Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Lamotrigine, an antiepileptic drug, and its chloride and nitrate salts

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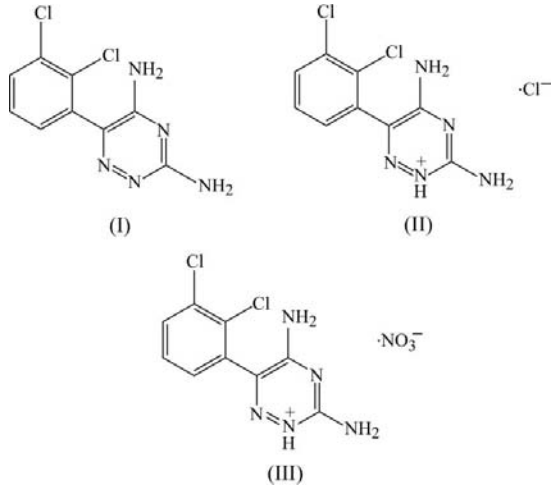
Received 22 July 2009
Accepted 4 August 2009
Online 19 August 2009

In lamotrigine [systematic name: 6-(2,3-dichlorophenyl)-1,2,4-triazine-3,5-diamine], $C_9H_7Cl_2N_5$, (I), the asymmetric unit contains one lamotrigine base molecule. In lamotriginium chloride [systematic name: 3,5-diamino-6-(2,3-dichlorophenyl)-1,2,4-triazin-2-ium chloride], $C_9H_8Cl_2N_5^+\cdot Cl^-$, (II), the asymmetric unit contains one lamotriginium cation and one chloride anion, while in lamotriginium nitrate, $C_9H_8Cl_2N_5^+\cdot NO_3^-$, (III), the asymmetric unit contains two crystallographically independent lamotriginium cations and two nitrate anions. In all three structures, N—H$\cdots$N hydrogen bonds form an $R_2^2(8)$ dimer. In (I) and (II), hydrophilic layers are sandwiched between hydrophobic layers in the crystal packing. In all three structures, hydrogen bonds lead to the formation of a supramolecular hydrogen-bonded network. The significance of this study lies in its illustration of the differences between the supramolecular aggregation in the lamotrigine base and in its chloride and nitrate salts.

Comment

Lamotrigine [6-(2,3-dichlorophenyl)-1,2,4-triazine-3,5-diamine] is an antiepileptic drug used in the treatment of epilepsy and bipolar disorder. The US Food and Drug Administration approved lamotrigine (marketed as Lamictal by Glaxo-SmithKline) for the treatment of epilepsy in 1994, and for the treatment of bipolar I disorder in 2003. Chemically unrelated to other antiepileptics, lamotrigine has relatively few side effects and does not require blood monitoring. The formation of multicomponent ionic crystals, or salts, is of fundamental importance to the development of most active pharmaceutical ingredients (APIs), where the approach is used for both purification and physical-property optimization. Salt formation is generally used to increase or decrease solubility, to improve stability or toxicity, or to reduce the hygroscopicity of APIs (Gould, 1986; Stahl & Wermuth, 2002). Chlorides, nitrates and sulfates are the most frequently occurring counter-ions of salts of drug molecules. The unit-cell dimensions [a = 6.386 (3) Å, b = 10.467 (3) Å and c = 14.856 (4) Å, β = 100.774 (3)° and V = 975.5 (6) Å^3, monoclinic space group *Aa* (Janes *et al.*, 1989)] of the lamotrigine base have been reported previously, but no coordinates were deposited in the Cambridge Structural Database [Allen, 2002; refcode EFEMUX (Janes *et al.*, 1989)]. The earlier reported unit cell appears to differ significantly from that found here. This paper reports the crystal structure of lamotrigine, (I), for the first time, and the crystal structures of the chloride, (II), and nitrate, (III), salts.



The asymmetric unit of (I) contains one lamotrigine base, while the asymmetric unit of (II) comprises one lamotriginium cation and one chloride anion and that of (III) contains two lamotriginium cations (*A* and *B*) and two nitrate anions (1 and 2). In both (II) and (III), protonation occurs at atom N2 of the triazine ring.

