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4-HYDROXY-2-QUINOLONES.

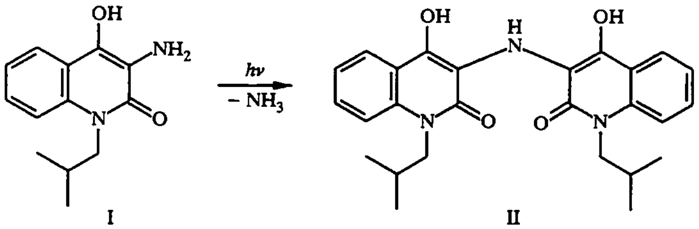
34.* STRUCTURE OF PHOTOCONDENSATION PRODUCTS OF 3-AMINO-1R-2-OXO-4-HYDROXYQUINOLINES

I. V. Ukrainets, S. G. Taran, L. V. Sidorenko,
O. V. Gorokhova, A. V. Turov, and A. A. Ogirenko

3-Amino-2-oxo-4-hydroxyquinolines when exposed to light are quantitatively converted to di(1R-2-oxo-4-hydroxyquinolin-3-yl)amines. We have carried out an x-ray diffraction study of one such compound.

In studying alternative routes to synthesis of 3-acylamino-2-oxo-4-hydroxyquinolines [2], we noted the instability of the intermediate 3-aminoquinolines. As we should expect, these compounds especially rapidly decompose in solution, which must be considered when crystallizing them. Furthermore, chromatographic monitoring of the purity of 3-amino-2-oxo-4-hydroxyquinolines has shown that even when isolated in pure form, when exposed to light they are completely converted to new compounds after even just a few hours. Accordingly, the goal of this work was to study the orientation of this reaction by establishing the structure of the compounds formed during it. We studied 3-amino-1-isobutyl-2-oxo-4-hydroxyquinoline (I) as an example. However, we note that 3-aminoquinolines with other 1-N-alkyl substituents and also the 1H derivative undergo a similar chemical conversion.

According to chromatographic/mass spectrometric analysis, the product of conversion of 3-aminoquinoline I is a pure compound with molecular mass 447 amu. On the other hand, the PMR spectrum of the compound obtained indicates that its molecule is symmetric. All these data taken together allowed us to hypothesize that the compound II formed may be characterized as di(1-isobutyl-2-oxo-4-hydroxyquinolin-3-yl)amine. We conclusively resolved the question of the structure of compound II with the help of x-ray diffraction.



As we see from the data presented in Fig. 1 and in Tables 1 and 2, the basis for the structure of diamine II is two practically identical molecules. The most appreciable difference between them is the fact that in the second molecule, the $C_{(50)}-C_{(51)}$ bond in the isobutyl moiety is somewhat shortened compared with the other isobutyl moieties, which allows us to suggest that these two atoms in the structure can be disordered, although we could not detect this disordering experimentally.

Both molecules in the structure of diamine II have point symmetry close to C_2. Strong intramolecular hydrogen bonds O–H...O are formed in them, fixing the observed conformation of the molecules and preventing free rotation of the bicyclic

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*For Communication 33, see [1].

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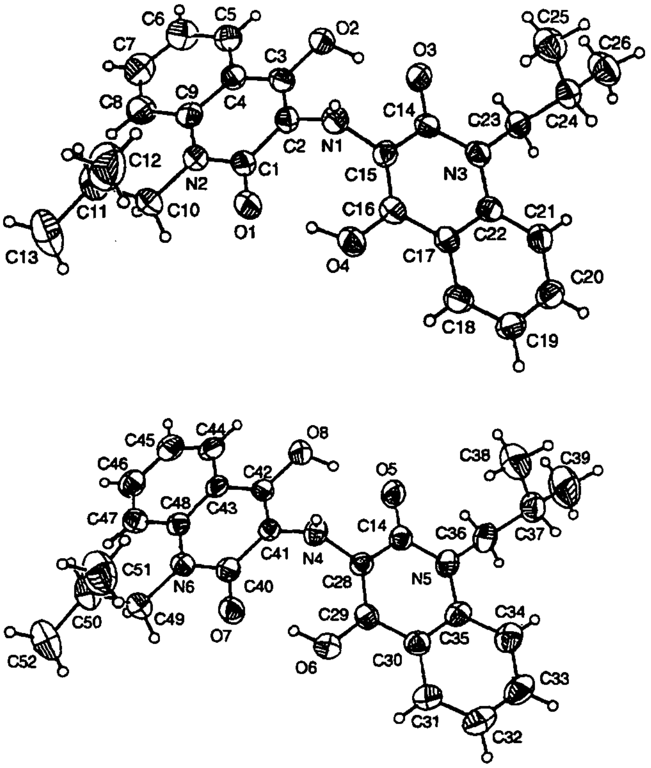

