Structural Insights into Supramolecular Interactions in Isostructural Salts of 2,4,6-Triaminopyrimidinium with Various Heterocyclic Carboxylates

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Abstract

2,4,6-triaminopyrimidine is an interesting and challenging molecule due to the multiple hydrogen bond donors and acceptors. Its non-covalent interactions with the variety of carboxylic acids provide several supramolecular aggregates with frequently occurring molecular synthons. The present work focused on the supramolecular interactions of 2,4,6-triaminopyrimidinium indole-3-propionate (I), 2,4,6-triaminopyrimidinium indole-3-acetate (II), 2,4,6-triaminopyrimidinium 5-bromothiophene-2-carboxylate (III) and 2,4,6-triaminopyrimidinium 5-chlorothiophene-2-carboxylate (IV). All the four compounds exhibit a robust homomeric and heteromeric $R_2^2(8)$ ring motifs. Compounds (I and II) develop sextruple (I) and quadruple (I & II) hydrogen bonded array through fused ring motifs. Compound II exhibits a rosette like architecture. Compound IV is isostructural and isomorphous with Compound III, exhibiting an identical crystal structure with different composition and identical supramolecular architectures. In compounds (III & IV), a linear hetero tetrameric motif is fomed and in addition, both compounds exhibit halogen$\cdots\pi$ interactions which enhance the crystal stability. All compounds develop a supramolecular hydrogen bonded pattern facilitated by several N—H$\cdots$O and N—H$\cdots$N hydrogen bonds with multiple furcated donors and acceptors.

1. Introduction

X-ray single crystal diffraction and structural analysis provides information on the molecular conformation and mutual arrangement of building blocks within the crystal structure. The investigation into supramolecular architectures within crystals remains a highly active and dynamic field. The intricate interplay of various noncovalent interactions governs the formation of these supramolecular architectures. Hydrogen bonding, known for its strength and directionality, emerges as a crucial factor in shaping the solid-state organization of molecular solids. The resulting crystal structure reflects a delicate equilibrium among a diverse range of intermolecular interactions. Crystal engineering typically involves the design and synthesis of crystalline materials by self-assembling molecular building blocks. The functionality of the material is often categorized within the framework of crystal engineering. This approach identifies a compatible arrangement of molecular or ionic components to form into a specific architecture by strategically engineering a network of desired supramolecular interactions (Bernstein *et al.*, 1995; Jeffrey & Saenger, 1991; Desiraju, 1995). In biological systems, pyrimidine and aminopyrimidine derivatives are essential constituents, serving as fundamental building blocks for nucleic acids. The functionality of nucleic acids is dictated by hydrogen bonding patterns crucial for the transfer of genetic information. Various derivatives of pyrimidine demonstrate a range of biological activities such as antibacterial, antimicrobial, anticancer, anti-HIV-1, and anti-rubella virus effects. Moreover, these derivatives play integral roles in energy transduction, act as metabolic cofactors, and contribute to cellular signaling processes (Desiraju,1995; Brown, 1987; Botta *et al.*, 2001).

2,4,6-Triaminopyrimidine (TAP) plays a crucial role in synthesizing anticancer drugs and lipophilic small molecule antifolates, offering potential applications in the treatment and prevention of opportunistic microbial infections associated with AIDS (Rosowsky *et al.*, 2003). Additionally, TAP exhibits diverse pharmacological effects, serving as a blocker of cation-specific paracellular conductance pathways in epithelial cells, inhibiting sodium transport in frog skin, and influencing transmembrane potential and contractility in guinea pig myocardium (Achelle *et al.*, 2012, Seenan *et al.*, 2020, Bowman *et al.*, 1978). Its interaction with carboxylic acids is integral to processes like protein-nucleic acid recognition and drug-protein recognition. The pyrimidine moiety of TAP forms hydrogen bonds with the carboxyl group of proteins (Vallee & Auld, 1993; Hunt *et al.*, 1980). Due to its numerous hydrogen-bond donors and acceptors, 2,4,6-triaminopyrimidine presents itself as an intriguing and challenging molecule.

This research investigates the potential of carboxylic acids to form stable supramolecular heterosynthons with pyrimidine derivatives, due to the strong hydrogen bonding capabilities of pyrimidines. Numerous crystal structures of TAP salts have been reported, including 2,4,6-triaminopyrimidinium (TAP^+) dichloride dihydrate (Portalone & Colapietro, 2007), TAP^+ malonate, TAP^+ succinate, TAP^+ glutarate, TAP^+ adipate, TAP^+ thiodipropionate (Pedireddi *et al.*, 1998), TAP zidovudine (Bhatt *et al.*, 2009), TAP^+ barbiturate (Marchi-Artzner *et al.*, 1998), TAP^+ sulfate pentahydrate (Nimthong *et al.*, 2013), TAP^+ 3,5-dihydroxybenzoate dihydrate, TAP^+ 3-nitrophthalate monohydrate, TAP^+ 5-amino-2,4,6-triiodoisophthalate dihydrate, TAP^+ 2,5-dibromoterephthalate dihydrate, TAP^+ naphthalene-1,5-disulfonate, TAP^+ sebacicate, TAP^+ 1,2,4-benzenetricarboxylate monohydrate, TAP^+ biphenyl-2,2',5,5'-tetracarboxylate monohydrate (Xing *et al.*, 2017), TAP^+ 3-nitrobenzoate (Mohana *et al.*, 2023), TAP^+ 6-chloronicotinate dihydrate, TAP^+ pyridine-2,6-dicarboxylate dihydrate, TAP^+ sulfate monohydrate and TAP^+ 3,5-dinitrobenzoate dihydrate (Sangavi *et al.*, 2023).

In this present work, four salts have been prepared, namely, 2,4,6- triaminopyrimidinium indole-3-propionate, indole-3-propionic acid (I), 2,4,6-triaminopyrimidinium indole-3-acetate (II), 2,4,6-triaminopyrimidinium 5-bromothiophene-2-carboxylate (III) and 2,4,6-triaminopyrimidinium 5-chlorothiphene-2-carboxylate (IV) (Scheme 1).

2. Experimental

2.1. Synthesis and crystallization

Compounds I - IV were prepared by mixing a hot ethanolic solution of 2,4,6-triaminopyrimidine (0.0312 mg) with hot ethanolic solution of indole-3-propionic acid (0.0473 mg)/indole-3-acetic acid (0.0437 mg)/5-bromothiophene-2-carboxylic acid (0.0517 mg) /5-chlorothiophene-2-carboxylic acid (0.0406 mg) in 1:1 molar ratio. The resultant mixture was warmed over a water bath for 20 min at 70 °C and then allowed to cool slowly at room temperature. After a few days, colorless prismatic crystals were separated out from the mother liquor.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. In all the structures (I—IV), the hydrogen atoms of N—H and NH_2 groups were located from the difference Fourier map and refined freely. Other hydrogen atoms were placed geometrically and refined using a riding model.

3. Results and discussion

The cocrystal of a salt of the compound I is built by one 2,4,6-triaminopyrimidinium cation (TAP) one molecule of indole-3-propionate IPA (A) anion and one molecule of indole-3-propionic acid IPA (B) in the asymmetric unit. The ORTEP view of the compound is shown in Fig 1a. The oxygen atoms of the carboxyl group in IPA (A) are deprotonated and the proton transferred to the pyrimidine nitrogen atom (N1) of TAP. Protonation at N1 site is reflected by a slight increase in C2—N1—C6 bond angle (120.78 (12)º) and for the non protonated ring N3, the C2—N3—C4 bond angle is

found to be 116.94 (12)° (Schwalbe & Williams, 1982). The carboxylate C—O bond length tend to be (C17A—O1A)/(C17A—O2A) = 1.2345 (18) Å/1.2809 (17) Å) respectively which also supported the deprotanation of IPA A ion.

The fundamental heterodimeric synthon $R^2_2(8)$ (Etter *et al.*, 1990) is formed via a pair of N—H···O hydrogen bonds involving the protonated ring N1 and N5-amino group of the cation and O2A and O1A atom of the carboxylate (A) anion (Table 2). Further $R^2_3(10)$ motif is formed through a couple of N—H···O and one O—H···O hydrogen bonds linking protonated ring N1 and N2-amino group of cation with bifurcated carboxylate O2A atom of anion A and bifurcated carboxylic O2B atom of IPA (B). The $R^2_4(8)$ ring motif is formed *via* a couple of N—H···O hydrogen bonds by linking amino N5 of TAP cation and carboxylate O1A and O1A^{ii} [Symmetry code: -*x*, -*y*+1, -*z*+1] of symmetry related IPA (A) anion.The three ring motifs fused together and thus facilitating the formation of sextruplet hydrogen bonded array (DDDAAA) by the reoccurrence of $R^2_2(8)$, $R_3^2(10)$ & $R_4^2(8)$ fused ring motifs. This arrangement is analogous to crystal structure of 2,4,6-triamnipyrimidinium sulfate monohydrate (Sangavi *et al.*, 2023). A discrete complementary base pairing is formed via N—H···N hydrogen bond involving amino N2 and N3^{i} [Symmetry code: -*x*+1, -*y*, -*z*+1] of sy,mmetry related TAP cation. This discrete base pair in turn links amino N4 of TAP with O2B^{i} [Symmetry code: -*x*+1, -*y*, -*z*+1] of carboxylic acid IPA(B) through N—H···O hydrogen bond to form $R_3^2(8)$ ring motif resulting a DADA quadruple hydrogen bonded pattern with a ring sequence of $R_3^2(8)$, $R_2^2(8)$ and $R_3^2(8)$. Similar type of complementary base pair is observed in the crystal structure of 2,4,6-triamnipyrimidinium succinate (Pedireddi *et al.*, 1998), 2,4,6-triamnipyrimidinium 3,5-dihydroxybenzoate (Xing *et al.*, 2017), 2,4,6-triamnipyrimidinium 3-nitrobenzoate (Mohana *et al.*, 2023) and 2,4,6-triamnipyrimidinium 6-chloronicotinate dihydrate (Sangavi *et al.*, 2023). This arrangement is observed in the crystal structures of 2,4,6-triamnipyrimidinium 3-nitrobenzoate (Mohana *et al.*, 2023). The alternative occurrence of sextuple and quadruple hydrogen bonded arrays generates the supramolecular hydrogen bonded pattern (Fig 1b). The crystal structure is further stabilized by C—H···π and carbonyl···π interactions. The carbonyl ···π interactions is observed between carbonyl group of IPA (B) and pyrimidinium cation (C17B—O2B···Cg7 = 3.4638 (14) Å; <C=O···Cg7= 124.14 (10)°; Symmetry code: 1+x, y, z).

The asymmetric unit of compound II is composed of two crystallographically independent TAP cations (A and B) and two indole-3-acetate anions [IAA (A) & IAA (B)]. The ORTEP view of the compound is shown in Fig 2a. Both the TAP moieties (A & B) are protonated at ring nitrogen N1 position and the same are evident from the widening of C—N—C bond angle C2A—N1A—C6A and C2B—N1B—C6B are 120.8 (2)° and 120.4 (2)° respectively where as the non protonated C—N—C bond angle in the same molecules are C2A—N3A—C4A = 117.7 (2)° and C2B—N3B—C4B = 117.1 (2)° respectively (Schwalbe & Williams, 1982). The deprotonated carboxylate C—O bond length in the IAA (A and B) moities C16A—O1A, C16A—O2A, C16B—O1B, C16B—O2B are found to be 1.259 (3) Å, 1.260 (3) Å, 1.265 (3) Å, 1.257 (3) Å) respectively.

The primary interaction between the cation and anion leads to the formation of two different heterodimeric R^2_2 (8) ring motifs *via* pair of N—H···O hydrogen bonds (Table 3). This hydrogen bond connects the protonated ring N1A and amino N2A of TAP (A) with carboxylate O1A & O2A atoms of IAA(A) and protonated ring N1B and amino N5B of TAP (B) with carboxylate O1B & O2B atoms of IAA(B). Among the two TAP (A & B), the cation B is self assembled centrosymmetrically and exist as a discrete base pair *via* a pair of N—H···N hydrogen bonds involving amino N4B and ring N3B^{v} [Symmetry code : 2-x,1-y,-z] of TAP (B) cation to form homomeric R^2_2 (8) ring. This type of complementary base pair observed in the crystal structure of 2,4,6-triamnipyrimidinium succinate (Pedireddi *et al.*, 1998), 2,4,6-triamnipyrimidinium 3,5-dihydroxybenzoate (Xing *et al.*, 2017), 2,4,6-triamnipyrimidinium 3-nitrobenzoate (Mohana *et al.*, 2023) and 2,4,6-triamnipyrimidinium 6- chloronicotinate dihydrate (Sangavi *et al.*, 2023). This discrete base pair hold two centrosymmetric IAA(A) anions on either side by the formation of R_3^2 (8) ring motif through a pair of N—

H···O hydrogen bonds linking amino N2B & N4B of TAP B cation and O2A of IAA (A) and O2A^{v} [Symmetry code : 2-x,1-y,-z] of symmetry related IAA (A)anion. The continuous occurrence of fused ring motifs R_3^2 (8), R_2^2 (8) and R_3^2 (8) develop a quadruple DADA hydrogen bonded array. This type of array is observed in the crystal structures of 2,4,6-triamnipyrimidinium 3-nitrobenzoate (Mohana *et al.*, 2023). The TAP (A and B) cations exhibit a heteromeric base pair like a discrete chain through N—H···N hydrogon bond linking amino N2B and N5A^{iv} [symmetry code:1-x,1-y,1-z]. The two base TAP (A & B) connect one IAA(B) anion *via* a pair of bifurcated N—H···O hydrogen bonds to form a R_3^2 (8) ring motif. These supramolecular assemblies involving two TAP (A & B) and two IAA (A & B) molecules centrosymmetrically paired through a pair of N—H···N hydrogen bonds by linking indole ring N6A of IAA (A) anion.and ring N3A^{vii} [Symmetry code: x,1+y,z] of TAP (A) cation. This hydrogen bond generates a huge ring motif R_6^6(30) and the suprmolecuar assemblies together propagates as a supramolecular sheet extending along *b* axis as shown in Fig 2b. These supramolecular sheets are fused by a rosette like architecture (Shao *et al.*, 2004; Hemamalini *et al.*, 2006; Prior *et al.*, 2013) involving R_4^2(16) ring motif at the centre and around which a pair of R_5^4(16) and R_4^4(20) ring motifs. This rosette is formed by the tetrameric molecular assemblies of TAP (A & B) and IAA(A & B) molecules involving two pairs of bifurcated N—H···O hydrogen bonds by linking amino N2A and amino N4A of TAP (A) cations with carboxylate O2B^{i} [Symmetry code:1-x,1-y,-z] and O2B^{ii} [Symmetry code: x,-1+y,1+z] of two symmetry related IAA(B) anion. The R_4^4(20) ring motif links two symmetry related TAP (A) cation with IAA(B) anion through N—H···O hydrogen bonding involving amino N5A and carboxylate O1B^{iv} [Symmetry code: 1-x,1-y,1-z]. The R_5^4(16) ring motif formed through a pair of N—H···N hydrogen bonds by coupling amino N5A and N5B^{iii} [Symmetry code: x,y,1+z] of two symmetry related TAP(A & B) cations as shown in Fig 2c. This hydrogen facilated the formation of R_3^2(8) ring motif through N—H···O hydrogen bond by connecting amino N5B of TAP(B) cation and carboxylate O1A^{vi} [Symmetry code: x,y,-1+z] of IAA(A) anion. The entire interaction develop a supramolecular hydrogen bonded network. The supramolecular hydrogen bonded network is further stabilized by two different π-π stacking interactions .One is observed between aromatic five membered rings IAA (B) anions [Cg1···Cg1^{viii} ; Symmetry code: -x,2-y,-z] and the another is observed between the aromatic five membered and six memebered ring of IAA(B) [Cg1···Cg2^{viii} ; Symmetry code: -x,2-y,-z] . The corresponding centroid to centroid distance for the two different stacking are 3.5353 (16) Å & 3.7027 (18) Å & and the slip angle of 12.64° & 21.28° respectively. The crystal structure is further strengthened by weak C—H···π interactions.

