*Chemistry of Natural Compounds, Vol. 31, No. 5, 1995*

# ALKALOIDS OF *Arundo donax*. III. RECONSIDERATION OF THE STRUCTURES OF DONAXARINE AND DONAXARIDINE

**V. U. Khuzhaev, B. Tashkhodzhaev, and S. F. Aripova**

UDC 547.944/945+548.737

*The alkaloid composition of the epigeal part of* Arundo donax *has been studied, and from an ether extract have been isolated the alkaloids donaxine, arundine, deoxyvasicinone, N-phenyl-β-naphthylamine, donaxarine, and donaxaridine. On the basis of an x-ray structural analysis, new structures have been established for donaxarine and donaxaridine: spiro[(N-methylpyrrolidin-2-one)-3,4'-(2'-methyl-3',1'-benzoxazine)] and 3-hydroxy-3-(2'-aminophenyl)-N-methyl-2-pyrrolidone, respectively. Spectral characteristics of the derivatives obtained are given and their chemical interconversions are described.*

Continuing an investigation of the alkaloid composition of plants of the genus *Arundo* [1-3], we have studied *A. donax* L., gathered from a new growth site (environs of the village of Kumkishlak, Kashkarandar'inskaya province). Chloroform extraction of the epigeal part showed the presence of a total of 0.3% of alkaloids — 0.1% of ether fraction and 0.2% of chloroform fraction. By chromatographing the ether fraction on an alumina column we isolated donaxine, arundine, deoxyvasicinone, N-phenyl-β-naphthylamine, and bases (**1**) with mp 218-220°C and (**2**) with mp 178-180°C. The known alkaloids were identified by direct TLC comparison with authentic specimens, mixed melting points, and IR spectra.

On acetylation with acetic anhydride in pyridine, base (**1**) formed an amorphous acetyl derivative, (**3**). The mass spectrum of (**3**) showed a peak of the molecular ion with *m/z* 274, and peaks of ions with *m/z* 231, 217, 189, and 146, formed by the splitting out of an acetyl group and further fragmentation of the molecular ion.

The acetylation of (**2**) with acetic anhydride in pyridine gave an oily diacetyl derivative, (**4**), as was confirmed by the spectral characteristics of the reaction product obtained. The IR spectrum of (**4**) showed additional absorption bands of an amide carbonyl (1697 $cm^{-1}$) and also the band of an ester carbonyl at 1741 $cm^{-1}$. The mass spectrum of (**4**) showed the peak of the molecular ion with *m/z* 290, and the peaks of ions with *m/z* 247 and 204 formed the the successive elimination of two acetyl groups. In the PMR spectrum of (**4**) we observed two three-proton singlets at 1.97 ppm ($O-CO-CH_3$) and 2.14 ppm ($N-CO-CH_3$).

On condensation with acetone in concentrated sulfuric acid, alkaloid (**2**) gave an acetonide in the form of a white amorphous substance (**5**), the mass spectrum of which showed the peak of the molecular ion with *m/z* 246, and the peaks of ions with *m/z* 204 and 174.

The alkaline saponification of (**1**) gave a base with mp 178-180°C the spectral characteristics of which were close to those of (**2**). A direct comparison (mixed melting point, IR spectrum) demonstrated their identity. The above chemical conversions showed that bases (**1**) and (**2**) were structurally close. The reverse passage from (**2**) to (**1**) was also achieved. Thus, the condensation of (**2**) with acetaldehyde produced a substance with mp 218-220°C identical with alkaloid (**1**) (TLC, mixed melting point, IR spectrum).

Two bases — donaxarine and donaxaridine — have been isolated previously from the plant *A. donax* gathered in the Shaartuzskii region of Tadzhikistan [4]. Structures for these alkaloids were proposed on the basis of spectral characteristics and chemical conversions. A comparison of the spectral characteristics of donaxarine and donaxaridine and their $R_f$ values in TLC, melting points, and the results of the chemical transformations with the corresponding properties of bases (**1**) and (**2**) indicated that we were dealing with the above-mentioned alkaloids.

