Thermal Rearrangement of Some 3-Alkyl-2-aryltetrahydro-1,3-oxazine *N*-Oxides. Synthesis of 2-Alkyl-7-aryltetrahydro-1,6,2-dioxazepines [1]

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Several 2-alkyl-7-aryltetrahydro-1,6,2-dioxazepines **5a-i** have been synthesized by a Meisenheimer-type rearrangement of 3-alkyl-2-aryltetrahydro-1,3-oxazine *N*-oxides **4a-i**. These dioxazepines have not been previously described and for the first time compounds with the tetrahydro-1,6,2-dioxazepine ring-system have been made available. The structure assigned to members of this novel heterocycle is based on elemental analysis, infrared, and ^{1}H nmr spectral evidence. Further definitive evidence for the structure of these heterocycles is provided by an X-ray crystallographic analysis of **5c**.

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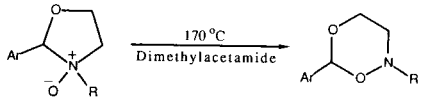
Saba and co-workers [2] have recently observed that when 3-alkyloxazolidine *N*-oxides bearing an aryl group at the 2-position (**1**) are heated to about 170° in dimethylacetamide, 2-alkyl-6-aryl-3,4-dihydro-2*H*-1,5,2-dioxazines **2** are obtained in moderate to good yields. The incorporation of the *N*-oxide function into the ring has precedent in pyrrolidines [3,4] and piperidines [5] and is analogous to rearrangements of acyclic tertiary amine *N*-oxides bearing an allyl or benzyl group to *O*-allyl or *O*-benzylhydroxylamines originally discovered by Meisenheimer [6].

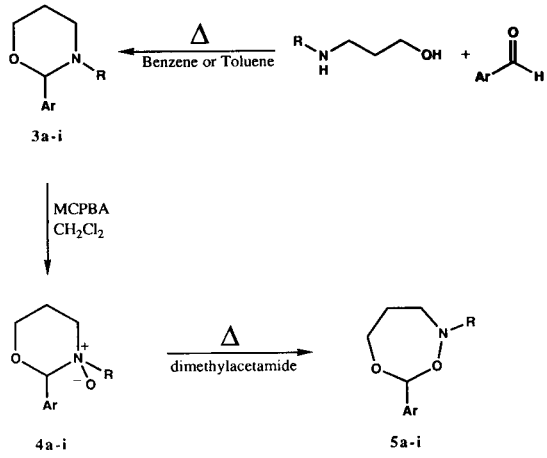


1 2

The rearrangement of appropriately substituted *N*-oxides to a ring-expanded product is of considerable mechanistic interest and synthetic utility. It was envisioned that the application of this reaction to substituted tetrahydro-1,3-oxazine *N*-oxides **4** could lead to tetrahydro-1,6,-2-dioxazepies **5**. We now wish to describe here a complementary procedure for the synthesis of a series of 2-alkyl-7-aryltetrahydro-1,6,2-dioxazepines **5a-i** as members of heterocycles containing a novel ring system.

The starting materials for the synthesis are 3-alkyl-2-aryltetrahydro-1,3-oxazines **3**. These compounds were easily prepared by heating equimolar mixtures of 3-alkylaminopropanols and aromatic aldehydes in benzene or toluene solution in a Dean Stark apparatus with azeotropic removal of the water formed [7] (Scheme). The 3-alkylaminopropanols were prepared according to a literature procedure by reduction with lithium aluminum hydride of the Michael adducts from the reaction of primary amines with ethyl acrylate [8]. 3-Ethyl and 3-isopropylaminopropanols were also prepared by adaptation of the method of Saavedra [9] by reductive alkylation of 3-aminopropanol with acetaldehyde and acetone using sodium borohydride which produced excellent yields of these products. The 3-alkyl-2-aryltetrahydro-1,3-oxazine *N*-oxides **4a-i** were made by oxidation of **3a-i** with 3-chloroperoxybenzoic acid (MCPBA) using a general procedure for oxidation of tertiary amines [10]. The crude *N*-oxides were generally used without further purification in the subsequent rearrangement step. Attempts to make 3-*tert*-butyl-2-aryltetrahydro-1,3-oxazines failed presumably due to steric problems.

