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The 1:1 complex of 4-chloro-3-nitrobenzoic acid and pyridazine

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.087
Data-to-parameter ratio = 13.5

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

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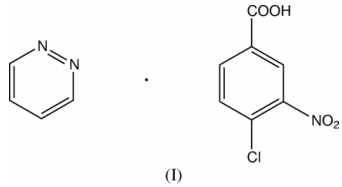
In the title compound, $C_7H_4ClNO_4\cdot C_4H_4N_2$, the two components are connected by an O—H···N hydrogen bond. C—H···O hydrogen bonds connect the $C_7H_4ClNO_4\cdot C_4H_4N_2$ units to afford a macrocycle with graph-set descriptor $R_4^4(16)$; this ring is located on an inversion center.

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, molecules of pyridazine and 4-chloro-3-nitrobenzoic acid are held together by a short O—H···N hydrogen bond (Table 2), forming a $C_7H_4ClNO_4\cdot C_4H_4N_2$ unit (Fig. 1).



(I)

All atoms of the unit except the O atoms of the nitro group are almost coplanar; the dihedral angle between the carboxyl group and the benzene ring is 1.1 (3)°, and 8.53 (11)° between the planes of the pyridazine and benzene rings. The nitro group is twisted out of the benzene ring plane, with a dihedral angle of 50.27 (13)°. C—H···O hydrogen bonds (Table 2) between the pyridazine ring and the carboxyl group connect the $C_7H_4ClNO_4\cdot C_4H_4N_2$ units, resulting in a centrosymmetric macrocycle with graph-set descriptor $R_4^4(16)$ (Bernstein *et al.*, 1995). These macrocyclic units are stacked along the a axis (Fig. 2). A short contact [Cl···O3^{ii}, 3.206 (2) Å; symmetry code: (ii) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$] is observed between macrocycles.

Experimental

Crystals of (I) were obtained by slow evaporation from a benzene solution of pyridazine with 4-chloro-3-nitrobenzoic acid in a molar ratio of 1:1.

Crystal data

$C_7H_4ClNO_4\cdot C_4H_4N_2$
M_r = 281.65
Monoclinic, $P2_1/n$
a = 3.7659 (4) Å
b = 27.486 (6) Å
c = 11.7723 (15) Å
β = 96.677 (10)°
V = 1210.3 (3) Å^3
Z = 4
D_x = 1.546 Mg m^{-3}
Mo Kα radiation
Cell parameters from 25 reflections
θ = 11.4–12.4°
μ = 0.33 mm^{-1}
T = 298 K
Prism, pale brown
0.35 × 0.30 × 0.25 mm

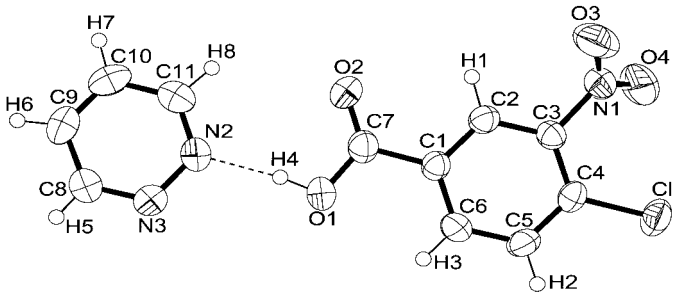


Figure 1
ORTEP-3 (Farrugia, 1997) drawing of (I) with the atom-labeling. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. An O—H···N hydrogen bond is indicated by a dashed line.

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.873, T_{max} = 0.921
4090 measured reflections
2767 independent reflections
1582 reflections with $I > 2\sigma(I)$

R_{int} = 0.022
θ_{max} = 27.5°
$h = -1 \rightarrow 4$
$k = 0 \rightarrow 35$
$l = -15 \rightarrow 15$
3 standard reflections
every 97 reflections
intensity decay: 1.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.087
S = 1.19
2767 reflections
205 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o) + 0.00013|F_o|^2]$

$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 9.3 (16) × 10^{-7}

Table 1
Selected geometric parameters (Å).

Cl—C4	1.725 (2)	N1—C3	1.474 (3)
O1—C7	1.307 (3)	N2—N3	1.333 (3)
O2—C7	1.206 (3)	N2—C11	1.309 (3)
O3—N1	1.218 (3)	N3—C8	1.315 (3)
O4—N1	1.209 (3)	C1—C7	1.499 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H4···N2	0.89 (3)	1.74 (3)	2.629 (3)	172 (3)
C10—H7···O2^{i}	0.91 (2)	2.55 (2)	3.310 (3)	142.4 (18)

Symmetry code: (i) $2-x, 1-y, 2-z$.

H atoms were found in difference Fourier maps and refined isotropically. Refined distances: C—H = 0.89 (3)–0.95 (2) and O—H = 0.89 (3) Å.

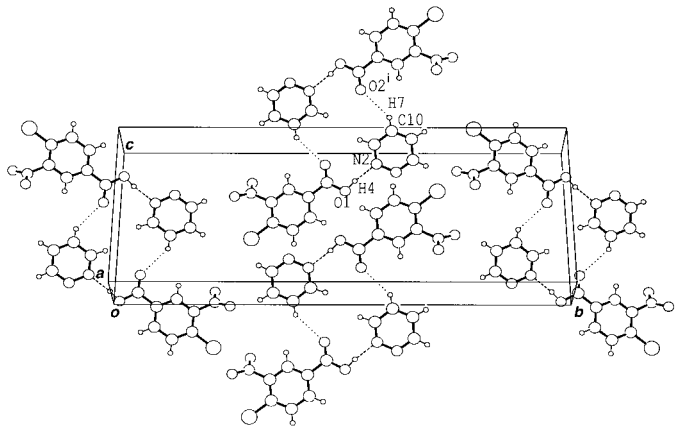


Figure 2
Packing diagram, showing the macrocycle formed *via* O—H···N and C—H···O hydrogen bonds indicated by dashed and dotted lines, respectively [symmetry code: (i) $2-x, 1-y, 2-z$].

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: *teXsan* for Windows; 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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