

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (II)
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
O1	0.0234 (2)	0.1323 (2)	0.0819 (2)	0.0892 (7)
O2	-0.4654 (2)	0.23501 (15)	-0.1220 (2)	0.0873 (7)
N1	-0.0929 (2)	0.1855 (2)	-0.1170 (2)	0.0626 (6)
N2	-0.3216 (2)	0.1064 (2)	-0.1349 (2)	0.0649 (7)
C1	0.0817 (3)	0.2181 (2)	0.0971 (3)	0.0676 (8)
C2	0.1739 (3)	0.2354 (3)	0.2011 (3)	0.0827 (10)
C3	0.2380 (3)	0.3206 (3)	0.2212 (3)	0.0954 (11)
C4	0.2107 (3)	0.3905 (3)	0.1390 (3)	0.0971 (11)
C5	0.1189 (3)	0.3736 (2)	0.0370 (3)	0.0851 (10)
C6	0.0519 (3)	0.2878 (2)	0.0115 (3)	0.0615 (7)
C7	-0.0442 (3)	0.2702 (2)	-0.0994 (2)	0.0608 (8)
C8	-0.0800 (3)	0.3517 (2)	-0.1841 (2)	0.0866 (10)
C9	-0.1871 (2)	0.1569 (2)	-0.2258 (2)	0.0615 (8)
C10	-0.1288 (3)	0.1291 (2)	-0.3080 (2)	0.0754 (9)
C11	-0.2254 (3)	0.0974 (2)	-0.4190 (2)	0.0792 (9)
C12	-0.2993 (3)	0.0135 (2)	-0.4037 (2)	0.0885 (10)
C13	-0.3548 (3)	0.0377 (2)	-0.3175 (2)	0.0782 (9)
C14	-0.2588 (2)	0.0727 (2)	-0.2072 (2)	0.0632 (8)
C15	-0.3111 (2)	0.0674 (2)	-0.0414 (3)	0.0618 (8)
C16	-0.2319 (3)	-0.0184 (2)	0.0096 (3)	0.0910 (10)
C17	-0.3833 (3)	0.1107 (2)	0.0170 (2)	0.0616 (8)
C18	-0.3814 (3)	0.0734 (2)	0.1181 (3)	0.0791 (9)
C19	-0.4496 (3)	0.1143 (3)	0.1723 (3)	0.0923 (11)
C20	-0.5215 (3)	0.1930 (3)	0.1276 (3)	0.0947 (12)
C21	-0.5278 (2)	0.2323 (2)	0.0279 (3)	0.0819 (10)
C22	-0.4578 (3)	0.1927 (3)	-0.0265 (2)	0.0683 (9)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (II)

O1—C1	1.346 (3)	C6—C7	1.471 (4)
O2—C22	1.340 (3)	C7—C8	1.514 (3)
N1—C7	1.284 (3)	C9—C14	1.512 (3)
N1—C9	1.477 (3)	C15—C17	1.467 (4)
N2—C15	1.288 (3)	C15—C16	1.497 (4)
N2—C14	1.475 (3)		
C7—N1—C9	122.4 (2)	N1—C9—C14	108.5 (2)
C15—N2—C14	125.1 (2)	N2—C14—C9	108.0 (2)
O1—C1—C6	121.7 (3)	N2—C15—C17	116.7 (3)
N1—C7—C6	116.8 (3)	N2—C15—C16	124.3 (3)
N1—C7—C8	124.4 (3)	O2—C22—C17	121.9 (3)
O1—C1—C6—C7	-0.4 (4)	N1—C9—C14—N2	63.1 (3)
C9—N1—C7—C6	-177.7 (2)	C14—N2—C15—C17	-178.8 (2)
C1—C6—C7—N1	-5.3 (3)	N2—C15—C17—C22	-1.7 (4)
C7—N1—C9—C14	-157.9 (2)	C15—C17—C22—O2	-0.5 (4)
C15—N2—C14—C9	-124.0 (3)		

H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; each H atom was given an isotropic displacement parameter equal to 1.2 (or 1.5 for methyl and —OH H atoms) times the equivalent isotropic displacement parameter of the atom to which it is attached. For compound (II) the structure was solved by direct methods in *Pc* and then transformed into the space group *P2₁/c*.

For both compounds, data collection: *MADNES* (Pflugrath & Messerschmidt, 1992); cell refinement: *MADNES*; data reduction: *SHELXTL-Plus* (Sheldrick, 1990); program(s) used to solve structures: *SHELXTL-Plus*; program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXL93*.

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Chemical Database Service (Allen *et al.*, 1991) at Daresbury. Financial assistance was provided by BP (JPC) and ICI (JCC).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1152). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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8-Hydroxy-7-nitroquinoline-5-sulfonic Acid Monohydrate

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Abstract

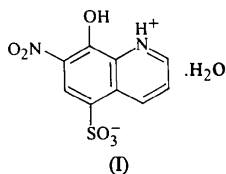
The title compound, $\text{C}_9\text{H}_6\text{N}_2\text{O}_6\text{S}\cdot\text{H}_2\text{O}$, is zwitterionic (*i.e.* 8-hydroxy-7-nitroquinolinium-5-sulfonate monohydrate) with a deprotonated sulfonic group and a protonated quinoline N atom which interacts with the hydroxy O atom. The H atom of the hydroxy group is distal to the N atom and is involved in an interaction with an O atom of the nitro group, which is oriented at $32.2(1)^\circ$ with respect to the quinoline ring system.

