

## Amine oxides

### XIV.\* Preparation, infrared spectra, and antimicrobial activity of tris(*N*-oxides) of bis(2-dimethylaminoethyl)alkylamines

\*F. DEVÍNSKY, \*F. BITTEREROVÁ, \*I. LACKO, and <sup>b</sup>D. MLYNARČÍK

<sup>a</sup>*Department of Inorganic and Organic Chemistry, Faculty of Pharmacy, Komenský University, CS-832 32 Bratislava*

<sup>b</sup>*Department of Biochemistry and Microbiology, Faculty of Pharmacy, Komenský University, CS-832 32 Bratislava*

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Preparation and infrared spectra of some bis(2-aminoethyl)alkylamines, bis(2-dimethylaminoethyl)alkylamines, and tris(*N*-oxides) of bis(2-dimethylaminoethyl)alkylamines have been described and the effect of structural changes on their antimicrobial activity against *Staphylococcus aureus*, *Escherichia coli*, and *Candida albicans* has been followed.

Описаны получение и инфракрасные спектры некоторых бис(2-аминоэтил)алкиламинов, бис(2-диметиламиноэтил)алкиламинов и трис(*N*-оксида) бис(2-диметиламиноэтил)алкиламинов. Рассмотрено также влияние структурных изменений на их антимикробную активность по отношению к *Staphylococcus aureus*, *Escherichia coli* и *Candida albicans*.

In the framework of study of the effect of structural changes in amine oxides on some of their physicochemical properties and biological activity, we investigated both monoamine oxides [1—10] and bis(*N*-oxides) [11—14]. These compounds exhibited besides very good surface-active properties also noticeable antimicrobial activities, in many cases higher than those of disinfectants belonging to organic ammonium salts.

In the present work we paid attention to another type of nonaromatic amine oxides, namely tris(*N*-oxides) which have not been dealt with in the literature so far. Only *Blaschke et al.* [15] described the preparation of tris(*N*-oxides) of some ethoxylated triamines usable in detergent compositions as tensides, however, they did not follow their biological activity.

In the preparation of the compounds mentioned above we started from one of the most frequently occurring and available polyamines, bis(2-aminoethyl)amine,

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\* For Part XIII see *Tetrahedron*, in press.

and from the assumption that increased number of amine-oxide groups in the molecule can positively affect not only the physicochemical properties but also the biological activity of such types of compounds. Therefore, we followed the effect of change in the length of the alkyl chain in tris(*N*-oxide) on their spectral properties and antimicrobial activity which was compared to that of monoamine oxides, derived from saturated heterocyclic amines, and of bis(*N*-oxides), derived from 1,6-hexanediamine, described in our previous papers [4, 11].

## Experimental

Infrared spectra were measured as a film (*I—XVIII*) or nujol mull on KBr windows; the spectra of tris(*N*-oxides) (*XIX—XXVII*) were measured besides nujol mull also in dichloromethane in KRS 5 cells with an IR-75 (Zeiss, Jena) spectrophotometer calibrated with polystyrene foil. Reading accuracy was  $\pm 1 \text{ cm}^{-1}$ .

The purity of all compounds was checked on TLC plates (*R<sub>f</sub>* was the average value of five measurements) after [7].

Antimicrobial activity was determined by the dilution test method and was expressed as minimum inhibitory concentration (MIC) [16].

Analytical, spectral, and antimicrobial characterization of the synthesized compounds is presented in Tables 1—5.

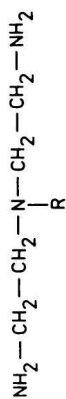
### *Bis(2-aminoethyl)alkylamines*

To the solution of bis(benzylidene-2-aminoethyl)amine (0.2 mol) (prepared by the reaction of benzaldehyde (0.4 mol) and bis(2-aminoethyl)amine (0.2 mol) in benzene in quantitative yield, m.p. 50—52 °C (petroleum ether)) in dry benzene (300 cm<sup>3</sup>) 1-bromoalkane (0.1 mol) was added at boiling within 1 h and the reaction mixture was refluxed for 8 h. After cooling the same amount of dry ether was added to the mixture and the precipitated ammonium salt (bis(benzylidene-2-aminoethyl)ammonium bromide) was filtered off. The solvents were distilled off under reduced pressure and the residue was suspended in water (100 cm<sup>3</sup>). Then concentrated H<sub>2</sub>SO<sub>4</sub> (0.6 mol) was added and the reaction mixture was distilled with water for ca. 3 h. After extraction of the distillation residue with ether, solid NaOH was added to the water layer of the distillation residue under cooling until separation of the amine. The product was extracted with ether and after drying of the extracts and distillation of the solvent, it was redistilled in inert atmosphere (Table 1).

