REACTION OF AMIDYL RADICALS IN SODIUM PEROXYDISULFATE – METAL CHLORIDE OXIDATION SYSTEMS

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Amidyl radicals $RC(O)\dot{N}R'$ are objects of intense research [1-3]. Methods have been developed for producing these radicals, usually based on the homolysis of N-haloamides of carboxylic acids [1, 4, 5], and their general reactions have been studied, in particular, the intra- and intermolecular addition to the C=C bond [1, 3], intramolecular cyclization into lactams during addition to aromatic ring, [2], rearrangement with 1,5-migration of H atom, and cyclization into 2-iminotetrahydrofurans [4, 6]. The structure of amidyl radicals in the ground state was established by theoretical calculations and EPR spectroscopy methods [7].

The reaction of amidyl radicals in oxidation systems containing compounds of elements of variable valence have been studied less [8].

We studied the single-electron oxidation of carboxylic acid amides (Ia-e) into amidyl radicals (IIa-e), and the reactions of the latter in the following sodium peroxydisulfate (SPDS)–metal chloride oxidation systems: SPDS–$CuCl_2$, SPDS–NaCl, and also in the SPDS–NaCl–NaOH system

$$\underset{\text{(Ia–e)}}{R(CH_2)_4CONHR'} \xrightarrow{S_2O_8^{2-}-Cl^-} \underset{\text{(IIa–e)}}{R(CH_2)_4C(O)\dot{N}R'}$$

R, R' = H, H (a); CH_3, H (b); C_2H_5, H (c); H, CH_3 (d); H, C_2H_5 (e).

We also studied the oxidative transformations of N,N-diethyl valeramide (If) and cyclohexanecarboxamide in the SPDS–$CuCl_2$ system.

During oxidation of amides (Ia-c) by an equimolar amount of SPDS in the presence of 1 equivalent of $CuCl_2$ or 2 equivalents of NaCl in aqueous solutions at 85-90°C, γ-lactones (IIIa-c) and δ-lactones (IVb, c) are formed*

$$\text{(Ia–c)} \xrightarrow{S_2O_8^{2-}-Cl^-} \underset{\text{(IIIa–c)}}{RCH_2\text{-}\langle O\rangle\text{=O}} + \underset{\text{(IVb,c)}}{R\text{-}\langle O\rangle\text{=O}},$$

A direct one-stage transformation of carboxylic acid amides into lactones has not previously been known, although γ- and δ-lactones can be obtained from amides by a series of successive transformations, including the halogenation of the amides into N-haloamides, their rearrangement during photolysis into amides of 4- and 5-halocarboxylic acids, and cyclization of the latter by treatment with alkali [4, 6].

Under oxidation conditions, amides (Ia-c) also become hydrolyzed into the corresponding alkanoic acids $R(CH_2)_4COOH$ (Va-c) (Table 1).

Thus, in the two systems studied, amide (Ia) converts regiospecifically into γ-valerolactone (IIIa), while from amides (Ib, c) a mixture of γ-lactones (IIIb, c) and δ-lactones (IVb, c) is formed with a marked predominance of γ-lactones. The total yield of the lactonization products in the SPDS–$CuCl_2$ system is ~40%, and it is independent of the length of the alkyl fragment in amides (Ia-c). Under similar conditions, monoalkyl amides (Id, e) convert mainly (>80%) into valeric acid, while the yield of the γ-valerolactone is 2-6%.

The mechanism of formation of the lactones includes a single-electron oxidation of the amides (Ia-e) by the action of the SPDS–Cl^- system into amidyl radicals (IIa-e):

*Preliminary communication, see [9].

