## LITERATURE CITED

- Zh. A. Krasnaya and T. S. Stytsenko, Izv. Akad. Nauk SSSR, Ser. Khim., 1060 (1981); 855 (1983).
- É. B. Lifshits, I. I. Levkoev, L. M. Yagupol'skii, and N. S. Barvyn', Zh. Nauchn. Prikl. Foto-Kinemat., No. 11, 175 (1966).

OXIDATIVE  $\delta$ -CHLORINATION OF ALIPHATIC ALCOHOLS IN THE SYSTEM LEAD TETRAACETATE—METAL CHLORIDE\*

N. I. Kapustina, A. V. Lisitsyn, and G. I. Nikishin UDC 542.943.7+542.944:547.26

Alkoxy radicals containing at least four carbon atoms in the chain are capable of undergoing rearrangement in solution with 1,5-H and 1,6-H migration  $(K_{1,5-H}/K_{1,6-H} \approx 10 \ [2])$ . Precursors of the alkoxy radicals may be alkanols, alkyl hypochlorites, alkyl nitrites, or alkyl hydroperoxides [3].

Functionalization of carbon-centered radicals at the radical center by external reagents gives functionally substituted alcohols, while intramolecular functionalization affords fivemembered cyclic ethers. For example, oxidation of aliphatic alcohols with five or more carbon atoms by lead tetraacetate (LTA) gives mostly  $\alpha$ -alkyltetrahydrofurans [4], whereas photochemical decomposition of alkyl hypochlorites and reduction of alkyl hydroperoxides by Fe(II) chloride produces mainly the  $\delta$ -chloroalkanols [5, 6].

We have now examined the oxidation of primary, secondary, and tertiary  $C_5-C_{11}$  aliphatic alcohols (I) in the system LTA-metal chloride, in order to determine the influence of the structure of the alcohol on the course of oxidation, and to synthesize  $\delta$ -chloroalkanols. The reaction was carried out in benzene at 80°C. The principal product of the reaction was found to be the  $\delta$ -chloroalkanol (II). To a small extent, (I) was oxidized to the  $\varepsilon$ -chloroalkanol (III), the carbonyl compound (IV), and the substituted alkyltetrahydrofuran (V), the overall yield of (III)-(IV) being 5-10%:



R<sup>1</sup>, R<sup>2</sup>, R<sup>3</sup>, R<sup>4</sup> and R<sup>5</sup>=H, alkyl C<sub>1</sub>--C<sub>5</sub>, C<sub>6</sub>H<sub>5</sub>; M = Li, Na, K, Mg, Ca; n = 1, 2.

EPR was used to detect the radical steps in the oxidation of alcohols. It was found that the reaction between LTA and 1-hexanol in benzene in the presence of C-phenyl-N-tertbutylnitrone (PBN) gave rise to a triplet of doublets in the EPR spectrum ( $a_{\rm N}$  = 13.8;  $a_{\beta}^{\rm H}$  = 1.9 Oe) for the spin adduct (SA) of the alkoxy radicals (A) with PBN. Radicals of type (A) were also detected in the reaction of 1-hexanol with the system LTA-LiCl in the presence of PBN. The formation of carbon-centered secondary radicals was observed in the oxidation of 1-hexanol with LTA in the presence of 2-methyl-2-nitrosopropane (MNP). The EPR spectrum of

\*For previous communication, see [1].

N. D. Zelinskii Institute of Organic Chemistry, Academy of Sciences of the USSR, Moscow. Translated from Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No. 1, pp. 98-105, January, 1989. Original article submitted September 11, 1987.

Experi- ment No.	Alcohol:LTA:LiCl ratio	Product, yield (as % on LTA)*						
		(II)	(III)	(IV)	(V)			
1 2 3 4 5 6	1:1:1 1:1:4 1:2:4 2:1:1 2:1:2 2:1:2 2:1:4	33 46 30 36 56 67	- 3 - 2 2 3	13 4 4 7 3 4	22 			

TABLE 1. Oxidative Chlorination of 1-Hexanol in the System LTA-LiCl (80°C, 5-10 min, benzene 20 ml, conversion of LTA 100%)

\*Here and subsequently, calculated on the basis of one mole of LTA per mole of product.

the SA of these radicals also consists of a triplet of doublets ( $a_{\rm N}$  = 13.5;  $a_{\beta}^{\rm H}$  = 1.6 Oe) due to splitting at the nitrogen of the nitroso-group and the  $\beta$ -protons of the captured radical. Since, in preparative experiments on the oxidation of 1-hexanol in the system LTA-LiCl, only  $\delta$ - and  $\epsilon$ -chloro-1-alkanols were obtained, we assume that this EPR spectrum is due to adducts of MNP with the radicals CH<sub>3</sub>CH<sub>2</sub>CHCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH (B) and CH<sub>3</sub>CH(CH<sub>2</sub>)<sub>3</sub>CH<sub>2</sub>OH (C).