Scheme



a. R = CH_3, Ar = C_6H_5
b. R = CH_3, Ar = 4-ClC_6H_4
c. R = CH_3, Ar = 4-$NO_2C_6H_4$
d. R = CH_3CH_2, Ar = C_6H_5
e. R = CH_3CH_2, Ar = 4-ClC_6H_4
f. R = CH_3CH_2, Ar = 4-$NO_2C_6H_4$
g. R = $(CH_3)_2CH$, Ar = C_6H_5
h. R = $(CH_3)_2CH$, Ar = 4-ClC_6H_4
i. R = $(CH_3)_2CH$, Ar = 4-$NO_2C_6H_4$

The pyrolysis of the oxazine *N*-oxides was conducted in dimethylacetamide at 170° for about 50 minutes. Removal of dimethylacetamide by vacuum distillation and column chromatography of the dark residue on basic alumina using anhydrous ether as the eluent afforded the dioxazepines. The dioxazepines eluted very quickly and are stable

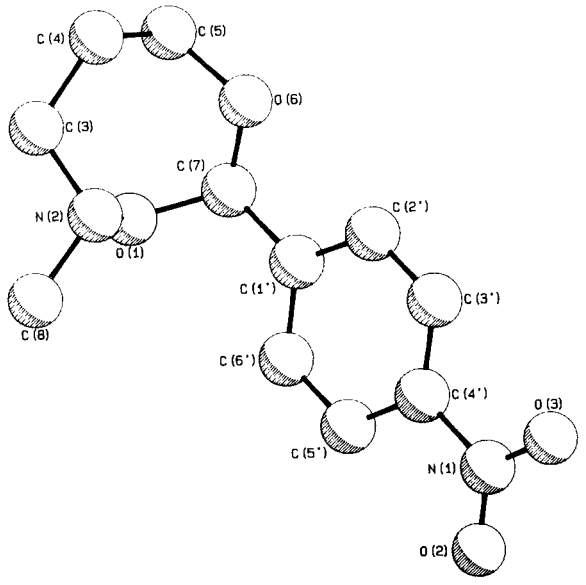


Figure 1. Numbering of atoms and conformation of the molecule.

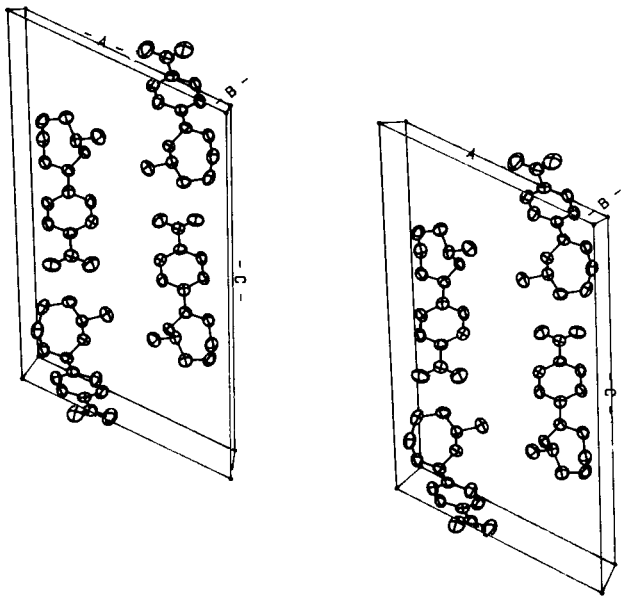


Figure 2. Stereoscopic view of the unit cell.

on silica gel tlc plates. The dioxazepines appeared as slightly uv active spots on tlc and were stainable with iodine (R_f values are about 0.85 using ethyl acetate-petroleum ether 1:1 as the solvent). The dioxazepines with a 4-nitro group on the aromatic ring were obtained in higher yields (about 75%), whereas the others were obtained in 45-65% yields. With the exception of **5c** which crystallized on standing, all the others were obtained as pale yellow oils.