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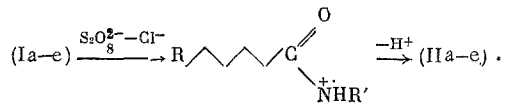
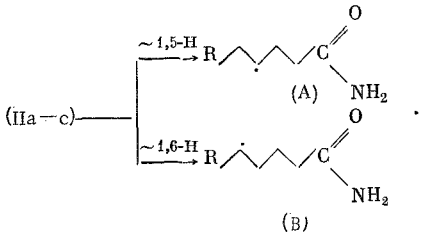
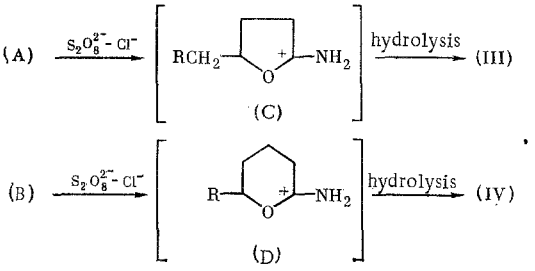


The formation of amidyl radicals during oxidation of the amides by SPDS was shown in [8].

The primary radicals (IIa-c) (R' = H) then rearrange with 1,5- and 1,6-migration of the H atom into 3- and 4-carboxamidoalkyl radicals (A) and (B):



As the result of the homolytic cyclization of (A) and (B) at the C = O group and the subsequent oxidation (cf. [10]), 2-amino-5-alkyl-2-tetrahydrofuryl cations (C) and 2-amino-6-alkyl-2-tetrahydropyranyl cations (D) are formed. Under the reaction conditions, their hydrolysis leads directly to γ-lactones (III) and δ-lactones (IV), which ensures a one-stage character of the lactonization process of the amides:



Lactones (III) and (IV) can also be formed during oxidation of radicals (A) and (B) into the corresponding carbonium ions or amides of chloroalkanoic acids and heterolytic cyclization of the latter. In accordance with the general principles of rearrangements with H atom transfer in free radicals, the 1,5-H-migration predominates over the 1,6-H-migration [11], which also determines the regiospecific or regioselective formation of γ-lactones (III).

In the case of secondary N-alkylamidyl radicals (IId, e) (R' = alkyl), which are more stable than the corresponding primary radicals (R' = H), the rearrangement into carboxamidoalkyl radicals is thermodynamically less favorable; secondary radicals (II) (R' = alkyl) are probably also more readily oxidized, and in the long run this also determines the low yield of the γ-lactone (IIIa) from N-alkylamides (Id, e).

The inertness of (Id, e) to oxidative lactonization under the given conditions does not agree with the literature data on the high tendency of N-chloro-N-methyl valeramide to convert into a γ-valerolactone in nonaqueous media [6].

The ratio between γ- and δ-lactones, derived from the amides substantially differs from their ratio observed in the case of analogous alkanoic acids [12], and, as shown in the case of oxidation of amide (Ib), is practically independent of the degree of conversion of the amide (see Table 1). In the reaction studied, CO_2, a product of the oxidative decarboxylation of alkanoic acids in the presence of SPDS [12-14], has also not been identified. We can thus exclude the possibility of formation of lactones (III) and (IV) as the result of the hydrolysis of amides (I) into the corresponding acids (V), and the lactonization of the latter under the reaction conditions. A control experiment showed that amide (Ia) is stable in the $NaHSO_4-CuCl_2$ system (see experimental part), and thus acids (V) are products of oxidative hydrolysis of amides (I).

By the action of equimolar amount of SPDS and $CuCl_2$, N,N-diethyl valeramide (If) is subjected to oxidative deethylation with the formation of N-ethyl valeramide (Ie) in a 94% yield, with 32% conversion of (If). γ-Valerolactone (IIIa) was not detected in the oxidation products of (If):

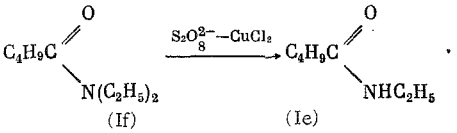


TABLE 1. Oxidation of Alkanoic Acid Amides (Ia-e) in $Na_2S_2O_8$–$CuCl_2$ and $Na_2S_2O_8$–NaCl Systems*

