STRUCTURE
OF ORGANIC COMPOUNDS

X-ray Mapping in Heterocyclic Design:
IX. X-ray Structure Investigation of Conjugated Aminodienes

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Abstract—The single-crystal structures of two aminodienes containing an oxazole fragment, namely, 1-piperidyl-4-[5-(4-nitrophenyl)-oxazol-2-yl]-buta-1,3-diene $C_{18}H_{19}N_3O_3$ (**IIa**) and 1-hexamethyleneimine-4-[5-(4-nitrophenyl)-oxazol-2-yl]-buta-1,3-diene $C_{19}H_{21}N_3O_3$ (**IIb**), are studied by X-ray diffraction. Structures **IIa** [$a = 16.181(6)$ Å, $b = 5.939(3)$ Å, $c = 17.337(9)$ Å, $\beta = 96.13(2)°$, $Z = 4$, and space group $P2_1$] and **IIb** [$a = 7.4704(11)$ Å, $b = 10.9904(19)$ Å, $c = 43.434(6)$ Å, $\beta = 91.24(1)°$, $Z = 8$, and space group $P2_1/c$] are solved by the direct method and refined to $R = 0.060$ and 0.238, respectively. Although the ring sizes of the cyclic amines in compounds **IIa** and **IIb** are different, the designs of two structures are identical. Each structure contains two topologically identical but crystallographically independent molecules. In structure **IIa**, the intramolecular hydrogen bonds between the N atoms of the oxazole fragments and the H atoms of the diene fragments are formed. In structure **IIb**, similar bonds are absent. © 2002 MAIK "Nauka/Interperiodica".

INTRODUCTION

This study continues the series of our structural investigations of heterocyclic compounds that are able to enter readily into different rearrangements and ring-transformation reactions [1–20]. By analogy with our previous studies, we determined step-by-step the structures of all the intermediates and final products of the multistage cyclization and recyclization reactions. The data on the molecular structures of 1-piperidyl-4-[5-(4-nitrophenyl)-oxazol-2-yl] $C_{18}H_{19}N_3O_3$ (**IIa**) and 1-hexamethyleneimine-4-[5-(4-nitrophenyl)-oxazol-2-yl]-buta-1,3-diene $C_{19}H_{21}N_3O_3$ (**IIb**), which are considered in this paper, are not available in the Cambridge Structural Database (version 11.01) [21].

Compound **IIa** was synthesized by scheme *1*[1]
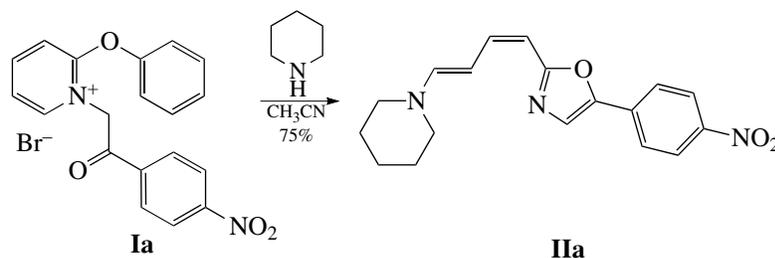
according to the following procedure: 1-(4-Nitrophenacyl)-2-phenoxypyridinium bromide (0.1 g, 0.24 mmol) was dissolved in acetonitrile. Piperidine (0.04 g, 0.48 mmol) was added on stirring at room temperature to the yellow solution obtained. The solution became dark red. The mixture was allowed to stand at room temperature for two days. Violet needle crystals that precipitated were filtered off and washed with an ether. An additional amount of pure product could be isolated by the dilution of the filtrate with water followed by filtration of the precipitate. The yield was 0.06 g (75%) of 1-piperidyl-4-[5-(4-nitrophenyl)-oxazol-2-yl]}-buta-

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[1] With the participation of T.A. Smirnova.

1,3-diene (**IIa**). The synthesis and other transformations of the initial pyridinium salt will be reported elsewhere.

