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6-Methoxyquinoline *N*-oxide–hydroquinone (2/1)

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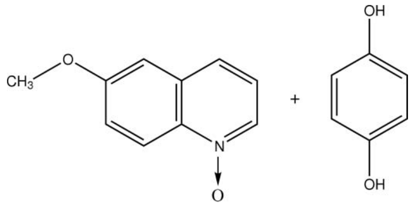
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.089; data-to-parameter ratio = 12.3.

The title cocrystal, $2C_{10}H_9NO_2{\cdot}C_6H_6O_2$, belongs to a series of molecular systems based on quinoline *N*-oxide. The hydroquinone molecule lies on an inversion centre. The cocrystal structure is held together by an O—H···O hydrogen bond, with a donor–acceptor distance of 2.6118 (18) Å. The dihedral angle formed by the mean planes of the two molecules is 83.5 (1)°. In the crystal structure, the title complex is stabilized by weak C—H···O hydrogen bonds. Molecules of methoxyquinoline *N*-oxide and hydroquinone are intertwined, forming chains along the [010] direction.

Related literature

For related literature, see: Moreno-Fuquen *et al.* (2005); Wallwork & Powell (1980).



Experimental

Crystal data

$2C_{10}H_9NO_2{\cdot}C_6H_6O_2$
M_r = 460.48
Triclinic, $P\bar{1}$
a = 7.8841 (19) Å
b = 8.480 (2) Å
c = 9.887 (3) Å
α = 95.72 (2)°
β = 109.85 (3)°
γ = 113.50 (2)°
V = 548.6 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 150 (2) K
0.18 × 0.14 × 0.04 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.963, T_{max} = 1.000
4934 measured reflections
1950 independent reflections
1425 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.089
S = 1.08
1950 reflections
159 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3H···O1	0.94 (2)	1.67 (2)	2.6118 (18)	176.2 (17)
C1—H1···O3^i	0.95	2.39	3.336 (3)	176

Symmetry code: (i) $-x+2, -y+2, -z+2$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS93* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PARST95* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2019).

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supplementary materials

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6-Methoxyquinoline *N*-oxide-hydroquinone (2/1)

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Comment

The title cocrystal, $C_{10}H_9NO_2$. 1/2($C_6H_6O_2$), belongs to a series of molecular systems based on quinoline *N*-oxide with diverse hydrogen-bond donors (Moreno-Fuquen *et al.*, 2005). The synthesis and characterization of the title 6-methoxyquinoline *N*-oxide (MQNO) and hydroquinone (HQ) complex (I) has a threefold purpose: (a) to analyze the type of hydrogen-bonding interactions in (I), (b) to explore possible non-linear optical properties and (c) to increase the information about the MQNO system, since the structural data alone do not contain enough information.

The isoquinoline *N*-oxide/2-nitrobenzoic acid complex, reported by our research group (Moreno-Fuquen *et al.*, 2005) and the free hydroquinone molecule (HQ) in the more stable form at room temperature (Wallwork & Powell, 1980) were used as reference systems to compare with the structural characteristics of the title complex. An ORTEP plot of the hydrogen bonded complex with the atomic numbering scheme is shown in Figure 1. The MQNO and HQ molecules are held together by an intermolecular hydrogen bond between the O1 atom of the N-oxide group of MQNO and the O3 atom of HQ molecule, with an O···O distance of 2.6118 (18) Å. The dihedral angle between the mean planes defined by the quinoline and hydroquinone ring atoms is 83.5 (1)°. The plane formed by methoxy group, C10—O2—C8, shows a slight deviation with respect to the mean plane of the quinoline ring atoms, showing a dihedral angle of 3.1 (1)°. The crystal packing of the title complex is stabilized by weak C1—H1···O3, [O3 with symmetry: -x+2, -y+2, -z+2] intermolecular hydrogen bond. Values of these interactions can be observed in Table 2. Molecules of HQ are intertwined with two other MQNO molecules by O···O interactions. These MQNO molecules are linked to other HQ molecules, disposed about an inversion centre (-x+2, y+2, z-2), by C—H···O interactions. All form chains along the [010] direction. The bond lengths and angles in both molecules are within the expected values. The presence of a centre of symmetry in the crystal precludes any nonlinear properties.