Fig. 1. Numbering scheme and thermal vibration ellipsoids for atoms of the two independent diamine molecules II.

moieties about the N – C bonds. The hydrogen bonds are characterized by appreciable lengthening of the O – H bonds (Table 1) and shortening of the H...O and O...O van der Waals distances. In this structure, these distances are as follows:

$H_{(4)}\cdots O_{(1)}$	1.60(4)	$O_{(4)}\cdots O_{(1)}$	2.584(3)
$H_{(2)}\cdots O_{(3)}$	1.53(4)	$O_{(2)}\cdots O_{(3)}$	2.554(3)
$H_{(6)}\cdots O_{(7)}$	1.62(4)	$O_{(6)}\cdots O_{(7)}$	2.564(3)
$H_{(8)}\cdots O_{(5)}$	1.64(4)	$O_{(8)}\cdots O_{(5)}$	2.594(3)

Furthermore, the values of the O–H...O angles (for $H_{(2)}$, $H_{(4)}$, $H_{(6)}$, and $H_{(8)}$ these are respectively 175, 167, 175 and 171°) are also typical for a hydrogen bond.

Both basis molecules contain moieties of condensed aromatic systems. In the first molecule, the moiety consisting of $N_{(2)}$ and $C_{(1)}-C_{(9)}$ atoms is planar within 0.04 Å; the moiety consisting of $N_{(3)}$, $C_{(14)}-C_{(22)}$ atoms is planar within 0.013 Å. The planes of these moieties form a 44.9° dihedral angle. We note that the $N_{(1)}$ atom deviates by 0.296 and 0.183 Å from the first and second plane respectively. For the second molecule, the indicated numbers are respectively 0.06 and 0.02 Å, 42.6°, 0.44 and 0.11 Å.

We can also note that in both molecules, the environment of the central nitrogen atoms is appreciably distorted compared with the trigonal–planar environment characteristic for atoms in an sp^2 hybridization state: while the C – N – C angles are close to 120°, the C–N–H angles lie within the range 108-111° (see Table 2), which is more typical for atoms in an sp^3 hybridization state. Such distortion of the bond angles may be due to the effect of the unshared electron pair.

Summarizing our investigation, we consider it advisable to give a number of recommendations concerning obtaining 3-amino-1R-2-oxo-4-hydroxyquinolines. Considering the high light-sensitivity of the indicated compounds, the last stage in their synthesis (hydrazinolysis of 1R-2-oxo-3-(1-pyridino)quinolin-4-olates [2]) must be carried out in a reaction vessel protected from light. Similar precautions should be observed in crystallization, drying, and storage of the compounds obtained. According to our data, 3-amino-2-oxo-4-hydroxyquinolines obtained with consideration of the indicated requirements can be stored without appreciable chemical changes for 1 to 1.5 months.

