organic compounds

Four 7-aryl-substituted pyrido[2,3-*d*]-pyrimidine-2,4(1*H*,3*H*)-diones: similar molecular structures but different crystal structures

Jorge Trilleras,[a] Jairo Quiroga,[a] Justo Cobo,[b] Michael B. Hursthouse[c] and Christopher Glidewell[d]*

[a]Grupo de Investigación de Compuestos Heterocíclicos, Departamento de Química, Universidad de Valle, AA 25360 Cali, Colombia, [b]Departamento de Química Inorgánica y Orgánica, Universidad de Jaén, 23071 Jaén, Spain, [c]School of Chemistry, University of Southampton, Highfield, Southampton SO17 1BJ, England, and [d]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland
Correspondence e-mail: cg@st-andrews.ac.uk

Molecules of 1,3-dimethyl-7-(4-methylphenyl)pyrido[2,3-*d*]-pyrimidine-2,4(1*H*,3*H*)-dione, $C_{16}H_{15}N_3O_2$, (I), are linked by paired C—H$\cdots$O hydrogen bonds to form centrosymmetric $R_2^2(10)$ dimers, which are linked into chains by a single π–π stacking interaction. A single C—H$\cdots$O hydrogen bond links the molecules of 7-(biphenyl-4-yl)-1,3-dimethylpyrido-[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione, $C_{21}H_{17}N_3O_2$, (II), into $C(10)$ chains, which are weakly linked into sheets by a π–π stacking interaction. In 7-(4-fluorophenyl)-3-methylpyrido-[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione, $C_{14}H_{10}FN_3O_2$, (III), an N—H$\cdots$O hydrogen bond links the molecules into $C(6)$ chains, which are linked into sheets by a π–π stacking interaction. The molecules of 7-(4-methoxyphenyl)-3-methyl-pyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione, $C_{15}H_{13}N_3O_3$, (IV), are also linked into $C(6)$ chains by an N—H$\cdots$O hydrogen bond, but here the chains are linked into sheets by a combination of two independent C—H$\cdots$$\pi$(arene) hydrogen bonds.

Comment

Pyrido[2,3-*d*]pyrimidine heterocycles (also known as 5-de-azapteridines) have received considerable attention over the past decade as a result of the wide range of biological activity that they exhibit (Devi *et al.*, 2003; Tu *et al.*, 2008), including, for example, bronchodilator, vasodilator, antiallergic, cardio-tonic, antihypertensive or hepatoprotective activities, as well as for their role in the treatment of proliferative diseases (Devi *et al.*, 2004). As part of a wide-ranging project on the synthesis and characterization of fused pyrimidine systems under solvent-free conditions, we report here the structures of four examples of 7-arylpyrido[2,3-*d*]pyrimidine derivatives

prepared by cyclocondensation reactions between 6-amino-pyrimidine-5-carbaldehydes and acetophenones, utilizing sol-vent-free fusion reactions promoted by BF_3–Et_2O catalysis.