Comment

Derivatives of 8-hydroxyquinoline (oxine) are known for their anti-amoebic, antibacterial and antifungal activities, which can be correlated to their metal chelating

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ability (Banergee & Saha, 1986). They are also well known analytical reagents. This work was undertaken as part of a study of substituted 8-hydroxyquinolines and their complexes. We have recently redetermined the crystal structure of 8-hydroxy-7-iodoquinoline-5-sulfonic acid (Balasubramanian & Thomas Muthiah, 1996) and a cobalt complex of 8-hydroxy-7-iodoquinoline-5-sulfonic acid (Balasubramanian & Thomas Muthiah, 1994). In order to study the effect of the nitro substituent, we have investigated the crystal structure of the title compound, (I).



The proton of the sulfonic group has migrated to N1, as in the crystal structure of ferron. This leads to an enhancement of the internal angle at N1 as compared with that in the neutral 8-hydroxyquinoline (Banergee & Saha, 1986). This trend has already been observed in the crystal structures of other 8-hydroxyquinoline derivatives (Balasubramanian & Thomas Muthiah, 1996). Bond lengths and bond angles involving the quinoline system are comparable with those reported for the N1-protonated quinoline system (Banergee, Basak, Mazumdar & Chaudhuri, 1984). The geometry around the S atom is distorted tetrahedral and the bond lengths and bond angles involving this group are in agreement with those reported for 8-hydroxy-7-iodoquinoline-5-sulfonic acid (Balasubramanian & Thomas Muthiah, 1996) and 8-hydroxyquinoline-5-sulfonic acid dihydrate (Banergee *et al.*, 1984). The bond lengths and bond angles of the nitro group are consistent with those of similar nitro groups (Carr, Finney & Lindley, 1977).

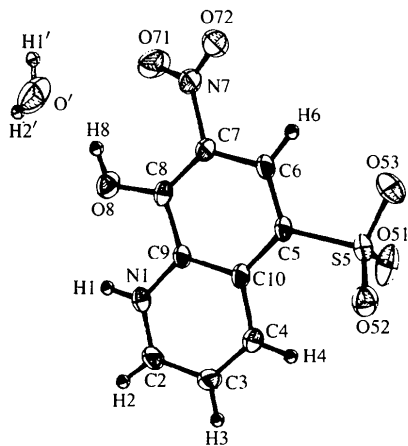


Fig. 1. An ORTEP (Johnson, 1965) view of the molecule with displacement ellipsoids at the 50% probability level.

The dihedral angle between the nitro group and the quinoline ring system is 32.2(1)°. N1—H is involved in bifurcated hydrogen bonding. N1—H forms an intramolecular contact with O8 leading to a five-membered hydrogen-bonded ring with an N1...O8 distance of 2.616(2) Å and an N1—H...O8 angle of 105(2)°. This intramolecular hydrogen bond is also observed in the crystal structures of ferron and 8-hydroxyquinoline-5-sulfonic acid dihydrate. N1—H also forms an intermolecular hydrogen bond with one of the sulfonic O atoms, O53(*x* - 1, 1 + *y*, *z*) [*D*...*A* = 2.715(2) Å, *D*—H...*A* = 149(2)°]. O8—H forms an intramolecular hydrogen bond with O71 of the nitro group, with a *D*...*A* distance of 2.792(3) Å and a *D*—H...*A* angle of 125(3)°, leading to a six-membered hydrogen-bonded ring system. C6—H is involved in two hydrogen bonds, one with the sulfonic O53 atom and the other with the nitro O72 atom, with *D*...*A* distances of 2.810(2) and 2.727(2) Å and *D*—H...*A* angles of 102(2) and 101(2)°, respectively. The water H atoms form hydrogen bonds with O8, O52 and O71.

Experimental

The compound was prepared by warming a mixture of a 150 ml water solution of 0.7 g of ferron and 2 ml of concentrated nitric acid in a water bath for 30 min. The crystals appeared on slow evaporation.