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Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, Tashkent, fax (3712) 89 14 75. Translated from Khimiya Prirodnykh Soedinenii, No. 5, pp. 720-727, September-October, 1995. Original article submitted March 22, 1995.

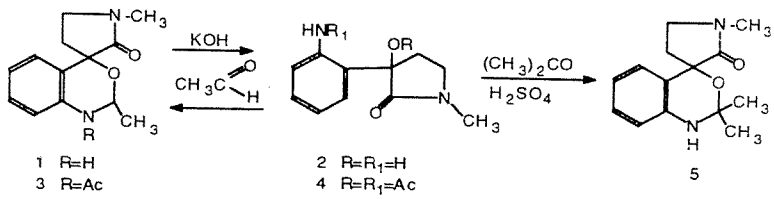
 0009-3130/95/3105-0604$12.50 

TABLE 1. Bond Lengths $r$ (Å) and Valence Angles $\omega$ in (**1**) and (**2**)

| Bond | $r_1$ | Angle | $\omega_1$ | Bond | $r_2$ | Angle | $\omega_2$ |
|---|---|---|---|---|---|---|---|
| O1-C2 | 1.244(4) | C3-O3′-C2 | 112.8(3) | O1-C2 | 1.235(4) | C2-N1-C5 | 113.5(2) |
| O3′-C3 | 1.251(3) | C2-N1-C5 | 114.0(4) | O2-C3 | 1.423(3) | C2-N10C6 | 125.1(3) |
| O3′C2′ | 1.425(5) | C2-N1-C6 | 124.0(3) | N1-C2 | 1.325(4) | C5-N10C6 | 121.3(3) |
| N1-C2 | 1.327(7) | C5-N1-C6 | 121.7(5) | N1-C5 | 1.454(4) | O1-C2-N1 | 125.5(3) |
| N1-C5 | 1.455(5) | C9′-N1-C2′ | 113.4(4) | N1-C6 | 1.429(4) | O1-C2-C3 | 124.9(3) |
| N1-C6 | 1.449(6) | O1-C2-N1 | 125.8(4) | N2-C2′ | 1.384(4) | N1-C2-C3 | 109.7(2) |
| N1-C9′ | 1.415(4) | O1-C2-C3 | 125.3(5) | C2-C3 | 1.534(4) | O2-C3-C2 | 109.2(2) |
| N1-C2′ | 1.453(6) | N1-C2-C3 | 108.9(3) | C3-C4 | 1.541(4) | O2-C3-C4 | 109.4(2) |
| C2-C3 | 1.548(5) | O3-C3-C2 | 107.1(2) | C3-C1 | 1.515(4) | C2-C3-C4 | 101.5(2) |
| C3-C4 | 1.529(6) | O3′-C3-C4 | 106.0(3) | C4-C5 | 1.516(4) | O2-C3-C1 | 111.7(2) |
| C3-C10 | 1.496(7) | C2-C3-C4 | 102.1(4) | C1′-C6′ | 1.384(4) | C2-C30C1 | 111.4(2) |
| C4-C5 | 1.516(7) | O3′-C3-C10′ | 111.0(4) | C1′-C2′ | 1.419(4) | C4-C3-C1 | 113.1(3) |
| C10′-C5′ | 1.412(5) | C2-C3-C10′ | 114.1(3) | C6′-C5′ | 1.376(4) | C3-C4-C5 | 105.0(2) |
| C10′-C9′ | 1.395(7) | C4-C3-C10′ | 115.6(3) | C5′-C4′ | 1.382(5) | N1-C5-C4 | 104.1(3) |
| C5′-C6′ | 1.379(9) | C3-C4-C5 | 105.6(3) | C4′-C3′ | 1.375(4) | C3-C1′-C6′ | 122.9(2) |
| C6′-C7′ | 1.389(9) | N1-C5-C4 | 103.4(4) | C3′-C2′ | 1.374(5) | C3-C1′-C2′ | 119.5(2) |
| C7′-C8′ | 1.373(5) | C3-C10′-C5′ | 121.4(4) | | | C6′-C1′-C2′ | 117.5(3) |
| C8′-C9′ | 1.394(9) | C3-C10′-C9′ | 121.0(3) | | | C1′-C6′-C5′ | 122.8(3) |
| C2′-C11′ | 1.519(5) | C5′C10′-C9′ | 117.6(5) | | | C6′-C5′-C4′ | 118.7(3) |
| | | C10-′C5′-C6′ | 121.5(5) | | | C5′-C4′-C3′ | 120.1(3) |
| | | C5′-C6-′C7′ | 119.5(4) | | | C4′-C3′-C2′ | 121.6(3) |
| | | C6′-C7′-C8′ | 120.2(6) | | | N2-C2′-C1′ | 121.1(3) |
| | | C7-′C8′-C9′ | 120.5(6) | | | N2-C2′-C3′ | 119.5(3) |
| | | N1′-C9′-C10′ | 120.0(5) | | | C1′-C2′-C3′ | 119.3(3) |
| | | N1-C9′-C8′ | 119.4(5) | | | | |
| | | C10′-C9′-C8′ | 120.6(3) | | | | |
| | | O3′-C2′-N1 | 108.6(3) | | | | |
| | | O3′-C2′-C11 | 108.5(4) | | | | |
| | | N1′-C2′-C11 | 109.9(4) | | | | |