In all three structures, the individual rings are almost planar, with maximum deviations from the least-squares planes of the benzene rings of 0.001 (2) Å in (I), 0.019 (4) Å in

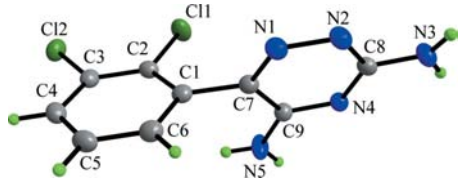


Figure 1
A view of the structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

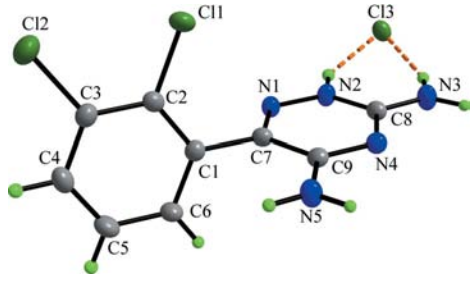


Figure 2
A view of the structure of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

  doi:10.1107/S0108270109030960

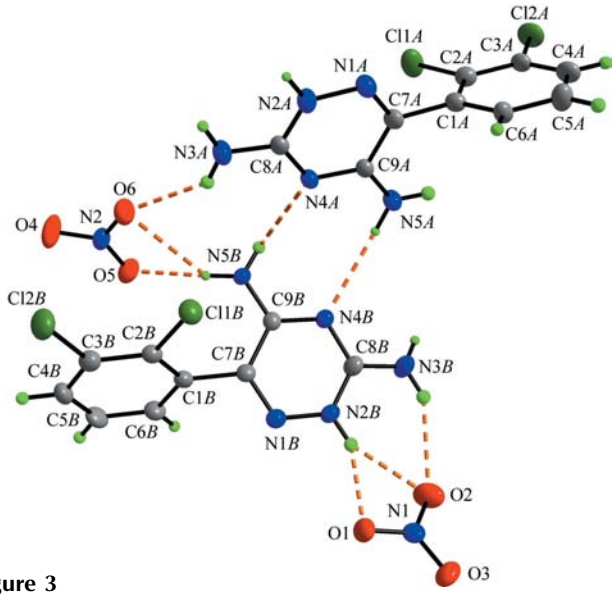


Figure 3
A view of the structure of (III), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

(II), and 0.018 (4) Å in cation *A* and 0.007 (4) Å in cation *B* of (III), and with maximum deviations from the least-squares planes of the triazine rings of 0.028 (2) Å in (I), 0.007 (3) Å in (II), and 0.036 (4) Å in cation *A* and 0.016 (4) Å in cation *B* of (III).

The dihedral angles between the rings are 69.8 (1)° in (I), 73.5 (1)° in (II), and 73.2 (1) (cation *A*) and 67.4 (1)° (cation *B*) in (III). The orientation of the benzene ring with respect to the triazine ring can be seen from the torsion angles C2—C1—C7—N1 and C2—C1—C7—C9 (Table 1). An r.m.s. overlay of the triazine rings of the three structures (Fig. 4) clearly shows the tilt of the dichlorophenyl rings. It is well known from the previously reported lamotrigine structures (Potter *et al.*, 1999; Janes & Palmer, 1995*a*,*b*) that the corresponding dihedral angles are generally observed in the range 50–80°. This relatively large value of the dihedral angle may be due to the presence of substitutents on the *ortho* positions with respect to the central C—C bond. However, in the crystal structure of 5-(4-chlorophenyl)-1,2,4-triazine (Atwood *et al.*, 1974), the twist is much smaller [dihedral angle = 9.3 (1)°] in the absence of such hindrance.

The molecular geometry of the three compounds, in terms of bond lengths and angles (Table 1), is in good agreement with the related lamotrigine structures lamotrigine isethionate (Potter *et al.*, 1999), lamotriginium benzoate dimethylformamide monosolvate (Sridhar & Ravikumar, 2005) and lamotriginium hydrogen phthalate dimethylformamide monosolvate (Sridhar & Ravikumar, 2007). The N—N and four C—N bond distances of the triazine ring are similar for all three structures and are intermediate between the expected single- (1.45 and 1.47 Å) and double-bond (1.20 and 1.27 Å) distances (Allen *et al.*, 1987).