### *Bis(2-dimethylaminoethyl)alkylamines*

Bis(2-aminoethyl)alkylamine (0.05 mol) was added to the methylation mixture (98—100 % formic acid (0.8 mol) and 36 % formaldehyde (0.44 mol)) and was allowed to react at 100 °C for 8 h. After cooling, concentrated hydrochloric acid (0.15 mol) was added

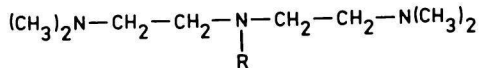
Table I  
 Characterization of bis(2-aminoethyl)alkylamines



Compound	R	Formula $M_r$	$w_i(\text{calc.})/\%$ $w_i(\text{found})/\%$			Yield %	B.p./°C M.p./°C	$R_f$ ( $p/\text{kPa}$ ) ( $n(D, 20^\circ\text{C})$ )
			C	H	N			
I	Octyl	$\text{C}_{12}\text{H}_{29}\text{N}_3$ 215.38	66.92 66.96	13.57 13.67	19.51 19.24	61	120 (1.4678)	0.84 (1.33)
II	Nonyl	$\text{C}_{13}\text{H}_{31}\text{N}_3$ 229.41	68.06 67.78	13.62 13.95	18.32 18.60	67	176—178	0.81 (1.4673)
III	Decyl	$\text{C}_{14}\text{H}_{33}\text{N}_3$ 243.44	69.07 69.00	13.66 13.32	17.26 17.15	50	166—169	0.80 (1.4684)
IV	Undecyl	$\text{C}_{15}\text{H}_{35}\text{N}_3$ 257.47	69.98 69.98	13.70 14.10	16.32 16.22	51	194—196 20—21	0.79 (0.93)
V	Dodecyl	$\text{C}_{16}\text{H}_{37}\text{N}_3$ 271.49	70.79 71.07	13.74 13.74	15.48 15.29	50	214 22	0.78 (1.60)
VI	Tridecyl	$\text{C}_{17}\text{H}_{39}\text{N}_3$ 285.52	71.51 71.79	13.77 13.99	14.72 14.85	62	168 23—24	0.77 (0.05)
VII	Tetradecyl	$\text{C}_{18}\text{H}_{41}\text{N}_3$ 299.55	72.17 71.95	13.80 14.01	14.03 13.80	51	215 25—26	0.76 (0.20)
VIII	Pentadecyl	$\text{C}_{19}\text{H}_{43}\text{N}_3$ 313.57	72.78 72.40	13.82 14.03	13.40 13.51	68	183 29—31	0.60 (0.06)
IX	Hexadecyl	$\text{C}_{20}\text{H}_{45}\text{N}_3$ 327.60	73.33 73.06	13.85 14.07	12.83 12.86	50	180—184 34—36	0.51 (0.08)

