5-(1-Hydroxyethylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione and four amino derivatives

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In the structures of 5-(1-hydroxyethylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione, $C_8H_{10}N_2O_4$, (I), 1,3-dimethyl-5-[1-(propylamino)ethylidene]pyrimidine-2,4,6(1H,3H,5H)-trione, $C_{11}H_{17}N_3O_3$, (II), and 5-[1-(2,2-dimethoxyethylamino)ethylidene]-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione, $C_{12}H_{19}N_3O_5$, (III), there are no direction-specific intermolecular interactions. The molecules in 5-[1-(benzylamino)ethylidene]-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione, $C_{15}H_{17}N_3O_3$, (IV), are linked into chains of edge-fused rings by a combination of one C—H···O hydrogen bond and one C—H···π(arene) hydrogen bond, while the molecules in 5-(1-hydrazinoethylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione, $C_8H_{12}N_4O_3$, (V), are linked into a continuous framework structure by three distinct N—H···O hydrogen bonds, each involving a different O-atom acceptor. Each of compounds (I)–(V) contains an intramolecular hydrogen bond, of the O—H···O type in (I) and of the N—H···O type in (II)–(V).

Comment

It has recently been reported (da Silva & Lima, 2003) that the title compound, (I), which is the enol tautomer of 1,3-dimethyl-5-acetylbarbituric acid, reacts readily with primary amines to form enamines. We report here the structures of (I), of three representative enamine derivatives, (II)–(IV), and of the hydrazine derivative (V) (Figs. 1–5).

The bond distances in (I) (Table 1), in particular the C4—O4 and C51—O51 distances on the one hand and the C4—C5 and C5—C51 distances on the other, clearly demonstrate the enol constitution, consistent with the location of atom H51 as deduced from a difference map. In each of compounds (II)–

(V), atom N51 has a planar configuration, and the C51—N51 distances are all typical of their type (Allen et al., 1987). Atom N52 in (V) has a distinctly pyramidal coordination environment, with a sum of bond angles, based on H-atom coordinates derived from difference maps, of 328.8°. The N51—N52 distance in (V) corresponds closely to the mean value of 1.420 Å for >N—N< distances where the coordination of one N atom is planar and that of the other is pyramidal.

(I)

(II)

(III)

(IV)

(V)
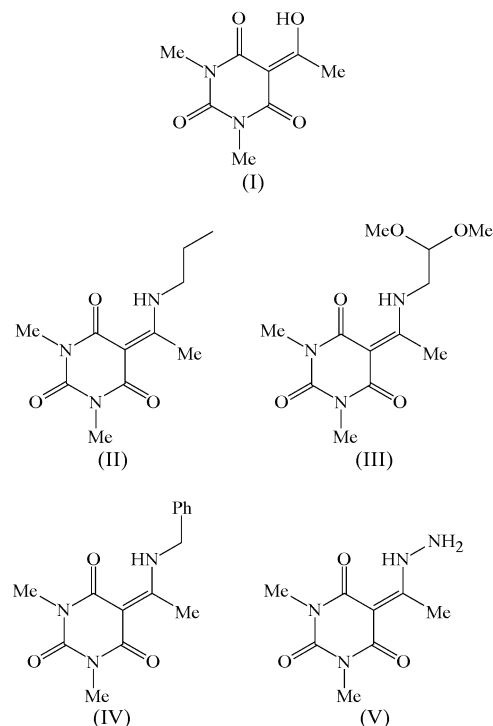
There is an intramolecular hydrogen bond in each of (I)–(V), of O—H···O type in (I) and of N—H···O type in (II)–(V) (Table 2). There are no direction-specific intermolecular interactions in (I)–(III), but the molecules in (IV) and (V) are linked by intermolecular hydrogen bonds (Table 2) into supramolecular structures that are one- and three-dimensional, respectively.