The synthesis of compound **IIb** by scheme *2* was described earlier in [22]:

Ia **IIa**
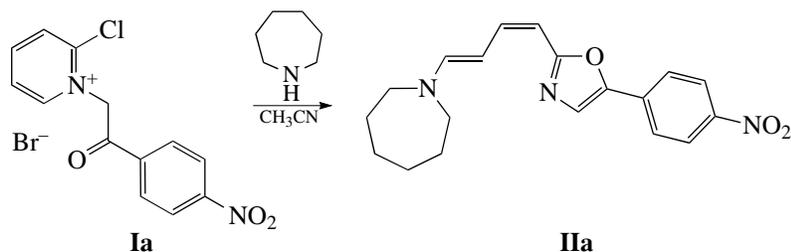
According to [23], compounds **IIa** and **IIb** exhibit an antimicrobal activity. For this reason, their crystal structures have been studied in detail. The structural characteristics of these compounds are also of particular interest from the standpoint of X-ray mapping of the structures, because we studied earlier the structures of a series of 2-substituted *N*-phenacylpyridinium salts and the products of their transformations, which belong to various classes of heterocycles, for example, pyridone derivatives such as oxazolopyridine [1, 2, 6], cationic thiazolopyridine [24], and indolizine [4, 11].

EXPERIMENTAL

The intensities of diffraction reflections for **IIa** were measured at room temperature on a CAD4 four-circle diffractometer [25] (MoK_α radiation, graphite monochromator, ω scan mode). The experimental data for

IIb were collected at room temperature in the dynamic mode on a KM4-CCD four-circle diffractometer [26] (MoK_α radiation, graphite monochromator, CCD

Table 2. Bond lengths d (Å) in structures **IIa** and **IIb** (molecules *A* and *B*)

Bond	d	
	IIa	**IIb**
N(1)–C(2)	1.486(8)	1.473(12)
N(1)–C(6)	1.403(8)	
N(1)–C(7)		1.473(12)
N(1)–C(8)	1.351(4)	1.43(2)
C(2)–C(3)	1.433(8)	1.436(10)
C(3)–C(4)	1.533(11)	1.436(10)
C(4)–C(5)	1.479(11)	1.436(10)
C(5)–C(6)	1.417(7)	1.436(10)
C(6)–C(7)		1.436(10)
C(8)–C(9)	1.335(4)	1.37(2)
C(9)–C(10)	1.409(4)	1.408(18)
C(10)–C(11)	1.345(4)	1.36(2)
C(11)–C(12)	1.421(4)	1.45(2)
C(12)–N(2)	1.309(3)	1.278(18)
C(12)–O(1)	1.372(3)	1.356(16)
C(13)–O(1)	1.380(3)	1.401(14)
C(13)–C(14)	1.350(4)	1.275(19)
C(13)–C(15)	1.441(4)	1.52(2)
C(14)–N(2)	1.360(4)	1.39(2)
C(15)–C(16)	1.407(7)	1.457(13)
C(15)–C(20)	1.380(7)	1.457(13)
C(16)–C(17)	1.359(10)	1.349(16)
C(17)–C(18)	1.344(7)	1.404(13)
C(18)–C(19)	1.400(7)	1.404(13)
C(18)–N(3)	1.458(4)	1.44(2)
C(19)–C(20)	1.356(11)	1.349(15)
N(3)–O(31)	1.214(2)	1.248(11)
N(3)–O(32)	1.214(2)	1.248(11)

Table 1. Crystallographic characteristics and details of the X-ray diffraction experiment and structure-refinement parameters for **IIa** and **IIb**