Experimental

The synthesis of the title compound (I) was carried out by slow evaporation of equimolecular quantities of HQ (0.703 g, 0.0064 mol) and MQNO (1.118 g) in 100 ml of dry acetonitrile. After two days, colourless plates of a good quality, suitable for X-ray analysis, were obtained. The initial reagents were purchased from Aldrich Chemical Co., and were used without additional purification.

Refinement

All non-hydrogen atoms were identified by direct methods and the positions of all the hydrogen atoms were obtained from the use of difference Fourier maps. In the final refinement, ring hydrogen atoms were constrained to geometrically sensible positions with a riding model, C—H = 0.93 Å, and U_{iso}(H)= 1.2U_{eq}(C). The H-atoms of the methyl group were assigned C—H distances of 0.96 Å, with U_{iso}(H)= 1.5U_{eq}(C). The H-atom H3H was allowed to refine freely.

Figures

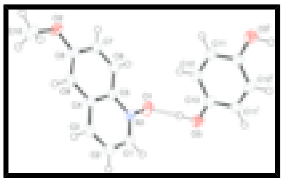

Fig. 1. An ORTEP plot of the title compound showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

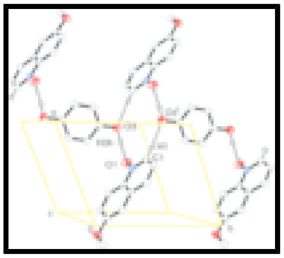

Fig. 2. View of the crystal structure of (I), showing the O—H···O and C—H···O interactions. Symmetry codes : (i), 2-x, 2-y, 2-z.

(I)

Crystal data

$2C_{10}H_9N_1O_2 \cdot C_6H_6O_2$	$Z = 1$
$M_r = 460.48$	$F_{000} = 242$
Triclinic, $P\bar{1}$	$D_x = 1.394$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 431(1) K
$a = 7.8841$ (19) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 8.480$ (2) Å	Cell parameters from 2421 reflections
$c = 9.887$ (3) Å	$\theta = 2.7$–28.5°
$\alpha = 95.72$ (2)°	$\mu = 0.10$ mm^{-1}
$\beta = 109.85$ (3)°	$T = 150$ (2) K
$\gamma = 113.50$ (2)°	Plate, colourless
$V = 548.6$ (3) Å^3	0.18 × 0.14 × 0.04 mm

Data collection

Oxford Diffraction Gemini diffractometer	1950 independent reflections
Radiation source: fine-focus sealed tube	1425 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\mathrm{int}} = 0.022$
$T = 150(2)$ K	$\theta_{\mathrm{max}} = 25.0$°
ω and π scans	$\theta_{\mathrm{min}} = 2.7$°
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)	$h = -9 \rightarrow 9$
$T_{\mathrm{min}} = 0.963$, $T_{\mathrm{max}} = 1.000$	$k = -10 \rightarrow 9$
4934 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.0161P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.089$	$\Delta\rho_{max} = 0.13$ e Å^{-3}
$S = 1.08$	$\Delta\rho_{min} = -0.17$ e Å^{-3}
1950 reflections	Extinction correction: none
159 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: structure-invariant direct methods	
Hydrogen site location: difference Fourier map	