(I) Ar = 4-MeC$_6$H$_4$
(II) Ar = 4-PhC$_6$H$_4$

(III) Ar' = 4-FC$_6$H$_4$
(IV) Ar' = 4-MeOC$_6$H$_4$

(V) Ar = 4-BrC$_6$H$_4$
(VI) Ar = 4-ClC$_6$H$_4$
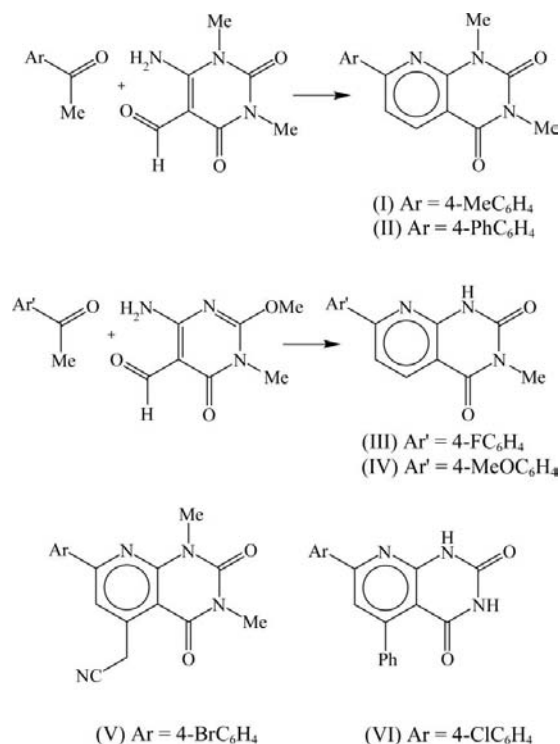
While the molecular structures of the pair of compounds 1,3-dimethyl-7-(4-methylphenyl)pyrido[2,3-*d*]pyrimidine-2,4-(1*H*,3*H*)-dione, (I) (Fig. 1), and 7-(1,1'-biphenyl-4-yl)-1,3-di-methylpyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione, (II) (Fig. 2), are broadly similar in constitution and very similar in conformation, they exhibit significant differences in their crystal structures, in particular in their space groups and in their pattern of supramolecular aggregation. Similarly, the pair of compounds 7-(4-fluorophenyl)-3-methylpyrido[2,3-*d*]pyri-midine-2,4(1*H*,3*H*)-dione, (III) (Fig. 3), and 7-(4-methoxy phenyl)-3-methylpyrido[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione, (IV) (Fig. 4), which are closely related to (I) and (II), have
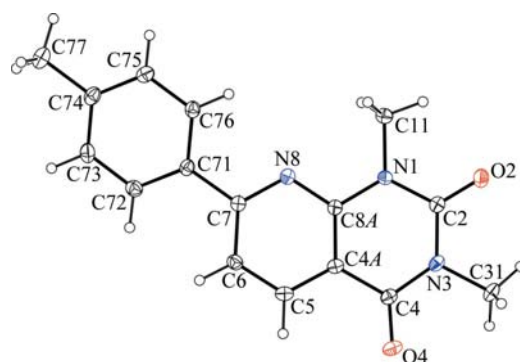

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

similar constitutions and conformations, but again they crystallize in different space groups and exhibit different patterns of supramolecular aggregation. Overall, no two of the compounds reported here crystallize in the same space group or have similar unit-cell dimensions or exhibit the same pattern of direction-specific intermolecular interactions.
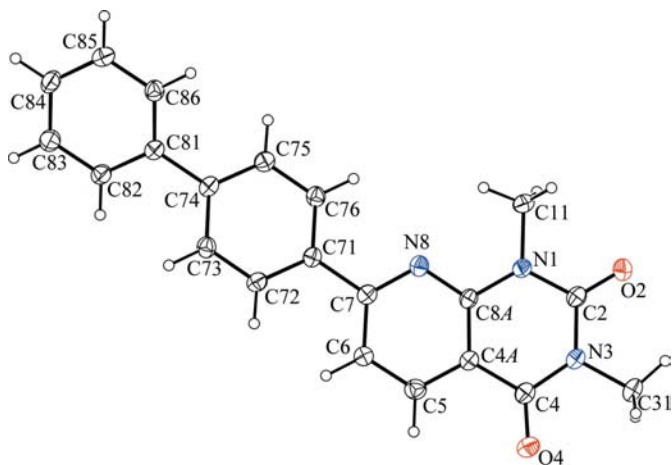

Figure 2
The molecular structure of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.
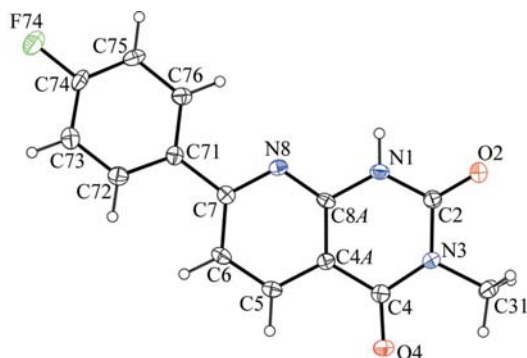