TABLE 1. Bond Lengths (Å) in Diamine II Molecule

Bond	l	Bond	l
$O_{(1)}$—$C_{(1)}$	1,249(4)	$O_{(5)}$—$C_{(27)}$	1,267(4)
$O_{(2)}$—$C_{(3)}$	1,347(4)	$O_{(6)}$—$C_{(29)}$	1,331(4)
$O_{(2)}$—$H_{(2)}$	1,030(4)	$O_{(6)}$—$H_{(6)}$	0,940(3)
$O_{(3)}$—$C_{(14)}$	1,261(4)	$O_{(7)}$—$C_{(40)}$	1,259(4)
$O_{(4)}$—$C_{(16)}$	1,341(4)	$O_{(8)}$—$C_{(42)}$	1,349(4)
$O_{(4)}$—$H_{(4)}$	1,000(3)	$O_{(8)}$—$H_{(8)}$	0,960(3)
$N_{(1)}$—$C_{(15)}$	1,430(4)	$N_{(4)}$—$C_{(41)}$	1,423(4)
$N_{(1)}$—$C_{(2)}$	1,432(4)	$N_{(4)}$—$C_{(28)}$	1,440(4)
$N_{(1)}$—$H_{(1)}$	0,910(3)	$N_{(4)}$—$H_{(4A)}$	0,890(3)
$N_{(2)}$—$C_{(1)}$	1,379(4)	$N_{(5)}$—$C_{(27)}$	1,381(4)
$N_{(2)}$—$C_{(9)}$	1,398(4)	$N_{(5)}$—$C_{(35)}$	1,405(4)
$N_{(2)}$—$C_{(10)}$	1,482(4)	$N_{(5)}$—$C_{(36)}$	1,480(4)
$N_{(3)}$—$C_{(14)}$	1,366(4)	$N_{(6)}$—$C_{(40)}$	1,390(4)
$N_{(3)}$—$C_{(22)}$	1,399(4)	$N_{(6)}$—$C_{(48)}$	1,394(4)
$N_{(3)}$—$C_{(23)}$	1,489(4)	$N_{(6)}$—$C_{(49)}$	1,483(4)
$C_{(1)}$—$C_{(2)}$	1,451(5)	$C_{(27)}$—$C_{(28)}$	1,423(4)
$C_{(2)}$—$C_{(3)}$	1,360(5)	$C_{(28)}$—$C_{(29)}$	1,331(4)
$C_{(3)}$—$C_{(4)}$	1,433(5)	$C_{(29)}$—$C_{(30)}$	1,441(4)
$C_{(4)}$—$C_{(5)}$	1,401(5)	$C_{(30)}$—$C_{(35)}$	1,393(5)
$C_{(4)}$—$C_{(9)}$	1,407(5)	$C_{(30)}$—$C_{(31)}$	1,410(5)
$C_{(5)}$—$C_{(6)}$	1,361(5)	$C_{(31)}$—$C_{(32)}$	1,374(5)
$C_{(6)}$—$C_{(7)}$	1,383(6)	$C_{(32)}$—$C_{(33)}$	1,364(6)
$C_{(7)}$—$C_{(8)}$	1,388(6)	$C_{(33)}$—$C_{(34)}$	1,381(6)
$C_{(8)}$—$C_{(9)}$	1,405(5)	$C_{(34)}$—$C_{(35)}$	1,408(5)
$C_{(10)}$—$C_{(11)}$	1,525(6)	$C_{(36)}$—$C_{(37)}$	1,529(6)
$C_{(11)}$—$C_{(12)}$	1,501(8)	$C_{(37)}$—$C_{(39)}$	1,512(7)
$C_{(11)}$—$C_{(13)}$	1,543(7)	$C_{(37)}$—$C_{(38)}$	1,528(7)
$C_{(14)}$—$C_{(15)}$	1,444(4)	$C_{(40)}$—$C_{(41)}$	1,437(4)
$C_{(15)}$—$C_{(16)}$	1,363(4)	$C_{(41)}$—$C_{(42)}$	1,363(4)
$C_{(16)}$—$C_{(17)}$	1,435(4)	$C_{(42)}$—$C_{(43)}$	1,435(4)
$C_{(17)}$—$C_{(18)}$	1,406(5)	$C_{(43)}$—$C_{(48)}$	1,400(4)
$C_{(17)}$—$C_{(22)}$	1,409(4)	$C_{(43)}$—$C_{(44)}$	1,409(5)
$C_{(18)}$—$C_{(19)}$	1,370(5)	$C_{(44)}$—$C_{(45)}$	1,373(5)
$C_{(19)}$—$C_{(20)}$	1,386(5)	$C_{(45)}$—$C_{(46)}$	1,390(5)
$C_{(20)}$—$C_{(21)}$	1,362(5)	$C_{(46)}$—$C_{(47)}$	1,379(5)
$C_{(21)}$—$C_{(22)}$	1,399(5)	$C_{(47)}$—$C_{(48)}$	1,405(5)
$C_{(23)}$—$C_{(24)}$	1,534(5)	$C_{(49)}$—$C_{(50)}$	1,511(5)
$C_{(24)}$—$C_{(26)}$	1,513(5)	$C_{(50)}$—$C_{(51)}$	1,477(6)
$C_{(24)}$—$C_{(25)}$	1,520(6)	$C_{(50)}$—$C_{(52)}$	1,528(6)

