Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Hydrogen-bonded structures of the isomeric 2-, 3- and 4-carbamoylpyridinium hydrogen chloranilates

Kazuma Gotoh, Hirokazu Nagoshi and Hiroyuki Ishida*

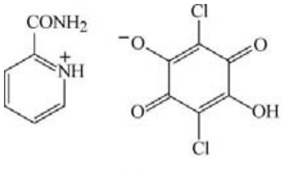
Department of Chemistry, Faculty of Science, Okayama University, Okayama 700-8530, Japan
Correspondence e-mail: ishidah@cc.okayama-u.ac.jp

Received 3 April 2009
Accepted 23 April 2009
Online 2 May 2009

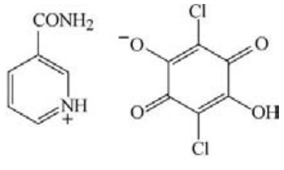
In the three isomeric salts, all $C_6H_7N_2O^+\cdot C_6HCl_2O_4^-$, of chloranilic acid (2,5-dichloro-3,6-dihydroxy-1,4-benzoquinone) with 2-, 3- and 4-carbamoylpyridine, namely, 2-carbamoylpyridinium hydrogen chloranilate (systematic name: 2-carbamoylpyridinium 2,5-dichloro-4-hydroxy-3,6-dioxocyclohexa-1,4-dienolate), (I), 3-carbamoylpyridinium hydrogen chloranilate, (II), and 4-carbamoylpyridinium hydrogen chloranilate, (III), acid–base interactions involving H-atom transfer are observed. The shortest interactions between the cation and the anion in (I) and (II) are pyridinium N—H···(O,O) bifurcated hydrogen bonds, which act as the primary intermolecular interaction in each crystal structure. In (III), an amide N—H···(O,O) bifurcated hydrogen bond, which is much weaker than the bifurcated hydrogen bonds in (I) and (II), connects the cation and the anion.

Comment

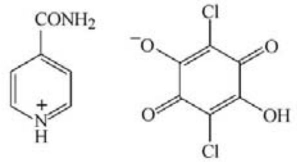
Chloranilic acid (2,5-dichloro-3,6-dihydroxy-1,4-benzoquinone), a strong dibasic acid with hydrogen-bond donor and acceptor groups, appears particularly attractive as a template for generating tightly bound self-assemblies with various pyridine derivatives (Ishida & Kashino, 1999*a*,*b*,*c*, 2002; Zaman *et al.*, 1999, 2000, 2001, 2004), and as a model compound for investigating H-atom transfer in O—H···N and N—H···O hydrogen-bond systems (Nihei *et al.*, 2000*a*,*b*; Ikeda *et al.*, 2005; Suzuki *et al.*, 2007; Gotoh *et al.*, 2008; Seliger *et al.*, 2009). Furthermore, salts and cocrystals of chloranilic acid with pyridine derivatives have recently attracted much interest with respect to organic ferroelectrics (Horiuchi, Ishii *et al.*, 2005; Horiuchi, Kumai & Tokura, 2005; Asaji *et al.*, 2007; Gotoh *et al.*, 2007; Horiuchi & Tokura, 2008). In the present study, we have prepared three isomeric salts of chloranilic acid with picolinamide, nicotinamide and isonicotinamide, namely, 2-carbamoylpyridinium hydrogen chloranilate, (I), 3-carbamoylpyridinium hydrogen chloranilate, (II), and 4-carbamoylpyridinium hydrogen chloranilate, (III), in order to extend our studies of *D*—H···*A* hydrogen bonding (*D* = N, O or C; *A* = N, O or Cl) in chloranilic acid–substituted-pyridine systems (Tabuchi *et al.*, 2005; Gotoh *et al.*, 2006, 2009).