Amide	Conversion, %	Reaction products and yield, % (based on converted amide)		
		γ-lactone	δ-lactone	acid
		SPDS–$CuCl_2$ system (1:1)		
Valeramide (Ia)	98	(IIIa), 45	–	(Va), 55
Caproamide (Ib)	90	(IIIb), 30	(IVb), 15	(Vb), 50
Enanthoamide (Ic)	48†	(IIIb), 38	(IVb), 18	(Vb), 42
	92	(IIIc), 22	(IVc), 15	(Vc), 50
N-Methyl valeramide (Ib)	60	(IIIa), 2	–	(Va), 90
N-Ethyl valeramide (Ie)	48	(IIIa), 6	–	(Va) 83
		SPDS–NaCl system (1:2)		
Valeramide (Ia)	80	(IIIa), 42	–	(Va), 45
Caproamide (Ib)	68	(IIIb), 40	(IVb), 19	(Vb), 41

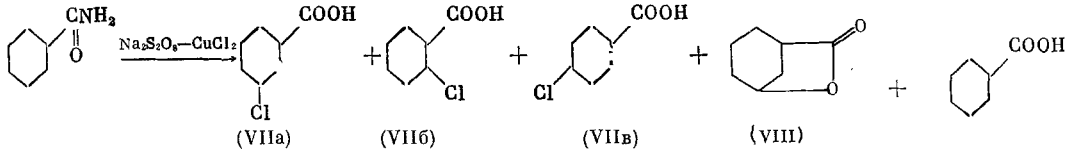
* 85-90°C, 10 h, 50 moles of amide (I), 50 mmoles of $Na_2S_2O_8$, 50 mmoles of $CuCl_2 \cdot 2H_2O$ or 100 mmoles of NaCl, 150 ml of water.
† Time of reaction 5 h.

TABLE 2. Oxidation of Alkanoic Acid Amides (Ia-c) in $Na_2S_2O_8$–NaCl–NaOH System*

Amide	Conversion, %	Reaction products and yield, % (based on converted amide)	
		amine	acid
Valeramide (Ia)	100	(IXa), 30	(Va), 55
Caproamide (Ib)	100	(IXb), 36	(Vb), 52
Enenthoamide (Ic)	100	(IXc), 35	(Vc), 49

* 85-90°C, 7 h, 50 mmoles of (I), 50 mmoles of $Na_2S_2O_8$, 100 mmoles of NaCl, 100 mmoles of NaOH, 200 ml of water.

By the action of equimolar amounts of SPDS and $CuCl_2$, cyclohexanecarboxamide (VI) converts into 3- and 2-chlorocyclohexanecarboxylic acids (VIIa, b), the lactone 6-oxabicyclo[3.2.1] octan-7-one (VIII), and cyclohexanecarboxylic acid, in a yield of 14.2, 10, and 40%, respectively, based on the converted amide (VI), at 80% conversion. 4-Chlorocyclohexanecarboxylic acid (VIIc) has also been identified in trace amounts (~1%):



(VIIa) (VIIб) (VIIв) (VIII)

The mechanism of formation of bicyclic lactone (VIII) is clearly similar to that proposed for lactones (IIIa-c). The conformational rigidity of (VIII) and steric unfavorableness of the transition states of the cyclization reaction favor direct chlorination of (VI) in the ring with the formation of (VIIb), and possibly partially also (VIIa, c).

In the SPDS–NaCl oxidation system in the presence of NaOH, the character of the oxidative transformations of amides (I) sharply changes, and they convert into primary amines (IX) with one less carbon atom than in the initial amides (I).

$$(Ia-c) \xrightarrow{Na_2S_2O_8-NaCl-NaOH} \underset{(IXa-c)}{R(CH_2)_4NH_2}$$

R=H (a), CH_3 (b), C_2H_5 (c).

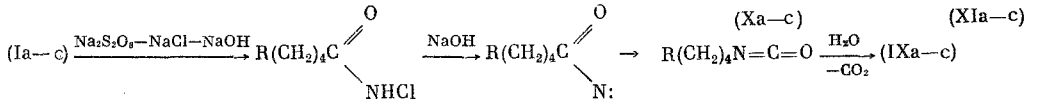
Under the reaction conditions, amides (Ia-c) in both the SPDS–$CuCl_2$ and SPDS–NaCl systems are hydrolyzed into acids (Va-c) (Table 2).