The asymmetric unit of compound III contains two crystallographically independent TAP cations (A and B) and two 5-bromothiophene-2-carboxylate an ions BTCA (A & B) (Figure 3a). In both A and B molecules, protonation occurs at N1 positon of TAP which are confirmed by the widening of C—N—C bond angle C2A—N1A—C6A & C2B—N1B—C6B are 120.4 (2)^{o} and 120.24 (2)^{o} respectively where as the N3 position remains as non protonated with C—N—C bond and angle (C2A—N3A—C4A & C2B—N3B—C4B) of 116.84 (19)^{o} and 116.98 (18)^{o} respectively (Schwalbe & Williams,1982).

The molecule interacts primarily through a pair of N—H···O hydrogen bonds leading to the formation of two different R_2^2 (8) ring motifs involving protonated ring N1A & N1B and amino N2A & N5B of cation and carboxylate O1A & O2A and O1B & O2B of anion (Table 4). Among the two BTCA anions, the anion B forms a R_2^1(6) ring motif *via* a bifurcated N—H···O hydrogen bond involving protonated ring N1B, amino N5B of cation B and carboxylate O1B of anion B. Similarly the BTCA anion A form a R_3^2(8) ring motif by linking TAP (A and B) cations and BTCA (A) anion through a bifurcated N—H···O by linking amino N5B of TAP (B) and carboxylate O2A^{vi} [Symmetry code: x,y,1+z] of BTCA (A) anion. This motif also links amino N2A and amino N5B^{i} [Symmetry code: x,y,-1+z] via N—H···N hydrogen bonds. Both the TAP cations exhibit a discrete complementary base pairing with the generation of homomeric R_2^2 (8)

ring motifs *via* a couple of N—H···N hydrogen bonding.This motif links two centrosymmetric TAP (A) cation through N2A—H2B···N3A^{ii} [Symmetry code: 1-x,2-y,-z] and centrosymmetric TAP (B) through N2B—H2C···N3B^{iii} [Symmetry code: -x,2-y,1-z]. Similar type of complementary base pairing is also observed in compound I. The discrete base pair formed by B cations are interlinked by huge R_4^4 (20) ring motif involving two centrosymmetric BTCA (A) anion *via* two pair of N—H···O hydrogen bonds involving amino N4B and N5B and symmetry related carboxylate O1A^{iv} [Symmetry code: -x,1-y,1-z] & O2A^{vi} [Symmetry code: x,y,1+z] of BTCA (A) anion. The alternate existence of the R_2^2 (8) and R_4^4 (20) ring motifs propagate as a supramolecular layer along *c* axis as shown in Fig 3b. The discrete base pair formed by TAP (A) cations are interconnected by R_3^2(6) ring motif in which O1B atom of BTCA (B) acts as a trifurcated hydrogen bonded donor facilitating the formation of three different motifs (R_2^2 (8) , R_2^1 (6) and R_3^2 (6)). The R_3^2(6) motif links tetrameric supermolecule involving two TAP (A & B) cations and BTCA (A & B) anions through four N—H···O hydrogen bonds. The existence of R_3^2(6) ring motif facilitated the generation of huge R_8^6(26) ring motif and its recurrence in alternative manner along with the acid base interaction (B molecule) leads to the formation of another supramolecular layer which is also extending along the *c* axis. These two sheets are inter linked by a linear heterotetrameric synthon by holding a base pair formed by TAP (B) cations and two symmetry related BTCA(B) anion. All the interaction generate a supramolecular sheet like architecture which is further stabilized by aromatic face to face π-π stacking interaction observed between five membered ring of BTCA (A) and pyrimidine ring of the TAP (A) molecule [Cg1···Cg3^{vii}; Symmetry code: 1-x,1-y,-z] with centroid to centroid distance of 3.7175 (15) Å, interplanar distance of 3.664 Å and the slip angle of 9.70°. The crystal structure is further strengthened by halogen···π interaction (symmetry code:x,-1+y,-1+z) with distance 3.6250 (9) Å and angle of 139.86 (8)^o. These values are agree with those reported (Shukla *et al.*, 2017) halogen···π interaction.

The crystal structure of compound (IV) is the isomorphous with the crystal structure of compound (III). Both the compounds are crystallized in triclinic P-1 space group with similar cell parameters . The asymmetric unit of compound IV consists of two TAP cations (A and B) and two 5-chlorothiophene-2-carboxylate anions (CTCA (A) and (B)) as shown in Fig 4a. The protonation of compound (IV) is similar to compound (III) and the parameters are also closely agree with the compound (III). The supramolecular hydrogen bonded interactions (Table 5) of this compounds are exactly isostructural (Gomathi *et al.*, 2014) with compound (III) except the existence of R_4^2(10) motif adjacent to R_2^2(8) instead of R_2^1(6) in compound (III) (Fig 4b). The values of aromatic stacking and halogen··· π interaction (Riley *et al.*, 2016) of this compound have a very good agreement with the crystal structure of (III).

In the crystal structures (I- IV), there is a consistent protonation at the N1 position of TAP with similar widening of C—N—C bond angles. Compounds (I—IV) exhibit an acid-base interactions to form a robust R_2^2(8) homomeric and heteromeric synthons involving primary acid base interaction and secondary complementary base pairing. The compounds (I and II) develop sextruple (compound I) and quadruple (compounds I & II) hydrogen bonded array. Compound (II) exhibits a rosette like architecture. All compounds develop a supramolecular hydrogen bonded pattern facilitated by several N—H···O and N—H···N hydrogen bonds with multiple furcated donors and acceptors. In compounds (II—IV) aromatic π-π stacking plays an additional role in stabilizing the compounds where in compound I, C—H···π governs the additional stability. Compound (IV) is isostructural and isomorphous with Compound (III), exhibiting identical crystal structure with different composition and identical supramolecular interactions and architectures. In compounds (III & IV), a linear hetero tetrameric motif is fomed and in addition, halogen··· π interactions enhanced the crystal stability. Crystal structure of four TAP salts have been synthesized and characterized by single-crystal X-ray diffraction technique and their non-covalent interactions and supramolecular patterns have been investigated.

Table 1

Experimental details

	(1)	(2)	(3)	(4)
Crystal data				
Chemical formula	$C_{11}H_{10}NO_2{\cdot}C_{11}H_{11}NO_2{\cdot}C_4H_8N_5$	$C_{10}H_8NO_2{\cdot}C_4H_8N_5$	$C_5H_2BrO_2S{\cdot}C_4H_8N_5$	$C_5H_2ClO_2S{\cdot}C_4H_8N_5$
M_r	503.56	300.33	332.19	287.73
Crystal system, space group	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	120	120	120	120
a, *b*, *c* (Å)	5.6392 (3), 13.4963 (7), 31.968 (2)	11.5487 (14), 11.5859 (10), 11.9737 (15)	8.4348 (5), 10.5501 (7), 14.0931 (9)	8.3306 (5), 10.4403 (5), 14.0361 (5)
α, β, γ (°)	90, 92.790 (6), 90	69.595 (9), 73.659 (11), 74.862 (9)	77.558 (5), 86.820 (5), 78.896 (5)	78.406 (4), 87.357 (4), 78.608 (4)
V (Å^3)	2430.2 (2)	1417.0 (3)	1201.61 (14)	1172.28 (10)
Z	4	4	4	4
Radiation type	Mo *K*α	Mo *K*α	Cu *K*α	Cu *K*α
μ (mm^{-1})	0.10	0.10	6.34	4.61
Crystal size (mm)	0.47 × 0.43 × 0.10	0.48 × 0.23 × 0.12	0.15 × 0.12 × 0.06	0.36 × 0.15 × 0.12
Data collection				
Diffractometer	SuperNova, Dual, Cu at zero, Atlas	SuperNova, Dual, Cu at zero, Atlas	SuperNova, Dual, Cu at zero, Atlas	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Multi-scan *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	Multi-scan *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min}, T_{max}	0.956, 0.990	0.954, 0.988	0.450, 0.702	0.288, 0.608
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17370, 6030, 4473	13004, 7007, 4307	10725, 4975, 4697	10298, 4863, 4401
R_{int}	0.045	0.052	0.022	0.026
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.667	0.667	0.631	0.632
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.115, 1.05	0.062, 0.145, 1.03	0.031, 0.083, 1.04	0.038, 0.108, 1.05
No. of reflections	6030	7007	4975	4863
No. of parameters	374	461	382	381
No. of restraints	1	0	0	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.26	0.24, −0.27	1.23, −1.27	0.68, −0.57

Computer programs: *CrysAlis PRO* (Agilent, 2014), SHELXT 2014/4 (Sheldrick, 2015a), *SHELXL2016*/6 (Sheldrick, 2015b), *SHELXL2018*/3 (Sheldrick, 2015b), *PLATON* (Spek, 2009), *Mercury* (Macrae *et al.*, 2008) and POVRay (Cason, 2004), *publCIF* (Westrip, 2010).

Table 2

Hydrogen-bond geometry (Å, º) for (1)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*B*—H1*B*···O2*A*	0.91 (2)	1.62 (2)	2.5178 (17)	172 (3)
N1—H1*A*···O2*A*	0.98 (2)	1.78 (2)	2.7577 (17)	174.8 (19)
N2—H2*B*···N3^{i}	0.92 (2)	2.08 (2)	2.9915 (19)	170.5 (17)
N2—H2*A*···O2*B*	0.90 (2)	2.03 (2)	2.7825 (18)	139.9 (16)
N5—H5*A*···O1*A*^{ii}	0.89 (2)	2.139 (19)	2.9149 (17)	144.7 (16)
N5—H5*B*···O1*A*	0.92 (2)	1.89 (2)	2.8166 (19)	177 (2)
N4—H4*A*···O2*B*^{i}	0.89 (2)	2.12 (2)	2.9968 (19)	172.5 (18)

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

Table 3

Hydrogen-bond geometry (Å, º) for (2)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O1*A*	1.02 (3)	1.68 (3)	2.696 (3)	172 (3)
N1*B*—H1*B*···O1*B*	0.99 (4)	1.74 (4)	2.733 (3)	179 (5)
N2*A*—H2*A*···O2*B*^{i}	0.87 (2)	2.07 (2)	2.886 (2)	156 (2)
N2*A*—H2*B*···O2*A*	0.91 (3)	1.90 (3)	2.798 (3)	171 (3)
N4*A*—H4*A*···O2*B*^{ii}	0.88 (3)	2.27 (3)	3.037 (3)	146 (3)
N5*A*—H5*D*···N5*B*^{iii}	0.90 (3)	2.47 (3)	3.282 (3)	152 (2)
N5*A*—H5*C*···O1*B*^{iv}	0.91 (3)	1.98 (3)	2.873 (3)	163 (2)
N1*B*—H1*B*···O1*B*	0.99 (3)	1.74 (3)	2.733 (2)	179 (3)
N2*B*—H2*C*···O2*A*	0.92 (3)	1.94 (3)	2.844 (3)	167 (3)
N2*B*—H2*D*···N5*A*^{iv}	0.90 (3)	2.41 (3)	3.278 (3)	161 (2)
N4*B*—H4*C*···N3*B*^{v}	0.91 (3)	2.09 (3)	3.000 (3)	174 (2)
N4*B*—H4*D*···O2*A*^{v}	0.89 (3)	2.34 (3)	3.042 (3)	135 (2)
N5*B*—H5*F*···O1*A*^{vi}	0.92 (3)	2.14 (2)	2.885 (2)	138 (2)
N5*B*—H5*E*···O2*B*	0.85 (3)	1.93 (3)	2.764 (3)	169 (3)
N6*A*—H6*A*···N3*A*^{vii}	0.91 (3)	2.03 (3)	2.932 (2)	173 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*, *y*−1, *z*+1; (iii) *x*, *y*, *z*+1; (iv) −*x*+1, −*y*+1, −*z*+1; (v) −*x*+2, −*y*+1, −*z*; (vi) *x*, *y*, *z*−1; (vii) *x*, *y*+1, *z*.

Table 4

Hydrogen-bond geometry (Å, º) for (3)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O2*A*	0.83 (3)	1.92 (3)	2.743 (3)	177 (3)
N1*B*—H1*B*···O1*B*	0.94 (3)	1.75 (3)	2.672 (2)	170 (3)
N2*A*—H2*A*···N5*B*^{i}	0.89 (3)	2.33 (3)	3.185 (3)	162 (3)
N2*A*—H2*B*···N3*A*^{ii}	0.88 (3)	2.13 (3)	3.013 (3)	178 (3)
N2*B*—H2*C*···N3*B*^{iii}	0.88 (3)	2.13 (3)	3.005 (3)	174 (3)

N2*B*—H2*D*···O2*B*	0.86 (3)	1.99 (3)	2.844 (2)	172 (3)
N4*A*—H4*A*···O2*B*	0.85 (4)	1.93 (4)	2.783 (2)	177 (3)
N4*B*—H4*D*···O1*A*^{iv}	0.83 (4)	2.16 (4)	2.964 (3)	162 (3)
N5*A*—H5*C*···O1*A*	0.88 (3)	2.04 (3)	2.911 (3)	174 (3)
N5*A*—H5*D*···O1*B*^{v}	0.83 (4)	2.37 (3)	2.978 (3)	131 (3)
N5*B*—H5*E*···O1*B*	0.84 (3)	2.56 (3)	3.217 (2)	136 (3)
N5*B*—H5*E*···O1*A*^{v}	0.84 (3)	2.52 (3)	3.103 (2)	128 (2)
N5*B*—H5*F*···O2*A*^{vi}	0.97 (3)	1.85 (3)	2.809 (3)	167 (3)

Symmetry codes: (i) *x*, *y*, *z*−1; (ii) −*x*+1, −*y*+2, −*z*; (iii) −*x*, −*y*+2, −*z*+1; (iv) −*x*, −*y*+1, −*z*+1; (v) −*x*+1, −*y*+1, −*z*+1; (vi) *x*, *y*, *z*+1.