Figure 3
The molecular structure of (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.
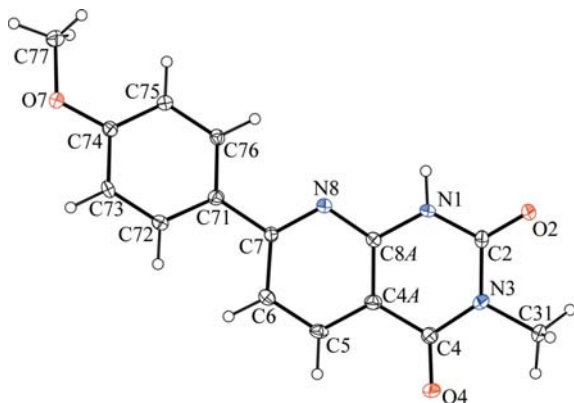

Figure 4
The molecular structure of (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

The molecular conformations are straightforwardly definable in terms of the interplanar angle between the pyridine ring and the pendent aryl ring (C71–C76), with an additional angle between the two rings of the biphenyl-4-yl substituent in (II) (Table 1). These angles indicate that the molecular skeletons do not deviate much from overall planarity, a point emphasized by the very small deviation, 0.012 (5) Å, of the methoxy C atom from the plane of the adjacent aryl ring in (IV). The bond distances and angles show no unexpected values.

In our analysis of the intermolecular interactions, we have discounted all intermolecular contacts involving methyl C—H bonds on the usual grounds that these bonds are of low acidity, while the rotation about the adjacent C—C bonds of methyl groups bonded to planar rings are hindered by extremely low barriers, typically a few J mol^{-1} (Tannenbaum *et al.*, 1956; Naylor & Wilson, 1957), and so are likely to be undergoing very fast rotation about the adjacent C—C bonds.

In (I), there is a single C—H···O hydrogen bond utilizing a pyridine C—H bond as the donor (Table 2), and this links pairs of molecules into centrosymmetric dimers characterized by an $R_2^2(10)$ (Bernstein *et al.*, 1995) motif. In addition, the pyridine rings of the molecules at (*x*, *y*, *z*) and (−*x*, −*y* + 1, −*z* + 1) are strictly parallel, with an interplanar spacing of 3.439 (2) Å; the corresponding ring-centroid separation is 3.693 (2) Å, with a near-ideal ring-centroid offset of 1.345 (2) Å. The combined effect of these two interactions is to link the molecules into a π-stacked chain of hydrogen-bonded dimers, running parallel to the [010] direction, with $R_2^2(10)$ rings centred at $(0, n, \frac{1}{2})$, where *n* represents an integer, alternating with π–π stacking interactions across $(0, n + \frac{1}{2}, \frac{1}{2})$, where *n* again represents an integer (Fig. 5).

The crystal structure of (II) also contains just one intermolecular C—H···O hydrogen bond, but this now involves an aryl C—H bond as the donor (Table 2), and its effect is to link molecules related by the 2_1 screw axis along $(\frac{1}{2}, y, \frac{3}{4})$ into a
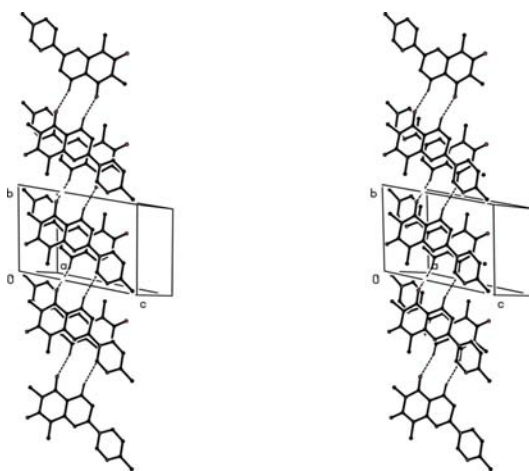

Figure 5
A stereoview of part of the crystal structure of (I), showing the formation of a chain of π-stacked hydrogen-bonded dimers along [010]. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

