OXIDATION OF SECONDARY ALIPHATIC AMINES IN SYSTEMS CONTAINING SODIUM PEROXYDISULFATE

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The kinetic aspects of the reaction of dialkylamines with free radicals [1], oxygen [2], peroxides [3], chlorine dioxide [4], potassium permanganate [5], potassium ferricyanide [6], and other oxidants have been studied in great detail [3, 7]. The mechanisms of these reactions, the structure of the intermediate dialkylamine cation radicals [8], and dialkylaminyl radicals [9] have been examined, and the potentials of formation of nitrogen-centered cation radicals from dialkyl amines have been determined [10].

The chemical transformations of secondary amines in oxidizing systems have been studied in much less detail. It is known that aldehydes are formed from dialkylamines under the effect of sodium peroxydisulfate in an alkaline medium [11] or silver picolinate [12].

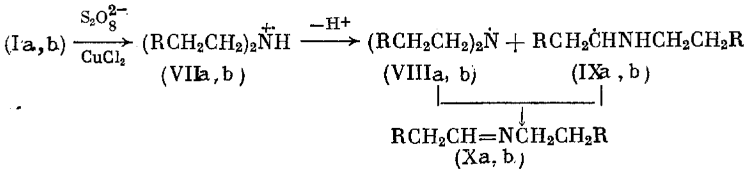
In continuing the sudies of the reactions of aminyl radicals obtained from primary amines in oxidizing systems [13-15], we studied the reactions of secondary aliphatic amines in $Na_2S_2O_8$–$CuCl_2$ and $Na_2S_2O_8$–NaCN systems.

We found that 2-chloroalkanals (IIa, b), 2,2-dichloroalkanals (IIIa, b), alkanoic acids (IVa, b), and N-alkylalkanamides (Va, b) are formed from amines $(RCH_2CH_2)_2NH$ (Ia, b) under the effect of the $Na_2S_2O_8$–$CuCl_2$ system when equimolar amounts of the substrate and components of the oxidizing system in water at 70-80°C are used. Alkylamine (VIb) (Table 1) was also identified in the products of oxidation of (Ib)

$$\underset{\text{(Ia, b)}}{(RCH_2CH_2)_2NH} \xrightarrow{Na_2S_2O_8-CuCl_2} \underset{\text{(IIa, b)}}{RCHClCHO} + \underset{\text{(IIIa, b)}}{RCCl_2CHO} +$$
$$+ \underset{\text{(IVa, b)}}{RCH_2COOH} + \underset{\text{(Va, b)}}{RCH_2CH_2NHC(O)CH_2R} + \underset{\text{(VIb)}}{RCH_2CH_2NH_2}$$

where R = Et (a), Pr (b).

We will assume that the mechanism of the reaction of dialkylamines (Ia, b) with the $Na_2S_2O_8$–$CuCl_2$ oxidizing system includes their one-electron oxidation to amine cation radicals (VIIa, b), where their deprotonation causes the formation of dialkylaminyl radicals (VIIIa, b) and possibly aminoalkyl radicals (IXa, b). Subsequent oxidation of both types of radicals results in azomethines (Xa, b).



$$\text{(Ia, b)} \xrightarrow[CuCl_2]{S_2O_8^{2-}} \underset{\text{(VIIa, b)}}{(RCH_2CH_2)_2\overset{+\cdot}{N}H} \xrightarrow{-H^+} \underset{\text{(VIIIa, b)}}{(RCH_2CH_2)_2\dot{N}} + \underset{\text{(IXa, b)}}{RCH_2\dot{C}HNHCH_2CH_2R} \longrightarrow \underset{\text{(Xa, b)}}{RCH_2CH{=}NCH_2CH_2R}$$

Kinetic studies showed tht deprotonation of (VIIa, b) into (IXa, b) is significantly less probable than the formation of dialkylamine radicals (VIIIa, b) from (VIIa, b) [16].