Table 2

Characterization of bis(2-dimethylaminoethyl)alkylamines



Compound	R	Formula $M_r$	$w_i(\text{calc.})/\%$ $w_i(\text{found})/\%$			Yield %	B.p./°C ( $p/\text{kPa}$ ) $n(\text{D}, 20^\circ\text{C})$	$R_f$
			C	H	N			
X	Octyl	$\text{C}_{16}\text{H}_{37}\text{N}_3$	70.79	13.74	15.48	87	159—161 (1.60) 1.4534	0.81
		271.49	70.60	14.54	15.29			
XI	Nonyl	$\text{C}_{17}\text{H}_{39}\text{N}_3$	71.51	13.77	14.72	66	172 (1.60) 1.4558	0.80
		285.52	71.37	14.01	14.57			
XII	Decyl	$\text{C}_{18}\text{H}_{41}\text{N}_3$	72.17	13.80	14.03	68	138 (0.09) 1.4551	0.79
		299.55	71.95	13.99	14.20			
XIII	Undecyl	$\text{C}_{19}\text{H}_{43}\text{N}_3$	72.78	13.82	13.40	64	152—155 (0.09) 1.4564	0.78
		313.57	72.53	14.05	13.52			
XIV	Dodecyl	$\text{C}_{20}\text{H}_{45}\text{N}_3$	73.33	13.85	12.83	63	163 (0.09) 1.4555	0.77
		327.60	73.65	13.84	12.99			
XV	Tridecyl	$\text{C}_{21}\text{H}_{47}\text{N}_3$	73.83	13.87	12.30	77	153 (0.03) 1.4570	0.76
		341.63	73.91	13.97	12.48			
XVI	Tetradecyl	$\text{C}_{22}\text{H}_{49}\text{N}_3$	74.30	13.89	11.81	70	186 (0.02) 1.4559	0.72
		355.66	74.64	13.66	11.81			
XVII	Pentadecyl	$\text{C}_{23}\text{H}_{51}\text{N}_3$	74.73	13.91	11.37	70	188—190 (0.07) 1.4590	0.65
		369.68	74.50	13.79	11.62			
XVIII	Hexadecyl	$\text{C}_{24}\text{H}_{53}\text{N}_3$	75.13	13.92	10.95	82	170 (0.03) 1.4565	0.59
		383.71	75.35	14.04	10.73			

and the volatile components were distilled off *in vacuo*. The solid residue was dissolved in water (100 cm<sup>3</sup>) and solid NaOH was added under cooling until separation of the amine. After extraction with ether, drying of the extracts, and distillation of the solvent, the product was once distilled and redistilled from sodium metal. Distillation was performed in inert atmosphere (Table 2).

### *Amine oxides*

Tris(*N*-oxides) (Table 3) were prepared by oxidation of bis(2-dimethylaminoethyl)alkylamines (0.01 mol) in methanol (30 cm<sup>3</sup>) with 30 % aqueous solution of hydrogen peroxide (0.1 mol) and crystallized from the mixture of dry acetone and ethanol (*v/v* = 10:3) to constant melting points. The general procedure for the preparation is described in [3]. The lower members of the series (XIX—XXI) were very hygroscopic, the compounds XXII—XXIV were little, and the compounds XXV—XXVII were not hygroscopic at all. All compounds were stored *in vacuo* over P<sub>4</sub>O<sub>10</sub>.

### **Results and discussion**

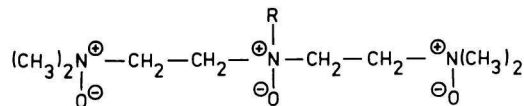
The yield of alkylation of bis(benzylidene-2-aminoethyl)amine and thus also the yield of bis(2-aminoethyl)alkylamines depend decisively on the nucleophilicity of the secondary nitrogen (the reaction proceeds *via* S<sub>N</sub>2 mechanism). Nucleophilicity can be increased by formation of an anion, *e.g.* by the reaction with NaH [17]. The yields in such alkylations varied about 50 %, *i.e.* were lower than those obtained by the method described. Besides, the final products were always contaminated with a small amount of by-products formed by reduction of the starting compound and some reaction intermediates. Lower yields may be ascribed also to insufficient neutralization of the amine during the formation of the anion.

Another method using anhydrous K<sub>2</sub>CO<sub>3</sub> [18] which afforded very high yields of the alkylation product in many cases, gave maximum 50 % yield of the product probably due to insufficient contact of the reactants (the reaction was carried out without solvent and stirring).

The described method for alkylation using molar excess of the secondary amine in dry benzene (toluene) was shown to be most suitable from the view-point of both the yield and purity of the products. Besides, it was not necessary to isolate the formed bis(benzylidene-2-aminoethyl)alkylamine as it was immediately used in the further step of the reaction.