Hence, the oxidative chlorination involves the generation of alkoxy radicals (A), their rearrangement with 1,5- and 1,6-migration of hydrogen to give the carbon-centered radicals (B) and (C), and oxidation of the latter with ligand transfer to the  $\delta$ -chloroalkanol (II) and the  $\epsilon$ -chloroalkanol (III).



The energy advantage of the six-membered cyclic transition state in 1,5-H migration over the seven-membered state in 1,6-H migration in radical (A) determines the regioselectivity of the  $\delta$ -functionalization of aliphatic alcohols [7].

The effects of the amounts of reactants used on the ratios of the products have been examined in the oxidation of 1-hexanol by the system LTA--LiCl (Table 1). As the amounts of alcohol and LiCl were increased relative to the LTA, the yields of 4-chloro-1-hexanol improved, the best results being obtained with an alcohol:LTA:LiCl ratio of 2:1:4. These conditions were used in the oxidations shown in Table 2 for primary, secondary, and tertiary alkanols.

Unsubstituted 1-alkanols produced the  $\delta$ -chloroalkanols in 55-65% yields (calculated on one mole of LTA per mole of product). A CH<sub>3</sub> group in the  $\delta$ -position of the 1-alkanol has a screening effect on the oxidation of the tertiary C-centered radical, hindering the formation of the  $\delta$ -chloroalcohol, with the simultaneous formation of increased amounts of the cyclic ether (V) (experiment 5). The CH<sub>3</sub> group in the  $\gamma$ -position in 3-methyl-1-pentanol has no effect on the oxidative chlorination, 3-methyl-4-chloro-1-pentanol being formed in two stereoisomeric forms in a ratio of 0.9:1.1 (experiment 6).

The oxidation of secondary unbranched  $C_6-C_8$  alcohols also affords mainly the  $\delta$ -chloroalkanols (experiments 7 and 8). However, 6-undecanol gives, in addition to 3-chloro-6-undecanol, significant amounts of 2-ethyl-5-pentyltetrahydrofuran as the cis and trans isomers (according to GC and PMR) in a ratio of  $\sim$ 1:1 (experiment 9). As in the case of primary alcohols, a CH<sub>3</sub> group in the  $\delta$ -position reduces the yield of the chloroalcohol (experiment 10).

Expt.		Product, yield (as % on LTA)					
No.	Alkanol	(11)	(III)	(IV)	(V)		
	Primary			]	1		
1	ОН	65	-	+	-		
2	ОН	67	3	4	+		
3	OH	57	8	· _	4		
4	ССССССССССССССССССССССССССССССССССССССС	65	7	+	+		
5	OH CH	43	2	-	10		
6		77 *	-	-	_		
	Secondary		ļ.	}			
7		66	-	6	-		
8	ОН	45	_	+	-		
9		32	2	+	23 **		
10	OH ·	23	+	-			
11	Ph OH	-	—	42	-		
12		-	-	70	-		
13	OH		-	50	-		
	Tertiary						
14	Л ОН	64	-	+			

TABLE 2. Oxidative Chlorination of Alkanols in the System LTA-LiCl (80°C, 5-40 min, benzene 20 ml, alcohol:LTA:LiCl = 2:1:4, alcohol 0.02 mole, conversion of LTA 100%)

\*Mixture of the two diastereoisomeric forms in a ratio of 0.9:1.1. \*\*Mixture of cis- and trans-2-ethyl-5-pentyltetrahydrofuran in a ratio of ~1:1.

The course of the reaction is different in secondary alcohols with branching at the  $\beta$ carbon, the alkoxy radicals generated from 2-methyl-3-octanol and 5-methyl-4-octanol undergoing quantitative fragmentation with the elimination of the secondary alkyl radical and the formation of capraldehyde and butyraldehyde respectively. 1-Phenyl-1-hexanol behaves similarly, being converted into benzaldehyde (Table 2, experiments 11-13).