TABLE 3. Spectral Characteristics of Amides C_4H_9CONRR' (Id-f), Methyl Esters of 2-, 3-, and 4-Chlorocyclohexanecarboxylic Acids (VIIa-c), and 6-Oxabicyclo[3.2.1]octan-7-one (VIII)

Compound	IR spectrum (ν, cm^{-1})	PMR spectrum (δ, ppm)	Mass spectrum, m/z (relative intensity, %)
(Id) (R=H, R′=CH_3)	1640, 3070, 3350	0,90 t (3H, CH_3CH_2), 1,50 m (4H, CH_2CH_2), 2,20 t (2H, CH_2CO), 2,60 s (3H, CH_3), 7,25 s (1H, H)	73(100), 55(90), 41(80), 43(80), 60(60), 54(50), 56(50), 100(40), 83(20)
(Ie) (R=H, R′=C_2H_5)	1640, 3070, 3340	0,92 t (3H, $CH_3CH_2CH_2$), 1,24 m (3H, CH_3CH_2N), 1,60 m (4H, CH_3CH_2), 2,23 t (2H, CH_2CO), 3,31 q (2H, CH_2N), 5,8 s (1H, NH)	87(100), 72(80), 44(40), 57(25), 41(21), 100(20), 85(15), 114(10), 129(M^+, 7), 128(6)
(If) (R=R′= $=C_2H_5$)	1640	0,9 t (3H, $CH_3CH_2CH_2$), 1,24 m (6H, CH_3CH_2N), 1,62 m (4H, $CH_3CH_2CH_2$), 2,20 t (2H, CH_2CO), 3,24 q (4H, CH_2N)	58(100), 44(90), 41(70), 57(60), 72(50), 56(40), 46(400), 100(40), 115(30), 128(30), 157(M^+, 10)
(VIIa), Methyl ester		3,73 m (1H, CHCl), 1,52 m (8H, CH_2), 2,92 m (1H, CHCO), 3,59 s (3H, CH_3O)	81(100), 80(60), 108(40), 41(15), 79(15), 109(10), 141(10), 140(7), 145(7)
(VIIb), Methyl ester			81(100), 121(20), 141(15), 117(15), 79(15), 80(10), 140(7), 123(7), 53(6), 105(5)
(VIIc), Methyl ester			81(100), 108(90), 80(90), 79(20), 109(15), 140(9), 141(8), 145(7), 147(3)
(VIII)	1770		67(100), 54(80), 55(50), 84(50), 82(50), 83(40), 70(25), 126(M^+, 2) *

*Similar as in [24].

The most probable mechanism for the formation of amines (IXa-c) is that including the oxidation of amides (Ia-c) or the amidyl radicals (IIa-c) into N-chloroamides (Xa-c), their dehydrochlorination into nitrenes (XIa-c), and a Hoffmann type rearrangement [15] of the latter into (IXa-c):



Thus, introduction of NaOH to the SPDS–NaCl oxidation system made it possible to entirely change the direction of the reaction of amides (I).

EXPERIMENTAL

The GLC analysis was carried out on an LKhM-8MD chromatograph with a flame-ionization detector in a N_2 current, using the following columns (stainless steel): 3000 × 3 mm with 10% Carbowax 20 M, treated by H_3PO_4 [16], on Celite-545 (52-60 mesh); 1700 × 3 mm with PEGS on Chromosorb G, treated with dimethyldichlorosilane; 2000 × 4 mm with 10% Carbowax 20 M, treated with Na_3PO_4, on Celite-545 (52-60 mesh). The PMR spectra of the solutions in CD_3CN in CCl_4 were measured on a Tesla BS-497 spectrometer (100 MHz) with reference to HMDS. The mass spectra were run on a Varian MAT CH-6 apparatus with direct introduction of the sample into the ion source, and with energy of ionizing electrons of 70 eV. The IR spectra were obtained on a Perkin–Elmer apparatus in a thin layer and in solution in CCl_4. The chromato-mass-spectrometric analysis was carried out on a Varian MAT CH-111 (Gnom) apparatus with chromatographic introduction of the sample into the ion source, with energy of the ionizing electrons of 80 eV.

