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The 1:1 adduct of 3,5-dinitrobenzoic acid and quinoline

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
R factor = 0.038
wR factor = 0.111
Data-to-parameter ratio = 19.8

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

In the crystal structure of the title adduct, $C_7H_4N_2O_6 \cdot C_9H_7N$, the 3,5-dinitrobenzoic acid and quinoline molecules are linked into a chain along the *b* axis by intermolecular O—H···N and C—H···O hydrogen bonds. Adjacent chains are linked *via* C—H···O hydrogen bonds into layers parallel to the *ab* plane. A three-dimensional network is formed *via* π–π interactions.

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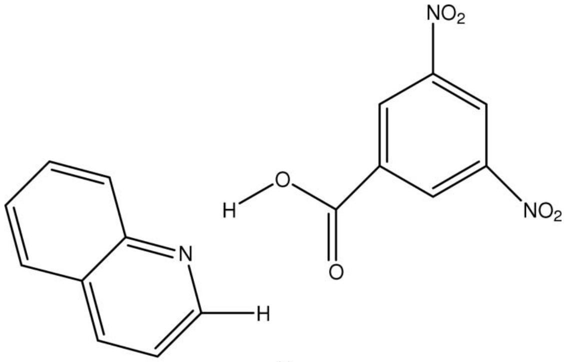
Comment

The adducts of organic acids with amine bases in the solid state have been of interest due to their hydrogen-bond interactions (Coupar *et al.*, 1997; Sobczyk *et al.*, 2000; Smith, Wermuth & White, 2001; Smith, Wermuth, Bott *et al.*, 2001; Fun *et al.*, 2003; Chantrapromma & Fun, 2004) and their important role in biological systems (Blow, 1976). Following our interest in the hydrogen-bonding interactions and phase transitions due to hydrogen bonding (Usman *et al.*, 2001; Fun *et al.*, 2003; How *et al.*, 2005), we have investigated the interaction between 3,5-dinitrobenzoic acid and several amine bases such as 2,4′-bipyridine (Chantrapromma *et al.*, 2002*b*), quinuclidine (Chantrapromma *et al.*, 2004) and hexamethylenetetramine (Fun *et al.*, 2003; Chantrapromma *et al.*, 2006; How *et al.*, 2005). The H-transfer process occurs in these adducts and the adduct of 3,5-dinitrobenzoic acid with hexamethylenetetramine undergoes a phase transition (Fun *et al.*, 2003; How *et al.*, 2005). We have been investigating the phase transition due to hydrogen bonding in adducts of acids and amines using single-crystal structure determination (Chantrapromma *et al.*, 2002*a*,*b*, 2004, 2006; Chantrapromma & Fun 2004; Fun *et al.*, 2003; How *et al.*, 2005). In our ongoing systematic studies on the interactions between 3,5-dinitrobenzoic acid and amine bases, the title compound, (I), was prepared.



(I)

The asymmetric unit of (I) (Fig. 1) comprises one 3,5-dinitrobenzoic acid and one quinoline molecule. No proton

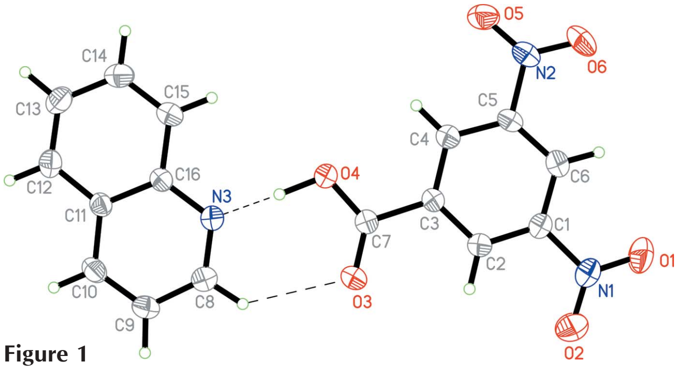


Figure 1
The asymmetric unit of (I), showing 80% probability displacement ellipsoids and the atomic numbering. Dashed lines indicate hydrogen bonds.