2-Chloro- and 2,2-dichloroalkanals (IIa, b), (IIIa, b), the basic products of the reaction, and amides (Va, b) are formed from azomethines (Xa, b) in the $Na_2S_2O_8$–$CuCl_2$ system (see Experimental)

$$\text{(Xa, b)} \xrightarrow[\text{2. Hydrolysis}]{\text{1. } Na_2S_2O_8-CuCl_2} \text{(IIa, b)} + \text{(IIIa, b)}$$

Amines (VIa) and RCH_2CHO alkanals (XIa, b) are obtained in the hydrolysis of (Xa, b) without an oxidant and are transformed into acids (IVa, b) in the conditions of the reac-

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TABLE 1. Oxidation of Dialkylamines (Ia, b) in the $Na_2S_2O_8$–$CuCl_2$ System (70-80°C, 7.5 h, (I) = $Na_2S_2O_8$ = $CuCl_2 \cdot 2H_2O$ = 100 mmoles, 75 ml of water)

Dialkylamine	Conversion, %	Products of the reaction, yield, % of transformed amine				
		2-chloroalkanal	2,2-dichloroalkanal	acid	amide	alkylamine
Dibutylamine (Ia)	40	(IIa), 24	(IIIa), 18	(IVa), 10	(Va), 8	–
Diamylamine (Ib)	35	(IIb), 10	(IIIb), 38	(IVb), 6	(Vb), 4	(VIb), 18

TABLE 2. Oxidation of Dialkylamines (Ia, b) in the $Na_2S_2O_8$–NaCN–NaOH System (70°C, 7.5 h, (I) = $Na_2S_2O_8$ = NaCN = NaOH = 100 mmoles, 100 ml of Water)

Dialkylamine	Conversion, %	Products of the reaction yield, % of transformed amine			
		cyanamide	urea	hydroxylamine	aminonitrile
Dibutylamine (Ia)	100	(XIIIa), 7	(XIVa), 30	(XVa), 20	(XVIa), 30
Diamylamine (Ib)	60	(XIIIb), 6	(XIVb), 42	(XVb), 16	(XVIb), 22

tion. The significant predominance of chloro-substituted alkanals (II) and (III) in a mixture with acids (IV) and their amides (V) permits concluding that oxidizing chlorination of azomethines (Xa, b) takes place with a higher rate than the hydrolysis of these substances.

Diamylaminyl radical (VIIIb) was also obtained in the thermolysis of N-chlorodiamylamine (XII) in the presence of the $Na_2S_2O_8$–$CuCl_2$ system [17], from which 28% (IIb), 12% (IIIb), 8% (IVb), 5% (Ib), and 3% (VIb) were formed in the conditions of the reaction (the yields are reported for the (XII) used)

$$\underset{\text{(XII)}}{(C_3H_7CH_2CH_2)_2NCl} \xrightarrow{\Delta} \text{(VIIIb)} \xrightarrow{Na_2S_2O_8-CuCl_2} \text{(IIb)} + \text{(IIIb)} + \text{(IVb)} + \text{(Ib)} + \text{(VIb)}$$

The composition and ratio of the products of this reaction are in good agreement with the mechanism examined above, which consists of the one-electron oxidation of the dialkylamines into the corresponding dialkylaminyl radicals.

In the development of the previously studied new reaction of oxidative N-cyanidation of primary amines [15], we studied the reaction of secondary amines (Ia, b) in the $Na_2S_2O_8$–NaCN–NaOH system and found that when equimolar quantities of the substrate and components of the oxidizing system in water are used at 70°C, dialkylcyanamides (XIIIa, b) N,N-dialkylureas (XIVa, b), N,N-dialkylhydroxylamines (XVa, b), and 2-(dialkylamino)alkanonitriles (XVIa, b) are formed (Table 2)

$$\text{(Ia, b)} \xrightarrow[NaCN-NaOH]{Na_2S_2O_8-} \underset{\text{(XIIIa, b)}}{(RCH_2CH_2)_2NCN} + \underset{\text{(XIVa, b)}}{(RCH_2CH_2)_2NCONH_2} + \underset{\text{(XVa, b)}}{(RCH_2CH_2)_2NOH} + \underset{\text{(XVIa, b)}}{(RCH_2CH_2)_2NCH(CN)CH_2R}$$