Yunusov et al. [4], basing themselves on the results of the potassium permanganate oxidation of donaxaridine, giving isatine, assigned it, and, consequently, donaxarine as well, to the oxindole type of bases [5]. We repeated this reaction, but found no isatine. Apparently, the reaction product from the oxidation of donaxaridine was not quite correctly characterized. Doubt has also been thrown on the conclusion that an acetyl derivative of donaxarine was obtained under the conditions described in [4]. The formation of an acetate from the structure proposed for donaxarine is unlikely.

All the above facts necessitated a reconsideration of the structures of donaxarine and donaxaridine. The structures of the alkaloids have now been established definitively by x-ray structural analysis.




1 R=H
3 R=Ac

2 $R=R_1=H$
4 $R=R_1=Ac$

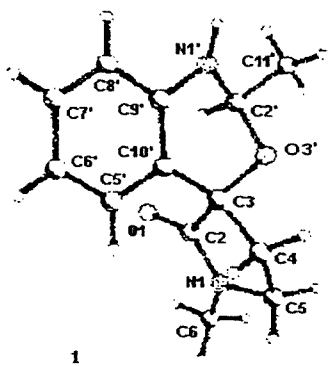
5

The forms of the donaxarine and donaxaridine molecules established by the x-ray structural method and given in Fig. 1 show that these alkaloids do not belong to the hydroxyindole group but are spiro[(N-methylpyrrolidin-2-one)-3,4′-(2′-methyl-3′,1′-benzoxazine)] and 3-hydroxy-3-(2′-aminophenyl)-N-methyl-2-pyrrolidone, respectively. According to their space groups (see the Experimental section) in each case the crystals contain a mixture of asymmetric molecules and, consequently, $[\alpha]_D = 0°$.

Donaxarine (**1**) is a tricyclic molecule (the product of the condensation of (**2**) with acetaldehyde). In the (**1**) molecule the benzene (planar) and pyrrolidone (4$\beta$-envelope) rings retain their conformations. The six-membered oxazine heterocycle adopts the O3′$\alpha$,C2′$\beta$-half-chair conformation through the condensation of its aromatic nucleus. Analysis of the crystal structure of base (**1**) shows that the molecules are present at the equilibrium distances of Van der Waals interactions.

