

Hydrogen-bonded structures of the isomeric compounds of quinoline with 2-chloro-5-nitrobenzoic acid, 3-chloro-2-nitrobenzoic acid, 4-chloro-2-nitrobenzoic acid and 5-chloro-2-nitrobenzoic acid

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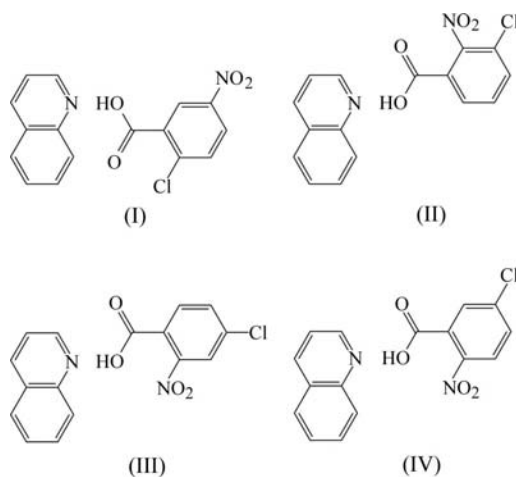
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The structures of four isomeric compounds, all $C_7H_4ClNO_4 \cdot C_9H_7N$, of quinoline with chloro- and nitro-substituted benzoic acid, namely, 2-chloro-5-nitrobenzoic acid–quinoline (1/1), (I), 3-chloro-2-nitrobenzoic acid–quinoline (1/1), (II), 4-chloro-2-nitrobenzoic acid–quinoline (1/1), (III), and 5-chloro-2-nitrobenzoic acid–quinoline (1/1), (IV), have been determined at 185 K. In each compound, a short hydrogen bond is observed between the pyridine N atom and a carboxyl O atom. The N \cdots O distances are 2.6476 (13), 2.5610 (13), 2.5569 (12) and 2.5429 (12) Å for (I), (II), (III) and (IV), respectively. Although in (I) the H atom in the hydrogen bond is located at the O site, in (II), (III) and (IV) the H atom is disordered in the hydrogen bond over two positions with (N site):(O site) occupancies of 0.39 (3):0.61 (3), 0.47 (3):0.53 (3) and 0.65 (3):0.35 (3), respectively.

Comment

Solid hydrogen-bonded compounds of 2-chloro-4-nitrobenzoic acid with amines and pyridine derivatives were studied by Kalenik *et al.* (1989), Habeeb & Awad (1995) and Awad & Habeeb (1996) using ^{35}Cl NQR and IR techniques. They reported that the hydrogen bonds formed between the acid and the bases vary from an O–H \cdots N to an O \cdots H–N type with increasing pK_a or proton affinities (PA) of the bases, and that the critical (inversion) point at 50% proton transfer, where a relatively short hydrogen bond and a broad single-minimum potential energy curve for the H atom or a double-minimum potential would be expected, exists in the compound of 3,5-dimethylpyridine ($pK_a = 6.15$ and PA = 218 kcal mol $^{-1}$). However, there is not yet any crystallographic evidence supporting these findings. Moreover, the shapes of the proton donor and acceptor molecules may be as important as the pK_a

or PA in determining the critical point. This has prompted us to carry out continuous studies on the structure of chloro- and nitro-substituted benzoic acid compounds with amines and pyridine derivatives (Ishida *et al.*, 2001a, 2002a,b,c; Ishida & Fukunaga, 2004; Gotoh & Ishida, 2007). In addition, the chloro- and nitro-substituted benzoic acid compounds with amine and pyridine systems are of interest in crystal engineering for the formation of chiral two-component molecular crystals (Koshima *et al.*, 1996; Ishida *et al.*, 2001b,c; Sugiyama *et al.*, 2002a,b). Previously, we have analysed the crystal structure of the 2-chloro-4-nitrobenzoic acid–3,5-dimethylpyridine system, but this compound consisted of a 2-chloro-4-nitrobenzoic acid molecule, a 2-chloro-4-nitrobenzoate anion and a 3,5-dimethylpyridinium cation in the asymmetric unit, and the hydrogen bond between the acid and the base was of the N–H \cdots O type (Ishida *et al.*, 2004).



