) |
| N3A | 0.0349 (7) | 0.0623 (9) | 0.0525 (8) | 0.0031 (6) | 0.0103 (6) | 0.0020 (7) |
| N4A | 0.0324 (6) | 0.0512 (8) | 0.0563 (8) | 0.0050 (5) | 0.0059 (6) | 0.0062 (6) |
| C1A | 0.0316 (7) | 0.0459 (8) | 0.0485 (8) | 0.0065 (6) | 0.0072 (6) | 0.0107 (7) |
| C2A | 0.0339 (8) | 0.0473 (9) | 0.0637 (10) | 0.0092 (6) | 0.0119 (7) | 0.0174 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3A | 0.0405 (8) | 0.0532 (9) | 0.0587 (10) | 0.0121 (7) | 0.0151 (7) | 0.0166 (8) |
| C4A | 0.0587 (11) | 0.0821 (13) | 0.0730 (12) | 0.0164 (10) | 0.0305 (10) | 0.0170 (10) |
| C5A | 0.0335 (8) | 0.0637 (11) | 0.0876 (13) | 0.0016 (7) | 0.0115 (8) | 0.0130 (10) |
| N1B | 0.0418 (7) | 0.0583 (8) | 0.0508 (8) | 0.0079 (6) | 0.0137 (6) | 0.0046 (6) |
| N2B | 0.0366 (7) | 0.0562 (8) | 0.0480 (7) | 0.0047 (6) | 0.0097 (6) | 0.0033 (6) |
| N3B | 0.0374 (8) | 0.0774 (10) | 0.0518 (9) | 0.0030 (7) | 0.0126 (6) | −0.0048 (8) |
| N4B | 0.0406 (7) | 0.0559 (8) | 0.0493 (7) | 0.0082 (6) | 0.0069 (6) | 0.0039 (6) |
| C1B | 0.0363 (8) | 0.0500 (8) | 0.0460 (8) | 0.0103 (6) | 0.0073 (6) | 0.0080 (7) |
| C2B | 0.0378 (8) | 0.0454 (8) | 0.0524 (9) | 0.0076 (6) | 0.0062 (7) | 0.0088 (7) |
| C3B | 0.0371 (8) | 0.0460 (8) | 0.0554 (9) | 0.0089 (6) | 0.0111 (7) | 0.0090 (7) |
| C4B | 0.0455 (9) | 0.0643 (11) | 0.0695 (11) | 0.0064 (8) | 0.0199 (8) | 0.0037 (9) |
| C5B | 0.0454 (9) | 0.0691 (11) | 0.0681 (11) | −0.0008 (8) | 0.0042 (8) | 0.0018 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C6 | 1.2665 (17) | N3A—C1A | 1.3189 (19) |
| O2—C6 | 1.2445 (18) | N3A—H3AA | 0.933 (19) |
| N5—C12 | 1.375 (2) | N3A—H3AB | 0.92 (2) |
| N5—C13 | 1.404 (2) | N4A—C2A | 1.3056 (19) |
| N5—H5N | 0.85 (2) | N4A—C1A | 1.3595 (18) |
| C6—C7 | 1.496 (2) | C2A—C3A | 1.431 (2) |
| C7—C8 | 1.390 (2) | C2A—C5A | 1.490 (2) |
| C7—C12 | 1.416 (2) | C3A—C4A | 1.494 (2) |
| C8—C9 | 1.376 (2) | C4A—H4AA | 0.9600 |
| C8—H8 | 0.9300 | C4A—H4AC | 0.9600 |
| C9—C10 | 1.375 (3) | C4A—H4AB | 0.9600 |
| C9—H9 | 0.9300 | C5A—H5AB | 0.9600 |
| C10—C11 | 1.366 (2) | C5A—H5AA | 0.9600 |
| C10—H10 | 0.9300 | C5A—H5AC | 0.9600 |
| C11—C12 | 1.402 (2) | N1B—C3B | 1.318 (2) |
| C11—H11 | 0.9300 | N1B—N2B | 1.3490 (17) |
| C13—C18 | 1.373 (3) | N2B—C1B | 1.331 (2) |
| C13—C14 | 1.380 (3) | N3B—C1B | 1.3412 (19) |
| C14—C15 | 1.380 (3) | N3B—H3BA | 0.89 (2) |
| C14—H14 | 0.9300 | N3B—H3BB | 0.97 (2) |
| C15—C16 | 1.356 (4) | N4B—C2B | 1.3142 (19) |
| C15—H15 | 0.9300 | N4B—C1B | 1.3572 (19) |
| C16—C17 | 1.364 (4) | C2B—C3B | 1.409 (2) |
| C16—H16 | 0.9300 | C2B—C5B | 1.494 (2) |