In (I) and (II), the crystal structures are stabilized by N—H···N and N—H···Cl hydrogen bonds, and in (III) by

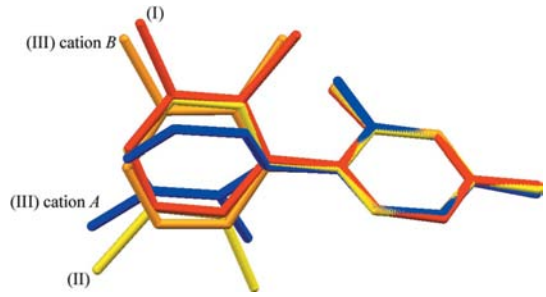


Figure 4
An r.m.s. overlay of (I), (II) (r.m.s. deviation = 0.045 Å), cation *A* of (III) (r.m.s. deviation = 0.055 Å) and cation *B* of (III) (r.m.s. deviation = 0.048 Å), showing the difference in the dichlorophenyl ring orientations by superposition of the triazine ring. For clarity, the chloride anion in (II), the two nitrate anions in (III) and all H atoms have been omitted.

N—H···N and N—H···O hydrogen bonds. Interestingly, in (I), all the N atoms of the triazine ring participate in hydrogen bonding, while in (II) and (III), all N atoms except atom N1 are involved in hydrogen bonding. In all three structures, lamotrigine–lamotrigine dimers are formed by N—H···N hydrogen bonds [$R_2^2(8)$ motif (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995)]. In (I), the dimer is formed between atom N3 and its inversion-related atom N4, while in (II) and (III) the dimer is formed between atoms N5 and N4 of the triazine rings. Interestingly, in (III), the dimer is formed between the two independent lamotriginium cations through a double intramolecular N—H···N hydrogen bond.

In (I), adjacent lamotrigine–lamotrigine dimers are interlinked by double N—H···N hydrogen bonds [$R_3^3(9)$ motif; Table 2], leading to one-dimensional supramolecular polymeric ribbons (Fig. 5). These ribbons are further interlinked by an N—H···Cl hydrogen bond involving atom N5 of the triazine ring and its glide-related atom Cl2 of the dichlorophenyl ring, leading to the formation of a dimer having an $R_2^2(16)$ motif.

The three N—H···N hydrogen bonds in (I) form alternate $R_2^2(8)$ and $R_3^3(9)$ motifs, which in turn produce an $R_4^4(14)$ motif and aggregate into a one-dimensional hydrogen-bonded polymeric ribbon running along the crystallographic *a* axis. These ribbons are further interlinked by N—H···Cl hydrogen-bonded dimers with an $R_4^4(16)$ motif, thereby leading to the formation of a supramolecular three-dimensional hydrogen-bonded network. In the crystal packing, the triazine rings are sandwiched between the dichlorophenyl rings.

The chloride ion of (II) is involved in four N—H···Cl hydrogen bonds (Table 3), forming intramolecular N—H···Cl hydrogen bonds with atoms N2 and N3, generating an $R_2^1(6)$ motif, and intermolecular N—H···Cl hydrogen bonds with atoms N3 and N5, generating two $R_3^2(8)$ motifs (Fig. 6).

Each centrosymmetric $R_2^2(8)$ dimer of (II) is flanked by two symmetry-related chloride anions at $(-x+1, y+\frac{1}{2}, -z+\frac{3}{2})$ and $(x, -y+\frac{1}{2}, z+\frac{1}{2})$, thereby generating an $R_3^2(8)$ motif. Thus, the combination of intermolecular N—H···Cl and N—H···N hydrogen bonds produces an $R_4^2(16)$ motif. The $R_2^2(8)$ and $R_3^2(8)$ motifs are arranged alternately and form hydrogen-

