organic compounds

Two- and three-dimensional hydrogen-bonded structures in the 1:1 proton-transfer compounds of 4,5-dichlorophthalic acid with the isomeric monoaminobenzoic acids

Graham Smith,[a]* Urs D. Wermuth[b] and Jonathan M. White[c]

[a]School of Physical and Chemical Sciences, Queensland University of Technology, GPO Box 2434, Brisbane, Queensland 4001, Australia, [b]School of Biomolecular and Physical Sciences, Griffith University, Nathan, Queensland 4111, Australia, and [c]BIO-21 Molecular Science and Biotechnology, University of Melbourne, Parkville, Victoria 3052, Australia

Correspondence e-mail: g.smith@qut.edu.au

The crystal structures of the 1:1 proton-transfer compounds of 4,5-dichlorophthalic acid with the three isomeric mono-aminobenzoic acids, namely the hydrate 2-carboxyanilinium 2-carboxy-4,5-dichlorobenzoate dihydrate, $C_7H_8NO_2^+ \cdot C_8H_3Cl_2O_4^- \cdot 2H_2O$, (I), and the anhydrous salts 3-carboxyanilinium 2-carboxy-4,5-dichlorobenzoate, $C_7H_8NO_2^+ \cdot C_8H_3Cl_2O_4^-$, (II), and 4-carboxyanilinium 2-carboxy-4,5-dichlorobenzoate, $C_7H_8NO_2^+ \cdot C_8H_3Cl_2O_4^-$, (III), have been determined at 130 K. Compound (I) has a two-dimensional hydrogen-bonded sheet structure, while (II) and (III) are three-dimensional. All three compounds feature sheet substructures formed through anilinium $N^+-H \cdots O_{carboxyl}$ and anion carboxylic acid $O-H \cdots O_{carboxyl}$ interactions and, in the case of (I), additionally linked through the donor and acceptor associations of the solvent water molecules. However, (II) and (III) have additional lateral extensions of these substructures though cyclic $R_2^2(8)$ associations involving the carboxylic acid groups of the cations. Also, (II) and (III) have cation–anion π–π aromatic ring interactions. This work provides further examples illustrating the regular formation of network substructures in the 1:1 proton-transfer salts of 4,5-dichlorophthalic acid with the bifunctional aromatic amines.