TABLE 2. Bond Angles in the Structure of Diamine II

Angle	ω	Angle	ω
1	2	3	4
$C_{(3)}$—$O_{(2)}$—$H_{(2)}$	109,0(2)	$C_{(29)}$—$O_{(6)}$—$H_{(6)}$	112,0(2)
$C_{(16)}$—$O_{(4)}$—$H_{(4)}$	110,0(2)	$C_{(42)}$—$O_{(8)}$—$H_{(8)}$	114,0(2)
$C_{(15)}$—$N_{(1)}$—$C_{(2)}$	119,4(3)	$C_{(41)}$—$N_{(4)}$—$C_{(28)}$	120,1(3)
$C_{(15)}$—$N_{(1)}$—$H_{(1)}$	110,0(2)	$C_{(41)}$—$N_{(4)}$—$H_{(4A)}$	110,0(2)
$C_{(2)}$—$N_{(1)}$—$H_{(1)}$	111,0(2)	$C_{(28)}$—$N_{(4)}$—$H_{(4A)}$	108,0(2)
$C_{(1)}$—$N_{(2)}$—$C_{(9)}$	122,0(3)	$C_{(27)}$—$N_{(5)}$—$C_{(35)}$	120,9(3)
$C_{(1)}$—$N_{(2)}$—$C_{(10)}$	117,6(3)	$C_{(27)}$—$N_{(5)}$—$C_{(36)}$	118,4(3)
$C_{(9)}$—$N_{(2)}$—$C_{(10)}$	120,4(3)	$C_{(35)}$—$N_{(5)}$—$C_{(36)}$	120,7(3)
$C_{(14)}$—$N_{(3)}$—$C_{(22)}$	121,7(3)	$C_{(40)}$—$N_{(6)}$—$C_{(48)}$	121,2(3)
$C_{(14)}$—$N_{(3)}$—$C_{(23)}$	117,6(3)	$C_{(40)}$—$N_{(6)}$—$C_{(49)}$	117,4(3)
$C_{(22)}$—$N_{(3)}$—$C_{(23)}$	120,7(3)	$C_{(48)}$—$N_{(6)}$—$C_{(49)}$	121,5(3)

TABLE 2 (continued)

