Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Morpholinium 4-chloro-2-nitrobenzoate

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Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.098
Data-to-parameter ratio = 13.4

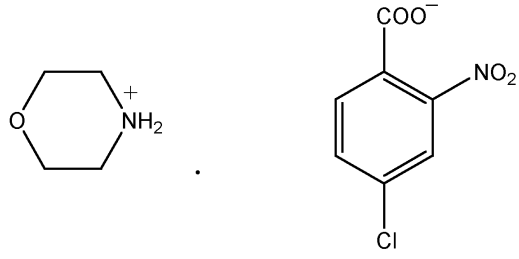
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 June 2001
Accepted 15 June 2001
Online 29 June 2001

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 three C—H···O interactions which connect the ring units to form a three-dimensional network.

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 4-chloro-2-nitrobenzoic acid–amine system.



(I)

An acid–base interaction involving a proton transfer is observed as expected from the high basicity of this present amine. 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. The dihedral angle between the nitro group and the benzene ring plane is 50.7 (2)° and that between the carboxyl group and the benzene ring is 43.6 (2)°. The macro rings are arranged along the a axis by a C—H···O interaction formed between the cation and the nitro group (C8—H7···O3^{iii}; Table 2). The rings are also linked along the b and c axes by two C—H···O interactions (C5—H2···O5^{ii} and C11—H12···O2^{iv}; Table 2), formed between the benzene ring and the cation, and between the cation and the carboxyl group (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.

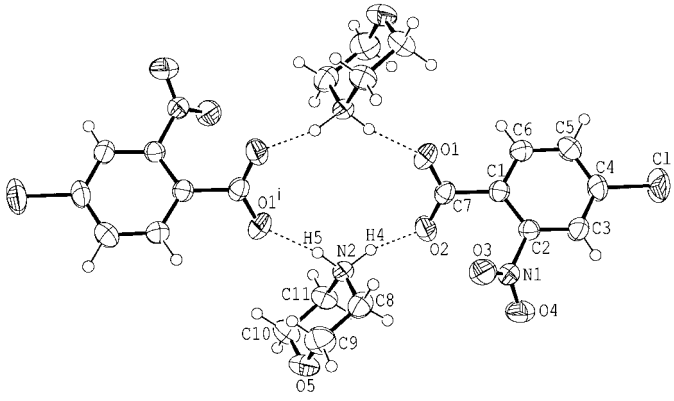


Figure 1
ORTEP-3 (Farrugia, 1997) drawing of a hydrogen-bonded ring with the atom-labeling. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. N—H···O hydrogen bonds are indicated by dashed lines [symmetry code: (i) $1-x, 1-y, 1-z$].

Crystal data

$C_4H_{10}NO^+\cdot C_7H_3ClNO_4^-$	D_x = 1.458 Mg m^{-3}
M_r = 288.69	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 25 reflections
a = 6.0475 (10) Å	θ = 11.0–12.1°
b = 25.808 (4) Å	μ = 0.31 mm^{-1}
c = 8.6101 (10) Å	T = 299 K
β = 101.853 (11)°	Plate, colorless
V = 1315.2 (3) Å^3	0.45 × 0.35 × 0.18 mm
Z = 4	

Data collection

Rigaku AFC-5*R* diffractometer	R_{int} = 0.028
ω–2θ scans	θ_{max} = 27.5°
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -1 \rightarrow 7$
T_{min} = 0.881, T_{max} = 0.946	$k = 0 \rightarrow 33$
3948 measured reflections	$l = -11 \rightarrow 11$
3006 independent reflections	3 standard reflections every 97 reflections
1740 reflections with $I > 2\sigma(I)$	intensity decay: 4.7%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = 0.01
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$wR(F^2)$ = 0.098	$\Delta\rho_{min}$ = −0.41 e Å^{-3}
S = 1.19	Extinction correction: Zachariasen (1967)
3005 reflections	Extinction coefficient: 9.9 (16) × 10^{-7}
225 parameters	
All H-atom parameters refined	
$w = 1/[\sigma^2(F_o) + 0.00032\|F_o\|^2]$	

Table 1
Selected geometric parameters (Å, °).

