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Quinoline *N*-oxide dihydrate from powder data

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Key indicators

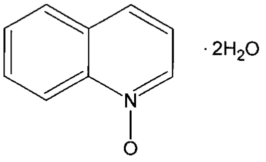
Powder X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
H-atom completeness 64%
R factor = 0.028
wR factor = 0.040

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

The structure of quinoline *N*-oxide dihydrate, $C_9H_7NO{\cdot}2H_2O$, has been determined from X-ray powder diffraction data, using grid search technique and a bond-restrained Rietveld refinement. The structure features an interesting system of co-operative hydrogen bonds, which apparently involves all four 'active' H atoms, and is based on the infinite chains formed by two alternating symmetry-independent water molecules. The chains are further interlinked through two more symmetry-independent hydrogen bonds which make use of the *N*-oxide O atom as hydrogen-bond acceptor.

Comment

Heteroaromatic *N*-oxides are characterized by the presence of a donor–acceptor bond between the N and O atoms, which is formed by the overlap of the non-bonding electron pair of the N atom with an empty orbital of the O atom. The N—O group in heteroaromatic systems can exhibit both electron-donor and electron-acceptor properties, depending upon the molecular structure and conditions. Thus, *N*-oxides react more readily with both electrophilic and nucleophilic agents than their parent compounds, which makes them convenient intermediates (Albini & Pietra, 1991). Steric accessibility of the N—O group also facilitates formation of complexes with different electron-deficient compounds. Moreover, *N*-oxides show a great variety of biological activities.


(I)

The molecular structure of the quinoline *N*-oxide as well as the O atoms of two symmetry-independent water molecules are shown in Fig. 1. All bond lengths and angles are within expected ranges.

The extensive hydrogen-bond system in the structure apparently involves all four symmetry-independent 'active' H atoms (Fig. 2). Two of them are responsible for the formation of infinite chains running along the $[10\overline{1}]$ direction of the crystal, and formed of alternating O*W*1 and O*W*2 molecules [$OW1\cdots OW2^{i}$ 2.660 (9) Å and $OW1\cdots OW2^{ii}$ 2.875 (9) Å; symmetry codes: (i) $x, y, z+1$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2}$]. Those OH groups of each of the water molecules, which are not involved within water chains, are most probably used as hydrogen-bond donors in the O—H···O bonds involving the *N*-oxide atom O6 as an acceptor. Thus, the O6 atom is, in fact, connected to the O*W*1 and O*W*2 molecules of two adjacent

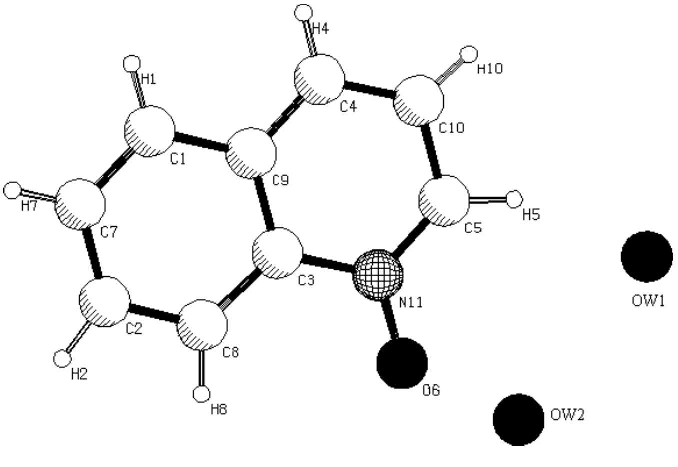


Figure 1
The molecular structure of (I), with the atomic numbering.

chains [O*W*1···O6 2.601 (7) Å and O*W*2···O6 2.705 (8) Å], which effectively interlinks the chains into a two-dimensional hydrogen-bonded system.

Experimental

Compound (I) was prepared in polycrystalline form according to the procedure of Ochiai (1967). The IR spectrum was measured in KBr using a Specord M-80 spectrometer. Electronic spectra were obtained using Specord UV–vis in ethanol (96%) [λ nm (log ε): 210 (*sh*), 231 (4.60), 325 (3.84), 3250 (*sh*), 325 (3.82), 335(3.89), 350 (*sh*, 3.83), 40 (*sh*, 3.79)], chloroform [λ nm (log ε): 250 (*sh*), 325 (3.82), 335 (3.89), 350 (*sh*, 3.83)] and dichloromethane [λ nm (log ε): 247 (4.03), 327 (*sh*, 3.77), 339 (3.80), 355 (*sh*, 3.69)]. IR and electronic spectra are in agreement with literature data (Ochiai, 1967). 1310 cm^{-1} and 1267 cm^{-1} correspond to the N—O bond vibrations and the broad band 3200–3600 cm^{-1} is caused by water.

Crystal data

$C_9H_7NO \cdot 2H_2O$
$M_r = 181.19$
Monoclinic, $P2_1/n$
$a = 9.484$ (3) Å
$b = 16.235$ (5) Å
$c = 6.907$ (2) Å
$\beta = 118.25$ (2)°
$V = 936.8$ (5) Å^3
$Z = 4$
$D_x = 1.285$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 29 reflections
$\theta = 5.4$–17.1°
$\mu = 0.81$ mm^{-1}
$T = 293$ (2) K
Light grey
Specimen shape: flat sheet
12 × 12 × 2 mm
Particle morphology: no specific habit

Data collection

X-ray powder DRON-4.07 diffractometer
Specimen mounting: the powder was sprinkled on the sample holder.
Specimen mounted in reflection mode
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 10$
$l = -4 \rightarrow 3$
$2\theta_{\min} = 10.0$, $2\theta_{\max} = 60.0$°
Increment in $2\theta = 0.02$°

