SYNTHESIS OF STABLE OXAZOLIDINE NITROXYL RADICALS WITH METHOXY GROUPS AT THE α-CARBON ATOMS TO THE RADICAL SITE

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The condensation of α-hydroxyaminoalcohols with aldehydes and acetone gave 3-hydroxyoxazolidines and tautomeric N-(2-hydroxyethyl)-α-arylnitrones, whose oxidation by PbO_2 in methanol leads to stable oxazolidine nitroxyl radicals with methoxy groups at C^2 and C^4.

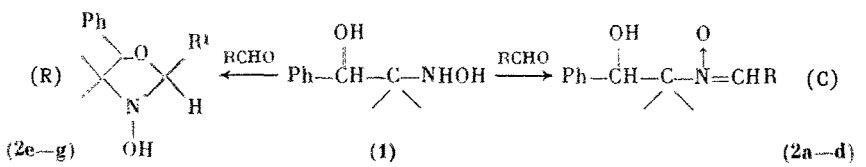
Keywords: *α-hydroxyaminoalcohols, 3-hydroxyoxazolidines, stable nitroxyl radicals, α-arylnitrones, lead dioxide, oxazolidines.*

In previous work [1-3], we showed that α,α-dialkoxy-substituted stable nitroxyl radicals (SNR) derived from imidazolidine, pyrrolidine, and imidazoline are formed upon the oxidation of aldonitrone or methoxynitrone derivatives of imidazoline, pyrroline, and 2H-imidazole by PbO_2 in methanol. We have examined the possibility of using this method for obtaining new oxazolidine nitroxyl radicals (NR) starting from 3-hydroxyoxazolidines containing a hydrogen atom at C^2 or C^4. We should note that the oxazolidine heterocyclic system is the basis for many SNR ("doxyls") [4, 5].

3-Hydroxyoxazolidines (**2**) were obtained as in the work of Kligel and Becker [6] by the condensation of α-hydroxyaminoalcohols (HAA) with aldehydes. HAA (**1**), in turn, was obtained by the reduction of 2-methyl-2-hydroxyamino-3-phenyl-3-propanone by $NaBH_4$.

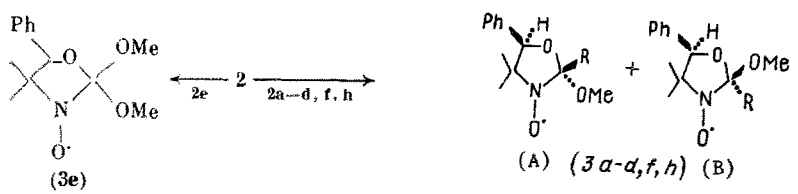
Ring—chain tautomerism is possible for the products of the condensation of α-hydroxyaminoalcohols with aldehydes [6]. According to the spectral data (bands at ~1570-1580 cm^{-1} in the IR spectrum (Table 1) and at ~300 nm in the UV spectra (Table 2) for the arylnitrone group and aldonitrone proton PMR signal at 7.5-8.0 ppm (Table 3)), the products of the condensation of α-hydroxyaminoalcohol **1** with aromatic aldehydes **2a-2d** exist in the open (chain) form, N-(1,1-dimethyl-2-hydroxy-2-phenylethyl)-α-arylnitrones (**C**).

The lack of such signals for the products of the condensation with formaldehyde **2e**, acetaldehyde **2f**, and glyoxylic acid **2g** and finding of a PMR signal for the proton at C^2 in the ring at 5 ppm indicate the existence of these compounds in the ring 4,4-dimethyl-5-phenyl-3-hydroxyoxazolidine form (**R**). Furthermore, the signals for the gem-methyl groups appear at higher field for the ring form (Table 3). Ester **2h** obtained by treating acid **2g** with diazomethane has an analogous structure.



R = Ph (a), 3-NO_2-C_6H_4 (b), 2-Py (c), 4-Py (d), H (e), CH_3 (f), COOH (g).