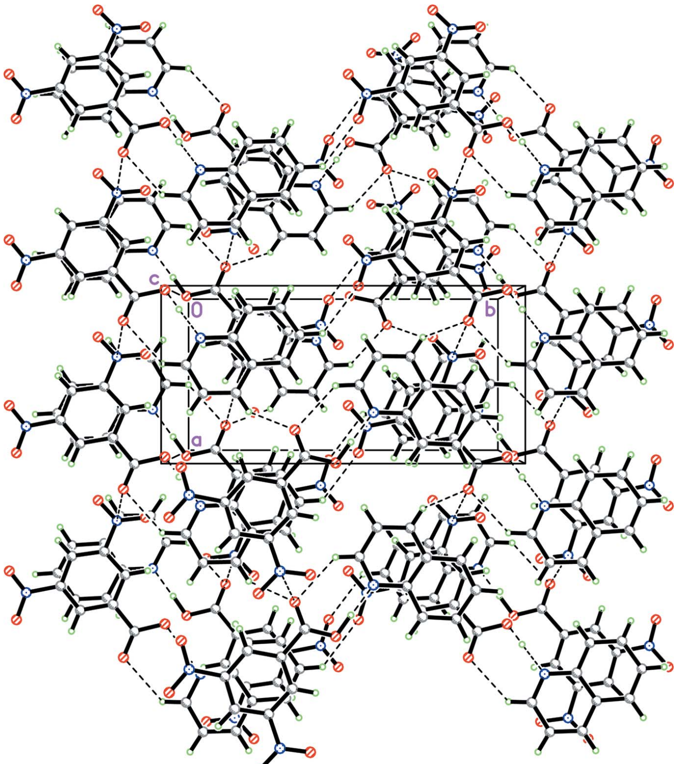


Figure 2
The crystal packing of (I), viewed down the *c* axis, illustrating the intermolecular hydrogen bonds (dashed lines).

transfer is observed in (I), which is in contrast with the similar compounds of 3,5-dinitrobenzoic acid with 1,4-diazabicyclo[2.2.2]octane (Chantrapromma & Fun 2004), quinuclidine (Chantrapromma *et al.*, 2004) and hexamethylenetetramine (Chantrapromma *et al.*, 2006; Fun *et al.*, 2003; How *et al.*, 2005).

All bond distances and angles in (I) show normal values (Allen *et al.*, 1987). Selected bond lengths are given in Table 1. The quinoline ring system is planar within ±0.011 (1) Å. In the 3,5-dinitrobenzoic acid molecule, the two nitro and carboxyl groups are twisted slightly away from the plane of the benzene ring with torsion angles O5—N2—C5—C4 of −3.77 (11)°, O1—N1—C1—C6 of 2.41 (12)° and C4—C3—C7—O4 of 2.58 (11)°. The dihedral angle between the benezene ring and the quinoline ring system is 7.20 (3)°. The geometric parameters in 3,5-dinitrobenzoic acid are comparable to those in its adducts with 1,4-diazabicyclol2.2.2]octane (Chantrapromma & Fun, 2004), quinuclidine (Chantrapromma *et al.*, 2004) and hexamethylenetetramine (Chantrapromma *et al.*, 2006).

In the crystal structure (Fig. 2), the 3,5-dinitrobenzoic acid and quinoline molecules are linked into a chain along the *b* axis by O4—H1O4···N3, C8—H8···O3 and C13—H13···O2^{ii} hydrogen bonds (Table 2); the screw-related molecules in adjacent chains are linked *via* C10—H10···O3^{i} hydrogen bonds (symmetry codes are given in Table 2) into layers parallel to the *ab* plane (Fig. 2). The benzene rings of the 3,5-dinitrobenzoic acid and quinoline molecules in adjacent layers are stacked with a centroid···centroid distance of 3.6202 (5) Å, indicating weak *π*-*π* interactions.

Experimental

Compound (I) was prepared by mixing thoroughly equimolar amounts of quinoline (0.60 ml, 5 mmol) and 3,5-dinitrobenzoic acid (1.06 g, 5 mmol) in ethanol (100 ml) with a few drops of water. The mixture was warmed until a clear solution was obtained. The solution was then filtered and left to evaporate slowly at ambient temperature. Colourless block-shaped single crystals of (I) were obtained from the solution after a few days (m.p. 419–421 K).

Crystal data

$C_7H_4N_2O_6 \cdot C_9H_7N$
M_r = 341.28
Monoclinic, $P2_1/c$
a = 7.0358 (1) Å
b = 14.3980 (3) Å
c = 14.6002 (2) Å
β = 90.175 (1)°
V = 1479.01 (4) Å^3
Z = 4
D_x = 1.533 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 5352 reflections
θ = 2.0–32.5°
μ = 0.12 mm^{-1}
T = 100.0 (1) K
Block, colourless
0.52 × 0.42 × 0.39 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.852, T_{max} = 0.955
31914 measured reflections
5352 independent reflections
4753 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 32.5°
$h = -10 \rightarrow 10$
$k = -21 \rightarrow 21$
$l = -22 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.111
S = 1.03
5352 reflections
270 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 0.323P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—N1	1.2255 (10)	O6—N2	1.2305 (9)
O2—N1	1.2252 (11)	N1—C1	1.4723 (10)
O3—C7	1.2225 (9)	N2—C5	1.4731 (10)
O4—C7	1.3081 (10)	N3—C8	1.3213 (10)
O5—N2	1.2252 (10)	N3—C16	1.3722 (10)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O4—H1O4⋯N3	0.854 (16)	1.749 (16)	2.6019 (9)	176 (2)
C8—H8⋯O3	0.989 (14)	2.465 (14)	3.2237 (10)	133 (1)
C10—H10⋯O3^{i}	0.958 (14)	2.454 (14)	3.2530 (10)	141 (1)
C13—H13⋯O2^{ii}	0.977 (14)	2.512 (14)	3.2129 (12)	129 (1)

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

All H atoms were located in a difference map and refined isotropically. As the O4—H1O4 distance was long [1.06 (2) Å], it was restrained to 0.85 (1) Å during the final cycles of refinement. The C—H distances lie in the range 0.94 (1)–0.99 (1) Å.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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