The oxidizing agent, AR grade sodium peroxydisulfate (SPDS), and cp grade $CuCl_2$, NaCl, and NaOH brand were used without further purification. The water was distilled once. The amides of valeric, caproic, and enanthoic acids (Ia-c) and cyclohexanecarboxamide (VI) were obtained by the reaction of the acid chlorides of the corresponding alkanoic acids with NH_4OH [17] and purified by recrystallization from ethanol. N-Methyl, N-ethyl and N-diethyl valeramides (Id-f) were obtained by the reaction of valeryl chloride with CH_3NH_2, $C_2H_5NH_2$,

and $(C_2H_5)_2NH$, respectively [17], and purified by distillation in vacuo. The melting and boiling points of the amides obtained correspond to those in the literature [18-22]. The spectral characteristics of amides (Id-f) are listed in Table 3.

Oxidation of Alkanoic Acid Amides (Ia-c) in SPDS–$CuCl_2$ and SPDS–NaCl Systems. A solution of 50 mmoles of SPDS in 50 ml of water was added dropwise (2.5-3 h) with vigorous stirring, at 85-90°C, to a mixture of 50 mmoles of amide (I) and 50 mmoles of $CuCl_2$ or 100 mmoles of NaCl in 100 ml of water. The mixture was stirred at 85-90°C for another 7 h, and then cooled. The precipitate was filtered, and washed with cold water and a small amount of ether. The precipitate was the unreacted amide (I). The filtrate was extracted with ether (3 × 100 ml), and chloroform (2 × 100 ml), and the extract was dried over $MgSO_4$ and evaporated. The residue was analyzed by GLC using a standard for the determination of lactones and acids, an acid with one less carbon atom. During the determination of unreacted amide (I) in the residue, the lower homologous amide was used as standard. To isolate lactones (III) and (IV), the residue was neutralized by a saturated solution of Na_2CO_3, extracted with ether (3 × 100 ml), dried over $MgSO_4$, and evaporated, and the residue was distilled in vacuo. The properties and the spectral characteristics of lactones (IIIa-c) and (IVb, c) are identical to those given in our previous paper [12].

Oxidation of Substituted Valeramides (Id-f) in SPDS–$CuCl_2$ System. The oxidation was carried out by the above procedure. The reaction mixture was further extracted by ether, the extract was dried over $MgSO_4$, and evaporated, and the residue was analyzed by GLC and then fractionated in vacuo.

Attempt to Hydrolyze Valeramide (Ia) in $NaHSO_4$–$CuCl_2$ System. A mixture of 100 mmoles of (Ia), 200 mmoles of $NaHSO_4$, and 100 mmoles of $CuCl_2$ in 250 ml of water was heated for 10 h at 85-90°C with vigorous stirring. It was then cooled, and extracted with ether (3 × 100 ml). The extract was dried over $MgSO_4$, and evaporated. In the residue 98 mmoles of unreacted (Ia) were found.

Oxidation of Cyclohexanecarboxamide (VI). A solution of 50 mmoles of $Na_2S_2O_8$ in 50 ml of water was added dropwise (2.5-3 h), at 85-90°C, with vigorous stirring, to a mixture of 50 mmoles of (VI) and 50 mmoles of $CuCl_2 \cdot 2H_2O$ in 100 ml of water. Stirring was continued for another 7 h at 85-90°C, and then the mixture was cooled, extracted with an ether–acetonitrile mixture (4:1) (4 × 100 ml), then with ether (2 × 100 ml) and chloroform (2 × 100 ml). The extracts were combined, dried over $MgSO_4$, and evaporated, and in the residue the amount of unreacted (VI) (20%) was determined by GLC (standard – caproamide). Part of the residue was methylated by an excess of CH_2N_2 [23] and analyzed by GLC (internal standard – methyl valerate) and chromato-mass-spectrometrically. In the methylation product, the methyl esters of cyclohexanecarboxylic and 2-, 3-, and 4-chlorocyclohexanecarboxylic acids (VIIa-c) were detected in a ratio of 40:2:14:10. Part of the unmethylated residue was neutralized by a saturated solution of $NaHCO_3$, and extracted with ether (4 × 20 ml). The extract was dried over $MgSO_4$, and evaporated in vacuo. From the residue, lactone (VIII) was obtained. The structure of esters (VIIa-c) and lactone (VIII) was confirmed by spectral data. The spectral characteristics of (VIIa-c) and (VIII) are listed in Table 3.

