Acta Cryst. (1997). C**53**, 1324–1325

5-Hydroxyimino-8-quinolone

NOBUO OKABE AND MAYUMI AKITA

Faculty of Pharmaceutical Sciences, Kinki University, Kowakae 3-4-1, Higashiosaka, Osaka 577, Japan

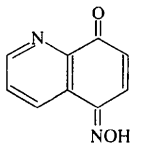
(Received 19 November 1996; accepted 23 April 1997)

Abstract

In the structure of the title compound, $C_9H_6N_2O_2$, the isonitroso (hydroxyimino) group is nearly coplanar with that of the quinoline ring. The molecules are linked by intermolecular O—H···N hydrogen bonds between the isonitroso O atom and the quinoline N atoms, with O···N 2.746 (4) Å.

Comment

The title compound, (I), is a derivative of 8-hydroxyquinoline which is carcinogenic and cytotoxic (Kitchin, Brown & Kulkarni, 1992; Jonas & Riley, 1991). Nitroso compounds, especially N-nitroso ones, are well known as potent carcinogens (Iishi, Tatsuta, Baba, Uehara & Nakaizumi, 1994). In order to clarify the physiological effect of nitroso compounds, it is important to obtain structural data of various nitroso compounds, although the physiological effect of the title compound is as yet unclear. Accordingly, the crystal structure of the title compound has been determined.



(I)

The molecular structure of (I) with the atomic labelling is shown in Fig. 1. The plane of the isonitroso group is almost coplanar with that of the quinoline ring; O5—N5—C5—C6 −0.8 (6)°. Molecules are connected by hydrogen bonds O5—H5···N1^{i} 2.746 (4) Å [symmetry code: (i) $\frac{1}{2}+x$, $\frac{1}{2}-y$, $-\frac{1}{2}+z$].

Compound (I) is a tautomer of 5-nitroso-8-hydroxyquinoline and the single crystal used in this study was obtained from the latter compound. The results of this indicate that the keto form at position 8 in (I) is caused by tautomerism from the enol form, which is the stable form in the crystal. Therefore, the keto form seems to be a structural feature of the nitroso derivative of 8-hydroxyquinolines like compound (I). On the contrary, in solution and solid-state studies of the other 8-hydroxyquinoline derivatives, *i.e.* [(8-hydroxy-5-quinolyl)imino]mercaptoacetic acid (El-Gahami, 1994), 8-hydroxyquinoline-5-sulfonylhydrazide and 8-hydroxyquinoline-5-(2-pyridyl)sulfonamide (Ibrahim, Makhlouf, Abdel-Hafez & Moharram, 1986), the enol form at the 8 position of all derivatives remains stable and it is always intramolecularly hydrogen bonded to the N atom of the quinoline ring. This difference between the 8-hydroxyquinoline derivatives seems to be worth considering when the derivatives are concerned with their physiological effect.

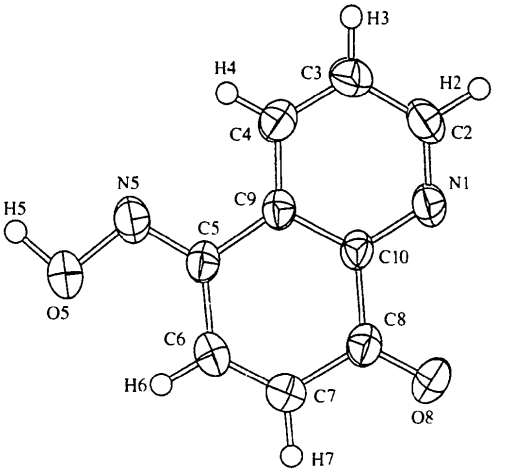


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the atomic numbering scheme. Ellipsoids for non-H atoms correspond to 50% probability levels.

Experimental

The light yellow plate crystal used for analysis was obtained by slow evaporation of 1-propanol solution of 5-hydroxyimino-8-quinolone at room temperature.