Table 5

Hydrogen-bond geometry (Å, º) for (4)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O2*A*	0.91 (3)	1.83 (3)	2.736 (2)	173 (3)
N1*B*—H1*B*···O1*B*	0.90 (3)	1.80 (3)	2.687 (2)	169 (3)
N2*A*—H2*A*···N5*B*^{i}	0.85 (3)	2.36 (3)	3.181 (3)	165 (3)
N2*A*—H2*B*···N3*A*^{ii}	0.87 (3)	2.14 (3)	3.014 (2)	177.8 (14)
N2*B*—H2*C*···O2*B*	0.86 (3)	1.99 (3)	2.844 (2)	173 (3)
N2*B*—H2*D*···N3*B*^{iii}	0.87 (3)	2.14 (3)	3.008 (2)	178 (3)
N4*A*—H4*B*···O2*B*	0.90 (3)	1.90 (3)	2.784 (2)	167 (2)
N4*B*—H4*D*···O1*A*^{iv}	0.85 (4)	2.15 (4)	2.969 (2)	161 (3)
N5*B*—H5*C*···O1*B*^{v}	0.87 (3)	2.21 (3)	2.906 (2)	136 (3)
N5*A*—H5*D*···O1*A*	0.87 (3)	2.05 (3)	2.911 (2)	169 (3)
N5*B*—H5*E*···O1*A*^{v}	0.87 (3)	2.45 (3)	3.077 (2)	130 (2)
N5*B*—H5*F*···O2*A*	0.95 (3)	1.89 (3)	2.815 (2)	163 (3)

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) −*x*+1, −*y*, −*z*+2; (iii) −*x*+2, −*y*, −*z*+1; (iv) −*x*+2, −*y*+1, −*z*+1; (v) −*x*+1, −*y*+1, −*z*+1.

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Figure 1

a. ORTEP view of the compound I with displacement ellipsoids drawn at 50% probability level. b. A view of three dimensional supramolecular ladder formed *via* N-H···O, N-H···N and O-H···O hydrogen bonds. [Symmetry codes: (i) -*x*+1, -*y*, -*z*+1; (ii) -*x*, -*y*+1, -*z*+1]. c. A view of three dimensional supramolecular ladder formed *via* N-H···O, N-H···N and O-H···O hydrogen bonds.

Figure 2

a. ORTEP view of the compound II with displacement ellipsoids drawn at 50% probability level. b.Three dimensional supramolecular aggregates formed via N-H···O and N-H···N hydrogen bonds. c.The generation of rosette like structure via N-H···O and N-H···N hydrogen bonds [Symmetry codes: (i) -*x*+1, -*y*+1, -*z*; (ii) *x*, *y*-1, *z*+1; (iii) *x*, *y*, *z*+1; (iv) -*x*+1, -*y*+1, -*z*+1; (v) -*x*+2, -*y*+1, -*z*; (vi) *x*, *y*, *z*-1; (vii) *x*, *y*+1, *z*].

Figure 3

a.ORTEP view of the compound III with displacement ellipsoids drawn at 50% probability level. b.A view of three dimensional supramolecular sheet formed via N-H···O and N-H···N hydrogen bonds [Symmetry codes: (i) *x*, *y*, *z*-1; (ii) -*x*+1, -*y*+2, -*z*; (iii) -*x*, -*y*+2, -*z*+1; (iv) -*x*, -*y*+1, -*z*+1; (v) -*x*+1, -*y*+1, -*z*+1; (vi) *x*, *y*, *z*+1].

Figure 4

a.ORTEP view of the compound IV with displacement ellipsoids drawn at 50% probability level. b.Three dimensional supramolecular sheet formed via N-H···O and N-H···N hydrogen bonds [Symmetry codes: (i) *x*, *y*, *z*+1; (ii) -*x*+1, -*y*, -*z*+2; (iii) -*x*+2, -*y*, -*z*+1; (iv) -*x*+2, -*y*+1, -*z*+1; (v) -*x*+1, -*y*+1, -*z*+1].

supporting information

Structural Insights into Supramolecular Interactions in Isostructural Salts of 2,4,6-Triaminopyrimidinium with Various Heterocyclic Carboxylates

Mohana Marimuthu, Gomathi Sundaramoorthy, Thomas Muthiah Packianathan* and Ray J. Butcher

Computing details

For all structures, data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: SHELXT 2014/4 (Sheldrick, 2015a). Program(s) used to refine structure: *SHELXL2016*/6 (Sheldrick, 2015b) for (1), (3), (4); *SHELXL2018*/3 (Sheldrick, 2015b) for (2). For all structures, molecular graphics: *PLATON* (Spek, 2009), *Mercury* (Macrae *et al.*, 2008) and POVRay (Cason, 2004); software used to prepare material for publication: *PLATON* (Spek, 2009), *publCIF* (Westrip, 2010).

(1)

Crystal data

$C_{11}H_{10}NO_2 \cdot C_{11}H_{11}NO_2 \cdot C_4H_8N_5$
$M_r = 503.56$
Monoclinic, $P2_1/c$
$a = 5.6392$ (3) Å
$b = 13.4963$ (7) Å
$c = 31.968$ (2) Å
$\beta = 92.790$ (6)°
$V = 2430.2$ (2) Å^3
$Z = 4$
$F(000) = 1064$
$D_x = 1.376$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 15769 reflections
$\theta = 3.0–41.2°$
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Plate, pale yellow
0.47 × 0.43 × 0.10 mm

Data collection

SuperNova, Dual, Cu at zero, Atlas diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Detector resolution: 10.6501 pixels mm^{-1}
ω scans
Absorption correction: multi-scan *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
$T_{min} = 0.956$, $T_{max} = 0.990$
17370 measured reflections
6030 independent reflections
4473 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 28.3°$, $\theta_{min} = 3.0°$
$h = -7 \rightarrow 4$
$k = -17 \rightarrow 17$
$l = -42 \rightarrow 42$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.115$

S = 1.05
6030 reflections
374 parameters
1 restraint
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 0.393P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ 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 (Å^2) for (1)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2250 (2)	0.23098 (9)	0.48773 (4)	0.0194 (3)
H1A	0.297 (4)	0.2747 (15)	0.4676 (7)	0.042 (6)*
N2	0.4890 (2)	0.11276 (10)	0.46916 (5)	0.0234 (3)
H2A	0.564 (3)	0.1575 (14)	0.4534 (6)	0.033 (5)*
H2B	0.562 (3)	0.0529 (14)	0.4744 (6)	0.036 (5)*
N3	0.2286 (2)	0.07378 (9)	0.51961 (4)	0.0200 (3)
N4	−0.0371 (3)	0.03859 (10)	0.56903 (5)	0.0238 (3)
H4A	0.030 (4)	−0.0206 (15)	0.5717 (6)	0.034 (5)*
H4B	−0.157 (3)	0.0529 (14)	0.5819 (6)	0.030 (5)*
N5	−0.0360 (3)	0.35429 (10)	0.50222 (5)	0.0248 (3)
H5A	−0.139 (4)	0.3828 (13)	0.5189 (6)	0.031 (5)*
H5B	0.063 (4)	0.3941 (15)	0.4873 (7)	0.048 (6)*
C2	0.3124 (3)	0.13818 (10)	0.49285 (5)	0.0192 (3)
C4	0.0415 (3)	0.10331 (11)	0.54137 (5)	0.0192 (3)
C5	−0.0613 (3)	0.19655 (11)	0.53671 (5)	0.0209 (3)
H5A1	−0.195106	0.214552	0.551972	0.025*
C6	0.0361 (3)	0.26194 (11)	0.50940 (5)	0.0202 (3)
O1A	0.2704 (2)	0.47885 (8)	0.45922 (4)	0.0286 (3)
O2A	0.4543 (2)	0.35032 (8)	0.43252 (4)	0.0276 (3)
N6A	0.0571 (3)	0.68220 (10)	0.35278 (5)	0.0245 (3)
H6A	−0.077 (4)	0.6670 (14)	0.3385 (7)	0.040 (6)*
C7A	0.1775 (3)	0.61903 (11)	0.37986 (5)	0.0223 (3)
H7A	0.123305	0.555184	0.387478	0.027*
C8A	0.3855 (3)	0.66059 (10)	0.39428 (5)	0.0184 (3)
C9A	0.3950 (3)	0.75556 (10)	0.37467 (5)	0.0187 (3)
C10A	0.5597 (3)	0.83268 (11)	0.37677 (5)	0.0229 (3)
H10A	0.701171	0.827100	0.393984	0.028*
C11A	0.5141 (3)	0.91720 (12)	0.35349 (6)	0.0290 (4)
H11A	0.625187	0.970142	0.354873	0.035*
C12A	0.3069 (3)	0.92625 (12)	0.32789 (6)	0.0280 (4)
H12A	0.280552	0.985172	0.312084	0.034*
C13A	0.1406 (3)	0.85168 (12)	0.32510 (5)	0.0260 (4)
H13A	0.000182	0.857701	0.307621	0.031*
C14A	0.1868 (3)	0.76702 (11)	0.34899 (5)	0.0201 (3)
C15A	0.5703 (3)	0.62031 (11)	0.42464 (5)	0.0208 (3)

H15A	0.521911	0.634835	0.453351	0.025*
H15B	0.720956	0.655993	0.420655	0.025*
C16A	0.6172 (3)	0.50954 (11)	0.42132 (6)	0.0234 (3)
H16A	0.631652	0.492649	0.391414	0.028*
H16B	0.771783	0.494804	0.436016	0.028*
C17A	0.4308 (3)	0.44322 (11)	0.43899 (5)	0.0228 (3)
O1B	0.7773 (3)	0.30849 (9)	0.38377 (5)	0.0410 (4)
H1B	0.667 (4)	0.320 (2)	0.4028 (8)	0.085 (10)*
O2B	0.8445 (2)	0.16603 (8)	0.41613 (4)	0.0329 (3)
N6B	1.5069 (3)	0.23563 (10)	0.24054 (5)	0.0254 (3)
H6B	1.632 (4)	0.2564 (15)	0.2260 (7)	0.053 (7)*
C7B	1.4121 (3)	0.28776 (12)	0.27251 (6)	0.0258 (4)
H7B	1.476118	0.347515	0.283999	0.031*
C8B	1.2144 (3)	0.24218 (11)	0.28546 (5)	0.0223 (3)
C9B	1.1790 (3)	0.15702 (11)	0.25921 (5)	0.0190 (3)
C10B	1.0061 (3)	0.08315 (11)	0.25574 (5)	0.0220 (3)
H10B	0.875002	0.083765	0.273318	0.026*
C11B	1.0283 (3)	0.00941 (11)	0.22648 (5)	0.0247 (4)
H11B	0.911301	−0.041166	0.223992	0.030*
C12B	1.2206 (3)	0.00752 (11)	0.20025 (5)	0.0247 (4)
H12B	1.232316	−0.044656	0.180504	0.030*
C13B	1.3922 (3)	0.07978 (11)	0.20261 (5)	0.0226 (3)
H13B	1.522789	0.078503	0.184934	0.027*
C14B	1.3676 (3)	0.15469 (11)	0.23178 (5)	0.0209 (3)
C15B	1.0615 (3)	0.27346 (11)	0.31986 (5)	0.0242 (4)
H15C	0.895401	0.278557	0.308536	0.029*
H15D	1.111851	0.340233	0.329542	0.029*
C16B	1.0693 (3)	0.20361 (12)	0.35730 (6)	0.0275 (4)
H16C	1.047994	0.134890	0.346951	0.033*
H16D	1.228523	0.207876	0.371647	0.033*
C17B	0.8861 (3)	0.22399 (11)	0.38862 (5)	0.0245 (4)

Atomic displacement parameters (Å²) for (1)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0220 (7)	0.0177 (6)	0.0188 (7)	0.0015 (5)	0.0045 (6)	0.0001 (5)
N2	0.0237 (7)	0.0188 (6)	0.0285 (8)	0.0016 (5)	0.0106 (6)	0.0014 (6)
N3	0.0242 (7)	0.0176 (6)	0.0185 (7)	−0.0011 (5)	0.0052 (6)	−0.0013 (5)
N4	0.0285 (8)	0.0202 (7)	0.0235 (7)	0.0007 (6)	0.0104 (6)	0.0006 (6)
N5	0.0264 (8)	0.0229 (7)	0.0257 (8)	0.0063 (6)	0.0074 (6)	0.0023 (6)
C2	0.0212 (8)	0.0185 (7)	0.0180 (8)	−0.0023 (6)	0.0014 (6)	−0.0034 (6)
C4	0.0209 (8)	0.0208 (7)	0.0161 (7)	−0.0022 (6)	0.0015 (6)	−0.0039 (6)
C5	0.0205 (8)	0.0233 (7)	0.0190 (8)	0.0010 (6)	0.0045 (6)	−0.0031 (6)
C6	0.0216 (8)	0.0215 (7)	0.0175 (8)	0.0009 (6)	−0.0002 (6)	−0.0032 (6)
O1A	0.0335 (7)	0.0203 (5)	0.0334 (7)	0.0040 (5)	0.0163 (6)	0.0031 (5)
O2A	0.0349 (7)	0.0181 (5)	0.0309 (7)	0.0039 (5)	0.0143 (6)	0.0016 (5)
N6A	0.0210 (7)	0.0269 (7)	0.0252 (8)	−0.0016 (6)	−0.0026 (6)	−0.0015 (6)
C7A	0.0257 (8)	0.0206 (7)	0.0208 (8)	−0.0005 (6)	0.0030 (7)	−0.0011 (6)
C8A	0.0209 (8)	0.0187 (7)	0.0159 (7)	0.0020 (6)	0.0034 (6)	−0.0013 (6)
C9A	0.0217 (8)	0.0186 (7)	0.0161 (7)	0.0033 (6)	0.0030 (6)	−0.0017 (6)
C10A	0.0232 (8)	0.0220 (7)	0.0236 (8)	−0.0003 (6)	0.0008 (7)	−0.0004 (7)

C11A	0.0334 (10)	0.0203 (7)	0.0335 (10)	−0.0019 (7)	0.0025 (8)	0.0041 (7)
C12A	0.0362 (10)	0.0232 (8)	0.0250 (9)	0.0086 (7)	0.0056 (8)	0.0058 (7)
C13A	0.0281 (9)	0.0295 (8)	0.0204 (8)	0.0096 (7)	0.0019 (7)	0.0000 (7)
C14A	0.0206 (8)	0.0231 (7)	0.0168 (8)	0.0039 (6)	0.0022 (6)	−0.0032 (6)
C15A	0.0210 (8)	0.0212 (7)	0.0202 (8)	0.0008 (6)	0.0009 (7)	0.0029 (6)
C16A	0.0249 (8)	0.0208 (7)	0.0249 (9)	0.0051 (6)	0.0058 (7)	0.0030 (7)
C17A	0.0270 (9)	0.0217 (7)	0.0199 (8)	0.0041 (6)	0.0048 (7)	0.0034 (7)
O1B	0.0578 (9)	0.0260 (6)	0.0419 (9)	0.0179 (6)	0.0313 (8)	0.0099 (6)
O2B	0.0406 (7)	0.0267 (6)	0.0330 (7)	0.0073 (5)	0.0174 (6)	0.0086 (6)
N6B	0.0246 (7)	0.0266 (7)	0.0256 (8)	−0.0034 (6)	0.0072 (6)	−0.0003 (6)
C7B	0.0277 (9)	0.0243 (8)	0.0257 (9)	−0.0009 (7)	0.0032 (7)	−0.0019 (7)
C8B	0.0273 (8)	0.0204 (7)	0.0194 (8)	0.0026 (6)	0.0039 (7)	0.0007 (6)
C9B	0.0215 (8)	0.0200 (7)	0.0154 (7)	0.0044 (6)	0.0005 (6)	0.0041 (6)
C10B	0.0236 (8)	0.0229 (7)	0.0196 (8)	0.0018 (6)	0.0036 (7)	0.0041 (7)
C11B	0.0277 (9)	0.0206 (7)	0.0256 (9)	−0.0023 (6)	−0.0007 (7)	0.0018 (7)
C12B	0.0334 (9)	0.0206 (7)	0.0198 (8)	0.0050 (7)	0.0002 (7)	−0.0003 (7)
C13B	0.0247 (8)	0.0261 (8)	0.0173 (8)	0.0072 (6)	0.0046 (7)	0.0028 (7)
C14B	0.0213 (8)	0.0214 (7)	0.0199 (8)	0.0015 (6)	0.0001 (6)	0.0038 (6)
C15B	0.0304 (9)	0.0205 (7)	0.0222 (8)	0.0026 (6)	0.0067 (7)	−0.0004 (7)
C16B	0.0347 (10)	0.0239 (8)	0.0249 (9)	0.0078 (7)	0.0099 (8)	0.0024 (7)
C17B	0.0323 (9)	0.0191 (7)	0.0227 (8)	0.0003 (6)	0.0063 (7)	−0.0018 (7)