Oxidation of Alkanoic Acid Amides (Ia-c) in the SPD–NaCl–NaOH System. Solutions of 100 mmoles of NaOH in 50 ml of water and 50 mmoles of SPDS in 50 ml of water were added dropwise from two separatory funnels at 85-90°C in the course of 2 h, with stirring, to a mixture of 50 mmoles of (Ia-c) and 100 mmoles of NaCl in 100 ml of water; the solution of the alkali was added twice as rapidly as the $Na_2S_2O_8$ solution. The mixture was heated for another 5 h at 85-90°C, cooled, and extracted with ether (3 × 50 ml). The extract was dried over $MgSO_4$, and evaporated. By distillation, amines (IXa-c) were isolated, which in their physical properties and spectral data were identical to actual samples. The aqueous layer was acidified by 1 N HCl, and extracted by ether (3 × 100 ml). The extract was dried over $MgSO_4$ and evaporated. Distillation in vacuo gave the alkanoic acids (Va-c) identical to actual samples.

CONCLUSIONS

1. Unsubstituted alkanoic acid amides $R(CH_2)_4CONH_2$ (R = H or alkyl) convert in $Na_2S_2O_8$–$CuCl_2$ and $Na_2S_2O_8$–NaCl oxidation systems via intermediate amidyl radicals $R(CH_2)_4C(O)NH$ into γ- and δ-lactones with a considerable predominance of γ-lactones.

2. Monoalkyl amides of alkanoic acids are lactonized to an inappreciable extent by the action of the $S_2O_8^{2-}$–Cl^- system; N,N-diethyl valeramide undergoes oxidative dealkylation into N-alkyl valeramide.

3. As the result of an oxidative, Hoffmann type rearrangement in a $Na_2S_2O_8$–NaCl–NaOH system, unsubstituted alkanoic acid amides convert into amines containing one carbon atom less than the initial amides.

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OXIDATION OF PRIMARY AMINES BY POTASSIUM FERRICYANIDE

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The use of organic amines and aminyl radicals has recently stimulated the development of research on their oxidative transformations during photolysis [1], in electrochemical reactions [2], in reactions with free radicals [3], and in redox systems containing elements with variable valence [4]. The reactions of secondary and tertiary amines were studied in the greatest detail, while those of primary amines characterized by high reactivity and lability in oxidation systems have been studied less.

We studied the oxidative transformations of primary n-alkyl-, sec-alkyl-, and tert-alkylamines by the action of a mild oxidation agent, $K_3Fe(CN)_6$, in an alkaline medium, already used for the oxidative dealkylation of primary and secondary amines [5, 6].

By the action of the $K_3Fe(CN)_6$–NaOH system in aqueous solutions at 70-80°C and with equimolar amounts of the reagents, primary n-alkylamines (Ia-c) are oxidized into nitriles (IIa-c) (Table 1):

$$\underset{\text{(Ia–c)}}{RCH_2NH_2} + 4Fe(CN)_6^{3-} + 4OH^- \rightarrow \underset{\text{(IIa–c)}}{RC\equiv N} + 4Fe(CN)_6^{4-} + 4H_2O$$

$$R = C_5H_{11}\text{ (a)},\ C_6H_{13}\text{ (b)},\ C_7H_{15}\text{ (c)}.$$

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