| C17—C18 | 1.378 (3) | C3B—C4B | 1.499 (2) |
| C17—H17 | 0.9300 | C4B—H4BC | 0.9600 |
| C18—H18 | 0.9300 | C4B—H4BB | 0.9600 |
| N1A—C3A | 1.301 (2) | C4B—H4BA | 0.9600 |
| N1A—N2A | 1.3493 (17) | C5B—H5BA | 0.9600 |
| N2A—C1A | 1.327 (2) | C5B—H5BC | 0.9600 |
| N2A—H2AA | 1.02 (2) | C5B—H5BB | 0.9600 |
| | | | |
| C12—N5—C13 | 129.15 (16) | C2A—N4A—C1A | 116.78 (14) |

| | | | |
|---|---|---|---|
| C12—N5—H5N | 114.8 (13) | N3A—C1A—N2A | 119.79 (13) |
| C13—N5—H5N | 116.0 (13) | N3A—C1A—N4A | 119.16 (14) |
| O2—C6—O1 | 122.75 (13) | N2A—C1A—N4A | 121.05 (13) |
| O2—C6—C7 | 119.44 (12) | N4A—C2A—C3A | 121.44 (13) |
| O1—C6—C7 | 117.81 (13) | N4A—C2A—C5A | 117.34 (15) |
| C8—C7—C12 | 118.97 (14) | C3A—C2A—C5A | 121.22 (14) |
| C8—C7—C6 | 117.89 (13) | N1A—C3A—C2A | 120.46 (14) |
| C12—C7—C6 | 123.13 (13) | N1A—C3A—C4A | 116.60 (16) |
| C9—C8—C7 | 121.86 (16) | C2A—C3A—C4A | 122.94 (15) |
| C9—C8—H8 | 119.1 | C3A—C4A—H4AA | 109.5 |
| C7—C8—H8 | 119.1 | C3A—C4A—H4AC | 109.5 |
| C10—C9—C8 | 118.98 (15) | H4AA—C4A—H4AC | 109.5 |
| C10—C9—H9 | 120.5 | C3A—C4A—H4AB | 109.5 |
| C8—C9—H9 | 120.5 | H4AA—C4A—H4AB | 109.5 |
| C11—C10—C9 | 120.92 (15) | H4AC—C4A—H4AB | 109.5 |
| C11—C10—H10 | 119.5 | C2A—C5A—H5AB | 109.5 |
| C9—C10—H10 | 119.5 | C2A—C5A—H5AA | 109.5 |
| C10—C11—C12 | 121.42 (16) | H5AB—C5A—H5AA | 109.5 |
| C10—C11—H11 | 119.3 | C2A—C5A—H5AC | 109.5 |
| C12—C11—H11 | 119.3 | H5AB—C5A—H5AC | 109.5 |
| N5—C12—C11 | 121.59 (15) | H5AA—C5A—H5AC | 109.5 |
| N5—C12—C7 | 120.56 (14) | C3B—N1B—N2B | 120.50 (13) |
| C11—C12—C7 | 117.79 (14) | C1B—N2B—N1B | 117.46 (12) |
| C18—C13—C14 | 118.46 (18) | C1B—N3B—H3BA | 119.5 (11) |
| C18—C13—N5 | 118.06 (18) | C1B—N3B—H3BB | 118.4 (11) |
| C14—C13—N5 | 123.37 (18) | H3BA—N3B—H3BB | 122.0 (16) |
| C15—C14—C13 | 119.9 (3) | C2B—N4B—C1B | 116.13 (13) |
| C15—C14—H14 | 120.0 | N2B—C1B—N3B | 118.09 (14) |
| C13—C14—H14 | 120.0 | N2B—C1B—N4B | 125.25 (13) |
| C16—C15—C14 | 121.5 (3) | N3B—C1B—N4B | 116.65 (14) |
| C16—C15—H15 | 119.3 | N4B—C2B—C3B | 120.74 (14) |
| C14—C15—H15 | 119.3 | N4B—C2B—C5B | 117.67 (14) |
| C15—C16—C17 | 118.7 (2) | C3B—C2B—C5B | 121.59 (14) |
| C15—C16—H16 | 120.6 | N1B—C3B—C2B | 119.91 (13) |
| C17—C16—H16 | 120.6 | N1B—C3B—C4B | 117.31 (15) |
| C16—C17—C18 | 120.8 (3) | C2B—C3B—C4B | 122.79 (14) |
| C16—C17—H17 | 119.6 | C3B—C4B—H4BC | 109.5 |
| C18—C17—H17 | 119.6 | C3B—C4B—H4BB | 109.5 |
| C13—C18—C17 | 120.6 (2) | H4BC—C4B—H4BB | 109.5 |
| C13—C18—H18 | 119.7 | C3B—C4B—H4BA | 109.5 |
| C17—C18—H18 | 119.7 | H4BC—C4B—H4BA | 109.5 |
| C3A—N1A—N2A | 116.69 (14) | H4BB—C4B—H4BA | 109.5 |