Reaction of the tertiary alcohol 2-methyl-2-hexanol with the LTA-LiCl system gives exclusively 2-methyl-5-chloro-2-hexanol. No fragmentation products of the intermediate alkoxy radical were found.

Oxidation of (I) with LTA in the presence of chlorides other metals showed that the chloride used has an effect on the course of the reaction (Table 3). The systems LTA-MgCl<sub>2</sub> and LTA-CaCl<sub>2</sub>, like LTA-CiCl, chlorinated 1-pentanol, 1-hexanol, and 1-heptanol in the  $\delta$ -position with high selectivity. In contrast, LTA-NaCl and LTA-KCl oxidized 1-heptanol for the most part to 2-propyltetrahydrofuran, and only to a small extent to 4-chloro-1-heptanol (Table 3, experiments 4 and 5). These differences in the mode of action of the oxidative systems are

TABLE 3. Oxidative Chlorination of Alkanols in the System  $LTA-MCl_2*$  (80°C, 10-20 min, benzene 20 ml, alcohol:LTA:MCl<sub>2</sub> = 2:1:2, alcohol 0.02 mole, conversion of LTA 100%)

_		Produc	Product, yield (as % on LTA)						
Expt. No.	Alkanol	LTA	- MgCl <sub>2</sub>	LTA — CaCl <sub>2</sub>					
		(II)	(III)	(11)	(III)				
1	ОН	57	3	47	_				
2	ОН	63	3	47	+				
3		52	9	46	+				
4		5**	-						
5	OH OH	ք ***							
6	ОН	52	+	54	+				
7	ОН	53	+	63	+				
			1		1				

\*Carbonyl compounds and alkyltetrahydrofurans were formed in all the experiments (overall yields 2-10%). \*\*LTA-NaCl system; in addition, 23% of 2-propyltetrahydrofuran was obtained. \*\*\*LTA-KCl system; a further 24% of 2-propyltetrahydrofuran was obtained.

most probably due to the differing solubilities of the chlorides used (Table 4). The higher solubility of Li, Mg, and Ca chlorides as compared with NaCl and KCl permits higher concentrations of the complex salt of six-coordinated Pb(IV) with the ligands AcO and Cl, formed from the LTA and the metal chloride to be reached. These salts function as efficient interceptors of radicals (B) and (C), converting them into the chloroalcohols. At the lower concentrations of the complex salts obtained with NaCl and KCl, these radicals are oxidized by LTA to  $\alpha$ -alkyltetrahydrofurans. It is noteworthy that none of the chlorides were soluble in benzene at 20-80°C in the absence of LTA. The solubility of LTA in benzene was 2.00-2.25  $10^{-1}$  mole/liter at 80°C, and 0.55-0.60  $\cdot 10^{-1}$  mole/liter at 20°C, values somewhat higher than those for the complexes formed.

The conversion of the alcohol in all the experiments (Tables 1-3) was slightly greater than the sum of the products. This is due to the ability of the alcohol to associate with metal chlorides, as shown experimentally: under the standard conditions ( $80^{\circ}C$ , 15 min, alcohol:LiCl = 1:4, benzene 20 ml), 20-25% of the alcohol was combined with the salt.

TABLE 4. Solubility of Metal Chlorides in the System LTA-Benzene (80°C, 10 min, benzene 20 ml, LTA: $MCl_n = 1:5$  (n = 1) or 1:3 (n = 2), LTA 0.01 mole).

MCI <sub>n</sub>	Solubility 10 <sup>-1</sup> , moles/liter	MCI <sub>n</sub>	Solubility 10 <sup>-1</sup> , moles/liter
LiCl	$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	NaCl KCl MgCl <sub>2</sub> CaCl <sub>2</sub>	$\begin{array}{c} 0,37{-}0,43\\ 0,11{-}0,12\\ 1,35{-}1,45\\ 1,08{-}1,13\end{array}$

\*Stirred for 5 min. †Stirred for 20 min. ‡Temperature 20°C.