The one-dimensional supramolecular structure of (IV) is generated by a combination of C—H···O and C—H···π(arene) hydrogen bonds (Table 2). Aryl atom C514 in the molecule at (x, y, z) acts as a hydrogen-bond donor to
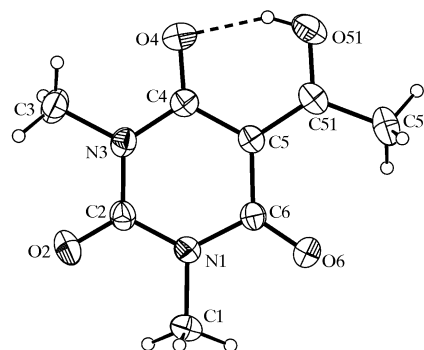

Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

carbonyl atom O4 in the molecule at $(1 - x, 1 - y, 1 - z)$, so generating a centrosymmetric $R_2^2(22)$ ring (Bernstein *et al.*, 1995) centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ (Fig. 6). In addition, methylene atom C53 in the molecule at (x, y, z) acts as a hydrogen-bond donor to the C511–C516 aryl ring in the molecule at $(1 - x, -y, 1 - z)$, so generating a second centrosymmetric ring, this time centred at $(\frac{1}{2}, 0, \frac{1}{2})$. The combination of these two centrosymmetric motifs thus generates a chain of edge-fused rings running parallel to the [010] direction, with the larger rings centred at $(\frac{1}{2}, \frac{1}{2} + n, \frac{1}{2})$ (n = zero or integer) and the smaller rings centred at $(\frac{1}{2}, n, \frac{1}{2})$ (n = zero or integer) (Fig. 6).

The three-dimensional supramolecular structure of (V) is of some complexity but can readily be analysed in terms of a series of one-dimensional substructures and their simple combinations. In the first substructure, atom N52 in the molecule at (x, y, z) acts as a hydrogen-bond donor, *via* atom H52D, to atom O4 in the molecule at $(-\frac{1}{2} + x, \frac{1}{2} - y, -z)$, so producing a $C(7)$ chain running parallel to the [100] direction and generated by the 2_1 screw axis along $(x, \frac{1}{4}, 0)$ (Fig. 7). In the second substructure, atom N52 at (x, y, z) acts as a donor, *via* atom H52E, to atom O2 in the molecule at $(2 - x, -\frac{1}{2} + y, \frac{1}{2} - z)$, so producing a $C(9)$ chain running parallel to the [010] direction, now generated by the 2_1 screw axis along $(1, y, \frac{1}{4})$ (Fig. 8).
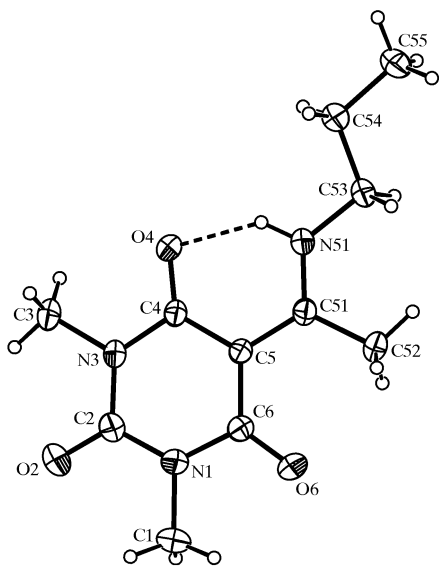
Figure 2
The molecule of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.
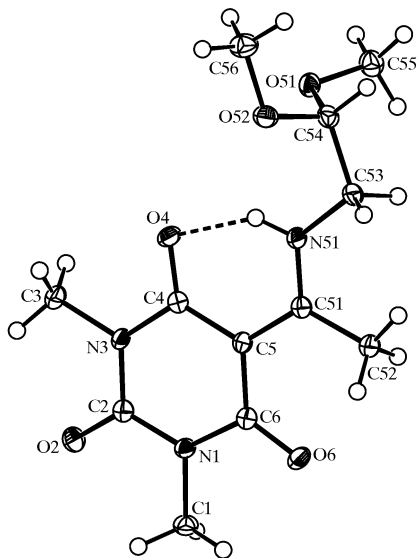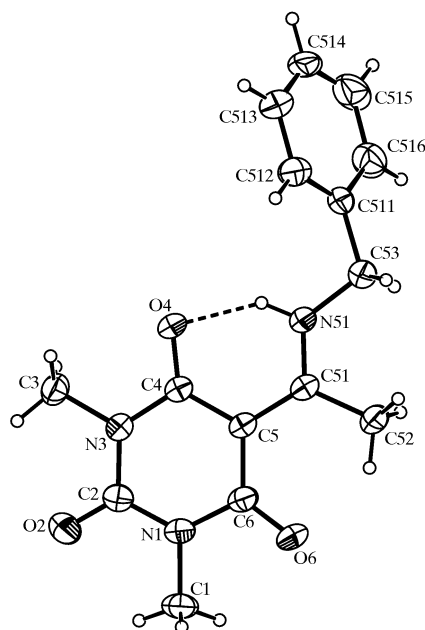
Figure 4
The molecule of (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Figure 3
The molecule of (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.
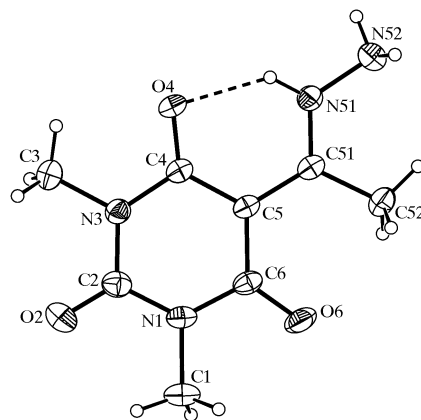
Figure 5
The molecule of (V), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Each of these chains, along [100] and [010], respectively, utilizes just one N—H···O hydrogen bond, but the combination of these two interactions produces a third chain motif, running parallel to the [001] direction. Atom N52 in the molecule at $(-\frac{1}{2} + x, \frac{1}{2} - y, -z)$ acts as a hydrogen-bond donor, *via* atom H52*E*, to atom O2 in the molecule at $(\frac{3}{2} - x, 1 - y, -\frac{1}{2} + z)$, while atom N52 at $(\frac{3}{2} - x, 1 - y, -\frac{1}{2} + z)$ acts as a donor, *via* atom H52*D*, to atom O4 at $(2 - x, \frac{1}{2} + y, -\frac{1}{2} - z)$; finally, atom N52 at $(2 - x, \frac{1}{2} + y, -\frac{1}{2} - z)$ acts as a donor, *via* atom H52*E*, to atom O2 in the molecule at $(x, y, -1 + z)$. In this manner a $C_2^2(16)$ chain is generated along [001] (Fig. 9).

