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The 1:1 complex of 2-chloro-4-nitrobenzoic acid and 1,2,3-benzotriazole

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Key indicators

Single-crystal X-ray study
T = 300 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.132
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.

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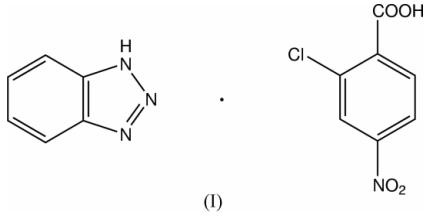
In the title compound, $C_7H_4ClNO_4\cdot C_6H_5N_3$, two acid and two base components are connected by O—H···N and N—H···O hydrogen bonds to afford a centrosymmetric macrocycle with graph-set descriptor $R_4^4(16)$. C—H···O hydrogen bonds connect the ring units to form a ribbon structure.

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*,*d*,*e*). In the crystal, two acid and two base components are held together by short O—H···N hydrogen bonds and relatively long N—H···O hydrogen bonds (Table 2) to afford a centrosymmetric macrocycle with graph-set descriptor $R_4^4(16)$ (Bernstein *et al.*, 1995) (Fig. 1), which is similar to that found in benzotriazole 3-nitrobenzoic acid (Hashizume *et al.*, 2001). The dihedral angle between the nitro group and the benzene ring is 10.03 (19)°, and that between the carboxyl group and the benzene ring is 22.79 (17)°. A C—H···O hydrogen bond (C5—H2···O4^{ii}; Table 2) connects the hydrogen-bonded rings, resulting in the formation of a molecular ribbon running parallel to the [011] direction (Fig. 2). The ribbons, related by a twofold screw axis, are stacked along the a axis. A short contact [Cl···O1^{iii}, 3.164 (3) Å; symmetry code: (iii) $\frac{1}{2}-x,\frac{1}{2}+y,\frac{3}{2}-z$] is observed between the ribbons.



(I)

Experimental

Crystals of (I) were obtained by slow evaporation from an acetonitrile solution of 1,2,3-benzotriazole and 2-chloro-4-nitrobenzoic acid in a molar ratio of 1:1.

Crystal data

$C_7H_4ClNO_4\cdot C_6H_5N_3$
M_r = 320.69
Monoclinic, $P2_1/n$
a = 7.0590 (15) Å
b = 11.7721 (13) Å
c = 16.4853 (17) Å
β = 93.717 (13)°
V = 1367.0 (4) Å^3
Z = 4
D_x = 1.558 Mg m^{-3}
Mo Kα radiation
Cell parameters from 25 reflections
θ = 10.5–12.5°
μ = 0.30 mm^{-1}
T = 300 K
Prism, colorless
0.40 × 0.30 × 0.25 mm

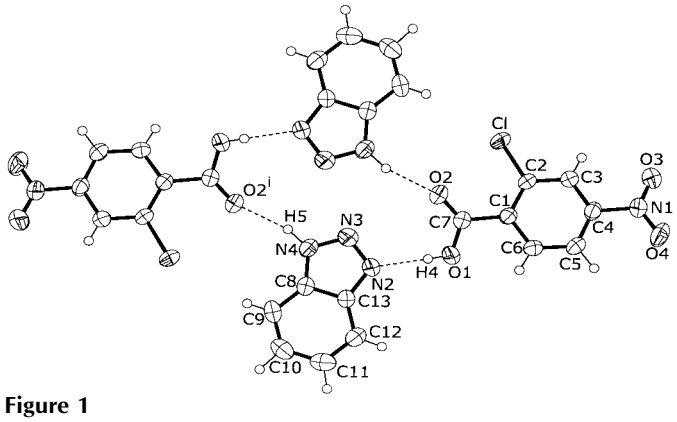


Figure 1
ORTEP-3 (Farrugia, 1997) drawing of a hydrogen-bonded ring of (I), with the atom-labeling. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. O—H···N and N—H···O hydrogen bonds are indicated by dashed lines [symmetry code: (i) $1-x, 1-y, 2-z$].

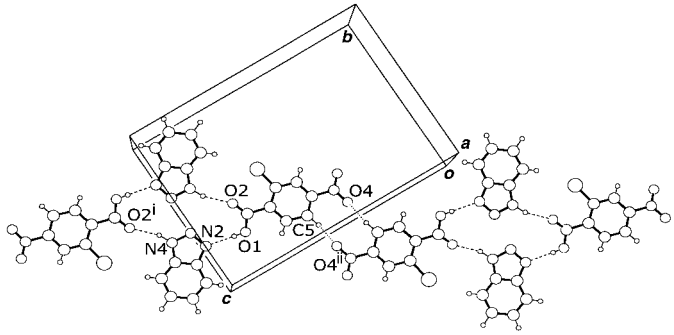


Figure 2
Packing diagram, showing a molecular ribbon formed *via* C—H···O hydrogen bonds (indicated by dotted lines). O—H···N and N—H···O hydrogen bonds are shown by dashed lines [symmetry codes are as in Table 2].

Data collection

Rigaku AFC-5*R* diffractometer	$R_{int} = 0.020$
ω–2θ scans	$\theta_{max} = 27.5^\circ$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -1 \rightarrow 9$
	$k = 0 \rightarrow 15$
$T_{min} = 0.913$, $T_{max} = 0.927$	$l = -21 \rightarrow 21$
3894 measured reflections	3 standard reflections
3134 independent reflections	every 97 reflections
1816 reflections with $I > 2\sigma(I)$	intensity decay: 1.4%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + 0.6722P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.052$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.132$	$\Delta\rho_{max} = 0.25$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.23$ e Å^{-3}
3134 reflections	Extinction correction: *SHELXL*
236 parameters	Extinction coefficient: 4.7 (11) $\times 10^{-3}$
All H-atom parameters refined	

Table 1
Selected geometric parameters (Å).

Cl—C2	1.723 (3)	N2—N3	1.302 (3)
O1—C7	1.303 (4)	N2—C13	1.379 (3)
O2—C7	1.196 (3)	N3—N4	1.336 (3)
O3—N1	1.215 (3)	N4—C8	1.360 (4)
O4—N1	1.214 (3)	C1—C7	1.505 (4)
N1—C4	1.478 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H4···N2	0.77 (5)	1.89 (5)	2.661 (3)	173 (5)
N4—H5···O2^{i}	0.95 (4)	2.00 (3)	2.909 (3)	158 (3)
C5—H2···O4^{ii}	0.97 (2)	2.48 (3)	3.265 (4)	138 (2)

Symmetry codes: (i) $1-x, 1-y, 2-z$; (ii) $1-x, -y, 1-z$.

H atoms were found in difference Fourier maps and refined isotropically. Refined distances: C—H = 0.89 (3)–1.03 (4), O—H = 0.78 (4) and N—H = 0.95 (4) 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: *SIR*92 (Altomare *et al.* 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 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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