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Four products from the cyanoacetylation of pyrimidines: hydrogen-bonded dimers, π-stacked hydrogen-bonded chains and hydrogen-bonded chains of edge-fused rings

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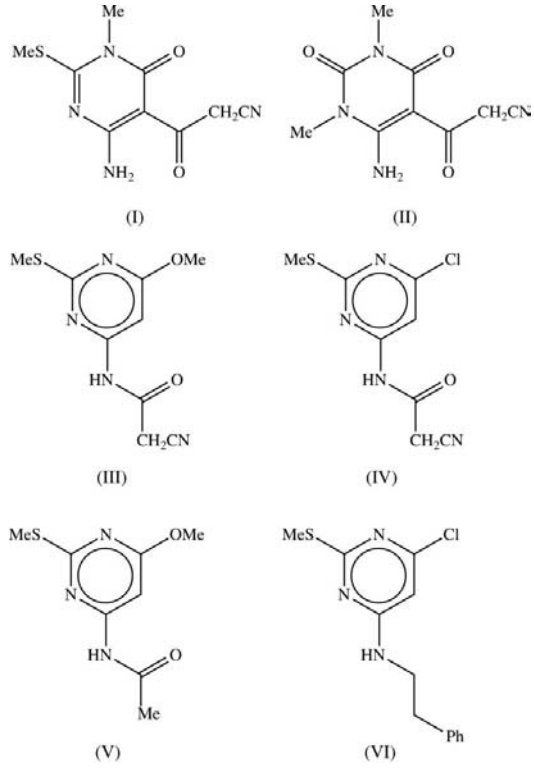
The molecules of 2-[6-amino-3-methyl-2-(methylsulfanyl)-4-oxo-3,4-dihydropyrimidin-5-ylcarbonyl]acetonitrile, $C_9H_{10}N_4O_2S$, (I), are linked in pairs by N—H···O hydrogen bonds to form cyclic centrosymmetric $R_2^2(4)$ dimers. Similar dimers formed by 2-(6-amino-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylcarbonyl)acetonitrile, $C_9H_{10}N_4O_3$, (II), are reinforced by paired N—H···N hydrogen bonds and linked into chains of rings by C—H···O hydrogen bonds. The molecules of 2-cyano-*N*-[6-methoxy-2-(methylsulfanyl)-pyrimidin-4-yl]acetamide, $C_9H_{10}N_4O_2S$, (III), are linked into simple *C*(6) chains by an N—H···N hydrogen bond, and the chains are weakly linked into sheets by a π–π stacking interaction. A combination of one two-centre N—H···N hydrogen bond and one three-centre C—H···(N,O) hydrogen bond links the molecules of 2-cyano-*N*-[6-chloro-2-(methylsulfanyl)pyrimidin-4-yl]acetamide, $C_8H_7ClN_4OS$, (IV), into a chain of alternating edge-fused $R_2^1(6)$ and $R_1^2(6)$ rings. The crystal structures reported in this study, and those of some related examples from the recent literature, show a wide variation in hydrogen-bonded aggregation consequent upon rather small changes in molecular constitution.