These three chain motifs (Figs. 7–9) together produce a three-dimensional framework structure, which is generated solely by the action of 2_1 screw axes and which utilizes only half of the molecules within the unit cell. There is, accordingly, a second such framework present, which is interwoven with the first framework and related to it by inversion. These two three-dimensional substructures are, however, linked by the final N—H···O hydrogen bond; this is, in fact, one component of an effectively planar three-centre N—H···(O)$_2$ system (Table 2). Atom N51 in the molecule at (x, y, z) acts as a

donor, not only intramolecularly to atom O4 but also to atom O6 in the molecule at $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, so producing a $C(6)$ chain running parallel to the [001] direction and generated by the *c*-glide plane at $y = \frac{1}{4}$ (Fig. 10). This final motif suffices to link the two frameworks and hence to link all of the molecules into a single three-dimensional structure.

Figure 8
Part of the crystal structure of (V), showing the formation of a $C(9)$ chain along [010]. For clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(2 - x, -\frac{1}{2} + y, \frac{1}{2} - z)$ and $(2 - x, \frac{1}{2} + y, \frac{1}{2} - z)$, respectively.
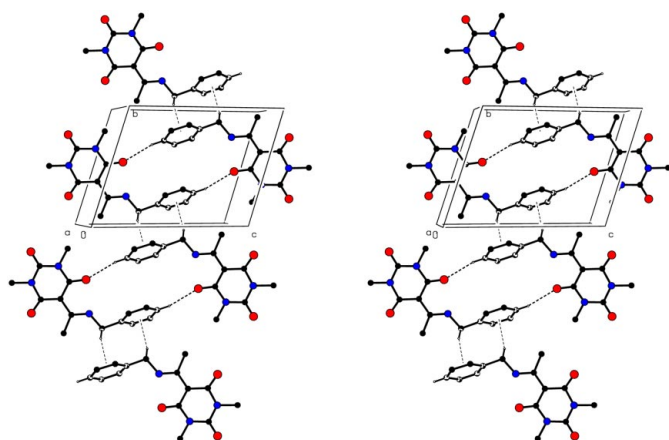
Figure 6
A stereoview of part of the crystal structure of (IV), showing the formation of a chain of edge-fused rings along [010]. For clarity, H atoms not involved in the motifs shown have been omitted.
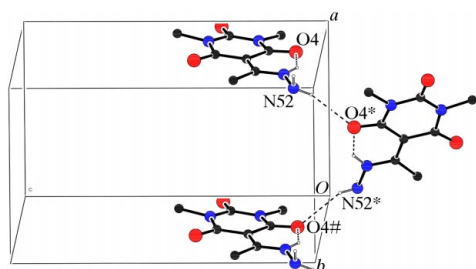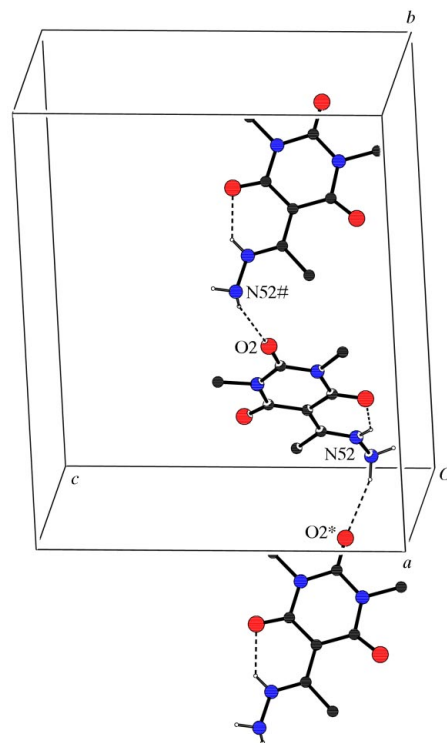
Figure 7
Part of the crystal structure of (V), showing the formation of a $C(7)$ chain along [100]. For clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(-\frac{1}{2} + x, \frac{1}{2} - y, -z)$ and $(-1 + x, y, z)$, respectively.
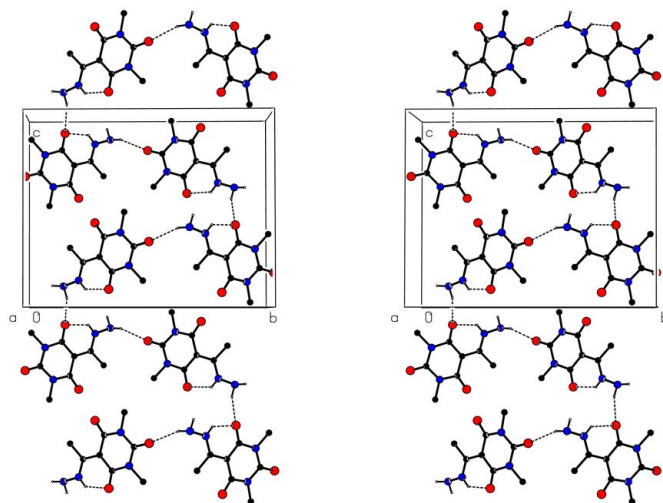
Figure 9
A stereoview of part of the crystal structure of (V), showing the formation of a $C_2^2(16)$ chain along [001]. For clarity, H atoms bonded to C atoms have been omitted.

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Figure 10
Part of the crystal structure of (V), showing the formation of a $C(6)$ chain along [001]. For clarity, H atoms bonded to C atoms have been omitted. Atom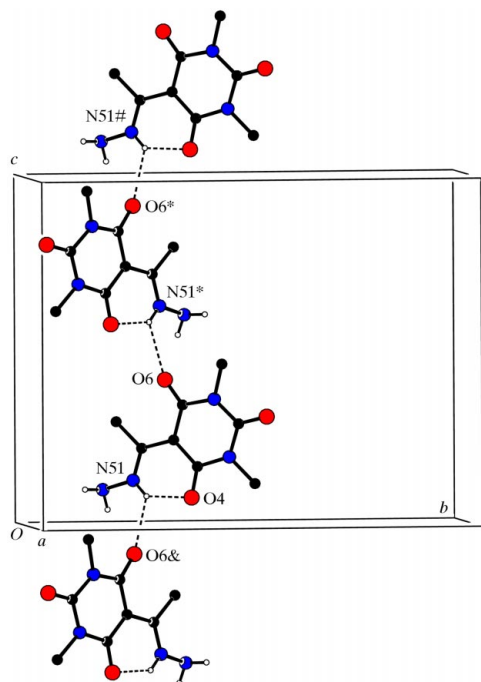s marked with an asterisk (*), a hash (#) or an ampersand (&) are at the symmetry positions $(x, \frac{1}{2} - y, \frac{1}{2} + z)$, $(x, y, 1 + z)$ and $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, respectively.