Geometric parameters (Å, º) for (1)

N1—C2	1.3530 (19)	C13A—C14A	1.392 (2)
N1—C6	1.364 (2)	C13A—H13A	0.9500
N1—H1A	0.98 (2)	C15A—C16A	1.523 (2)
N2—C2	1.325 (2)	C15A—H15A	0.9900
N2—H2A	0.90 (2)	C15A—H15B	0.9900
N2—H2B	0.92 (2)	C16A—C17A	1.511 (2)
N3—C2	1.3230 (19)	C16A—H16A	0.9900
N3—C4	1.352 (2)	C16A—H16B	0.9900
N4—C4	1.334 (2)	O1B—C17B	1.3008 (19)
N4—H4A	0.89 (2)	O1B—H1B	0.905 (17)
N4—H4B	0.83 (2)	O2B—C17B	1.2085 (19)
N5—C6	1.328 (2)	N6B—C14B	1.366 (2)
N5—H5A	0.89 (2)	N6B—C7B	1.371 (2)
N5—H5B	0.92 (2)	N6B—H6B	0.91 (2)
C4—C5	1.390 (2)	C7B—C8B	1.356 (2)
C5—C6	1.374 (2)	C7B—H7B	0.9500
C5—H5A1	0.9500	C8B—C9B	1.431 (2)
O1A—C17A	1.2346 (19)	C8B—C15B	1.491 (2)
O2A—C17A	1.2788 (18)	C9B—C10B	1.395 (2)
N6A—C14A	1.367 (2)	C9B—C14B	1.411 (2)
N6A—C7A	1.371 (2)	C10B—C11B	1.376 (2)
N6A—H6A	0.89 (2)	C10B—H10B	0.9500
C7A—C8A	1.360 (2)	C11B—C12B	1.403 (2)
C7A—H7A	0.9500	C11B—H11B	0.9500
C8A—C9A	1.429 (2)	C12B—C13B	1.373 (2)
C8A—C15A	1.492 (2)	C12B—H12B	0.9500
C9A—C10A	1.394 (2)	C13B—C14B	1.387 (2)

C9A—C14A	1.408 (2)	C13B—H13B	0.9500
C10A—C11A	1.379 (2)	C15B—C16B	1.523 (2)
C10A—H10A	0.9500	C15B—H15C	0.9900
C11A—C12A	1.399 (3)	C15B—H15D	0.9900
C11A—H11A	0.9500	C16B—C17B	1.499 (2)
C12A—C13A	1.376 (2)	C16B—H16C	0.9900
C12A—H12A	0.9500	C16B—H16D	0.9900

C2—N1—C6	120.73 (14)	C8A—C15A—H15B	108.4
C2—N1—H1A	118.5 (12)	C16A—C15A—H15B	108.4
C6—N1—H1A	120.7 (12)	H15A—C15A—H15B	107.5
C2—N2—H2A	122.1 (12)	C17A—C16A—C15A	115.41 (13)
C2—N2—H2B	117.7 (12)	C17A—C16A—H16A	108.4
H2A—N2—H2B	118.1 (17)	C15A—C16A—H16A	108.4
C2—N3—C4	116.84 (13)	C17A—C16A—H16B	108.4
C4—N4—H4A	120.1 (13)	C15A—C16A—H16B	108.4
C4—N4—H4B	118.7 (13)	H16A—C16A—H16B	107.5
H4A—N4—H4B	121.0 (18)	O1A—C17A—O2A	123.45 (15)
C6—N5—H5A	120.3 (12)	O1A—C17A—C16A	120.39 (14)
C6—N5—H5B	116.7 (13)	O2A—C17A—C16A	116.14 (14)
H5A—N5—H5B	118.9 (17)	C17B—O1B—H1B	113.7 (18)
N3—C2—N2	120.11 (14)	C14B—N6B—C7B	108.79 (14)
N3—C2—N1	123.14 (14)	C14B—N6B—H6B	126.5 (14)
N2—C2—N1	116.75 (14)	C7B—N6B—H6B	124.4 (14)
N4—C4—N3	116.16 (14)	C8B—C7B—N6B	110.55 (14)
N4—C4—C5	120.89 (15)	C8B—C7B—H7B	124.7
N3—C4—C5	122.91 (14)	N6B—C7B—H7B	124.7
C6—C5—C4	118.16 (14)	C7B—C8B—C9B	106.18 (14)
C6—C5—H5A1	120.9	C7B—C8B—C15B	127.63 (15)
C4—C5—H5A1	120.9	C9B—C8B—C15B	126.19 (14)
N5—C6—N1	116.09 (14)	C10B—C9B—C14B	118.67 (14)
N5—C6—C5	125.75 (15)	C10B—C9B—C8B	134.21 (15)
N1—C6—C5	118.15 (13)	C14B—C9B—C8B	107.12 (13)
C14A—N6A—C7A	109.02 (14)	C11B—C10B—C9B	118.97 (15)
C14A—N6A—H6A	126.4 (13)	C11B—C10B—H10B	120.5
C7A—N6A—H6A	124.3 (13)	C9B—C10B—H10B	120.5
C8A—C7A—N6A	110.48 (14)	C10B—C11B—C12B	121.22 (15)
C8A—C7A—H7A	124.8	C10B—C11B—H11B	119.4
N6A—C7A—H7A	124.8	C12B—C11B—H11B	119.4
C7A—C8A—C9A	105.75 (14)	C13B—C12B—C11B	121.14 (15)
C7A—C8A—C15A	129.33 (14)	C13B—C12B—H12B	119.4
C9A—C8A—C15A	124.91 (14)	C11B—C12B—H12B	119.4
C10A—C9A—C14A	118.72 (14)	C12B—C13B—C14B	117.48 (15)
C10A—C9A—C8A	133.44 (15)	C12B—C13B—H13B	121.3
C14A—C9A—C8A	107.83 (13)	C14B—C13B—H13B	121.3
C11A—C10A—C9A	119.02 (16)	N6B—C14B—C13B	130.18 (15)
C11A—C10A—H10A	120.5	N6B—C14B—C9B	107.34 (14)
C9A—C10A—H10A	120.5	C13B—C14B—C9B	122.48 (14)
C10A—C11A—C12A	121.09 (15)	C8B—C15B—C16B	114.14 (13)
C10A—C11A—H11A	119.5	C8B—C15B—H15C	108.7
C12A—C11A—H11A	119.5	C16B—C15B—H15C	108.7

C13A—C12A—C11A	121.43 (15)	C8B—C15B—H15D	108.7
C13A—C12A—H12A	119.3	C16B—C15B—H15D	108.7
C11A—C12A—H12A	119.3	H15C—C15B—H15D	107.6
C12A—C13A—C14A	117.12 (16)	C17B—C16B—C15B	114.66 (14)
C12A—C13A—H13A	121.4	C17B—C16B—H16C	108.6
C14A—C13A—H13A	121.4	C15B—C16B—H16C	108.6
N6A—C14A—C13A	130.48 (15)	C17B—C16B—H16D	108.6
N6A—C14A—C9A	106.92 (14)	C15B—C16B—H16D	108.6
C13A—C14A—C9A	122.60 (15)	H16C—C16B—H16D	107.6
C8A—C15A—C16A	115.51 (14)	O2B—C17B—O1B	123.17 (16)
C8A—C15A—H15A	108.4	O2B—C17B—C16B	122.13 (14)
C16A—C15A—H15A	108.4	O1B—C17B—C16B	114.70 (14)
C4—N3—C2—N2	−177.11 (15)	C8A—C9A—C14A—C13A	−179.61 (14)
C4—N3—C2—N1	2.3 (2)	C7A—C8A—C15A—C16A	37.8 (2)
C6—N1—C2—N3	−2.4 (2)	C9A—C8A—C15A—C16A	−143.28 (15)
C6—N1—C2—N2	176.97 (15)	C8A—C15A—C16A—C17A	−75.62 (19)
C2—N3—C4—N4	−178.30 (14)	C15A—C16A—C17A—O1A	−8.8 (2)
C2—N3—C4—C5	−0.3 (2)	C15A—C16A—C17A—O2A	172.86 (15)
N4—C4—C5—C6	176.40 (15)	C14B—N6B—C7B—C8B	0.8 (2)
N3—C4—C5—C6	−1.5 (2)	N6B—C7B—C8B—C9B	−1.48 (19)
C2—N1—C6—N5	179.91 (15)	N6B—C7B—C8B—C15B	178.81 (15)
C2—N1—C6—C5	0.5 (2)	C7B—C8B—C9B—C10B	−177.52 (18)
C4—C5—C6—N5	−178.01 (16)	C15B—C8B—C9B—C10B	2.2 (3)
C4—C5—C6—N1	1.4 (2)	C7B—C8B—C9B—C14B	1.61 (18)
C14A—N6A—C7A—C8A	0.16 (19)	C15B—C8B—C9B—C14B	−178.67 (15)
N6A—C7A—C8A—C9A	−0.31 (18)	C14B—C9B—C10B—C11B	1.6 (2)
N6A—C7A—C8A—C15A	178.78 (15)	C8B—C9B—C10B—C11B	−179.37 (16)
C7A—C8A—C9A—C10A	179.54 (17)	C9B—C10B—C11B—C12B	−0.1 (2)
C15A—C8A—C9A—C10A	0.4 (3)	C10B—C11B—C12B—C13B	−0.6 (2)
C7A—C8A—C9A—C14A	0.34 (17)	C11B—C12B—C13B—C14B	−0.2 (2)
C15A—C8A—C9A—C14A	−178.80 (14)	C7B—N6B—C14B—C13B	−179.06 (16)
C14A—C9A—C10A—C11A	−0.4 (2)	C7B—N6B—C14B—C9B	0.27 (18)
C8A—C9A—C10A—C11A	−179.54 (16)	C12B—C13B—C14B—N6B	−178.99 (16)
C9A—C10A—C11A—C12A	−0.3 (3)	C12B—C13B—C14B—C9B	1.8 (2)
C10A—C11A—C12A—C13A	0.3 (3)	C10B—C9B—C14B—N6B	178.13 (14)
C11A—C12A—C13A—C14A	0.3 (2)	C8B—C9B—C14B—N6B	−1.16 (17)
C7A—N6A—C14A—C13A	179.36 (16)	C10B—C9B—C14B—C13B	−2.5 (2)
C7A—N6A—C14A—C9A	0.06 (18)	C8B—C9B—C14B—C13B	178.24 (14)
C12A—C13A—C14A—N6A	179.83 (16)	C7B—C8B—C15B—C16B	−112.7 (2)
C12A—C13A—C14A—C9A	−1.0 (2)	C9B—C8B—C15B—C16B	67.7 (2)
C10A—C9A—C14A—N6A	−179.58 (14)	C8B—C15B—C16B—C17B	−169.81 (15)
C8A—C9A—C14A—N6A	−0.24 (17)	C15B—C16B—C17B—O2B	167.65 (16)
C10A—C9A—C14A—C13A	1.1 (2)	C15B—C16B—C17B—O1B	−12.5 (2)

Hydrogen-bond geometry (Å, º) for (1)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*B*—H1*B*···O2*A*	0.91 (2)	1.62 (2)	2.5178 (17)	172 (3)
N1—H1*A*···O2*A*	0.98 (2)	1.78 (2)	2.7577 (17)	174.8 (19)
N2—H2*B*···N3^{i}	0.92 (2)	2.08 (2)	2.9915 (19)	170.5 (17)

N2—H2*A*···O2*B*	0.90 (2)	2.03 (2)	2.7825 (18)	139.9 (16)
N5—H5*A*···O1*A*^{ii}	0.89 (2)	2.139 (19)	2.9149 (17)	144.7 (16)
N5—H5*B*···O1*A*	0.92 (2)	1.89 (2)	2.8166 (19)	177 (2)
N4—H4*A*···O2*B*^{i}	0.89 (2)	2.12 (2)	2.9968 (19)	172.5 (18)

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

(2)

Crystal data

$C_{10}H_8NO_2 \cdot C_4H_8N_5$
$M_r = 300.33$
Triclinic, $P\overline{1}$
$a = 11.5487$ (14) Å
$b = 11.5859$ (10) Å
$c = 11.9737$ (15) Å
$\alpha = 69.595$ (9)°
$\beta = 73.659$ (11)°
$\gamma = 74.862$ (9)°
$V = 1417.0$ (3) Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.408$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7851 reflections
$\theta = 2.9$–31.3°
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Block, colourless
0.48 × 0.23 × 0.12 mm