Crystal data

C₉H₆N₂O₆S.H₂O
M_r = 288.23
 Triclinic
P $\bar{1}$
a = 7.8849(12) Å
b = 8.2625(15) Å
c = 9.975(2) Å
 α = 86.11(2)°
 β = 66.88(2)°
 γ = 65.741(13)°
V = 541.5(2) Å³
Z = 2
D_x = 1.768 Mg m⁻³

Mo *K* α radiation
 λ = 0.71073 Å
 Cell parameters from 15 reflections
 θ = 2–23°
 μ = 0.335 mm⁻¹
T = 298(2) K
 Prismatic
 0.25 × 0.23 × 0.20 mm
 Dark brown

Data collection

CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 ψ scan (MolEN; Fair, 1990)
 T_{\min} = 0.90, T_{\max} = 0.95
 1975 measured reflections
 1833 independent reflections
 1649 observed reflections
 $[I > 2\sigma(I)]$

R_{int} = 0.007
 θ_{max} = 24.98°
 $h = -8 \rightarrow 9$
 $k = -9 \rightarrow 9$
 $l = 0 \rightarrow 11$
 2 standard reflections monitored every 100 reflections
 intensity decay: negligible

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0302$
 $wR(F^2) = 0.0783$

$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.1837P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.101$
 1833 reflections
 204 parameters
 H atoms located from
 difference Fourier map,
 refined isotropically

$(\Delta/\sigma)_{\max} = -0.012$
 $\Delta\rho_{\max} = 0.217 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.275 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Atomic scattering factors
 from *International Tables*
 for *Crystallography* (1992),
 Vol. C, Tables 4.2.6.8 and
 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
N1	-0.1875 (2)	0.9383 (2)	0.3290 (2)	0.0286 (3)
C2	-0.1442 (3)	0.9126 (3)	0.1883 (2)	0.0342 (4)
C3	0.0334 (3)	0.7721 (3)	0.0990 (2)	0.0350 (5)
C4	0.1598 (3)	0.6584 (2)	0.1589 (2)	0.0295 (4)
C5	0.2275 (3)	0.5613 (2)	0.3849 (2)	0.0248 (4)
S5	0.46253 (6)	0.37538 (6)	0.28737 (5)	0.0300 (2)
O51	0.6031 (2)	0.4520 (2)	0.2071 (2)	0.0543 (4)
O52	0.4289 (2)	0.2778 (2)	0.1928 (2)	0.0388 (3)
O53	0.5119 (3)	0.2743 (2)	0.4018 (2)	0.0552 (5)
C6	0.1631 (3)	0.5931 (2)	0.5323 (2)	0.0267 (4)
C7	-0.0141 (3)	0.7450 (2)	0.6128 (2)	0.0264 (4)
N7	-0.0714 (2)	0.7654 (2)	0.7703 (2)	0.0308 (4)
O71	-0.1633 (3)	0.9180 (2)	0.8341 (2)	0.0490 (4)
O72	-0.0238 (2)	0.6305 (2)	0.83082 (15)	0.0396 (3)
C8	-0.1325 (3)	0.8675 (2)	0.5494 (2)	0.0262 (4)
O8	-0.3001 (2)	1.0156 (2)	0.6092 (2)	0.0373 (3)
C9	-0.0676 (3)	0.8271 (2)	0.3953 (2)	0.0243 (4)
C10	0.1107 (2)	0.6789 (2)	0.3108 (2)	0.0241 (4)
O'	-0.5392 (3)	1.1988 (3)	0.8649 (2)	0.0617 (5)

Table 2. Selected geometric parameters (Å , $^\circ$)

N1—C2	1.317 (3)	S5—O53	1.451 (2)
N1—C9	1.368 (2)	C7—N7	1.455 (2)
C5—S5	1.786 (2)	N7—O72	1.221 (2)
S5—O51	1.443 (2)	N7—O71	1.231 (2)
S5—O52	1.444 (1)	C8—O8	1.323 (2)
C2—N1—C9	122.8 (2)	O53—S5—C5	104.10 (9)
C6—C5—S5	118.6 (1)	C8—C7—N7	120.5 (2)
C10—C5—S5	121.5 (1)	C6—C7—N7	116.4 (2)
O51—S5—O52	112.8 (1)	O72—N7—O71	124.0 (2)
O51—S5—O53	113.1 (1)	O72—N7—C7	118.1 (2)
O52—S5—O53	112.96 (9)	O71—N7—C7	117.9 (2)
O51—S5—C5	105.26 (9)	O8—C8—C7	130.1 (2)
O52—S5—C5	107.72 (8)	O8—C8—C9	114.6 (2)

Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: VJ1030). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Non-Peptide Angiotensin II Receptor Antagonist: 2-Butyl-6-dimethoxymethyl-5-phenyl-1-[2'-(1H-tetrazol-5-yl)biphenyl-4-yl]methyl}-1H-imidazo[5,4-b]pyridine

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Abstract

The title compound, $\text{C}_{33}\text{H}_{33}\text{N}_7\text{O}_2$, is one of a series of imidazo[4,5-*b*]pyridine-based angiotensin II receptor antagonists showing high antihypertensive activity. The biphenyltetrazole moiety assumes the same conformation as in related compounds, but its relative orientation with respect to the central fused ring is different to that in these compounds, indicating that there is considerable conformational flexibility about the methylene bridge joining the two ring systems.

Comment

Non-peptide angiotensin II (AII) receptor antagonists such as losartan are being actively investigated for treatment of hypertension in humans (Duncia *et al.*, 1992). Most of them contain a biphenyltetrazole moiety linked to a heterocycle by a methylene group. The title