Special details

Geometry. All su's (except the su in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell su's are taken into account individually in the estimation of su's in distances, angles and torsion angles; correlations between su's in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell su's is used for estimating su's involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.57431 (15)	0.73269 (13)	0.89892 (11)	0.0391 (3)
O2	−0.15919 (15)	0.09469 (12)	0.36825 (11)	0.0373 (3)
O3	0.95539 (17)	0.80704 (13)	1.03706 (12)	0.0420 (3)
N1	0.52157 (17)	0.71447 (15)	0.75311 (13)	0.0290 (3)
C1	0.6280 (2)	0.84352 (18)	0.70459 (17)	0.0340 (4)
H1	0.7459	0.9466	0.7747	0.041*
C2	0.5681 (2)	0.82872 (19)	0.55213 (18)	0.0358 (4)
H2	0.6460	0.9213	0.5191	0.043*
C3	0.3984 (2)	0.68218 (19)	0.44994 (17)	0.0334 (4)
H3	0.3562	0.6741	0.3463	0.040*
C4	0.2859 (2)	0.54249 (17)	0.49912 (15)	0.0259 (3)
C5	0.3516 (2)	0.55940 (17)	0.65351 (15)	0.0264 (3)
C6	0.2472 (2)	0.42169 (18)	0.70749 (16)	0.0309 (4)
H6	0.2926	0.4336	0.8118	0.037*
C7	0.0807 (2)	0.27146 (18)	0.60868 (16)	0.0317 (4)
H7	0.0105	0.1778	0.6446	0.038*
C8	0.0105 (2)	0.25262 (17)	0.45318 (16)	0.0287 (3)
C9	0.1095 (2)	0.38516 (17)	0.39870 (16)	0.0282 (3)
H9	0.0605	0.3721	0.2941	0.034*
C10	−0.2312 (2)	0.0611 (2)	0.20879 (17)	0.0397 (4)

H10A	−0.2812	0.1459	0.1767	0.060*
H10B	−0.3419	−0.0611	0.1609	0.060*
H10C	−0.1197	0.0755	0.1800	0.060*
C11	0.8351 (2)	0.33265 (18)	0.94786 (15)	0.0312 (4)
H11	0.7208	0.2176	0.9120	0.037*
C12	0.8066 (2)	0.48315 (17)	0.96332 (15)	0.0296 (4)
H12	0.6735	0.4707	0.9378	0.036*
C13	0.9725 (2)	0.65287 (17)	1.01619 (15)	0.0296 (4)
H3H	0.816 (3)	0.775 (2)	0.988 (2)	0.062 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0352 (6)	0.0466 (6)	0.0248 (6)	0.0164 (5)	0.0068 (5)	−0.0011 (5)
O2	0.0316 (6)	0.0305 (5)	0.0313 (6)	0.0033 (5)	0.0080 (5)	0.0004 (4)
O3	0.0328 (7)	0.0298 (6)	0.0471 (7)	0.0129 (5)	0.0036 (5)	−0.0013 (5)
N1	0.0259 (7)	0.0309 (6)	0.0253 (7)	0.0135 (6)	0.0069 (5)	0.0003 (5)
C1	0.0259 (8)	0.0247 (7)	0.0433 (10)	0.0085 (7)	0.0112 (7)	0.0025 (7)
C2	0.0317 (9)	0.0293 (8)	0.0442 (10)	0.0115 (7)	0.0158 (7)	0.0122 (7)
C3	0.0337 (9)	0.0341 (8)	0.0334 (9)	0.0154 (7)	0.0144 (7)	0.0123 (7)
C4	0.0255 (8)	0.0266 (7)	0.0271 (8)	0.0140 (6)	0.0106 (6)	0.0056 (6)
C5	0.0228 (8)	0.0276 (7)	0.0279 (8)	0.0132 (6)	0.0091 (6)	0.0023 (6)
C6	0.0310 (8)	0.0374 (8)	0.0234 (8)	0.0155 (7)	0.0110 (7)	0.0067 (7)
C7	0.0309 (8)	0.0320 (8)	0.0317 (9)	0.0113 (7)	0.0158 (7)	0.0104 (7)
C8	0.0237 (8)	0.0269 (7)	0.0296 (9)	0.0091 (6)	0.0094 (6)	0.0017 (6)
C9	0.0283 (8)	0.0307 (8)	0.0228 (8)	0.0140 (7)	0.0081 (6)	0.0039 (6)
C10	0.0314 (9)	0.0388 (8)	0.0312 (9)	0.0082 (7)	0.0067 (7)	−0.0041 (7)
C11	0.0273 (8)	0.0271 (8)	0.0263 (8)	0.0043 (6)	0.0076 (6)	0.0035 (6)
C12	0.0244 (8)	0.0337 (8)	0.0244 (8)	0.0100 (7)	0.0084 (6)	0.0034 (6)
C13	0.0323 (9)	0.0285 (8)	0.0219 (8)	0.0122 (7)	0.0079 (6)	0.0026 (6)