In this communication, we report the four isomeric compounds 2-chloro-5-nitrobenzoic acid–quinoline (1/1), (I), 3-chloro-2-nitrobenzoic acid–quinoline (1/1), (II), 4-chloro-2-nitrobenzoic acid–quinoline (1/1), (III), and 5-chloro-2-nitrobenzoic acid–quinoline (1/1), (IV). The pK_a values are 4.90,

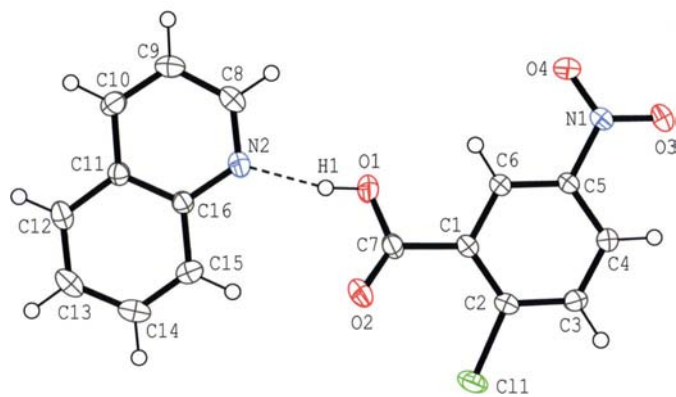


Figure 1

A molecular view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The O–H \cdots N hydrogen bond is indicated by a dashed line.

2.22, 1.82, 1.97 and 1.86, respectively, for quinoline, 2-chloro-5-nitrobenzoic acid, 3-chloro-2-nitrobenzoic acid, 4-chloro-2-nitrobenzoic acid and 5-chloro-2-nitrobenzoic acid. The ΔpK_a [$pK_a(\text{base}) - pK_a(\text{acid})$] values are 2.68, 3.08, 2.93 and 3.04, respectively, for (I), (II), (III) and (IV), which are smaller than the value of 4.18 for the 2-chloro-4-nitrobenzoic acid–3,5-dimethylpyridine system.

The molecular structures of compounds (I), (II), (III) and (IV) are shown in Figs. 1, 2, 3 and 4, respectively. The base and the acid in each compound are held together by a short hydrogen bond between the N atom of the base and the carboxyl group of the acid. In (I), no acid–base interaction is observed and the H atom in the hydrogen bond is located at the O site (Table 1). In (II), (III) and (IV), relatively short hydrogen bonds between the acid and the base are observed; the N···O distances are 2.5610 (13), 2.5569 (12) and 2.5429 (12) Å, respectively, for (II), (III) and (IV) (Tables 2–4), which are much shorter than that of 2.6476 (13) Å in (I). In the short hydrogen bonds, the H atoms are disordered. This feature is clearly shown in difference Fourier maps (Fig. 5). In (I), only one peak is observed between the acid and base, while in (II), (III) and (IV), two distinct peaks are observed,

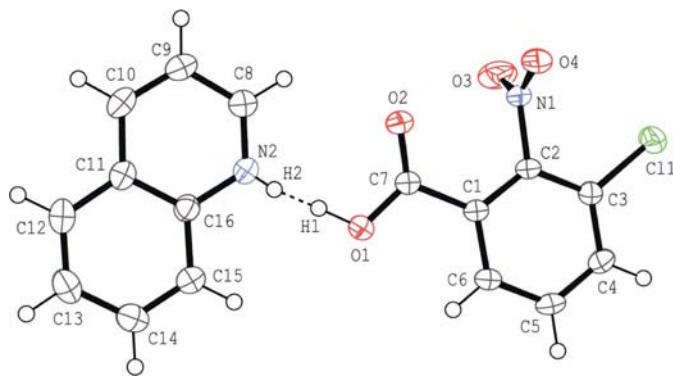


Figure 2

A molecular view of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The N···H···O hydrogen bond is indicated by a dashed line.

