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Two polymorphs, with Z' = 1 and 2, of 2-amino-4-chloro-6-morpholinopyrimidine in $P2_1/c$, and 2-amino-4-chloro-6-piperidinopyrimidine, which is isomorphous and almost isostructural with the Z' = 2 polymorph

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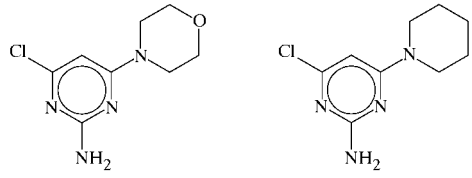
Crystallization of 2-amino-4-chloro-6-morpholinopyrimidine, $C_8H_{11}ClN_4O$, (I), yields two polymorphs, both with space group $P2_1/c$, having Z' = 1 (from diethyl ether solution) and Z' = 2 (from dichloromethane solution), denoted (I*a*) and (I*b*), respectively. In polymorph (I*a*), the molecules are linked by an N—H···O and an N—H···N hydrogen bond into sheets built from alternating $R_2^2(8)$ and $R_6^6(40)$ rings. In polymorph (I*b*), one molecule acts as a triple acceptor of hydrogen bonds and the other acts as a single acceptor; one N—H···O and three N—H···N hydrogen bonds link the molecules in a complex chain containing two types of $R_2^2(8)$ and one type of $R_4^4(18)$ ring. 2-Amino-4-chloro-6-piperidinopyrimidine, $C_9H_{13}ClN_4$, (II), which is isomorphous with polymorph (I*b*), also has Z' = 2 in $P2_1/c$, and the molecules are linked by three N—H···N hydrogen bonds into a centrosymmetric four-molecule aggregate containing three $R_2^2(8)$ rings.

Comment

Aminochloropyrimidines are key intermediates for the synthesis of O^6-benzyloxy-5-nitrosopyrimidines, which can themselves be converted into a wide range of alkoxy- and amino-substituted O^6-benzyloxy-5-nitrosopyrimidines (Quesada, Marchal, Melguizo *et al.*, 2002; Quesada, Marchal, Nogueras *et al.*, 2002), which are important as potential, or proven, *in vitro* inhibitors of the human DNA-repair protein O^6-alkylguanine–DNA–transferase (Chae *et al.*, 1995; Quesada, Marchal, Melguizo *et al.*, 2002). We report here the molecular and supramolecular structures of two examples of this class of pyrimidine, namely 2-amino-4-chloro-6-morpholinopyrimidine, (I), which crystallizes in two polymorphic forms, and 2-amino-4-chloro-6-piperidinopyrimidine, (II), which is isomorphous and almost isostructural with one of the polymorphs of (I).



The simpler polymorph of compound (I), *i.e.* (I*a*) (Fig. 1), has Z' = 1 in $P2_1/c$, and the molecules are linked into sheets by two hydrogen bonds, one each of the N—H···O and N—H···N types (Table 1). Amino atom N2 in the molecule at (x, y, z) acts as hydrogen-bond donor, *via* H1, to the morpholine atom O64 in the molecule at ($2-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$), while atom N2 at ($2-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$) likewise acts as donor to O64 at (x, $-1+y$, z), so producing a $C(9)$ chain running parallel to the [010] direction and generated by the 2_1 screw axis along (1, y, $\frac{1}{4}$) (Fig. 2). Adjacent [010] chains are linked into (102) sheets by means of N—H···N hydrogen bonding. Amine atom N2 in the molecule at (x, y, z), which lies in the [010] chain along (1, y, $\frac{1}{4}$), acts as hydrogen-bond donor, *via* H2, to atom N3 in the molecule at ($1-x$, $1-y$, $1-z$), which lies in the antiparallel chain along (0, $-y$, $\frac{3}{4}$), so forming an $R_2^2(8)$ ring centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$). In a similar way, the molecule at ($2-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$), a component of the (1, y, $\frac{1}{4}$) chain, is linked *via* the $R_2^2(8)$ motif to the molecule at ($1+x$, $\frac{1}{2}-y$, $-\frac{1}{2}+z$), which lies in the chain along (2, $-y$, $-\frac{1}{4}$). In this manner, [010] chains are linked into a (102) sheet, built from alternating $R_2^2(8)$ and $R_6^6(40)$ rings (Fig. 2). If the individual molecules are regarded as the nodes of the net defining the (102) sheet, then this net is of the (6,3)-type (Batten & Robson, 1998), while if the $R_2^2(8)$ dimers are