We might have assumed that, as in the oxidation of 3-imidazoline 3-oxides [7], the position of the ring—chain tautomeric equilibrium for **2** would not affect their capacity to undergo conversion to NR. Indeed, the oxidation of both ring 3-hydroxyoxazolidines **2e**, **2f**, and **2h** and acyclic α-hydroxyarylnitrones **2a-2d** leads to NR **3a-3d**, **3e**, **3f**, and **3h**.



(3e)

(A) (3a-d,f,h) (B)

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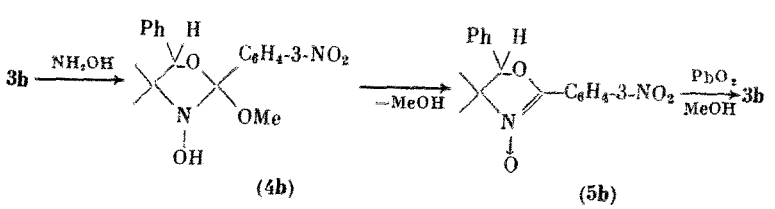
TABLE 1. Elemental Analysis Data, Melting Points, Yields, and IR Spectra (in KBr Pellets[a] and in CCl_4[b]) of Compounds Synthesized*

Compound	Yield, %	Mp, °C	Found/Calculated, %			Chemical formula	IR spectrum, ν, cm^{-1}
			C	H	N		
1	82	136–138	65.7	8.2	7.3	$C_{10}H_{15}NO_2$	3260, 3395 (NHOH)[a]
			66.3	8.3	7.7		
2a	60	126–128	76.1	7.3	5.3	$C_{17}H_{19}NO_2$	3240, 1100 (OH), 1570 (C=N) [a]
			75.8	7.0	5.2		
2b	84	170–171	64.6	5.8	8.7	$C_{17}H_{18}N_2O_4$	3290, 1100 (OH), 1570 (C=N), 1340, 1530 (NO_2) [a]
			65.0	5.7	8.9		
2c	60	147–149	71.0	6.8	10.0	$C_{16}H_{18}N_2O_2$	3280, 1105 (OH), 1570 (C=N) [a]
			71.1	6.7	10.4		
2d	40	168–170	69.9	6.8	10.0	$C_{16}H_{18}N_2O_2$	3280, 1105 (OH), 1580 (C=N) [a]
			71.1	6.7	10.0		
2e	80	87–89	67.9	7.8	6.7	$C_{11}H_{15}NO_2$	3610, 1110 (OH)[b]
			68.4	7.8	7.2		
2f	100	85.5–87	69.3	8.3	6.9	$C_{12}H_{17}NO_2$	3600, 1110 (OH)[b]
			69.6	8.2	6.7		
2g	70	124–126	60.8	6.7	5.6	$C_{12}H_{15}NO_2$	3480 (OH), 1680 (C=O) [a]
			60.8	6.3	5.9		
2h	100	95–98	62.3	7.3	5.7	$C_{13}H_{17}NO_4$	3590, 1100 (OH), 1750 (C=O) [b]
			62.2	6.8	5.6		
3aA	30	Oil	72.0	6.3	4.4	$C_{18}H_{20}NO_3$	2860 (OMe), 1190 [b]
			72.5	6.7	4.7		
3aB	35	Oil	72.9	6.3	4.4	$C_{18}H_{20}NO_3$	2860 (OMe) [b]
			72.5	6.7	4.7		
3bA	30	89–90	63.0	5.8	8.0	$C_{18}H_{19}N_2O_5$	2840 (OMe), 1530, 1350 (NO_2), 1190 [a]
			63.0	5.5	8.2		
3bB	55	88–90	63.3	5.9	7.8	$C_{18}H_{19}N_2O_5$	2840 (OMe), 1530, 1350 (NO_2) [a]
			63.0	5.5	8.2		
3cA	32	Oil	67.9	6.7	9.5	$C_{17}H_{19}N_2O_2$	2840 (OMe), 1190[b]
			68.2	6.4	9.4		
3d	60	106–108	68.0	6.3	8.9	$C_{17}H_{19}N_2O_3$	2840 (OMe), 1190[b]
			68.2	6.3	9.4		
3e	60	31–33	62.7	7.6	5.6	$C_{13}H_{18}NO_4$	2840 (OMe) [b]
			61.9	7.1	5.6		
3h	96	118–120	59.7	6.7	5.1	$C_{14}H_{19}NO_5$	2840 (OMe), 1750 (C=O) [b]
			60.0	6.4	5.0		
7	82	167–168	74.3	5.8	6.2	$C_{15}H_{15}NO_2$	1600 (C=N) [a]
			74.7	5.8	6.2		
8	90	85–90	73.2	7.0	5.7	$C_{15}H_{17}NO_2$	3350, 3460 (OH) [a]
			74.0	7.0	5.7		
10	100	100–101	68.7	8.0	7.1	$C_{11}H_{15}NO_2$	3600, 1100 (OH)[b]
			68.4	7.8	7.2		
11	60	30–31	61.4	7.4	5.3	$C_{13}H_{18}NO_4$	2850 (OMe) [b]
			61.9	7.1	5.6		