The analytical and spectral data (ir and ^{1}H nmr) strongly support the proposed structures **5a-i**. The ir spectra show a band at 923-926 cm^{-1} for the N-O bond [3,4,11]. The most diagnostic absorption in the ^{1}H nmr of the dioxazepines is the presence of a sharp singlet near δ 6 for the benzylic hydrogen flanked by two oxygens. The same hydrogen appears as a singlet at about δ 5 in **3a-i**. This significant downfield shift of the signal is in accord with the proposed structures for **5a-i**. Definitive proof for the structure of the dioxazepines came from a single X-ray crystal analysis of **5c** as described in the next section.

X-Ray Diffraction Analysis of 2-Methyl-7-(4-nitrophenyl)-tetrahydro-1,6,2-dioxazepine (**5c**).

The following data were obtained: $C_{11}N_2O_4H_{14}$, Mr = 238.25, monoclinic crystals, $P2_1/c$, a = 12.908(3), b = 4.948(1), c = 20.513(4)Å, β = 116.30(2)°, V = 1174.4(3)Å^3, Z = 4, D_c = 1.35 g-cm^{-3}, F(000) = 504, μ = 8.84 cm^{-1}, transparent colorless plates, 0.05, 0.15, 0.20 mm, θ_{max} = 60°. Unit cell parameters, orientation matrix, and intensity data collection were carried out on an Enraf-Nonius CAD4 diffractometer equipped with PDP-11 digital computer, CuKα radiation (λ = 1.5418Å) and a graphite monochromator. Cell parameters were measured using 25 reflections in the 2θ range 20-40°. Three intensity and orientation control reflections were recorded after every 200 reflections, no significant change in their intensity was observed during data collection. 807 reflections were considered as "observed" with the criterion I >

Table 1. Final fractional coordinates and equivalent isotropic temperature factors for non-H atoms with e.s.d.'s in parantheses.

$$B_{eq} = 4/3\Sigma_i\Sigma_j\beta_{ij}a_ia_j$$

	x	y	z	B_{eq}(Å^2)
O(1)	0.3061(3)	-0.4839(7)	0.6956(2)	3.06(5)
O(2)	0.3728(4)	0.308(2)	0.4378(3)	6.7(1)
O(3)	0.1933(5)	0.385(2)	0.3898(3)	6.8(1)
O(6)	0.1102(3)	-0.504(1)	0.6133(2)	3.96(7)
N(1)	0.2790(4)	0.268(1)	0.4334(2)	4.4(1)
N(2)	0.2834(3)	-0.2367(9)	0.7275(2)	3.11(7)
C(1')	0.2356(4)	-0.303(1)	0.5726(2)	3.28(9)
C(2')	0.1414(4)	-0.171(1)	0.5196(3)	3.5(1)
C(3')	0.1549(4)	0.016(1)	0.4746(3)	3.7(1)
C(3)	0.2326(5)	-0.345(1)	0.7730(3)	4.3(1)
C(4)	0.1093(4)	-0.437(1)	0.7292(3)	4.5(1)
C(4')	0.2629(4)	0.071(1)	0.4813(2)	3.34(8)
C(5)	0.0940(5)	-0.634(1)	0.6707(3)	4.4(1)
C(5')	0.3590(4)	-0.058(2)	0.5329(3)	5.0(1)
C(6')	0.3443(4)	-0.244(1)	0.5782(3)	4.2(1)
C(7)	0.2225(4)	-0.510(1)	0.6223(2)	3.18(8)
C(8)	0.3975(6)	-0.124(2)	0.7718(4)	4.9(1)

Table 2. Bond lengths (Å), angles (°), and selected torsion angles (°); (e.s.d.'s in parentheses).

O(1)	N(2)	1.477(6)	C(1')	C(2')	1.383(6)
O(1)	C(7)	1.415(4)	C(1')	C(6')	1.388(8)
O(2)	N(1)	1.190(8)	C(1')	C(7)	1.507(7)
O(3)	N(1)	1.217(7)	C(2')	C(3')	1.372(9)
O(6)	C(5)	1.438(9)	C(3')	C(4')	1.366(8)
O(6)	C(7)	1.378(6)	C(3)	C(4)	1.510(7)
N(1)	C(4')	1.463(8)	C(4)	C(5)	1.49(1)
N(2)	C(3)	1.458(8)	C(4')	C(5')	1.379(7)
N(2)	C(8)	1.455(7)	C(5')	C(6')	1.38(1)

