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Morpholinium 5-chloro-2-nitrobenzoate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.057
wR factor = 0.090
Data-to-parameter ratio = 13.3

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

In the title compound, $C_4H_{10}NO^+\cdot C_7H_3ClNO_4^-$, two cations and two anions are connected by N—H···O hydrogen bonds to afford a ring with descriptor $R_4^4(12)$, which is located on an inversion center. There are four C—H···O interactions which connect the ring units to form a three-dimensional network.

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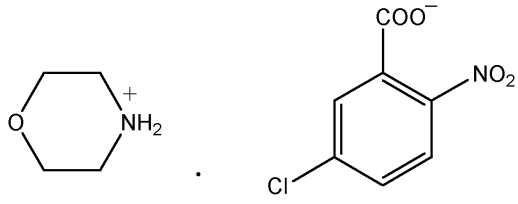
Comment

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). To our knowledge, this is the first structural report of a 5-chloro-2-nitrobenzoic acid–amine system.



(I)

In the crystal, two cations and two anions are held together by short N—H···O hydrogen bonds (Table 2), forming a centrosymmetric hydrogen-bonded ring (Fig. 1) with graph-set descriptor $R_4^4(12)$ (Bernstein *et al.*, 1995). 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 is 51.8 (2)° and that between the carboxyl group and the benzene ring is 46.1 (2)°. There are four C—H···O interactions involving O atoms of the nitro group and of the cation. The $R_4^4(12)$ rings are arranged along the *a*, *b* and *c* axes through C6—H3···O5^{iii} and C9—H9···O5^{iv} interactions, a C11—H13···O3^{v} interaction, and a C4—H2···O4^{ii} interaction (symmetry codes are as in Table 2), respectively, to form a three-dimensional network (Fig. 2).

Experimental

Crystals of (I) were obtained by slow evaporation from an acetonitrile solution of morpholine and the benzoic acid in a molar ratio of 1:1.

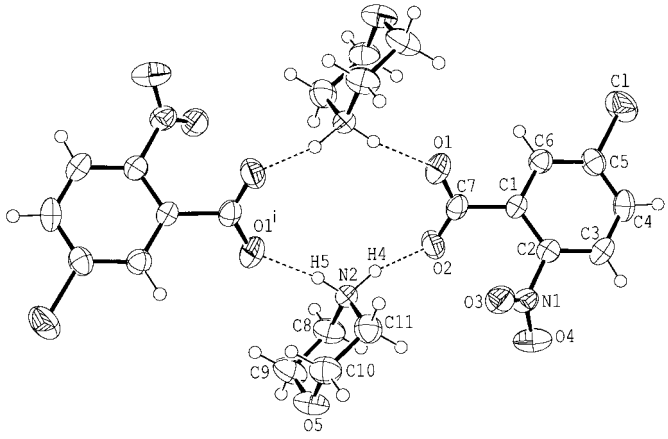


Figure 1
ORTEP-3 (Farrugia, 1997) drawing of a hydrogen-bonded ring with the atom-labeling. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. N—H···O hydrogen bonds are indicated by dashed lines [symmetry code: (i) $1-x, -y, -z$].

Crystal data

$C_4H_{10}NO^+\cdot C_7H_3ClNO_4^-$
M_r = 288.69
Monoclinic, $P2_1/n$
a = 10.6482 (15) Å
b = 5.9079 (12) Å
c = 21.084 (3) Å
β = 102.722 (10)°
V = 1293.8 (3) Å^3
Z = 4
D_x = 1.482 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 11.1–12.0°
μ = 0.31 mm^{-1}
T = 298 K
Prismatic, colorless
0.50 × 0.30 × 0.20 mm

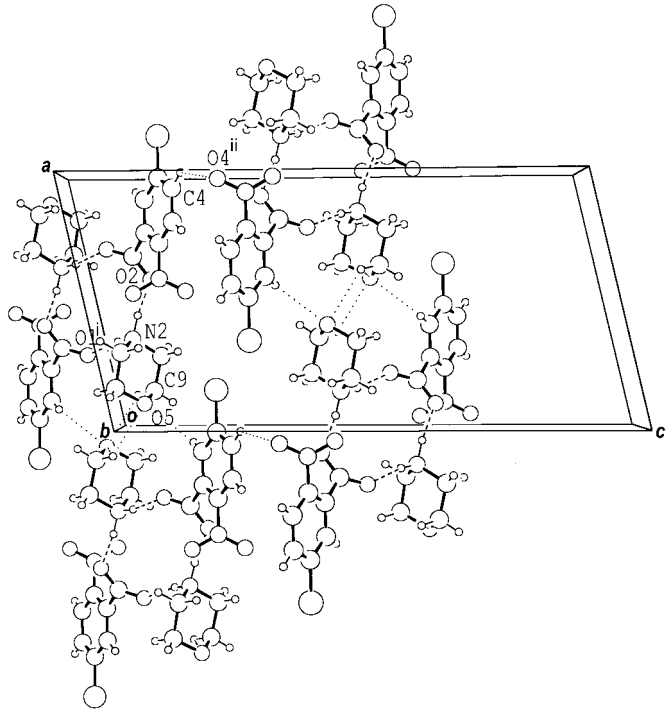


Figure 2
Packing diagram showing a three-dimensional hydrogen-bond network formed *via* N—H···O and C—H···O hydrogen bonds indicated by dashed and dotted lines, respectively [symmetry codes are as in Table 2].

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.894, T_{max} = 0.939
3789 measured reflections
2980 independent reflections
1667 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°
$h = -1 \rightarrow 13$
$k = 0 \rightarrow 7$
$l = -27 \rightarrow 27$
3 standard reflections
every 97 reflections
intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.090
S = 1.14
2979 reflections
224 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o) + 0.00029|F_o|^2]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl—C5	1.742 (3)	C3—C4	1.376 (4)
O1—C7	1.238 (3)	C4—C5	1.367 (4)
O2—C7	1.240 (3)	C5—C6	1.380 (4)
O3—N1	1.215 (3)	O5—C9	1.417 (4)
O4—N1	1.222 (3)	O5—C10	1.423 (3)
N1—C2	1.474 (3)	N2—C8	1.479 (3)
C1—C2	1.383 (3)	N2—C11	1.492 (4)
C1—C6	1.387 (3)	C8—C9	1.503 (4)
C1—C7	1.523 (3)	C10—C11	1.505 (4)
C2—C3	1.377 (4)		
C9—O5—C10	109.7 (2)	O5—C9—C8	111.7 (3)
C8—N2—C11	110.3 (2)	O5—C10—C11	110.9 (2)
N2—C8—C9	109.5 (2)	N2—C11—C10	109.5 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H4···O2	1.02 (3)	1.71 (3)	2.696 (3)	164 (3)
N2—H5···O1^{i}	0.89 (3)	1.86 (3)	2.734 (3)	169 (2)
C4—H2···O4^{ii}	0.91 (3)	2.74 (3)	3.408 (4)	132 (2)
C6—H3···O5^{iii}	0.95 (2)	2.63 (2)	3.339 (3)	131 (2)
C9—H9···O5^{iv}	1.04 (3)	2.68 (3)	3.455 (4)	132 (2)
C11—H13···O3^{v}	1.01 (2)	2.51 (2)	3.447 (4)	155 (2)

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

H atoms were located in difference Fourier maps and refined isotropically. Refined distances: C—H = 0.89 (2)–1.04 (3) Å and N—H = 0.89 (3)–1.02 (3) Å.

Data collection: *MSC*/*FC 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: *TEXSAN for Windows*; 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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