*3cB is unstable as a pure compound. IR spectrum: 2850 (OMe)[b].



We note that C^2 in stable NR **3e** is surrounded by three alkoxy groups. Diastereomers **A** and **B** were isolated as pure compounds upon chromatography of NR **3a-3d**, **3f**, and **3h** on silica gel. Diastereomers **A** in each pair of NR had the higher R_f value. The formation of a pair of unstable diastereomers of **3f** was observed using thin-layer chromatography and ESR

TABLE 2. UV Data for **2a-2d** and **7**

Compound	λ, nm	lg ε	Compound	λ, nm	lg ε
2a	298	4.25	**2c**	308	4.19
2b	278	4.17	**2d**	305	4.03
	300	4.19	**7**	298	4.39

TABLE 3. PMR Spectral Data for Diamagnetic Compounds, δ, ppm (*J*, Hz)

Compound	Substituent					
	R	H[a]	gem Me_2	PhCH	Ph	OH
1[b]	–	–	0.93 s	4.8s	7.33 m	
2a[c]	7.45 m	7.26–7.62	1.65 s	4.88d	7.26 s	5.83 d
	8.20 m		1.47 s	(5)		(5)
2b[c]	9.53 s					
	8.60 d (8)	7.93 s	1.57 s	5.2 d (5)	7.37 s	5.83 d
	8.26 d (8)		1.33 s			(5)
	7.7 m					
2c[d]	9.23 d (9)					
	8.73 d (6)	7.83 s	1.63 s	5.03 s	7.37 s	5.8 s
	7.6–8.0 m					
	7.18–7.6 m					
2d[d]	8.60 d (6)	7.37s	1.63 s	5.07 s	7.37 s	–
	7.98 d (6)					
2e[d]	4.9		1.42 s	4.98 s	7.33 s	–
			0.75 s			
2f[d]	1.52 d	5.4 d	1.28 s	4.7 s	7.33 s	–
	(6.7)	(6.7)	1.51 s			
2g[c]	–	5.23 s	1.20 s	4.95 s	7.33 s	–
			0.67 s			
2h[d]	3.87s	5.37 s	1.35 s	5.12 s	7.31 s	–
			0.78 s			
7[c]	8.3 m	7.7 s	4.13 m[e]	5.8 m	7.31 s	5.8
	7.31m					
8[c]	7.36s	3.86 s[f]	2.86 d[g] (7)	4.9 m	7.33 s	4.9 s[h]
						8.0
10[d]	5.7 d (5.8)[i]		2.03 s	4.03 t	7.33 s	6.53
			1.88 s	(5.8)		

[a]The signal for the proton at C^2 of the heterocycle in **2e-2h** and the signal for the aldol proton in **2a-2d**.

[b]In $(CD_3)_2CO$.

[c]In DMSO.

[d]In $CDCl_3$.