To achieve high yields of amine oxides, it is necessary to use 3- to 5-fold excess of the oxidant because the middle tertiary nitrogen is oxidized relatively slowly. On staying the formed amine oxides in the presence of trace amounts of impurities slowly decompose. Decomposition can be detected by TLC or infrared spectroscopy (slow vanishing of the band belonging to  $\nu(\text{N—O})$ ). Therefore, final purifica-

Table 3

Characterization of tris(*N*-oxides) of bis(2-dimethylaminoethyl)alkylamines

Compound	R	Formula $M_r$	$w_i(\text{calc.})/\%$ $w_i(\text{found})/\%$			Yield %	M.p. °C	$R_f$
			C	H	N			
XIX	Octyl	$\text{C}_{16}\text{H}_{37}\text{N}_3\text{O}_3^a$	54.06	11.62	11.82	85	124—125	0.68
		319.49	53.81	11.80	11.73			
XX	Nonyl	$\text{C}_{17}\text{H}_{39}\text{N}_3\text{O}_3^b$	52.68	11.70	10.84	97	120—121	0.66
		333.52	52.81	11.42	10.61			
XXI	Decyl	$\text{C}_{18}\text{H}_{41}\text{N}_3\text{O}_3^b$	53.83	11.80	10.46	90	121—123	0.64
		347.55	53.69	11.69	10.27			
XXII	Undecyl	$\text{C}_{19}\text{H}_{43}\text{N}_3\text{O}_3^b$	54.91	11.88	10.11	84	116—117	0.63
		361.57	54.97	11.75	9.99			
XXIII	Dodecyl	$\text{C}_{20}\text{H}_{45}\text{N}_3\text{O}_3^a$	58.36	12.00	10.21	86	119—120	0.62
		375.60	58.58	12.07	10.08			
XXIV	Tridecyl	$\text{C}_{21}\text{H}_{47}\text{N}_3\text{O}_3^a$	59.26	12.08	9.87	94	114—115	0.61
		389.63	58.93	12.34	9.59			
XXV	Tetradecyl	$\text{C}_{22}\text{H}_{49}\text{N}_3\text{O}_3^a$	60.10	12.15	9.55	95	116—118	0.57
		403.66	59.97	12.10	9.30			
XXVI	Pentadecyl	$\text{C}_{23}\text{H}_{51}\text{N}_3\text{O}_3^a$	60.89	12.22	9.26	89	112—113	0.47
		417.68	61.21	12.44	9.46			
XXVII	Hexadecyl	$\text{C}_{24}\text{H}_{53}\text{N}_3\text{O}_3^c$	64.10	12.33	9.34	82	115—116	0.46
		431.71	63.95	12.35	9.12			

a) Dihydrate; b) trihydrate; c) monohydrate.

tion of the prepared amine oxides is of key importance for their stability. Aqueous and ethanolic solutions of these compounds are stable.

In the infrared spectra (Table 4) of bis(2-aminoethyl)alkylamines (*I—IX*) two intensive bands belonging to  $\nu_{as}(\text{NH}_2)$  and  $\nu_s(\text{NH}_2)$  were observed in the region of  $\tilde{\nu} = 3360\text{—}3340\text{ cm}^{-1}$  and  $\tilde{\nu} = 3290\text{—}3225\text{ cm}^{-1}$  to be shifted to lower wavenumbers (especially  $\nu_s(\text{NH}_2)$ ) more than reported in the literature [19]. The sharp band of medium intensity  $\nu(\text{C—N})$  appeared at  $\tilde{\nu} = 1117\text{—}1100\text{ cm}^{-1}$  and was also shifted to higher wavenumbers when compared to the literature [19]. The sharp band of medium intensity belonging to deformation “rocking” vibrations of the aliphatic chain was observed at  $\tilde{\nu} = 716\text{—}710\text{ cm}^{-1}$ . In contrast to the literature data [19—21] for i.p. deformation (scissoring) vibration of the  $\text{NH}_2$  group at about  $\tilde{\nu} = 1620\text{ cm}^{-1}$  ( $1615\text{ cm}^{-1}$ ) (broader intensive band), we have observed with the primary amines (*I—IX*) evident splitting of the band to a doublet of relatively sharp bands of medium intensity with maxima at  $\tilde{\nu} = 1677\text{—}1651\text{ cm}^{-1}$  and  $\tilde{\nu} = 1591\text{—}1571\text{ cm}^{-1}$ . At  $\tilde{\nu} = 1620\text{ cm}^{-1}$  ( $1615\text{ cm}^{-1}$ ) we have not observed any bands. The broad intensive band with the maximum approximately at  $\tilde{\nu} = 840\text{ cm}^{-1}$  belonging to  $\delta(\text{NH}_2)_{\text{wag}} + \delta(\text{NH}_2)_{\text{twist}}$  was observed in the spectra of all these amines (*I—IX*) and is characteristic of primary amines.