(I)



(II)



(III)

Compound (I) contains one protonated picolinamide cation and one chloranilate monoanion, which are linked by a bifurcated pyridinium N1—H1···(O1,O4) hydrogen bond (Table 1). There is a dihedral angle of 64.42 (6)° between the plane of the pyridine N1/C7–C11 ring and that of the anion C1–C6 ring (Fig. 1). In the cation, the acetamide C12/O5/N2 plane makes a dihedral angle of 11.62 (4)° with the pyridine ring plane, which is comparable with the equivalent angles of 9.84 (8) and 19.35 (4)° in compounds (II) and (III), respectively. This suggests that the contribution of the intramolecular N—H···O hydrogen bond (N1—H1···O5) to the molecular conformation of (I) is small. Similar angles of 11.44 (6), 2.74 (6) and 10.61 (3)° between the carboxy plane and the pyridine ring plane are observed in, respectively, 2-carboxypyridinium hydrogen chloranilate (Gotoh *et al.*, 2009), 3-carboxypyridinium hydrogen chloranilate (Ishida, 2009*a*) and 4-carboxypyridinium hydrogen chloranilate monohydrate (Ishida, 2009*b*), which also suggests a weak intramolecular N—H···O hydrogen bond between the pyridininum N—H group and the carboxy O atom in 2-carboxypyridinium hydrogen chloranilate. The two components in (I) are further connected through a pair of O2—H2···(O3,O5^{iii}) bifurcated hydrogen bonds (symmetry code as in Table 1), to form a centrosymmetric 2+2 aggregate (Fig. 2). One of the amide

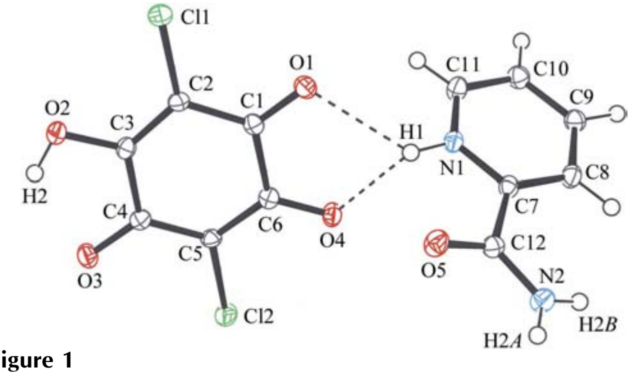


Figure 1
A molecular view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The bifurcated N—H···(O,O) hydrogen bond is indicated by dashed lines.

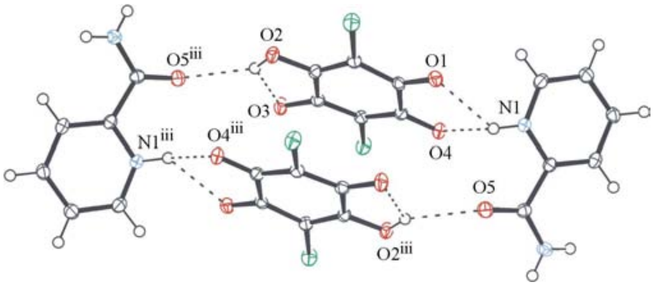


Figure 2
The 2+2 aggregate of (I), formed by N—H···(O,O) and O—H···(O,O) bifurcated hydrogen bonds (dashed lines). [Symmetry code: (iii) $-x + 1$, $-y + 1$, $-z$.]

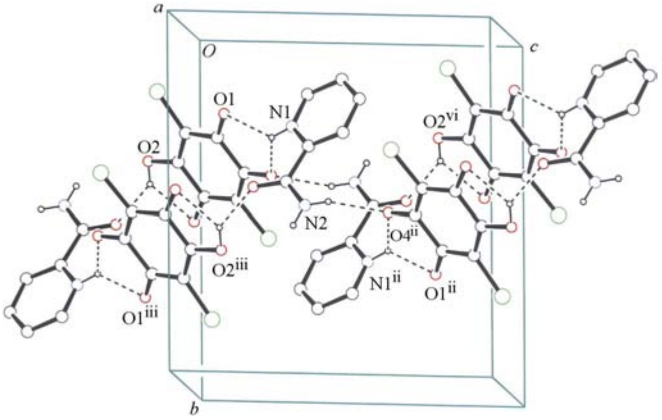


Figure 3
A partial packing diagram for (I), viewed approximately along the a axis, showing the hydrogen-bonded chain structure. Dashed lines show N—H···O and O—H···O hydrogen bonds. H atoms not involved in the hydrogen bonds have been omitted. [Symmetry codes: (ii) $-x + 2$, $-y + 1$, $-z + 1$; (iii) $-x + 1$, $-y + 1$, $-z$; (vi) $x + 1$, y, $z + 1$.]