[e]Signal for the protons of the CH_2 group in the α position to the nitrone fragment.

[f]Signal for the protons of the N—CH_2—Ph group.

[g]Signal for the protons of the CH—CH_2—N group.

[h]Signal for the hydroxyamino group.

[i]Signal for the protons at C^4 in the heterocycle.

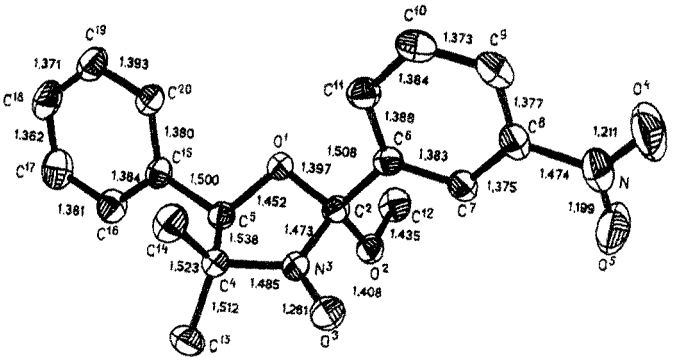


Fig. 1. Structure of nitroxyl radical of 2S-(3-nitrophenyl)-2-methoxy-4,4-dimethyl-(5_s)-phenyloxazolidine 3-oxide (**3Ab**). Thermal oscillation ellipsoids (30%) are shown. The error in the bond lengths was 0.003-0.006 Å.

TABLE 4. ESR Spectral Data (a_N, G) for Oxazolidine Radicals in $CHCl_3$

Compound	A	B	Compound	A	B
3a	12.97	13.52	**3e**		13.31
3b	12.78	13.12	**3f**		13.5
3c	12.97	13.11	**3h**	12.61	
3d		13.13	**11**		12.96
			12	11.91	12.26

spectroscopy. The formation of trace amounts of NR detected only by ESR spectroscopy occurred upon the oxidation of **2** in ethanol and 2-propanol by analogy to our previous work [2, 3].

Comparison of the pairs of NR diastereomers **3A** and **3B** showed a number of general features:

1) The a_N coupling constant for the **3A** diastereomer is less than the corresponding value for the **3B** diastereomer.

2) The IR spectra of NR **3A** has a strong band at 1190 cm^{-1}, which is lacking in the IR spectra of NR **3B**.

3) In each NR diastereomer pair, **3A** proved more stable upon storage.

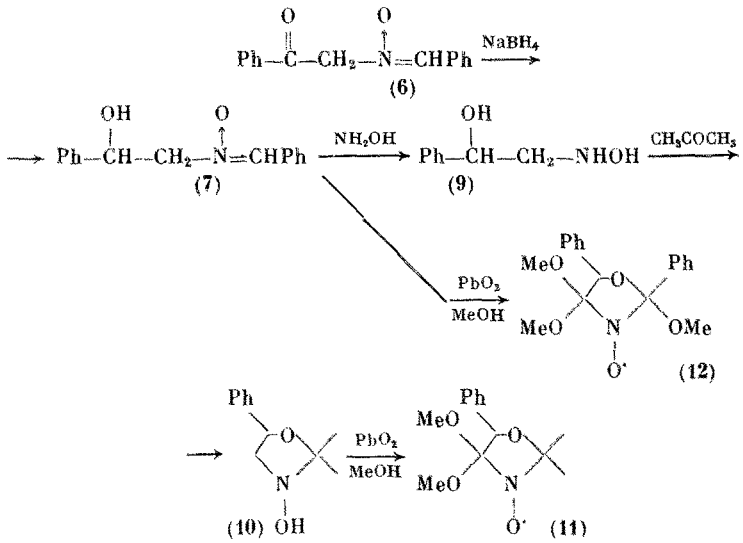
X-ray diffraction structural analysis showed that the phenyl substituent at C^5 and 3-nitrophenyl substituent at C^2 in NR **3Ab** are located *cis* relative to the $C^2O^1C^5$ plane (Fig. 1). In accord with these results and the general features indicated above, we assigned a structure for group **A** analogous to that shown in Fig. 1, while the structure for group **B** has the phenyl group at C^5 and methoxy group at C^2 in *cis* orientation.