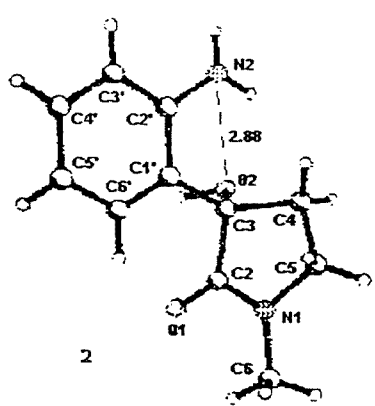
Donaxaridine (**2**) — the product of the saponification of (**1**) — is a bicyclic molecule consisting of 2-aminophenyl and substituted pyrrolidone rings linked through C1′−C3. The mutual positions of the planar 2-aminophenyl and 4$\beta$-envelope pyrrolidone rings favor the formation of an intramolecular H-bond between the unshared pairs of the O2 atom and the H atom of the $NH_2$ group, as is shown by the O2···N2 and O2···H−N distances of 2.88 and 2.34 Å, respectively, and the O2−H−N angle of 118°. In the crystals of (**2**), hydrogen bonds of the O···H−O type are formed: O1···O2 and O1···H−O2 distances 2.77 and 2.10 Å, respectively, and the O2−H···O1 angle 118°.

Donaxarine




1

Donaxaridine




2

The geometric parameters of the molecules (bond lengths and valence angles) in structures (**1**) and (**2**) are given in Table 1. They agree well with the corresponding standard values [6]. A slight lengthening of the C=O bond to 1.244(4) Å in both molecules shows the existenceof conjugation between the electron system of the carbonyl group and the unshared pair

TABLE 2. Chemical Shifts of the $^{13}C$ Atoms of Base (2) (in $CDCl_3+CD_3OD$, ppm, 0 — TMS)

| CH | | $CH_2$ | | $CH_3$ | | Quat. carbons | |
|---|---|---|---|---|---|---|---|
| carbon atom | ppm | carbon atom | ppm | carbon atom | ppm | carbon atom | ppm |
| C-6′ | 125.7[a] | | | | | C-2 | 175.5 |
| C-5′ | 117.7[b] | C-4 | 32.9 | N-$CH_3$ | 29.87 | C-3 | 79.5 |
| C-4′ | 128.9[a] | C-5 | 45.6 | | | C-1′ | 125.1 |
| C-3′ | 117.9[b] | | | | | C-2′ | 145.8 |

a,b) Alternative assignments of the signals are possible

TABLE 3. Coordinates of the Atoms ($\times 10^4$) and Temperature Factors ($Å^2 \times 10^3$) in (1) and (2)

| Atom | Compound 1 | | | | Atom | Compound 2 | | | |
|---|---|---|---|---|---|---|---|---|---|
| | $x$ | $y$ | $z$ | $U_{eq}$ | | $x$ | $y$ | $z$ | $U_{eq}$ |
| O1 | 2427(3) | 3754(2) | 9472(2) | 51 | O1 | 6428(2) | 1361(3) | 5654(1) | 66 |
| O3′ | 1977(3) | 6631(2) | 8681(2) | 49 | O2 | 3215(2) | 2279(3) | 4872(1) | 57 |
| N1 | 4006(5) | 4421(2) | 8334(2) | 45 | N1 | 6043(3) | 4140(4) | 6232(1) | 62 |
| N1′ | -295(4) | 6614(2) | 8913(2) | 46 | N2 | 129(3) | 2199(4) | 5403(1) | 67 |
| C2 | 2799(6) | 4439(2) | 8681(2) | 41 | C2 | 5533(3) | 2628(4) | 5832(2) | 51 |
| C3 | 1932(5) | 5474(3) | 7958(2) | 40 | C3 | 3653(3) | 2691(4) | 5620(2) | 48 |
| C4 | 2764(5) | 5774(3) | 6964(3) | 48 | C4 | 3292(4) | 4809(4) | 5753(2) | 62 |
| C5 | 4186(6) | 5377(4) | 7432(3) | 57 | C5 | 4733(4) | 5481(4) | 6305(2) | 66 |
| C10′ | 516(5) | 5063(3) | 7570(2) | 38 | C1′ | 2823(3) | 1337(4) | 6075(1) | 44 |
| C5′ | 209(6) | 4069(4) | 6728(3) | 50 | C6′ | 3681(3) | 314(4) | 6647(2) | 50 |
| C6′ | -1096(7) | 3698(4) | 6361(3) | 57 | C5′ | 2919(4) | -875(4) | 7072(2) | 58 |
| C7′ | -2138(8) | 4316(4) | 6821(3) | 60 | C4′ | 1228(4) | -1084(5) | 6915(2) | 61 |
| C8′ | -1867(7) | 5275(4) | 7651(3) | 54 | C3′ | 335(40) | -74(5) | 6356(2) | 59 |
| C9′ | -548(6) | 5635(3) | 8045(3) | 41 | C2′ | 1085(3) | 1137(4) | 5935(2) | 49 |
| C6 | 5106(6) | 3627(4) | 8875(4) | 57 | C6 | 7709(4) | 4501(6) | 6557(2) | 88 |
| C2′ | 1062(5) | 6587(3) | 9533(3) | 44 | | | | | |
| C11′ | 1301(6) | 7763(4) | 10331(3) | 59 | | | | | |