| C1A—N2A—N1A | 123.40 (13) | C2B—C5B—H5BA | 109.5 |
| C1A—N2A—H2AA | 121.3 (11) | C2B—C5B—H5BC | 109.5 |
| N1A—N2A—H2AA | 115.3 (11) | H5BA—C5B—H5BC | 109.5 |
| C1A—N3A—H3AA | 120.2 (11) | C2B—C5B—H5BB | 109.5 |
| C1A—N3A—H3AB | 117.7 (11) | H5BA—C5B—H5BB | 109.5 |
| H3AA—N3A—H3AB | 122.0 (16) | H5BC—C5B—H5BB | 109.5 |

| | | | |
|---|---|---|---|
| O2—C6—C7—C8 | 12.1 (2) | C3A—N1A—N2A—C1A | −1.2 (2) |
| O1—C6—C7—C8 | −168.01 (14) | N1A—N2A—C1A—N3A | −175.83 (13) |
| O2—C6—C7—C12 | −168.55 (15) | N1A—N2A—C1A—N4A | 4.4 (2) |
| O1—C6—C7—C12 | 11.4 (2) | C2A—N4A—C1A—N3A | 176.54 (14) |
| C12—C7—C8—C9 | −0.2 (2) | C2A—N4A—C1A—N2A | −3.7 (2) |
| C6—C7—C8—C9 | 179.15 (15) | C1A—N4A—C2A—C3A | 0.2 (2) |
| C7—C8—C9—C10 | −1.2 (3) | C1A—N4A—C2A—C5A | 179.96 (14) |
| C8—C9—C10—C11 | 0.8 (3) | N2A—N1A—C3A—C2A | −2.3 (2) |
| C9—C10—C11—C12 | 1.1 (3) | N2A—N1A—C3A—C4A | 178.65 (14) |
| C13—N5—C12—C11 | −16.2 (3) | N4A—C2A—C3A—N1A | 2.9 (2) |
| C13—N5—C12—C7 | 166.56 (17) | C5A—C2A—C3A—N1A | −176.87 (15) |
| C10—C11—C12—N5 | −179.81 (17) | N4A—C2A—C3A—C4A | −178.16 (15) |
| C10—C11—C12—C7 | −2.5 (3) | C5A—C2A—C3A—C4A | 2.1 (2) |
| C8—C7—C12—N5 | 179.39 (15) | C3B—N1B—N2B—C1B | 0.2 (2) |
| C6—C7—C12—N5 | 0.0 (2) | N1B—N2B—C1B—N3B | 177.90 (14) |
| C8—C7—C12—C11 | 2.0 (2) | N1B—N2B—C1B—N4B | −1.0 (2) |
| C6—C7—C12—C11 | −177.32 (14) | C2B—N4B—C1B—N2B | 0.9 (2) |
| C12—N5—C13—C18 | 148.89 (19) | C2B—N4B—C1B—N3B | −178.02 (14) |
| C12—N5—C13—C14 | −34.9 (3) | C1B—N4B—C2B—C3B | 0.0 (2) |
| C18—C13—C14—C15 | −0.7 (4) | C1B—N4B—C2B—C5B | −179.67 (15) |
| N5—C13—C14—C15 | −176.9 (2) | N2B—N1B—C3B—C2B | 0.6 (2) |
| C13—C14—C15—C16 | −0.1 (5) | N2B—N1B—C3B—C4B | −178.99 (13) |
| C14—C15—C16—C17 | 0.8 (5) | N4B—C2B—C3B—N1B | −0.7 (2) |
| C15—C16—C17—C18 | −0.8 (5) | C5B—C2B—C3B—N1B | 178.95 (15) |
| C14—C13—C18—C17 | 0.7 (3) | N4B—C2B—C3B—C4B | 178.86 (15) |
| N5—C13—C18—C17 | 177.1 (2) | C5B—C2B—C3B—C4B | −1.5 (2) |
| C16—C17—C18—C13 | 0.0 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N5—H5*N*···O1 | 0.85 (2) | 1.92 (2) | 2.619 (2) | 138 (2) |
| N2*A*—H2*AA*···O1$^{i}$ | 1.02 (2) | 1.58 (2) | 2.593 (2) | 179 (2) |
| N3*A*—H3*AA*···O2$^{i}$ | 0.93 (2) | 1.90 (2) | 2.834 (2) | 175 (2) |
| N3*A*—H3*AB*···N1*B*$^{ii}$ | 0.92 (2) | 2.07 (2) | 2.986 (2) | 172 (2) |
| N3*B*—H3*BA*···N2*B*$^{iii}$ | 0.89 (2) | 2.20 (2) | 3.089 (2) | 179 (2) |
| N3*B*—H3*BB*···O2 | 0.97 (2) | 1.98 (2) | 2.915 (2) | 163 (2) |
| C4*B*—H4*BC*···N4*A*$^{ii}$ | 0.96 | 2.52 | 3.483 (2) | 177 |

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