Compound	$C_{18}H_{19}N_3O_3$ (**IIa**)	$C_{19}H_{21}N_3O_3$ (**IIb**)
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1$	$P2_1/c$
a, Å	16.181(6)	7.470(1)
b, Å	5.939(3)	10.990(2)
c, Å	17.337(9)	43.434(6)
β, deg	96.13(2)	91.24(1)
V, Å^3	1656.5(13)	3565.2(10)
Z	4	8
ρ_{calcd}, g/cm^3	1.305	1.265
$\mu(MoK_\alpha)$, cm^{-1}	0.091	0.087
Crystal size, mm	$0.35 \times 0.40 \times 0.45$	$0.10 \times 0.12 \times 0.02$
θ_{max}, deg	28	30
No. of reflections with $I \geq 2\sigma(I)$/No. of parameters	4218/290	2131/177
R_1/wR_2	0.062/0.130	0.238/0.462
$\Delta\rho_{max}/\Delta\rho_{min}$, e/Å^3	0.172/–0.130	0.322/–0.320
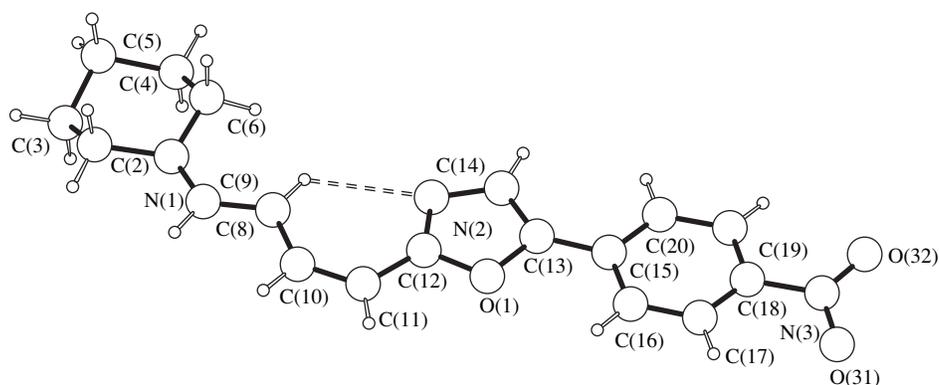
Fig. 1. Molecular structure of **IIa** and the atomic numbering.
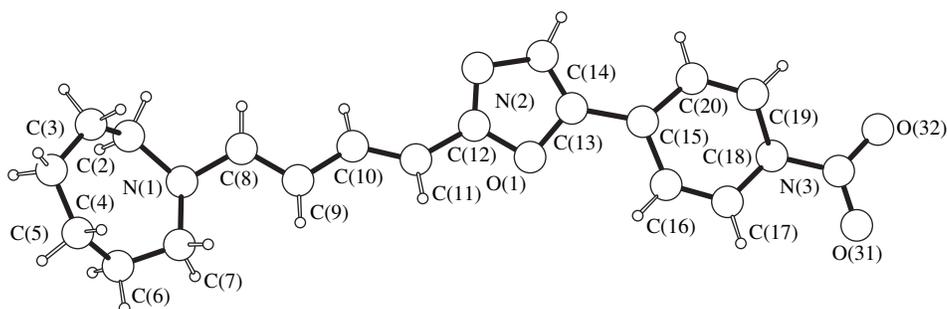
Fig. 2. Molecular structure of **IIb** and the atomic numbering.

detector, crystal-to-detector distance 51.7 mm). The unit cell parameters were determined and refined using 25 reflections in the θ range 14°–16° for **IIa** and 9640 reflections with $I > 6\sigma(I)$ from the whole data-collection range for **IIb**. The main experimental parameters and crystal data for compounds **IIa** and **IIb** are summarized in Table 1.

Since the crystals of the compounds studied were small and had small coefficients of linear absorption, the correction for absorption was not introduced. The primary processing of the experimental data for **IIa** was performed with the WinGX98 program package [27]. The integral intensities for **IIb** were obtained and primarily processed with the KM4 program package [28]. All the following calculations were performed with the SHELX97 program package [29]. The crystal structures were solved by the direct method. All the non-hydrogen atoms in structures **IIa** and **IIb** were refined in the anisotropic and isotropic approximations, respectively. The system of atomic numbering in the two compounds is the same. Since structures **IIa** and **IIb** contained two crystallographically independent molecules (A and B), the full-matrix least-squares refinement was performed under the restriction that the lengths of the analogous bonds in each structure were identical. The thermal parameters of the corresponding atoms were also kept equal. The hydrogen atoms in both structures were located from geometric considerations and refined