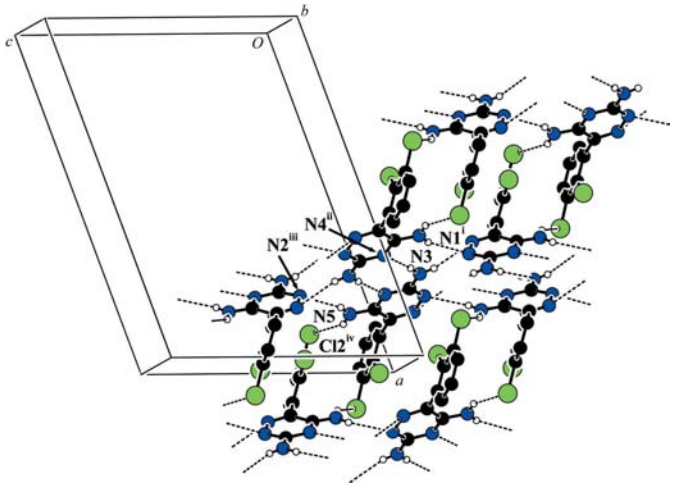


Figure 5
A packing diagram for (I), viewed approximately down the *b* axis, showing how the hydrogen-bonded triazine rings are sandwiched between the dichlorophenyl rings. Dashed lines indicate N—H···N hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted for clarity. Only atoms involved in the hydrogen bonding are labelled. [Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z-\frac{1}{2}$; (ii) $-x+\frac{3}{2}, -y+\frac{3}{2}, -z$; (iii) $x, -y+1, z+\frac{1}{2}$; (iv) $-x+2, y, -z+\frac{1}{2}$.]

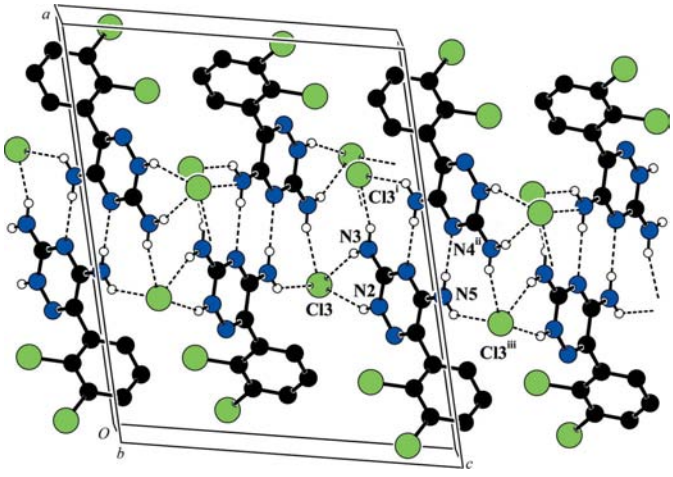


Figure 6
A packing diagram for (II), viewed approximately down the *b* axis, showing how the hydrophilic layer around $a = \frac{1}{2}$ is sandwiched between the hydrophobic layers around $a = \frac{1}{4}$ and $\frac{3}{4}$. Dashed lines indicate N—H···N and N—H···Cl hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted for clarity. Only atoms involved in the hydrogen bonding are labelled. [Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+1, -z+2$; (iii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.]

bonded columns in the crystal packing. Each hydrogen-bonded column is further interlinked to adjacent columns by double N—H···Cl hydrogen bonds, thereby generating an infinite two-dimensional supramolecular hydrogen-bonded sheet with alternate cations and anions parallel to the (100) plane (Fig. 6). Furthermore, in the crystal packing, hydrophilic layers around $a = \frac{1}{2}$ are sandwiched between hydrophobic layers about $a = \frac{1}{4}$ and $\frac{3}{4}$.

In (III), all the O atoms of the two nitrate anions participate in hydrogen-bonding networks (Table 4). Atoms N5*A*, N2*B*, N3*B* and N5*B* are involved in three-centred hydrogen-