of the N1 atom. The errors in the determination of the bond lengths and valence angles are of the order of 0.007 Å and 0.4° for (1) and 0.004 Å and 0.3° for (2).

Since bases (1) and (2) have proved to be alkaloids with a new structure, we obtained the $^{13}C$ NMR spectral characteristics of (2) (Table 2). The $^{13}C$ NMR spectrum contains the signals of 11 carbon atoms. With respect to the magnitudes of the chemical shifts and in the light of the nature of substitution, the signals in the spectrum can be grouped in the following way: in the aromatic part of the spectrum, two signals of quaternary carbons and four signals of tertiary carbons are observed. In the nonaromatic part of the spectrum there are two signals of quaternary carbons, two signals of secondary carbons, and one signal of a primary carbon (Table 2).

## EXPERIMENTAL

**General Observations.** Melting points were determined on a Boetius stage, and specific optical rotations on a Jasco J-20 spectropolarimeter. UV spectra were taken on a Perkin-Elmer $L_{16}$ Fourier UV spectrometer, using ethanol solutions in a quartz cell with a layer thickness of 1 cm; IR spectra on a Perkin-Elmer Fourier spectrometer (model 2000) using tablets with KBr; mass spectra on a Kratos MS25F spectrometer; and NMR spectra on a Tesla BS 567 A/100 instrument. The purity of the alkaloids was checked with the aid of TLC on plates with Chemapol type KSK, LS 5/40, silica gel and alumina (Brockmann activity grade II). Revealing agents: the Dragendorff reagents and iodine vapor. Column chromatography was conducted on sorbents (silica gel and alumina) with a particle size of 100-125 μm.

**Isolation of the Total Alkaloids.** The air-dry comminuted epigeal part of the plant (50 kg), previously moistened with 8% ammonia solution, was extracted with chloroform exhaustively (6 extractions). Treatment of the combined concen-

trated chloroform extract gave 50 g of an ether fraction and 100 g of a chloroform fraction of the total alkaloids. On treatment with acetone, the 50 g of ether fraction yielded 25 g of donaxine with mp 133-134°C (acetone). Chromatography on a column of alumina (1:30) was applied to 25 g of material from the donaxine mother solution. The alkaloids were eluted with ether and chloroform. Ether fractions gave N-phenyl-β-naphthylamine, arundine, deoxyvasicinone, and donaxine. Ether–chloroform fractions yielded donaxarine, with $R_f$ 0.85 (benzene–methanol (9:1)), and the chloroform fraction donaxaridine with $R_f$ 0.75 (chloroform–methanol (9:1)) and 0.6 (benzene–methanol (9:1)).