The data reported indicate that dialkylamines (Ia, b), similar to n-alkylamines [15], undergo oxidative N-cyanidation with the formation of cyanamides (XIIIa, b) and the products of their hydrolysis, ureas (XIVa, b). The mechanism of the reaction most probably consists of one-electron oxidation (Ia, b) to dialkylaminyl radicals (VIIIa, b) and their subsequent oxidation into dialkylamine cations (XVIIa. b)

$$\text{(Ia, b)} \rightarrow \text{(VIIIa, b)} \xrightarrow{Na_2S_2O_8-NaCN} \underset{\text{(XVIIa, b)}}{(RCH_2CH_2)_2\overset{+}{N}}$$

The possibility of the intermediate formation of the dibutylamine cation was established in studying the kinetics of oxidation of (Ia) under the effect of Ph_2ICl [16].

The reaction of (XVIIa, b) with the nucleophilic CN$^-$, OH$^-$ (or H_2O) present in the medium results in covalently bound products of the reaction "with ligand transfer": cyanamides (XIIIa, b) and hydroxylamines (XVa, b)

$$\text{(XVIIa, b)}\left|\begin{array}{l}\xrightarrow{\text{CN}^-}\text{(XIIIa, b)}\xrightarrow{\text{Hydrolysis}}\text{(XIVa, b)}\\ \xrightarrow{\text{OH}^-\text{ or }H_2O}\text{(XVa, b)}\end{array}\right.$$

Azomethines (Xa, b) are formed in deprotonation of (VIIIa, b) or (XVIIa, b), and their subsequent hydrolysis in the conditions of the reaction results in aldehydes (XIa, b). When they react with the initial (Ia, b) in a Strecker reaction, 2-(dialkylamino)alkanonitriles (XVIa, b) are formed

$$\text{(Ia, b)}+\text{(XIa, b)}\rightarrow\underset{\text{(XVIIIa, b)}}{[(RCH_2CH_2)_2NCH(OH)CH_2R]}\xrightarrow{CN^-}\text{(XVIa, b)}$$

This mechanism of the formation of (XVIa, b) was confirmed by a control experiment (cf. Experimental). It was also shown that tributylamine (XIX) is almost inert in the conditions of oxidation in the $Na_2S_2O_8$–NaCN–$\overset{+}{N}$aOH system. This permits excluding the alternative mechanism of formation of (XVIa, b) as a result of hydrocyanidation of $(RCH_2CH_2)_2\overset{+}{N}=CHCH_2R$, the product of dehydration of amino alcohols (XVIIIa, b).

A comparison of the results on oxidative N-cyanidation of primary alkylamines, where the total yield of cyanamides and ureas attains 70% [15], and the similar reactions of secondary dialkylamines indicates that the latter take place less selectively.

This is apparently due to the greater tendency of dialkylaminyl radicals (in comparison to alkylaminyl radicals) to oxidize into the corresponding amine cations [7], which react nonselectively with the nucleophilic CN$^-$ and OH$^-$ (or H_2O) present in the reaction medium.

The composition of the products of oxidation of secondary dialkylamines in the $Na_2S_2O_8$–$CuCl_2$ system is thus in best agreement with the initial formation of azomethines, the precursors of all identified products of the reaction, as a result of oxidative deprotonation, while oxidative substitution of the H atom in the nitrogen predominates in the $Na_2S_2O_8$–NaCN–NaOH oxidative system. This difference is apparently primarily related to the well-known tendency of copper ions to induce oxidative deprotonation [18].

The structure of all products of the reaction was confirmed by GLC, using PMR, IR, mass spectral, and chromato-mass spectral data for comparison with previously known samples. Reference samples were prepared by reverse synthesis: 2,2-dichloroalkanals (IIIa, b) [19], amides (Va, b) [15], N,N-dibutylurea (XIVa) [20], and N,N-dibutylhydroxylamine (XVa) [21].