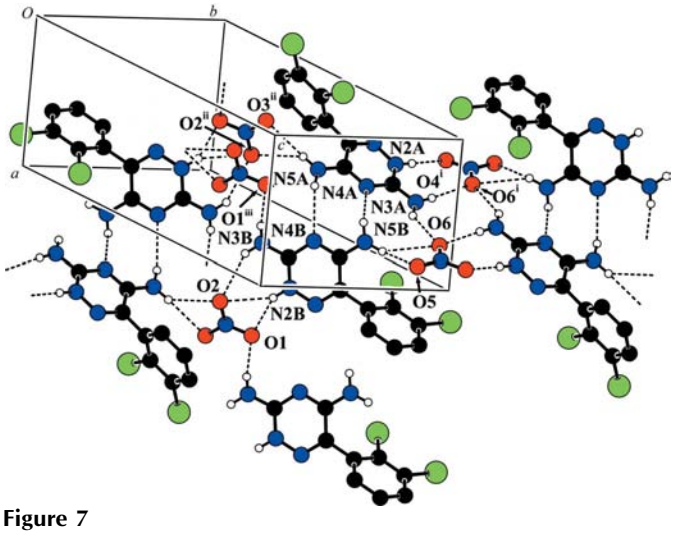


Figure 7
A packing diagram for (III), viewed down the *a* axis, showing the extended two-dimensional supramolecular hydrogen-bonded network parallel to the $(01\bar{1})$ plane. Dashed lines indicate N—H···N and N—H···O hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted for clarity. Only atoms involved in the hydrogen bonding are labelled. [Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $-x+2, -y+1, -z+1$; (iii) $x-1, y, z$.]

bonding patterns (Jeffrey & Saenger, 1991). Nitrate anion 1 is involved in six hydrogen bonds, while anion 2 is involved in five hydrogen bonds with the cation pairs. Nitrate anion 2 and its inversion-related anion act as bridging units and interconnect the lamotriginium–lamotriginium dimer pairs, which in turn generate $R_1^2(4)$, $R_4^2(8)$ and $R_2^2(8)$ motifs and form hydrogen-bonded columns. Each column is arranged in a hexameric hydrogen-bonded network consisting of two sets of alternate cation and anion pairs (Fig. 7). This hydrogen-bonded network is further interconnected by intramolecular N—H···O hydrogen bonds involving atoms N2*B* and N3*B* of the lamotriginium cation and atoms O1 and O2 of nitrate anion 1, thereby generating $R_1^2(3)$, $R_1^2(4)$ and $R_2^2(8)$ motifs. In addition, inversion-related nitrate anions link the hexameric hydrogen-bonded columns through atoms N5*A* and N3*B* of the cation pairs.

The combination of N—H···N and N—H···O hydrogen bonds in (III), involving the cation pair, two nitrate anions and their symmetry-related anions, leads to an infinite two-dimensional supramolecular hydrogen-bonded network parallel to the $(01\bar{1})$ plane. No specific hydrophilic and hydrophobic layers are observed in (III). It is very interesting to note that in (II) and (III), the two Cl atoms of the lamotriginium cations are not involved in any interactions. A short interatomic contact is observed between atom Cl2*A* of the lamotriginium cation and atom O2 of the nitrate anion [Cl2*A*···O2$(-x+1, -y+1, -z+1)$ = 2.946 (5) Å], which is a consequence of the dense packing of the components induced by the complex hydrogen bonding.

Experimental

To obtain crystals of (I) suitable for X-ray study, lamotrigine (Jubilant Organosys Ltd, Mysore, India; 98.75% purity) was dissolved in

methanol and the solution allowed to evaporate slowly. Crystals of (II) and (III) were obtained by slow evaporation from aqueous solutions of a 1:1 stoichiometric ratio, of lamotrigine and hydrochloric acid for (II), and of lamotrigine and nitric acid for (III).

Compound (I)

Crystal data

$C_9H_7Cl_2N_5$
M_r = 256.10
Monoclinic, $C2/c$
a = 19.136 (3) Å
b = 8.6409 (12) Å
c = 13.5549 (18) Å
β = 109.172 (2)°
V = 2117.0 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.59 mm^{-1}
T = 294 K
0.21 × 0.11 × 0.07 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
9654 measured reflections
1869 independent reflections
1785 reflections with $I > 2/s(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.082
S = 1.05
1869 reflections
161 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Compound (II)

Crystal data

$C_9H_8Cl_2N_5^+\cdot Cl^-$
M_r = 292.55
Monoclinic, $P2_1/c$
a = 16.664 (5) Å
b = 5.5139 (17) Å
c = 13.392 (4) Å
β = 102.588 (5)°
V = 1201.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.75 mm^{-1}
T = 294 K
0.19 × 0.12 × 0.07 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
10623 measured reflections
2119 independent reflections
1645 reflections with $I > 2/s(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.124
S = 1.07
2119 reflections
174 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å ,°) for (I), (II) and (III).