$C(10)$ chain running parallel to the [010] direction. The pyridine and phenyl rings in the molecules at (x, y, z) and $(-x, -y + 1, -z + 1)$, respectively, are not parallel, but the dihedral angle between them is only $12.8 (2)°$; the ring-centroid separation is $3.718 (2)$ Å and the interplanar distance is *ca* 3.53 Å, corresponding to a ring-centroid offset of *ca* 1.17 Å. Thus, these centrosymmetrically related molecules are weakly linked by the π–π stacking interaction and the overall effect of this is to link the hydrogen-bonded chain into a sheet parallel to $(10\bar{2})$ (Fig. 6).

In each of (III) and (IV), a single N—H$\cdots$O hydrogen-bond links the molecules into a $C(6)$ chain. Although the same hydrogen-bond donor and acceptor are utilized in each structure, the construction and orientation of the chains is different. In (III), the chain consists of molecules related by a 2_1 screw axis parallel to the [010] direction, while the chain in
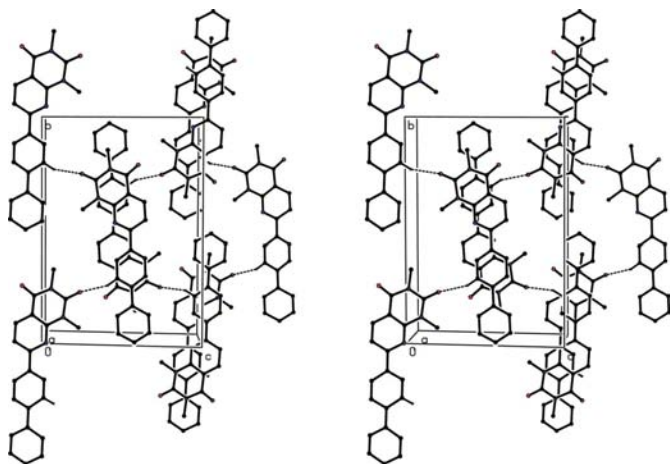
(IV) consists of molecules related by translation along [100]. In addition, the subsequent linking of the chains is different in the two structures. The pyridine ring in the molecule of (III) at (x, y, z) makes angles of $1.1 (2)°$ with the phenyl ring in each of the molecules at $(x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1)$ and $(x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1)$, with ring-centroid separations of $3.759 (2)$ and $3.748 (2)$ Å, respectively. The interplanar spacings are *ca* 3.315 and *ca* 3.39 Å, respectively, corresponding to ring-centroid offsets of *ca* 1.77 and *ca* 1.60 Å, respectively. The co-operative action of these two independent stacking interactions along [100] links the hydrogen-bonded chains along [010] into a sheet parallel to (001) (Fig. 7). In the structure of (IV), by contrast, the chains generated by the C—H$\cdots$O hydrogen bond are linked by two independent C—H$\cdots\pi$(arene) hydrogen bonds, both involving the same ring as the acceptor. The C71–C76 ring in the molecule at (x, y, z) acts as a hydrogen-bond acceptor from atom C72 in the molecule at $(x + \frac{1}{2}, -y, z)$ and from atom C75 in the molecule at $(x - \frac{1}{2}, -y + 1, z)$, so forming a chain running parallel to the [1$\bar{1}$0] direction; the two C—H$\cdots\pi$ hydrogen bonds lie on opposite faces of the ring, with C$\cdots$Cg$\cdots$C and H$\cdots$Cg$\cdots$H angles of 179 and 177°, respectively. The combination of the [100] and [1$\bar{1}$0] chains then generates a sheet parallel to (001) (Fig. 8).

