Furthermore, the dimeric units act as a bridge between the linear chains, through two weak hydrogen bonds between a carbonyl oxygen and an amino group [$N \cdots O = 3 \cdot 202$ (7), $H \cdots O = 2 \cdot 34$ Å, $\angle N—H \cdots O = 150°$].

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Structure of 3-Hydroxy-2-methyl-1,4-benzoquinone 4-Oxime Monohydrate

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Abstract. (I) $C_7H_7NO_3.H_2O$, $M_r = 171 \cdot 15$, monoclinic, $P2_1/c$, $a = 3 \cdot 931$ (2), $b = 13 \cdot 741$ (3), $c = 14 \cdot 214$ (3) Å, $\beta = 92 \cdot 08$ (2)°, $V = 767 \cdot 28$ Å^3, $Z = 4$, $D_x = 1 \cdot 482$ Mg m^{-3}, λ(Mo $K\alpha$) = $0 \cdot 71069$ Å, $\mu = 0 \cdot 11$ mm^{-1}, $F(000) = 360$, $T = 295$ K, $R = 0 \cdot 065$ for 1353 observed unique reflections with $I/\sigma(I) > 3 \cdot 0$. The molecule is essentially planar. The mean values of the two short and the four long bond distances in the hexagonal ring are $1 \cdot 327$ (18) and $1 \cdot 449$ (12) Å, respectively. In the oxime group the dimensions are C—N $1 \cdot 284$ (6), N—O $1 \cdot 364$ (6) Å and C—N—O $113 \cdot 8$ (4)°, while the phenolic and quinonic C—O bond distances are $1 \cdot 371$ (5) and $1 \cdot 247$ (5) Å, respectively. The crystal structure is stabilized by a network of hydrogen bonds formed between the molecules of the title compound both directly as well as through the solvate water molecules.

Introduction. The structural character of what are frequently referred to as 2- and 4-nitrosophenols has been the subject of discussion for a considerable time and has been investigated using spectroscopic and X-ray crystallographic techniques (Hadzi, 1956;

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Romers & Fischmann, 1960; Bartindale, Crowder & Morley, 1959; Romers, 1964). The X-ray crystallographic studies have demonstrated that invariably they are 1,2- or 1,4-quinone oximic in character and the isolation in two forms in some cases has been shown to be due to the orientation of the N—OH group relative to the C=O group. A predominantly quinone oximic structure has also been established from X-ray crystallographic studies of metal complexes of the deprotonated anionic form (Charalambous, Stoten & Henrick, 1989). A more complex situation exists in the case of the so-called nitrosoresorcinols which have an additional hydroxy group. No structural studies on these compounds have been reported and here we report on the structure of 3-hydroxy-2-methyl-1,4-benzoquinone 4-oxime (I) which exists as a monohydrate.

(I)

Experimental. 3-Hydroxy-2-methyl-1,4-benzoquinone 4-oxime was synthesized by nitrosation of 3-hydroxy-2-methylphenol using amyl nitrite/sodium

Table 1. *Data-collection parameters*

Crystal size (mm)	$0.40 \times 0.24 \times 0.24$
hkl range	$\pm 4, +16, \pm 16$
Scan type	$\theta/2\theta$
Scan speed (° s⁻¹)	0.05
2θ range (°)	6–50
Number of standard reflections	3
Variation in intensity (%)	< 5
Number of reflections	1635
Number of observed reflections [$I > 3\sigma(I)$]	1353
Number of variables	137
Max. shift/e.s.d.	0.02
Residual electron density (e Å⁻³)	0.31, −0.27
R	0.065
wR	0.078