Parameter	(I)	(II)	(III), cation *A*	(III), cation *B*
N1—N2	1.350 (2)	1.352 (4)	1.357 (5)	1.338 (5)
C7—N1	1.312 (2)	1.293 (4)	1.289 (5)	1.294 (5)
N2—C8	1.340 (2)	1.342 (4)	1.340 (5)	1.345 (5)
C8—N4	1.351 (2)	1.337 (4)	1.341 (5)	1.347 (5)
N4—C9	1.325 (2)	1.326 (4)	1.333 (5)	1.325 (5)
N2—C8—N4	126.1 (2)	121.3 (3)	117.4 (4)	119.2 (4)
C2—C1—C7—N1	111.6 (2)	−72.4 (4)	−106.7 (5)	113.6 (4)
C2—C1—C7—C9	−72.0 (2)	104.9 (3)	75.2 (5)	−70.7 (5)

Table 2
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3N···N1^{i}	0.80 (2)	2.54 (2)	3.284 (2)	155 (2)
N3—H4N···N4^{ii}	0.85 (2)	2.33 (2)	3.178 (2)	177 (2)
N5—H5N···N2^{iii}	0.83 (2)	2.40 (2)	3.152 (2)	151 (2)
N5—H6N···Cl2^{iv}	0.85 (3)	2.65 (2)	3.4121 (18)	151 (2)

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

Table 3
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2N···Cl3	0.85 (4)	2.47 (4)	3.233 (3)	150 (3)
N3—H3N···Cl3	0.84 (4)	2.36 (4)	3.160 (4)	160 (3)
N3—H4N···Cl3^{i}	0.89 (4)	2.30 (4)	3.165 (4)	164 (3)
N5—H5N···N4^{ii}	0.88 (3)	2.32 (3)	3.189 (4)	173 (3)
N5—H6N···Cl3^{iii}	0.87 (4)	2.48 (4)	3.149 (3)	135 (3)

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

Compound (III)

Crystal data

$C_9H_8Cl_2N_5^+\cdot NO_3^-$
M_r = 319.11
Triclinic, $P\overline{1}$
a = 7.1356 (9) Å
b = 13.3711 (17) Å
c = 14.0294 (18) Å
α = 88.285 (2)°
β = 85.502 (2)°
γ = 75.516 (2)°
V = 1291.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 294 K
0.20 × 0.18 × 0.09 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
12130 measured reflections
4540 independent reflections
3759 reflections with $I > 2/s(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.184
S = 1.10
4540 reflections
401 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

All N-bound H atoms were located in difference Fourier maps and their positions and isotropic displacement parameters were located and refined. All other H atoms were located in a difference density map but were positioned geometrically and included as riding atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). In (III), the N3*A*—H4, N5*A*—H6, N2*B*—H7, N3*B*—H8, N5*B*—H10 and N5*B*—H11 distances were restrained to a set value of 0.89 (1) Å. The highest unassigned peak is located 0.96 Å from atom H4*A* in (III).

For all compounds, data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

Table 4
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
N2A—H2···O4^{i}	0.85 (4)	1.91 (4)	2.753 (5)	173 (4)
N3A—H3···O6^{i}	0.82 (5)	2.14 (5)	2.960 (6)	174 (4)
N3A—H4···O6	0.89 (3)	2.24 (4)	2.979 (5)	140 (5)
N5A—H5···N4B	0.95 (4)	2.48 (4)	3.340 (5)	151 (3)
N5A—H6···O3^{ii}	0.89 (4)	2.35 (3)	3.155 (5)	151 (5)
N5A—H6···O2^{ii}	0.89 (4)	2.39 (4)	3.146 (5)	143 (5)
N2B—H7···O1	0.89 (4)	2.05 (3)	2.858 (5)	151 (5)
N2B—H7···O2	0.89 (4)	2.34 (3)	3.146 (5)	151 (5)
N3B—H8···O2	0.89 (5)	2.39 (3)	3.177 (6)	149 (5)
N3B—H9···O1^{iii}	0.82 (5)	2.18 (5)	2.978 (6)	163 (4)
N5B—H10···N4A	0.88 (3)	2.55 (5)	3.076 (5)	119 (4)
N5B—H11···O5	0.89 (3)	2.08 (2)	2.909 (5)	155 (4)
N5B—H11···O6	0.89 (6)	2.56 (3)	3.290 (5)	140 (3)

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

The authors thank Dr J. S. Yadav, Director, IICT, Hyderabad, for his kind encouragement.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DN3123). Services for accessing these data are described at the back of the journal.

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