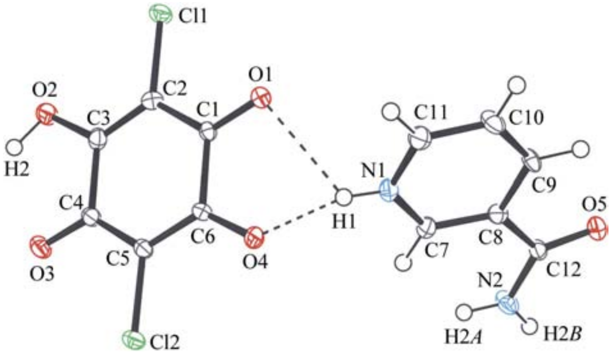


Figure 4
A molecular view of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The bifurcated N—H···(O,O) hydrogen bond is indicated by dashed lines.

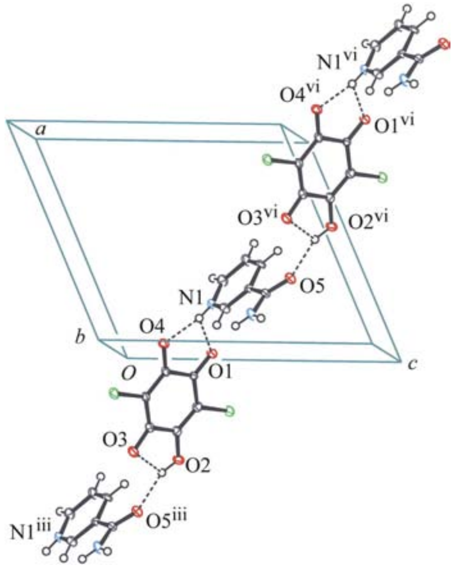


Figure 5
The supramolecular chain of (II), formed by N—H···(O,O) and O—H···(O,O) bifurcated hydrogen bonds (dashed lines). [Symmetry codes: (iii) $x - 1$, $y - 1$, $z - 1$; (vi) $x + 1$, $y + 1$, $z + 1$.]

N—H···O hydrogen bonds (N2—H2B···O4$^{\rm ii}$; Table 1) connects the aggregates into a chain along the [101] direction (Fig. 3). The second amide N—H···O hydrogen bond (N2—H2A···O1$^{\rm i}$; Table 1) further connects the chains, forming a three-dimensional network. A weak C—H···(O,O) bifurcated interaction is also present (Table 1).

Although all intermolecular N—H···O and O—H···O hydrogen bonds corresponding to those in (I) are observed in (II) (Table 2), all the $D\cdots A$ distances of the hydrogen bonds in (II) are shorter than those in (I), so that the molecular packing motifs are quite different from each other (Figs. 4–6). In (II), the cations and anions are separately stacked in columns along the b axis. Between the two components a bifurcated pyridinium N1—H1···(O1,O4) hydrogen bond and an O2—H2···(O3,O5$^{\rm iii}$) hydrogen bond (symmetry code as in Table 2) are present as the primary interactions, forming a zigzag supramolecular chain along the [111] direction (Fig. 5). The dihedral angle between the plane of the pyridine N1/C7–C11 ring and that of the anion C1–C6 ring is 71.59 (10)°. This packing motif is similar to that of 3-carboxypyridinium hydrogen chloranilate, which crystallizes in the same space group, *i.e.* Pc, and is effectively isostructural (Tabuchi *et al.*, 2005). The cations and anions are linked by the equivalent hydrogen bonds to form a chain. In 3-carboxypyridinium hydrogen chloranilate, the carboxy group of the cation and the carbonyl O atom of the anion form an additional strong O—H···O hydrogen bond, which further connects the supramolecular chains related to each other by a c-glide plane to form a three-dimensional hydrogen-bond network. On the other hand, in (II) the amide group of the cation forms two weak N—H···O hydrogen bonds as the secondary interaction. One amide N—H···O hydrogen bond (N2—H2A···O1$^{\rm i}$; Table 2) connects neighbouring chains related by a b translation, giving a wavy layer expanding parallel to the $(10\overline{1})$ plane (Fig. 6). Neighbouring layers related to each other by a c-glide plane are further linked by the second amide N—H···O hydrogen bond (N2—H2B···O4$^{\rm ii}$; Table 2) to form a three-dimensional hydrogen-bond network.