EXPERIMENTAL

The GLC analysis was conducted on a LKhM-8MD chromatograph with a flame ionization detector in a N_2 current; columns (stainless steel): 300 × 0.4 cm with 10% Carbowax 20 M, treated with Na_3PO_4 and NaOH on Celite-545 (52-60 mesh); 300 × 0.3 cm with 2% DS-550 on Chromosorb R (120-140 mesh), washed with acid and treated with Me_2SiCl_2; 100 × 0.3 cm with 5% OV-225 on Chromaton Super (120-140 mesh) treated with Me_2SiCl_2. The PMR spectra of solutions in $CDCl_3$ and $(CD_3)_2SO$ were made on Varian DA-60-IL (60 MHz), Tesla BS-467 (60 MHz), and Bruker WM-250 (250 MHz) spectrometers using TMS as the internal standard. The mass spectra were made on a Varian MATCH-6 spectrometer with direct introduction of the sample into the ion source and ionizing electron energy of 70 eV. The chromato-mass spectrometer analysis was conducted on a Varian MATCH-111 with chromatographic introduction of the sample into the ion source and ionizing electron energy of 80 eV. The IR spectra were made on Specord 75-IR and UR-20 spectrometers in a thin layer in solutions in $CHCl_3$ and CCl_4 and compression with KBr.

Cp $Na_2S_2O_8$, cp NaOH, NaCN, and $CuCl_2\cdot 2H_2O$ were used without additional purification. The water was distilled once. Amines (Ia, b) were purified by distillation at atmospheric pressure in an Ar current.

Oxidation of Dialkylamines (Ia, b) in the $Na_2S_2O_8$–$CuCl_2$ System. A solution of 100 mmoles of $Na_2S_2O_8$ in 50 ml of water was added to a mixture of 100 mmoles of amine (Ia, b) and a solution of 100 mmoles of $CuCl_2\cdot 2H_2O$ in 50 ml of water over 1.5 h at 70°C with in-

TABLE 3. Spectral Characteristics of the Products of the Reactions

Compound	IR spectrum, ν, см$^{-1}$	PMR spectrum (δ, ppm)	Mass spectrum, m/z (rel. intensity, %)
2-Chlorobutanal (IIa)	1730 (C=O)	4,05 m (1H, CHCl), 9,43 d (1H, CHO)	–
2-Chloropentanal (IIb)	1730 (C=O)	4,05 m (1H, CHCl), 9,43 d (1H, CHO)	–
2,2-Dichlorobutanal (IIIa)[*]	1747 (C=O)	1,20 t (3H, CH$_3$), 2,30 q (2H, CH$_2$), 9,18 s(1H, CHO)	См. [19]
2,2-Dichloropentanal (IIIb)	1747 (C=O)	0,95 t (3H, CH$_3$), 1,45m (2H, CH$_2$), 2,30 t (2H, CH$_2$CCl$_2$), 9,20 s(1H, CHO)	129(2), 127(14), 125(23), 114(13), 112(20), 91(40), 89(66), 89(66), 83(22), 75(22), 63(54), 55(100), 53(43), 44(70), 43(95), 42(42), 41(73)
N-Butylbutanamide (Va)	3280, 3080 (NH) 1645, 1550 (CONH)	0,90 t (6H, CH$_3$), 1,45 m (6H, CH$_2$), 2,10 t (2H, CH$_2$CO) 3,20 t (2H, CH$_2$N), 7,15 br, s (1H, H)	143, M$^+$(15), 128(8), 115(14), 101(11), 100(19), 88(9), 86(7), 73(18), 71(44), 57(29), 44(100), 43(50), 41(33)
N-Pentylpentanamide (Vb)		[15]	
N-Chlorodiamylamine (XII)		2,85 t (4H, CH$_2$N)	
N,N-Dibutyl cyanamide (XIIIa)	2170 (CN)	0,90 t (6H, CH$_3$), 1,35 m (8H, CH$_2$), 3,17 t (4H, CH$_2$N)	154(M$^+$)
N,N-Dipentylcyanamide (XIIIb)	2170 (CN)	0,90 t (6H, CH$_3$), 1,35 m (12H, CH$_2$), 3,17 t (4H, CH$_2$N)	182 (M$^+$)
N,N-Dibutylurea (XIVa)	3490, 3350, 3210, (NH), 1640, 1590 (CONH$_2$)	0,90 t (6H, CH$_3$), 1,35 (8H, CH$_2$), 3,17 t (4H, CH$_2$N), 5,10 br.s (2H, NH$_2$)	172, M$^+$ (26), 143(3), 129(85), 100(12), 86(100), 69(21), 57(52), 56(20), 55(32)
N,N-Dipentylurea (XIVb)	3390, 3350, 3210 (NH) 1640, 1590 (CONH$_2$)	0.90 t (6H, CH$_3$), 1,35 m (12H, CH$_2$), 3,17 t (4H, CH$_2$N), 5,10 br.s (2H, NH$_2$)	200 (M$^+$)
N,N-Dibutylhydroxylamine (XVa)		0,90 t (6H, CH$_3$), 1,30 m (4H, CH$_2$), 1,57 m (4H, CH$_2$), 2,93[†] m (4H, CH$_2$N) Position of OH not determined	145, M$^+$ (8), 128(8), 126(3), 102(59), 100(50), 84(80), 70(30), 57(100)
N,N-Dipentylhydroxylamine (XVb)		0,90 t (6H, CH$_3$), 1,45 m (12H, CH$_2$), 2,93[†] m (4H, CH$_2$N), position of OH not determined	173 (M$^+$)
2-(N,N-Dibutylamino)pentanenitrile (XVIa)	2240 (CN)	0,90 m (9H, CH$_3$), 1,40 m (10H, CH$_2$), 1,70 m (2H, CH$_2$CH), 2,35m and 2,58 m (4H, CH$_2$N), 3,58 t (1H, CH)	210, M$^+$ (5), 183(50), 168(66), 167(64), 154(14), 140(100), 126(37), 125(23), 112(21), 98(82), 86(39), 84(98), 72(21), 70(32), 69(25), 68(25), 57(96), 56(58), 55(62)
2-(N,N-Dipentylamino)hexanenitrile (XVIb)	2240 (CN)	0,90 m (98, CH$_3$), 1,40 m (16H, CH$_2$), 1,70 m (2H, CH$_2$CH), 2,35 m and 2,58 m (4H, CH$_2$N), 3,60 t (1H, CH)	252 (M$^+$)