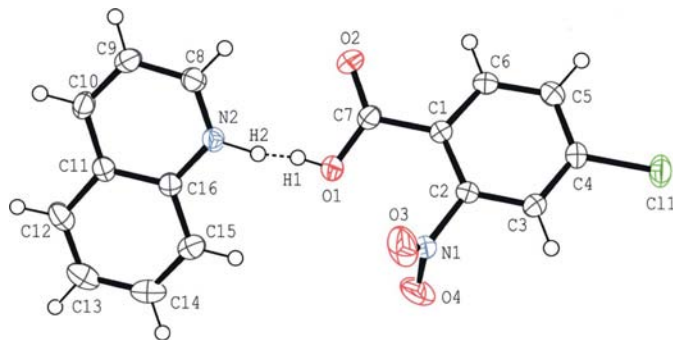


Figure 3

A molecular view of (III), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The N···H···O hydrogen bond is indicated by a dashed line.

indicating that the H atom is disordered over two positions. The occupancies of the N site and the O site refined to 0.39 (3):0.61 (3), 0.47 (3):0.53 (3) and 0.65 (3):0.35 (3), respectively, for (II), (III) and (IV). The C1–C6 benzene ring and the N2/C8–C16 quinoline system are approximately coplanar in (I) and (II), with dihedral angles of 1.92 (4) and 4.71 (5)°, respectively, between them, while in (III) and (IV), the two ring planes are twisted, with dihedral angles of 31.65 (4) and 54.43 (5)°, probably due to steric repulsion between the nitro group and the quinoline system. On the other hand, the dihedral angle between the CO₂ plane and the quinoline system in (I) is 22.48 (14)°, which is larger than the corresponding dihedral angles of 6.18 (16), 18.77 (13) and 5.41 (15)° in (II), (III) and (IV), respectively. It is of note that compound (I) differs from (II), (III) and (IV) in the orientation of the carboxyl group with respect to the quinoline system; in (I), the C7=O2 carbonyl group points to the C15–H15 group, but in (II), (III) and (IV), it points to the C8–H8 group, forming a weak C–H···O hydrogen bond (C8–H8···O2; Tables 2–4). In the

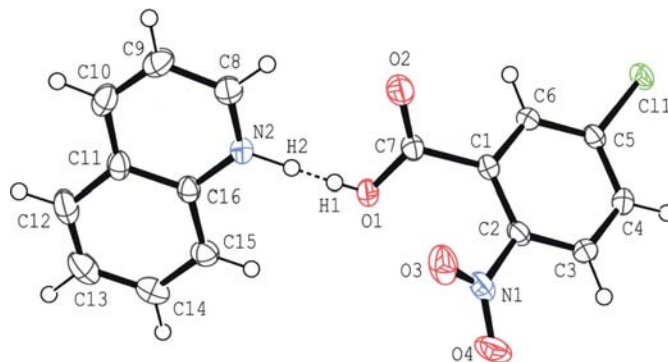


Figure 4

A molecular view of (IV), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The N···H···O hydrogen bond is indicated by a dashed line.

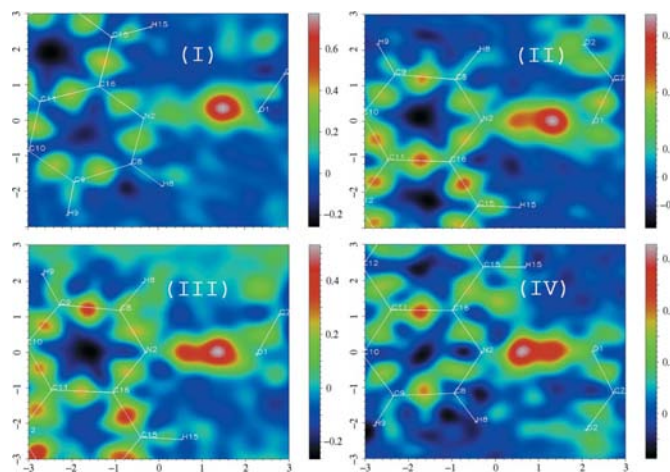


Figure 5

Difference Fourier maps of (I), (II), (III) and (IV) associated with the N···H···O hydrogen bond between the acid and the base. Maps were calculated on the O1/N2/C8/C16 mean plane from a model containing all atoms apart from the H atom in the hydrogen bond.