N(2)	O(1)	C(7)	109.8(3)	C(2')	C(3')	C(4')	119.7(4)
C(5)	O(6)	C(7)	114.0(4)	N(2)	C(3)	C(4)	112.5(4)
O(2)	N(1)	O(3)	122.6(7)	C(3)	C(4)	C(5)	114.0(5)
O(2)	N(1)	C(4')	120.2(5)	N(1)	C(4')	C(3')	120.5(4)
O(3)	N(1)	C(4')	117.2(6)	N(1)	C(4')	C(5')	118.5(5)
O(1)	N(2)	C(3)	102.4(4)	C(3')	C(4')	C(5')	121.1(6)
O(1)	N(2)	C(8)	104.3(4)	O(6)	C(5)	C(4)	110.5(6)
C(3)	N(2)	C(8)	111.0(4)	C(4')	C(5')	C(6')	118.7(5)
C(2')	C(1')	C(6')	118.1(5)	C(1')	C(6')	C(5')	121.4(4)
C(2')	C(1')	C(7)	121.9(5)	O(1)	C(7)	O(6)	113.8(4)
C(6')	C(1')	C(7)	120.0(4)	O(1)	C(7)	C(1')	113.3(4)
C(1')	C(2')	C(3')	121.5(5)	O(6)	C(7)	C(1')	108.7(4)

C(7)	O(1)	N(2)	C(3)	104.2(4)	C(4)	C(5)	O(6)	C(7)	90.5(6)
O(1)	N(2)	C(3)	C(4)	-75.1(6)	C(5)	O(6)	C(7)	O(1)	-36.3(7)
N(2)	C(3)	C(4)	C(5)	54.2(7)	O(6)	C(7)	O(1)	N(2)	-54.6(5)
C(3)	C(4)	C(5)	O(6)	-70.4(7)					

3.0σ(I). All reflections were corrected for Lorentz and polarization effects, but no corrections for absorption were made.

The SDP package [12] was used for all the calculations. The structure was solved using MULTAN [13]. The usual sequence of isotropic and anisotropic refinements were followed, after which all H atoms were located on a difference electron density map. In the last cycle the H atoms were fixed at idealized position with fixed Debye-Waller temperature parameters at 6.0 Å^2. Convergence was reached with R = 0.068 and R_w = 0.059. The weighting scheme [14] of the type $w = 1/[\sigma F^2 + (0.02F)^2 + 3]$ was applied with Dunitz-Seiler [15] modified weights $w' = w \exp[20(\sin\theta/\lambda)^2]$. Scattering factors for neutral atoms [16] were used.

Atomic parameters are given in Table 1; the bond distances, bond angles and relevant torsion angles are presented in Table 2. Atomic numbering is given in Figure 1 and the stereoscopic view of the unit cell is shown in Figure 2. The bond distances and angles are normal with the exception of O(6) - C(7), 1.378 Å (Table 2), considerably shorter than 1.421 Å found in 2-ethyl-6-(4-nitrophenyl)-tetrahydro-2*H*-1,5,2-dioxazine [17], which may impose some constraint on the ring. The minimum deviation from the mean plane of this ring is 0.017(5) Å for C(7) and the maximum is −0.521(3) Å for O(1); O(6) deviates from the plane by 0.436(5) Å. The planar phenyl ring is in the equatorial position. The dihedral angle between the plane of the phenyl and the heterocyclic ring is 114.9(1)°.

EXPERIMENTAL

Melting points were determined on a mel-temp capillary melting point apparatus and are uncorrected. The ir spectra were obtained with a Perkin Elmer 710B spectrophotometer. The ^{1}H nmr spectra were recorded at 100 MHz on a Varian XL 100 spectrometer, using tetramethylsilane as internal standard and deuteriochloroform as the solvent. Chemical shifts are reported in ppm (δ) and J values are in Hz. Elemental analyses were performed by Quantitative Technologies Inc., Bound Brook, New Jersey. For column chromatography, basic alumina (Aldrich grade) was used. Thin layer chromatographic analyses were performed on precoated silica gel plastic sheets (Eastman Chromatogram Sheets), 0.1 mm thickness with fluorescent indicator. All chemicals and anhydrous solvents were purchased from Aldrich.

General Procedure for the Synthesis of 3-Alkyl-2-aryltetrahydro-1,3-oxazines **3a-i**.

