AcOH system changes the course of the reaction and leads to the formation of $CH_3COCHClCOCH_3$, which can be oxidatively added to 1-hexene to give 3-acetyl-3,5-dichloro-2-nonanone.

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DIRECT OXIDATION OF ALKANOIC ACIDS TO LACTONES

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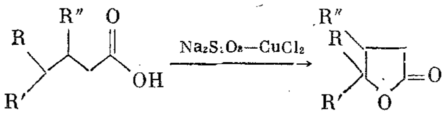
The reaction of carboxylic acids with oxidizing agents, for example, compounds of Pb(IV), Ag(II), Co(III), and of other metals of variable valence [1-3], or with oxidizing systems such as $S_2O_8^{2-}$—Ag^+, $S_2O_8^{2-}$—Ag^+—Cu^{2+}, etc. [4, 5], leads to their oxidative decarboxylation and generation of free radicals. The retention of the carboxylic grouping of the acids in reactions of this type was observed only during their conversion into lactones, and only in two cases: by the action of peroxydisulfuryl difluoride [6], and in the oxidation of alkanoic acids with a tertiary γ-C atom by $KMnO_4$ in an alkaline medium [7].

We found that by using the sodium peroxydisulfate—copper chloride oxidizing system with equimolar amounts of the reagents, and by carrying out the reaction in water at 80-90°C, alkanoic acids (Ia-g) very selectively convert into γ-lactones (IIa-g)*



(Ia— g) (IIa—g)

R, R′, R″ = H, H, H (a); CH_3, H, H (b); C_2H_5, H, H (c); C_3H_7, H, H (d); C_4H_9, H, H (e); H, H, CH_3 (f); C_4H_9, C_2H_5, H (g).

In the case of acids (Ib-e), not only γ-lactones (IIb-e) are formed, but also the corresponding δ-lactones: δ-valerolactone (IIIb), δ-methyl-, δ-ethyl-, and δ-propyl-δ-valerolactones (IIIc-e). Acids (Ia-g) partially undergo decarboxylation (Table 1).

The yield of γ-lactones (II) obtained from the n-alkanoic acids studied increases on transition from (Ia) to (Ib), and then decreases in the series of acids (Ic-e). 3-Methylbutanoic acid (If) is oxidized to β-methyl-γ-butyrolactone (IIf) less effectively than acid

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*Preliminary communication, see [8].

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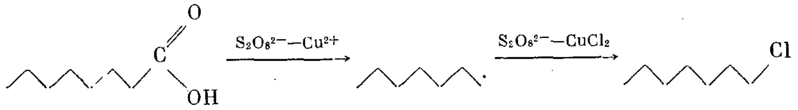
(Ib) into γ-methyl-γ-butyrolactone (IIb). In the ability to be oxidized to γ-lactone (IIg), 4-ethyloctanoic acids (Ig) is inferior only to acid (Ib). Hence, the selectivity of oxidation of (I) into (II) is largely influenced by the substituent at the C^4 atom of the acids: the contribution of the oxidative lactonization reaction is lowest when this atom is primary in (Ia) and (If), and appreciably increases when this atom is secondary in (Ib–e) or tertiary in (Ig).

With increase in the yield of lactones (II) and (III), the contribution of the oxidative decarboxylation reaction of acids (I) with the formation of CO_2 increases symbatically. In the oxidation of acid (Ie), 1-chloroheptane was also identified, which is a product of a reaction between the heptyl radical, formed during the decarboxylation of (If), and the $Na_2S_2O_8$–$CuCl_2$ oxidizing system