Table 2. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for non-H atoms*

	x	*y*	*z*	U_{eq}
C(1)	0.4151 (13)	0.2417 (3)	0.1996 (3)	0.0371 (16)
C(2)	0.3384 (12)	0.3310 (3)	0.2500 (3)	0.0359 (15)
C(3)	0.1796 (12)	0.3307 (3)	0.3318 (3)	0.0370 (16)
C(4)	0.0755 (15)	0.2378 (3)	0.3688 (3)	0.0400 (17)
C(5)	0.1525 (15)	0.1486 (4)	0.3182 (4)	0.0463 (19)
C(6)	0.3135 (14)	0.1505 (3)	0.2388 (3)	0.0419 (18)
C(7)	0.1000 (17)	0.4209 (4)	0.3855 (4)	0.0474 (20)
N	0.5747 (11)	0.2536 (3)	0.1232 (3)	0.0428 (15)
O(1)	0.6475 (10)	0.1687 (3)	0.0784 (2)	0.0571 (14)
O(2)	0.4374 (9)	0.4157 (2)	0.2130 (2)	0.0504 (13)
O(3)	−0.0746 (10)	0.2317 (3)	0.4444 (2)	0.0547 (14)
O(*W*)	0.7510 (11)	0.0561 (3)	0.5369 (3)	0.0783 (17)

Table 3. *Intermolecular distances* (Å) *less than the sum of the corresponding van der Waals radii*

N⋯O(3ⁱⁱ)	2.942 (5)	O(*W*)⋯O(*W*ᵛ)	2.687 (6)
N⋯O(*W*ⁱⁱⁱ)	2.981 (6)	O(*W*)⋯O(*W*ᵛⁱ)	2.731 (6)
O(1)⋯O(3ⁱⁱ)	2.616 (5)	C(4)⋯H(O1ᵛⁱⁱ)	2.75 (6)
O(2)⋯O(*W*ⁱⁱⁱ)	2.856 (5)	O(3)⋯H(O1ᵛⁱⁱⁱ)	1.67 (6)
O(3)⋯O(*W*ⁱ)	2.844 (6)	O(*W*)⋯H(O2ⁱˣ)	1.95 (7)

Symmetry code: (i) $-1 + x, y, z$; (ii) $1 + x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iv) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (v) $1 - x, -y, 1 - z$; (vi) $2 - x, -y, 1 - z$; (viii) $-1 + x, \frac{1}{2} - y, \frac{1}{2} + z$.

ethoxide followed by acidification of the resultant sodium complex. The precipitate obtained from the acidification reaction was recrystallized from methanol/water (2:1).

Data collected on a Philips PW1100 diffractometer and corrected for Lorentz and polarization effects but not for absorption. Unit-cell dimensions obtained by least-squares refinement of the setting angles of 25 reflections in θ range 9–10°. Structure solved by direct methods using *MULTAN* 77 (Main, Lessinger, Woolfson, Germain & Declerq, 1977). An *E* map of highest figure of merit revealed all non-H atoms. In subsequent difference Fourier synthesis all H atoms except those of the water molecule were located. Full-matrix least-squares refinement (on *F*) was carried out using *SHELX*76 (Sheldrick, 1976) with anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms

with weight of $2.67/[\sigma^2(F) + 0.000787F^2]$ applied to the individual reflections. Scattering factors for H atoms were from Cromer & Mann (1968). No extinction correction was applied. All relevant parameters are listed in Table 1. Geometrical calculations and plotting of figures were carried out with *NRCVAX* (PC version; Gabe, Lee & Le Page, 1985).

Atomic coordinates and equivalent isotropic temperature factors are given in Table 2.*

Discussion. A perspective view of the molecule with the atomic numbering scheme and some bond distances is shown in Fig. 1. The molecule is planar

* Lists of structure factors, anisotropic thermal parameters, bond angles and interatomic distances involving H atoms, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54363 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
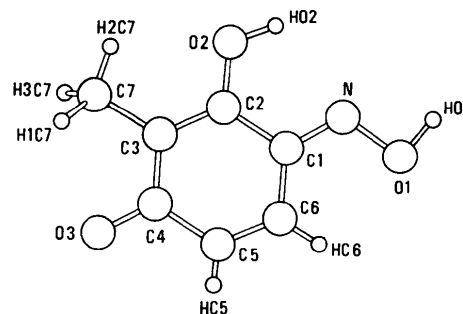

