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Three ethyl 5-amino-1-aryl-1*H*-imidazole-4-carboxylates: hydrogen-bonded supramolecular structures in one, two and three dimensions

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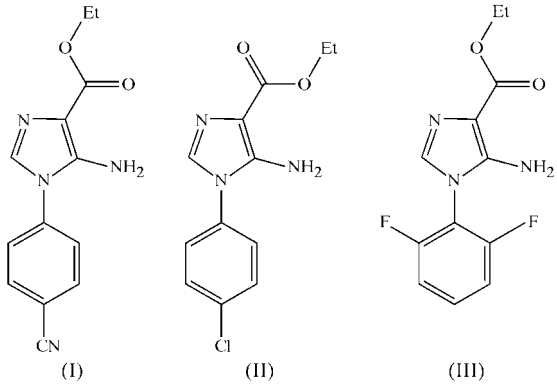
The molecules of ethyl 5-amino-1-(4-cyanophenyl)-1*H*-imidazole-4-carboxylate, $C_{13}H_{12}N_4O_2$, are linked into a chain of alternating $R_2^2(10)$ and $R_4^4(34)$ rings by a combination of N—H···N and C—H···N hydrogen bonds. In ethyl 5-amino-1-(4-chlorophenyl)-1*H*-imidazole-4-carboxylate, $C_{12}H_{12}ClN_3O_2$, where the ethyl group is disordered over two sets of sites, a combination of N—H···O, N—H···N, C—H···N and C—H···π(arene) hydrogen bonds links the molecules into complex sheets. Two intermolecular hydrogen bonds, one each of N—H···N and C—H···O types, link the molecules of ethyl 5-amino-1-(2,6-difluorophenyl)-1*H*-imidazole-4-carboxylate, $C_{12}H_{11}F_2N_3O_2$, into a continuous three-dimensional framework structure.

Comment

Imidazole rings appear frequently in biologically active compounds, both natural and man-made (ten Have *et al.*, 1997). In particular, N-substituted imidazoles (Khabnadideh *et al.*, 2003) have been found to exhibit a variety of pharmacological properties, including antiparasitic, antifungal and antimicrobial properties (Gangneux *et al.*, 1999; Gupta *et al.*, 2004; Foroumadi *et al.*, 2005). In continuation of our studies on agents having inhibitory activity against *Mycobacterium tuberculosis* and anti-leishmanicidal activity (Costa *et al.*, 2006), we have prepared a series of ethyl 5-amino-1-aryl-1*H*-imidazole-4-carboxylates, and report here the structures of three such compounds, namely ethyl 5-amino-1-(4-cyanophenyl)-1*H*-imidazole-4-carboxylate, (I) (Fig. 1), ethyl 5-amino-1-(4-chlorophenyl)-1*H*-imidazole-4-carboxylate, (II) (Fig. 2), and lastly ethyl 5-amino-1-(2,6-difluorophenyl)-1*H*-imidazole-4-carboxylate, (III) (Fig. 3), where small changes in the substituents on the aryl ring lead to significant changes in the supramolecular structures.



In each of compounds (I)–(III), the two rings are far from being coplanar; the dihedral angles between the rings are 40.4 (2), 48.0 (2) and 56.9 (2)° in (I)–(III), respectively. However, the principal point of interest in the conformations concerns the ester portion of the molecules. In each compound, there is a short intramolecular N—H···O hydrogen bond (Tables 1–3) and this may control the conformation of the carboxyl fragment, which is, in each case, almost coplanar with the imidazole ring, as shown by the torsion angles (Table 4). However, while in compounds (I) and (III) it is carbonyl atom O41 that participates in the intramolecular hydrogen bond, in compound (II) it is ethoxy atom O42. Similarly, the ethoxycarbonyl groups in compounds (I)

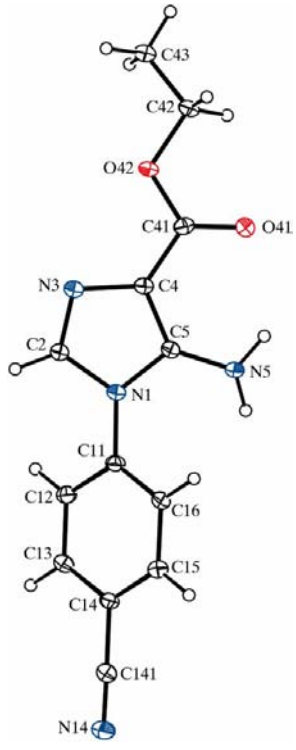


Figure 1
A molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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and (III) adopt a nearly planar conformation, while in compound (II), where this fragment is disordered over two sets of sites with equal occupancy, neither conformation of this group is even close to planarity (Table 4). Apart from the long C14—C141 bond and the short C141—N14 bond characteristic of nitriles, as found in compound (I), none of the other bond distances presents any unusual features.