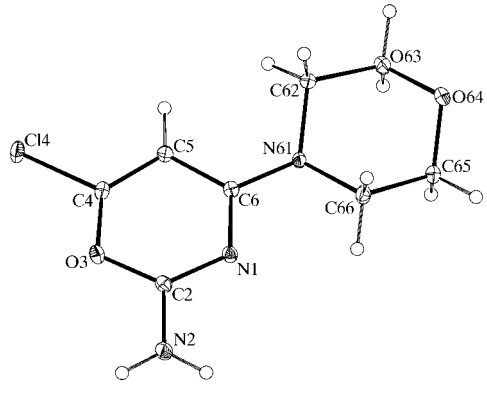


Figure 1
The molecule of compound (I) in the Z' = 1 polymorph, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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taken to be the nodes, the net is of the (4,4)-type. There are no direction-specific interactions between adjacent sheets.

In the $Z' = 2$ polymorph of compound (I), *viz*. (I*b*), the two independent molecules (Fig. 3) exhibit different hydrogen-bonding behaviour and the hydrogen bonds (Table 2) generate a rather elaborate one-dimensional structure. In both molecules, the amino group acts as a double donor, but molecule 1 (containing atom N11; Fig. 3) acts as a triple acceptor of hydrogen bonds, *via* atoms N11, N13 and O164, whereas in molecule 2, there is just a single acceptor, namely N23. These facts alone preclude any additional symmetry. Within the selected asymmetric unit (Fig. 3), the molecules are linked by paired N—H···N hydrogen bonds, forming an $R_2^2(8)$ dimer in which the orientation of the morpholine substituents precludes even approximate additional symmetry. In analysing the structure of this polymorph, it is convenient to employ the substructure approach (Gregson *et al.*, 2000), where the key substructural unit is the $R_2^2(8)$ dimer; the formation of this dimer leaves two N—H bonds, N12—H12 and N22—H21, available for the formation of further hydrogen bonds. Amino atom N12 in the type-1 molecule at (x, y, z) acts as hydrogen-bond donor, *via* H12, to atom N13 in the type-1 molecule at ($2 - x$, $1 - y$, $1 - z$), so generating a centrosymmetric $R_2^2(8)$ motif, centred at (1, $\frac{1}{2}$, $\frac{1}{2}$), while atom N22 in the type-2 molecule at (x, y, z) acts as donor, *via* H21, to atom O164 in the type-1 molecule at ($1 - x$, $-y$, $1 - z$), so generating a centrosymmetric $R_4^4(18)$ motif centred at ($\frac{1}{2}$, 0, $\frac{1}{2}$). Propagation by inversion of these hydrogen bonds thus leads to the formation of a chain of rings running parallel to the [110] direction (Fig. 4), such that the type-2 morpholine groups containing atom O264 are pendent from the chain with no plausible hydrogen-bond donors within hydrogen-bonding distance.

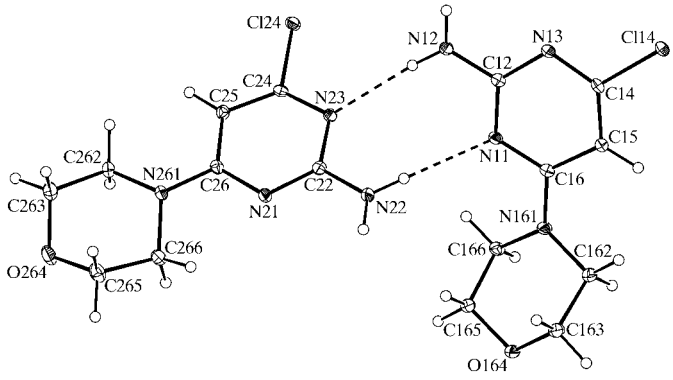

Figure 3
The two independent molecules of compound (I) in the $Z' = 2$ polymorph, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