Comment

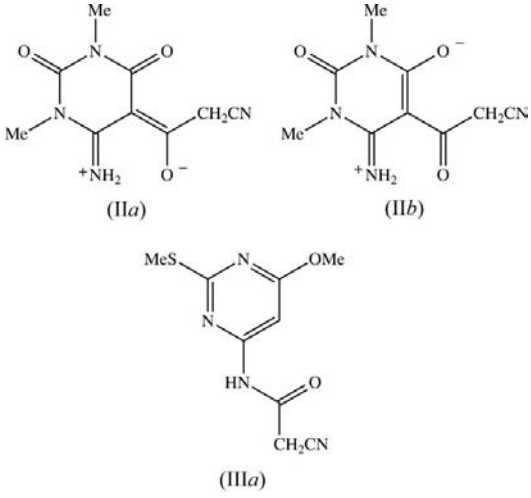
As part of a wider programme on the development of synthetic routes to new heterocyclic systems likely to exhibit biological activity, we have attempted to develop a route to the functionalization of the C5 position of pyrimidines. Accordingly, we have investigated the reactions of pyrimidines and their simple derivatives with cyanoacetic acid in the presence of acetic anhydride as an activator. When pyrimidin-4(3*H*)-ones are used as substrates, substitution does indeed occur at C5 to provide derivatives such as title compounds (I) and (II) (Figs. 1 and 2). However, when the substrates are aromatic pyrimidines, no ring substitution occurs. Instead, acylation occurs at the exocyclic amino group, giving products such as title compounds (III) and (IV) (Figs. 3 and 4). We report here the molecular and supramolecular structures of compounds (I)–(IV), which exhibit a range of different hydrogen-bonded structures, and we compare the structure of (III) with that of the closely related analogue, (V) (Low *et al.*, 1996), and with that of (VI) (Lynch & McClenaghan, 2001).



In each of compounds (I) and (II), the exocyclic carbonyl group is nearly coplanar with the ring, with a deviation of carbonyl atom O51 from the mean plane of the ring of only 0.080 (2) Å in (I) and 0.018 (2) Å in (II); this is probably associated both with the polarization of the molecular–electronic structure, discussed below, and with an intramolecular N—H···O hydrogen bond (Table 2), which is charge-assisted (Gilli *et al.*, 1994) as a consequence of the polarization. In each of (III) [which is isomeric with (I)], (IV) and (V), the conformation of the side chain at C6 may in part be controlled by a rather short intramolecular C—H···O contact. Here, the deviation of carbonyl atom O61 from the ring plane, *viz.* 0.038 (2) Å in (III) and 0.427 (3) Å in (IV), may be contrasted with the exact planarity in (V), where all of the non-H atoms lie on a mirror plane in the *Pnma* space group (Low *et al.*, 1996). The conformation adopted by the methylsulfanyl group in (IV) differs markedly from the conformations in (I) and (III) (Figs. 1, 2 and 4, and Table 1), although it is similar to that in (V).

The bond distances in compounds (I)–(III) (Table 1) show some unexpected features. We consider firstly the pair (I) and

(II). In each of compounds (I) and (II), the exocyclic C6—N6 bond is short for its type (mean value 1.353 Å; Allen *et al.*, 1987) and certainly much shorter than the corresponding bonds in compounds (III) and (IV). In addition, the C5—C6 bond, which is formally a double bond, is long for its type (expected value 1.331 Å; Allen *et al.*, 1987) and is only slightly shorter than the C4—C5 and C5—C51 bonds, which are both formally single bonds. Finally, the C51—O51 bond is long for its type (mean value 1.210 Å; Allen *et al.*, 1987), although the length of the C4—O4 bond is typical of its type. Overall, these effects are somewhat more marked in (II) than in (I), and they indicate that polarized forms such as (II*a*) and (II*b*) (see scheme below) are contributors to the overall molecular–electronic structure.



In compound (III), there is some evidence for a measure of bond fixation within the pyrimidine ring. Thus, the N1—C2 and N3—C4 bonds are slightly shorter than the C2—N3 and C6—N1 bonds, by *ca* 0.02 and 0.03 Å, respectively, while C5—C6 is slightly shorter (*ca* 0.02 Å) than C4—C5, indicative of a modest contribution to the overall structure of the form (III*a*) in addition to the dominant delocalized form (III). For compound (IV), this pattern in the C—N bonds is not apparent, while the C4—C5 and C5—C6 bond distances are not distinguishable within experimental uncertainty. The lower precision of the determination precludes any mean-