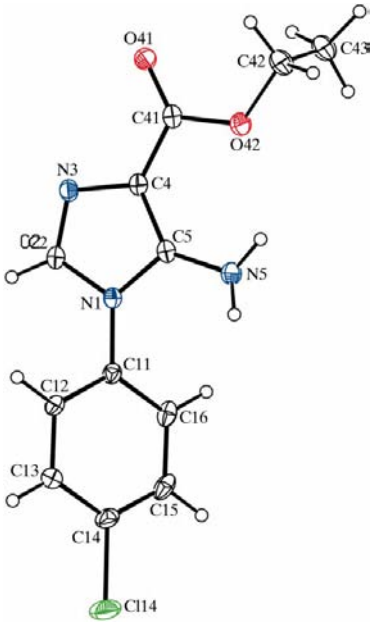

Figure 2
A molecule of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level; for the sake of clarity, only one orientation of the disordered ethyl group is shown.

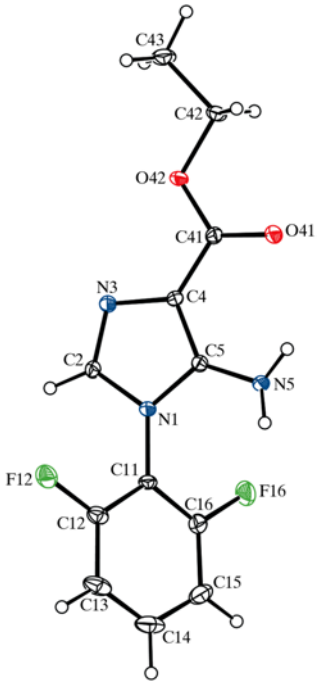

Figure 3
A molecule of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

The molecules of compound (I) are linked by a combination of N—H···N and C—H···N hydrogen bonds (Table 1) into chains of edge-fused rings. Atoms N5 and C15 in the molecule at (x, y, z) act as hydrogen-bond donors, respectively, to N3 in the molecule at $(1 + x, y, z)$ and N14 in the molecule at $(2 - x, 1 - y, -z)$, so forming a chain of edge-fused centrosymmetric rings running parallel to the [100] direction, with $R_2^2(10)$ (Bernstein *et al.*, 1995) rings centred at $(n, \frac{1}{2}, 0)$ (where n is zero or an integer) and $R_4^4(34)$ rings centred at $(n + \frac{1}{2}, \frac{1}{2}, 0)$ (where n is zero or an integer) (Fig. 4). There are no direction-specific interactions between the chains, so the supramolecular structure of compound (I) is one-dimensional.

The supramolecular structure of compound (II) takes the form of sheets generated by a combination of N—H···O, N—H···N, C—H···N and C—H···π(arene) hydrogen bonds (Table 2), and the formation of the sheet is readily analysed in terms of two distinct low-dimensional substructures. The simpler of these substructures is a finite (zero-dimensional) dimer motif; atom C12 in the molecule at (x, y, z) acts as a

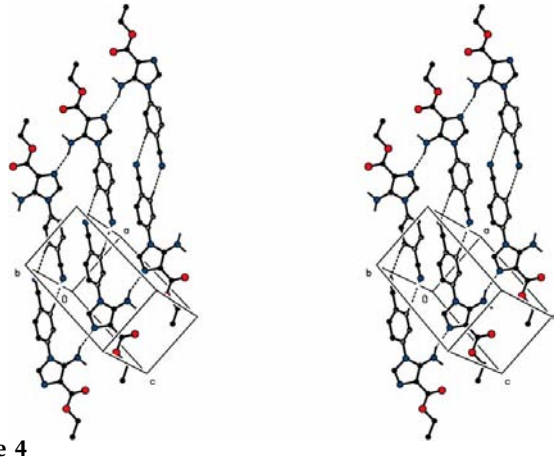

Figure 4
A stereoview of part of the crystal structure of compound (I), showing the formation of a chain of hydrogen-bonded $R_2^2(10)$ and $R_4^4(34)$ rings along [100]. For the sake of clarity, H atoms bonded to C atoms and not involved in the motifs shown have been omitted.