Angle	ω	Angle	ω
1	2	3	4
O(1)—C(1)—N(2)	120,2(3)	O(5)—C(27)—N(5)	118,7(3)
O(1)—C(1)—C(2)	121,8(3)	O(5)—C(27)—C(28)	122,4(3)
N(2)—C(1)—C(2)	118,0(3)	N(5)—C(27)—C(28)	118,9(3)
C(3)—C(2)—N(1)	122,7(3)	C(29)—C(28)—C(27)	121,4(3)
C(3)—C(2)—C(1)	120,7(3)	C(29)—C(28)—N(2)	121,0(3)
N(1)—C(2)—C(1)	116,5(3)	C(27)—C(28)—N(4)	117,4(3)
O(2)—C(3)—C(2)	122,7(3)	O(6)—C(29)—C(28)	124,1(3)
O(2)—C(3)—C(4)	116,8(3)	O(6)—C(29)—C(30)	116,6(3)
C(2)—C(3)—C(4)	120,5(3)	C(28)—C(29)—C(30)	119,2(3)
C(5)—C(4)—C(9)	119,3(3)	C(35)—C(30)—C(31)	119,6(3)
C(5)—C(4)—C(3)	121,8(3)	C(35)—C(30)—C(29)	119,5(3)
C(9)—C(4)—C(3)	118,8(3)	C(31)—C(30)—C(29)	120,7(3)
C(6)—C(5)—C(4)	121,6(4)	C(32)—C(31)—C(30)	120,2(4)
C(5)—C(6)—C(7)	119,1(4)	C(33)—C(32)—C(31)	119,9(4)
C(6)—C(7)—C(8)	121,5(4)	C(32)—C(33)—C(34)	121,8(4)
C(7)—C(8)—C(9)	119,5(4)	C(33)—C(34)—C(35)	119,2(4)
N(2)—C(9)—C(8)	121,4(3)	C(30)—C(35)—N(5)	119,6(3)
N(2)—C(9)—C(4)	119,8(3)	C(30)—C(35)—C(34)	119,3(3)
C(8)—C(9)—C(4)	118,8(3)	N(5)—C(35)—C(34)	121,2(3)
N(2)—C(10)—C(11)	114,0(3)	N(5)—C(36)—C(37)	115,2(3)
C(12)—C(11)—C(10)	113,2(4)	C(39)—C(37)—C(36)	108,9(4)
C(12)—C(11)—C(13)	111,2(5)	C(39)—C(37)—C(38)	110,5(4)
C(10)—C(11)—C(13)	107,8(4)	C(36)—C(37)—C(38)	113,2(3)
O(3)—C(14)—N(3)	120,2(3)	O(7)—C(40)—N(6)	119,3(3)
O(3)—C(14)—C(15)	120,8(3)	O(7)—C(40)—C(41)	122,4(3)
N(3)—C(14)—C(15)	118,9(3)	N(6)—C(40)—C(41)	118,3(3)
C(16)—C(15)—N(1)	121,2(3)	C(42)—C(41)—N(4)	122,2(3)
C(16)—C(15)—C(14)	120,7(3)	C(42)—C(41)—C(40)	121,1(3)
N(1)—C(15)—C(14)	118,0(3)	N(4)—C(41)—C(40)	116,6(3)
O(4)—C(16)—C(15)	123,0(3)	O(8)—C(42)—C(41)	122,6(3)
O(4)—C(16)—C(17)	117,3(3)	O(8)—C(42)—C(43)	117,3(3)
C(15)—C(16)—C(17)	119,7(3)	C(41)—C(42)—C(43)	120,0(3)
C(18)—C(17)—C(22)	119,2(3)	C(48)—C(43)—C(44)	119,5(3)
C(18)—C(17)—C(16)	121,5(3)	C(48)—C(43)—C(42)	118,9(3)
C(22)—C(17)—C(16)	119,4(3)	C(44)—C(43)—C(42)	121,6(3)
C(19)—C(18)—C(17)	120,9(4)	C(45)—C(44)—C(43)	121,3(4)
C(18)—C(19)—C(20)	119,2(4)	C(44)—C(45)—C(46)	118,7(4)
C(21)—C(20)—C(19)	121,3(4)	C(47)—C(46)—C(45)	121,6(4)
C(20)—C(21)—C(22)	120,7(3)	C(46)—C(47)—C(48)	120,1(4)
N(3)—C(22)—C(21)	121,9(3)	N(6)—C(48)—C(43)	120,5(3)
N(3)—C(22)—C(17)	119,5(3)	N(6)—C(48)—C(47)	120,6(3)
C(21)—C(22)—C(17)	118,6(3)	C(43)—C(48)—C(47)	118,9(3)
N(3)—C(23)—C(24)	114,5(3)	N(6)—C(49)—C(50)	114,1(3)
C(26)—C(24)—C(25)	110,6(4)	C(51)—C(50)—C(49)	114,7(4)
C(26)—C(24)—C(23)	108,2(3)	C(51)—C(50)—C(52)	112,5(4)
C(25)—C(24)—C(23)	112,5(3)	C(49)—C(50)—C(52)	107,5(4)

TABLE 3. Atomic Coordinates (Å × 10^4) and Equivalent Thermal Corrections (Å^2 × 10^2) in the Structure of Diamine II (isotropic thermal corrections are given for hydrogen atoms)