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[*] ^{13}C NMR spectrum (δ, ppm): 8.75, 34.16, 89.54, 184.88.

[†] The position of this signal is a function of the pH: 2.65 (pH > 10), 2.80 (pH 8), 2.93 (pH 6), 3.30 ppm (pH 3).

tense stirring, mixed for another 6 h at the same temperature, cooled, and extracted with 3 × 100 ml of ether. The extract was dried with $CaSO_4$ and evaporated. 2-Chloroalkanals (IIa, b), 2,2-dichloroalkanals (IIIa, b), acids (IVa, b), and amides (Va, b) were detected in the residue by GLC. The aqueous layer from the oxidation of (Ia) was alkalized with NH_4OH and then KOH and extracted with 3 × 100 ml of water. The extract was dried with $CaSO_4$, evaporated, and the amount of unreacted (Ia) was determined in the residue by GLC. The aqueous layer from oxidation of (Ib) and the crystals which precipitated from it were treated with NH_4OH until they totally dissolved, then with KOH, and were extracted with 3 × 100 ml of ether. The extract was dried with $CaSO_4$, saturated with dry HCl, and filtered. The residue was dissolved in KOH, extracted with ether, the extract was dried, evaporated, and the amount of unreacted (Ib) and amylamine (VIb) formed was determined by GLC. The yields of the products and conversion of (Ia, b) are reported in Table 1. The spectra characteristics are generalized in Table 3.

Reverse synthesis of 2,2-dichlorobutanal (IIa) and 2,2-dichloropentanal (IIb) was conducted by chlorination of 1-butanol and 1-pentanol in DMF according to the method in [19], yielding 50 and 60%, bp 115-116°C (760 mm) and 65°C (60 mm), respectively.

Synthesis of N-chlorodiamylamine (XII) was conducted by chlorination of (Ib) in pentane with Cl_2 according to the method in [22].

Behavior of N-Chlorodiamylamine (XII) in the $Na_2S_2O_8$–$CuCl_2$ System. A solution of 50 mmoles of $N_2S_2O_8$ in 15 ml of water was added to a mixture of a solution of 50 mmoles of freshly prepared (XII) in 20 ml of pentane and a solution of 50 mmoles of $CuCl_2 \cdot 2H_2O$ in 20 ml of water at 70°C over 1 h with intense stirring, stirring was continued for another 6 h at the same temperature, and the mixture was cooled and extracted with ether. GLC revealed 14 mmoles (28%) of (IIb), 6 mmoles (12%) of (IIIb), and 4 mmoles (8%) of (IVb) in the residue. The aqueous layer was alkalized with NH_4OH and then KOH and extracted with ether. The extract was dried, evaporated, and 2.5 mmoles (5%) of (Ib) and 1.5 mmoles (3%) of (VIb) were determined in the residue by GLC.