Data collection

SuperNova, Dual, Cu at zero, Atlas diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Detector resolution: 10.6501 pixels mm^{-1}
ω scans
Absorption correction: multi-scan *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
$T_{min} = 0.954$, $T_{max} = 0.988$
13004 measured reflections
7007 independent reflections
4307 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.9°$
$h = -15 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.145$
$S = 1.03$
7007 reflections
461 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ 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) for (2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1A	0.63615 (16)	0.30030 (18)	0.62051 (17)	0.0208 (4)
H1A	0.644 (3)	0.392 (3)	0.577 (3)	0.061 (9)*
N2A	0.63925 (19)	0.2710 (2)	0.43978 (19)	0.0257 (5)
H2A	0.629 (2)	0.228 (2)	0.398 (2)	0.031 (7)*
H2B	0.660 (2)	0.347 (3)	0.399 (3)	0.043 (8)*
N3A	0.60521 (16)	0.10818 (17)	0.61650 (17)	0.0209 (4)
N4A	0.5679 (2)	−0.04931 (19)	0.7937 (2)	0.0310 (5)
H4A	0.548 (3)	−0.085 (3)	0.749 (3)	0.050 (9)*
H4B	0.559 (3)	−0.082 (3)	0.874 (3)	0.070 (11)*
N5A	0.64327 (19)	0.3363 (2)	0.7959 (2)	0.0270 (5)
H5D	0.665 (3)	0.410 (3)	0.749 (3)	0.056 (10)*
H5C	0.642 (2)	0.309 (3)	0.878 (3)	0.048 (8)*
C2A	0.62629 (19)	0.2243 (2)	0.5595 (2)	0.0211 (5)
C4A	0.5935 (2)	0.0661 (2)	0.7390 (2)	0.0233 (5)
C5A	0.6072 (2)	0.1376 (2)	0.8060 (2)	0.0243 (5)
H5A	0.601031	0.105141	0.891712	0.029*
C6A	0.6300 (2)	0.2565 (2)	0.7435 (2)	0.0220 (5)
N1B	0.65132 (18)	0.61064 (17)	−0.10067 (16)	0.0211 (4)
H1B	0.563 (3)	0.649 (3)	−0.085 (3)	0.060 (9)*
N2B	0.6484 (2)	0.5680 (2)	0.10206 (18)	0.0258 (5)
H2C	0.685 (3)	0.548 (3)	0.167 (3)	0.060 (9)*
H2D	0.566 (2)	0.593 (2)	0.113 (2)	0.037 (7)*
N3B	0.83497 (16)	0.53634 (17)	−0.02755 (16)	0.0225 (4)
N4B	1.01985 (18)	0.5148 (2)	−0.1620 (2)	0.0297 (5)
H4C	1.059 (2)	0.500 (2)	−0.101 (2)	0.031 (7)*
H4D	1.064 (3)	0.529 (3)	−0.238 (3)	0.047 (8)*
N5B	0.6422 (2)	0.6387 (2)	−0.29810 (19)	0.0262 (5)
H5F	0.679 (2)	0.637 (2)	−0.377 (3)	0.037 (7)*
H5E	0.570 (3)	0.680 (3)	−0.285 (3)	0.046 (9)*
C2B	0.7144 (2)	0.5707 (2)	−0.0094 (2)	0.0213 (5)
C4B	0.8970 (2)	0.5428 (2)	−0.1444 (2)	0.0229 (5)
C5B	0.8378 (2)	0.5737 (2)	−0.2402 (2)	0.0248 (5)
H5B	0.882802	0.570268	−0.319063	0.030*
C6B	0.7124 (2)	0.6094 (2)	−0.2174 (2)	0.0226 (5)
O1A	0.67827 (15)	0.53560 (14)	0.50482 (14)	0.0267 (4)
O2A	0.72548 (14)	0.49730 (14)	0.32602 (14)	0.0264 (4)
N6A	0.72289 (18)	0.92150 (18)	0.48589 (18)	0.0260 (5)
H6A	0.687 (3)	0.984 (3)	0.520 (3)	0.056 (9)*
C7A	0.6674 (2)	0.8778 (2)	0.4250 (2)	0.0246 (5)
H7A	0.587994	0.912863	0.407782	0.029*
C8A	0.7420 (2)	0.7773 (2)	0.3929 (2)	0.0215 (5)
C9A	0.8515 (2)	0.7565 (2)	0.4361 (2)	0.0213 (5)
C10A	0.9621 (2)	0.6692 (2)	0.4327 (2)	0.0287 (5)
H10A	0.974438	0.604622	0.396544	0.034*
C11A	1.0518 (2)	0.6783 (2)	0.4822 (2)	0.0370 (6)
H11A	1.126927	0.620103	0.478996	0.044*
C12A	1.0347 (2)	0.7718 (3)	0.5372 (2)	0.0396 (7)
H12A	1.098518	0.775944	0.570598	0.048*

C13A	0.9274 (2)	0.8579 (2)	0.5438 (2)	0.0332 (6)
H13A	0.915715	0.921144	0.581422	0.040*
C14A	0.8365 (2)	0.8488 (2)	0.4933 (2)	0.0251 (5)
C15A	0.7189 (2)	0.7050 (2)	0.3221 (2)	0.0234 (5)
H15B	0.786892	0.706469	0.249761	0.028*
H15A	0.642586	0.748720	0.292155	0.028*
C16A	0.7070 (2)	0.5694 (2)	0.3902 (2)	0.0211 (5)
O1B	0.40872 (14)	0.71706 (16)	−0.05616 (15)	0.0301 (4)
O2B	0.42128 (15)	0.79833 (15)	−0.25663 (14)	0.0294 (4)
N6B	0.0357 (2)	0.8516 (2)	0.1637 (2)	0.0315 (5)
H6B	−0.013 (3)	0.833 (3)	0.233 (3)	0.052 (9)*
C7B	0.0746 (2)	0.7887 (2)	0.0777 (2)	0.0286 (6)
H7B	0.046635	0.716292	0.083241	0.034*
C8B	0.1587 (2)	0.8451 (2)	−0.0164 (2)	0.0246 (5)
C9B	0.1726 (2)	0.9511 (2)	0.0113 (2)	0.0231 (5)
C10B	0.2421 (2)	1.0462 (2)	−0.0491 (2)	0.0288 (5)
H10B	0.295011	1.048856	−0.126424	0.035*
C11B	0.2321 (2)	1.1357 (2)	0.0060 (2)	0.0357 (6)
H11B	0.278486	1.200916	−0.034338	0.043*
C12B	0.1546 (3)	1.1328 (2)	0.1208 (2)	0.0370 (7)
H12B	0.150596	1.195287	0.156885	0.044*
C13B	0.0848 (2)	1.0410 (2)	0.1814 (2)	0.0332 (6)
H13B	0.031985	1.039132	0.258598	0.040*
C14B	0.0942 (2)	0.9514 (2)	0.1258 (2)	0.0257 (5)
C15B	0.2217 (2)	0.8090 (2)	−0.1297 (2)	0.0298 (6)
H15C	0.201125	0.880232	−0.200565	0.036*
H15D	0.188453	0.737884	−0.128431	0.036*
C16B	0.3608 (2)	0.7721 (2)	−0.1485 (2)	0.0241 (5)

Atomic displacement parameters (Å²) for (2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.0267 (10)	0.0215 (10)	0.0165 (10)	−0.0068 (8)	−0.0043 (8)	−0.0070 (9)
N2A	0.0391 (12)	0.0241 (11)	0.0186 (11)	−0.0118 (9)	−0.0078 (9)	−0.0062 (10)
N3A	0.0253 (10)	0.0191 (10)	0.0198 (10)	−0.0055 (8)	−0.0070 (8)	−0.0045 (8)
N4A	0.0458 (13)	0.0213 (11)	0.0286 (13)	−0.0140 (10)	−0.0124 (11)	−0.0012 (10)
N5A	0.0380 (12)	0.0285 (12)	0.0190 (11)	−0.0115 (10)	−0.0070 (9)	−0.0078 (10)
C2A	0.0185 (11)	0.0251 (12)	0.0211 (12)	−0.0017 (9)	−0.0062 (9)	−0.0085 (10)
C4A	0.0240 (11)	0.0221 (12)	0.0236 (13)	−0.0031 (9)	−0.0071 (10)	−0.0056 (10)
C5A	0.0280 (12)	0.0264 (12)	0.0187 (12)	−0.0068 (10)	−0.0076 (10)	−0.0035 (10)
C6A	0.0218 (11)	0.0259 (12)	0.0185 (12)	−0.0029 (9)	−0.0041 (9)	−0.0080 (10)
N1B	0.0263 (10)	0.0227 (10)	0.0140 (10)	−0.0027 (8)	−0.0049 (8)	−0.0058 (8)
N2B	0.0245 (11)	0.0371 (12)	0.0141 (10)	−0.0041 (9)	−0.0030 (9)	−0.0075 (9)
N3B	0.0227 (10)	0.0259 (10)	0.0190 (10)	−0.0029 (8)	−0.0033 (8)	−0.0088 (9)
N4B	0.0247 (11)	0.0401 (13)	0.0206 (12)	−0.0017 (9)	−0.0027 (9)	−0.0093 (10)
N5B	0.0294 (11)	0.0344 (12)	0.0159 (11)	−0.0025 (10)	−0.0065 (9)	−0.0098 (10)
C2B	0.0297 (12)	0.0193 (11)	0.0161 (12)	−0.0043 (9)	−0.0059 (10)	−0.0062 (10)
C4B	0.0271 (12)	0.0178 (11)	0.0219 (12)	−0.0021 (9)	−0.0035 (10)	−0.0061 (10)
C5B	0.0315 (13)	0.0252 (12)	0.0159 (12)	−0.0024 (10)	−0.0046 (10)	−0.0064 (10)
C6B	0.0329 (12)	0.0184 (11)	0.0172 (12)	−0.0050 (10)	−0.0064 (10)	−0.0050 (10)
O1A	0.0415 (10)	0.0238 (9)	0.0144 (8)	−0.0099 (7)	−0.0042 (7)	−0.0036 (7)

O2A	0.0396 (10)	0.0257 (9)	0.0167 (8)	−0.0070 (7)	−0.0066 (7)	−0.0084 (7)
N6A	0.0299 (11)	0.0226 (10)	0.0296 (12)	−0.0020 (9)	−0.0098 (9)	−0.0119 (10)
C7A	0.0245 (12)	0.0260 (12)	0.0234 (13)	−0.0046 (10)	−0.0090 (10)	−0.0045 (11)
C8A	0.0265 (12)	0.0208 (11)	0.0165 (11)	−0.0043 (9)	−0.0043 (9)	−0.0047 (10)
C9A	0.0254 (11)	0.0203 (11)	0.0160 (11)	−0.0058 (9)	−0.0024 (9)	−0.0031 (10)
C10A	0.0285 (13)	0.0258 (13)	0.0233 (13)	−0.0030 (10)	−0.0009 (10)	−0.0019 (11)
C11A	0.0237 (13)	0.0387 (15)	0.0350 (15)	−0.0033 (11)	−0.0069 (11)	0.0046 (13)
C12A	0.0319 (14)	0.0469 (17)	0.0380 (16)	−0.0142 (13)	−0.0166 (12)	0.0023 (14)
C13A	0.0395 (14)	0.0345 (14)	0.0320 (15)	−0.0163 (12)	−0.0146 (12)	−0.0051 (12)
C14A	0.0287 (12)	0.0238 (12)	0.0213 (12)	−0.0062 (10)	−0.0075 (10)	−0.0022 (10)
C15A	0.0313 (12)	0.0210 (12)	0.0160 (12)	−0.0055 (10)	−0.0065 (10)	−0.0015 (10)
C16A	0.0236 (11)	0.0224 (12)	0.0168 (12)	−0.0037 (9)	−0.0061 (9)	−0.0042 (10)
O1B	0.0285 (9)	0.0409 (10)	0.0217 (9)	−0.0009 (8)	−0.0093 (7)	−0.0109 (8)
O2B	0.0332 (9)	0.0361 (10)	0.0205 (9)	−0.0049 (8)	−0.0072 (7)	−0.0101 (8)
N6B	0.0304 (12)	0.0353 (12)	0.0240 (12)	−0.0096 (10)	−0.0029 (10)	−0.0025 (10)
C7B	0.0299 (13)	0.0224 (12)	0.0332 (14)	−0.0055 (10)	−0.0097 (11)	−0.0053 (12)
C8B	0.0241 (12)	0.0253 (12)	0.0253 (13)	−0.0040 (10)	−0.0104 (10)	−0.0049 (11)
C9B	0.0240 (12)	0.0246 (12)	0.0189 (12)	−0.0047 (10)	−0.0080 (10)	−0.0013 (10)
C10B	0.0338 (13)	0.0287 (13)	0.0235 (13)	−0.0072 (11)	−0.0101 (11)	−0.0029 (11)
C11B	0.0481 (16)	0.0291 (14)	0.0332 (15)	−0.0159 (12)	−0.0158 (13)	−0.0009 (12)
C12B	0.0497 (16)	0.0307 (14)	0.0370 (16)	−0.0028 (13)	−0.0191 (13)	−0.0131 (13)
C13B	0.0360 (14)	0.0384 (15)	0.0253 (14)	−0.0009 (12)	−0.0117 (11)	−0.0095 (12)
C14B	0.0254 (12)	0.0264 (12)	0.0233 (13)	−0.0030 (10)	−0.0089 (10)	−0.0031 (11)
C15B	0.0326 (13)	0.0321 (14)	0.0302 (14)	−0.0050 (11)	−0.0135 (11)	−0.0110 (12)
C16B	0.0293 (12)	0.0246 (12)	0.0235 (13)	−0.0038 (10)	−0.0077 (10)	−0.0125 (11)

Geometric parameters (Å, º) for (2)

N1A—C6A	1.367 (3)	C7A—C8A	1.358 (3)
N1A—C2A	1.368 (3)	C7A—H7A	0.9500
N1A—H1A	1.03 (3)	C8A—C9A	1.430 (3)
N2A—C2A	1.323 (3)	C8A—C15A	1.494 (3)
N2A—H2A	0.87 (2)	C9A—C10A	1.408 (3)
N2A—H2B	0.91 (3)	C9A—C14A	1.411 (3)
N3A—C2A	1.331 (3)	C10A—C11A	1.372 (3)
N3A—C4A	1.353 (3)	C10A—H10A	0.9500
N4A—C4A	1.338 (3)	C11A—C12A	1.400 (4)
N4A—H4A	0.88 (3)	C11A—H11A	0.9500
N4A—H4B	0.89 (4)	C12A—C13A	1.375 (4)
N5A—C6A	1.343 (3)	C12A—H12A	0.9500
N5A—H5D	0.90 (3)	C13A—C14A	1.392 (3)
N5A—H5C	0.91 (3)	C13A—H13A	0.9500
C4A—C5A	1.401 (3)	C15A—C16A	1.519 (3)
C5A—C6A	1.373 (3)	C15A—H15B	0.9900
C5A—H5A	0.9500	C15A—H15A	0.9900
N1B—C2B	1.363 (3)	O1B—C16B	1.266 (3)
N1B—C6B	1.380 (3)	O2B—C16B	1.259 (3)
N1B—H1B	0.99 (3)	N6B—C14B	1.368 (3)
N2B—C2B	1.332 (3)	N6B—C7B	1.373 (3)
N2B—H2C	0.92 (3)	N6B—H6B	0.86 (3)
N2B—H2D	0.90 (3)	C7B—C8B	1.357 (3)

N3B—C2B	1.321 (3)	C7B—H7B	0.9500
N3B—C4B	1.366 (3)	C8B—C9B	1.437 (3)
N4B—C4B	1.342 (3)	C8B—C15B	1.491 (3)
N4B—H4C	0.91 (3)	C9B—C10B	1.400 (3)
N4B—H4D	0.89 (3)	C9B—C14B	1.415 (3)
N5B—C6B	1.333 (3)	C10B—C11B	1.377 (3)
N5B—H5F	0.92 (3)	C10B—H10B	0.9500
N5B—H5E	0.85 (3)	C11B—C12B	1.407 (4)
C4B—C5B	1.393 (3)	C11B—H11B	0.9500
C5B—C6B	1.373 (3)	C12B—C13B	1.375 (4)
C5B—H5B	0.9500	C12B—H12B	0.9500
O1A—C16A	1.258 (3)	C13B—C14B	1.383 (3)
O2A—C16A	1.264 (2)	C13B—H13B	0.9500
N6A—C14A	1.369 (3)	C15B—C16B	1.524 (3)
N6A—C7A	1.374 (3)	C15B—H15C	0.9900
N6A—H6A	0.91 (3)	C15B—H15D	0.9900