In view of the different patterns of supramolecular aggregation found here for (I)–(IV), it is of interest briefly to compare these structures with those of the closely related compounds (V) [Cambridge Structural Database (Allen, 2002) refcode QOQQIW (Sarkhel *et al.*, 2001)] and (VI) (refcode XEBCUD; Wang *et al.*, 2006). The crystal structure of (V) was described (Sarkhel *et al.*, 2001) as containing C—H$\cdots$O and C—H$\cdots$Br hydrogen bonds and π–π stacking interactions, although the structural effects of these interactions were not specified. However, it is now well established that Br and Cl bonded to carbon are both exceptionally poor acceptors of hydrogen bonds, even from O—H and N—H units, so that the C—H$\cdots$Br contacts in (V) and the C—H$\cdots$Cl contacts in (VI) are likely to be no more than normal van der Waals contacts (Brammer *et al.*, 2001; Thallypally &

Figure 6
A stereoview of part of the crystal structure of (II), showing the formation of a sheet parallel to $(10\bar{2})$ built from π-stacked hydrogen-bonded chains along [010]. For the sake of clarity, H atoms not involved in the motif shown have been omitted.
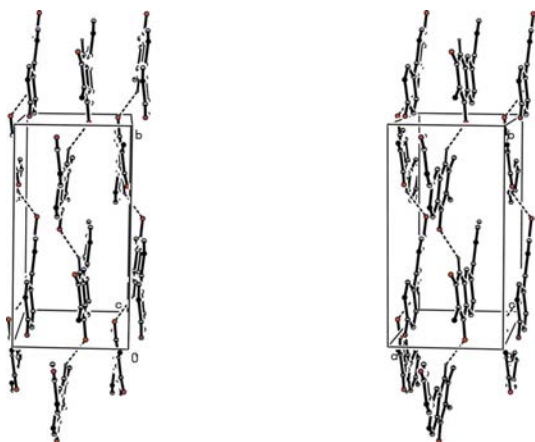

Figure 7
A stereoview of part of the crystal structure of (III), showing the formation of a sheet parallel to (001) built from π-stacked hydrogen-bonded chains along [010]. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

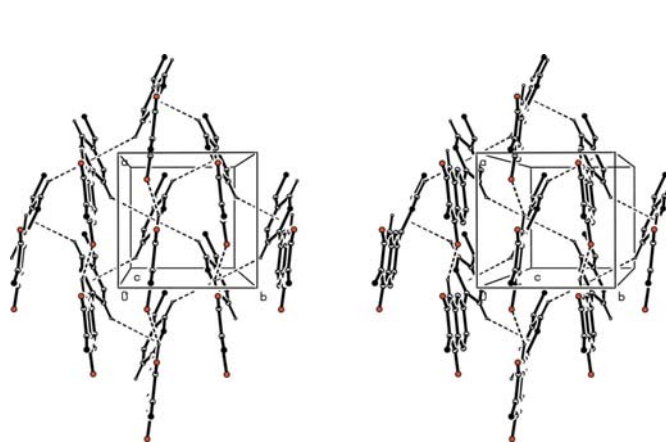**Figure 8**
A stereoview of part of the crystal structure of (IV), showing the formation of a sheet parallel to (001) built from one C—H$\cdots$O hydrogen bond and two C—H$\cdots\pi$(arene) hydrogen bonds. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

Nangia, 2001). Accordingly, the structure of (V) consists of $C(10)$ hydrogen-bonded chains, running parallel to the [001] direction and built from a single C—H···O hydrogen bond; antiparallel pairs of these chains, related by inversion, are linked by the π–π stacking interaction (Fig. 9). The original report on (VI) (Wang *et al.*, 2006) stated that 'the crystal structure is stabilized by N—H···O, C—H···O and C—H···Cl hydrogen bonds', but it gave no information as to the actions of these interactions. Re-examination of this structure shows that most of the so-called hydrogen bonds listed by the authors have H···A distances far too long to be of structural significance. In the event, only two hydrogen bonds linking the
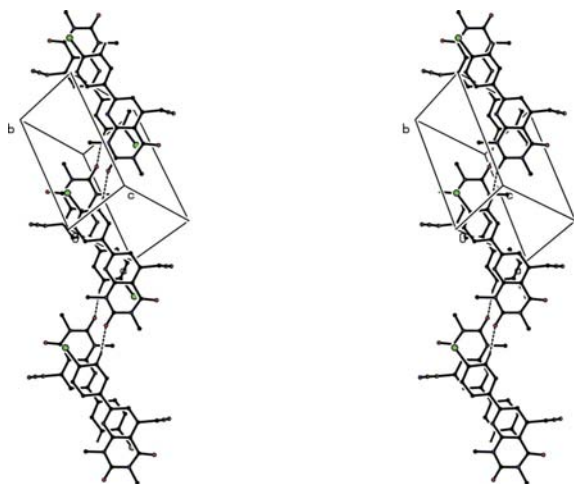
heterocyclic components are significant, and they link these molecules into chains of centrosymmetric rings running parallel to the [001] direction; the dimethylformamide molecules are pendent from the chain, but they play no other role in the hydrogen bonding. Within the chain, $R_2^2(8)$ rings containing paired N—H···O hydrogen bonds are centred at $(0, 0, n)$, where n represents an integer, and these alternate with $R_2^2(16)$ rings containing paired C—H···O hydrogen bonds, which are centred at $(0, 0, \frac{1}{2} + n)$, where n represents an integer (Fig. 10). These chains are further linked by a π–π stacking interaction to form a sheet parallel to (100).