δ-Chloro-	Pp °C	Foun	d/Calcu %	ulated	, Empiri-	IR sp trum,	ec- ∨,cm <sup>-]</sup>	
alkanol*	(p, mm Hg)	С	н	CI	cal for mula	C-C	с-он	δ, ppm
CI OH	71(7)	<u>48,77</u> 49,21	8,90	28.17 28,70	C <sub>5</sub> H <sub>11</sub> OCl	615	3350	1,45 d (3H, CH <sub>3</sub> , J=6,75 Hz), 1,70m (4H, CH <sub>2</sub> ), 3,20 s (1H, OH), 3,55 t (2H, CH <sub>2</sub> OH, $J=$
<b>Л</b> ЛЛОН	87 (7)	53.05	9,58	25,47	C6H15OCI	610	3340	=5,89 Hz), 4,00 m (1H, CHCl) 0,95 t (3H, CH <sub>3</sub> ,
Ċ1		52,94	9,52	26,01				J=7.54 HZ), $1.45-1.87$ m (4H, CH <sub>2</sub> ), 3.40 s (1H, OH). 3.58 t (2H, CH <sub>2</sub> OH, J=5.83 Hz), $3.82$ m
Cl OH	98(7)	55,55 55,80	9,40 9,96	24.09 23,60	C7H15OCl	610	3340	(1H, CHCl) 0.84t (3H. CH <sub>3</sub> , J=7.56 Hz), $1.12-1.82$ m (6H, CH <sub>2</sub> ), 3.53t (2H, CH <sub>2</sub> OH, J=584 Hz) $2.58$ c
CI OH	79(5)	<u>52.70</u> 52,94	<u>9,51</u> 9,52	25,30 26,01	C6H13OCl	575`	3350	(1H, OH), 3,85 s (1H, CHCl) 1,55 s (6H, CH <sub>3</sub> ), 1,75 m (4H, CH <sub>2</sub> ), 2,60 s (1H OH)
						640	2220	$3,60 \pm (2H, CH_2OH, J=5,83 Hz)$
C1 OH		_	-			040	3330	$\begin{array}{c} 0.35 \text{ (3H, CH_3, }\\ J=7.53 \text{ Hz}), 1.20-\\ 1.90 \text{ m} (14\text{H}, \text{CH}_2),\\ 3.64 \text{ t} (2\text{H}, \text{CH}_2\text{OH})\\ J=5.82 \text{ Hz}), 3.9 \text{ m}\\ (1\text{H}, \text{CHCl}) \end{array}$
Cl OH	-		-		-	640	3330	0,98 d.d (3H, CH <sub>3</sub> CH, $J=6,76$ Hz ), 1,48 d.d (3H, CH <sub>3</sub> CHCl, $J=7.51$ Hz ), 1,70 m (2H, CH <sub>2</sub> ), 1,70 m (1H, CH <sub>2</sub> ), 3.67 t (2H, CH <sub>2</sub> OH, $J=6,76$ Hz) 4,10 d.m(1H, CHCl)
сі /// он	71 (6)	53,50 52,94	9,32 9,52	25,82 26,01	C <sub>6</sub> H <sub>13</sub> OCl	625	3380	1,15 d (3H, $CH_3CHOH$ , $J=$ =6,40), 1,55d (3H, $CH_3CHCl$ , $J=$ =6,80 Hz), 1,75 m (4H, CH <sub>2</sub> ), 3,40 s (1H, OH), 3,85 m (2H, CHOH, CHCl)
CI OH	68 (0,3)	58,91 58,46	10,32 10,32	20,93 21,01	C <sub>8</sub> H <sub>17</sub> OCl	615	3355	0,88 t (3H, CH <sub>3</sub> , J=7,50 Hz), 1,20- 2,05 m (8H, CH <sub>2</sub> ), 1,47 d (3H, CH <sub>3</sub> CHCI, J=6,74 Hz), 2,46 s(1H, OH), 3,55 m (1H, CHOH), 4,00 m (1H, CHCI)

# TABLE 5. Properties of $\delta\text{-Chloroalkanols}$

TABLE 5 (continued)