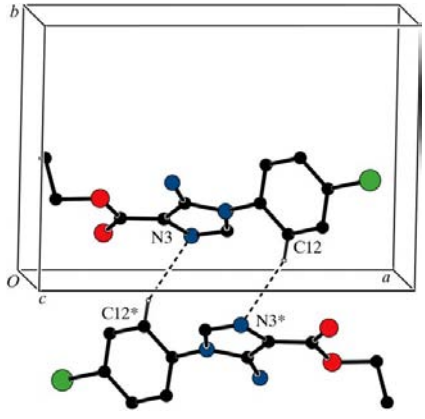

Figure 5
Part of the crystal structure of compound (II), showing the formation of an $R_2^2(12)$ dimer centred at $(\frac{1}{2}, 0, \frac{1}{2})$. For the sake of clarity, H atoms not involved in the motifs shown have been omitted, and only one orientation of the disordered ethyl group is shown. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, -y, 1 - z)$.

hydrogen-bond donor to atom N3 in the molecule at $(1 - x, -y, 1 - z)$, so generating by inversion an $R_2^2(12)$ dimer centred at $(\frac{1}{2}, 0, \frac{1}{2})$ (Fig. 5). In the second substructure, atom N5 in the molecule at (x, y, z) acts as a hydrogen-bond donor, *via* atoms H5*A* and H5*B*, respectively, to atoms O41 and N3, both in the molecule at $(x, \frac{1}{2} - y, \frac{1}{2} + z)$, so forming a $C(5)C(6)[R_2^2(7)]$ chain of rings running parallel to the [001] direction and generated by the *c*-glide plane at $y = \frac{1}{4}$. At the same time, atom C2 in the molecule at (x, y, z) acts as a hydrogen-bond donor to the C11–C16 ring in the molecule at $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, so both reinforcing and adding complexity to the [001] chain (Fig. 6). Propagation by the space group of this chain motif then directly links the dimer centred at $(\frac{1}{2}, 0, \frac{1}{2})$ to those centred at $(\frac{1}{2}, -\frac{1}{2}, 0)$, $(\frac{1}{2}, -\frac{1}{2}, 1)$, $(\frac{1}{2}, \frac{1}{2}, 0)$ and $(\frac{1}{2}, \frac{1}{2}, 1)$, so generating a rather complex sheet lying parallel to (100) (Fig. 7). However, there are no direction-specific interactions between adjacent sheets, so that the supramolecular structure of compound (II) is two-dimensional.

There are only two hydrogen bonds (Table 3) in the structure of compound (III), but as propagated by the space group they link all the molecules into a single three-dimensional framework, whose formation is readily analysed in terms of simple one-dimensional substructures. In the first substructure, atom N5 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom N3 in the molecule at $(x, 1 - y, \frac{1}{2} + z)$, so forming a $C(5)$ chain running parallel to the [001] direction and generated by the *c*-glide plane at $y = \frac{1}{2}$ (Fig. 8). The second substructure is built using the C—H···O hydrogen bond, where atom C2 in the molecule at (x, y, z) acts as donor to carbonyl atom O41 in the molecule at $(\frac{1}{2} + x, -\frac{1}{2} + y, z)$, so generating by translation a $C(6)$ chain running parallel to the $[1\bar{1}0]$ direction (Fig. 9). The action of the *c*-glide plane upon the chain along $[1\bar{1}0]$ generates an identical $C(6)$ chain, this time running parallel to the [110] direction. Successive [110] and $[1\bar{1}0]$ chains are linked by the [001] chain, and the combination of these three chain motifs is thus sufficient to generate a continuous three-dimensional structure.

Accordingly, the supramolecular structures of compounds (I), (II) and (III) are, respectively, one-, two- and three-dimensional.