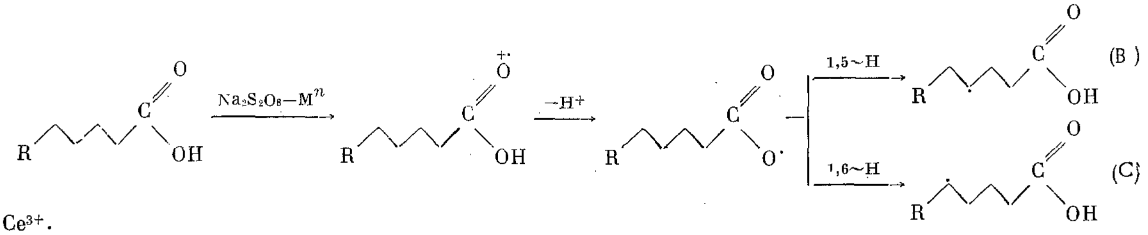


To find the mechanism of the oxidative lactonization of acids (I) into γ-lactones (II), and also to find other effective oxidizing systems, we studied the oxidation of acid (Ib) by the action of $Na_2S_2O_8$ in the presence of different salts (Table 2). From the data obtained it follows that acid (Ib) is most effectively and selectively oxidized to lactone (IIb) by the action of the $Na_2S_2O_8$–$CuCl_2$, $Na_2S_2O_8$–$Ce_2(SO_4)_3$, and $Na_2S_2O_8$–NaCl systems. Thus, increase in the amount of $CuCl_2$ leads to decrease in the selectivity of formation of (IIb), and increase in the contribution of the decarboxylation reaction. In the presence of Fe(II), Fe(III), and Co(II) compounds, the selectivity of oxidation of (Ib) to (IIb) markedly decreases. By the action of $Na_2S_2O_8$–NaCl system (1:2), acid (Ic) is 25% converted to lactones (IIc) and (IIIc) in a yield of 60 and 20%, respectively.

It should be noted that acid (Ib) is inert to oxidation, and does not lactonize by the action of $KMnO_4$–NaOH system, which was previously used for the preparation of lactones from substituted alkanoic acids with tertiary γ-carbon atom [7].

The results show that the ability to oxidize alkanoic acids into γ-lactones is a specific property of the $Na_2S_2O_8$–$CuCl_2$ and $N_2S_2O_8$–NaCl, and also $Na_2S_2O_8$–$Ce_2(SO_4)_3$ systems.

We believe that the mechanism of formation of lactones includes one-electron oxidation of the alkanoic acids by the action of the $Na_2S_2O_8$–M^nX_n systems to acyloxyl radicals (A), which are stabilized by coordination with a metal of variable valency, and can undergo an intramolecular rearrangement by the 1,5- and 1,6-migration of the H atom, to form 3- and 4-carboxyalkyl radicals (B) and (C)



M = Cu^{2+}, Ce^{3+}.

By the oxidation of radicals (B) and (C) into the corresponding carbonium ions, and cyclization of the latter, or by homolytic cyclization of (B) and (C) by intramolecular addition to the C=O group and subsequent oxidation ([9]), γ- and δ-lactones, respectively, are formed.

In the $Na_2S_2O_8$–$CuCl_2$ and $Na_2S_2O_8$–NaCl systems, acylhypochlorites $RCH_2CH_2(CH_2)C(O)OCl$ can also be formed (in analogy with [10]). During the homolytic dissociation of these acylhypochlorites at the O–Cl bond, radicals (A) are formed, which after rearrangement into (B) and (C), convert into γ- and δ-lactones.

An alternative reaction scheme is that with the formation from $Na_2S_2O_8$, thermally or Cu^{2+} or Ce^{3+}-catalyzed, of sulfate anion radicals, and in the $Na_2S_2O_8$–$CuCl_2$ and $Na_2S_2O_8$–NaCl systems, also of Cl radicals, which nonselectively abstract H atoms from the alkyl fragment of the alkanoic acid with formation of carboxy-substituted alkyl radicals

TABLE 1. Oxidation of Alkanoic Acids (Ia-g) in the $Na_2S_2O_8$–$CuCl_2$ System*

Acid	Conversion, %	Reaction products and their yield, % based on reacted acid		
		CO_2	γ-lactone	δ-lactone
Butanoic (Ia)	44	36	(IIa), 34	–
Pentanoic (Ib)	45	11	(IIb), 77	(IIIb), 9
Hexanoic (Ic)	45	13	(IIc), 60	(IIIc), 22
Heptanoic (Id)	40	25	(IId), 45	(IIId), 17
Octanoic (Ie)	31	30†	(IIe), 30	(IIIe), 12
3-Methylbutanoic (If)	30	47	(IIf), 50	–
4-Ethyloctanoic (Ig)	22	27	(IIg), 64	–

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*85-90°C, 5 h, 100 mmoles of (I), 100 mmoles of $Na_2S_2O_8$, 100 mmoles of $CuCl_2 \cdot 2H_2O$, 110 ml of water.

†1-Chloroheptane, obtained in a yield of 23% based on reacted (Ie), was also identified.