Geometric parameters (Å, °)

O1—N1	1.3313 (15)	C6—C7	1.360 (2)
O2—C8	1.3684 (17)	C6—H6	0.9500
O2—C10	1.4363 (18)	C7—C8	1.414 (2)
O3—C13	1.3698 (16)	C7—H7	0.9500
O3—H3H	0.94 (2)	C8—C9	1.367 (2)
N1—C1	1.3333 (19)	C9—H9	0.9500
N1—C5	1.3914 (19)	C10—H10A	0.9800
C1—C2	1.394 (2)	C10—H10B	0.9800
C1—H1	0.9500	C10—H10C	0.9800
C2—C3	1.364 (2)	C11—C12	1.3839 (19)
C2—H2	0.9500	C11—C13^{i}	1.385 (2)
C3—C4	1.413 (2)	C11—H11	0.9500
C3—H3	0.9500	C12—C13	1.393 (2)
C4—C5	1.4069 (19)	C12—H12	0.9500
C4—C9	1.421 (2)	C13—C11^{i}	1.385 (2)

C5—C6	1.408 (2)		
C8—O2—C10	117.18 (12)	C6—C7—C8	120.93 (14)
C13—O3—H3H	107.1 (10)	C6—C7—H7	119.5
O1—N1—C1	120.45 (12)	C8—C7—H7	119.5
O1—N1—C5	118.38 (12)	C9—C8—O2	125.29 (13)
C1—N1—C5	121.16 (13)	C9—C8—C7	120.61 (13)
N1—C1—C2	120.69 (13)	O2—C8—C7	114.10 (13)
N1—C1—H1	119.7	C8—C9—C4	119.74 (14)
C2—C1—H1	119.7	O2—C10—H10A	109.5
C3—C2—C1	120.46 (15)	O2—C10—H10B	109.5
C3—C2—H2	119.8	H10A—C10—H10B	109.5
C1—C2—H2	119.8	O2—C10—H10C	109.5
C2—C3—C4	119.66 (15)	H10A—C10—H10C	109.5
C2—C3—H3	120.2	H10B—C10—H10C	109.5
C4—C3—H3	120.2	C12—C11—C13^{i}	121.05 (13)
C5—C4—C3	118.74 (13)	C12—C11—H11	119.5
C5—C4—C9	118.68 (13)	C13^{i}—C11—H11	119.5
C3—C4—C9	122.58 (14)	C11—C12—C13	120.21 (13)
N1—C5—C4	119.22 (13)	C11—C12—H12	119.9
N1—C5—C6	120.04 (13)	C13—C12—H12	119.9
C4—C5—C6	120.73 (13)	O3—C13—C11^{i}	118.11 (13)
C7—C6—C5	119.29 (14)	O3—C13—C12	123.13 (13)
C7—C6—H6	120.4	C11^{i}—C13—C12	118.74 (12)
C5—C6—H6	120.4		

