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NTA–1,10-phenanthroline–water (1/1/1)

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Key indicators

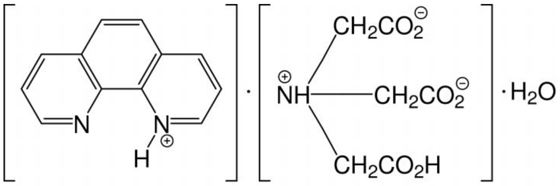
Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.102
Data-to-parameter ratio = 15.0

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

The title complex, $C_6H_9NO_6 \cdot C_{12}H_8N_2 \cdot H_2O$, is composed of nitrilotriacetic acid (NTA), 1,10-phenanthroline and water in the molar ratio of 1:1:1. The crystal structure, determined at 180 K, contains an extensive network of hydrogen bonds and both intra- and intermolecular H-atom transfers are observed.

Comment

With a view towards the prediction of solid-state organic crystal structures, we are currently studying the supramolecular structures of cocrystals containing organic acids and organic bases. Nitrilotriacetic acid (NTA), which posesses three carboxylic acid groups, is commonly found as a multidentate ligand in many metal–chelate compounds (Kaneyoshi *et al.*, 1999; Martell, 1975). Organic cocrystals involving NTA, however, have not been reported previously. We describe here the structure of a cocrystal of NTA, incorporating the organic base 1,10-phenanthroline, determined at 180 K.



(I)

The asymmetric unit and atomic numbering scheme for (I) are shown in Fig. 1. An intramolecular hydrogen bond is observed in the acid molecule, although the interaction is somewhat distorted from linearity [N1···O1 2.631 (1) Å and N1—H1···O1 115.6 (13)°]. The location of H1 in a difference Fourier map suggests that intramolecular H-atom transfer occurs from the carboxylic acid group to the N atom [O1—C2 1.235 (2) Å and O2—C2 1.266 (2) Å]. Intermolecular H-atom transfer is also observed from the O5—C6—O6 carboxylic acid group to N2 in the phenanthroline base [O5—C6 1.248 (2) Å and O6—C6 1.253 (2) Å]. The O3—C4—O4 group is the only carboxylic acid group which remains protonated (Fig. 1).

Crinkled supramolecular tapes are formed along the *c* direction from acid molecules linked *via* $O4^{i}$—H100···O2 hydrogen bonds [symmetry code: (i) $-x+\frac{3}{2}$, $-y+\frac{1}{2}$, $z+\frac{1}{2}$]. Adjacent tapes are bound *via* hydrogen bonds through water molecules (Table 1) such that supramolecular sheets are formed parallel to the (100) plane (Fig. 2). The 1,10-phenanthroline molecule, furthermore, is bonded to O6 *via* an $N2^{ii}$—H2···O6 hydrogen bond [Table 1; symmetry code: (ii) $1-x$, y,

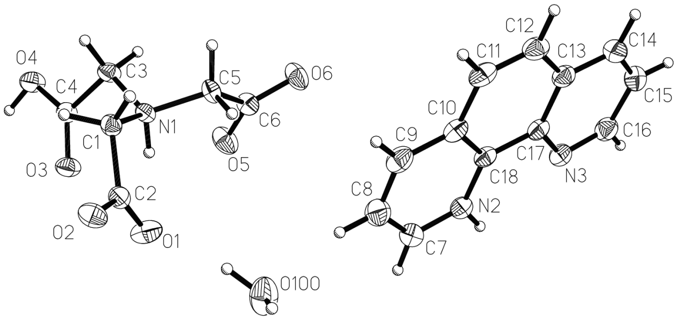


Figure 1
The molecular unit of (I) showing displacement ellipsoids at the 50% probability level (*XP*; Sheldrick, 1993).

$\frac{1}{2} - z$]. The 1,10-phenanthroline molecules adopt a face-to-face stacking arrangement along the *c* direction.

Experimental

Nitrilotriacetic acid and 1,10-phenanthroline were obtained from Aldrich. The title complex was prepared by slow evaporation of an acid–base (1:1) solution in *N*,*N*-dimethylformamide (DMF) at room temperature.

Crystal data

$C_6H_8NO_6 \cdot C_{12}H_9N_2 \cdot H_2O$
$M_r = 389.36$
Orthorhombic, *Pbcn*
$a = 22.7045$ (6) Å
$b = 11.1044$ (3) Å
$c = 14.2366$ (2) Å
$V = 3589.33$ (14) Å^3
$Z = 8$
$D_x = 1.441$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 22 808 reflections
$\theta = 1.0$–27.5°
$\mu = 0.11$ mm^{-1}
$T = 180$ (2) K
Block, colourless
0.46 × 0.46 × 0.30 mm