C10···N2···O1 angle, which may be a measure of the N···H···O hydrogen-bond strength, (I) also differs from the other compounds; the angles are 165.01 (15), 177.33 (5), 173.02 (4) and 176.76 (5)° for (I), (II), (III) and (IV), respectively.

Packing diagrams of the four compounds are given in Figs. 6–9. In (I), the acid and base units held by the hydrogen bond

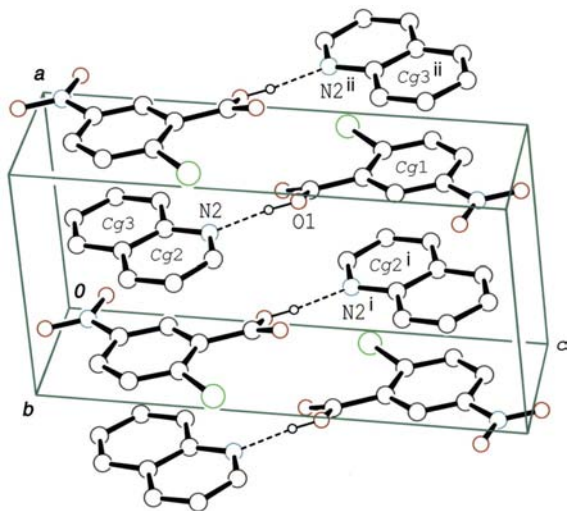


Figure 6
A packing diagram of (I), showing π – π interactions between the benzene ring and the quinoline system. Cg1, Cg2 and Cg3 are the centroids of the C1–C6, N2/C8–C11/C16 and C11–C16 rings, respectively. H atoms not involved in the O–H···N hydrogen bond have been omitted. [Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + 2, -y + 1, -z + 1$.]

are stacked in an antiparallel manner along the *a* axis through π – π stacking interactions between the C1–C6 benzene ring and the N2/C8–C16 quinoline system (Fig. 6). The centroid–centroid distances are in the range 3.6269 (8)–3.7302 (8) Å. The detailed geometries are given in Table 5. In (II), the acid and base units are linked by a C–H···O hydrogen bond (C5–H5···O2ⁱ; Table 2), forming a molecular tape running

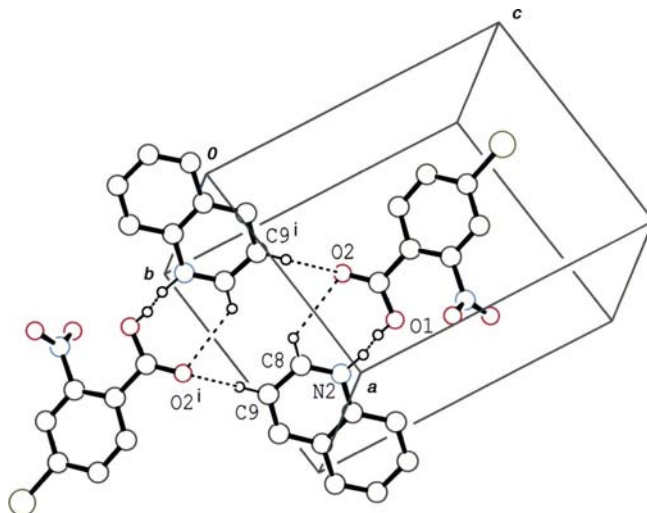


Figure 8
A packing diagram of (III), showing a centrosymmetric 2+2 aggregate. N···H···O and C–H···O hydrogen bonds are shown by dashed lines. H atoms not involved in the hydrogen bonds have been omitted. [Symmetry code: (i) $-x + 1, -y + 1, -z$.]