Figure 9
A stereoview of part of the crystal structure of QOQQIW (Sarkhel *et al.*, 2001), showing the formation of a π-stacked pair of $C(10)$ hydrogen-bonded chains. The original atom coordinates have been employed and, for the sake of clarity, H atoms not involved in the motif shown have been omitted.
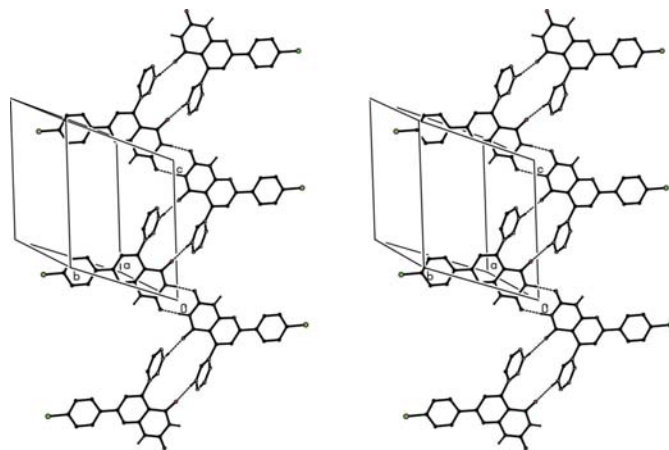
Figure 10
A stereoview of part of the crystal structure of XEBCUD (Wang *et al.*, 2006), showing the formation of a chain of hydrogen-bonded $R_2^2(8)$ and $R_2^2(16)$ rings. The original atom coordinates have been employed and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted, as have the dimethylformamide molecules.

Experimental

Equimolar quantities of the appropriate 6-aminopyrimidine-5-carbaldehyde, 6-amino-5-formyl-1,3-dimethylpyrimidine-2,4(1H,3H)-dione for (I) and (II) or 6-amino-5-formyl-2-methoxy-3-methylpyrimidin-4(3H)-one for (III) and (IV), and the appropriate 4-substituted acetophenone 4-RC$_6$H$_4$COCH$_3$, where R is Me for (I), Ph for (II), F for (III) and MeO for (IV), were mixed in the absence of solvent. Three drops of BF$_3$–Et$_2$O were added to each mixture, which was then heated in an oil bath at 443 K for 30 s. The resulting dark-brown liquids were diluted with ethanol and cooled to ambient temperature. The solid products were collected by filtration, washed with ethanol and then recrystallized to give the pure pyrido[2,3-d]pyrimidine derivatives as crystals suitable for single-crystal X-ray diffraction. For (I), yellow solid, crystallized from ethanol/DMF, yield 50%, m.p. 458–460 K; HR–MS found: 281.1159; C$_{16}$H$_{15}$N$_3$O$_2$ requires: 281.1164. For (II), yellow solid, purified by column chromatography using chloroform as eluant, and recrystallized from ethanol/DMF, yield 60%, m.p. 506–509 K; HR–MS found: 343.1313; C$_{21}$H$_{17}$N$_3$O$_2$ requires: 343.1321. For (III), yellow solid, crystallized from DMF, yield 60%, m.p. 568–570 K; HRMS found: 271.0752; C$_{14}$H$_{10}$FN$_3$O$_2$ requires: 271.0757. For (IV), yellow solid, crystallized from DMF, yield 70%, m.p. >573 K. HR–MS found: 283.0966; C$_{15}$H$_{13}$N$_3$O$_3$ requires: 283.0957.