δ-Chloro-	Bp, °C (p, mm Hg)	Found/Calculated,		Empiri-	IR spec- trum,∨,cm <sup>-1</sup>		PMR spectrum.	
alkanol*		С	н	CI	mula	c—c	с—он	δ, ppm
OH I Cl	-	_	-	_	_	630	3385	0,85 t (6H, CH <sub>3</sub> , J=7,52 Hz), 1.20- 2.30 m (12H, CH <sub>2</sub> ), 3,10 s (1H, OH)
СІ	_	-	_	-	-	630	3338	3,60 m (1H, CHOH), 3,85 m (1H, CHCl), 0,86 t (3H, CH <sub>3</sub> , J = 7,53 Hz), 1.20– 1.80 m (8H, CH <sub>2</sub> ), 1.55 s (6H, CH <sub>2</sub> ), 3,55 m (1H, CHOH)
СІ	66 (8)	55,45 55,80	<u>10,01</u> 9,96	23,70 23,60	C7H15OC	-	-	1,15 s (6H, CH <sub>3</sub> CCH <sub>3</sub> ), 1,45 d (3H, CH <sub>3</sub> CHCl. J=6,76 Hz), 1,65m (4H, CH <sub>2</sub> ), 2.20s (1H, OH), 3,95 m (1H, CHCl)

\*Contained 1-3% of the  $\varepsilon$ -chloroalkanol as impurity.

## EXPERIMENTAL

GLC analyses were carried out on an LKhM-80 chromatograph with a flame ionization detector, on 1 m  $\times$  3 mm columns with 5% XE-60 and 5% Carbowax 20M on Chromatone N-AW-HMDS (0.16-0.20 mm), carrier gas nitrogen. PMR spectra were obtained on a Bruker WM-250 instrument, and IR spectra on a Specord M 80, in thin films. The starting alcohols were dried and redistilled. The lead tetraacetate was of pure reagent grade, washed with glacial acetic acid and dried over caustic alkali. Its purity was determined by iodometry [8]. The benzene was dried over sodium and redistilled. 5-Chloro-1-hexanol was obtained independently by reduction of 5hexenoic acid to the corresponding unsaturated alcohol, followed by addition of HC1.

EPR spectra were obtained on an RE-1306 spectrometer during the oxidation of 1-hexanol by LTA in benzene at 20°C in the presence of the spin traps C-phenyl-N-tert-butylnitrone (PBN) and 2-methyl-2-nitrosopropane (MNP): [LTA] =  $10^{-2}$ , [PBN] =  $10^{-2}-10^{-3}$ , [MNP] =  $10^{-2}-10^{-3}$ , [C<sub>6</sub>H<sub>13</sub>OH] = 0.1-0.5 M. In a glass ampul was placed 0.1-0.2 ml of a benzene solution containing the alcohol, LTA, and the spin trap. The ampul was then degassed by freezing, evacuating, and thawing, sealed in vacuo, and placed in the resonator of the spectrometer. When the alcohol was oxidized with the system LTA-LiC1,  $10^{-1}-10^{-2}$  M LiC1 was also introduced into the ampul.

To determine the solubilities of the metal chlorides, a method was used which was based on precipitation with excess  $AgNO_3$  solution, followed by titration of the excess as described in [9].

Oxidation of Aliphatic Alcohols by the System LTA-MCl<sub>n</sub> (general method). A mixture of the alcohol, LTA, and MCl<sub>n</sub> in benzene was stirred vigorously at 80°C until all the LTA had reacted (5-40 min), the deep brown color changing to pale yellow. The mixture was then cooled, and GLC (internal standard) used to determine the conversion of the alcohol and the yields of the products:  $\delta$ -chloroalkanol (II),  $\varepsilon$ -chloroalkanol (III), carbonyl compound (IV), and alkyltetrahydrofuran (V) (Tables 1-3). To isolate the chloroalcohol, the mixture was filtered, the solid washed with ether (2 × 30 ml), and the combined filtrates washed with water (3 × 20 ml), dried over Na<sub>2</sub>SO<sub>4</sub>, and distilled. The properties of the  $\delta$ -chloroalkanols are given in Table 5. The  $\epsilon$ -chloroalkanols were identified by GLC (comparison with marker) and PMR, the carbonyl compounds by GLC (comparison with marker) and as their 2,4-dinitrophenyl-hydrazones, and the alkyltetrahydrofurans were isolated preparatively and identified by PMR spectroscopy.

2-Ethyltetrahydrofuran, PMR spectrum ( $\delta$ , ppm): 0.92 t (3H, CH<sub>3</sub>, J = 7.14 Hz), 1.40-1.65 m (4H, (CH<sub>2</sub>)<sub>2</sub>CHO), 1.80-2.03 m (2H, CH<sub>2</sub>CH<sub>3</sub>), 3.73 m (2H, CH<sub>2</sub>O), 3.85 m (1H, CH).