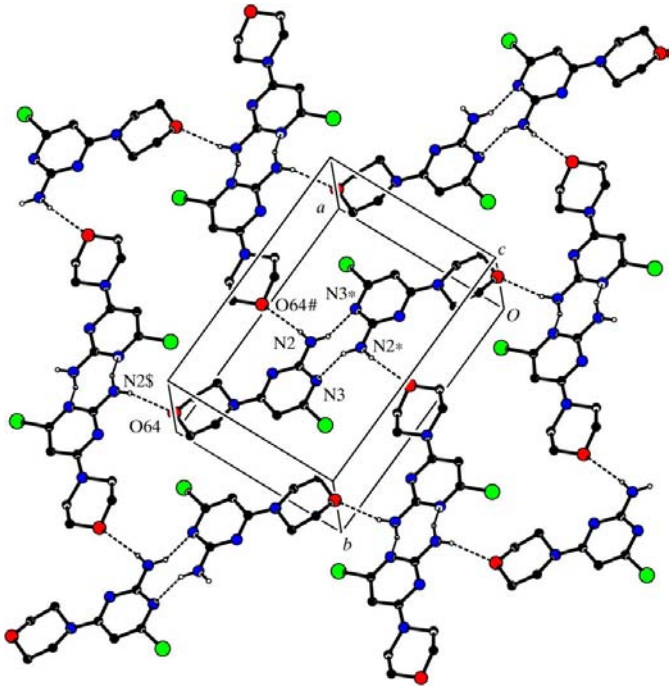

Figure 2
Part of the crystal structure of the $Z' = 1$ polymorph of (I), showing the formation of a (102) sheet built from alternating $R_2^2(8)$ and $R_6^6(40)$ rings. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*), hash (#) or dollar sign ($) are at the symmetry positions ($1 - x$, $1 - y$, $1 - z$), ($2 - x$, $-\frac{1}{2} + y$, $\frac{1}{2} - z$) and ($2 - x$, $\frac{1}{2} + y$, $\frac{1}{2} - z$), respectively.

Compound (II) has so far been isolated in only one crystalline form and this is isomorphous with the $Z' = 2$ polymorph of compound (I); the cell dimensions of these two $P2_1/c$, $Z' = 2$ phases are very similar and it is possible to refine either structure starting from the coordinates of the other, paying due regard to the difference in atom types between the morpholine substituent in (I) and the piperidine substituent in (II). Since, however, there can be no N—H···O hydrogen bonds in (II) (Table 3), the supramolecular aggregation in (II) must be different from that in either polymorph of (I); such hydrogen bonds are present in both polymorphs of (I) and hence the two $Z' = 2$ phases are not, strictly speaking, properly isostructural, although they are nearly so. The asymmetric unit selected for compound (II) resembles that for the $Z' = 2$

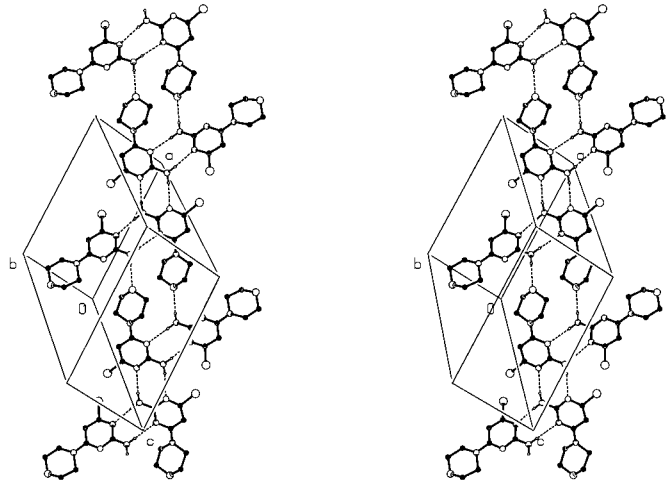

Figure 4
Stereoview of part of the crystal structure of the $Z' = 2$ polymorph of (I), showing the formation of a chain of edge-fused $R_2^2(8)$ and $R_4^4(18)$ rings along [110]. For the sake of clarity, H atoms bonded to C atoms have been omitted.