within a rider model together with the corresponding carbon atoms. The thermal parameters of the hydrogen atoms were calculated from those of the corresponding carbon atoms $[U_{iso}(H) = 1.2U_{iso/eq}(C)]$. The structural data for crystals **IIa** and **IIb** (CIF files) were deposited in the Cambridge Structural Database [21], CCDC nos. 191971 and 191972. The interatomic distances in structures **IIa** and **IIb** are listed in Table 2. The drawings of the molecular structures **IIa** and **IIb** obtained with the PLUTON96 program [30] are shown in Figs. 1 and 2, respectively. The interatomic and intermolecular contacts involving hydrogen atoms for structures **IIa** and **IIb**, which were calculated using the PARST95 program [31], are listed in Tables 3 and 4, respectively.

RESULTS AND DISCUSSION

The compositions of the compounds studied differ only in the size of the cyclic amine, which is the six-membered piperidine ring in **IIa** and the seven-membered hexamethyleneimine ring in **IIb**. However, the three-dimensional structures of the compounds studied differ fundamentally. Compound **IIa** is the 1*E*,3*Z*-isomer, whereas compound **IIb** has the 1*E*,3*E*-configuration of the substituted diene fragment. The formation of the *E,Z*- and *E,E*-isomers was discussed earlier in [22].

Table 3. Parameters of interatomic contacts in **IIa**

D–H	$d(D$–H$)$	$d(D \cdots A)$	$d(H \cdots A)$	ωDHA	A	Symmetry operation
C(9A)–H(9A)	0.93	3.07(1)	2.490(8)	120.8(5)	N(2A)	$[x; y; z]$
C(16A)–H(16A)	0.93	2.88(1)	2.590(6)	98.7(5)	O(1A)	$[x; y; z]$
C(17A)–H(17A)	0.93	2.71(1)	2.408(8)	98.4(6)	O(31A)	$[x; y; z]$
C(19A)–H(19A)	0.93	2.71(1)	2.445(7)	96.5(5)	O(32A)	$[x; y; z]$
C(9B)–H(9B)	0.93	3.10(1)	2.501(8)	122.4(5)	N(2B)	$[x; y; z]$
C(17B)–H(17B)	0.93	2.72(1)	2.411(7)	99.1(5)	O(31B)	$[x; y; z]$
C(19B)–H(19B)	0.93	2.74(1)	2.484(8)	96.0(6)	O(32B)	$[x; y; z]$
C(20B)–H(20B)	0.93	2.84(1)	2.505(6)	101.1(6)	O(1B)	$[x; y; z]$
C(3A)–H(3A1)	0.97	3.76(1)	2.975(6)	139.2(6)	N(3B)	$[1-x; 1/2+y; 2-z]$
C(3A)–H(3A1)	0.97	3.71(1)	2.781(7)	160.6(6)	O(32B)	$[1-x; 1/2+y; 2-z]$
C(3A)–H(3A2)	0.97	3.73(1)	2.786(8)	164.1(6)	O(32B)	$[1-x; y-1/2; 2-z]$
C(5A)–H(5A1)	0.97	3.61(1)	2.745(7)	148.6(6)	O(31B)	$[1-x; y-1/2; 2-z]$
C(11A)–H(11A)	0.93	3.33(1)	2.675(7)	128.3(5)	O(31A)	$[2-x; y+3/2; 2-z]$
C(10A)–H(10A)	0.93	3.38(1)	2.757(5)	125.4(5)	O(31A)	$[2-x; y+3/2; 2-z]$
C(17A)–H(17A)	0.93	3.62(1)	2.848(5)	140.6(5)	O(1A)	$[2-x; y-1/2; 2-z]$
C(19A)–H(19A)	0.93	3.36(1)	2.529(6)	148.8(5)	O(31B)	$[x; y-2; z]$
C(3B)–H(3B1)	0.97	3.61(1)	2.763(6)	146.1(6)	O(32A)	$[2-x; y+1/2; 1-z]$
C(5B)–H(5B2)	0.97	3.69(1)	2.959(8)	132.9(7)	O(31A)	$[2-x; y-1/2; 1-z]$
C(11B)–H(11B)	0.93	3.32(1)	2.694(7)	125.1(5)	O(32B)	$[1-x; y-3/2; 1-z]$
C(10B)–H(10B)	0.93	3.32(1)	2.704(5)	124.2(5)	O(32B)	$[1-x; y-3/2; 1-z]$
C(14B)–H(14B)	0.93	3.66(1)	2.988(6)	130.4(5)	O(32A)	$[x; 1+y; z]$
C(17B)–H(17B)	0.93	3.41(1)	2.561(6)	151.1(5)	O(32A)	$[x; y+2; z]$
C(19B)–H(19B)	0.93	3.59(1)	2.780(5)	145.6(5)	O(1B)	$[1-x; y+1/2; 1-z]$