Figure 7
A packing diagram of (II), viewed down the *a* axis. Dashed lines show N···H···O and C–H···O hydrogen bonds, and Cl···O short contacts. H atoms not involved in the hydrogen bonds have been omitted. [Symmetry codes: (i) $x, y + 1, z$; (ii) $-x, -y + 1, -z$.]

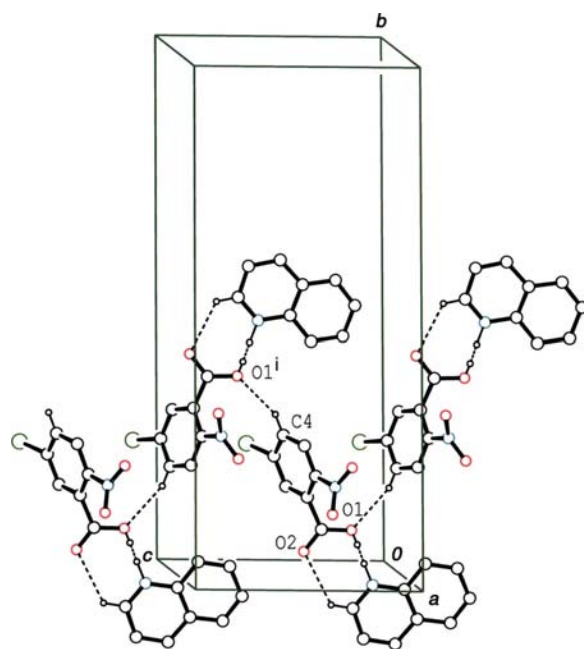


Figure 9
A packing diagram of (IV), viewed approximately down the *a* axis, showing a molecular chain running along the *c* axis. N···H···O and C–H···O hydrogen bonds are shown by dashed lines. H atoms not involved in the hydrogen bonds have been omitted. [Symmetry code: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.]

along the *b* axis (Fig. 7). A short Cl...O contact [Cl1...O4ⁱⁱ = 3.0389 (10) Å; symmetry code: (ii) $-x, -y + 1, -z$] is observed between the tapes. Furthermore, the acid and base are alternately stacked along the *a* axis through π - π stacking interactions between the benzene ring and the quinoline system (Table 5). In (III), the hydrogen-bonded acid and base units related to each other by an inversion centre are linked by a C-H...O hydrogen bond (C9-H9...O2ⁱ; Table 3), forming a centrosymmetric 2+2 aggregate (Fig. 8). Furthermore, the two components are separately stacked in columns in an anti-parallel manner along the *b* axis through π - π interactions (Table 5). In (IV), the acid molecules are connected by a C-H...O hydrogen bond (C4-H4...O1ⁱ; Table 4) into a chain running along the *c* axis (Fig. 9). A π - π interaction between the quinoline systems is observed (Table 5). A short Cl...O contact [Cl1...O4ⁱⁱ = 2.9953 (12) Å; symmetry code: (ii) $x - 1, -y + \frac{1}{2}, z + \frac{1}{2}$] is also present between the chains.

In this communication, we show that compounds (II), (III) and (IV) have a short double-well N...H...O hydrogen bond. Although these compounds have ΔpK_a values of 2.93–3.08, larger than that of 2.68 in (I), more systematic structural studies are required to confirm whether this ΔpK_a range is appropriate to determine the critical point in the chloro- and nitro-substituted benzoic acid–pyridine systems.

Experimental

Crystals of all four title compounds were obtained by slow evaporation from acetonitrile solutions of quinoline with the corresponding chloro- and nitro-substituted benzoic acid in a 1:1 molar ratio at room temperature [150 ml acetonitrile solution of quinoline (0.455 g) and 2-chloro-5-nitrobenzoic acid (0.709 g) for (I), 30 ml solution of quinoline (0.204 g) and 3-chloro-2-nitrobenzoic acid (0.318 g) for (II), 20 ml solution of quinoline (0.205 g) and 4-chloro-2-nitrobenzoic acid (0.320 g) for (III), and 30 ml solution of quinoline (0.288 g) and 5-chloro-2-nitrobenzoic acid (0.449 g) for (IV)].