TABLE 2. Oxidation of Valeric Acid (Ib) by $Na_2S_2O_8$–M^nX_n Systems

M^nX_n	Molar ratio $Na_2S_2O_8:M^nX_n$	Conversion of (Ib), %	Reaction products and their yield, % based on reacted (Ib)		
			CO_2	γ-lactone (IIb)	other products
$CuCl_2$	1 : 1	45	11	77	(IIIb), 9
$CuCl_2$	1 : 2	52	40	38	(IIIb), 4
$CuSO_4$	1 : 1	8	60	Traces	–
$CuCl_2$–$FeSO_4$	1 : 1 : 0,1	55	†	55	(IIIb), 4
$FeCl_2$	1 : 1	44	†	40	(IIIb), 4
$FeCl_3$	1 : 1	51	28	47	(IIIb), 8
$CoCl_2$	1 : 1	52	†	30	(IIIb), traces
$Ce_2(SO_4)_3$	1 : 1	44	14	80	–
$NiCl_2$	1 : 1	65	8	55	$Cl(CH_2)_4COOH$ (IVa), 14 $C_2H_5CHClCH_2COOH$ (IVb), 15
NaCl	1 : 2	37	8	76	(IIIb), 8

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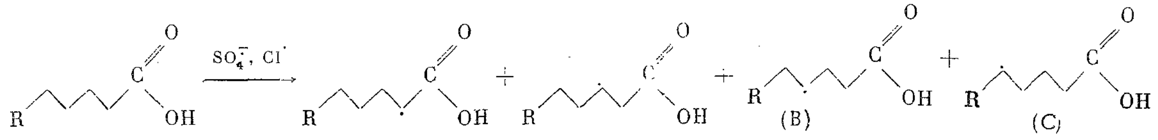
*85-90°C, 5 h, 100 mmoles of acid (Ib), 100 mmoles of $Na_2S_2O_8$, 110 ml of water.

†Not determined.



From radicals (B) and (C) γ- and δ-lactones are formed.

To more precisely clarify the proposed oxidation mechanism of acids (I) into lactones (II) and (III), we studied the reaction of methyl valerate (V) with the $Na_2S_2O_8$–$CuCl_2$ system. Under the conditions when there is practically no hydrolysis of ester (V) to acid (Ib), lactones (IIb) and (IIIb) are not formed, and the conversion of (V) is 8%. Under similar reaction conditions, the conversion of (Ib) is 29%, and the yield of (IIb) is 24%. Obviously, the $CH_3CH_2CH_2CH_2$ fragments in (Ib) and (V) practically do not differ in reactivity with respect to $SO_4^{-\bullet}$ and Cl• radicals, and radicals $CH_3\dot{C}HCH_2CH_2C(O)OCH_3$, analogs of (B), formed from ester (V), can also in similar $Na_2S_2O_8$-containing oxidizing systems be oxidized to a γ-lactone (IIb) (cf. [11]). We believe that due to the observed inertness of (V) to the action of the $Na_2S_2O_8$–$CuCl_2$ system, the mechanism of formation of (IIb) as the result of an initial attack of $SO_4^{-\bullet}$ or Cl• radicals at the alkyl fragment of (Ib) is not possible. This scheme is also improbable because of the high regioselectivity of formation of γ-lactones (II), compared with δ-lactones (III). Since the abstraction of H atoms by electrophilic radicals, in general, proceeds preferentially at the (ω = 1)-C atom of the alkyl residue [12], in the reaction of acid (Ic) with electrophilic $SO_4^{-\bullet}$ and Cl•, obviously, a radical $CH_3\dot{C}H(CH_2)_3COOH$, a predecessor of δ-lactone (IIIc), stabilized by the reaction with the CH_3 group, should mainly be formed; this radical is, however, obtained in a much smaller amount than the corresponding γ-lactone (IIc).

We thus believe that the most probable mechanism is the one whose key stage is the generation of O-centered acyloxyl radicals and their rearrangement with 1,5- and 1,6-migration of the H atom into the 3- and 4-carboxyalkyl radicals. In accordance with the general principles of rearrangements in radical systems, the 1,5-migration predominates over the 1,6-migration [13], which also determines the regioselective formation of γ-lactones (II). According to this scheme, the maximal yield of γ-lactone is observed in the oxidation of acid (Ib) when 1-methyl-3-carboxypropyl radical is formed, which is stabilized by conjugation with the CH_3 group. The low yield of (IIa) from (Ia) is due to unfavorable rearrangement of radical $CH_3CH_2CH_2C(O)O^{\cdot}$ into a primary 3-carboxypropyl radical.

In contrast to the $Na_2S_2O_8$–$CuCl_2$, $Na_2S_2O_8$–NaCl, and $Na_2S_2O_8$–$Ce_2(SO_4)_3$ systems, in the oxidation of acid (Ib) in the $Na_2S_2O_8$–$NiCl_2$ system, according to identification of chloroacids (IVa, b), a definite contribution is apparently introduced by a mechanism including an initial abstraction of the H atom from the alkyl fragment.