Compound (I)

Crystal data

C$_{16}$H$_{15}$N$_3$O$_2$	$\gamma = 101.152\ (6)°$
$M_r = 281.31$	$V = 677.71\ (11)$ Å^3
Triclinic, $P\bar{1}$	$Z = 2$
$a = 6.9953\ (5)$ Å	Mo $K\alpha$ radiation
$b = 7.9221\ (7)$ Å	$\mu = 0.09$ mm^{-1}
$c = 12.5140\ (13)$ Å	$T = 120$ K
$\alpha = 93.466\ (4)°$	$0.20 \times 0.10 \times 0.02$ mm
$\beta = 92.993\ (6)°$	

Data collection

Bruker–Nonius KappaCCD diffractometer	11686 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	2989 independent reflections
	1699 reflections with $I > 2\sigma(I)$
$T_{min} = 0.972$, $T_{max} = 0.998$	$R_{int} = 0.080$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$	193 parameters
$wR(F^2) = 0.200$	H-atom parameters constrained
$S = 1.03$	$\Delta\rho_{max} = 0.45$ e Å^{-3}
2989 reflections	$\Delta\rho_{min} = -0.35$ e Å^{-3}

Compound (II)

Crystal data

$C_{21}H_{17}N_3O_2$
$M_r = 343.38$
Monoclinic, $P2_1/c$
$a = 7.2314$ (5) Å
$b = 17.5834$ (18) Å
$c = 12.4864$ (15) Å
$\beta = 97.782$ (8)°

$V = 1573.1$ (3) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 120$ K
$0.45 \times 0.27 \times 0.15$ mm

Data collection

Bruker–Nonius KappaCCD
  diffractometer
Absorption correction: multi-scan
  (*SADABS*; Sheldrick, 2003)
  $T_{min} = 0.947$, $T_{max} = 0.986$

40144 measured reflections
3612 independent reflections
1851 reflections with $I > 2\sigma(I)$
$R_{int} = 0.092$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.244$
$S = 1.03$
3612 reflections

237 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Compound (III)

Crystal data

$C_{14}H_{10}FN_3O_2$
$M_r = 271.25$
Orthorhombic, $P2_12_12_1$
$a = 6.7658$ (8) Å
$b = 12.978$ (2) Å
$c = 13.162$ (2) Å

$V = 1155.7$ (3) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.12$ mm^{-1}
$T = 120$ K
$0.10 \times 0.10 \times 0.10$ mm

Data collection

Bruker–Nonius KappaCCD
  diffractometer
Absorption correction: multi-scan
  (*SADABS*; Sheldrick, 2003)
  $T_{min} = 0.968$, $T_{max} = 0.988$

11820 measured reflections
1538 independent reflections
833 reflections with $I > 2\sigma(I)$
$R_{int} = 0.164$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.155$
$S = 1.00$
1538 reflections

182 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

Compound (IV)

Crystal data

$C_{15}H_{13}N_3O_3$
$M_r = 283.28$
Orthorhombic, $Pca2_1$
$a = 6.6312$ (11) Å
$b = 6.8121$ (15) Å
$c = 27.653$ (7) Å

$V = 1249.2$ (5) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 120$ K
$0.28 \times 0.24 \times 0.22$ mm

Data collection

Bruker–Nonius KappaCCD
  diffractometer
Absorption correction: multi-scan
  (*SADABS*; Sheldrick, 2003)
  $T_{min} = 0.961$, $T_{max} = 0.977$

8019 measured reflections
1439 independent reflections
1181 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.147$
$S = 1.14$
1439 reflections
192 parameters

1 restraint
H-atom parameters constrained
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Table 1
Selected interplanar angles (°) for (I)–(IV).