Atom	*x/a*	*y/b*	*z/c*	U_{eq}
1	2	3	4	5
$O_{(2)}$	3727(2)	9280(2)	4609(2)	60(1)
$O_{(3)}$	4339(2)	8644(2)	6035(1)	61(1)
$O_{(4)}$	1751(2)	11183(2)	7388(2)	57(1)
$N_{(1)}$	3261(3)	10592(2)	5953(2)	49(1)
$N_{(2)}$	604(3)	11319(2)	4680(2)	51(1)
$N_{(3)}$	3729(2)	8397(2)	7397(2)	48(1)
$C_{(1)}$	1323(3)	11240(2)	5346(2)	50(1)
$C_{(2)}$	2442(3)	10551(2)	5308(2)	46(1)
$C_{(3)}$	2723(3)	9955(2)	4657(2)	50(1)
$C_{(4)}$	1963(3)	10044(2)	3971(2)	50(1)
$C_{(5)}$	2249(4)	9463(3)	3281(2)	60(1)
$C_{(6)}$	1574(4)	9596(3)	2607(3)	74(1)
$C_{(7)}$	572(4)	10315(3)	2610(3)	72(1)
$C_{(8)}$	222(4)	10887(3)	3289(2)	64(1)
$C_{(9)}$	922(3)	10757(3)	3984(2)	50(1)
$C_{(10)}$	-531(4)	12031(3)	4721(3)	66(1)
$C_{(11)}$	-356(4)	13024(3)	4247(3)	81(1)
$C_{(12)}$	654(7)	13584(4)	4550(4)	118(2)
$C_{(13)}$	-1631(7)	13652(5)	4303(5)	139(3)
$C_{(14)}$	3748(3)	8970(2)	6681(2)	46(1)
$C_{(15)}$	3096(3)	9956(2)	6678(2)	45(1)
$C_{(16)}$	2409(3)	10289(2)	7367(2)	46(1)
$C_{(17)}$	2391(3)	9677(2)	8114(2)	45(1)
$C_{(18)}$	1705(3)	9997(3)	8839(2)	56(1)
$C_{(19)}$	1694(4)	9405(3)	9549(2)	63(1)
$C_{(20)}$	2359(4)	8474(3)	9546(2)	60(1)
$C_{(21)}$	3042(3)	8145(3)	8856(2)	52(1)
$C_{(22)}$	3070(3)	8731(3)	8122(2)	45(1)
$C_{(23)}$	4413(3)	7386(2)	7379(2)	50(1)
$C_{(24)}$	5734(3)	7333(3)	7681(2)	55(1)
$C_{(25)}$	6585(4)	8019(3)	7173(3)	75(1)
$C_{(26)}$	6284(5)	6258(3)	7655(4)	84(2)
$O_{(5)}$	2645(2)	5034(2)	10652(1)	61(1)
$O_{(6)}$	1474(2)	6398(2)	8073(2)	60(1)
$O_{(7)}$	2921(2)	4974(2)	7436(1)	60(1)
$O_{(8)}$	4377(2)	3783(2)	10000(1)	55(1)
$N_{(4)}$	3401(3)	5428(2)	9004(2)	46(1)
$N_{(5)}$	809(3)	6012(2)	10595(2)	50(1)
$N_{(6)}$	3802(2)	3368(2)	7560(2)	46(1)
$C_{(27)}$	1898(3)	5563(3)	10214(2)	49(1)
$C_{(28)}$	2161(3)	5743(2)	9352(2)	42(1)
$C_{(29)}$	1313(3)	6258(2)	8887(2)	45(1)
$C_{(30)}$	184(3)	6732(3)	9287(2)	50(1)
$C_{(31)}$	-654(4)	7360(3)	8833(3)	64(1)
$C_{(32)}$	-1654(4)	7894(3)	9232(3)	74(1)
$C_{(33)}$	-1850(4)	7803(3)	10069(3)	70(1)
$C_{(34)}$	-1073(4)	7174(3)	10536(3)	61(1)
$C_{(35)}$	-28(3)	6635(2)	10141(2)	48(1)

TABLE 3 (continued)