EXPERIMENTAL

The GLC analysis was carried out on an LKhM-8MD chromatograph with a flame-ionization detector in an N_2 current, using a 300 × 0.3 cm column (stainless-steel) with a 10% Carbowax 20 M on a Celite-545 (52-60 mesh) pretreated by H_3PO_4 [14]. The PMR spectra of the solutions 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 ionic source, and with an energy of the ionizing electrons of 70 eV. The IR spectra were obtained on a Perkin–Elmer apparatus in a thin layer and in CCl_4 solution. The chromato-mass-spectrometric analysis was carried out on a Varian MAT CH-111 (Gnom) apparatus, with chromatographic introduction of the sample into the ionic source, and with energy of ionizing electrons of 80 eV. AR grade $Na_2S_2O_8$ was used as the oxidizing agent, and pure grade $CuCl_2$, $FeCl_2$, $FeSO_4$, $FeCl_3$, $CoCl_2$, $NiCl_2$, $Ce_2(SO_4)_3$, NaCl, and $KMnO_4$ preparations were used without additional purification. Pure grade alkanoic acids (Ia-g) were purified by distillation in vacuo. Once-distilled water was used.

Oxidation of Alkanoic Acids (Ia-g). A solution of 100 mmoles of $Na_2S_2O_8$ in 50 ml of water was added dropwise at 85-90°C (0.5-1 h) to a mixture of 100 mmoles of the alkanoic acid and other components (in amounts indicated in Tables 1 and 2) in 90 ml of water, and the mixture was heated for 4-4.5 h at 85-90°C with vigorous stirring. The mixture was cooled and extracted by ether (3 × 100 ml). The ether extract was dried over $MgSO_4$, and evaporated, and the residue was analyzed by GLC, using propionic acid as a standard. To isolate lactones (II) and (III), the residue was neutralized by a saturated solution of Na_2CO_3, and extracted by ether (3 × 100 ml). The ether extract was dried over $MgSO_4$, evaporated, and distilled in vacuo. The properties and spectral characteristics of the reaction products are given in Table 3.

Reaction between Methyl Valerate (V) and 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 in the course of 30 min at 85-90°C to a mixture of 100 mmoles of (V) and 100 mmoles of $CuCl_2 \cdot 2H_2O$ in 90 ml of water. The mixture was heated at 85-90°C for 90 min, cooled, and extracted by ether (3 × 100 ml). The extract was dried over $MgSO_4$, and evaporated, and by GLC the residue was found to contain 92 mmoles of ester (V).

Oxidation of Valeric Acid (Ib) by the $Na_2S_2O_8$–$NiCl_2$ System. A solution of 100 mmoles of $Na_2S_2O_8$ in 50 ml of water was added dropwise at 85-90°C in the course of 1 h to a mixture of 100 mmoles of (Ib) and 100 mmoles of $NiCl_2$ in 90 ml of water. The mixture was heated for 4 h at 85-90°C with vigorous stirring, cooled, treated with a saturated solution of Na_2CO_3 to pH 9, and extracted by ether (3 × 30 ml). The extract was dried over $MgSO_4$, evaporated, and in the residue the amount of γ-valerolactone (IIb) was determined by GLC. The aqueous phase was acidified to pH 3-4, and extracted by ether (3 × 50 ml). The extract was dried over $MgSO_4$, evaporated, and the residue was distilled in vacuo to yield 3- and 5-chlorovaleric acids (IVa, b).

Reaction between (Ib) and the $KMnO_4$–NaOH System. A solution of 100 mmoles of $KMnO_4$ in 100 ml of water was added at 85-90°C, with stirring, in the course of 1 h to a mixture of 100 mmoles of (Ib) and 120 mmoles of NaOH in 150 ml of water. The mixture was heated for 4 h at 85-90°C, cooled, treated with Na_2CO_3 to the discoloration of the solution, acidified to pH 3-4 by dilute H_2SO_4, and extracted by ether (3 × 100 ml). The ether extract was dried over $MgSO_4$, and evaporated, and the residue was analyzed by GLC and spectral methods. Ninety five mmoles of unreacted acid (Ib) were found.