Experimental

Samples of compounds (I)–(V) were prepared as described by da Silva & Lima (2003). Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of solutions in ethanol.

Compound (I)

Crystal data

$C_8H_{10}N_2O_4$
$M_r = 198.18$
Monoclinic, $P2_1/c$
$a = 8.6066 (5)$ Å
$b = 9.1751 (5)$ Å
$c = 11.9799 (7)$ Å
$\beta = 109.321 (2)°$
$V = 892.73 (9)$ Å^3
$Z = 4$

$D_x = 1.475$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3198 reflections
$\theta = 2.9$–$32.5°$
$\mu = 0.12$ mm^{-1}
$T = 291 (2)$ K
Block, colourless
$0.52 \times 0.33 \times 0.20$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ–ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.934$, $T_{max} = 0.976$
7606 measured reflections

3198 independent reflections
1926 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 32.5°$
$h = -13 \rightarrow 5$
$k = -13 \rightarrow 9$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.207$
$S = 1.03$
3198 reflections
131 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.102P)^2 + 0.1435P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Compound (II)

Crystal data

$C_{11}H_{17}N_3O_3$
$M_r = 239.28$
Monoclinic, Pn
$a = 4.1085 (3)$ Å
$b = 8.4497 (5)$ Å
$c = 17.1272 (11)$ Å
$\beta = 94.721 (2)°$
$V = 592.56 (7)$ Å^3
$Z = 2$

$D_x = 1.341$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2129 reflections
$\theta = 2.4$–$32.5°$
$\mu = 0.10$ mm^{-1}
$T = 291 (2)$ K
Lath, colourless
$0.49 \times 0.40 \times 0.12$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ–ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.931$, $T_{max} = 0.988$
5869 measured reflections

2129 independent reflections
1671 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 32.5°$
$h = -6 \rightarrow 6$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.126$
$S = 1.01$
2129 reflections
158 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0836P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Compound (III)

Crystal data

$C_{12}H_{19}N_3O_5$
$M_r = 285.30$
Trigonal, $R\bar{3}$
$a = 18.4577 (4)$ Å
$c = 19.7736 (4)$ Å
$V = 5834.1 (2)$ Å^3
$Z = 18$
$D_x = 1.462$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 2965 reflections
$\theta = 3.1$–$27.5°$
$\mu = 0.12$ mm^{-1}
$T = 120 (2)$ K
Block, colourless
$0.40 \times 0.40 \times 0.30$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ–ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.950$, $T_{max} = 0.966$
14 896 measured reflections

2965 independent reflections
2329 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.5°$
$h = -19 \rightarrow 23$
$k = -23 \rightarrow 19$
$l = -22 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.115$
$S = 1.11$
2965 reflections
186 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 2.9334P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Compound (IV)

Crystal data

$C_{15}H_{17}N_3O_3$
$M_r = 287.32$
Triclinic, $P\bar{1}$
$a = 7.0696 (5)$ Å
$b = 9.0734 (7)$ Å
$c = 11.9214 (8)$ Å
$\alpha = 70.995 (2)°$
$\beta = 82.084 (2)°$
$\gamma = 84.729 (2)°$
$V = 715.23 (9)$ Å^3

$Z = 2$
$D_x = 1.334$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3241 reflections
$\theta = 2.4$–$27.5°$
$\mu = 0.10$ mm^{-1}
$T = 291 (2)$ K
Plate, colourless
$0.40 \times 0.34 \times 0.06$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3241 independent reflections
φ–ω scans	1951 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$R_{int} = 0.022$
	$\theta_{max} = 27.5°$
$T_{min} = 0.959$, $T_{max} = 0.994$	$h = -9 \rightarrow 8$
6134 measured reflections	$k = -11 \rightarrow 11$
	$l = -15 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.091P)^2 + 0.0203P]$
$R[F^2 > 2\sigma(F^2)] = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.167$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
3241 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}
193 parameters	
H-atom parameters constrained	

Compound (V)

