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The Crystal Structures of 2-Quinolone and 8-Acetoxy-2-quinolone

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The crystal structures of 2-quinoline (**1**) and 8-acetoxy-2-quinolone (**2**) were determined by X-ray analysis. The structural features of the ring portion of both compounds are very similar to these of procaterol hydrochloride hemihydrate.

Keywords——X-ray analysis; 2-quinolone; procaterol; crystal structures; MULTAN program

Some heteroaromatic ring compounds have very interesting biological activities; for instance, the 2-quinolone ring skeleton of procaterol hydrochloride hemihydrate, a novel potent bronchodilator,[1] plays an important role in its characteristic activity,[2] and completely adopts the 2-one form where π-electrons in the ring system are rather localized. It would be very interesting to know whether this 2-one form is inherent or is due to the substituted functional groups. Therefore, X-ray analyses of 2-quinolone (**1**) and 8-acetoxy-2-quinolone (**2**) (Chart 1) were carried out, in the present work.

The molecular structures of **1** and **2** with atomic numberings are shown in Figs. 1 and 2, respectively. Bond distances and angles are listed in Table III. The O(1)–C(2) bond distances of the two compounds, 1.242(6) Å and 1.246(5) Å, strongly indicate double bond character and therefore, the 2-quinolone ring completely adopts the 2-one form. The 2-quinolone ring of each compound is slightly bent at the C(9)–C(10) bond; the dihedral angles between the benzene ring and hetero ring planes of **1** and **2** are 178.3° and 178.8°, respectively. π-Electrons in the ring system are rather localized on the C(3)–C(4), C(5)–C(6), C(7)–C(8) and C(9)–C(10) bonds in the crystals. Such structural features of the ring portion in both compounds are very similar to those of procaterol hydrochloride hemihydrate.[3]

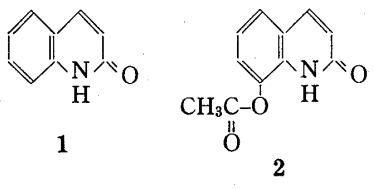

Chart 1

TABLE I. Atomic Coordinates ($\times 10^4$) of **1** for Non-hydrogen Atoms with Their e.s.d.'s in Parentheses

	x	y	z
O (1)	2395(9)	3721(3)	6(3)
N	−988(10)	3170(4)	−1188(3)
C (2)	1117(12)	3872(4)	−864(4)
C (3)	1746(13)	4778(4)	−1598(5)
C (4)	364(13)	4855(5)	−2579(5)
C (5)	−3122(13)	4138(5)	−3913(4)
C (6)	−5139(13)	3348(6)	−4137(5)
C (7)	−5862(13)	2497(5)	−3423(5)
C (8)	−4493(13)	2460(5)	−2430(4)
C (9)	−1743(11)	4096(5)	−2900(4)
C (10)	−2430(12)	3222(5)	−2177(4)

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Table II. Atomic Coordinates ($\times 10^4$) of **2** for Non-hydrogen Atoms with Their e.s.d.'s in Parentheses

	x	y	z
O (1)	−1393(3)	4856(4)	5397(1)
O (2)	654(2)	2312(3)	3719(1)
O (3)	896(3)	4391(3)	3082(1)
N	−1095(3)	3598(4)	4523(1)
C (2)	−1882(3)	4179(5)	4962(2)
C (3)	−3262(4)	3900(6)	4885(2)
C (4)	−3735(4)	3180(5)	4408(2)
C (5)	−3348(4)	1808(5)	3454(2)
C (6)	−2490(4)	1254(5)	3043(2)
C (7)	−1156(4)	1460(5)	3119(2)
C (8)	−692(3)	2237(4)	3608(1)
C (9)	−2899(3)	2598(5)	3957(1)
C (10)	−1556(3)	2820(5)	4031(1)
C (11)	1379(4)	3430(4)	3409(2)
C (12)	2799(4)	3258(6)	3537(2)

Table III. The Bond Distances (Å) and Angles (°) of **1** and **2** for Non-hydrogen Atoms with Their e.s.d.'s in Parentheses

			1	**2**
N	− C (2)		1.366(7)	1.373(5)
N	− C (10)		1.396(7)	1.379(5)
C (2)	− C (3)		1.448(8)	1.438(6)
C (3)	− C (4)		1.375(9)	1.333(6)
C (4)	− C (9)		1.410(9)	1.424(6)
C (5)	− C (6)		1.376(9)	1.366(6)
C (5)	− C (9)		1.407(7)	1.397(6)
C (6)	− C (7)		1.392(9)	1.384(7)
C (7)	− C (8)		1.383(8)	1.373(6)
C (8)	− C (10)		1.376(8)	1.394(5)
C (9)	− C (10)		1.416(8)	1.394(5)
C (2)	− O (1)		1.242(6)	1.246(5)
C (8)	− O (2)			1.399(5)
C (11)	− O (2)			1.365(5)
C (11)	− O (3)			1.187(5)
C (11)	− C (12)			1.487(6)
C (2)	− N	− C (10)	125.7(5)	124.1(3)
O (1)	− C (2)	− N	121.1(5)	120.4(4)
O (1)	− C (2)	− C (3)	123.2(5)	124.1(4)
N	− C (2)	− C (3)	115.8(5)	115.5(4)
C (2)	− C (3)	− C (4)	120.0(5)	121.6(4)
C (3)	− C (4)	− C (9)	122.5(5)	121.8(4)
C (6)	− C (5)	− C (9)	118.3(5)	120.8(4)
C (5)	− C (6)	− C (7)	123.6(6)	120.3(4)
C (6)	− C (7)	− C (8)	117.8(6)	120.0(3)
C (7)	− C (8)	− C (10)	120.6(5)	120.4(4)
C (4)	− C (9)	− C (5)	123.5(5)	123.8(4)
C (4)	− C (9)	− C (10)	118.0(5)	117.4(4)
C (5)	− C (9)	− C (10)	118.5(5)	118.8(4)
N	− C (10)	− C (8)	120.9(5)	120.7(3)
N	− C (10)	− C (9)	117.9(5)	119.6(3)
C (8)	− C (10)	− C (9)	121.2(5)	119.7(3)
O (2)	− C (8)	− C (7)		120.5(3)
O (2)	− C (8)	− C (10)		118.7(3)
C (8)	− O (2)	− C (11)		117.8(3)
O (2)	− C (11)	− O (3)		122.3(4)
O (2)	− C (11)	− C (12)		111.4(3)
O (3)	− C (11)	− C (12)		126.3(4)