Synthesis of N-butylidenebutylamine (Xa) was conducted by the reaction of butylamine (VIa) with butanal (XIa) according to [14], and (Xa) was separated by distillation over KOH, yield of 80%, bp of 41°C (11 mm).

Oxidation of N-Butylidenebutylamine (Xa) with the $Na_2S_2O_8$–$CuCl_2$ System. The reaction and treatment of the reaction mixture were conducted with the general method for oxidation of amines. Here 45% butylamine (VIa), 15% butanal (IXa), 15% of the starting (Xa), 7% 2-chlorobutanal (IIa), 15% 2,2-dichlorobutanal (IIIa), 5% butanoic acid (IVa), and 5% N-butylbutanamide (Va) were isolated from the reaction mixture.

Oxidation of Secondary Amines (Ia, b) in the $Na_2S_2O_8$–NaCN–NaOH System. A solution of 100 mmoles of $Na_2S_2O_8$ in 50 ml of water was added to a mixture of 100 mmoles of amine (Ia, b) and a solution of 100 mmoles of NaCN and 100 mmoles of NaOH in 50 ml of water over 1.5 h at 70°C with intense stirring, and stirring was continued for another 6 h at the same temperature; the mixture was cooled and extracted with ether (3 × 100 ml). The extract was dried with $CaSO_4$ and evaporated. The products of the reaction and the unreacted amine (Ia, b) were determined in the residue by quantitative PMR spectroscopy using a spectrometer with a working frequency of 250 MHz and abs. $MeNO_2$ as the standard. The composition of the reaction mixtures is reported in Table 2. The spectral characteristics of the products of the reaction are reported in Table 3.

Behavior of tributylamine (XIX) in the $Na_2S_2O_8$–NaCN–NaOH system. The reaction and treatment of the reaction mixture were conducted as above. Here 95% of unreacted (XIX) was isolated, and no products of its oxidation were detected.

Reverse synthesis of N,N-dibutylurea (XIVa) were conducted as described in [20] with the reaction of (Ia) with $CO(NH_2)_2$ urea. At the end of the reaction, the mxiture was cooled, extracted with ether, and the extract was dried and evaporated. Yield of (XIVa) of 90%.

Reverse Synthesis of N,N-Dibutylhydroxylamine (XVa). [21]. N,N-Dibutylhydroxylamine (XVa), mp 52-53°C, was obtained by the reaction of butanoic acid (IVa), butanal oxime, and $NaBH_4$.

Reverse Synthesis of 2-(N,N-Dibutylamino)pentanenitrile (XVIa). A solution of 20 mmoles of NaCN in 20 ml of water was added to a mixture of 20 mmoles of dibutylamine (Ia), 20 mmoles of butanal (XIa), and 20 mmoles of NaOH in 30 ml of water at 70°C over 1 h with intense stirring, held for another 4 h at the same temperature, cooled and extracted with ether (3 × 50 ml). The extract was dried and evaporated, and the residue was distilled at low pressure. Here 10 mmoles (50%) of (XVIa) were obtained, bp of 86°C (2 mm).

CONCLUSIONS

1. In the $Na_2S_2O_8$—$CuCl_2$ system, secondary aliphatic amines $(RCH_2CH_2)_2NH$ are transformed into 2-chloro- and 2,2-dichloroalkanals, alkanoic acids and their alkylamides; their most probable precursors are $RCH_2CH=NCH_2CH_2R$ azomethines, formed as a result of oxidative deprotonation.

2. In the $Na_2S_2O_8$—NaCN—NaOH system, oxidation of dialkylamines by a mechanism of oxidative substitution with the formation of the products of N-cyanidation: cyanamides and ureas, and the products of N-hydroxylation, dialkylhydroxylamines, predominate.

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