C6A—N1A—C2A	120.4 (2)	C14A—C9A—C8A	107.17 (18)
C6A—N1A—H1A	118.2 (16)	C11A—C10A—C9A	119.3 (2)
C2A—N1A—H1A	121.4 (16)	C11A—C10A—H10A	120.4
C2A—N2A—H2A	120.0 (17)	C9A—C10A—H10A	120.4
C2A—N2A—H2B	121.5 (17)	C10A—C11A—C12A	121.2 (2)
H2A—N2A—H2B	118 (2)	C10A—C11A—H11A	119.4
C2A—N3A—C4A	117.74 (18)	C12A—C11A—H11A	119.4
C4A—N4A—H4A	117 (2)	C13A—C12A—C11A	121.3 (2)
C4A—N4A—H4B	120 (2)	C13A—C12A—H12A	119.4
H4A—N4A—H4B	122 (3)	C11A—C12A—H12A	119.4
C6A—N5A—H5D	119.9 (18)	C12A—C13A—C14A	117.5 (2)
C6A—N5A—H5C	118.6 (17)	C12A—C13A—H13A	121.3
H5D—N5A—H5C	121 (2)	C14A—C13A—H13A	121.3
N2A—C2A—N3A	120.83 (19)	N6A—C14A—C13A	129.9 (2)
N2A—C2A—N1A	116.9 (2)	N6A—C14A—C9A	107.49 (19)
N3A—C2A—N1A	122.2 (2)	C13A—C14A—C9A	122.6 (2)
N4A—C4A—N3A	116.1 (2)	C8A—C15A—C16A	116.09 (19)
N4A—C4A—C5A	121.2 (2)	C8A—C15A—H15B	108.3
N3A—C4A—C5A	122.7 (2)	C16A—C15A—H15B	108.3
C6A—C5A—C4A	117.6 (2)	C8A—C15A—H15A	108.3
C6A—C5A—H5A	121.2	C16A—C15A—H15A	108.3
C4A—C5A—H5A	121.2	H15B—C15A—H15A	107.4
N5A—C6A—N1A	116.6 (2)	O1A—C16A—O2A	124.0 (2)
N5A—C6A—C5A	124.2 (2)	O1A—C16A—C15A	119.41 (18)
N1A—C6A—C5A	119.25 (19)	O2A—C16A—C15A	116.63 (19)
C2B—N1B—C6B	120.10 (19)	C14B—N6B—C7B	109.2 (2)
C2B—N1B—H1B	119.8 (17)	C14B—N6B—H6B	122 (2)
C6B—N1B—H1B	119.8 (17)	C7B—N6B—H6B	129 (2)
C2B—N2B—H2C	121.6 (19)	C8B—C7B—N6B	110.1 (2)
C2B—N2B—H2D	119.6 (16)	C8B—C7B—H7B	124.9
H2C—N2B—H2D	119 (2)	N6B—C7B—H7B	124.9
C2B—N3B—C4B	117.06 (19)	C7B—C8B—C9B	106.56 (19)
C4B—N4B—H4C	121.3 (15)	C7B—C8B—C15B	127.6 (2)
C4B—N4B—H4D	119.0 (18)	C9B—C8B—C15B	125.8 (2)

H4C—N4B—H4D	118 (2)	C10B—C9B—C14B	118.8 (2)
C6B—N5B—H5F	119.3 (16)	C10B—C9B—C8B	134.3 (2)
C6B—N5B—H5E	119.6 (19)	C14B—C9B—C8B	106.8 (2)
H5F—N5B—H5E	118 (3)	C11B—C10B—C9B	118.5 (2)
N3B—C2B—N2B	120.2 (2)	C11B—C10B—H10B	120.8
N3B—C2B—N1B	123.1 (2)	C9B—C10B—H10B	120.8
N2B—C2B—N1B	116.7 (2)	C10B—C11B—C12B	121.5 (2)
N4B—C4B—N3B	116.1 (2)	C10B—C11B—H11B	119.3
N4B—C4B—C5B	121.2 (2)	C12B—C11B—H11B	119.2
N3B—C4B—C5B	122.7 (2)	C13B—C12B—C11B	121.0 (2)
C6B—C5B—C4B	118.0 (2)	C13B—C12B—H12B	119.5
C6B—C5B—H5B	121.0	C11B—C12B—H12B	119.5
C4B—C5B—H5B	121.0	C12B—C13B—C14B	117.5 (2)
N5B—C6B—C5B	125.3 (2)	C12B—C13B—H13B	121.2
N5B—C6B—N1B	116.0 (2)	C14B—C13B—H13B	121.2
C5B—C6B—N1B	118.6 (2)	N6B—C14B—C13B	130.1 (2)
C14A—N6A—C7A	108.55 (18)	N6B—C14B—C9B	107.3 (2)
C14A—N6A—H6A	126.1 (18)	C13B—C14B—C9B	122.6 (2)
C7A—N6A—H6A	125.1 (18)	C8B—C15B—C16B	115.32 (19)
C8A—C7A—N6A	110.5 (2)	C8B—C15B—H15C	108.4
C8A—C7A—H7A	124.7	C16B—C15B—H15C	108.4
N6A—C7A—H7A	124.7	C8B—C15B—H15D	108.4
C7A—C8A—C9A	106.24 (18)	C16B—C15B—H15D	108.4
C7A—C8A—C15A	127.8 (2)	H15C—C15B—H15D	107.5
C9A—C8A—C15A	125.91 (18)	O2B—C16B—O1B	124.0 (2)
C10A—C9A—C14A	118.1 (2)	O2B—C16B—C15B	117.0 (2)
C10A—C9A—C8A	134.68 (19)	O1B—C16B—C15B	119.0 (2)

C4A—N3A—C2A—N2A	179.7 (2)	C7A—N6A—C14A—C13A	178.5 (3)
C4A—N3A—C2A—N1A	0.0 (3)	C7A—N6A—C14A—C9A	−0.5 (3)
C6A—N1A—C2A—N2A	−176.69 (19)	C12A—C13A—C14A—N6A	−179.3 (3)
C6A—N1A—C2A—N3A	3.0 (3)	C12A—C13A—C14A—C9A	−0.4 (4)
C2A—N3A—C4A—N4A	177.6 (2)	C10A—C9A—C14A—N6A	−179.7 (2)
C2A—N3A—C4A—C5A	−2.5 (3)	C8A—C9A—C14A—N6A	0.5 (3)
N4A—C4A—C5A—C6A	−178.2 (2)	C10A—C9A—C14A—C13A	1.3 (4)
N3A—C4A—C5A—C6A	1.9 (3)	C8A—C9A—C14A—C13A	−178.6 (2)
C2A—N1A—C6A—N5A	178.57 (19)	C7A—C8A—C15A—C16A	−115.0 (3)
C2A—N1A—C6A—C5A	−3.5 (3)	C9A—C8A—C15A—C16A	67.9 (3)
C4A—C5A—C6A—N5A	178.9 (2)	C8A—C15A—C16A—O1A	22.2 (3)
C4A—C5A—C6A—N1A	1.1 (3)	C8A—C15A—C16A—O2A	−158.79 (19)
C4B—N3B—C2B—N2B	−179.27 (19)	C14B—N6B—C7B—C8B	0.4 (3)
C4B—N3B—C2B—N1B	0.3 (3)	N6B—C7B—C8B—C9B	−0.7 (3)
C6B—N1B—C2B—N3B	4.0 (3)	N6B—C7B—C8B—C15B	−178.4 (2)
C6B—N1B—C2B—N2B	−176.36 (19)	C7B—C8B—C9B—C10B	−178.4 (3)
C2B—N3B—C4B—N4B	176.1 (2)	C15B—C8B—C9B—C10B	−0.7 (4)
C2B—N3B—C4B—C5B	−5.2 (3)	C7B—C8B—C9B—C14B	0.8 (2)
N4B—C4B—C5B—C6B	−175.8 (2)	C15B—C8B—C9B—C14B	178.5 (2)
N3B—C4B—C5B—C6B	5.7 (3)	C14B—C9B—C10B—C11B	0.6 (3)
C4B—C5B—C6B—N5B	−178.2 (2)	C8B—C9B—C10B—C11B	179.7 (2)
C4B—C5B—C6B—N1B	−1.1 (3)	C9B—C10B—C11B—C12B	0.3 (4)
C2B—N1B—C6B—N5B	173.9 (2)	C10B—C11B—C12B—C13B	−0.9 (4)

C2B—N1B—C6B—C5B	−3.5 (3)	C11B—C12B—C13B—C14B	0.4 (4)
C14A—N6A—C7A—C8A	0.4 (3)	C7B—N6B—C14B—C13B	179.9 (2)
N6A—C7A—C8A—C9A	−0.1 (3)	C7B—N6B—C14B—C9B	0.1 (3)
N6A—C7A—C8A—C15A	−177.6 (2)	C12B—C13B—C14B—N6B	−179.3 (2)
C7A—C8A—C9A—C10A	179.9 (3)	C12B—C13B—C14B—C9B	0.5 (4)
C15A—C8A—C9A—C10A	−2.5 (4)	C10B—C9B—C14B—N6B	178.8 (2)
C7A—C8A—C9A—C14A	−0.2 (3)	C8B—C9B—C14B—N6B	−0.6 (2)
C15A—C8A—C9A—C14A	177.4 (2)	C10B—C9B—C14B—C13B	−1.0 (3)
C14A—C9A—C10A—C11A	−1.4 (4)	C8B—C9B—C14B—C13B	179.6 (2)
C8A—C9A—C10A—C11A	178.4 (3)	C7B—C8B—C15B—C16B	−118.5 (3)
C9A—C10A—C11A—C12A	0.8 (4)	C9B—C8B—C15B—C16B	64.2 (3)
C10A—C11A—C12A—C13A	0.1 (4)	C8B—C15B—C16B—O2B	−147.0 (2)
C11A—C12A—C13A—C14A	−0.2 (4)	C8B—C15B—C16B—O1B	33.4 (3)

Hydrogen-bond geometry (Å, º) for (2)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O1*A*	1.02 (3)	1.68 (3)	2.696 (3)	172 (3)
N1*B*—H1*B*···O1*B*	0.99 (4)	1.74 (4)	2.733 (3)	179 (5)
N2*A*—H2*A*···O2*B*^{i}	0.87 (2)	2.07 (2)	2.886 (2)	156 (2)
N2*A*—H2*B*···O2*A*	0.91 (3)	1.90 (3)	2.798 (3)	171 (3)
N4*A*—H4*A*···O2*B*^{ii}	0.88 (3)	2.27 (3)	3.037 (3)	146 (3)
N5*A*—H5*D*···N5*B*^{iii}	0.90 (3)	2.47 (3)	3.282 (3)	152 (2)
N5*A*—H5*C*···O1*B*^{iv}	0.91 (3)	1.98 (3)	2.873 (3)	163 (2)
N1*B*—H1*B*···O1*B*	0.99 (3)	1.74 (3)	2.733 (2)	179 (3)
N2*B*—H2*C*···O2*A*	0.92 (3)	1.94 (3)	2.844 (3)	167 (3)
N2*B*—H2*D*···N5*A*^{iv}	0.90 (3)	2.41 (3)	3.278 (3)	161 (2)
N4*B*—H4*C*···N3*B*^{v}	0.91 (3)	2.09 (3)	3.000 (3)	174 (2)
N4*B*—H4*D*···O2*A*^{v}	0.89 (3)	2.34 (3)	3.042 (3)	135 (2)
N5*B*—H5*F*···O1*A*^{vi}	0.92 (3)	2.14 (2)	2.885 (2)	138 (2)
N5*B*—H5*E*···O2*B*	0.85 (3)	1.93 (3)	2.764 (3)	169 (3)
N6*A*—H6*A*···N3*A*^{vii}	0.91 (3)	2.03 (3)	2.932 (2)	173 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*, *y*−1, *z*+1; (iii) *x*, *y*, *z*+1; (iv) −*x*+1, −*y*+1, −*z*+1; (v) −*x*+2, −*y*+1, −*z*; (vi) *x*, *y*, *z*−1; (vii) *x*, *y*+1, *z*.

(3)

Crystal data

$C_5H_2BrO_2S·C_4H_8N_5$
M_r = 332.19
Triclinic, $P\overline{1}$
a = 8.4348 (5) Å
b = 10.5501 (7) Å
c = 14.0931 (9) Å
α = 77.558 (5)°
β = 86.820 (5)°
γ = 78.896 (5)°
V = 1201.61 (14) Å^3

Z = 4
F(000) = 664
D_x = 1.836 Mg m^{-3}
Cu *K*α radiation, λ = 1.54184 Å
Cell parameters from 4975 reflections
θ = 3.2–76.8°
μ = 6.34 mm^{-1}
T = 120 K
Prism, pale yellow
0.15 × 0.12 × 0.06 mm

Data collection

SuperNova, Dual, Cu at zero, Atlas diffractometer
Radiation source: sealed X-ray tube
Detector resolution: 10.6501 pixels mm^{-1}
ω scans

Absorption correction: multi-scan
CrysAlis PRO, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min} = 0.450, T_{max} = 0.702
10725 measured reflections
4975 independent reflections
4697 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 76.8°, θ_{min} = 3.2°
h = −10→10
k = −13→11
l = −17→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.083
S = 1.04
4975 reflections
382 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 1.147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 1.23 e $Å^{-3}$
$\Delta\rho_{min}$ = −1.27 e $Å^{-3}$
Extinction correction: *SHELXL2016*/6 (Sheldrick 2016), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00064 (12)

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) for (3)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.02698 (4)	0.06825 (3)	−0.14984 (2)	0.03511 (10)
S1	0.15487 (7)	0.21229 (5)	−0.00622 (4)	0.02401 (13)
O1A	0.2619 (2)	0.37579 (16)	0.11715 (11)	0.0246 (3)
O2A	0.2815 (2)	0.55073 (17)	−0.00169 (12)	0.0270 (4)
C11A	0.2507 (3)	0.4364 (2)	0.03004 (16)	0.0207 (4)
C7A	0.1964 (3)	0.3697 (2)	−0.04261 (16)	0.0208 (4)
C8A	0.1677 (4)	0.4164 (2)	−0.13938 (18)	0.0316 (5)
H8A	0.185221	0.500976	−0.173009	0.038*
C9A	0.1096 (4)	0.3277 (2)	−0.18526 (18)	0.0319 (5)
H9A	0.083391	0.345353	−0.252089	0.038*
C10A	0.0965 (3)	0.2144 (2)	−0.12113 (16)	0.0231 (4)
Br2	1.31377 (3)	0.54736 (3)	0.55182 (2)	0.03121 (9)
S2	0.94653 (6)	0.62021 (5)	0.59563 (4)	0.01968 (12)
O1B	0.60183 (19)	0.72305 (17)	0.62508 (11)	0.0248 (3)
O2B	0.55136 (19)	0.83865 (17)	0.47353 (11)	0.0234 (3)