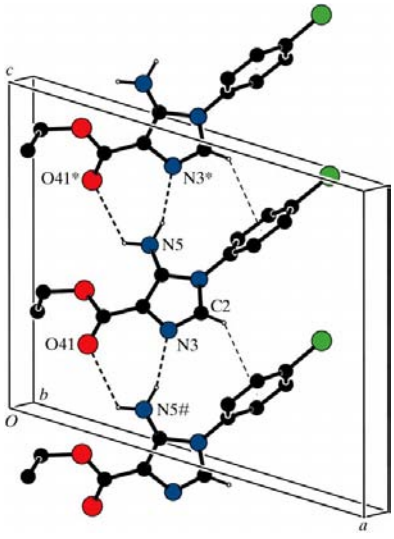

Figure 6
Part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded chain along [001]. For the sake of clarity, H atoms not involved in the motifs shown have been omitted, and only one orientation of the disordered ethyl group is shown. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, \frac{1}{2} - y, \frac{1}{2} + z)$ and $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, respectively.

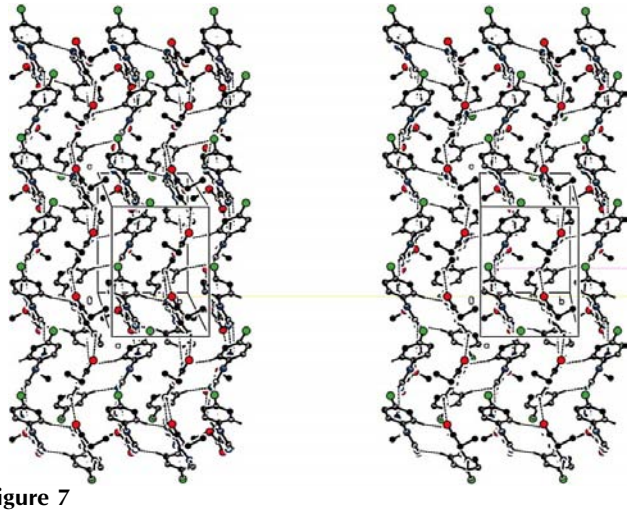

Figure 7
A stereoview of part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded sheet parallel to (100). For the sake of clarity, H atoms not involved in the motifs shown have been omitted, and only one orientation of the disordered ethyl group is shown.

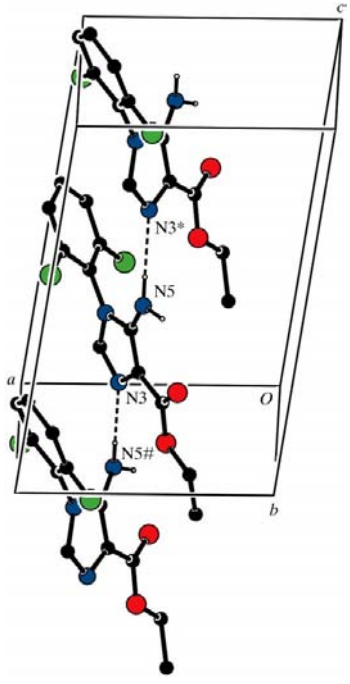

Figure 8
Part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded $C(5)$ chain along [001]. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, \frac{1}{2} - y, \frac{1}{2} + z)$ and $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, respectively.

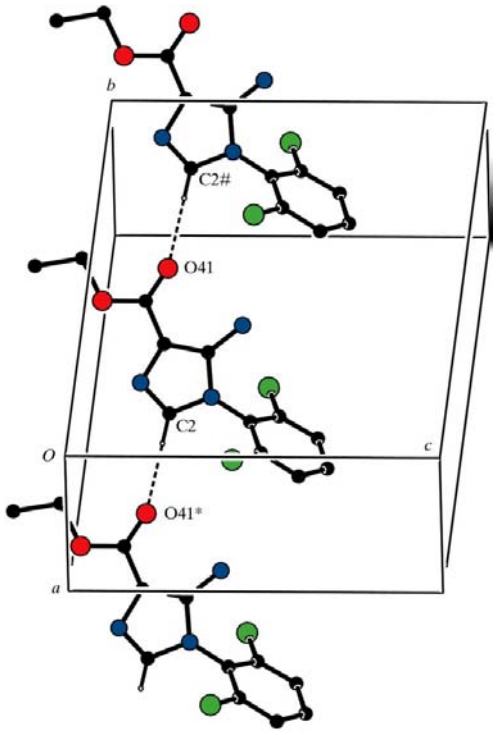

Figure 9
Part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded $C(6)$ chain along $[1\bar{1}0]$. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(\frac{1}{2}+x, -\frac{1}{2}+y, z)$ and $(-\frac{1}{2}+x, -\frac{1}{2}+y, z)$, respectively.