polymorph of (I) in that it forms an $R_2^2(8)$ dimer which itself exhibits no symmetry (Fig. 5). Pairs of these dimers are linked into a centrosymmetric four-molecule aggregate centred at $(1, \frac{1}{2}, \frac{1}{2})$; atom N12 at (x, y, z) acts as hydrogen-bond donor, *via* H12, to atom N13 at $(2 - x, 1 - y, 1 - z)$, but the N22—H21 bond does not participate in the hydrogen bonding, so that this aggregate is strictly finite (Fig. 6).

It is notable that even in (II), where there is an exact match between the number of hard hydrogen-bond donors and the number of hard hydrogen-bond acceptors, not all of these donors and acceptors are involved in the supramolecular aggregation. Thus, atom N21 does not accept any hydrogen bond, nor is N22—H21 involved. In both (I*a*) and (I*b*), there is an excess of acceptors over donors, and in (I*a*), atom N1 is unused as an acceptor, while in (I*b*) both N21 and O264 are unused. The structures of (I*a*), (I*b*) and (II) do not contain any soft hydrogen bonds of the C—H$\cdots A$ (A = N or O) or D—H$\cdots\pi$ types (D = C or N), nor do they contain any π–π-stacking interactions; they thus have hydrogen-bonded supramolecular structures which are two-dimensional, one-dimensional and (finite) zero-dimensional, respectively.

In each of (I*b*) and (II), there are rather short contacts between pairs of Cl atoms across the inversion centre at $(1, \frac{1}{2}, \frac{1}{2})$ (Figs. 4 and 6); in (I*b*), the Cl14$\cdots$Cl24^{i} distance is 3.375 (2) Å [symmetry code: (i) $2 - x$, $1 - y$, $1 - z$], associated with a C—Cl$\cdots$Cl angle of 145.14 (5)°, while in (II), the corresponding values are 3.425 (2) Å and 150.08 (5)°. For Cl bonded to neutral arene groups, Bondi (1964) recommended a van der Waals radius of 1.77 Å; the Cl$\cdots$Cl contact distances in (I*b*) and (II) are thus significantly shorter than twice the van der Waals radius. Database analysis of such halogen–halogen contacts has shown that when the $X\cdots X$ contact distance (X = halogen) is significantly less than the van der Waals sum, the associated C—$X\cdots X$ angles are clustered around 180 and 90° (Ramasabbu *et al.*, 1986); the C—Cl$\cdots$Cl angles observed are not particularly close to either cluster, and almost certainly their values are constrained by the formation of the centrosymmetric hydrogen-bonded $R_2^2(8)$ rings.

Within the molecules, the morpholine and piperidine substituents adopt the usual chair conformations, but with the N atoms in these rings almost planar; the sum of the angles at the N atom ranges from 352.3 (2)° in (I*a*) to 359.9 (2)° in molecule 1 of (I*b*). Consistent with this planarity, the associated C—N distances for the bond linking the two rings within each molecule range from 1.352 (2) to 1.372 (2) Å, with a mean of 1.361 (2) Å, typical of Ar—N(C)$_2$ distances involving di-*N*-substituted anilines with a planar N atom (mean value 1.371 Å; Allen *et al.*, 1987). By way of comparison, the C—NH$_2$ distances here range from 1.338 (2) to 1.358 (2) Å, with a mean value of 1.348 (2) Å, again typical of Ar—NH$_2$ distances in simple anilines with an unsubstituted planar N atom (mean value 1.355 Å; Allen *et al.*, 1987). The other intramolecular distances and angles show no unusual features; in particular, there is no evidence for any bond fixation within the pyrimidine rings.