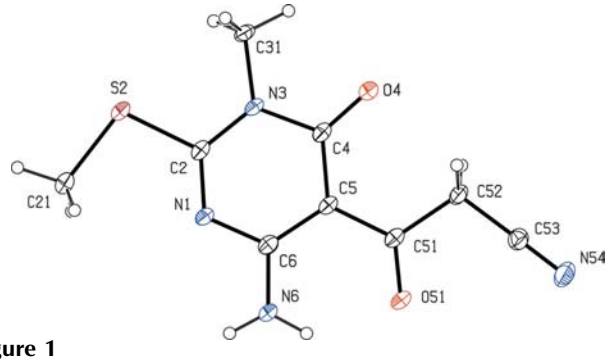


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

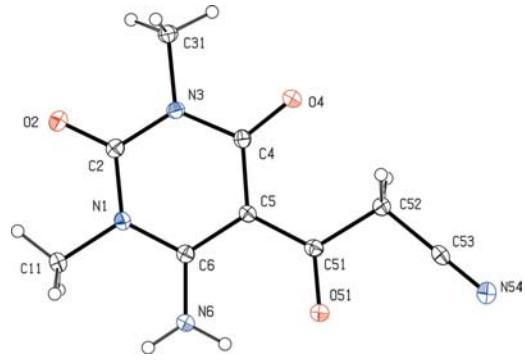


Figure 2
The molecular structure of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

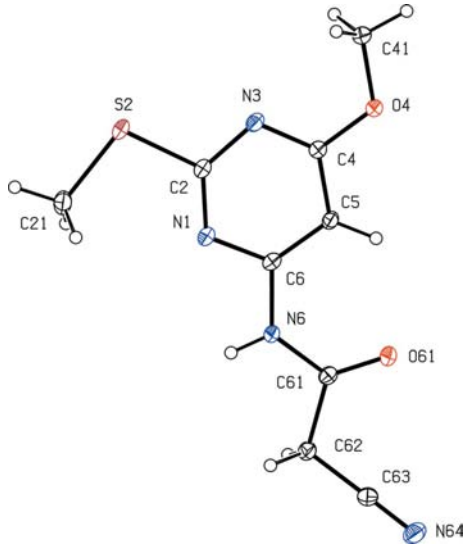


Figure 3
The molecular structure of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

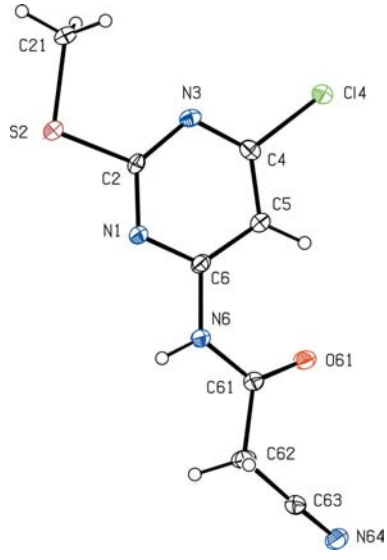


Figure 4
The molecular structure of compound (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

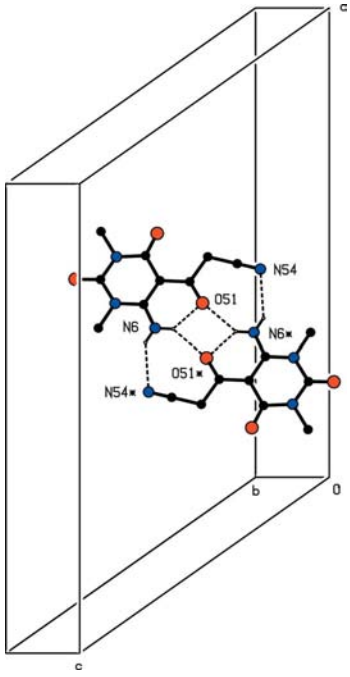


Figure 5
Part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded dimer. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, -y, 1 - z)$.