Experimental

The title compounds were obtained following a published procedure (Shaw *et al.*, 1980). A solution of ethyl 2-amino-2-cyanoacetate (0.0273 mol) and triethyl orthoformate (0.0273 mol) in acetonitrile (15 ml) was stirred at room temperature for 5 min and the appropriate aniline [4-cyanoaniline for (I), 4-chloroaniline for (II) and 2,6-difluoroaniline for (III)] (0.0273 mol) was then added. The reaction mixtures were heated under reflux for 4 h; after cooling the mixtures, the solid products were collected by filtration, washed with cold acetonitrile and recrystallized from ethanol to give crystals suitable for single-crystal X-ray diffraction. Compound (I): yield 65%; m.p. 516–518 K; IR (KBr disk, cm^{-1}): 3426 (NH_2), 2231 (CN), 1669 (C═O); NMR (DMSO-d_6): δ(H) 1.26 (*t*, CH_3, J = 7.0 Hz), 4.20 (*q*, CH_2, J = 7.0 Hz), 5.99 (*s*, NH_2), 7.36 (*s*, imidazole CH), 7.45 (*d*, J = 8.0 Hz), 7.76 (*d*, J = 8.0 Hz); δ(C) 14.5 (CH_3), 58.5 (CH_2), 109.6, 121.3 (CN), 127.1, 130.9, 133.4, 132.6, 145.7, 163.7 (C═O); MS (*m*/*z*, %): 256 (60, M^+), 210 [82, $(M-46)^+$], 184 [30, $(M-72)^+$], 129 [100 $(M-127)^+$], 102 [70 $(M-154)^+$]. Compound (II): yield 70%; m.p. 522–524 K; IR (KBr disk, cm^{-1}): 3437 (NH_2), 1696 (C═O); NMR (DMSO-d_6): δ(H) 1.26 (*t*, CH_3, J = 7.0 Hz), 4.20 (*q*, CH_2, J = 7.0 Hz), 6.02 (*s*, NH_2), 7.36 (*s*, imidazole CH), 7.52 (*dd*, J = 3.0 and 9.0 Hz), 7.64 (*dd*, J = 3.0 and 9.0 Hz); δ(C) 14.5 (CH_3), 58.5 (CH_2), 109.6, 126.9, 129.7, 131.0, 132.8, 133.0, 145.8, 163.7 (C═O); MS (*m*/*z*, %): 219 [82 $(M-46)^+$], 193 [30 $(M-72)^+$], 138 [100 $(M-127)^+$], 111 [76 $(M-154)^+$]. Compound (III): yield 65%; m.p. 529–531 K; IR (KBr disk, cm^{-1}): 3417 (NH_2), 1675 (C═O); NMR (DMSO-d_6): δ(H) 1.27 (*t*, CH_3, J = 6.5 Hz), 4.20 (*q*, CH_2, J = 6.0 Hz), 6.20 (*s*, NH_2), 7.32 (*s*, imidazole CH), 7.39 (*dd*, J = 8.0, 8.5 Hz), 7.66 (*dd*, J = 6.5, 7.0 Hz); δ(C) 14.5 (CH_3), 58.5 (CH_2), 108.4, 110.8 [*t*, ^2J(CF) = 17.2 Hz], 112.7 [*d*, ^2J(CF) = 19.7 Hz], 131.9 [*t*, ^3J(CF) = 10.3 Hz], 131.2, 146.9, 158.0 [*d*, ^1J(CF) = 250.1 Hz], 163.5 (C═O); MS (*m*/*z*, %): 267 (60, M^+), 221 [34 $(M-46)^+$], 202 [70 $(M-65)^+$], 195 [24 $(M-72)^+$], 140 [100 $(M-127)^+$], 113 [18 $(M-154)^+$].