**Donaxarine (1),** $C_{13}H_{16}N_2O_2$, mp 218-220°C, $[\alpha]_D$ 0°.

UV spectrum (EtOH, $\nu_{max}$, nm): 292, 250, 206.

IR spectrum (KBr, $cm^{-1}$): 3266, 2881, 1687, 1603, 1497, 981, 746.

Mass spectrum (EI, 70 eV), *m/z* (%): 232 ($M^+$100), 217 $(M-15)^+$, 189 $(M-43)^+$, 174 $(M-58)^+$, 160 $(M-72)^+$, 146, 130, 118, 58, 44.

PMR spectrum ($CDCl_3$, ppm, 100 MHz): 1.29 (d, 3H, $CH_3$), 2.41 (t, 2H, $CH_2$), 2.90 (s, 3H, $NCH_3$), 3.45 (m, 2H, $CH_2$), 4.66 (q, H, CH), 6.70-7.16 (m, 4H, Ar-H), 8.12 (br.s, H, NH).

**Donaxaridine (2),** $C_{11}H_{14}N_2O$, mp 178-180°C, $[\alpha]_D$ 0°.

UV spectrum (EtOH, $\nu_{max}$, nm): 292, 242.

IR spectrum (KBr, $cm^{-1}$): 3452, 3362, 1674, 1622, 1578, 1452, 1270, 1100, 860, 753.

Mass spectrum (EI, 70 eV), *m/z* (%): 206 ($M^+$), 188 $(M-18)^+$, 146, 135, 130, 120 (100), 92, 77, 58, 44.

PMR spectrum ($CDCl_3$, ppm, 100 MHz): 2.47 (m, 2H, $CH_2$), 2.87 (s, 3H, N-$CH_3$), 3.17 (m, 2H, $CH_2$), 4.60 (br.s, 2H), 6.52-7.16 (m, 4H, Ar-H).

**Acetyldonaxarine (3).** A mixture of 0.01 g of donaxarine, 0.5 ml of acetic anhydride, and 0.3 ml of pyridine was kept at room temperature until the solid had passed into the solvent completely (10 days). The reaction mixture was evaporated under vacuum, the residue was dissolved in water, and the solution was made alkaline with ammonia and extracted with chloroform. Elimination of the chloroform left amorphous N-acetyldonaxarine (**3**), TLC, $R_f$ 0.95 (benzene–methanol (9:1)). Mass spectrum (EI, 70 eV), *m/z* (%): 274 ($M^+$, 30), 231, 217, 189 (100), 146, 118.

**Saponification of Donaxarine.** A mixture of 0.04 g of donaxarine and 5 ml of 20% methanolic caustic potash was heated for 5 h. The residue after evaporation of the solvent was diluted with 5 ml of water and extracted with chloroform. The chloroform residue was treated with 4 ml of 10% sulfuric acid. The acid solution was made alkaline with ammonia and extracted with chloroform. The residue (0.02 g) was crystallized from acetone, mp 178-180°C, TLC, $R_f$ 0.60 (benzene–methanol (9:1)). A mixture with donaxaridine gave no depression of the melting point.

**O,N-Diacetyldonaxaridine (4).** A mixture of 0.05 g of donaxaridine, 2 ml of acetic anhydride, and 0.5 ml of pyridine was heated in the water bath for 3 h. Then the reaction mixture was left at room temperature for two days. The solvent was eliminated under vacuum, and the residue was chromatographed on a column of alumina. From benzene eluates we isolated the oily O,N-diacetyldonaxaridine (**4**), TLC, $R_f$ 0.95 (benzene–methanol (9:1)).

IR spectrum (KBr, $cm^{-1}$): 3265, 1741, 1697, 1610, 1540, 1232, 1092, 964, 756.

Mass spectrum (EI, 70 eV), *m/z* (%): 290 ($M^+$60), 247 (20), 188 (100), 146, 120.

PMR spectrum ($CDCl_3$, ppm, 100 MHz): 1.97 (s, 3H, $CH_3$), 2.14 (s, 3H, N-$CH_3$).