The reaction of both **3Ab** and **3Bb** with hydroxylamine does not stop at the formation of the corresponding isomeric reduction products, namely, 3-hydroxy derivatives **4b**, but rather leads in both cases to the same product, oxazoline 3-oxide **5b**, whose subsequent oxidation in methanol gives a mixture of starting NR **3b**.

Thus, oxazolidine NR are formed upon oxidation of the products of the condensation of 2-methyl-2-hydroxyamino-3-phenylpropanol **1** with aldehydes having both cyclic **2R** and acyclic **2C** structure. These NR include stable radicals with one or two methoxy groups at C^2. We should also note that there are no significant limitations to the nature of substituent R such as those found for the oxidation of tetrasubstituted 3-imidazoline 3-oxides [2] and 2H-imidazole N-oxides [3]. This difference is attributed to the circumstance that the oxidation of **2** proceeds through the intermediate formation of oxazoline 3-oxides **5** with an intracyclic alkoxynitrone group in the heterocycle, which, as shown previously, significantly facilitates the oxidative methoxylation to give α-methoxy derivatives [2].

In accord with previous findings [1, 8], the introduction of methoxy groups, as in the case of the introduction of other electron-withdrawing substituents with an $-I$ effect at the α-carbon to the radical site, leads to a decrease in the coupling constants (Table 4). Thus, while the a_N value for 2,4,4-trimethyl-2-heptyloxazolidine 3-oxide is 14.4 G [9], these values for **3f**, **3e**, and **3h** are 13.5, 13.31, and 12.6 G, respectively.

In order to obtain stable oxazolidine NR with methoxy groups at the carbon atom at the other side of the radical site **11** and **12**, we attempted to oxidize 4-unsubstituted 3-hydroxyoxazolidines or their open tautomers, which were synthesized according to the scheme below. We should note that further reduction to N-(2-hydroxy-2-phenylethyl)-N-hydroxy-N-benzylamine (**8**) is possible upon the reduction of N-(1-oxo-1-phenylethyl)-α-phenylnitrone (**6**).



Hydroxylamino-2-phenylethanol **9** formed upon treating nitrone **7** with hydroxylamine was described by Vanderbiet and Hasst [10] as the oxalate and hydrochloride derivatives. In light of the instability of hydroxylamino-2-phenylethanol **9** as a pure compound, we used it immediately upon isolation in the condensation with acetone.

NR **11** was formed upon the oxidation of ring product **10** and isolated as a pure compound, while the formation of unstable **12A** and **12B** upon the oxidation of acyclic nitrone **7** was detected only using ESR spectroscopy.

EXPERIMENTAL

The IR spectra were taken on a UR-20 spectrometer for KBr pellets (c = 0.25%) or in CCl_4 solution (c = 1%). The UV spectra were taken on a Specord UV-VIS spectrometer in ethanol. The PMR spectra were taken on a Varian A56-60A spectrometer for 5-10% solutions; internal standard HMDS. The ESR spectra were taken in chloroform on a Bruker ESP 300 spectrometer. The x-ray diffraction structural analysis was carried out on a Syntex P2 spectrometer. The thin-layer chromatography was carried out on Silufol UV 254 plates using 25:1 chloroform—ethanol as the eluent. The IR spectral data, melting points, yields, and elemental analysis results for the compounds synthesized are given in Table 1. The UV spectral for conjugated arylnitrones **2a-2d** are given in Table 2. The synthesis of N-(1-oxo-1-phenylethyl)-α-phenylnitrone **6** was given in our previous work [11].