Comment

The known crystal structures of the anhydrous 1:1 proton-transfer compounds of 4,5-dichlorophthalic acid (DCPA) with nitrogen Lewis bases show a number of structural types. With the difunctional bases brucine (Smith *et al.*, 2007) and 1,10-phenanthroline (Smith *et al.*, 2008b), discrete cation–anion $N^+-H \cdots O_{carboxyl}$ hydrogen-bonded units are found. With
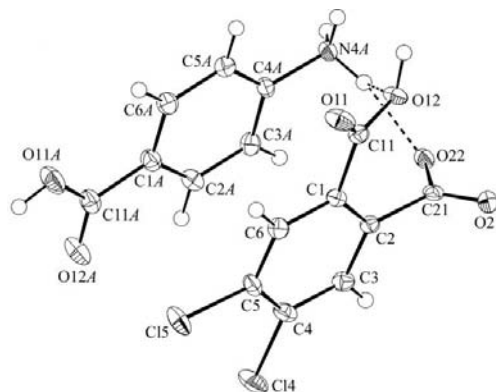
2-aminopyrimidine (Smith *et al.*, 2008b), discrete duplex cyclic hydrogen-bonded cation–anion units are found, while with the similar associative functional group-substituted but bicyclic heteroaromatic bases 8-hydroxyquinoline and 8-amino-quinoline (Smith *et al.*, 2008a), the primary cation–anion units are extended into one-dimensional hydrogen-bonded chains through linear associations involving the second functional group of the cation species. In all of these examples, the DCPA anion species are essentially planar, having a short intra-molecular $O-H \cdots O$ hydrogen bond (*ca* 1.4 Å) between the *syn*-related carboxylic acid H atom and the carboxyl group. This is also the case in the naphthalene-1,8-bis(dimethyl-ammonium) salt of DCPA (Mallinson *et al.*, 2003) and in the tetramethylammonium salt (Bozkurt *et al.*, 2006), although in these compounds the structure is three-dimensional. However, in the unusual 1:1:1 adduct hydrate compound of DCPA with quinaldic acid (Smith *et al.*, 2008a), the cation–anion units are extended into a two-dimensional sheet structure by linear $S(n)$ (n = 6 or 7) (Etter *et al.*, 1990) DCPA anion carboxylic acid $O-H \cdots O_{carboxyl}$ interactions. In these, the carboxylic acid and carboxylate groups of the DCPA anion are, of necessity, non-coplanar with the parent benzene ring. Similar two-dimensional sheet substructures are also found in the proton-transfer Lewis base salts of the nitro-substituted hydrogen phthalates and hydrogen isophthalates (Glidewell *et al.*, 2003, 2005; Smith, Wermuth *et al.*, 2008). The DCPA anion is similarly nonplanar in bis(2-ethylanilinium) 4,5-dichlorophthalate (Büyükgüngör & Odabaşoğlu, 2007), but this involves DCPA as a dianion.
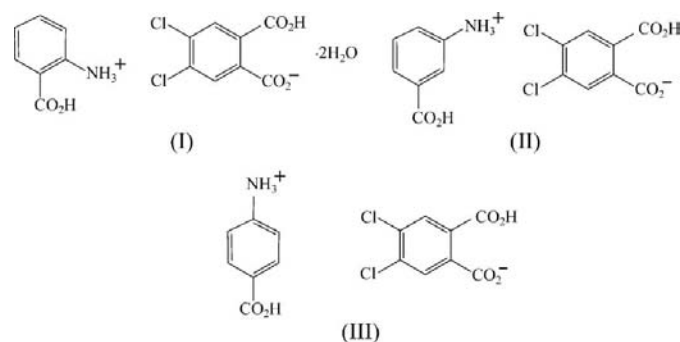
It has been found that, with the monoamino-substituted benzoic acids, three-dimensional framework structures are predominant in salts with strong aromatic carboxylic or sulfonic acids, such as 3,5-dinitrosalicylic acid (Smith *et al.*, 1995a), pyrazine-2,3-dicarboxylic acid (Smith *et al.*, 1995b) and 5-sulfosalicylic acid (Smith, 2005). Common also in the structures of the aromatic Lewis base salts of these acids are π–π associations between alternating cation and anion ring systems. It was therefore of interest to observe the effect of combining the potential chain- and sheet-forming properties of the carboxylic acid and carboxylate groups of the DCPA anion with the structure-extending features of both the aminium and carboxylic acid groups of the protonated monoamino-substituted benzoic acids, such as are found in the series reported here.
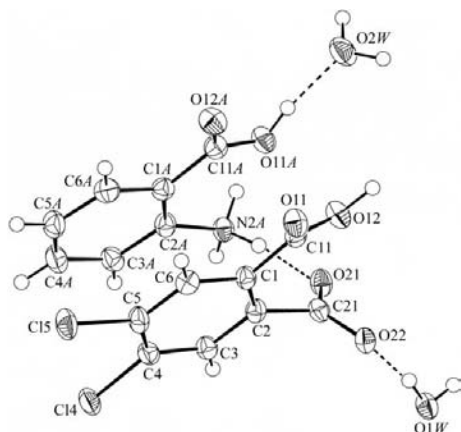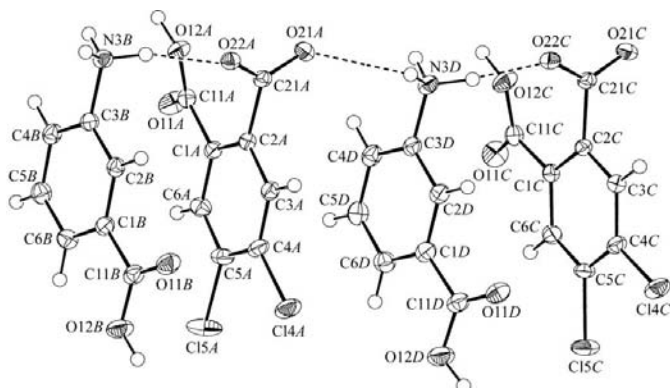
Figure 1
The molecular configuration and atom-numbering scheme for the 2-carboxyanilinium cation, the 2-carboxy-4,5-dichlorobenzoate anion and the two solvent water molecules of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Inter-species hydrogen bonds are shown as dashed lines.