Atom	*x/a*	*y/b*	*z/c*	U_{eq}
1	2	3	4	5
C(36)	531(4)	5800(3)	11488(2)	58(1)
C(37)	890(4)	6591(3)	12047(2)	67(1)
C(38)	2287(5)	6759(4)	11946(3)	89(2)
C(39)	499(7)	6293(5)	12932(3)	120(2)
C(40)	3436(3)	4295(3)	7884(2)	48(1)
C(41)	3671(3)	4442(2)	8722(2)	42(1)
C(42)	4198(3)	3684(3)	9200(2)	44(1)
C(43)	4566(3)	2729(2)	8860(2)	45(1)
C(44)	5077(3)	1919(3)	9342(2)	57(1)
C(45)	5402(4)	1001(3)	9017(3)	62(1)
C(46)	5198(4)	874(3)	8200(3)	63(1)
C(47)	4691(3)	1648(3)	7711(2)	54(1)
C(48)	4357(3)	2590(2)	8038(2)	45(1)
C(49)	3562(4)	3237(3)	6691(2)	55(1)
C(50)	4699(4)	3330(3)	6088(2)	76(1)
C(51)	5338(5)	4252(4)	6141(3)	109(2)
C(52)	4290(6)	3204(5)	5227(3)	101(2)
H(2)	3930(32)	9031(25)	5195(22)	72
H(4)	1586(32)	11438(25)	6814(22)	68
H(1)	4075(32)	10538(24)	5748(20)	59
H(5)	2929(34)	8950(26)	3324(21)	72
H(6A)	1790(37)	9259(29)	2111(24)	89
H(7)	95(37)	10500(28)	2156(24)	86
H(8A)	-450(35)	11399(27)	3261(22)	77
H(10A)	-693(33)	12181(26)	5320(23)	79
H(10B)	-1213(36)	11686(27)	4479(22)	79
H(11)	54(38)	13000(29)	3614(25)	97
H(12A)	701(49)	14236(41)	4216(32)	142
H(12B)	1505(51)	13175(40)	4495(32)	142
H(12C)	326(52)	13742(39)	5142(32)	142
H(13A)	-2268(60)	13304(47)	4077(38)	167
H(13B)	-1424(56)	14310(45)	3987(36)	167
H(13C)	-1857(57)	13733(44)	4957(35)	167
H(18)	1323(33)	10648(26)	8809(21)	67
H(19)	1213(34)	9628(26)	10030(22)	75
H(20)	2351(33)	8057(26)	10051(22)	71
H(21)	3504(31)	7494(25)	8882(19)	62
H(23A)	4462(30)	7216(23)	6781(21)	60
H(23B)	3872(31)	6960(24)	7728(20)	60
H(24)	5691(30)	7540(24)	8296(21)	66
H(25A)	6210(37)	8739(30)	7193(23)	90
H(25B)	6685(37)	7770(29)	6588(25)	90
H(25C)	7421(39)	7991(29)	7395(23)	90
H(26A)	5739(40)	5812(31)	7973(26)	101
H(26B)	7094(43)	6172(31)	7888(25)	101
H(26C)	6444(40)	6050(31)	7044(26)	101
H(6)	2041(34)	5895(26)	7836(21)	71
H(8)	3801(32)	4276(25)	10261(20)	66
H(4A)	3946(31)	5557(24)	9358(20)	55
H(31)	-508(34)	7407(27)	8247(23)	76
H(32)	-2200(37)	8334(29)	8895(23)	89
H(33)	-2545(37)	8159(27)	10392(23)	84

TABLE 3 (continued)

Atom	*x/a*	*y/b*	*z/c*	U_{eq}
1	2	3	4	5
H(34)	-1185(33)	7131(26)	11124(22)	73
H(36A)	945(32)	5120(27)	11623(21)	70
H(36B)	-337(35)	5703(25)	11569(21)	70
H(37)	519(34)	7265(28)	11869(22)	81
H(38A)	2478(41)	6982(31)	11313(27)	107
H(38B)	2743(43)	6125(34)	12012(28)	107
H(38C)	2512(39)	7316(33)	12309(26)	107
H(39A)	-321(53)	6124(43)	12974(34)	144
H(39B)	693(52)	6783(40)	13307(33)	144
H(39C)	972(51)	5568(40)	13042(32)	144
H(44)	5184(32)	2031(25)	9897(21)	69
H(45)	5745(34)	445(27)	9386(21)	75
H(46)	5440(34)	242(27)	7952(22)	76
H(47)	4563(31)	1525(24)	7122(21)	65
H(49A)	2887(33)	3737(25)	6552(20)	66
H(49B)	3231(32)	2566(26)	6666(20)	66
H(50)	5362(25)	2711(23)	6220(5)	91
H(51A)	6163(19)	4242(4)	5697(10)	131
H(51B)	5632(8)	4296(4)	6765(14)	131
H(51C)	4693(14)	4904(15)	6001(4)	131
H(52A)	3853(46)	2593(37)	5162(28)	121
H(52B)	4899(47)	3344(36)	4829(30)	121
H(52C)	3735(45)	3862(35)	5069(28)	121