X-Ray Diffraction Structural Analysis of 2S-(3-Nitrophenyl)-2-methoxy-4,4-dimethyl-5S-phenyloxazolidine 3-Oxide (3Ab). The unit cell data for the monoclinic crystals are as follows: a = 7.2071(8), b = 29.736(2), c = 8.3846(7) Å, β = 104.768(8)°, V = 1737.6 Å^3, space group $P2_1/a$, $C_{18}N_{19}N_2O_5$, Z = 4, d_{calc} = 1.31 g/cm^3, λCuK_α. The intensities of 2367 independent reflections with $2\theta < 116°$ were measured by $2\theta/\omega$ scanning. A total of 1540 observed reflections were used in the calculations. The structure was solved by the direct method and refined by the method of least squares anisotropically for the nonhydrogen atoms and isotropically for the hydrogen atoms in the block diagonal approximation to R = 0.039 and R_w = 0.045, $w^{-1} = \sigma_f^2 + 0.00017F^2$.

The oxazolidine ring is nonplanar and exists as a half-chair with a C_2 axis traversing N^3 and the midpoint of the O^1—O^5 bond. The phenyl, nitrophenyl, and nitroxyl groups are planar. The bond lengths in the nitroxyl group are very similar to the values for this group in 2,2,5,5-tetramethyl-4-phenyl-3-imidazoline-1-oxyl 3-oxide [12]. The orientation of the phenyl groups is characterized by O^1—C^5—C^{15}—C^{20} torsion angles equal to 28.5 and −22.6°, respectively.

2-Hydroxyamino-2-methyl-3-phenylpropanol (1). A solution of 1.8 g (49 mmoles) $NaBH_4$ in 100 ml ethanol was added in portions to a solution of 17.5 g (98 mmoles) 2-hydroxyamino-2-methyl-3-phenyl-3-propanone [11] in 150 ml ethanol until the starting ketone was consumed as indicated by thin-layer chromatography. The reaction mixture was maintained for 1 h. The solution was filtered and the solvent was distilled off. The residue was dissolved in 300 ml ethyl acetate. This solution was washed with two 10-ml portions of saturated aqueous sodium chloride and dried over $MgSO_4$. The solvent was evaporated and the residue was triturated with 5 ml ether. Product **1** precipitated and was filtered off and recrystallized from acetonitrile.

Condensation of 2-Hydroxyamino-2-methyl-3-phenylpropanol (1) with Aldehydes. A sample of 5 mmoles aldehyde was added to a solution of 5 mmoles hydroxyaminoalcohol **1** in 100 ml ethanol. (In the case of formaldehyde, we used 1 ml 37% aqueous solution of this compound per g hydroxyaminoalcohol. In the preparation of 3-hydroxy-2,4,4-trimethyl-5-phenyloxazolidine **2f**, the reaction mixture was saturated with gaseous acetaldehyde). After the complete consumption of starting **1** as indicated

by thin-layer chromatography, the solvent was evaporated. If the residue did not crystallize spontaneously, it was triturated with acetone in the case of the products of condensation with aromatic aldehydes **2c** and **2d** and with hexane in the case of the products of condensation with aliphatic aldehydes **2e** and **2f**. The products were recrystallized from 1:1 hexane—ethyl acetate, N-(1,1-dimethyl-2-hydroxy-2-phenylethyl)-α-3-nitrophenylnitrone (**2b**) was recrystallized from ethanol.

3-Hydroxy-4,4-dimethyl-2-methoxycarbonyl-5-phenyloxazolidine (2h). An ethereal solution of diazomethane was added with stirring to 5 g (21 mmoles) 3-hydroxy-4,4,-dimethyl-2-carboxy-5-phenyloxazolidine (**2g**) until this reagent fully entered solution. The reaction mixture was left for 30 min and ether was distilled off. The residue was recrystallized from 1:1 hexane—ethyl acetate.