Figure 2
The molecular configuration and atom-numbering scheme for the two 3-carboxyanilinium cations (B and D) and the two 2-carboxy-4,5-dichlorobenzoate anions (A and C) in the asymmetric unit of (II). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate inter-species hydrogen bonds.

The products from the 1:1 stoichiometric reaction of DCPA with the three isomeric aminobenzoic acids, namely 2-carboxyanilinium 2-carboxy-4,5-dichlorobenzoate dihydrate, (I), 3-carboxyanilinium 2-carboxy-4,5-dichlorobenzoate, (II), and 4-carboxyanilinium 2-carboxy-4,5-dichlorobenzoate, (III), were expected to have higher-dimensional hydrogen-bonded structures. However, this was not the case in all three compounds, with the structure of the dihydrate, (I), found to be two-dimensional and significantly different from the three-dimensional structures of (II) and (III). All three compounds have at least one direct N^+—H$\cdots$O$_{carboxyl}$ hydrogen-bond interaction (Figs. 1–3); in the case of (III), this is a three-centre cyclic $R_1^2(7)$ N—H$\cdots$O,O association involving carboxylic acid and carboxylate O atoms. Additionally, in (I), the two solvent water molecules act as double donors and acceptors in the extension of the structure into a two-dimensional sheet (Fig. 4). This involves carboxylic acid O—

Figure 3
The molecular configuration and atom-numbering scheme for the 4-carboxyanilinium cation and 2-carboxy-4,5-dichlorobenzoate anion in (III). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The dashed line indicates the inter-species hydrogen bond.
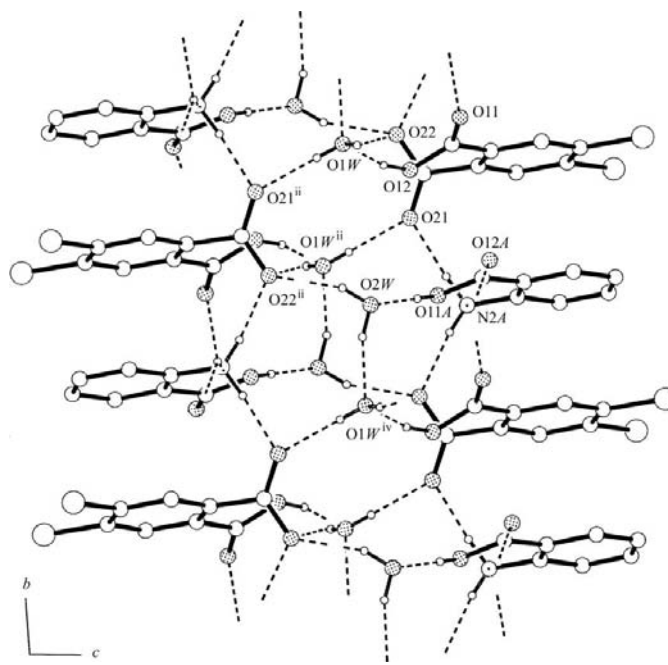
H$\cdots$O$_{water}$, water O—H$\cdots$O$_{carboxyl}$ and water O—H$\cdots$O$_{water}$ associations.