Fig. 1. A perspective view of (I) with the atomic numbering scheme. Bond distances (Å): C(1)—C(2) 1.458 (6), C(2)—C(3) 1.340 (6), C(3)—C(4) 1.445 (6), C(4)—C(5) 1.458 (7), C(5)—C(6) 1.314 (7), C(6)—C(1) 1.434 (6), C(1)—N 1.284 (6), N—O(1) 1.364 (6), C(2)—O(2) 1.341 (5), C(3)—C(7) 1.495 (7), C(4)—O(3) 1.247 (5).
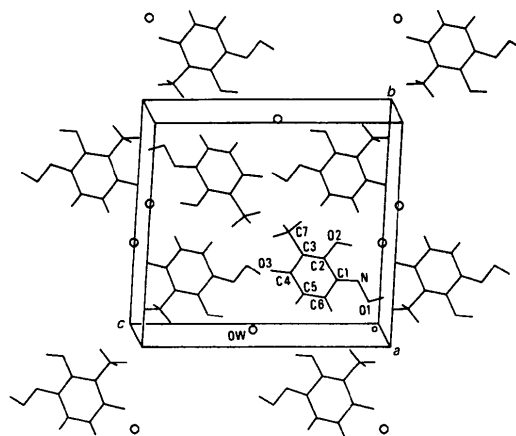

Fig. 2. A view of the unit cell of (I).

within experimental error, with the exception of the O atom of the —NOH group which shows somewhat greater deviation from the mean plane [0·036 (9) Å]. This may be attributed to the involvement of this atom in the hydrogen bond O(1)—H(O1)···O(3ⁱⁱ) (see Table 3). The bond distances C(1)—N, C(2)—C(3), C(5)—C(6) and C(4)—O(3) have pronounced double-bond character [> C—N 1·322 (3), > C=C 1·337 (6), > C=O 1·23 (1) Å; *International Tables for X-ray Crystallography*, 1968, Vol. III] and indicate a 1,4-quinone oximic structure. The observed bond distances are also in very good agreement with the corresponding ones found in related compounds, *e.g.* in 2,5-dimethyl-*p*-benzoquinone [> C=O 1·223 (3), > C=C 1·347 (3) Å; Hirshfeld & Rabinovich, 1967] and in *β*-5-*n*-propoxy-1,2-quinone 2-oxime [> C=O 1·270 (8), > C—N 1·319 (8) Å; Romers, 1964].

Fig. 2 shows the molecular packing of the crystal. Two kinds of intermolecular hydrogen bonds [O(1)—H(O1)···O(3) and O(2)—H(O2)···O(*W*)] have been confirmed in the structure (see Table 3). In addition the very short contact distances O(*W*)···N and O(*W*)···O(3) [2·981 (6) and 2·844 (6) Å, respectively] indicate the involvement of the water molecule in the formation of the hydrogen bonds

O(*W*)—H···N and O(*W*)—H—O(3), which contribute further to the stabilization of the structure.

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High-Temperature Synthesis and Structure Redetermination of Te₄I₄

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Abstract. $M_r = 1018·02$, triclinic, $P\bar{1}$, $a = 8·201$ (1), $b = 9·952$ (2), $c = 7·995$ (2) Å, $\alpha = 103·05$ (2), $\beta = 104·37$ (1), $\gamma = 90·08$ (1)°, $V = 614·7$ (2) Å³, $Z = 2$, $D_x = 5·500$ g cm⁻³, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 193·17$ cm⁻¹, $F(000) = 840$, $T = 293$ K, final $R = 0·061$ for 2560 observed reflections. To a first approximation, every Te atom is surrounded by two Te atoms and two I atoms; the four-coordinate geometries about Te atoms are square planar and trigonal bipyramidal with a distant equatorial electron pair. There are several Te···I and I···I interactions between neighbouring Te₄I₄ molecules, forming some chains along the *a* and *b* axes, respectively.

Experimental. Te₄I₄ was prepared by direct combination of elements. Co and Te powders (99·8 and 99·999% respectively, Shanghai Chemicals Supply

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