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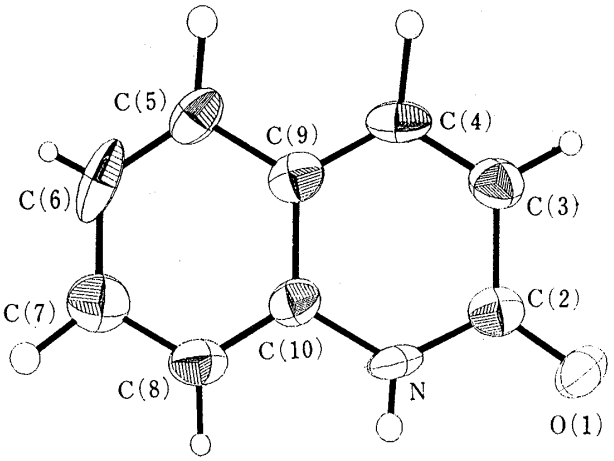

Fig. 1. Molecular Structure and Atomic Numbering of **1**

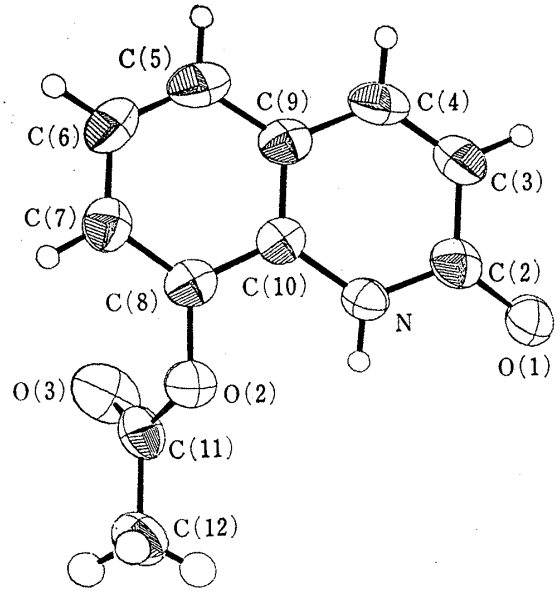

Fig. 2. Molecular Structure and Atomic Numbering of **2**

Experimental

Intensity data were collected on a Syntex R3 four-circle diffractometer with graphite-monochromated Mo Kα radiation using the ω scan mode. All the computations were made on a NOVA-3 computer coupled to the diffractometer, using the Syntex XTL programs.

Crystal Data for 1——Chemical formula C_9H_7NO, M.W.=145.2, orthorhombic, space group $P2_12_12_1$, a=4.730(4), b=12.056(9), c=12.299(15) Å, Z=4, Dx=1.37 g/cm^3, and μ(Mo Kα)=1.0 cm^{-1}. A total of 1214 independent reflections were collected within 2θ less than 60°, among which 496 reflections ($I>1.96\sigma(I)$) were used as observed.

Crystal Data for 2——Chemical formula $C_{11}H_9NO_3$, M.W.=203.2, orthorhombic, space group Pbca, a=10.212(4), b=8.045(3), c=23.027(10) Å, Z=8, Dx=1.43 g/cm^3, and μ(Mo Kα)=1.1 cm^{-1}. A total of 1236 independent reflections were collected within 2θ less than 45°, among which 995 reflections ($I>1.96\sigma(I)$) were used as observed.

Structure Determination and Refinement for 1——The structure of **1** was solved by the direct method using the MULTAN program.[4] The resulting E map revealed the positions of all non-hydrogen atoms. All the hydrogen atoms were found on the difference Fourier map. The refinement of atomic parameters was carried out by a block-diagonal least-squares method. Thermal parameters were refined anisotropically for all the non-hydrogen atoms and isotropically for the hydrogen atoms. The final R-value was 0.080. The final atomic coordinates for the non-hydrogen atoms are shown in Table I.[5]

Structure Determination and Refinement for 2——The structure of **2** was determined by the direct method using the MULTAN program[4] and refined by the least-squares method as described in the preceding section. The final R-value was 0.046. The final atomic coordinates for the non-hydrogen atoms are shown in Table II.[5]

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References and Notes

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5) Tables of the observed and calculated structure factors, anisotropic temperature factors, the coordinates of the hydrogen atoms, and bond distances and angles including the hydrogen atoms of **1** and **2** are available from the authors on request.

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