	(I)	(II)	(III)	(IV)
Pyridine/C71–C76	19.7 (2)	12.8 (2)	15.3 (2)	21.7 (2)
C71–C76/C81–C86		33.0 (2)		
Pyridine/C81–C86		20.3 (2)		

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

Cg represents the centroid of the C71–C76 ring.

Compound	$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
(I)	C5—H5$\cdots$O4^i	0.95	2.39	3.306 (3)	161
(II)	C75—H75$\cdots$O2^{ii}	0.95	2.39	3.294 (3)	158
	C76—H76$\cdots$N8	0.95	2.48	2.805 (4)	100
(III)	N1—H1$\cdots$O4^{iii}	0.88	2.08	2.874 (4)	149
	C76—H76$\cdots$N8	0.95	2.42	2.753 (5)	100
(IV)	N1—H1$\cdots$O4^{iv}	0.88	1.99	2.765 (5)	146
	C72—H72$\cdots$Cg^v	0.95	2.65	3.448 (5)	142
	C75—H75$\cdots$Cg^{vi}	0.95	2.75	3.548 (5)	142

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

With the exception of the methyl H atoms bonded to C31 in compound (III), all H atoms were clearly located in difference maps. H atoms bonded to C31 in compound (III) were placed in calculated positions, and all H atoms were then treated as riding atoms in geometrically idealized positions. The H atoms bonded to three-connected C atoms in aromatic or heteroaromatic rings were placed along the external bisectors of the ring angles, with C—H distances of 0.95 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms bonded to ring N atoms were placed along the external bisectors of the ring angles, with N—H distances of 0.88 Å and with $U_{iso}(H) = 1.2U_{eq}(N)$. The methyl groups were all permitted to rotate about the adjacent C—X bonds (X = C or O) but not to tilt, with all of the X—C—H and H—C—H angles held fixed in each such group, and with C—H distances of 0.98 Å and with $U_{iso}(H) = 1.5U_{eq}(C)$. In the absence of significant resonant scattering, the Friedel-equivalent reflections were merged for compounds (III) and (IV). Hence, the absolute configuration of the molecules in (III) and the correct orientation of the structure of (IV) with respect to the polar-axis direction are both undetermined.

For all compounds, data collection: *COLLECT* (Hooft, 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: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FA3182). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Brammer, L., Bruton, E. A. & Sherwood, P. (2001). *Cryst. Growth Des.* **1**, 277–290.

Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.

Devi, I., Borah, H. N. & Bhuyan, P. J. (2004). *Tetrahedron Lett.* **45**, 2405–2408.

Devi, I., Kumar, B. S. D. & Bhuyan, P. J. (2003). *Tetrahedron Lett.* **44**, 8307–8310.

Duisenberg, A. J. M., Hooft, R. W. W., Schreurs, A. M. M. & Kroon, J. (2000). *J. Appl. Cryst.* **33**, 893–898.

Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.

Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.

Hooft, R. W. W. (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.

Naylor, R. E. & Wilson, E. B. (1957). *J. Chem. Phys.* **26**, 1057–1060.

Sarkhel, S., Srivastava, P., Saxena, A. S., Ram, V. J., Prasad, O., Shiro, M. & Maulik, P. R. (2001). *Acta Cryst.* E**57**, o408–o410.

Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Tannenbaum, E., Myers, R. J. & Gwinn, W. D. (1956). *J. Chem. Phys.* **25**, 42–47.

Thallypally, P. K. & Nangia, A. (2001). *CrystEngComm*, **3**, 114–119.

Tu, S., Li, C., Shi, F., Zhou, D., Shao, Q., Cao, L. & Jiang, B. (2008). *Synthesis*, **3**, 369–376.

Wang, X.-S., Zeng, Z.-S., Zhang, M.-M., Li, Y.-L. & Tu, S.-J. (2006). *Acta Cryst.* E**62**, o476–o477.