An equimolar mixture of an aromatic aldehyde and 3-alkylaminopropanol was heated under reflux in benzene or toluene with azeotropic removal of the water formed using a Dean-Stark apparatus. The reflux was continued until the theoretical quantity of water was collected. The solvent was then evaporated on the rotary evaporator and the residue was fractionally distilled or crystallized as appropriate. Tetrahydro-1,3-oxazines **3a**, **c**, **f** and **g** are known compounds.

3-Methyl-2-(4-chlorophenyl)tetrahydro-1,3-oxazine (**3b**).

A solution of 3-methylaminopropanol 1.78 g, (0.02 mole) and 4-chlorobenzaldehyde 2.81 g, (0.02 mole) in benzene (50 ml) was heated as described above gave 4.06 g (96%), bp 112-113°/0.04 torr; ^{1}H nmr: δ 2.02 (s, 3H, CH_3), 1.50-4.24 (complex m, 6H, OCH_2, NCH_2, CH_2), 4.64 (s, 1H, OCHN), 7.44 (q, 4H, J = 8, ArH).

Anal. Calcd. for $C_{11}H_{14}ClNO$: C, 62.41; H, 6.67; N, 6.62. Found: C, 62.51; H, 6.69; N, 6.64.

3-Ethyl-2-phenyltetrahydro-1,3-oxazine (**3d**).

A solution of 3-ethylaminopropanol 2.06 g (0.02 mole) and benzaldehyde 2.12 g (0.02 mole) in benzene (50 ml) was heated as described above gave 3.63 g (95%), bp 67°/0.08 torr; ^{1}H nmr: δ 0.92 (t, 3H, CH_3-ethyl), 1.49-4.24 (complex m, 8H, OCH_2, NCH_2, CH_2, NCH_2-ethyl), 4.92 (s, 1H, OCHN), 7.46 (m, 5H, ArH).

Anal. Calcd. for $C_{12}H_{17}NO$: C, 75.35; H, 8.96; N, 7.32. Found: C, 75.19; H, 9.08; N, 7.34.

3-Ethyl-2-(4-chlorophenyl)tetrahydro-1,3-oxazine (**3e**).

A solution of 3-ethylaminopropanol 2.06 g (0.02 mole) and 4-chlorobenzaldehyde 2.81 g (0.02 mole) in benzene (50 ml) was heated as described above gave 4.33 g (96%), bp 93-94°/0.04 torr; ^{1}H nmr: δ 0.90 (t, 3H, CH_3-ethyl), 1.42-4.20 (complex m, 8H, OCH_2, NCH_2, CH_2, NCH_2-ethyl), 4.92 (s, 1H, OCHN), 7.44 (q, 4H, J = 8, ArH).

Anal. Calcd. for $C_{12}H_{16}NOCl$: C, 63.85; H, 7.15; N, 6.21. Found: C, 63.95; H, 7.19; N, 6.16.

3-Isopropyl-2-(4-chlorophenyl)tetrahydro-1,3-oxazine (**3h**).

A solution of 3-isopropylaminopropanol 2.34 g (0.02 mole) and 4-chlorobenzaldehyde 2.81 g (0.02 mole) in toluene (50 ml) was heated as described above gave 4.41 g (92%), bp 93-95°/0.03 torr. ^{1}H nmr: δ 0.90, 1.30 (d, 6H, J = 7, CH_3-isopropyl), 1.71-4.14 (complex m, 7H, OCH_2, NCH_2, CH_2, CH-isopropyl), 4.98 (s, 1H, OCHN), 7.48 (q, 4H, J = 8, ArH).

Anal. Calcd. for $C_{13}H_{18}NOCl$: C, 65.13; H, 7.57; N, 5.84. Found: C, 64.80; H, 7.49; N, 5.81.

3-Isopropyl-2-(4-nitrophenyl)tetrahydro-1,3-oxazine (**3i**).

A solution of 3-isopropylaminopropanol 2.34 g (0.02 mole) and 4-nitrobenzaldehyde 3.02 g (0.02 mole) in toluene (50 ml) was

heated as described above gave 4.65 g (93%), mp 69-71° (ligroin) ^{1}H nmr: δ 0.94, 1.07 (d, 6H, J = 7, CH_3-isopropyl), 1.76-4.10 (complex m, 7H, OCH_2, NCH_2, CH_2, CH-isopropyl), 5.16 (s, 1H, OCHN), 7.79 (d, 2H, J = 8, ArH), 8.33 (d, 2H, J = 8, ArH).