In the spectra of bis(2-dimethylaminoethyl)alkylamines (*X—XVIII*) the band belonging to  $\text{NH}_2$  group vanished (when compared to the spectra of the previous group of compounds), however, a new sharp intensive band  $\nu(\text{N—CH}_3)$  appeared in the region of  $\tilde{\nu} = 2765\text{—}2762\text{ cm}^{-1}$ . The position and shape of the bands  $\nu(\text{C—N})$  at  $\tilde{\nu} = 1120\text{—}1115\text{ cm}^{-1}$  and  $\rho(\text{CH}_2)$  at  $\tilde{\nu} = 720\text{—}710\text{ cm}^{-1}$  remained practically the same as with the compounds *I—IX*.

The spectra of amine oxides (*XIX—XXVII*) exhibited a new doublet of sharp bands with approximately the same medium intensity (the band I was more intensive) at  $\tilde{\nu} = 968\text{—}955\text{ cm}^{-1}$  and  $\tilde{\nu} = 941\text{—}930\text{ cm}^{-1}$  (nujol mull) and at  $\tilde{\nu} = 965\text{—}945\text{ cm}^{-1}$  and  $\tilde{\nu} = 933\text{—}923\text{ cm}^{-1}$  (dichloromethane), respectively. The appearance of the broad intensive band with the maximum approximately at  $\tilde{\nu} = 3400\text{ cm}^{-1}$  (both in nujol and dichloromethane) indicated the presence of crystalline water determined by elemental analysis. The presence of crystalline water in the molecule of amine oxides has been already described [9, 13, 22]. The weak intensity bands belonging to  $\rho(\text{CH}_2)$  exhibited maxima in the region of  $\tilde{\nu} = 720\text{—}710\text{ cm}^{-1}$ .

The change in the length of the alkyl chain essentially influenced only the positions of the absorption bands  $\nu_{as}(\text{NH}_2)$ ,  $\nu_s(\text{NH}_2)$ , and  $\delta(\text{NH}_2)$ . In these cases lengthening of the chain (in lower members of the series) was reflected in shifts of the band maxima to higher wavenumbers, with the alkyl chain of C-10—C-11 these values decreased and with C-12—C-14 they became constant. This phenomenon can be probably ascribed to changes in steric arrangement of the molecules via the mechanism described in [3]. It is of interest that the position of the band  $\nu(\text{N—O})$

Table 4

Infrared spectral data ( $\tilde{\nu}/\text{cm}^{-1}$ ) of bis(2-aminoethyl)alkylamines, bis(2-dimethylaminoethyl)alkylamines, and tris(*N*-oxides) of bis(2-dimethylaminoethyl)alkylamines