Crystal data

$C_8H_{12}N_4O_3$	Mo $K\alpha$ radiation
$M_r = 212.22$	Cell parameters from 2160 reflections
Orthorhombic, *Pbca*	$\theta = 2.4$–$32.5°$
$a = 7.9669$ (5) Å	$\mu = 0.12$ mm^{-1}
$b = 17.2670$ (11) Å	$T = 291$ (2) K
$c = 13.6223$ (9) Å	Needle, colourless
$V = 1873.9$ (2) Å^3	$0.60 \times 0.07 \times 0.06$ mm
$Z = 8$	
$D_x = 1.504$ Mg m^{-3}	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	2160 independent reflections
φ–ω scans	1284 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$R_{int} = 0.057$
	$\theta_{max} = 27.5°$
$T_{min} = 0.928$, $T_{max} = 0.993$	$h = -10 \rightarrow 9$
13 048 measured reflections	$k = -22 \rightarrow 20$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.0725P)^2]$
$wR(F^2) = 0.129$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.94$	$(\Delta/\sigma)_{max} < 0.001$
2160 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
139 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}

Table 1
Selected bond distances (Å) for compounds (I)–(V).

	(I)	(II)	(III)	(IV)	(V)
C2—O2	1.208 (2)	1.219 (2)	1.2211 (18)	1.218 (2)	1.221 (2)
C4—O4	1.253 (2)	1.232 (2)	1.2442 (16)	1.239 (2)	1.239 (2)
C4—C5	1.437 (2)	1.440 (2)	1.4422 (18)	1.439 (2)	1.435 (2)
C5—C51	1.396 (2)	1.427 (2)	1.4308 (18)	1.422 (2)	1.429 (3)
C51—O51	1.311 (2)	–	–	–	–
C51—N51	–	1.313 (2)	1.3172 (17)	1.314 (2)	1.312 (2)
N51—N52	–	–	–	–	1.415 (2)
C6—O6	1.225 (2)	1.223 (2)	1.2278 (16)	1.222 (2)	1.224 (2)

For (I) and (V), space groups $P2_1/c$ and *Pbca*, respectively, were uniquely assigned from the systematic absences. For (II), the systematic absences permitted *Pn* and *P2/n* as possible space groups; *Pn* was selected and confirmed by the structure analysis. For (III), the systematic absences permitted $R3$ and $R\bar{3}$ as possible space groups; $R\bar{3}$ was selected and confirmed by the structure analysis. Crystals of

Table 2
Hydrogen-bond parameters (Å, °) for compounds (I)–(V).

Compound	D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
(I)	O51—H51$\cdots$O4	0.82	1.70	2.450 (2)	151
(II)	N51—H51$\cdots$O4	0.86	1.85	2.563 (2)	139
(III)	N51—H51$\cdots$O4	0.88	1.86	2.5829 (14)	139
(IV)	N51—H51$\cdots$O4	0.86	1.85	2.559 (2)	139
	C514—H514$\cdots$O4^i	0.93	2.48	3.392 (2)	168
	C53—H53$B\cdots Cg^{ii}$	0.97	2.66	3.557 (2)	154
(V)	N51—H51$\cdots$O4	0.86	1.92	2.600 (2)	135
	N51—H51$\cdots$O6^{iii}	0.86	2.35	2.974 (2)	129
	N52—H52$D\cdots$O4^{iv}	0.86	2.45	3.230 (2)	152
	N52—H52$E\cdots$O2^v	0.86	2.32	3.144 (2)	159

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, -y, 1-z$; (iii) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (iv) $-\frac{1}{2}+x, \frac{1}{2}-y, -z$; (v) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$. Note: Cg is the centroid of the C511–C516 ring.

(IV) are triclinic; space group $P\bar{1}$ was selected and confirmed by the structure analysis. All H atoms were located from difference maps and then treated as riding atoms, with C—H distances of 0.98 (CH$_3$), 0.99 (CH$_2$) or 1.00 Å (aliphatic CH) and N—H distances of 0.88 Å (at 120 K), and with C—H distances of 0.93 (aromatic), 0.96 (CH$_3$) or 0.97 Å (CH$_2$), N—H distances of 0.86 Å and O—H distances of 0.82 Å (at 291 K) [U_{iso}(H) = 1.2U_{eq}(C,N,O) or 1.5U_{eq}(C$_{methyl}$)]. In the absence of significant anomalous dispersion, the Flack (1983) parameter for (II) was inconclusive (Flack & Bernardinelli, 2000); it was therefore not possible to determine the correct orientation of the structure with respect to the polar axis directions (Jones, 1986) and, accordingly, Friedel pairs were merged prior to the final refinements.

For compounds (I), (II), (IV) and (V), data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

For compound (III), data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS*97; program(s) used to refine structure: *OSCAIL* and *SHELXL*97; molecular graphics: *PLATON*; software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA*.

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