Note: D is a donor, A is an acceptor, and H is a hydrogen atom; the d distances and ω angles are given in Å and degrees, respectively.

The structure of molecules **IIa** is convenient for the formation of the C(9)H(9)$\cdots$N(2) intramolecular hydrogen bond (Table 3, Fig. 1), which cannot be formed in the molecules of compound **IIb** (Fig. 2). Moreover, molecule **IIb** is longer than molecule **IIa** (the difference in the lengths of the similar N(1)$\cdots$N(3) fragments is 0.836 Å). As a consequence of these two factors, the molecular packings and the habits of the crystals are different. For example, compound **IIa** crystallizes as dark red well-edged prisms, whereas crystals **IIb** are very fine plates that are colored dark red, almost black, and exhibit metallic iridescence. Because of the very small size of crystals **IIb**, we managed to collect a relatively satisfactory set of intensities (suitable only for the determination of the model) using a highly sensitive CCD detector. It was assumed earlier [22] that the intense crystal color is due to the intramolecular charge transfer from the amino group to the nitro group via the azapolyene system according to scheme *3* (illustrated by the example of 1*E*,3*Z*-isomer).
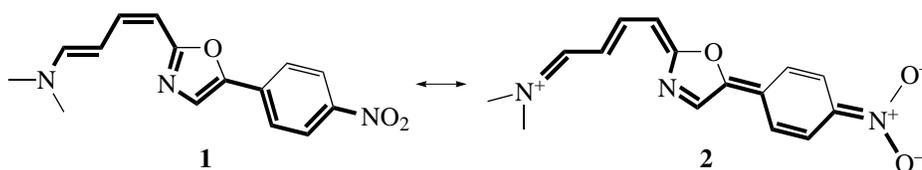
The data of the X-ray diffraction study reveal the degree of contribution of the charge-transfer structures to the structures of the dienes under consideration. Now, we compare the bond lengths in the conjugation chains between the amino and nitro groups obtained in this study with those in the hypothetical resonance structures **1** and **2**. First, let us consider the nitrophenyl group. The C–N distances between the phenyl radical and the NO$_2$ group and the N–O distances in crystals **IIa** and **IIb** are slightly different, but in both crys-