O1—N1—C1—C2	−177.08 (12)	N1—C5—C6—C7	179.62 (12)
C5—N1—C1—C2	1.9 (2)	C4—C5—C6—C7	−0.3 (2)
N1—C1—C2—C3	0.4 (2)	C5—C6—C7—C8	−0.5 (2)
C1—C2—C3—C4	−1.7 (2)	C10—O2—C8—C9	4.1 (2)
C2—C3—C4—C5	0.6 (2)	C10—O2—C8—C7	−175.94 (11)
C2—C3—C4—C9	−179.12 (13)	C6—C7—C8—C9	0.3 (2)
O1—N1—C5—C4	176.07 (11)	C6—C7—C8—O2	−179.64 (12)
C1—N1—C5—C4	−2.91 (19)	O2—C8—C9—C4	−179.34 (12)
O1—N1—C5—C6	−3.88 (18)	C7—C8—C9—C4	0.7 (2)
C1—N1—C5—C6	177.14 (12)	C5—C4—C9—C8	−1.50 (19)
C3—C4—C5—N1	1.64 (19)	C3—C4—C9—C8	178.22 (13)
C9—C4—C5—N1	−178.63 (11)	C13^{i}—C11—C12—C13	−0.2 (2)
C3—C4—C5—C6	−178.41 (12)	C11—C12—C13—O3	−178.59 (12)
C9—C4—C5—C6	1.3 (2)	C11—C12—C13—C11^{i}	0.2 (2)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3H···O1	0.94 (2)	1.67 (2)	2.6118 (18)	176.2 (17)
C1—H1···O3^{ii}	0.95	2.39	3.336 (3)	176

Symmetry codes: (ii) −*x*+2, −*y*+2, −*z*+2.

Fig. 1

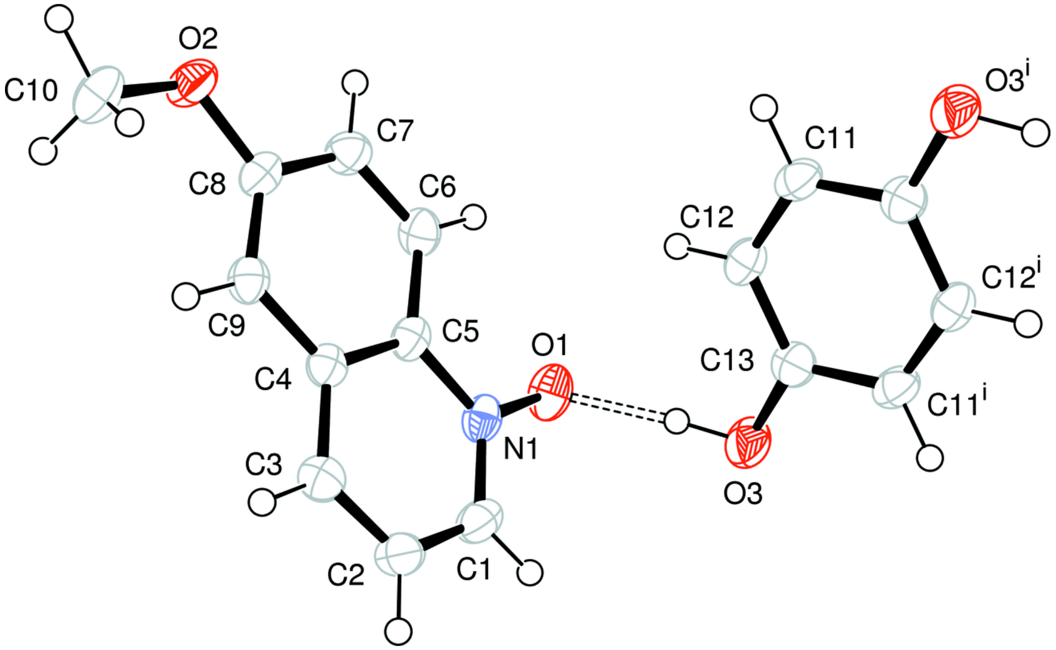

Fig. 2