Compound (I)

Crystal data

C ₇ H ₄ ClNO ₄ ·C ₉ H ₇ N	$\gamma = 79.842 (4)^\circ$
$M_r = 330.73$	$V = 706.71 (17) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 6.9254 (10) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 7.4746 (11) \text{ \AA}$	$\mu = 0.29 \text{ mm}^{-1}$
$c = 14.310 (2) \text{ \AA}$	$T = 185 \text{ K}$
$\alpha = 75.861 (4)^\circ$	$0.33 \times 0.28 \times 0.20 \text{ mm}$
$\beta = 89.207 (3)^\circ$	

Data collection

Rigaku R-Axis RAPIDII diffractometer	14562 measured reflections
Absorption correction: numerical (ABSCOR; Higashi, 1995)	4109 independent reflections
$T_{\min} = 0.932, T_{\max} = 0.943$	3703 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.096$	$\Delta\rho_{\text{max}} = 0.44 \text{ e \AA}^{-3}$
$S = 1.06$	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
4109 reflections	
212 parameters	

Table 1
Hydrogen-bond geometry (Å, °) for (I).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...N2	0.85 (2)	1.81 (2)	2.6476 (13)	166 (2)

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

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...N2	0.93 (3)	1.63 (3)	2.5610 (13)	176 (3)
N2—H2...O1	0.68 (4)	1.89 (4)	2.5610 (13)	169 (5)
C5—H5...O2 ⁱ	0.95	2.41	3.3408 (15)	167
C8—H8...O2	0.95	2.44	3.1247 (17)	129

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

Compound (II)

Crystal data

C ₇ H _{3.61} ClNO ₄ ·C ₉ H _{7.39} N	$\gamma = 85.3277 (19)^\circ$
$M_r = 330.73$	$V = 720.94 (5) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.6022 (3) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 7.6251 (4) \text{ \AA}$	$\mu = 0.29 \text{ mm}^{-1}$
$c = 12.9978 (5) \text{ \AA}$	$T = 185 \text{ K}$
$\alpha = 82.300 (2)^\circ$	$0.37 \times 0.20 \times 0.16 \text{ mm}$
$\beta = 75.1792 (12)^\circ$	

Data collection

Rigaku R-Axis RAPID diffractometer	18189 measured reflections
Absorption correction: numerical (ABSCOR; Higashi, 1995)	4204 independent reflections
$T_{\min} = 0.932, T_{\max} = 0.955$	3577 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.106$	$\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
$S = 1.06$	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
4204 reflections	
215 parameters	

Compound (III)

Crystal data

C ₇ H _{3.53} ClNO ₄ ·C ₉ H _{7.47} N	$\gamma = 78.671 (3)^\circ$
$M_r = 330.73$	$V = 738.06 (11) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.9684 (8) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 8.6178 (7) \text{ \AA}$	$\mu = 0.28 \text{ mm}^{-1}$
$c = 11.1202 (9) \text{ \AA}$	$T = 185 \text{ K}$
$\alpha = 88.959 (2)^\circ$	$0.45 \times 0.45 \times 0.35 \text{ mm}$
$\beta = 80.348 (3)^\circ$	

Data collection

Rigaku R-Axis RAPID diffractometer	11936 measured reflections
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	4237 independent reflections
$T_{\min} = 0.804, T_{\max} = 0.906$	3681 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.032$

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

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...N2	0.75 (4)	1.81 (4)	2.5569 (12)	175 (3)
N2—H2...O1	0.97 (3)	1.59 (3)	2.5569 (12)	173 (3)
C8—H8...O2	0.95	2.59	3.2445 (15)	126
C9—H9...O2 ⁱ	0.95	2.52	3.3527 (16)	146

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

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.106$
 $S = 1.08$
 4237 reflections
 215 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.42 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$

Compound (IV)