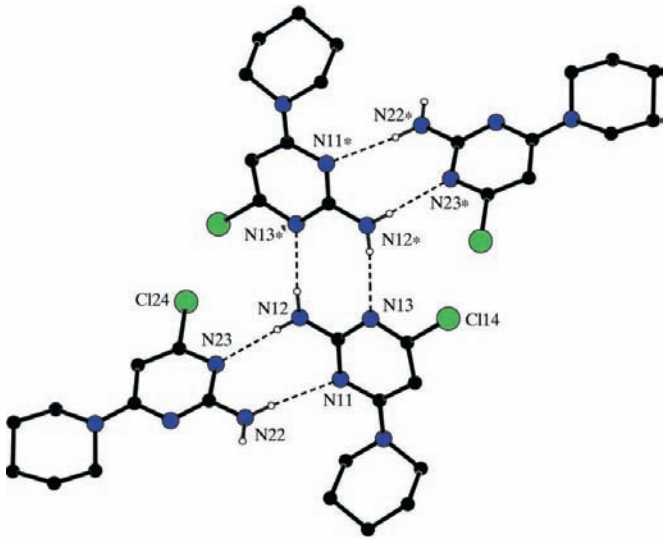

Figure 6
Part of the crystal structure of (II), showing the formation of a four-molecule aggregate containing three $R_2^2(8)$ rings. For the sake of clarity, H atoms bonded to C atoms and the unit-cell box have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(2 - x, 1 - y, 1 - z)$.

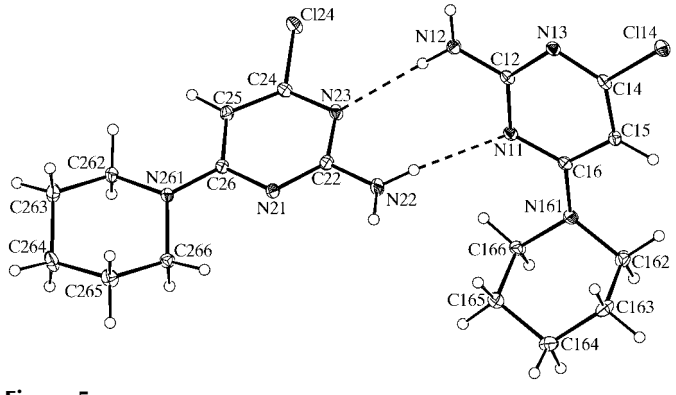

Figure 5
The two independent molecules of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

The very different cell dimensions of polymorphs (I*a*) and (I*b*) effectively rule out any possibility of a simple displacive phase transition between these two polymorphs. While we have not investigated the relative stability of these two polymorphs, we note that (I*a*) has a significantly higher density than (I*b*) and thus is probably the thermodynamically more stable form (Burger & Ramberger, 1979). Although polymorph (I*b*) was the first to be isolated, both forms can readily be reproduced by crystallization from the appropriate solvent; hence there is no question here of any disappearing polymorph (Dunitz & Bernstein, 1995).

Experimental

Samples of (I) and (II) were synthesized from 2-amino-4,6-dichloropyrimidine (purchased from Aldrich). Morpholine [for (I)] or piperidine [for (II)] (3.1 mmol) and triethylamine (6.5 mmol) were added to a stirred solution of 2-amino-4,6-dichloropyrimidine (3.0 mmol) in 3-methylbutan-2-ol (20 ml). The mixtures were heated

under reflux, with stirring, for 2 h. After cooling, the solvent was removed, excess distilled water was added, and the resulting solid was filtered off and washed with water. After drying, the solid products were recrystallized first from ethyl acetate and then from dichloromethane; m.p. 485 K [for (I)] and 431 K [for (II)]. NMR for (I), ^{1}H ($CHCl_3$, p.p.m.): δ 3.55 (4H, *t*, *J* = 5.2 Hz, N–CH_2), 3.74 (4H, *t*, *J* = 5.2 Hz, O–CH_2), 4.90 (2H, *br*, NH_2, exchanges with D_2O), 5.94 (1H, *s*, C5–H); ^{13}C ($CDCl_3$, p.p.m.): δ 45.3, 66.6, 82.4, 160.2, 162.3, 163.3; NMR for (II), ^{1}H ($CHCl_3$, p.p.m.): δ 1.58–1.68 (6H, *m*, 3 × CH_2), 3.54 (4H, *t*, *J* = 45.0 Hz, N–CH_2), 4.76 (2H, *br*, NH_2, exchanges with D_2O), 5.96 (1H, *s*, C5–H); ^{13}C ($CDCl_3$, p.p.m.): δ 24.6, 25.5, 45.3, 82.4, 160.2, 162.2, 163.3. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of solutions in diethyl ether [for (I*a*)] or dichloromethane [for (I*b*) and (II)].