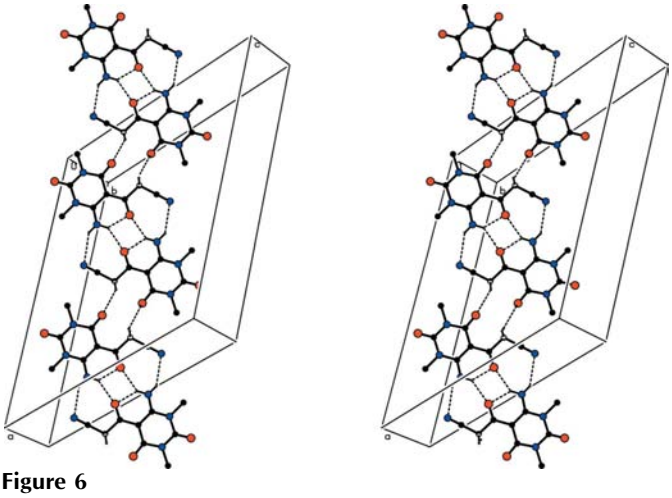


Figure 6
A stereoview of part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded chain of rings along [110]. For the sake of clarity, H atoms not involved in the hydrogen bonding have been omitted.

ingful metrical comparisons at this level. In compound (V), which was determined from diffraction data collected at 294 K (Low *et al.*, 1996), the distances provide no evidence for any bond fixation in the ring, while in (VI), which crystallizes with $Z' = 2$ (Lynch & McClenaghan, 2001), no obvious pattern can be discerned in the ring bond distances.

The hydrogen-bonded supramolecular structures of (I) and (II) show both similarities and differences. In each compound, a planar three-centre N—H···$(O)_2$ hydrogen bond links the molecules in pairs to form a centrosymmetric dimer characterized by an $R_2^2(4)$ (Bernstein *et al.*, 1995) motif (Fig. 5).

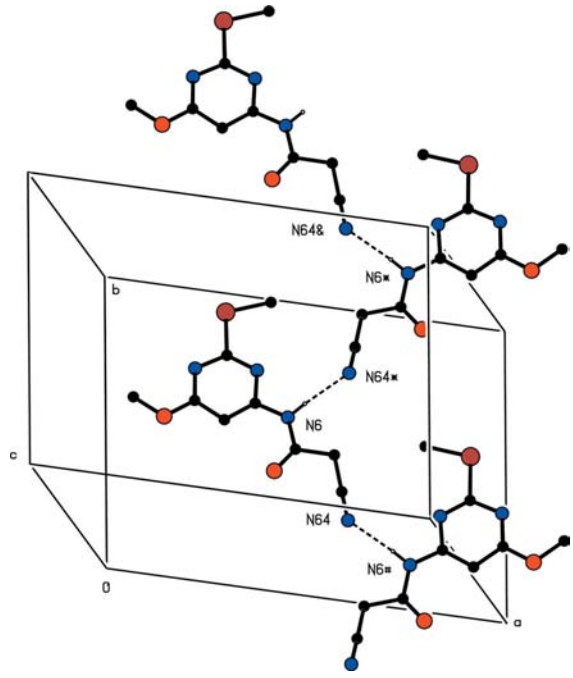


Figure 7
Part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded $C(6)$ chain along [010]. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*), a hash (#) or an ampersand (&) are at the symmetry positions $(\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$, $(\frac{3}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z)$ and $(x, 1 + y, z)$, respectively.

Within this dimer, there is a pair of fairly long, possibly adventitious, N—H···N hydrogen bonds involving two symmetry-related cyano N54 atoms as acceptors. The cyclic dimers formed by (II) are linked into chains of rings by a single C—H···O hydrogen bond (eighth entry in Table 2); atom C52 in the molecule at (x, y, z) acts as donor, *via* atom H52*B*, to carbonyl atom O4 in the molecule at $(\frac{3}{2} - x, \frac{1}{2} - y, 1 - z)$, so forming a centrosymmetric $R_2^2(12)$ motif. Propagation by inversion of the hydrogen bonds links the molecules of (II) into a rather complex chain of edge-fused rings running parallel to the [110] direction (Fig. 6).