Although the anhydrous compounds, (II) and (III), are structurally similar, (III) has a single cation–anion pair in the asymmetric unit (Fig. 3), whereas that of (II) comprises two independent but conformationally identical 2-carboxyanilinium cations (B and D) and two DCPA monoanions (A and C) (Fig. 2). In each compound, the substructures comprise a network of glide-related DCPA anions linked by common phthalate–carboxylic acid O—H$\cdots$O$_{carboxyl}$ hydrogen bonds, graph set $S(7)$ [for (II), O12A—H$\cdots$O22A^i and O12C—

Figure 4
Hydrogen bonding in the two-dimensional sheet structure in (I), viewed down the b axial direction, at x = 0. H atoms not involved in the hydrogen bonding have been omitted. Hydrogen bonds are shown as dashed lines. For symmetry codes, see Table 1.
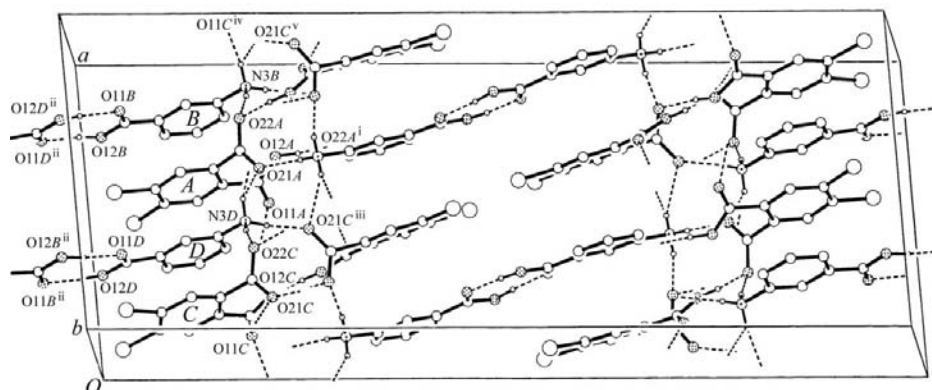
Figure 5
The two-dimensional hydrogen-bonded cation–anion sheet structure in (II), viewed down the b axial direction, showing the cyclic inversion-related $R_2^2(8)$ carboxylic acid dimeric links across c. H atoms not involved in the hydrogen bonding have been omitted. Hydrogen bonds are shown as dashed lines. For symmetry codes, see Table 2.
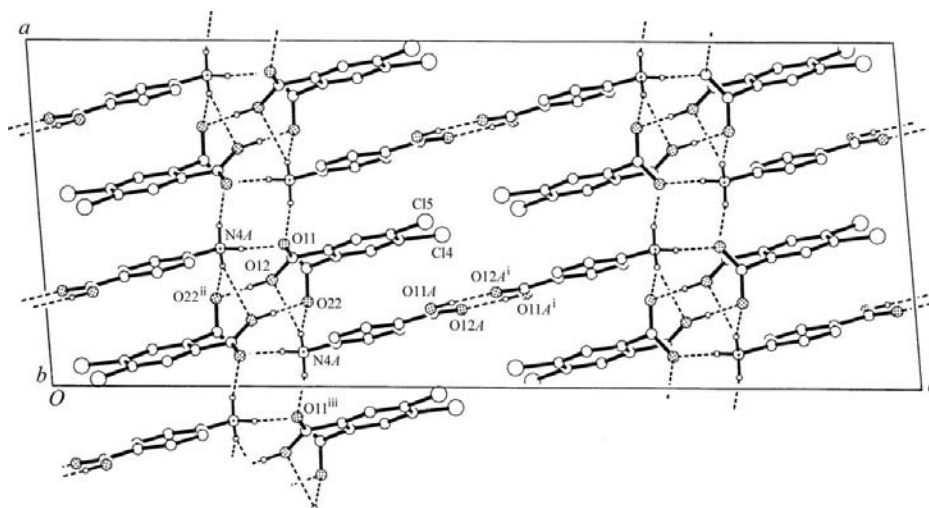

Figure 6
A view of the $C(6)$ chain extension in the two-dimensional hydrogen-bonded sheet structure of (III), viewed down the b cell direction. H atoms not involved in the hydrogen bonding have been omitted. Hydrogen bonds are shown as dashed lines. For symmetry codes, see Table 3.