Compound (III) also crystallizes in a noncentrosymmetric space group, *i.e.* Cc, where the basic hydrogen-bonded structure is quite different from those of (I) and (II). In (III), primary hydrogen bonds are formed between the cations

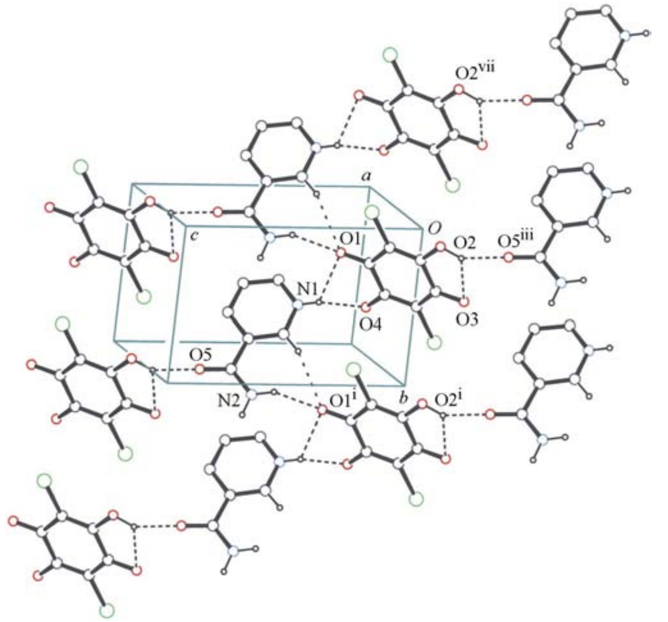


Figure 6
A partial packing diagram for (II), viewed approximately along the *a* axis, showing the hydrogen-bonded layer structure. Dashed lines show N—H···O, O—H···O and C—H···O hydrogen bonds. H atoms not involved in the hydrogen bonds have been omitted. [Symmetry codes: (i) $x, y + 1, z$; (iii) $x - 1, y - 1, z - 1$; (vii) $x, y - 1, z$.]

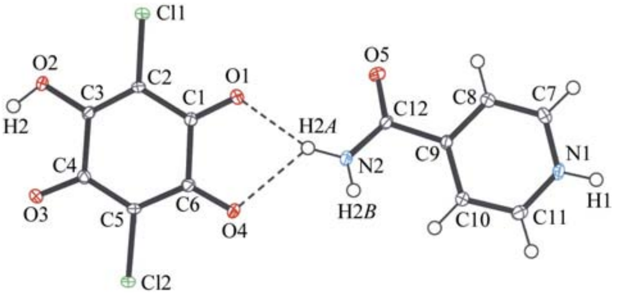


Figure 7
A molecular view of (III), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The bifurcated N—H···(O,O) hydrogen bond is indicated by dashed lines.

(N1—H1···O5^{i}; symmetry code as in Table 3) and between the anions [O2—H2···(O3,O4^{iii}); Table 3], and each component affords a supramolecular zigzag chain along the [101] direction (Figs. 7 and 8). The cation and anion chains are alternately arranged and linked together through amide N—H···O hydrogen bonds [N2—H2*A*···(O1,O4) and N2—H2*B*···O3^{ii}] and C—H···O hydrogen bonds (Table 3), forming a layer parallel to the (10$\overline{1}$) plane. In the layer, the cations and anions are approximately coplanar, with a dihedral angle of 4.61 (5)° between the planes of the N1/C7–C11 and C1–C6 rings. A short Cl···Cl contact [Cl1···Cl2^{v} = 3.1634 (5) Å; symmetry code: (v) $x + \frac{1}{2}, y + \frac{1}{2}, z$] is also observed in the layer. Between the layers, no significant interactions are observed; the shortest contact is C4···C7^{vi} = 3.167 (2) Å [symmetry code: (vi) $x + \frac{1}{2}, y - \frac{1}{2}, z + 1$].