The same $R_2^2(4)$ motif as found in (II) is also seen in (I), but the N—H···N and C—H···O contacts in (I) corresponding to the other intermolecular hydrogen bonds in (II) have H···*A* distances much longer than those in (II) (Table 2), such that they are not structurally significant (first and fourth entries in Table 2). Thus, the hydrogen bonding in (I) leads to finite dimeric units, while that in (II) generates a chain of rings.

In compound (III), a single N—H···N hydrogen bond links the molecules related by a 2_1 screw axis into $C(6)$ chains running parallel to the [010] direction (Fig. 7), and the hydrogen-bonded chains are weakly linked into sheets by a π–π stacking interaction (not shown in Fig. 7). The molecules at (x, y, z) and $(1 - x, y, \frac{3}{2} - z)$ form parts of the hydrogen-bonded chains around the 2_1 screw axes along $(\frac{3}{4}, y, \frac{3}{4})$ and $(\frac{1}{4}, y, \frac{3}{4})$, respectively. The pyrimidine rings of these molecules make a dihedral angle of 5.9 (2)°; the ring centroid separation is 3.819 (2) Å and the interplanar spacing is *ca* 3.39 Å. The resulting π–π stacking interaction thus links two molecules,

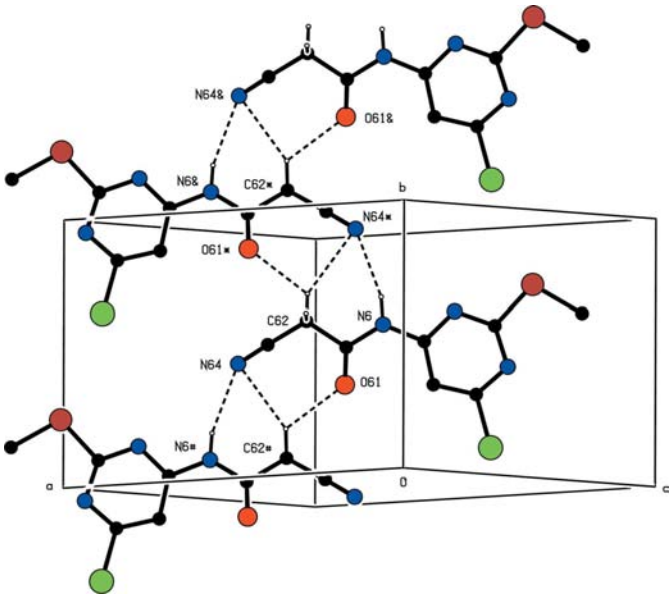


Figure 8
Part of the crystal structure of compound (IV), showing the formation of a hydrogen-bonded chain of alternating $R_2^1(6)$ and $R_1^2(6)$ rings along [010]. For the sake of clarity, H atoms bonded to atoms C5 and C21 have been omitted. Atoms marked with an asterisk (*), a hash (#) or an ampersand (&) are at the symmetry positions $(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$, $(1-x, -\frac{1}{2}+y, \frac{1}{2}-z)$ and $(x, 1+y, z)$, respectively.

related by the twofold rotation axis along $(\frac{1}{2}, y, \frac{3}{4})$, and propagation of this interaction by the twofold rotation axes links the hydrogen-bonded chain along $(\frac{3}{4}, y, \frac{3}{4})$ (Fig. 7) to those along $(\frac{1}{4}, y, \frac{3}{4})$ and $(\frac{5}{4}, y, \frac{3}{4})$, thereby generating a sheet of π-stacked hydrogen-bonded chains parallel to (001).

As in compound (III), a single N—H···N hydrogen bond in (IV) links molecules related by a 2_1 screw axis into $C(6)$ chains running parallel to the [010] direction. However, this chain is reinforced by two further interactions which together form a planar three-centre C—H···(N,O) hydrogen bond, and the combined effect of all three interactions is the generation of a chain of edge-fused rings in which $R_2^1(6)$ and $R_1^2(6)$ rings alternate (Fig. 8 and Table 2). There are no direction-specific interactions between adjacent chains.