H$\cdots$O22C^{iii} (for symmetry codes, see Table 2); for (III), O12—H$\cdots$O22^{ii} (for symmetry code, see Table 3)]. The associated anilinium cations alternate with the DCPA anions down the a cell directions in the respective unit cells, giving sheet substructures which extend across ab (Figs. 5 and 6).

The cation and anion aromatic rings are π–π associated. For (II), the ring centroid separation ($Cg\cdots Cg$) and inter-ring dihedral angle (α) for the planes C1B–C6B and C1A–C6A are 3.6357 (9) Å and 3.15 (1)°, respectively, while for the planes C1D–C6D and C1C–C6C, $Cg\cdots Cg$ = 3.5261 (9) Å and α = 3.81 (1)°. For (III), for the planes C1–C6 and C1A–C6A, $Cg\cdots Cg$ = 3.5867 (11) Å and α = 2.76 (1)°. This feature is absent in the structure of (I).

The three-dimensional structures in (II) and (III) are generated by classical (Leiserowitz, 1976) centrosymmetric head-to-head cyclic $R_2^2(8)$ associations through the carboxylic acid groups of the anilinium cations, which extend across the c axial directions in each. Also present in (II) and (III) but not in (I) are short intermolecular inversion-related Cl$\cdots$Cl interactions: in (II), Cl4$A\cdots$Cl4A^{ii} = 3.3243 (6) Å and

Cl5$A\cdots$Cl5A^{vii} = 3.2295 Å [symmetry code: (vii) $-x$, $-y + 1$, $-z$], and in (III), Cl4$\cdots$Cl4^{v} = 3.2488 (7) Å [symmetry code: (v) $-x + 1$, $-y$, $-z + 1$]. Similarly, there are unusual short intermolecular Cl$\cdots$O$_{carboxyl}$ interactions: in (II), Cl5$A\cdots$O11D^{viii} = 3.2279 (14) Å [symmetry code: (viii) x, $y - 1$, z], and in (III), Cl4$\cdots$O12A^{vi} = 3.2213 (15) Å [symmetry code: (vi) $-x + \frac{1}{2}$, $-y + \frac{1}{2}$, $-z + 1$].

Unlike the anion species found in the majority of the known structures of 1:1 acid salts of DCPA with aromatic Lewis bases, in which the presence of short intramolecular carboxylic acid O—H$\cdots$O$_{carboxyl}$ hydrogen bonds keeps the anions essentially planar, in compounds (I)–(III) this is not the case. The anion species in all three compounds are nonplanar, with the two carboxyl groups rotated out of the plane of the benzene ring. The torsion angles associated with these groups, $viz.$ C2—C1—C11—O11 and C1—C2—C21—O22, are, respectively, for (I), -157.0 (2) and 70.7 (3)°; for (II), -24.0 (2) and -79.61 (19)° (anion A), and -15.9 (2) and -86.93 (19)° (anion C); for (III), 20.0 (2) and 77.7 (2)°. The carboxylic acid substituent groups of the aminium cations in all three compounds are essentially

coplanar with the aromatic rings: the C2—C1—C11—O12 torsion angle for (I) is −173.2 (2)°; for (II) 178.83 (14) (cation *B*) and 176.65 (15)° (cation *D*); for (III) −179.66 (17)°.

This present series provides further examples which demonstrate the utility of the hydrogen 4,5-dichlorophalate anion species in the stabilization of 1:1 hydrogen-bonded proton-transfer structures with aromatic Lewis base cations. In all three cases, the less common nonplanar DCPA anion type is observed, which gives structure extension in the anhydrous compounds through carboxylic acid–carboxylate hydrogen-bonding interactions and two-dimensional substructures. The secondary associative carboxylic acid substituent groups of the cations are also important in the formation of higher-order structure types.

Experimental

Compounds (I)–(III) were synthesized by heating together, for 10 min under reflux, 1 mmol quantities of 4,5-dichlorophthalic acid and, respectively, 2-amino-, 3-amino- or 4-aminobenzoic acid in 50 ml of 50% ethanol–water for (I) or 80% methanol–water for (II) and (III). Compound (I) was obtained as large colourless needles (m.p. 437–438 K), (II) as pale-brown blocks (m.p. >573 K) and (III) as colourless blocks (m.p. >573 K) after partial room-temperature evaporation of the solvents.

