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The 1:1 complex of hydroquinone and 3-methy-4-nitropyridine 1-oxide

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Key indicators

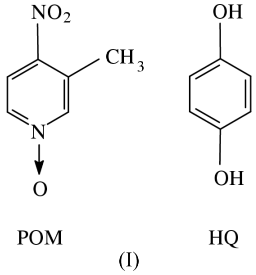
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.127
Data-to-parameter ratio = 11.6

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 complex, $C_6H_6O_2 \cdot C_6H_6N_2O_3$, the molecules are linked by intermolecular hydrogen bonds between the O—H and N—O groups. The O atom of the *N*-oxide group of 3-methyl-4-nitropyridine 1-oxide acts as an acceptor for hydrogen bonds from O—H groups of two symmetry-related hydroquinone molecules [O···O 2.732 (2) and 2.810 (2) Å]. The angles between the rings of the 3-methyl-4-nitropyridine 1-oxide and two hydroquinone molecules are 6.1 (3) and 0.6 (3)°. The crystal structure exhibits overlap between the aromatic rings of the molecules in the [11$\bar{1}$] direction.

Comment

The design of organic crystals formed by non-covalent intermolecular interactions has been one of the main goals of crystal engineering (Schmidt, 1971; Desiraju, 1995). This type of solid has attracted great interest because of its increasing technological applications (Lehn, 1990). The present work is part of a series of structural studies on molecular complexes, formed by hydrogen bonds, with potential non-linear optical applications (Moreno-Fuquen *et al.*, 1998). The title molecular complex, (I), is formed by hydroquinone (HQ) (Lindeman *et al.*, 1981; Cambridge Structural Database refcode HYQUIN05; Allen *et al.*, 1991), which crystallizes in a centrosymmetric space group, and 3-methyl-4-nitropyridine 1-oxide (POM) (Hamzaoui *et al.*, 1996; refcode MNPYDO03), whose non-linear optical response has already been reported (Zyss *et al.*, 1981). Although the title complex is centrosymmetric, information about its crystal structure is important for the study of the general behavior of POM with respect to its formation of hydrogen-bond complexes. The title cocrystal is held together by hydrogen bonds between the O—H groups of two different HQ molecules and the N—O group of POM. The O···O distances are 2.732 (2) Å for O1···O5 and 2.810 (2) Å for O1···O4. The O1···HO5—O5 angle is 156 (2)° and the O1···HO4—O4 angle is 168 (3)°. A view of the hydrogen-bonded complex is shown in Fig. 1 and the unit cell contents are shown in Fig. 2.



(I)

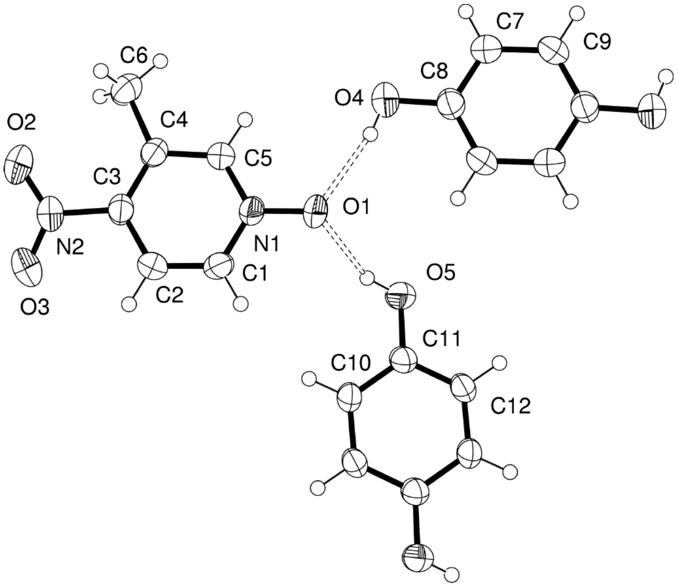


Figure 1
An *ORTEP*-3 (Farrugia, 1997) plot of the title complex, with the atomic labeling scheme. Displacement ellipsoids are plotted at the 50% probability level.