 $\frac{2-\text{Propyltetrahydrofuran, bp 113-114°C. PMR spectrum (\delta, ppm): 0.88 t (3H, CH<sub>3</sub>, J = 7.51 Hz), 1.25-1.60 m (4H, (CH<sub>2</sub>)<sub>2</sub>CHO), 1.85-1.98 m (4H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 3.65 m (1H, CH), 3.80 m (2H, CH<sub>2</sub>O).$ 

2-Ethyl-5-pentyltetrahydrofuran, PMR spectrum (δ, ppm): 0.88 t (6H, CH<sub>3</sub>), 1.20-1.50 m (10 H, (CH<sub>2</sub>)<sub>3</sub>CH<sub>3</sub>, (CH<sub>2</sub>)<sub>2</sub>CHO), 1.55-1.65 m (2H, CH<sub>2</sub>(CH<sub>2</sub>)<sub>3</sub>CH<sub>3</sub>), 1.86-2.07 m (2H, CHCH<sub>2</sub>CH<sub>3</sub>), 3.70-3.90 m (2H, CH).

Determination of the Solubilities of Metal Chlorides in the System LTA-Benzene (Table 4). A mixture of 0.01 mole of LTA and 0.05 mole of MC1 or 0.03 mole of MCl<sub>2</sub> in 20 ml of benzene was stirred at 80°C for 10 min, and 5 ml samples withdrawn. The solvent was removed from the samples in a rotary evaporator. The dry residue was then dissolved in 1-2 ml of a mixture of concentrated nitric acid and 20% H<sub>2</sub>O<sub>2</sub> (in a ratio of 2:1) to reduce the Pb(IV) to Pb(II). When large amounts of the chloride were present (LiCl, MgCl<sub>2</sub>, and CaCl<sub>2</sub>), the solution was diluted with water to 100 ml, 10 ml withdrawn and treated with 5 ml of 0.01 N silver nitrate solution. When small amounts of chloride were present (NaCl and KCl), the undiluted solution was used, this being treated with 10 ml of 0.01 N silver nitrate. When the silver chloride had coagulated, the mixture was filtered, the filtrate treated with 2-3 drops of a solution of iron alum (a saturated solution of iron alum treated with concentrated nitric acid until the brown color disappeared), and titrated with 0.01 N ammonium thiocyanate until a persistent pale pink end-point was reached [9]. The solubility of LTA was measured as described in [8].

The metal chloride solubilities were determined with the assistance of A. Yu. Popkov.

### CONCLUSIONS

1. In the oxidation of primary, secondary, and tertiary aliphatic alcohols with the system  $Pb(OAc)_{\mu}$ -metal chloride, the principal product is the  $\delta$ -chloroalkanol.

2. EPR using spin traps has been used to identify the alkoxyl and secondary C-centered radicals generated in the oxidation of an alcohol with Pb(OAc)<sub>4</sub> and with the system  $Pb(OAc)_4$ -LiC1.

## LITERATURE CITED

- G. I. Nikishin, N. I. Kapustina, and A. V. Lisitsyn, Izv. Akad. Nauk SSSR, Ser. Khim., 1. 1689 (1986).
- 2. J. Y. Nedelec, M. Gruselle, A. Triki, and D. Lefort, Tetrahedron, 33, 39 (1977).
- Z. Ceković, Rad. Jugosl. Akad. Znan. Umjet., 2, 21 (1983). 3.
- M. L. Mihailović and Z. Čeković, Synthesis, 209 (1970). 4.
- 5. C. Walling and D. Bristol, J. Org. Chem., <u>37</u>, 3514 (1972).
- 6.
- Ž. Čeković, and G. Djokić, Tetrahedron, <u>37</u>, 4263 (1981).
  M. Green, J. M. Moldovan, and J. G. McGrew, J. Org. Chem., <u>39</u>, 2166 (1974). 7.
- L. F. Fieser and M. Fieser, Reagents for Organic Synthesis, Vol. 3, Wiley, New York 8. (1972).
- 9. V. F. Gillebrand, G. É. Lendel', G. A. Brait, and D. I. Gofman, A Practical Manual of Inorganic Analysis [in Russian], 3rd ed., Khimiya, Moscow (1966), p. 813.