TABLE 3. Physicochemical Properties and **Spectral** Characteristics of Lactones (II), (III), and Chloro-Acids (IV)

Compound	bp, °C (p, mm Hg)	Empirical formula	*Found/Calculated, %*		IR spectrum (ν, cm^{-1})	PMR spectrum (δ, ppm)	Mass spectrum, m/z (relative intensity, %)
			C	H			
(IIa)	100(27)	$C_4H_6O_2$	55,69 55,81	6,89 7,00	1780	2,10 m (2H, $CH_2\underline{CH_2}CH_2$), 2,35 t (2H, CH_2CO) 4,28 t (2H, CH_2O)	–
(IIb)	105–107(26)	$C_5H_8O_2$	59,53 60,00	7,72 8,00	1780	1,25 d (3H, CH_3) 2,25 m (4H, CH_2CH_2) 4,50 m (1H, CHO)	Similar to that in [15]
(IIc)	102–104(20)	$C_6H_{10}O_2$	62,50 63,16	8,58 8,62	1770	1,10 t (3H, CH_3) 1,70 m (2H, $CH_3\underline{CH_2}CH$) 2,25 m (4H, CH_2CH_2) 4,50 m (1H, CHO)	»
(IId)	109(16)	$C_7H_{12}O_2$	65,16 65,62	9,15 9,47	1770	1,05 t (3H, CH_3) 1,65m (4H, $CH_3CH_2CH_2$) 2,25 m(4H, CH_2CH_2) 4,50 m(1H, CHO)	»
(IIe)	115(36)	$C_5H_8O_2$	59,64 60,00	7,85 8,00	1780	1,17d (3H, CH_3) 2,05d and 2,43 d (2H, CH_2CO) 2,55 m(1H, CH) 3,75 m and 4,22 m(2H, CH_2O)	41(100), 42(95), 56(65), 39(50), 55(20), 57(7), 100(M^+, 7) 85(2)
(IIf)	104(10)	$C_{10}H_{18}O_2$	70,28 70,59	10,46 10,56	1775	0,90t (6H, CH_3) 1,25–1,8 m(10 H) 2,40 t (2H, CH_2CO)	29(100), 41(95), 57(90), 113(90), 55(86), 43(40), 141(40), 56(35), 85(25), 69(25)
(IIIb)	–	–	–	–	1730	–	Similar to that in [15]
(IIIc)	–	–	–	–	1725	–	»
(IIId)	–	–	–	–	1730	–	»
(IVa)	149–152(13)	$C_5H_9ClO_2$	43,82 43,95	6,48 6,58	790, 1710	2,00 d (4H, CH_2CH_2) 3,60 t (2H, CH_2Cl) 2,30 t (2H, CH_2CO)	73(100), 55(90), 41(80) 43(70), 60(70), 56(60). 100(60), 54(50), 42(50)
(IVb)	143–145(15)	$C_5H_9ClO_2$	43,82 43,95	6,48 6,58	790, 1710	1,08 t (3H, CH_3) 2,05m (2H, CH_3CH_2) 4,10 m (1H, CHCl)	60(100), 42(75), 41(70), 55(65), 56(60), 43(60), 73(30), 100(20)

CONCLUSIONS

1. By the action of the $Na_2S_2O_8$—$CuCl_2$ system, alkanoic acids $RR'CHCH(R'')CH_2COOH$ convert into γ-lactones $RR'\overline{C CH(R'')CH_2C(O)O}$ with high regioselectivity.

2. A reaction mechanism was proposed, including the formation from alkanoic acids of alkylcarbonyloxyl radicals, their rearrangement with 1,5-migration of the H atom into 3-carboxylalkyl radicals, and oxidative cyclization of the latter into γ-lactones.

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CYCLOKETONIZATION AND LINEAR POLYKETONIZATION OF α,ω-DICARBOXYLIC ACIDS.

COMMUNICATION 7. PREPARATION AND REACTIONS OF ZINC SALTS OF UNBRANCHED DICARBOXYLIC ACIDS

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V. N. Lutovinova, and A. L. Liberman

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Ketonization of Mn and Cd salts of dicarboxylic acids of composition C_6 and higher is a complex process including two main reactions, namely, cycloketonization and linear polyketonization, and two secondary reactions, namely, crotonic condensation of cyclopentanone and cyclohexanone on MnO [1, 2] and crotonic condensation of the polyketodicarboxylic acids formed during linear polyketonization in the presence of CdO [2-4]. The investigation of the ketonization of Zn salts of dicarboxylic acids was intended to elucidate the generalness and specific differences of the mechanism of ketonization of dicarboxylic acid salts with different cations and also the possibility of elucidating the yields of cycloalkanones due to inhibition of linear polyketonization and secondary reactions. The data [5-7] on the ketonization of Zn salts of dicarboxylic acids are very contradictory.

In the present paper, we have studied the ketonization of Zn salts of adipic, pimelic, suberic, azelaic, and sebacic acids. The reaction was carried out according to [8] with due regard for the improvements mentioned in [4] and [9]. To obtain Zn salts of dicarboxylic

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