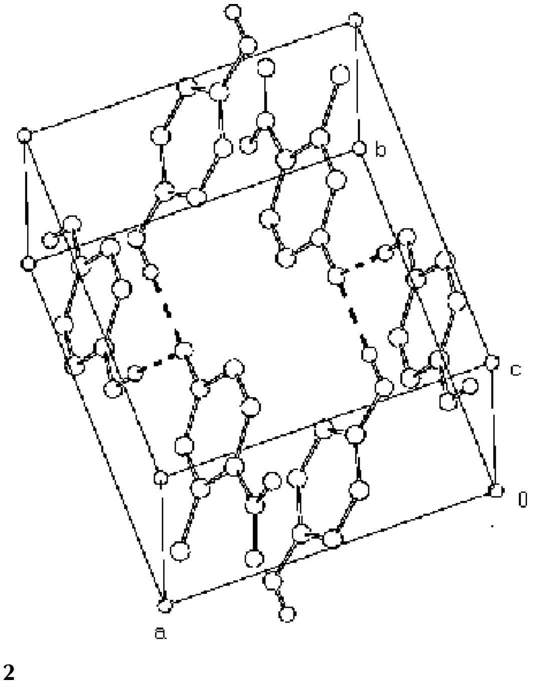


Figure 2
A *ZORTEP* (Zsolnai, 1995) diagram of (I), showing the crystal packing.

The presence of hydrogen bonds induces some changes in the structure of the molecules in the complex with respect to the structures of the separate components. Thus, the C—O bonds in (I) (Table 1) are 1.375 (2) and 1.371 (2) Å for C8—O4 and C11—O5, respectively, while in free hydroquinone, the bond length is 1.392 (4) Å. On the other hand, the bond lengths and angles for POM remain similar in the complex (Hamzaoui *et al.*, 1996). The interplanar angles between the rings of POM and the two HQ molecules are 6.1 (3) and 0.6 (3)°. The aromatic rings of POM and HQ show an ABAB disposition and are overlapped, with a mean distance between the rings of 3.47 (2) Å along the $[11\overline{1}]$ direction. The complex consists of zigzag chains, with molecules linked by intermolecular hydrogen bonds.

Experimental

Crystals of the title POM–HQ complex, (I), were obtained by slow evaporation from an equimolar solution of POM and HQ in acetonitrile. Orange–red crystals with a melting point of 384 (1) K were obtained. The initial reagents were purchased from Aldrich and were used without additional purification.

Crystal data

$C_6H_6O_2\cdot C_6H_6N_2O_3$
$M_r = 264.24$
Triclinic, $P\overline{1}$
$a = 7.8593\ (16)$ Å
$b = 8.429\ (3)$ Å
$c = 9.039\ (3)$ Å
$\alpha = 84.39\ (3)^\circ$
$\beta = 89.92\ (2)^\circ$
$\gamma = 88.91\ (3)^\circ$
$V = 595.8\ (3)$ Å^3
$Z = 2$
$D_x = 1.473$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 8$–20°
$\mu = 0.12$ mm^{-1}
$T = 293\ (2)$ K
Irregular block, orange–red
0.20 × 0.14 × 0.14 mm

Data collection

Rigaku AFC-7*S* diffractometer
$\omega/2\theta$ scans
2250 measured reflections
2091 independent reflections
1848 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$
$\theta_{\rm max} = 25.0^\circ$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 0$
$l = -10 \rightarrow 10$
3 standard reflections
frequency: 150 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.127$
$S = 1.04$
2091 reflections
181 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.077P)^2 + 0.1509P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.29$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.034 (7)

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.346 (2)	C11—O5	1.370 (2)
C5—N1	1.346 (2)	O4—HO4	0.82 (3)
N1—O1	1.3165 (17)	O5—HO5	0.83 (2)
C8—O4	1.375 (2)		
N1—C1—C2	119.55 (14)	O4—C8—C9^{i}	122.79 (16)
N1—C5—C4	122.49 (15)	O5—C11—C10	123.05 (15)
O1—N1—C5	120.00 (14)	O5—C11—C12	117.85 (14)
O1—N1—C1	119.01 (13)	C8—O4—HO4	106 (2)
C5—N1—C1	120.98 (14)	C11—O5—HO5	113.9 (16)
O4—C8—C7	117.43 (17)		
N1—C1—C2—C3	−1.6 (3)	C9—C7—C8—O4	−178.64 (16)
C4—C5—N1—O1	−179.30 (14)	C12^{ii}—C10—C11—O5	178.54 (15)
C2—C1—N1—O1	−179.41 (14)	O5—C11—C12—C10^{ii}	−178.61 (15)
C2—C1—N1—C5	1.6 (2)		

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1-x, -y, 1-z$.

The ring and methyl H atoms were added at geometrically idealized positions and were allowed for as riding; C—H = 0.93–0.96 Å, $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$ of the carrier atom. Hydroxyl atoms HO4 and HO5

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were located from a Fourier difference map and their coordinates were refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *ZORTEP* (Zsolnai, 1995); software used to prepare material for publication: *SHELXL*97.

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