Anal. Calcd. for $C_{13}H_{18}N_2O_3$: C, 62.38; H, 7.25; N, 11.20. Found: C, 62.33; H, 7.17; N, 11.11.

General Procedure for the Synthesis of 2-Alkyl-7-aryltetrahydro-1,6,2-dioxazepines **5a-i**.

To a stirred ice bath cooled solution of the 3-alkyl-2-aryltetrahydro-1,3-oxazines **3** (10 mmoles) in anhydrous methylene chloride (10 ml) under a nitrogen atmosphere, a solution of MCPBA (Aldrich 80-85%) 2.14 g, (10 mmoles) in anhydrous methylene chloride (90 ml) was added dropwise. The mixture was stirred for a total of 3 hours during which time the ice bath was allowed to warm up to room temperature. The solution was then passed through a dry column containing basic alumina (about 20 times the total weight of starting materials). Anhydrous chloroform (100 ml) was then passed to remove trace amounts of unreacted **3**, followed by methanol/chloroform (1:3; v/v; 300 ml) to elute the *N*-oxide **4**. Evaporation of the solvents on the rotary evaporator afforded the crude *N*-oxide as a viscous residue in about 75-85% yield. The *N*-oxide was then dissolved in anhydrous dimethylacetamide (20 ml) and the solution was heated in an oil bath at 170° for 50 minutes. The solution was then cooled to room temperature and dimethylacetamide was removed under reduced pressure at bath temperature of 40°. The dark brown residue was then chromatographed on basic alumina. Elution with anhydrous ether followed by evaporation of the solvent yielded the dioxazepines **5**. With the exception of **5c**, the dioxazepines were pale yellow oils.

2-Methyl-7-phenyltetrahydro-1,6,2-dioxazepine (**5a**).

This compound was obtained in a yield of 48%, mp of picrate 68-70°; ^{1}H nmr: δ 2.00 (m, 2H, CH_2), 2.60 (s, 3H, CH_3), 2.94 (t, J = 6, 2H, NCH_2), 4.06 (t, J = 5, 2H, OCH_2), 5.88 (s, 1H, OCHO), 7.50 (m, 5H, ArH).

Anal. Calcd. for $C_{17}H_{18}N_4O_9$: C, 48.34; H, 4.30; N, 13.27. Found: C, 48.24; H, 4.21; N, 13.24.

2-Methyl-7-(4-chlorophenyl)tetrahydro-1,6,2-dioxazepine (**5b**).

This compound was obtained in a yield of 55%, mp of picrate, 105-107°; ^{1}H nmr: δ 2.00 (m, 2H, CH_2), 2.68 (s, 3H, CH_3), 2.92 (t, J = 6, 2H, NCH_2), 4.06 (t, J = 5, 2H, OCH_2), 5.86 (s, 1H, OCHO), 7.50 (q, J = 8, 4H, ArH).

Anal. Calcd. for $C_{17}H_{17}ClN_4O_9$: C, 44.70; H, 3.76; N, 12.27. Found: C, 44.77; H, 3.62; N, 12.31.

2-Methyl-7-(4-nitrophenyl)tetrahydro-1,6,2-dioxazepine (**5c**).

This compound was obtained in a yield of 75% mp of picrate, 123.5-124.5°; ^{1}H nmr: δ 2.00 (m, 2H, CH_2), 2.68 (s, 3H, CH_3), 2.94 (t, J = 5, 2H, NCH_2), 4.10 (t, J = 5, 2H, OCH_2), 5.92 (s, 1H, OCHO), 7.74, 8.32 (d, J = 8, 4H, ArH).

Anal. Calcd. for $C_{17}H_{17}N_5O_{11}$: C, 43.69; H, 3.67; N, 14.99. Found: C, 43.81; H, 3.44; N, 15.18.

2-Ethyl-7-phenyltetrahydro-1,6,2-dioxazepine (**5d**).