N-(2-Hydroxy-2-phenylethyl)-α-phenylnitrone (7). A solution of 0.5 g (14 mmoles) $NaBH_4$ in 20 ml ethanol was added in portions to a solution of 3 g (13 mmoles) N-(2-oxo-2-phenylethyl))-α-phenylnitrone (**6**) in 50 ml ethanol until all starting ketonitrone (**6**) was consumed as indicated by thin-layer chromatography. Ethanol was distilled off and the residue was dissolved in 100 ml ethyl acetate. This solution was washed with two 5-ml portions of water and then dried over $MgSO_4$. The solvent was evaporated and the solid residue was recrystallized from ether.

N-(2-Hydroxy-2-phenylethyl)phenylmethylhydroxylamine (8). A solution of 1 g (28 mmoles) $NaBH_4$ in 20 ml ethanol was added in 0.5-ml portions to a solution of 3 g (13 mmoles) N-(1-oxo-1-phenylethyl)-α-phenylnitrone (**6**) in 50 ml ethanol until both starting ketonitrone **6** and N-(2-hydroxy-2-phenylethyl)-α-phenylnitrone **7** disappeared. Ethanol was distilled off and the residue was dissolved in chloroform. The solution was dried over $MgSO_4$ and filtered. The solvent was distilled off and the solid residue was recrystallized from ether.

3-Hydroxy-2,2-dimethyl-5-phenyloxazolidine (10). A sample of 7 g nitrone **7** was added with stirring to a solution of NH_2OH obtained from a solution of 7 g (100 mmoles) hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) in methanol and $NaOCH_3$ in 100 ml methanol. After complete consumption of starting nitrone **7** as indicated by thin-layer chromatography, the solvent was distilled off and the residue was dissolved in chloroform. The solution was dried over $MgSO_4$ and filtered. The solvent was distilled off and the residue was triturated with 1:1 hexane—ether. The precipitate formed was washed with ether to give 0.7 g (5 mmoles) 2-hydroxylamine-1-phenylethanol (**9**), which was dissolved in 50 ml acetone and left for 30 min. The solvent was distilled off. The residue was triturated with hexane and recrystallized from 1:1 hexane—ethyl acetate.

Preparation of Stable Oxazolidine Nitroxyl Radicals (3a-3d, 3e, 3f, 3h, 11, and 12). All the oxazolidine NR were obtained by oxidation of starting 3-hydroxyoxazolidines **2e**, **2f**, **2h**, **10**, or α-arylnitrones **3a-3d**, or **7** by the action of PbO_2 in methanol (using 1 g of the starting reagent and 5 g PbO_2 in 100 methanol). After complete consumption of the starting reagent as indicated by thin-layer chromatography, the oxidizing agent was filtered off and the solvent was distilled off. The residue was subjected to chromatography on a silica gel column using chloroform as the eluent. Diastereomeric doxyls **3a-3c** were separated by chromatography on a silica gel column using 1:1 hexane—ether as the eluent. NR **3b**, **3d**, **3e**, **3h**, and **12** were recrystallized upon trituration with hexane and recrystallization from 1:1 hexane—ethyl acetate. 2,2-Dimethyl-4,4-dimethoxy-5-phenyloxazolidine 3-oxide (**11**) was recrystallized from hexane.

Reduction of NR 3b by Hydroxylamine. A solution of NH_2OH in methanol obtained from a methanolic solution of 1 g (14 mmoles) $NH_2OH \cdot HCl$ and $NaOCH_3$ was added dropwise to a solution of 0.1 g (0.3 mmole) **3b** in 10 ml methanol. After starting reagent **3b** was completely consumed as indicated by thin-layer chromatography, the solvent was distilled off and the residue was dissolved in ethyl acetate. The solution was dried over $MgSO_4$ and filtered. Ethyl acetate was evaporated and the residue of **5b** was dried in vacuo. IR spectrum in CCl_4 (ν, cm^{-1}): 1560 (C=N), 1530, 1350 (NO_2). PMR spectrum in $CDCl_3$ (δ, ppm, J, Hz): 9.3 s, 9.0 d (J 8), 8.32 d (J 8), 7.2-7.8 m (3-NO_2—C_6H_4), 7.33 s (Ph), 5.63 s (H), 1.8 s, 1.33 s (2Me).

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