Polymorph (I*a*)

Crystal data

$C_8H_{11}ClN_4O$
M_r = 214.66
Monoclinic, $P2_1/c$
a = 9.4143 (4) Å
b = 13.0869 (4) Å
c = 7.5774 (2) Å
β = 96.4260 (13)°
V = 927.70 (5) Å^3
Z = 4
D_x = 1.537 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 2102 reflections
θ = 3.1–27.5°
μ = 0.38 mm^{-1}
T = 120 (2) K
Block, colourless
0.42 × 0.22 × 0.16 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.856, T_{max} = 0.941
6031 measured reflections
2102 independent reflections
1705 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 27.5°
h = −12 → 12
k = −16 → 12
l = −8 → 9

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.092
S = 1.05
2102 reflections
127 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 0.4718P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °) for (I*a*).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O64^{i}	0.88	2.44	3.288 (2)	163
N2—H2···N3^{ii}	0.88	2.36	3.119 (2)	145

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

Polymorph (I*b*)

Crystal data

$C_8H_{11}ClN_4O$
M_r = 214.66
Monoclinic, $P2_1/c$
a = 13.9215 (3) Å
b = 8.1803 (2) Å
c = 19.6390 (4) Å
β = 121.6130 (12)°
V = 1904.65 (8) Å^3
Z = 8
D_x = 1.497 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 4346 reflections
θ = 3.0–27.5°
μ = 0.37 mm^{-1}
T = 150 (1) K
Plate, colourless
0.30 × 0.25 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.961, T_{max} = 0.995
17 326 measured reflections
4346 independent reflections
3612 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 27.5°
h = −18 → 16
k = −10 → 10
l = −25 → 25

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.04
4346 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.6560P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Table 2
Hydrogen-bonding geometry (Å, °) for (I*b*).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N12—H11···N23	0.88	2.19	3.061 (2)	174
N22—H22···N11	0.88	2.35	3.211 (2)	165
N12—H12···N13^{i}	0.88	2.18	3.036 (2)	165
N22—H21···O164^{ii}	0.88	2.23	3.051 (2)	155

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

Compound (II)

Crystal data

$C_9H_{13}ClN_4$
M_r = 212.68
Monoclinic, $P2_1/c$
a = 13.8139 (2) Å
b = 8.48040 (10) Å
c = 19.8379 (3) Å
β = 120.6770 (9)°
V = 1998.74 (5) Å^3
Z = 8
D_x = 1.414 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 4533 reflections
θ = 3.0–27.5°
μ = 0.35 mm^{-1}
T = 120 (1) K
Block, colourless
0.45 × 0.35 × 0.25 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.826, T_{max} = 0.911
15 693 measured reflections
4533 independent reflections
3726 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.5°
h = −17 → 16
k = −10 → 11
l = −25 → 25

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.087
S = 1.04
4533 reflections
250 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.5327P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N12—H11···N23	0.88	2.19	3.0479 (16)	163
N22—H22···N11	0.88	2.40	3.2601 (17)	167
N12—H12···N13^{i}	0.88	2.12	2.9862 (16)	169

Symmetry code: (i) $2-x, 1-y, 1-z$.

Polymorphs (I*a*) and (I*b*) and compound (II) all crystallize in space group $P2_1/c$, the space group being uniquely assigned from the systematic absences in each case. All H atoms were treated as riding atoms, with C—H distances of 0.95 (heteroaromatic) or 0.99 Å (CH_2), and N—H distances of 0.88 Å.

For polymorphs (I*a*) and (I*b*) and compound (II), data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1595). Services for accessing these data are described at the back of the journal.

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