Crystal data

$\text{C}_7\text{H}_{3.35}\text{ClNO}_4 \cdot \text{C}_9\text{H}_{7.65}\text{N}$
 $M_r = 330.73$
 Monoclinic, $P2_1/c$
 $a = 7.0456 (4) \text{ \AA}$
 $b = 22.6829 (13) \text{ \AA}$
 $c = 9.6273 (6) \text{ \AA}$
 $\beta = 106.3182 (18)^\circ$

$V = 1476.61 (15) \text{ \AA}^3$
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.28 \text{ mm}^{-1}$
 $T = 185 \text{ K}$
 $0.33 \times 0.25 \times 0.16 \text{ mm}$

Data collection

Rigaku R-AXIS RAPIDII diffractometer
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.831, T_{\max} = 0.956$

19004 measured reflections
 4277 independent reflections
 3709 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.093$
 $S = 1.03$
 4277 reflections
 215 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.44 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$

For (I), the H atom of the carboxyl group was found in a difference Fourier map and was refined isotropically; the refined distance is given in Table 1. For (II), (III) and (IV), H atoms in the $\text{N} \cdots \text{H} \cdots \text{O}$ hydrogen bonds were found to be disordered over two positions in difference Fourier maps. Since the site-occupancy factors and isotropic displacement parameters were strongly correlated, the positional parameters and occupancy factors were refined, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N or O})$; the refined distances are given in Tables 2, 3 and 4. Other H atoms of all compounds were positioned geometrically ($\text{C—H} = 0.95 \text{ \AA}$) and treated as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

For all compounds, data collection: *PROCESS-AUTO* (Rigaku/MSK, 2004); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *WinGX* (Farrugia, 1999); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2009).

Table 4
Hydrogen-bond geometry (Å, °) for (IV).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...N2	0.67 (5)	1.88 (5)	2.5429 (12)	177 (6)
N2—H2...O1	1.01 (3)	1.54 (3)	2.5429 (13)	177 (3)
C4—H4...O1 ⁱ	0.95	2.41	3.2748 (14)	151
C8—H8...O2	0.95	2.56	3.2165 (15)	127

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

Table 5

Geometries (Å, °) of π - π stacking interactions for compounds (I)–(IV).

$CgI \cdots CgJ$, α , CgI -Perp, CgJ -Perp and Slippage are the centroid-centroid distance between rings I and J (Å), the inter-ring dihedral angle (°), the perpendicular distance of CgI from the plane of ring J (Å), the perpendicular distance of CgJ from the plane of ring I (Å) and the slippage distance (Å), respectively. $Cg1$, $Cg2$ and $Cg3$ are the centroids of the C1–C6, N2/C8–C11/C16 and C11–C16 rings, respectively.

	$CgI \cdots CgJ$	α	CgI -Perp	CgJ -Perp	Slippage
(I)					
$Cg1 \cdots Cg2^i$	3.6560 (8)	2.07 (5)	3.4271 (4)	3.4283 (4)	
$Cg1 \cdots Cg2^{ii}$	3.6851 (8)	2.07 (5)	3.44488 (4)	3.4722 (4)	
$Cg1 \cdots Cg3^i$	3.7302 (8)	1.75 (5)	3.4319 (4)	3.4669 (4)	
$Cg1 \cdots Cg3^{ii}$	3.6269 (8)	1.75 (5)	3.4485 (4)	3.4336 (4)	
(II)					
$Cg1 \cdots Cg2^{iii}$	3.7950 (7)	5.73 (6)	3.3985 (5)	3.3506 (5)	
$Cg1 \cdots Cg3^i$	3.6881 (7)	3.87 (6)	3.4523 (5)	3.5279 (5)	
(III)					
$Cg1 \cdots Cg1^{iv}$	3.7241 (7)	0	3.5065 (4)	3.5066 (4)	1.254
$Cg2 \cdots Cg3^v$	3.6697 (7)	0.75 (5)	3.3383 (4)	3.3225 (5)	
(IV)					
$Cg3 \cdots Cg3^{vi}$	3.8506 (8)	0	3.4475 (5)	3.4475 (5)	1.715

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

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD3308). Services for accessing these data are described at the back of the journal.

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