Cl—C4	1.735 (2)	C3—C4	1.375 (3)
O1—C7	1.238 (3)	C4—C5	1.384 (4)
O2—C7	1.231 (3)	C5—C6	1.380 (4)
O3—N1	1.213 (2)	O5—C9	1.420 (4)
O4—N1	1.219 (2)	O5—C10	1.418 (3)
N1—C2	1.472 (3)	N2—C8	1.483 (3)
C1—C2	1.390 (3)	N2—C11	1.478 (3)
C1—C6	1.385 (3)	C8—C9	1.496 (4)
C1—C7	1.519 (3)	C10—C11	1.509 (4)
C2—C3	1.379 (3)		
C9—O5—C10	109.6 (2)	O5—C9—C8	111.5 (3)
C8—N2—C11	110.1 (2)	O5—C10—C11	111.2 (2)
N2—C8—C9	110.1 (2)	N2—C11—C10	109.5 (2)

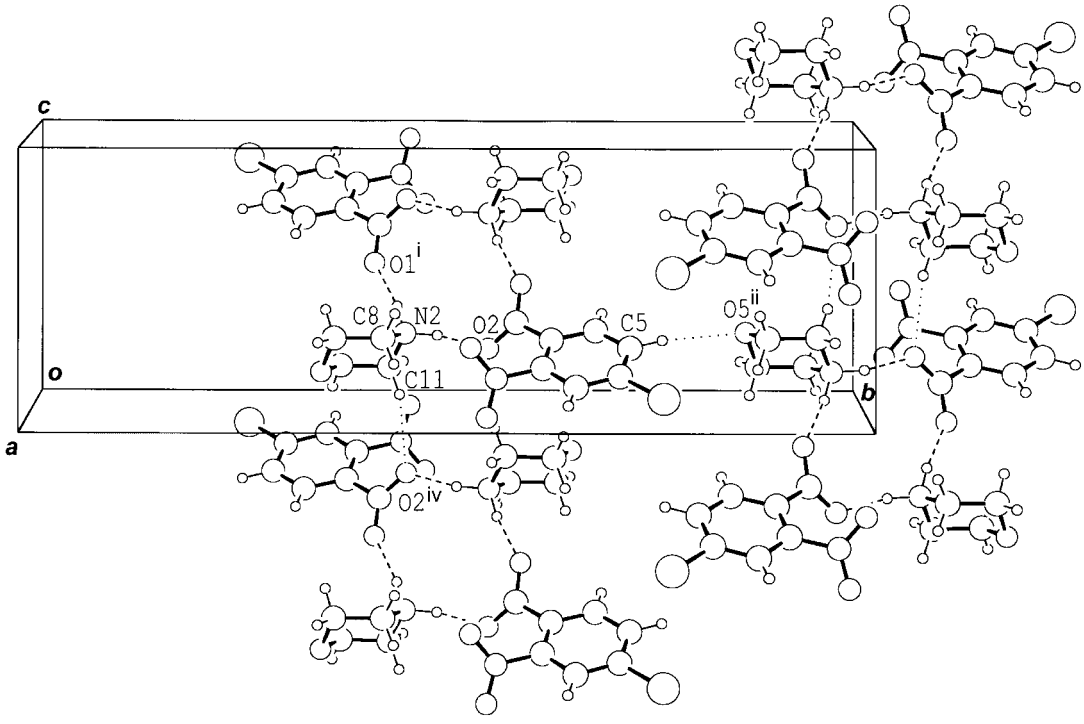


Figure 2
Packing diagram showing a three-dimensional hydrogen-bond network formed *via* N—H···O and C—H···O hydrogen bonds indicated by dashed and dotted lines, respectively [symmetry codes are as in Table 2].

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H4···O2	1.02 (3)	1.72 (3)	2.710 (2)	163 (2)
N2—H5···O1^{i}	0.95 (3)	1.80 (3)	2.733 (3)	166 (2)
C5—H2···O5^{ii}	1.01 (2)	2.57 (2)	3.575 (2)	171.4 (19)
C8—H7···O3^{iii}	1.00 (2)	2.54 (3)	3.391 (3)	143.6 (19)
C11—H12···O2^{iv}	0.97 (2)	2.55 (3)	3.398 (3)	146.4 (17)

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

H atoms were located in difference Fourier maps and refined isotropically. Refined distances: C—H = 0.88 (2)–1.06 (3) Å and N—H = 0.95 (3)–1.02 (3) Å.

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: *SAPI*91 (Fan, 1991); 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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