The molecules in compound (V) (Low *et al.*, 1996) are linked by N—H···O hydrogen bonds into $C(4)$ chains, and it is interesting to note the contrast between the hydrogen bonding in (III) and (V). Although both compounds contain a carbonyl O atom, this is utilized as a hydrogen-bond acceptor only in (V), where the cyano group is absent. In both (III) and (IV), where a cyano group is present, N—H···O hydrogen bonds are absent. Compound (VI) contains neither carbonyl nor cyano groups and each type of molecule is linked into a $C(6)$ chain by N—H···N hydrogen bonds, both utilizing a ring N atom as acceptor (Lynch & McClenaghan, 2001).

Experimental

The appropriate pyrimidine, *viz*. 6-amino-3-methyl-2-(methylsulfanyl)pyrimidin-4(3*H*)-one for (I), 6-amino-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione for (II), 6-amino-4-methoxy-2-(methylsulfanyl)-pyrimidine for (III) and 6-amino-4-chloro-2-(methylsulfanyl)pyrimidine for (IV) (50 mmol), was added to a solution of cyanoacetic acid (50 mmol) in acetic anhydride (50 ml) at 323 K. The mixtures were heated to 358 K for 5 min, whereupon the products started to crystallize. After a further 5 min, the mixtures were allowed to cool to ambient temperature, and the resulting solid products were collected by filtration, washed with methanol and dried in air. Recrystallization from dimethylformamide for (I) and (II) or from ethanol for (III) and (IV) gave crystals suitable for single-crystal X-ray diffraction. Analysis for (I): colourless crystals, yield 60%, m.p. 519–520 K (HRMS: *m*/*z* found 238.0524; $C_9H_{10}N_4O_2S$ requires 238.0523); (II): yellow crystals, yield 87%, m.p. 435–436 K (HRMS: *m*/*z* found 222.0742; $C_9H_{10}N_4O_3$ requires 222.0753); (III): yellow crystals, yield 60%, m.p. 429–430 K (HRMS: *m*/*z* found 238.0524; $C_9H_{10}N_4O_2S$ requires 238.0533); (IV): yellow crystals, yield 70%, m.p. 529–530 K (HRMS: *m*/*z* found 242.0029; $C_8H_7{}^{35}ClN_4OS$ requires 242.0028).

Compound (I)

Crystal data

$C_9H_{10}N_4O_2S$
M_r = 238.27
Triclinic, $P\overline{1}$
a = 6.6449 (3) Å
b = 8.7262 (3) Å
c = 9.4092 (2) Å
α = 76.390 (3)°
β = 81.079 (2)°
γ = 82.654 (4)°
V = 521.48 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 120 (2) K
0.56 × 0.33 × 0.32 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.849, T_{max} = 0.910
14244 measured reflections
2386 independent reflections
1902 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.107
S = 1.09
2386 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Compound (II)

Crystal data

$C_9H_{10}N_4O_3$
M_r = 222.21
Monoclinic, $C2/c$
a = 22.488 (3) Å
b = 5.0709 (5) Å
c = 18.6139 (17) Å
β = 116.956 (7)°
V = 1892.0 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 120 (2) K
0.44 × 0.31 × 0.08 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.952, T_{max} = 0.990
20693 measured reflections
2171 independent reflections
1603 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.136
S = 1.13
2171 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for compounds (I)–(IV).