Data collection

Nonius KappaCCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.932$, $T_{\max} = 0.967$
22 344 measured reflections
4114 independent reflections
3414 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\max} = 27.5°$
$h = -21 \rightarrow 29$
$k = -11 \rightarrow 14$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.102$
$S = 1.03$
4114 reflections
275 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 1.3895P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.013$
$\Delta\rho_{\max} = 0.39$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O4—H100$\cdots$O2$^{\rm i}$	1.00 (2)	1.49 (2)	2.4897 (13)	175 (2)
N1—H1$\cdots$O1	0.946 (15)	2.080 (16)	2.6310 (14)	115.6 (13)
N2—H2$\cdots$O6$^{\rm ii}$	0.954 (15)	1.801 (16)	2.6874 (15)	153.2 (17)
O100—H101$\cdots$O1	0.95 (2)	1.90 (2)	2.8119 (17)	161 (2)
O100—H102$\cdots$O5$^{\rm iii}$	0.95 (2)	1.87 (2)	2.8072 (16)	171 (3)

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

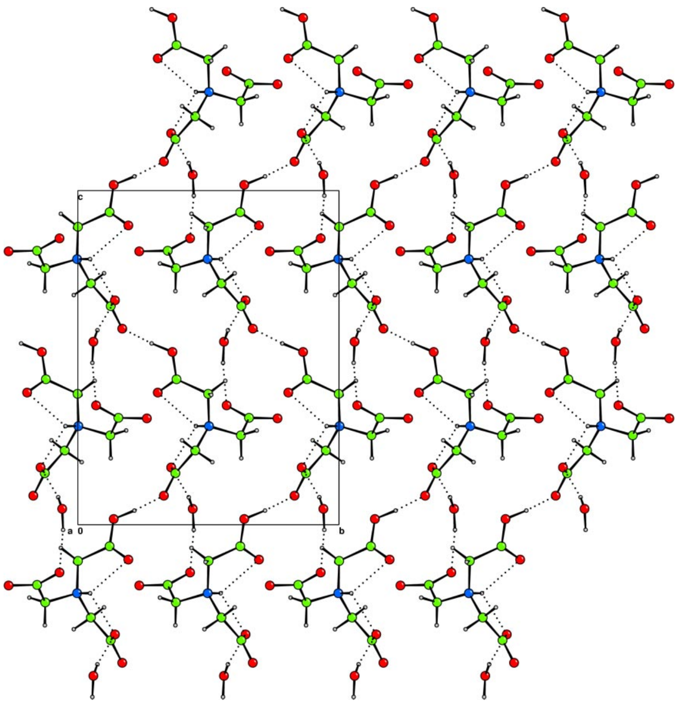


Figure 2
Projection onto (100) showing the NTA–water supramolecular sheet (*CAMERON*; Watkin *et al.*, 1996).

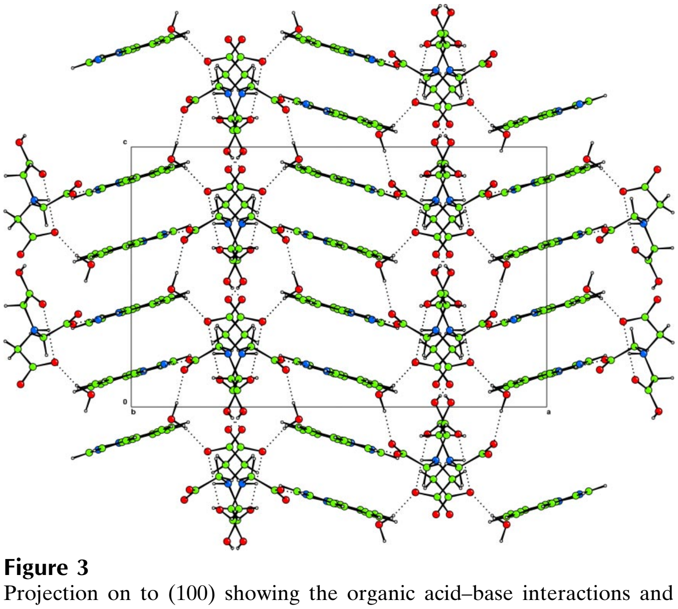


Figure 3
Projection on to (100) showing the organic acid–base interactions and face-to-face stacking of 1,10-phenanthroline molecules (*CAMERON*; Watkin *et al.*, 1996).

All H atoms not bonded to carbon were located from difference Fourier maps and were refined with isotropic displacement parameters. H100 was refined without restraint. H1 and H2 were refined with the N—H distances restrained to be equivalent with an s.u. of 0.01 Å. H101 and H102, associated with the water molecule, were refined with the O—H distances restrained to be equivalent with an s.u. of 0.01 Å, and the H101$\cdots$H102 distance was restrained to be 1.633 times the value of the O—H distance, with an s.u. of 0.02 Å; these restraints ensure a reasonable geometry for the water molecule. All H atoms bonded to C atoms were placed geometrically and refined using a riding model with the $U_{\rm iso}$ values for each H atom taken as 1.2 $U_{\rm eq}$ of the carrier atom.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1993); software used to prepare material for publication: *SHELXL*97.

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References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Kaneyoshi, M., Bond, A. D. & Jones, W. (1999). *Acta Cryst.* C**55**, 1260–1262.

Martell, A. E. (1975). *Pure Appl. Chem.* **44**, 81–113.

Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *HKL DENZO* and *SCALEPACK*. University of Texas, Southwestern Medical Center at Dallas, USA.

Sheldrick, G. M. (1993). *XP*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.

Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.