C11B	0.6436 (3)	0.7657 (2)	0.53808 (15)	0.0181 (4)
C7B	0.8162 (3)	0.7265 (2)	0.51181 (15)	0.0186 (4)
C8B	0.8949 (3)	0.7699 (2)	0.42654 (16)	0.0211 (4)
H8B	0.842271	0.828710	0.371803	0.025*
C9B	1.0623 (3)	0.7181 (2)	0.42881 (17)	0.0241 (4)
H9B	1.135481	0.737702	0.376391	0.029*
C10B	1.1052 (3)	0.6364 (2)	0.51582 (18)	0.0227 (4)
N1A	0.3668 (2)	0.70486 (18)	0.11369 (13)	0.0198 (4)
C2A	0.4144 (3)	0.8212 (2)	0.07309 (16)	0.0215 (4)
N3A	0.4690 (2)	0.89510 (19)	0.12434 (13)	0.0216 (4)
C4A	0.4776 (3)	0.8492 (2)	0.22232 (15)	0.0191 (4)
C5A	0.4315 (3)	0.7309 (2)	0.26914 (15)	0.0197 (4)
H5A	0.438853	0.701501	0.337654	0.024*
C6A	0.3746 (2)	0.6576 (2)	0.21230 (15)	0.0187 (4)
N2A	0.4055 (3)	0.8609 (2)	−0.02342 (15)	0.0285 (4)
N4A	0.5342 (3)	0.9261 (2)	0.27279 (14)	0.0242 (4)
N5A	0.3245 (2)	0.54320 (19)	0.24493 (15)	0.0234 (4)
N1B	0.2956 (2)	0.81688 (18)	0.66917 (13)	0.0179 (3)
C2B	0.1767 (3)	0.8884 (2)	0.60620 (15)	0.0169 (4)
N3B	0.0212 (2)	0.90751 (17)	0.63179 (13)	0.0174 (3)
C4B	−0.0183 (2)	0.8431 (2)	0.72132 (15)	0.0169 (4)
C5B	0.0972 (3)	0.7664 (2)	0.78848 (15)	0.0186 (4)
H5B	0.066027	0.723553	0.851199	0.022*
C6B	0.2562 (3)	0.7555 (2)	0.76045 (15)	0.0173 (4)
N2B	0.2237 (2)	0.93808 (19)	0.51704 (13)	0.0193 (4)
N4B	−0.1758 (2)	0.8567 (2)	0.74525 (14)	0.0217 (4)
N5B	0.3803 (2)	0.6903 (2)	0.81891 (14)	0.0219 (4)
H1A	0.337 (4)	0.660 (3)	0.079 (2)	0.027 (7)*
H2A	0.382 (4)	0.806 (3)	−0.058 (2)	0.034 (8)*
H2B	0.440 (4)	0.933 (3)	−0.053 (2)	0.031 (8)*
H4A	0.543 (4)	0.897 (3)	0.334 (3)	0.035 (8)*
H4B	0.579 (4)	0.990 (3)	0.241 (2)	0.020 (7)*
H5C	0.299 (3)	0.497 (3)	0.205 (2)	0.020 (7)*
H5D	0.336 (4)	0.505 (3)	0.303 (3)	0.036 (9)*
H1B	0.401 (4)	0.791 (3)	0.647 (2)	0.028 (7)*
H2D	0.324 (4)	0.916 (3)	0.502 (2)	0.023 (7)*
H2C	0.148 (4)	0.986 (3)	0.477 (2)	0.027 (7)*
H4D	−0.203 (4)	0.803 (3)	0.793 (3)	0.034 (8)*
H4C	−0.243 (4)	0.900 (3)	0.702 (2)	0.031 (8)*
H5E	0.474 (4)	0.677 (3)	0.795 (2)	0.032 (8)*
H5F	0.357 (4)	0.631 (3)	0.879 (2)	0.032 (8)*

Atomic displacement parameters (Å²) for (3)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.05728 (19)	0.02952 (15)	0.02619 (14)	−0.02413 (13)	0.00237 (11)	−0.00887 (10)
S1	0.0354 (3)	0.0217 (3)	0.0171 (2)	−0.0125 (2)	0.0030 (2)	−0.00309 (19)
O1A	0.0326 (9)	0.0245 (8)	0.0178 (7)	−0.0114 (7)	−0.0009 (6)	−0.0010 (6)
O2A	0.0432 (10)	0.0222 (8)	0.0183 (7)	−0.0151 (7)	−0.0024 (7)	−0.0011 (6)
C11A	0.0214 (10)	0.0229 (10)	0.0177 (10)	−0.0050 (8)	0.0007 (8)	−0.0031 (8)
C7A	0.0236 (10)	0.0192 (10)	0.0200 (10)	−0.0062 (8)	0.0002 (8)	−0.0029 (8)

C8A	0.0532 (16)	0.0197 (11)	0.0231 (11)	−0.0125 (11)	−0.0091 (10)	0.0005 (9)
C9A	0.0508 (16)	0.0237 (12)	0.0231 (11)	−0.0102 (11)	−0.0111 (10)	−0.0032 (9)
C10A	0.0276 (11)	0.0227 (11)	0.0218 (10)	−0.0084 (9)	0.0014 (8)	−0.0077 (8)
Br2	0.01766 (13)	0.02918 (15)	0.04900 (18)	0.00050 (9)	−0.00452 (10)	−0.01627 (11)
S2	0.0179 (2)	0.0191 (2)	0.0216 (2)	−0.00378 (18)	−0.00101 (18)	−0.00284 (19)
O1B	0.0188 (7)	0.0339 (9)	0.0179 (7)	−0.0041 (6)	0.0010 (6)	0.0014 (6)
O2B	0.0190 (7)	0.0288 (8)	0.0186 (7)	−0.0010 (6)	0.0009 (6)	0.0004 (6)
C11B	0.0180 (10)	0.0191 (10)	0.0182 (9)	−0.0062 (8)	0.0008 (7)	−0.0039 (8)
C7B	0.0190 (10)	0.0184 (10)	0.0190 (10)	−0.0045 (8)	0.0004 (8)	−0.0044 (8)
C8B	0.0219 (10)	0.0204 (10)	0.0213 (10)	−0.0051 (8)	0.0038 (8)	−0.0047 (8)
C9B	0.0219 (11)	0.0232 (11)	0.0289 (11)	−0.0066 (8)	0.0061 (9)	−0.0088 (9)
C10B	0.0173 (10)	0.0201 (10)	0.0335 (12)	−0.0044 (8)	0.0013 (8)	−0.0110 (9)
N1A	0.0266 (9)	0.0176 (9)	0.0156 (8)	−0.0073 (7)	−0.0016 (7)	−0.0012 (7)
C2A	0.0264 (11)	0.0206 (10)	0.0168 (10)	−0.0057 (8)	−0.0013 (8)	−0.0007 (8)
N3A	0.0299 (10)	0.0204 (9)	0.0151 (8)	−0.0089 (7)	−0.0016 (7)	−0.0009 (7)
C4A	0.0198 (10)	0.0205 (10)	0.0157 (10)	−0.0028 (8)	0.0008 (7)	−0.0022 (8)
C5A	0.0209 (10)	0.0214 (10)	0.0150 (9)	−0.0039 (8)	0.0005 (7)	−0.0001 (8)
C6A	0.0169 (9)	0.0197 (10)	0.0168 (9)	−0.0016 (8)	0.0018 (7)	0.0002 (8)
N2A	0.0476 (13)	0.0241 (10)	0.0162 (9)	−0.0166 (9)	−0.0034 (8)	−0.0002 (8)
N4A	0.0320 (10)	0.0258 (10)	0.0161 (9)	−0.0105 (8)	−0.0019 (7)	−0.0021 (7)
N5A	0.0307 (10)	0.0191 (9)	0.0200 (9)	−0.0089 (8)	−0.0001 (7)	0.0002 (8)
N1B	0.0165 (8)	0.0200 (9)	0.0165 (8)	−0.0044 (7)	0.0007 (6)	−0.0014 (7)
C2B	0.0190 (9)	0.0132 (9)	0.0188 (10)	−0.0044 (7)	0.0001 (7)	−0.0032 (7)
N3B	0.0182 (8)	0.0170 (8)	0.0161 (8)	−0.0042 (6)	0.0012 (6)	−0.0014 (6)
C4B	0.0195 (10)	0.0148 (9)	0.0173 (10)	−0.0045 (8)	0.0023 (8)	−0.0047 (8)
C5B	0.0209 (10)	0.0190 (10)	0.0159 (9)	−0.0055 (8)	0.0024 (8)	−0.0029 (8)
C6B	0.0214 (10)	0.0151 (9)	0.0161 (9)	−0.0041 (7)	0.0002 (7)	−0.0044 (7)
N2B	0.0164 (9)	0.0217 (9)	0.0172 (8)	−0.0038 (7)	0.0013 (7)	0.0014 (7)
N4B	0.0181 (9)	0.0269 (10)	0.0180 (9)	−0.0053 (7)	0.0018 (7)	0.0004 (8)
N5B	0.0188 (9)	0.0256 (10)	0.0189 (9)	−0.0025 (7)	−0.0008 (7)	−0.0007 (7)

Geometric parameters (Å, º) for (3)

Br1—C10A	1.879 (2)	N1A—C2A	1.362 (3)
S1—C10A	1.713 (2)	N1A—C6A	1.372 (3)
S1—C7A	1.727 (2)	C2A—N3A	1.327 (3)
O1A—C11A	1.255 (3)	C2A—N2A	1.336 (3)
O2A—C11A	1.265 (3)	N3A—C4A	1.362 (3)
C11A—C7A	1.496 (3)	C4A—N4A	1.352 (3)
C7A—C8A	1.365 (3)	C4A—C5A	1.396 (3)
C8A—C9A	1.417 (3)	C5A—C6A	1.386 (3)
C9A—C10A	1.353 (3)	C6A—N5A	1.335 (3)
Br2—C10B	1.869 (2)	N1B—C6B	1.364 (3)
S2—C10B	1.710 (2)	N1B—C2B	1.371 (3)
S2—C7B	1.720 (2)	C2B—N2B	1.324 (3)
O1B—C11B	1.267 (3)	C2B—N3B	1.330 (3)
O2B—C11B	1.254 (3)	N3B—C4B	1.355 (3)
C11B—C7B	1.484 (3)	C4B—N4B	1.340 (3)
C7B—C8B	1.373 (3)	C4B—C5B	1.403 (3)
C8B—C9B	1.412 (3)	C5B—C6B	1.367 (3)
C9B—C10B	1.361 (3)	C6B—N5B	1.350 (3)

C10A—S1—C7A	91.15 (11)	C2A—N1A—C6A	120.4 (2)
O1A—C11A—O2A	124.9 (2)	N3A—C2A—N2A	119.2 (2)
O1A—C11A—C7A	118.3 (2)	N3A—C2A—N1A	123.4 (2)
O2A—C11A—C7A	116.80 (19)	N2A—C2A—N1A	117.4 (2)
C8A—C7A—C11A	129.4 (2)	C2A—N3A—C4A	116.84 (19)
C8A—C7A—S1	110.56 (18)	N4A—C4A—N3A	115.67 (19)
C11A—C7A—S1	120.01 (16)	N4A—C4A—C5A	121.24 (19)
C7A—C8A—C9A	114.1 (2)	N3A—C4A—C5A	123.1 (2)
C10A—C9A—C8A	110.9 (2)	C6A—C5A—C4A	117.77 (19)
C9A—C10A—S1	113.29 (18)	N5A—C6A—N1A	115.8 (2)
C9A—C10A—Br1	125.79 (18)	N5A—C6A—C5A	125.7 (2)
S1—C10A—Br1	120.90 (13)	N1A—C6A—C5A	118.6 (2)
C10B—S2—C7B	90.63 (11)	C6B—N1B—C2B	120.24 (18)
O2B—C11B—O1B	125.2 (2)	N2B—C2B—N3B	120.70 (19)
O2B—C11B—C7B	118.04 (19)	N2B—C2B—N1B	116.71 (19)
O1B—C11B—C7B	116.76 (19)	N3B—C2B—N1B	122.59 (19)
C8B—C7B—C11B	128.1 (2)	C2B—N3B—C4B	116.98 (18)
C8B—C7B—S2	111.68 (16)	N4B—C4B—N3B	117.04 (19)
C11B—C7B—S2	120.14 (16)	N4B—C4B—C5B	119.93 (19)
C7B—C8B—C9B	112.9 (2)	N3B—C4B—C5B	123.02 (19)
C10B—C9B—C8B	111.2 (2)	C6B—C5B—C4B	117.62 (19)
C9B—C10B—S2	113.57 (17)	N5B—C6B—N1B	116.61 (19)
C9B—C10B—Br2	126.09 (17)	N5B—C6B—C5B	124.0 (2)
S2—C10B—Br2	120.33 (14)	N1B—C6B—C5B	119.32 (19)

Hydrogen-bond geometry (Å, º) for (3)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O2*A*	0.83 (3)	1.92 (3)	2.743 (3)	177 (3)
N1*B*—H1*B*···O1*B*	0.94 (3)	1.75 (3)	2.672 (2)	170 (3)
N2*A*—H2*A*···N5*B*^{i}	0.89 (3)	2.33 (3)	3.185 (3)	162 (3)
N2*A*—H2*B*···N3*A*^{ii}	0.88 (3)	2.13 (3)	3.013 (3)	178 (3)
N2*B*—H2*C*···N3*B*^{iii}	0.88 (3)	2.13 (3)	3.005 (3)	174 (3)
N2*B*—H2*D*···O2*B*	0.86 (3)	1.99 (3)	2.844 (2)	172 (3)
N4*A*—H4*A*···O2*B*	0.85 (4)	1.93 (4)	2.783 (2)	177 (3)
N4*B*—H4*D*···O1*A*^{iv}	0.83 (4)	2.16 (4)	2.964 (3)	162 (3)
N5*A*—H5*C*···O1*A*	0.88 (3)	2.04 (3)	2.911 (3)	174 (3)
N5*A*—H5*D*···O1*B*^{v}	0.83 (4)	2.37 (3)	2.978 (3)	131 (3)
N5*B*—H5*E*···O1*B*	0.84 (3)	2.56 (3)	3.217 (2)	136 (3)
N5*B*—H5*E*···O1*A*^{v}	0.84 (3)	2.52 (3)	3.103 (2)	128 (2)
N5*B*—H5*F*···O2*A*^{vi}	0.97 (3)	1.85 (3)	2.809 (3)	167 (3)

Symmetry codes: (i) *x*, *y*, *z*−1; (ii) −*x*+1, −*y*+2, −*z*; (iii) −*x*, −*y*+2, −*z*+1; (iv) −*x*, −*y*+1, −*z*+1; (v) −*x*+1, −*y*+1, −*z*+1; (vi) *x*, *y*, *z*+1.