Compound (I)

Crystal data

$C_7H_8NO_2{}^+ \cdot C_8H_3Cl_2O_4{}^- \cdot 2H_2O$	$\gamma = 86.190\ (6)°$
$M_r = 408.18$	$V = 843.37\ (10)\ \text{Å}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 6.7684\ (5)\ \text{Å}$	Cu $K\alpha$ radiation
$b = 6.9561\ (4)\ \text{Å}$	$\mu = 3.90\ \text{mm}^{-1}$
$c = 18.0001\ (14)\ \text{Å}$	$T = 180\ (2)\ \text{K}$
$\alpha = 86.525\ (6)°$	$0.40 \times 0.26 \times 0.14\ \text{mm}$
$\beta = 87.481\ (6)°$	

Data collection

Oxford Diffraction Gemini S Ultra CCD area-detector diffractometer	7458 measured reflections
	3190 independent reflections
	2691 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{min} = 0.260$, $T_{max} = 0.580$	$R_{int} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.157$	
$S = 1.09$	
3190 reflections	$\Delta\rho_{max} = 0.42\ \text{e Å}^{-3}$
271 parameters	$\Delta\rho_{min} = -0.48\ \text{e Å}^{-3}$

Compound (II)

Crystal data

$C_7H_8NO_2{}^+ \cdot C_8H_3Cl_2O_4{}^-$	$V = 3064.4\ (3)\ \text{Å}^3$
$M_r = 372.15$	$Z = 8$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 12.9028\ (7)\ \text{Å}$	$\mu = 0.46\ \text{mm}^{-1}$
$b = 7.4091\ (4)\ \text{Å}$	$T = 130\ (2)\ \text{K}$
$c = 32.2323\ (17)\ \text{Å}$	$0.50 \times 0.40 \times 0.35\ \text{mm}$
$\beta = 96.012\ (1)°$	

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O11*A*—H11*A*$\cdots$O2*W*	0.88 (5)	1.70 (5)	2.574 (3)	171 (5)
O12—H12$\cdots$O1*W*^{i}	0.90 (5)	1.67 (5)	2.562 (3)	179 (7)
O1*W*—H11*W*$\cdots$O22	0.85 (5)	1.88 (4)	2.708 (2)	167 (4)
O1*W*—H12*W*$\cdots$O21^{ii}	0.78 (5)	1.90 (5)	2.679 (2)	176 (5)
O2*W*—H21*W*$\cdots$O22^{iii}	0.87 (5)	2.09 (5)	2.875 (3)	150 (4)
O2*W*—H22*W*$\cdots$O1*W*^{iv}	0.90 (4)	1.92 (4)	2.789 (3)	163 (3)
N2*A*—H21*A*$\cdots$O21	0.94 (3)	1.80 (3)	2.742 (3)	174 (3)
N2*A*—H22*A*$\cdots$O11^{v}	0.85 (3)	2.26 (3)	2.938 (3)	137 (3)
N2*A*—H22*A*$\cdots$O12*A*^{vi}	0.85 (3)	2.37 (3)	3.026 (3)	135 (2)
N2*A*—H23*A*$\cdots$O22^{vii}	0.93 (3)	1.90 (3)	2.808 (3)	163 (3)

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

Data collection

Bruker SMART CCD area-detector diffractometer	17962 measured reflections
	6995 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{min} = 0.74$, $T_{max} = 0.85$	6125 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.104$	
$S = 1.06$	
6995 reflections	$\Delta\rho_{max} = 0.45\ \text{e Å}^{-3}$
474 parameters	$\Delta\rho_{min} = -0.41\ \text{e Å}^{-3}$