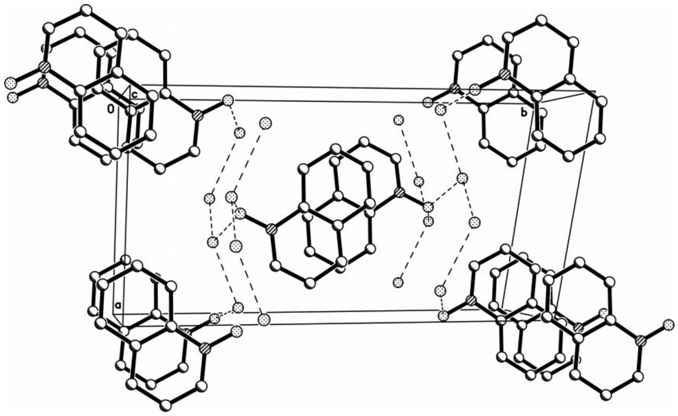


Figure 2
The crystal packing for the crystal of quinoline *N*-oxide dihydrate. The short O···O contacts corresponding to the hydrogen bonds are shown as dashed lines.

Refinement

Refinement on $I_{\rm net}$
$R_p = 0.028$
$R_{\rm wp} = 0.040$
$R_{\rm exp} = 0.012$
$S = 3.35$
$2\theta_{\min} = 10$, $2\theta_{\max} = 60$°
Increment in $2\theta = 0.02$°
Wavelength of incident radiation: 1.5418 Å
Profile function: split-type pseudo-Voigt (Toraya, 1986)
126 parameters
H atoms treated by a mixture of independent and constrained refinement
$(\Delta/\sigma)_{\max} = 0.02$
Preferred orientation correction: Spherical harmonics expansion (Ahtee *et al.*, 1989) up to the 6th order

Table 1
Selected geometric parameters (Å, °).

O6—N11	1.269 (7)	O6···O*W*2	2.705 (8)
N11—C3	1.431 (7)	O*W*1—O*W*2$^{\rm i}$	2.660 (9)
N11—C5	1.33 (1)	O*W*1···O*W*2$^{\rm ii}$	2.875 (9)
O6···O*W*1	2.601 (7)		
C3—N11—O6	119.4 (5)	C3—N11—C5	117.6 (7)
C5—N11—O6	123.0 (7)		

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

X-ray powder diffraction patterns were obtained with a Guinier–de Wolff camera and the X-ray powder diffraction system DRON-4.07, equipped with standard resolution goniometer GUR-9 and scintillation counter. From the Guinier photographs, a monoclinic unit cell was determined with the indexing program *TREOR* (Werner *et al.*, 1985), with $M_{20} = 39$ and $F_{29} = 59$ (0.0076, 65) using the first 29 peak positions. The second pattern was measured with the DRON system in the range 10–60° and was used for structure solution and refinement. The powder was sprinkled on the sample to avoid preferred orientation. During the exposures, the specimen was spun in its plane to improve particle statistics. A correct solution was found in the monoclinic space group $P2_1/n$. The strength of the restraints was a function of interatomic separation and, for intramolecular bond lengths, corresponded to an r.m.s. deviation of 0.03 Å. An additional restraint was applied to the planarity of the quinoline *N*-oxide fragment. Isotropic atomic displacement parameters were refined for all non-H atoms. H atoms were placed in geometrically calculated positions and allowed to refine using bond restraints, with a common isotropic displacement parameter $U_{\rm iso}$(H) fixed at 0.05 Å^2. The H atoms of lattice water molecules were not included in the refinement. The diffraction profiles are shown in Fig. 3.

Acta Cryst. (2002). E**58**, o920–o922 Svetlana N. Ivashevskaja *et al.* • $C_9H_7NO \cdot 2H_2O$ **o921**

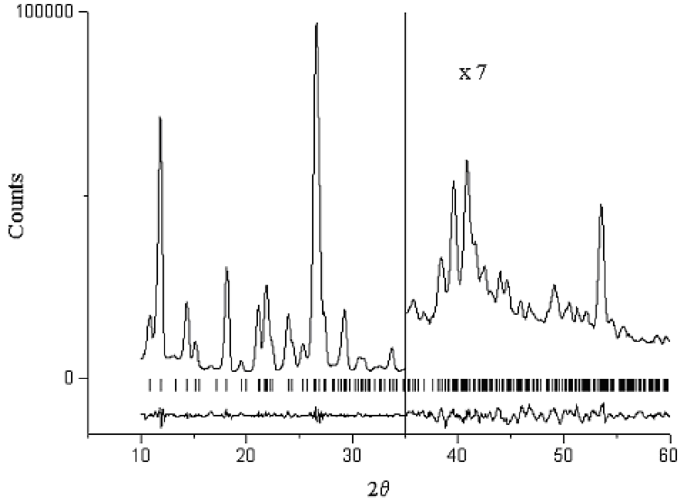


Figure 3
The Rietveld plot for (I), showing the observed and difference profiles. The reflection positions are shown above the difference profile.

Data collection: local program; data reduction: local program; program(s) used to solve structure: *MRIA* (Zlokazov & Chernyshev, 1992); program(s) used to refine structure: *MRIA*; molecular graphics: *PCMODEL* (Serena Software, 1999); software used to prepare material for publication: *MRIA* and *PARST* (Nardelli, 1983).

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o922 Svetlana N. Ivashevskaja *et al.* • $C_9H_7NO{\cdot}2H_2O$ *Acta Cryst.* (2002). E**58**, o920–o922