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Imidazolium 2-chloro-4-nitrobenzoate

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Key indicators

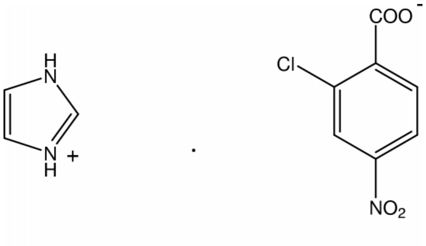
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.076
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The cations and anions of the title compound, $C_3H_5N_2^+\cdot C_7H_3ClNO_4^-$, are connected by N—H···O hydrogen bonds to afford a 2_1 helical chain. There are two important C—H···O interactions which link the chains.

Comment

The title compound, (I), was investigated as part of a study on *D*—H···*A* hydrogen bonding (*D*: N, O or C; *A*: N, O or Cl) in chloro- and nitro-substituted benzoic acid–amine systems (Ishida *et al.* 2001*a*,*b*,*c*). This is the first report of an enantiomorphic crystal in chloro- and nitro-substituted benzoic acid-amine systems.



(I)

An acid–base interaction involving a proton transfer is observed as expected from the high basicity of this amine (Fig. 1). The cations and anions are held together by short N—H···O hydrogen bonds (Table 2), forming a 2_1 helical chain along the *b* axis (Fig. 2). The nitro and carboxyl groups are considerably twisted out of the benzene ring plane; the dihedral angle between the nitro group and the benzene ring plnae is 11.9 (2)° and that between the carboxyl group and the benzene ring plane is 63.9 (2)°. The dihedral angle between the imidazolium ion and the benzene ring is 14.8 (2)°. There are two important C—H···O interactions (Table 2) which connect the helical chains.

Usually, the 2-chloro-4-nitrobenzoate ion is classified as an achiral molecule because of the rotational flexibility of the nitro and carboxyl groups around the C—N and C—C bond axes, respectively. In crystals, however, the rotation of these groups is hindered by intermolecular interactions and hence the dihedral angles of these groups with respect to the benzene ring may be restricted to certain values other than 0 and 90°. The present result provides evidence of resolution in the chirality of the benzoate ion by a helical chain formation *via* hydrogen bonding in the solid state.

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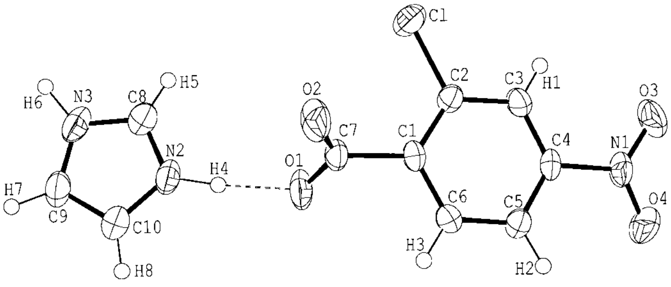

Figure 1
ORTEP-3 (Farrugia, 1997) drawing of (I) with the atom labeling. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

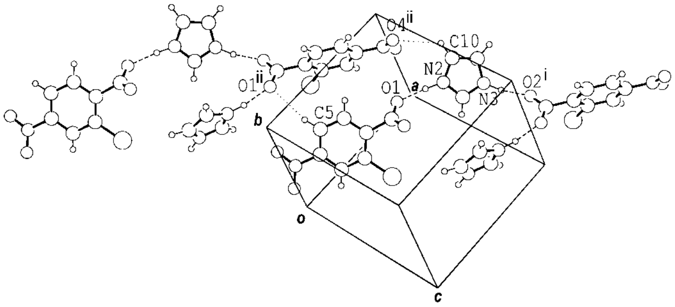

Figure 2
Packing diagram showing a 2_1 helical structure formed *via* N—H···O hydrogen bonds indicated by dashed lines. C—H···O interactions which connect the helical chains are indicated by dotted lines (symmetry codes are as in Table 2).

Experimental

Crystal data

$C_3H_5N_2^+ \cdot C_7H_3ClNO_4^-$
$M_r = 269.64$
Monoclinic, $P2_1$
$a = 9.078$ (2) Å
$b = 5.928$ (2) Å
$c = 10.7711$ (18) Å
$\beta = 98.998$ (17)°
$V = 572.5$ (2) Å^3
$Z = 2$
$D_x = 1.564$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 11.9$–12.4°
$\mu = 0.34$ mm^{-1}
$T = 298$ K
Prismatic, colorless
0.40 × 0.30 × 0.20 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scans (North *et al.*, 1968)
$T_{\min} = 0.874$, $T_{\max} = 0.934$
3945 measured reflections
2652 independent reflections
2141 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.014$
$\theta_{\max} = 30.0°$
$h = -4 \rightarrow 12$
$k = -3 \rightarrow 8$
$l = -15 \rightarrow 15$
3 standard reflections every 97 reflections intensity decay: 3.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.076$
$S = 1.04$
2652 reflections
186 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0320P)^2 + 0.0911P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.012 (3)
Absolute structure: Flack (1983), 836 Friedel pairs
Flack parameter = 0.02 (6)

Table 1
Selected geometric parameters (Å, °).

Cl—C2	1.737 (2)	N3—C9	1.358 (4)
O1—C7	1.241 (2)	C1—C2	1.385 (3)
O2—C7	1.245 (2)	C1—C6	1.393 (3)
O3—N1	1.206 (3)	C1—C7	1.524 (2)
O4—N1	1.221 (2)	C2—C3	1.395 (2)
N1—C4	1.473 (2)	C3—C4	1.375 (3)
N2—C8	1.311 (3)	C4—C5	1.372 (3)
N2—C10	1.368 (3)	C5—C6	1.390 (3)
N3—C8	1.321 (3)	C9—C10	1.349 (3)
C8—N2—C10	108.37 (19)	C10—C9—N3	107.2 (2)
C8—N3—C9	108.3 (2)	C9—C10—N2	106.8 (2)
N2—C8—N3	109.3 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H4···O1	1.00 (3)	1.67 (3)	2.661 (2)	169 (3)
N3—H6···O2^{i}	1.03 (3)	1.65 (3)	2.665 (3)	169 (3)
C5—H2···O1^{ii}	0.96 (2)	2.50 (2)	3.444 (3)	168.0 (15)
C10—H8···O4^{ii}	0.97 (3)	2.64 (3)	3.361 (3)	131 (2)

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

H atoms were refined isotropically.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1990); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN for Windows*.

X-ray measurements were made at the X-ray Laboratory of Okayama University.

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