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O12*A*—H12*A*$\cdots$O22*A*^{i}	0.98 (3)	1.50 (3)	2.4730 (16)	176 (3)
O12*B*—H12*B*$\cdots$O11*D*^{ii}	0.96 (3)	1.67 (3)	2.6216 (18)	176 (4)
O12*C*—H12*C*$\cdots$O22*C*^{iii}	0.99 (3)	1.51 (3)	2.4714 (16)	163 (3)
O12*D*—H12*D*$\cdots$O11*B*^{ii}	0.92 (3)	1.76 (3)	2.6718 (18)	176 (3)
N3*B*—H31*B*$\cdots$O11*C*^{iv}	0.92 (2)	1.95 (2)	2.8378 (19)	160 (2)
N3*B*—H31*B*$\cdots$O21*C*^{v}	0.92 (2)	2.54 (2)	2.9778 (18)	109.6 (16)
N3*D*—H31*D*$\cdots$O11*A*^{vi}	0.90 (2)	2.09 (2)	2.9097 (19)	150.8 (18)
N3*D*—H31*D*$\cdots$O21*A*	0.90 (2)	2.41 (2)	2.8919 (19)	113.9 (17)
N3*B*—H32*B*$\cdots$O21*A*^{i}	0.86 (2)	1.94 (2)	2.7977 (19)	176 (2)
N3*D*—H32*D*$\cdots$O22*C*	0.99 (2)	1.77 (2)	2.7454 (19)	172 (2)
N3*B*—H33*B*$\cdots$O22*A*	0.96 (2)	1.89 (2)	2.8416 (18)	169 (2)
N3*D*—H33*D*$\cdots$O21*C*^{iii}	0.92 (2)	1.80 (2)	2.7176 (17)	174 (2)

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

Compound (III)

Crystal data

$C_7H_8NO_2{}^+ \cdot C_8H_3Cl_2O_4{}^-$	$V = 3068.1\ (5)\ \text{Å}^3$
$M_r = 372.15$	$Z = 8$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 12.8552\ (12)\ \text{Å}$	$\mu = 0.46\ \text{mm}^{-1}$
$b = 7.5003\ (7)\ \text{Å}$	$T = 130\ (2)\ \text{K}$
$c = 31.932\ (3)\ \text{Å}$	$0.25 \times 0.20 \times 0.15\ \text{mm}$
$\beta = 94.785\ (2)°$	

Data collection

Bruker SMART CCD area-detector diffractometer	9325 measured reflections
	3467 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{min} = 0.81$, $T_{max} = 0.93$	2929 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.031$

organic compounds

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.086$
$S = 1.50$
3467 reflections
237 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.31$ e Å⁻³
$\Delta\rho_{min} = -0.32$ e Å⁻³

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O11A—H11$A\cdots$O12A^i	0.86 (3)	1.78 (3)	2.6435 (19)	178 (2)
O12—H12$\cdots$O22^{ii}	0.95 (3)	1.52 (3)	2.4726 (18)	180 (4)
N4A—H41$A\cdots$O11^{iii}	0.94 (2)	1.98 (2)	2.895 (2)	163.9 (19)
N4A—H41$A\cdots$O21^{iv}	0.94 (2)	2.47 (2)	2.874 (2)	106.0 (16)
N4A—H42$A\cdots$O12	0.94 (2)	2.54 (2)	2.953 (2)	106.6 (16)
N4A—H42$A\cdots$O22	0.94 (2)	1.95 (2)	2.874 (2)	166.6 (18)
N4A—H43$A\cdots$O21^{ii}	0.94 (2)	1.87 (2)	2.805 (2)	174 (2)

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

H atoms potentially involved in hydrogen-bonding interactions in all three compounds were located by difference methods and their positional and isotropic displacement parameters were refined. Other H atoms were included in calculated positions, with C—H = 0.93–0.95 Å, and treated as riding, with U_{iso}(H) = $1.2U_{eq}$(C).

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007) for (I); *SMART* (Bruker, 2000) for (II) and (III). Cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007) for (I); *SMART* for (II) and (III). Data reduction: *CrysAlis RED* for (I); *SAINT* (Bruker, 1999) for (II) and (III). For all compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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