This compound was obtained in a yield of 45%, mp of picrate, 70-71°; ^{1}H nmr: δ 1.12 (t, J = 6, 3H, CH_3), 1.98 (m, 2H, CH_2), 2.86 (m, 4H, NCH_2, NH_2-ethyl), 4.07 (t, J = 6, 2H, OCH_2), 5.88 (s, 1H, OCHO), 7.5 (m, 5H, ArH).

Anal. Calcd. for $C_{18}H_{20}N_4O_9$: C, 49.54; H, 4.63; N, 12.84. Found: C, 49.50; H, 4.63; N, 12.79.

2-Ethyl-7-(4-chlorophenyl)tetrahydro-1,6,2-dioxazepine (**5e**).

This compound was obtained in a yield of 60%, mp of picrate, 114-115°; ^{1}H nmr: δ 1.10 (t, J = 6, 3H, CH_3), 2.00 (m, 2H, CH_2), 2.86 (m, 4H, NCH_2, NCH_2-ethyl), 4.06 (t, J = 6, 2H, OCH_2), 5.86 (s, 1H, OCHO), 7.48 (q, J = 8, 4H, ArH).

Anal. Calcd. for $C_{18}H_{19}ClN_4O_9$: C, 45.92; H, 4.08; N, 11.90. Found: C, 45.75; H, 3.86; N, 11.84.

2-Ethyl-7-(4-nitrophenyl)tetrahydro-1,6,2-dioxazepine (**5f**).

This compound was obtained in a yield of 70%, mp of picrate, 124.5-125°; ^{1}H nmr: δ 1.04 (t, J = 6, 3H, CH_3), 2.00 (m, 2H, CH_2), 2.90 (m, 4H, NCH_2, NCH_2-ethyl), 4.08 (t, J = 6, 2H, OCH_2), 5.92 (s, 1H, OCHO), 7.78, 8.32 (d, J = 8, 4H, ArH).

Anal. Calcd. for $C_{18}H_{19}N_5O_{11}$: C, 44.91; H, 3.99; N, 14.55. Found: C, 45.00; H, 3.69; N, 14.49.

2-Isopropyl-7-phenyltetrahydro-1,6,2-dioxazepine (**5g**).

This compound was obtained in a yield of 50%, mp of picrate 69-71°; ^{1}H nmr: δ 1.10 (d, 6H, J = 6, CH_3-isopropyl), 1.98 (m, 2H, CH_2), 2.96 (m, 3H, CH-isopropyl, NCH_2), 4.04 (m, 2H, OCH_2), 5.86 (s, 1H, OCHO), 7.49 (m, 5H, ArH).

Anal. Calcd. for $C_{19}H_{22}N_4O_9$: C, 50.66; H, 4.93; N, 12.44. Found: C, 50.58; H, 4.90; N, 12.37.

2-Isopropyl-7-(4-chlorophenyl)tetrahydro-1,6,2-dioxazepine (**5h**).

This compound was obtained in a yield of 62%, mp of picrate 103-104°; ^{1}H nmr: δ 1.10 (d, 6H, J = 6, CH_3-isopropyl), 1.96 (m, 2H, CH_2), 2.96 (m, 3H, CH-isopropyl, NCH_2), 4.04 (m, 2H, OCH_2), 5.86 (s, 1H, OCHO), 7.50 (q, J = 8, 4H, ArH).

Anal. Calcd. for $C_{19}H_{21}ClN_4O_9$: C, 47.06; H, 4.37; N, 11.56. Found: C, 47.02; H, 4.29; N, 11.50.

2-Isopropyl-7-(4-nitrophenyl)tetrahydro-1,6,2-dioxazepine (**5i**).

This compound was obtained in a yield of 72%, mp of picrate 121-121.5°; ^{1}H nmr: δ 1.10 (d, J = 6, 6H, $2CH_3$), 2.00 (m, 2H, CH_2), 3.02 (m, 3H, CH-isopropyl, NCH_2), 4.05 (m, 2H, OCH_2), 5.90 (s, 1H, OCHO), 7.78, 8.34 (d, J = 8, 4H, ArH).

Anal. Calcd. for $C_{19}H_{21}N_5O_{11}$: C, 46.06; H, 4.28; N, 14.14. Found: C, 46.23; H, 3.98; N, 14.14.

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