**Donaxaridine Acetonide (5).** One drop of conc. sulfuric acid was added to a solution of 0.5 g of donaxaridine in 25 ml of acetone, the mixture was shaken, and, after 50 min, it was neutralized with ammonia. Then it was left for 12 h at room temperature, the acetone was driven off, and the residue was chromatographed on a column of alumina; benzene eluates yielded the product of the condensation of donaxaridine with acetone (**5**), TLC, $R_f$ 0.70 (benzene–methanol (9:1)).

Mass spectrum (EI, 70 eV), *m/z*, (%): 246 ($M^+$, 10), 204, 174 (100).

**Condensation of Donaxaridine with Acetaldehyde.** A mixture of 0.03 g of donaxaridine, 4 ml of methanol, and 20 ml of acetaldehyde was heated in the water bath for 3 h, and then the solvent was evaporated off and the residue was treated with acetone. This led to the formation of crystals of donaxarine with mp 218-220°C (acetone), $R_f$ 0.85 (benzene–methanol (9:1)) (mixed melting point, IR spectrum).

**X-Ray Structural Analysis.** The crystals were first investigated by the photo method. The space group and the parameters of the unit cell were determined from precession x-ray diagrams and were refined on a Syntex $P2_1$ diffractometer.

The main crystallographic characteristics of the structures investigated are given below:

| 1 | 2 |
|---|---|
| $a$ = 10.064(2) Å | $a$ = 8.236(3) Å |
| $b$ = 10.339(2) Å | $b$ 6.998(2) Å |
| $c$ = 11.410(3) Å | $c$ = 18.815(5) Å |
| $\beta$ = 97.64(2)° | $\beta$ = 99.56(2)° |
| $d_{calc}$ = 1.16 g/cm$^3$ | $d_{calc}$ = 1.28 g/cm$^3$ |
| 950, $I > 2\sigma(I)$ | 1166, $I > 2\sigma(I)$ |
| R = 0.042 | R = 0.052 |
| space gr.$P2_1/n$ | space gr. $P2_1/n$ |
| $Z$ = 4 | $Z$ = 4 |

A three-dimensional set of intensities was obtained on the same diffractometer by the $\theta/2\theta$ method of scanning, using $CuK_\alpha$ radiation (graphite monochromator), $\sin\theta/\lambda < 0.56$. The structure search was conducted by the SHELXS-86 program [7] (PC DOS version), where we succeeded in finding a model of the molecule in the automatic regime. Subsequent Fourier syntheses permitted the localization of all the nonhydrogen atoms. The structures were refined by the method of least squares (MLS) in the full-matrix anisotropic approximation by the SHELX-76 program [8]. The coordinates of the H atoms were calculated geometrically and were refined isotropically. The coordinates of the nonhydrogen atoms of structures (**1**) and (**2**) from the last stage of the MLS are given in Table 3. All the calculations were performed on a personal computer of the IBM PC AT type.

## REFERENCES


1. V. U. Khuzhaev, S. F. Aripova, and R. Sh. Shakirov, Khim. Prir. Soedin., 685 (1994).
2. V. U. Khuzhaev, S. F. Aripova, and R. Sh. Shakirov, Khim. Prir. Soedin., 687 (1994).
3. V. U. Khuzhaev, S. F. Aripova, and R. Sh. Shakirov, Khim. Prir. Soedin., 328 (1994).
4. K. A. Ubaidullaev, R. Sh. Shakirov, and S. Yu. Yunusov, Khim. Prir. Soedin., 553 (1976).
5. A. Chatterjee and S. Bose, J. Indian Chem. Soc., **31**, 17 (1954).
6. F. N. Allen, O. Kennard, and D. G. Watson, J. Chem. Soc., Perkin Trans. II, 1 (1987).
7. G. M. Sheldrick, Acta Cryst, **A46**, 467 (1990).
8. G. M. Sheldrick, SHELX-76 Program for Crystal Structure Determination, Cambridge, U.K.