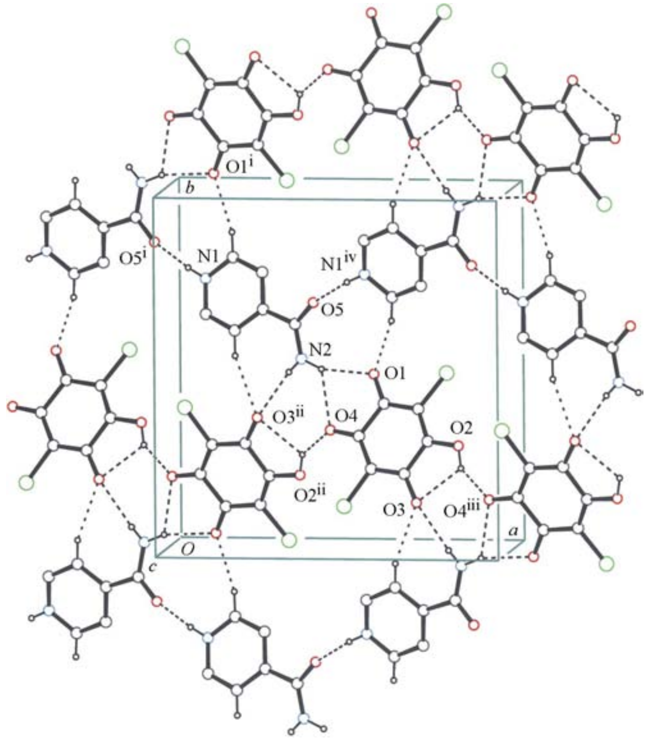


Figure 8
A packing diagram for (III), viewed along the *c* axis, showing the hydrogen-bonded layer structure. Dashed lines show N—H···O, O—H···O and C—H···O hydrogen bonds. H atoms not involved in the hydrogen bonds have been omitted. [Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (iv) $x + \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$.]

Experimental

Crystals of (I) and (III) were obtained by slow evaporation from methanol solutions [40 and 100 ml for (I) and (III), respectively] of chloranilic acid with picolinamide or isonicotinamide in a 1:1 molar ratio [0.300 g of chloranilic acid and 0.176 g of picolinamide for (I), and 0.100 g of chloranilic acid and 0.058 g of isonicotinamide for (III)] at room temperature. Crystals of (II) were obtained by slow evaporation from a water–methanol (1:1 *v*/*v*) solution (140 ml) of chloranilic acid (0.302 g) and nicotinamide (0.179 g) at room temperature.

Compound (I)

Crystal data

$C_6H_7N_2O^+\cdot C_6HCl_2O_4^-$
M_r = 331.11
Monoclinic, $P2_1/c$
a = 9.3420 (5) Å
b = 12.4483 (8) Å
c = 11.4167 (7) Å
β = 108.8014 (17)°
V = 1256.83 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 100 K
0.27 × 0.25 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID-II diffractometer
Absorption correction: numerical (*ABSCOR*; Higashi, 1995)
T_{min} = 0.875, T_{max} = 0.902
11008 measured reflections
3596 independent reflections
2830 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O1	0.90 (3)	2.32 (3)	3.005 (2)	133 (2)
N1—H1$\cdots$O4	0.90 (3)	2.07 (3)	2.713 (2)	128 (3)
N1—H1$\cdots$O5	0.90 (3)	2.32 (3)	2.690 (2)	105 (2)
N2—H2$A\cdots$O1^{i}	0.84 (2)	2.20 (3)	3.033 (2)	168 (2)
N2—H2$B\cdots$O4^{ii}	0.85 (2)	2.12 (2)	2.919 (2)	156 (3)
O2—H2$\cdots$O3	0.90 (3)	2.00 (3)	2.5942 (19)	123 (3)
O2—H2$\cdots$O5^{iii}	0.90 (3)	2.35 (3)	3.098 (2)	142 (3)
C10—H10$\cdots$O3^{iv}	0.95	2.38	3.026 (2)	125
C10—H10$\cdots$O5^{v}	0.95	2.48	3.310 (2)	146