Table 4. Parameters of interatomic contacts in **IIb**

D–H	$d(D$–H$)$	$d(D \cdots A)$	$d(H \cdots A)$	ωDHA	A	Symmetry operation
C(7A)–H(7A2)	0.97	3.82(6)	2.89(1)	161(3)	O(31B)	[x; y; z]
C(10A)–H(10A)	0.93	3.13(3)	2.93(2)	94(1)	N(2A)	[x; y; z]
C(16A)–H(16A)	0.93	2.91(2)	2.59(1)	101(1)	O(1A)	[x; y; z]
C(17A)–H(17A)	0.93	2.75(2)	2.48(2)	97(1)	O(31A)	[x; y; z]
C(19A)–H(19A)	0.93	2.73(2)	2.43(2)	99(1)	O(32A)	[x; y; z]
C(3B)–H(3B1)	0.97	3.65(4)	2.75(3)	154(2)	O(32A)	[x; y; z]
C(7B)–H(7B2)	0.97	3.85(6)	2.93(3)	158(2)	O(31A)	[x; y; z]
C(10B)–H(10B)	0.93	3.09(2)	2.85(2)	96(1)	N(2B)	[x; y; z]
C(16B)–H(16B)	0.93	2.97(2)	2.65(1)	101(1)	O(1B)	[x; y; z]
C(17B)–H(17B)	0.93	2.70(2)	2.40(2)	98(1)	O(31B)	[x; y; z]
C(19B)–H(19B)	0.93	2.78(3)	2.51(2)	97(1)	O(32B)	[x; y; z]
C(2A)–H(2A1)	0.97	3.54(2)	2.63(1)	157(1)	O(31A)	[1 − x; y − 1/2; 1/2 − z]
C(8A)–H(8A)	0.93	3.52(3)	2.72(2)	145(1)	O(31A)	[1 − x; y − 1/2; 1/2 − z]
C(2A)–H(2A2)	0.97	3.71(4)	2.94(3)	137(2)	O(32B)	[x − 1; y; z]
C(6A)–H(6A2)	0.97	3.51(2)	2.73(2)	138(1)	O(32A)	[x − 1; y; z]
C(16A)–H(16A)	0.93	3.64(3)	2.86(2)	142(1)	N(2A)	[x − 1; y; z]
C(2B)–H(2B1)	0.97	3.34(2)	2.51(2)	144(1)	O(31B)	[2 − x; y − 1/2; 1/2 − z]
C(8B)–H(8B)	0.93	3.50(3)	2.64(1)	155(1)	O(31B)	[2 − x; y − 1/2; 1/2 − z]
C(14A)–H(14A)	0.93	3.74(3)	2.93(2)	146(1)	O(1B)	[2 − x; y − 1/2; 1/2 − z]
C(16B)–H(16B)	0.93	3.57(2)	2.69(2)	159(1)	N(2B)	[2 − x; y + 1/2; 1/2 − z]
C(11B)–H(11B)	0.93	3.81(3)	2.98(2)	150(1)	N(2A)	[2 − x; y + 1/2; 1/2 − z]
C(6B)–H(6B2)	0.97	3.54(3)	2.73(1)	142(1)	O(32B)	[2 − x; y + 1/2; 1/2 − z]

Note: D is a donor, A is an acceptor, and H is a hydrogen atom; the d distances and ω angles are given in Å and degrees, respectively.

tals, their values correspond better to structure **1**. The phenyl ring is slightly distorted, and its geometric parameters correspond better to structure **2**, because the C(16)–C(17) and C(19)–C20) bonds [1.35(2) Å] are significantly shorter than all the other bonds in the ring [1.40(2)–1.46(2) Å]. Second, we consider the oxazole fragment. The five-membered rings in molecules **IIa** and **IIb** have different geometries. Actually, in molecule **IIa**, the double bonds in the C(12)N(2)C(14)C(13) azadiene fragment are evidently delocalized, since the C(12)–N(2), N(2)–C(14), and C(13)–C(14) distances are smoothened [1.31(1), 1.36(1), and 1.35(2) Å, respectively]. In the similar azadiene structural fragment of molecule **IIb**, the corresponding bond lengths alternate to a larger degree [1.28(2), 1.39(2), and 1.28(2) Å, respectively], which counts in favor of the contribution of structure **1** to the structure of the five-membered ring. Third, consider the diene fragment. The lengths of the formally single and double bonds in the butadiene fragments of both molecules agree more closely with structure **1**, but the bond-length alternation in this fragment of compound **IIa** is more pronounced than that in **IIb**. It is difficult to draw a more definite conclusion regarding the contribution of structures **1** and **2** to the geometry of the molecules because of the poor quality of the experimental data for crystal **IIb**.

It follows from the totality of data obtained that structure **2**, in which charge transfer is observed, contributes to the structures of both the compounds considered; however, the real molecular structures are intermediate between the resonance structures **1** and **2**.

Conformational analysis of the cyclic amines was not the object of this study; however, the parameters of ring puckering can be calculated from the atomic coordinates.

ACKNOWLEDGMENTS

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Translated by I. Polyakova