Crystal data

$C_9H_6N_2O_2$	Mo $K\alpha$ radiation
M_r = 174.16	λ = 0.71069 Å
Monoclinic	Cell parameters from 25 reflections
$P2_1/n$	θ = 15.15–16.50°
a = 7.946 (2) Å	μ = 0.102 mm^{-1}
b = 7.190 (3) Å	T = 296 K
c = 13.593 (2) Å	Plate
β = 94.58 (1)°	0.5 × 0.3 × 0.1 mm
V = 774.2 (4) Å^3	Light yellow
Z = 4	
D_x = 1.494 Mg m^{-3}	
D_m not determined	

Data collection

Rigaku AFC-5*R* diffractometer	R_{int} = 0.029
ω–2θ scans	θ_{max} = 27.5°
Absorption correction: none	h = 0 → 10
2054 measured reflections	k = 0 → 8
1922 independent reflections	l = −17 → 16
983 reflections with $I > \sigma(I)$	3 standard reflections every 150 reflections
	intensity decay: none

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.003$
$R = 0.063$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
$wR = 0.058$	$\Delta\rho_{min} = -0.25$ e Å^{-3}
$S = 1.46$	Extinction correction: none
983 reflections	Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
142 parameters	
H atoms refined isotropically	
$w = 4F_o^2/\sigma^2(F_o^2)$	

Table 1. *Selected geometric parameters (Å, °)*

O5—N5	1.368 (3)	C6—C7	1.328 (5)
O8—C8	1.218 (4)	C7—C8	1.463 (4)
N5—C5	1.308 (4)	C8—C10	1.481 (5)
C5—C6	1.449 (5)	C9—C10	1.395 (4)
C5—C9	1.462 (4)		
C2—N1—C10	118.2 (3)	C6—C5—C9	118.3 (3)
O5—N5—C5	111.4 (3)	O8—C8—C7	121.5 (3)
N5—C5—C6	125.2 (3)	O8—C8—C10	121.9 (3)
N5—C5—C9	116.5 (3)	C7—C8—C10	116.6 (3)

All H atoms were located from difference Fourier maps and included in the refinement calculations isotropically.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELX*86 (Sheldrick, 1985) and *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1032). Services for accessing these data are described at the back of the journal.

References

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors*. Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

El-Gahami, M. A. (1994). *Bull. Chem. Soc. Jpn*, **67**, 2417–2419.

Ibrahim, S. A., Makhlouf, M. Th., Abdel-Hafez, A. & Moharram, A. M. (1986). *J. Inorg. Biochem.* **28**, 57–65.

Iishi, H., Tatsuta, M., Baba, M., Uehara, H. & Nakaizumi, A. (1994). *Cancer Res.* **54**, 3167–3170.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Jonas, S. K. & Riley, P. A. (1991). *Cell Biochem. Func* **9**, 245–253.

Kitchin, K. T., Brown, J. L. & Kulkarni, A. P. (1992). *Mutat Res.* **266**, 253–272.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Sheldrick, G. M. (1985). *SHELXS*86. *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.

Acta Cryst. (1997). C**53**, 1325–1327

The Stereochemical Assignment of the Ozonide 5-(2-Naphthyl)-3-phenyl-1,2,4-trioxolane-3-carbonitrile

KUNSOO LEE,[a] TAESUNG HUH[b] AND HOSEOP YUN[a]

[a]*Department of Chemistry, Ajou University, Suwon 442-749, Korea, and* [b]*Department of Chemistry, Catholic University, Suwon 422-743, Korea. E-mail: hsyun@madang.ajou.ac.kr*

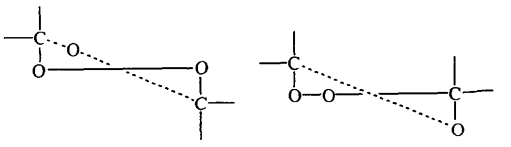
(Received 21 January 1997; accepted 28 April 1997)

Abstract

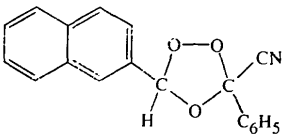
The title compound, $C_{19}H_{13}NO_3$, has been structurally characterized. The naphthyl and phenyl groups adopt a *trans* arrangement. The five atoms in the ozonide ring have a half-chair conformation. The distances and angles of the ring correspond well with those found in unstrained ozonides.

Comment

Although conformational and stereochemical assignments of ozonides have attracted much attention (Bailey, 1978; Bailey & Ferrel, 1978; Criegee & Wenner, 1949; Lattimer, Kuczkowski & Gillies, 1974), only simple ozonides with low molecular weights have been isolated as liquids and their structures elucidated by microwave spectroscopy (Mazur & Kuczkowski, 1977; Kuczkowski, Gillies & Gallaher, 1993). Both experimental results and theoretical calculations favour the half-chair conformation, (I). By contrast, several ozonides derived from cyclic or bicyclic olefins have been reported to adopt a symmetrical ether-O envelope conformation, (II).



(I) (II)



(III)

Stereochemical assignments for *cis–trans* isomeric ozonides bearing H atoms at the ozonide rings have been made with the help of ^{1}H NMR spectroscopy. It