	(I)	(II)	(III)	(IV)
N1—C2	1.308 (2)	1.397 (2)	1.320 (2)	1.339 (4)
C2—N3	1.357 (2)	1.372 (2)	1.343 (2)	1.349 (4)
N3—C4	1.426 (2)	1.400 (2)	1.324 (2)	1.318 (4)
C4—C5	1.439 (2)	1.438 (2)	1.397 (2)	1.379 (5)
C5—C6	1.414 (2)	1.421 (2)	1.375 (2)	1.390 (5)
C6—N1	1.372 (2)	1.368 (2)	1.356 (2)	1.339 (4)
C4—O4	1.227 (2)	1.231 (2)	1.344 (2)	
C5—C51	1.454 (2)	1.434 (2)		
C51—O51	1.234 (2)	1.243 (2)		
C6—N6	1.333 (2)	1.320 (2)	1.392 (2)	1.398 (4)
N1—C2—S2—C21	0.20 (16)		1.00 (15)	−176.6 (3)
C6—N6—C61—O61			2.2 (3)	−8.3 (6)

Table 2
Hydrogen bonds and short inter- and intramolecular contacts (Å, °) for compounds (I)–(IV).

Compound	D—H···A	D—H	H···A	D···A	D—H···A
(I)	N6—H6A···N54^{i}	0.86	2.69	3.354 (2)	135
	N6—H6B···O51	0.86	1.97	2.627 (2)	132
	N6—H6B···O51^{i}	0.86	2.29	3.019 (2)	142
	C52—H52B···O4^{ii}	0.99	2.78	3.068 (3)	97
(II)	N6—H6A···N54^{iii}	0.86	2.47	3.206 (3)	144
	N6—H6B···O51	0.86	1.92	2.586 (2)	134
	N6—H6B···O51^{iii}	0.86	2.17	2.894 (2)	141
	C52—H52B···O4^{iv}	0.99	2.40	3.227 (3)	141
(III)	N6—H6···N64^{v}	0.86	2.13	2.993 (3)	176
	C5—H5···O61	0.95	2.30	2.872 (2)	118
(IV)	N6—H6···N64^{vi}	0.86	2.34	3.149 (4)	158
	C5—H5···O61	0.95	2.25	2.816 (5)	117
	C62—H62B···O61^{vi}	0.99	2.33	2.949 (5)	120
	C62—H62B···N64^{vi}	0.99	2.55	3.334 (5)	136

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

Compound (III)

Crystal data

$C_9H_{10}N_4O_2S$
M_r = 238.27
Monoclinic, $C2/c$
a = 17.106 (3) Å
b = 11.8218 (13) Å
c = 13.7187 (19) Å
β = 127.836 (8)°
V = 2191.0 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 120 (2) K
0.78 × 0.52 × 0.33 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.807, T_{max} = 0.911
24464 measured reflections
2505 independent reflections
2078 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
S = 1.09
2505 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Compound (IV)

Crystal data

$C_8H_7ClN_4OS$
M_r = 242.69
Monoclinic, $P2_1/c$
a = 12.8655 (7) Å
b = 8.4390 (2) Å
c = 9.9327 (15) Å
β = 108.827 (13)°
V = 1020.72 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.56 mm^{-1}
T = 120 (2) K
0.35 × 0.27 × 0.17 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.843, T_{max} = 0.910
22495 measured reflections
2336 independent reflections
1676 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.175
S = 1.08
2336 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

Crystals of (I) are triclinic. Space group $P\overline{1}$ was selected and confirmed by the structure analysis. For each of (II) and (III), the systematic absences permitted $C2/c$ and Cc as possible space groups. In each case, $C2/c$ was selected and confirmed by the structure analysis. For (IV), space group $P2_1/c$ was uniquely assigned from the systematic absences. For three low-angle reflections, *viz*. $\overline{1}12$ in (I), $\overline{2}04$ in (III) and 202 in (IV), satisfactory integration could not be achieved, and they were therefore discarded from the data sets. All H atoms were located in difference maps and then treated as riding atoms in geometrically idealized positions, with distances C—H = 0.95 (aromatic and heteroaromatic), 0.98 (CH_3) or 0.99 Å (CH_2), and N—H = 0.86 Å, and with U_{iso}(H) = kU_{eq}(carrier), where k = 1.5 for the methyl groups and 1.2 for all other H atoms.

For all compounds, data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2000); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GA3078). Services for accessing these data are described at the back of the journal.

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