EXPERIMENTAL

The PMR spectrum of diamine II was recorded on a Bruker WP-100 SY, internal standard TMS. The mass spectrum was recorded on a Finnigan MAT Incos 50 quadrupole spectrometer in the full scanning mode in the range 33-700 *m/z*, ionization by electron impact at 70 eV, direct injection, heating rate ~5°/sec.

Di(1-isobutyl-2-oxo-4-hydroxyquinolin-3-yl)amine (II). 2.32 g (0.01 moles) of freshly obtained 3-amino-1-isobutyl-2-oxo-4-hydroxyquinoline I [2] was allowed to stand at room temperature in an illuminated location for 4-5 h, after which it was treated with 20 ml of a 2% aqueous solution of AcOH, filtered, washed with water, and dried. Obtained: 2.23 g (quantitative yield) diamine II. T_{mp} 183-184°C (diethyl ether). R_f 0.65 (Silufol UV-254, hexane–ether–2-propanol, 6:6:1). For comparison: the starting amine I in the same system has the value R_f 0.38. PMR spectrum (DMSO-D_6): 13.47 (2H, s, OH); 8.10-7.25 (8H, m, H_{arom}); 5.61 (1H, s, NH); 4.22 (4H, d, NCH_2); 2.12 (2H, m, $CH(Me)_2$); 0.89 ppm (12H, d, CH_3). Mass spectrum, *m/z* (relative intensity, %): 447 (100) $[M]^+$, 187 (40), 161 (94), 146 (33), 120 (37), 77 (29), 57 (43), 41 (70). Found, %: C 69.81; H 6.50; N 9.40. $C_{26}H_{29}N_3O_4$. Calculated, %: C 69.78; H 6.53; N 9.39.

The x-ray diffraction study was done on a Syntex $P2_1$ automatic four-circle diffractometer in monochromatic molybdenum radiation (λ = 0.71073 Å). The unit cell parameters were refined from 24 reflections in the interval of angles $28 \leq 2\theta \leq 30°$. The intensities of 3791 reflections for determination of the structure were measured by the $\theta/2\theta$ scanning method at a scanning rate from 3 to 30°/min in the interval $5 \leq 2\theta \leq 55°$. In calculating the structure factors, we took into account the Lorentz and polarization corrections, with no correction for absorption. The structure was deciphered by the direct method and refined by the least-squares method using 3775 independent nonequivalent reflections with $I(hkl) > 4\sigma I(hkl)$ in the full-matrix anisotropic approximation for nonhydrogen atoms of the structure (the hydrogen atoms were found by a difference synthesis and refined in the isotropic approximation). In the least-squares refinement process, we took into account the correction for extinction of the radiation. The final value of the *R* factor was 4.18% from the 3775 observed reflections (4.43% using the entire data file of reflections). The atomic coordinates are presented in Table 3.

Basic crystallographic data on the structure of diamine II: triclinic crystal system; at 20°C, a = 10.729(2), b = 13.434(3), c = 16.304(4) Å, α = 87.06(2)°, β = 85.20(2)°, γ = 85.10(2)°, V = 2331.0(8) Å^3, d_{calc} = 1.275 g/cm^3, $F(000)$ = 952, absorption coefficient μ (MoKα) 0.087 mm^{-1}, space group P-1, Z = 4.

REFERENCES

1. I. V. Ukrainets, S. G. Taran, O. V. Gorokhova, O. L. Kodolova, and A. V. Turov, Khim. Geterotsikl. Soedin., No. 7, 928 (1997).
2. I. V. Ukrainets, S. G. Taran, L. V. Sidorenko, O. V. Gorokhova, A. A. Ogirenko, A. A. Turov, and N. I. Filimonova, Khim. Geterotsikl. Soedin., No. 8, 1113 (1996).