(4)

Crystal data

$C_5H_2ClO_2S·C_4H_8N_5$	*a* = 8.3306 (5) Å
M_r = 287.73	*b* = 10.4403 (5) Å
Triclinic, $P\overline{1}$	*c* = 14.0361 (5) Å

α = 78.406 (4)°
β = 87.357 (4)°
γ = 78.608 (4)°
V = 1172.28 (10) Å^3
Z = 4
F(000) = 592
D_x = 1.630 Mg m^{-3}

Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 4863 reflections
θ = 3.2–76.9°
μ = 4.61 mm^{-1}
T = 120 K
Block, yellow
0.36 × 0.15 × 0.12 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
ω scans
Absorption correction: multi-scan *CrysAlis PRO*, Agilent Technologies, Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET) (compiled Aug 13 2014,18:06:01) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

T_{min} = 0.288, T_{max} = 0.608
10298 measured reflections
4863 independent reflections
4401 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 76.9°, θ_{min} = 3.2°
h = −10→10
k = −10→13
l = −14→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.108
S = 1.05
4863 reflections
381 parameters
0 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.7344P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 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 (Å^2) for (4)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.97092 (8)	0.92958 (5)	1.14832 (4)	0.03655 (15)
S1	0.84726 (6)	0.79481 (5)	1.00626 (3)	0.02449 (13)
O1A	0.74189 (18)	0.62714 (14)	0.88237 (10)	0.0253 (3)
O2A	0.72412 (19)	0.45143 (14)	1.00078 (10)	0.0267 (3)
C11A	0.7541 (2)	0.56700 (19)	0.96914 (14)	0.0211 (4)
C7A	0.8095 (2)	0.63513 (19)	1.04192 (14)	0.0215 (4)
C8A	0.8426 (3)	0.5877 (2)	1.13853 (16)	0.0293 (4)
H8A	0.828531	0.501787	1.171564	0.035*
C9A	0.8999 (3)	0.6785 (2)	1.18457 (16)	0.0309 (4)
H9A	0.928577	0.661076	1.251303	0.037*
C10A	0.9086 (2)	0.7934 (2)	1.12145 (15)	0.0250 (4)
Cl2	−0.31592 (6)	0.44909 (5)	0.45244 (5)	0.03503 (15)
S2	0.03987 (5)	0.38673 (4)	0.40489 (3)	0.02099 (12)
O1B	0.39270 (16)	0.28712 (15)	0.37649 (10)	0.0243 (3)
O2B	0.44317 (16)	0.16724 (14)	0.52650 (10)	0.0237 (3)

C11B	0.3500 (2)	0.24198 (19)	0.46245 (14)	0.0191 (3)
C7B	0.1743 (2)	0.27972 (18)	0.48818 (14)	0.0191 (3)
C8B	0.0952 (2)	0.2339 (2)	0.57201 (14)	0.0225 (4)
H8B	0.149814	0.174212	0.626188	0.027*
C9B	−0.0752 (2)	0.2838 (2)	0.57023 (16)	0.0257 (4)
H9B	−0.148465	0.261771	0.622115	0.031*
C10B	−0.1203 (2)	0.3673 (2)	0.48452 (16)	0.0245 (4)
N1A	0.6369 (2)	0.29387 (16)	0.88583 (12)	0.0205 (3)
N3A	0.5296 (2)	0.10389 (16)	0.87502 (11)	0.0214 (3)
N2A	0.6005 (2)	0.13785 (19)	1.02267 (13)	0.0274 (4)
N4A	0.4573 (2)	0.07309 (17)	0.72668 (13)	0.0239 (3)
N5A	0.6783 (2)	0.45540 (17)	0.75449 (13)	0.0233 (3)
C2A	0.5874 (2)	0.17809 (19)	0.92624 (14)	0.0212 (4)
C4A	0.5168 (2)	0.15016 (18)	0.77724 (13)	0.0196 (4)
C5A	0.5634 (2)	0.26873 (19)	0.73059 (14)	0.0206 (4)
H5A	0.552917	0.298355	0.662196	0.025*
C6A	0.6255 (2)	0.34172 (19)	0.78741 (14)	0.0194 (3)
N1B	0.70232 (19)	0.18810 (16)	0.32963 (12)	0.0192 (3)
N3B	0.97979 (19)	0.08982 (15)	0.36771 (11)	0.0185 (3)
N2B	0.7728 (2)	0.06219 (16)	0.48077 (12)	0.0205 (3)
N4B	1.1806 (2)	0.13731 (18)	0.25558 (13)	0.0230 (3)
N5B	0.6189 (2)	0.31598 (18)	0.17952 (13)	0.0232 (3)
C2B	0.8214 (2)	0.11275 (18)	0.39271 (14)	0.0181 (3)
C4B	1.0205 (2)	0.15386 (18)	0.27877 (13)	0.0185 (3)
C5B	0.9052 (2)	0.23341 (18)	0.21140 (13)	0.0191 (3)
H5B	0.937720	0.275665	0.149122	0.023*
C6B	0.7431 (2)	0.24803 (18)	0.23886 (13)	0.0186 (3)
H1A	0.674 (4)	0.345 (3)	0.922 (2)	0.038 (8)*
H2A	0.620 (4)	0.191 (3)	1.057 (2)	0.035 (7)*
H2B	0.561 (3)	0.068 (3)	1.051 (2)	0.025 (6)*
H4A	0.417 (3)	−0.003 (3)	0.7624 (19)	0.025 (6)*
H4B	0.436 (3)	0.102 (3)	0.663 (2)	0.026 (6)*
H5D	0.710 (3)	0.500 (3)	0.794 (2)	0.025 (6)*
H5C	0.660 (3)	0.500 (3)	0.695 (2)	0.026 (6)*
H1B	0.600 (4)	0.213 (3)	0.352 (2)	0.040 (8)*
H2C	0.672 (4)	0.087 (3)	0.496 (2)	0.035 (7)*
H2D	0.846 (3)	0.019 (3)	0.5240 (19)	0.022 (6)*
H4C	1.251 (4)	0.095 (3)	0.300 (2)	0.027 (6)*
H4D	1.208 (4)	0.192 (3)	0.207 (3)	0.043 (8)*
H5F	0.646 (4)	0.377 (3)	0.124 (2)	0.036 (7)*
H5E	0.520 (4)	0.332 (3)	0.202 (2)	0.033 (7)*

Atomic displacement parameters (Å²) for (4)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0527 (3)	0.0301 (3)	0.0345 (3)	−0.0201 (2)	−0.0006 (2)	−0.0123 (2)
S1	0.0321 (3)	0.0223 (2)	0.0213 (2)	−0.01014 (18)	0.00240 (18)	−0.00517 (17)
O1A	0.0296 (7)	0.0261 (7)	0.0211 (7)	−0.0092 (6)	−0.0009 (5)	−0.0029 (5)
O2A	0.0374 (8)	0.0247 (7)	0.0209 (6)	−0.0132 (6)	−0.0016 (6)	−0.0041 (5)
C11A	0.0199 (8)	0.0226 (9)	0.0213 (9)	−0.0045 (7)	0.0011 (7)	−0.0054 (7)
C7A	0.0201 (8)	0.0220 (9)	0.0228 (9)	−0.0038 (7)	−0.0001 (7)	−0.0055 (7)

C8A	0.0399 (12)	0.0213 (9)	0.0275 (10)	−0.0081 (8)	−0.0068 (9)	−0.0029 (8)
C9A	0.0396 (12)	0.0270 (10)	0.0275 (10)	−0.0068 (9)	−0.0073 (9)	−0.0069 (8)
C10A	0.0239 (9)	0.0253 (10)	0.0289 (10)	−0.0063 (7)	0.0001 (8)	−0.0106 (8)
Cl2	0.0169 (2)	0.0324 (3)	0.0582 (3)	0.00100 (18)	−0.0057 (2)	−0.0191 (2)
S2	0.0177 (2)	0.0200 (2)	0.0255 (2)	−0.00281 (16)	−0.00296 (16)	−0.00501 (17)
O1B	0.0173 (6)	0.0316 (7)	0.0210 (6)	−0.0034 (5)	0.0007 (5)	0.0004 (5)
O2B	0.0181 (6)	0.0292 (7)	0.0206 (6)	−0.0009 (5)	−0.0010 (5)	−0.0010 (5)
C11B	0.0175 (8)	0.0207 (8)	0.0206 (8)	−0.0053 (7)	−0.0001 (7)	−0.0059 (7)
C7B	0.0174 (8)	0.0178 (8)	0.0230 (9)	−0.0039 (7)	0.0006 (7)	−0.0060 (7)
C8B	0.0220 (9)	0.0227 (9)	0.0239 (9)	−0.0051 (7)	0.0027 (7)	−0.0069 (7)
C9B	0.0217 (9)	0.0257 (10)	0.0325 (10)	−0.0066 (7)	0.0072 (8)	−0.0120 (8)
C10B	0.0161 (8)	0.0212 (9)	0.0398 (11)	−0.0035 (7)	0.0000 (8)	−0.0145 (8)
N1A	0.0232 (8)	0.0195 (8)	0.0195 (7)	−0.0060 (6)	−0.0021 (6)	−0.0028 (6)
N3A	0.0260 (8)	0.0206 (8)	0.0177 (7)	−0.0063 (6)	−0.0013 (6)	−0.0018 (6)
N2A	0.0409 (10)	0.0252 (9)	0.0187 (8)	−0.0145 (8)	−0.0040 (7)	−0.0017 (7)
N4A	0.0279 (8)	0.0251 (8)	0.0202 (8)	−0.0092 (7)	−0.0020 (6)	−0.0038 (6)
N5A	0.0281 (8)	0.0209 (8)	0.0210 (8)	−0.0078 (7)	−0.0010 (6)	−0.0013 (7)
C2A	0.0220 (9)	0.0209 (9)	0.0201 (9)	−0.0042 (7)	−0.0005 (7)	−0.0027 (7)
C4A	0.0180 (8)	0.0211 (9)	0.0187 (8)	−0.0015 (7)	−0.0004 (7)	−0.0036 (7)
C5A	0.0199 (8)	0.0225 (9)	0.0186 (8)	−0.0034 (7)	−0.0003 (7)	−0.0030 (7)
C6A	0.0166 (8)	0.0195 (8)	0.0199 (8)	−0.0013 (6)	0.0014 (6)	−0.0011 (7)
N1B	0.0165 (7)	0.0210 (8)	0.0199 (7)	−0.0035 (6)	−0.0002 (6)	−0.0034 (6)
N3B	0.0173 (7)	0.0174 (7)	0.0207 (7)	−0.0034 (6)	−0.0002 (6)	−0.0034 (6)
N2B	0.0168 (8)	0.0217 (8)	0.0209 (8)	−0.0023 (6)	0.0002 (6)	−0.0006 (6)
N4B	0.0164 (7)	0.0291 (9)	0.0219 (8)	−0.0036 (6)	0.0014 (6)	−0.0019 (7)
N5B	0.0174 (8)	0.0275 (8)	0.0227 (8)	−0.0016 (6)	−0.0019 (6)	−0.0024 (7)
C2B	0.0180 (8)	0.0159 (8)	0.0213 (8)	−0.0046 (6)	−0.0010 (7)	−0.0046 (6)
C4B	0.0189 (8)	0.0165 (8)	0.0215 (9)	−0.0043 (7)	0.0009 (7)	−0.0064 (7)
C5B	0.0194 (8)	0.0201 (8)	0.0179 (8)	−0.0036 (7)	0.0007 (7)	−0.0042 (7)
C6B	0.0206 (9)	0.0175 (8)	0.0189 (8)	−0.0036 (7)	−0.0007 (7)	−0.0067 (7)

Geometric parameters (Å, º) for (4)

Cl1—C10A	1.720 (2)	N1A—C2A	1.361 (3)
S1—C10A	1.714 (2)	N1A—C6A	1.372 (2)
S1—C7A	1.728 (2)	N3A—C2A	1.324 (3)
O1A—C11A	1.252 (2)	N3A—C4A	1.362 (2)
O2A—C11A	1.269 (2)	N2A—C2A	1.337 (3)
C11A—C7A	1.492 (3)	N4A—C4A	1.348 (3)
C7A—C8A	1.368 (3)	N5A—C6A	1.335 (3)
C8A—C9A	1.413 (3)	C4A—C5A	1.397 (3)
C9A—C10A	1.353 (3)	C5A—C6A	1.387 (3)
Cl2—C10B	1.718 (2)	N1B—C6B	1.362 (2)
S2—C10B	1.717 (2)	N1B—C2B	1.371 (2)
S2—C7B	1.7236 (19)	N3B—C2B	1.337 (2)
O1B—C11B	1.266 (2)	N3B—C4B	1.355 (2)
O2B—C11B	1.250 (2)	N2B—C2B	1.322 (3)
C11B—C7B	1.486 (2)	N4B—C4B	1.344 (2)
C7B—C8B	1.369 (3)	N5B—C6B	1.349 (3)
C8B—C9B	1.412 (3)	C4B—C5B	1.400 (3)
C9B—C10B	1.356 (3)	C5B—C6B	1.375 (3)

C10A—S1—C7A	90.95 (10)	C2A—N1A—C6A	120.59 (17)
O1A—C11A—O2A	125.25 (18)	C2A—N3A—C4A	116.78 (17)
O1A—C11A—C7A	118.03 (17)	N3A—C2A—N2A	119.10 (18)
O2A—C11A—C7A	116.72 (17)	N3A—C2A—N1A	123.42 (17)
C8A—C7A—C11A	129.13 (19)	N2A—C2A—N1A	117.47 (18)
C8A—C7A—S1	110.81 (15)	N4A—C4A—N3A	115.69 (17)
C11A—C7A—S1	120.03 (15)	N4A—C4A—C5A	121.19 (17)
C7A—C8A—C9A	113.66 (19)	N3A—C4A—C5A	123.12 (17)
C10A—C9A—C8A	111.33 (19)	C6A—C5A—C4A	117.80 (17)
C9A—C10A—S1	113.25 (16)	N5A—C6A—N1A	116.21 (17)
C9A—C10A—Cl1	126.07 (17)	N5A—C6A—C5A	125.52 (18)
S1—C10A—Cl1	120.67 (13)	N1A—C6A—C5A	118.27 (17)
C10B—S2—C7B	90.48 (10)	C6B—N1B—C2B	120.42 (16)
O2B—C11B—O1B	125.24 (17)	C2B—N3B—C4B	116.74 (16)
O2B—C11B—C7B	117.78 (17)	N2B—C2B—N3B	120.65 (17)
O1B—C11B—C7B	116.97 (17)	N2B—C2B—N1B	116.83 (17)
C8B—C7B—C11B	128.11 (18)	N3B—C2B—N1B	122.52 (17)
C8B—C7B—S2	111.38 (14)	N4B—C4B—N3B	116.96 (17)
C11B—C7B—S2	120.40 (14)	N4B—C4B—C5B	119.64 (17)
C7B—C8B—C9B	113.55 (18)	N3B—C4B—C5B	123.40 (17)
C10B—C9B—C8B	110.91 (18)	C6B—C5B—C4B	117.41 (17)
C9B—C10B—S2	113.68 (15)	N5B—C6B—N1B	117.04 (17)
C9B—C10B—Cl2	125.95 (16)	N5B—C6B—C5B	123.62 (18)
S2—C10B—Cl2	120.37 (13)	N1B—C6B—C5B	119.31 (17)

Hydrogen-bond geometry (Å, º) for (4)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O2*A*	0.91 (3)	1.83 (3)	2.736 (2)	173 (3)
N1*B*—H1*B*···O1*B*	0.90 (3)	1.80 (3)	2.687 (2)	169 (3)
N2*A*—H2*A*···N5*B*^{i}	0.85 (3)	2.36 (3)	3.181 (3)	165 (3)
N2*A*—H2*B*···N3*A*^{ii}	0.87 (3)	2.14 (3)	3.014 (2)	177.8 (14)
N2*B*—H2*C*···O2*B*	0.86 (3)	1.99 (3)	2.844 (2)	173 (3)
N2*B*—H2*D*···N3*B*^{iii}	0.87 (3)	2.14 (3)	3.008 (2)	178 (3)
N4*A*—H4*B*···O2*B*	0.90 (3)	1.90 (3)	2.784 (2)	167 (2)
N4*B*—H4*D*···O1*A*^{iv}	0.85 (4)	2.15 (4)	2.969 (2)	161 (3)
N5*B*—H5*C*···O1*B*^{v}	0.87 (3)	2.21 (3)	2.906 (2)	136 (3)
N5*A*—H5*D*···O1*A*	0.87 (3)	2.05 (3)	2.911 (2)	169 (3)
N5*B*—H5*E*···O1*A*^{v}	0.87 (3)	2.45 (3)	3.077 (2)	130 (2)
N5*B*—H5*F*···O2*A*	0.95 (3)	1.89 (3)	2.815 (2)	163 (3)

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) −*x*+1, −*y*, −*z*+2; (iii) −*x*+2, −*y*, −*z*+1; (iv) −*x*+2, −*y*+1, −*z*+1; (v) −*x*+1, −*y*+1, −*z*+1.