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3-Cyanopyridine–2-chloro-4-nitrobenzoic acid (1/1)

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In the title compound, $C_6H_4N_2.C_7H_4ClNO_4$, the two components are connected by an O—H···N hydrogen bond. In the crystal structure, the molecules are stacked in columns held together by three different π–π stacking interactions.

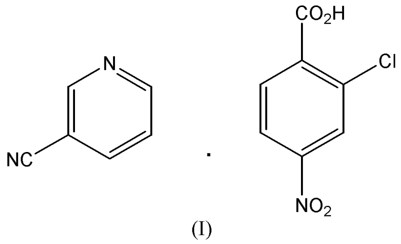
Key indicators

Single-crystal X-ray study
T = 300 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.119
Data-to-parameter ratio = 16.0

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

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*, 2002*a*,*b*,*c*). To our knowledge, this is the first structural report of a carboxylic acid–3-cyanopyridine system.



(I)

In the crystal structure, molecules of 3-cyanopyridine and 2-chloro-4-nitrobenzoic acid are held together by an O—H···N hydrogen bond [O2—H1 = 0.97 (4) Å, H1···N2 = 1.68 (4) Å, O2···N2 = 2.641 (3) Å and O2—H1···N2 = 176 (4)°], forming a 3-cyanopyridine–2-chloro-4-nitrobenzoic acid (1/1) unit, (I) (Fig. 1). No other significant hydrogen bonds are found. In the crystal structure (Fig. 2), the molecules are stacked in columns held together by three different π–π stacking interactions, two of which are strong and the other weak. The interplanar and centroid–centroid separations between the pyridine rings related by an inversion center are 3.316 (2) and 3.786 (2) Å, respectively, and those between the benzene rings related by an inversion center are 3.851 (2) and

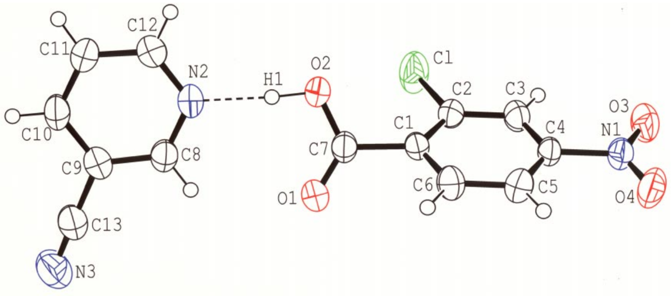


Figure 1
An *ORTEP*-3 (Farrugia, 1997) drawing of (I), with the atom-labeling. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. An O—H···N hydrogen bond is indicated by a dashed line.

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3.997 (2) Å. The pyridine ring and the benzene ring at ($\frac{1}{2}+x$, $\frac{1}{2}-y$, $\frac{1}{2}+z$) are almost parallel; the dihedral angle between them is 3.21 (15)° and the interplanar and centroid–centroid separations are *ca* 3.39 and 3.990 (2) Å, respectively.

Experimental

Crystals of (I) were obtained by slow evaporation of an aqueous solution of 3-cyanopyridine with 2-chloro-4-nitrobenzoic acid in a 1:1 molar ratio.

Crystal data

$C_6H_4N_2{\cdot}C_7H_4ClNO_4$
$M_r = 305.67$
Monoclinic, $P2_1/n$
$a = 10.012$ (3) Å
$b = 10.318$ (3) Å
$c = 13.974$ (2) Å
$\beta = 110.767$ (15)°
$V = 1349.7$ (6) Å^3
$Z = 4$

$D_x = 1.504$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 11.5$–$12.1°$
$\mu = 0.30$ mm^{-1}
$T = 300$ K
Prism, colorless
0.40 × 0.30 × 0.30 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.898$, $T_{\max} = 0.915$
3633 measured reflections
3103 independent reflections
1692 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.021$
$\theta_{\max} = 27.5°$
$h = -1 \rightarrow 13$
$k = 0 \rightarrow 13$
$l = -18 \rightarrow 17$
3 standard reflections every 97 reflections intensity decay: 4.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.119$
$S = 1.01$
3103 reflections
194 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.45P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl—C2	1.729 (3)	C1—C7	1.509 (3)
O1—C7	1.200 (3)	N2—C8	1.326 (3)
O2—C7	1.300 (3)	N2—C12	1.330 (3)
O3—N1	1.221 (3)	N3—C13	1.140 (4)
O4—N1	1.220 (3)	C9—C13	1.437 (4)
N1—C4	1.476 (3)		
C8—N2—C12	118.4 (2)	O1—C7—O2	124.5 (2)

The H atom attached to the O atom was refined isotropically. Other H atoms were treated as riding atoms, with C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

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.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

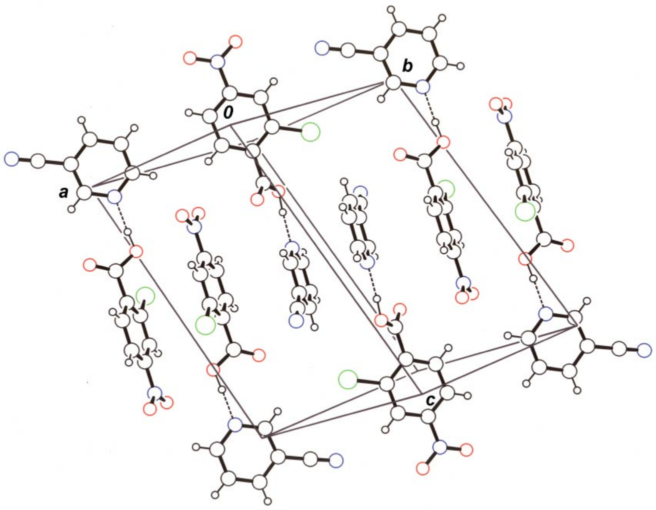

Figure 2
Packing diagram of (I), showing O—H···N hydrogen bonds (dashed lines) and π–π stacking interactions.

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