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O1	0.84 (5)	2.47 (5)	2.984 (3)	121 (4)
N1—H1$\cdots$O4	0.84 (5)	1.82 (5)	2.638 (3)	164 (4)
N2—H2$A\cdots$O1^{i}	0.88 (4)	2.22 (4)	2.991 (3)	146 (4)
N2—H2$B\cdots$O4^{ii}	0.96 (7)	1.95 (6)	2.899 (3)	172 (6)
O2—H2$\cdots$O3	0.92 (6)	2.11 (5)	2.613 (3)	114 (5)
O2—H2$\cdots$O5^{iii}	0.92 (6)	1.95 (6)	2.754 (3)	146 (5)
C7—H7$\cdots$O1^{i}	0.95	2.47	3.282 (3)	143
C11—H11$\cdots$O2^{iv}	0.95	2.47	3.206 (4)	134
C11—H11$\cdots$O5^{v}	0.95	2.48	3.370 (3)	157

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O5^{i}	0.92 (2)	1.79 (2)	2.6966 (18)	173 (2)
N2—H2$A\cdots$O1	0.94 (3)	2.07 (3)	2.9598 (19)	158 (2)
N2—H2$A\cdots$O4	0.94 (3)	2.32 (3)	2.9469 (18)	124 (2)
N2—H2$B\cdots$O3^{ii}	0.82 (3)	2.16 (3)	2.9424 (18)	161 (3)
O2—H2$\cdots$O3	0.89 (3)	2.21 (3)	2.6728 (16)	112 (2)
O2—H2$\cdots$O4^{iii}	0.89 (3)	1.93 (3)	2.6953 (15)	144 (2)
C7—H7$\cdots$O1^{i}	0.95	2.37	3.2959 (19)	164
C10—H10$\cdots$O3^{ii}	0.95	2.45	3.3599 (19)	159

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

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.111$
$S = 1.11$
3596 reflections
206 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.54$ e Å^{-3}

Compound (II)

Crystal data

$C_6H_7N_2O^+ \cdot C_6HCl_2O_4^-$
$M_r = 331.11$
Monoclinic, Pc
$a = 9.9861$ (5) Å
$b = 6.1438$ (3) Å
$c = 11.6027$ (5) Å
$\beta = 114.8583$ (16)°
$V = 645.90$ (5) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.53$ mm^{-1}
$T = 100$ K
0.38 × 0.27 × 0.08 mm

Data collection

Rigaku R-AXIS RAPID-II diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.753$, $T_{\max} = 0.959$
5606 measured reflections
3191 independent reflections
2991 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.149$
$S = 1.01$
3191 reflections
206 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.45$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}
Absolute structure: Flack (1983), with 1354 Friedel pairs
Flack parameter: 0.09 (6)

Compound (III)

Crystal data

$C_6H_7N_2O^+ \cdot C_6HCl_2O_4^-$
$M_r = 331.11$
Monoclinic, Cc
$a = 12.9482$ (4) Å
$b = 13.4993$ (5) Å
$c = 7.0726$ (3) Å
$\beta = 92.0130$ (11)°
$V = 1235.47$ (8) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.55$ mm^{-1}
$T = 100$ K
0.30 × 0.15 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID-II diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.767$, $T_{\max} = 0.936$
5840 measured reflections
3185 independent reflections
3029 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.016$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.061$
$S = 1.08$
3185 reflections
206 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), with 1411 Friedel pairs
Flack parameter: 0.00 (3)

H atoms attached to O and N atoms were found in a difference Fourier map and refined isotropically (refined O—H and N—H distances are given in Tables 1–3). Other H atoms were treated as riding, with C—H distances of 0.95 Å and with U_{iso}(H) values of 1.2U_{eq}(C). For compounds (II) and (III), the correct orientations of the structures with respect to the polar-axis directions were determined by use of the Flack x parameters (Flack, 1983). The Hooft y parameters (Hooft *et al.*, 2008) were evaluated to be 0.14 (2) and −0.008 (18), respectively, for (II) and (III).