Compound	$\nu_{\text{as}}(\text{NH}_2)$ $\nu_{\text{s}}(\text{NH}_2)$	$\delta(\text{NH}_2)$	$\nu(\text{C—N})$	$\rho(\text{CH}_2)$	Compound	$\nu(\text{N—CH}_3)$	$\nu(\text{C—N})$	$\rho(\text{CH}_2)$	Compound	$\nu(\text{N—O})$ I <sup>a</sup> $\nu(\text{N—O})$ II <sup>a</sup>
I	3348	1657	1106	713	X	2764	1120	720	XIX	955/945
	3284	1576 <sup>b</sup>								941/933
II	3346	1651	1100	713	XI	2764	1115	710	XX	955/947
	3279	1571 <sup>b</sup>								940/932
III	3360	1661 <sup>b</sup>	1107	711	XII	2763	1115	715	XXI	956/949
	3290	1583								938/930
IV	3358	1677 <sup>b</sup>	1105	710	XIII	2765	1115	715	XXII	958/950
	3286	1591								936/928
V	3342	1663 <sup>b</sup>	1110	716	XIV	2765	1115	720	XXIII	968/965
	3274	1583								931/924
VI	3345	1665	1107	715	XV	2763	1115	716	XXIV	962/954
	3249	1584 <sup>b</sup>								935/930
VII	3340	1669	1111	715	XVI	2762	1115	720	XXV	963/956
	3225	1587 <sup>b</sup>								934/928
VIII	3342	1659	1117	711	XVII	2762	1115	718	XXVI	962/955
	3230	1586 <sup>b</sup>								932/926
IX	3340	1657 <sup>b</sup>	1115	715	XVIII	2762	1115	720	XXVII	960/954
	3231	1574								930/923

a) Nujol mull/dichloromethane; b) more intensive band of the doublet.



with tris(*N*-oxides) was not influenced by the length of the alkyl chain (contrary to our previous observations).

Antimicrobial activity of tris(*N*-oxides) against representative gram-positive cocci (*S. aureus*), gram-negative rods (*E. coli*), and yeasts (*C. albicans*) was established by the dilution method (Table 5). We have found that changes in the structure (lengthening of the alkyl chain) brought about an increase in the activity. The compound *XXII* (alkyl chain C-11) was the first one in the series exhibiting activity, maximum activity was observed with *XXVI* and *XXVII* (alkyl chain C-15—C-16). This observation is somewhat surprising because by increasing the number of amine-oxide groups in the molecule (turning from mono- to bis(*N*-oxides)) maximum activity was achieved already with the alkyl chain of C-10—C-11 [11] (monoamine oxides C-13—C-15 [4]). It is also noteworthy that though the compounds tested were highly active against gram-positive bacteria and yeasts, even more active than some organic ammonium salts, e.g. Ajatin (benzylododecyl-

Table 5  
Antimicrobial activity of tris(*N*-oxides) of bis(2-dimethylaminoethyl)alkylamines<sup>a</sup>

Compound	MIC/( $\mu\text{g cm}^{-3}$ ) MIC/( $\text{mmol dm}^{-3}$ )	
	<i>S. aureus</i>	<i>C. albicans</i>
<i>XIX</i>	>1000	>1000
	>2.8127	>2.8127
<i>XX</i>	>1000	>1000
	>2.5803	>2.5803
<i>XXI</i>	>1000	>1000
	>2.4903	>2.4903
<i>XXII</i>	500	600
	1.2029	1.4436
<i>XXIII</i>	100	400
	0.2429	0.9717
<i>XXIV</i>	30	300
	0.0705	0.7050
<i>XXV</i>	8	50
	0.0182	0.1137
<i>XXVI</i>	9	40
	0.0198	0.0882
<i>XXVII</i>	9	9
	0.0200	0.0200
Ajatin <sup>b</sup>	10	10
	0.0260	0.0260

<sup>a</sup>) Activity calculated on amine oxides mono-, di-, and trihydrates; <sup>b</sup>) activity against *E. coli* 100/0.2600.

dimethylammonium bromide) used as the standard, their activity against gram-negative bacteria *E. coli*, expressed in MIC, was higher than  $1000 \mu\text{g cm}^{-3}$  (with the exception of XXII where  $\text{MIC} = 900 \mu\text{g cm}^{-3}$  ( $2.1653 \text{ mmol dm}^{-3}$ )). It means that all compounds tested were inactive against *E. coli*. From this point of view the tris(*N*-oxides) studied can be regarded as compounds with selective antimicrobial activity. In comparison with mono- [4] and bis(*N*-oxides) [11] it can be stated that increased number of amine-oxide groups in the molecule did not lead to substantial increase in the activity against *S. aureus* and *C. albicans*, but the activity against *E. coli* was practically lost.

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