Compound (I)

Crystal data

$C_{13}H_{12}N_4O_2$
M_r = 256.27
Triclinic, $P\bar{1}$
a = 6.3212 (5) Å
b = 9.4121 (11) Å
c = 10.2496 (12) Å
α = 90.311 (5)°
β = 94.183 (7)°
γ = 92.946 (7)°
V = 607.35 (11) Å^3
Z = 2
D_x = 1.401 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 120 (2) K
Needle, colourless
0.64 × 0.04 × 0.03 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.946, T_{max} = 0.997
11051 measured reflections
2688 independent reflections
1720 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 27.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.147
S = 1.04
2688 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0736P)^2 + 0.0026P]$ 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 1
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5A···O41	0.90	2.21	2.866 (2)	129
N5—H5B···N3^{i}	0.90	2.22	3.073 (2)	158
C15—H15···N14^{ii}	0.95	2.52	3.397 (3)	154

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

Compound (II)

Crystal data

$C_{12}H_{12}ClN_3O_2$
M_r = 265.70
Monoclinic, $P2_1/c$
a = 13.1153 (6) Å
b = 8.7114 (4) Å
c = 11.5273 (4) Å
β = 107.064 (3)°
V = 1259.05 (9) Å^3
Z = 4
D_x = 1.402 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 120 (2) K
Plate, colourless
0.48 × 0.32 × 0.08 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.869, T_{max} = 0.976
17524 measured reflections
2864 independent reflections
2210 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.126
S = 1.05
2864 reflections
171 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.7518P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

Cg is the centroid of the C11–C16 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···O42	0.90	2.24	2.834 (2)	123
N5—H5*A*···O41^{i}	0.90	2.51	3.071 (2)	121
N5—H5*B*···N3^{i}	0.90	2.09	2.952 (2)	161
C12—H12···N3^{ii}	0.95	2.59	3.462 (3)	153
C2—H2···*Cg*^{iii}	0.95	2.78	3.500 (2)	133

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

Compound (III)

Crystal data

$C_{12}H_{11}F_2N_3O_2$
M_r = 267.24
Monoclinic, *Cc*
a = 7.9045 (4) Å
b = 12.9950 (7) Å
c = 11.7737 (5) Å
β = 98.810 (4)°
V = 1195.11 (10) Å^3
Z = 4
D_x = 1.485 Mg m^{-3}
Mo *Kα* radiation
μ = 0.12 mm^{-1}
T = 120 (2) K
Block, colourless
0.35 × 0.17 × 0.12 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.966, T_{max} = 0.985
8086 measured reflections
1362 independent reflections
1175 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 27.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.080
S = 1.07
1362 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.2764P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···O41	0.88	2.30	2.903 (3)	125
N5—H5*B*···N3^{i}	0.88	2.07	2.946 (3)	173
C2—H2···O41^{ii}	0.95	2.23	3.175 (3)	176

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

Table 4
Selected torsion angles (°) for compounds (I)–(III).

	(I)	(II)	(III)
N3—C4—C41—O41	179.3 (2)	3.6 (3)	−177.6 (2)
N3—C4—C41—O42	−2.6 (3)	−176.39 (17)	2.2 (3)
C4—C41—O42—C42	−179.12 (18)	−171.1 (2)	−179.5 (2)
C4—C41—O42—C42*A*	–	164.8 (3)	–
C41—O42—C42—C43	161.84 (18)	−99.3 (3)	−171.2 (2)
C41—O42—C42*A*—C43*A*	–	−155.7 (4)	–

Note: in compound (II), the ethyl group is disordered over two sets of sites (see *Comment*).

Crystals of compound (I) are triclinic; the space group $P\overline{1}$ was selected and subsequently confirmed by the structure analysis. For compound (II), the space group $P2_1/c$ was uniquely assigned from the systematic absences. For compound (III), the systematic absences permitted *Cc* and *C*2/*c* as possible space groups; *Cc* was selected and confirmed by the structure analysis. All H atoms were located in difference maps and then treated as riding atoms, with C—H = 0.95 (aromatic and heteroaromatic), 0.98 (CH_3) or 0.99 Å (CH_2) and N—H = 0.88–0.90 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N). It was apparent from an early stage that the ethyl group in compound (II) was disordered over two sets of sites; refinement of the site-occupancy factors gave values which were experimentally indistinguishable from 0.5, and consequently these factors were thereafter fixed at 0.5. In the absence of significant resonant scattering, it was not possible to establish the correct orientation of the structure of compound (III) with respect to the polar-axis directions; accordingly, the Friedel equivalent reflections were merged prior to the final refinements.

For all compounds, 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 *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); 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: SK3077). Services for accessing these data are described at the back of the journal.

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