For all compounds, data collection: *PROCESS-AUTO* (Rigaku/MSC, 2004); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 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); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2009).

This work was supported by a Grant-in-Aid for Scientific Research (C) (No. 19550018) from the Japan Society for the Promotion of Science.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD3286). Services for accessing these data are described at the back of the journal.

References

Asaji, T., Seliger, J., Žager, V., Sekiguchi, M., Watanabe, J., Gotoh, K., Ishida, H., Vrtnik, S. & Dolinsek, J. (2007). *J. Phys. Condens Matter*, **19**, 226203.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Gotoh, K., Asaji, T. & Ishida, H. (2007). *Acta Cryst.* C**63**, o17–o20.

Gotoh, K., Asaji, T. & Ishida, H. (2008). *Acta Cryst.* C**64**, o550–o553.

Gotoh, K., Nagoshi, H. & Ishida, H. (2009). *Acta Cryst.* E**65**, o614.

Gotoh, K., Tabuchi, Y., Akashi, H. & Ishida, H. (2006). *Acta Cryst.* E**62**, o4420–o4421.

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.

Hooft, R. W. W., Straver, L. H. & Spek, A. L. (2008). *J. Appl. Cryst.* **41**, 96–103.

Horiuchi, S., Ishii, F., Kumai, R., Okimoto, Y., Tachibana, H., Nagaosa, N. & Tokura, Y. (2005). *Nat. Mater.* **4**, 163–166.

Horiuchi, S., Kumai, R. & Tokura, Y. (2005). *J. Am. Chem. Soc.* **127**, 5010–5011.

Horiuchi, S. & Tokura, Y. (2008). *Nat. Mater.* **7**, 357–366.

Ikeda, R., Takahashi, S., Nihei, T., Ishihara, H. & Ishida, H. (2005). *Bull. Chem. Soc. Jpn*, **78**, 1241–1245.

Ishida, H. (2009*a*). Private communication (deposition number 720198). CCDC, Cambridge, England.

Ishida, H. (2009*b*). Private communication (deposition number 720199). CCDC, Cambridge, England.

Ishida, H. & Kashino, S. (1999*a*). *Acta Cryst.* C**55**, 1149–1152.

Ishida, H. & Kashino, S. (1999*b*). *Acta Cryst.* C**55**, 1714–1717.

Ishida, H. & Kashino, S. (1999*c*). *Acta Cryst.* C**55**, 1923–1926.

Ishida, H. & Kashino, S. (2002). *Z. Naturforsch. Teil A*, **57**, 829–836.

Nihei, T., Ishimaru, S., Ishida, H., Ishihara, H. & Ikeda, R. (2000*a*). *Chem. Phys. Lett.* **329**, 7–14.

Nihei, T., Ishimaru, S., Ishida, H., Ishihara, H. & Ikeda, R. (2000*b*). *Chem. Lett.* pp. 1346–1347.

Rigaku/MSC (2004). *PROCESS-AUTO* and *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.

Seliger, J., Žagar, V., Gotoh, K., Ishida, H., Konnai, A., Amino, D. & Asaji, T. (2009). *Phys. Chem. Chem. Phys.* **11**, 2281–2286.

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

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Suzuki, H., Mori, H., Yamashita, J., Matsuda, M., Tajima, H. & Mochida, T. (2007). *Chem. Lett.* pp. 402–403.

Tabuchi, Y., Takahashi, A., Gotoh, K., Akashi, H. & Ishida, H. (2005). *Acta Cryst.* E**61**, o4215–o4217.

Zaman, Md. B., Tomura, M. & Yamashita, Y. (1999). *Chem. Commun.* pp. 999–1000.

Zaman, Md. B., Tomura, M. & Yamashita, Y. (2000). *Org. Lett.* **2**, 273–275.

Zaman, Md. B., Tomura, M. & Yamashita, Y. (2001). *J. Org. Chem.* **66**, 5987–5995.

Zaman, Md. B., Udachin, K. A. & Ripmeester, J. A. (2004). *Cryst. Growth Des.* **4**, 585–589.