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REACTION OF ALKOXYHYDROPEROXIDES WITH METAL SALTS
ALKYL HALIDE PREPARATION

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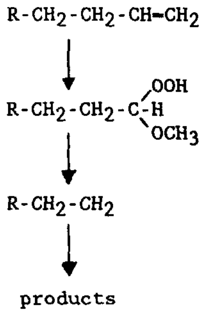
A synthesis is described of alkyl chlorides by reaction of methoxy hydroperoxides, prepared from 1-alkenes by ozonisation in methanol, with ferric chloride: $RCH{=}CH_2 \rightarrow RCH(OOH)OCH_3 \rightarrow R\bullet \rightarrow RCl$. Yields of 1-chlorotetradecane were about 60%; those of other halides from 38 to 57%.

INTRODUCTION

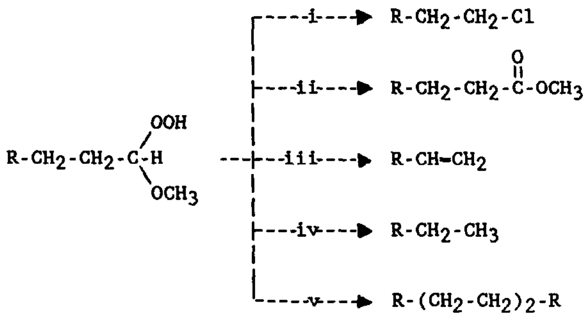
When alkenes are ozonised in alcoholic solution alkoxyhydroperoxides are formed.[1] Their reactivity towards metal salts has been explained by postulating carbon-carbon bond splitting, followed by radical formation (Scheme 1). Much of the chemistry describing radical reactions in organic synthesis has been reviewed recently.[2] Depending on the nature and the oxidation state of the transition metal, and on the nature of the counter ion, a range of products may be formed from the intermediate radical shown in Scheme 1. We have described procedures for preparing chloro-acetals,[3] olefinic acetals[3] and esters[4], chloro-esters, and hydroxy-acids and dicarboxylic acids[5] in modest to good yields (Scheme 2). For simplicity of reaction and for optimal yields the alkoxyhydroperoxides should be derived either from 1-alkenes, such as 1-hexadecene, or from alkenes which are completely symmetrical about the olefinic bond, including cycloalkenes (compare Ref. 4).

We now present work on the reaction of alkoxyhydroperoxides with a range of transition metal halides, intended to optimise formation of alkyl halides. Alkyl chlorides particularly were chosen because of their ubiquitous roles in organic chemistry,[6] and our interest in their preparation and utility.

SCHEME 1



SCHEME 2 Reagents: (i) $FeCl_3.6H_2O$; (ii) Δ/H^+; (iii) $FeSO_4.7H_2O + CuAc_2.H_2O$; (iv) $Fe(NO_3)_3.9H_2O$; (v) $FeSO_4$



RESULTS

Ozonisation of 1-hexadecene in methanol afforded 1-hydroperoxy-1-methoxypentadecane as a pure crystalline compound (R = $C_{12}H_{25}$ in Schemes 1 and 2). This was used in a series of model experiments with transition metal chlorides in order to study its conversion to 1-chlorotetradecane. The fragmentation reaction described in our earlier papers[3-5] operated again with loss of one carbon atom leading to the intermediate radical, and with formation of 1-chlorotetradecane. In clean reactions only 1-chlorotetradecane and methyl pentadecanoate were formed. In other reactions where a number of unidentified products were observed analytical values were determined only for these two products as percentages of total GLC peak areas.

From our independent parallel experiments it was found that heating 1-hydroperoxy-1-methoxypentadecane in methanolic ferric chloride or hydrogen chloride solution led to formation of methyl pentadecanoate. Controlled pyrolysis of crystalline 1-hydroperoxy-1-methoxypentadecane afforded mixtures of methyl pentadecanoate and free pentadecanoic acid. This reaction was verified for a series of 1-hydroperoxy-1-methoxyalkanes derived from homologous 1-alkenes. Griesbaum and co-workers also observed the acid-catalysed conversion of more reactive methoxyhydroperoxides to methyl esters in methanolic solution at ambient temperatures.[7a,b]

We investigated the following variables in the conversion of 1-hydroperoxy-1-methoxypentadecane to 1-chlorotetradecane with transition metal chlorides in solution: the influence of transition metal chlorides besides iron and copper, molar ratios and concentrations of reactants, the use of other solvents besides methanol, and finally the influence of the solvents used at the ozonisation stage, because these can be either participating solvents such as alcohols and ketones, or non-participating solvents such as hexane and light petroleum. The following is a summary of the results.

Table 1 Influence of Metal Halides (3 mol/mol) on Yields of 1-Chlorotetradecane and Methyl Pentadecanoate in Methanol

Metal halide	*Yield by GLC**	
	$C_{14}H_{29}Cl$	$C_{14}H_{29}COOCH_3$
$FeCl_3$	75	25
$FeCl_3.6H_2O$	75	25
$CrCl_3.6H_2O$	75	12
$CuCl_2.2H_2O$	58	42
CuCl	27	37
$MnCl_2.4H_2O$	28	48
$CoCl_2.6H_2O$	11	32
$NiCl_2.6H_2O$	38	46
$ZnCl_2$	14	42
VCl_3	5	7
$AlCl_3.6H_2O$	0	34

*Peak areas. Other products (to 100%) remained unidentified

Appreciable yields (60-70%) of 1-chlorotetradecane were obtained whenever the molar ratio of ferric chloride to methoxyhydroperoxide was 2:1 or more. The concentrations of the reactants appeared to have little or no influence on the final conversion yields. Ferric and chromic chlorides in methanol were superior to other metal chlorides in the conversion of 1-hydroperoxy-1-methoxypentadecane to 1-chlorotetradecane (Table 1).

The influence of different alcohol solvents up to 1-octanol in the conversion of 1-hydroperoxy-1-methoxypentadecane to 1-chlorotetradecane with ferric chloride was minimal. The yields in the higher alcohols were gradually depressed slightly, but they were possibly influenced most by the decreasing solubility of the metal salt in the higher alcohols. The effect of water on the conversion to alkyl chloride in aqueous methanol was also minimal. Up to 25% water (v/v) was tolerated without appreciable decline in the yield of 1-chlorotetradecane, but on further increase in the water content yields dropped appreciably below the 70% level. The cause may be increasing inhomogeneity in the mixture, lack of miscibility and contact between the reactants, and side reactions with water as a further complication.[8] Results using cupric chloride in aqueous methanol were similarly affected.

At the ozonisation stage of reactive intermediate formation not only alcohols but also ketonic and carboxylic acid solvents can participate. These three classes of solvents provide alkoxyhydroperoxides, ozonides and acetoxyhydroperoxides respectively,[8] all of which are potential reactants in the metal salt systems described here. A series of experiments was performed with 1-hexadecene ozonized in a number of solvents, followed by conversion with ferric chloride (2 molar equivalents) in the same or a different solvent (Table 2).

Table 2 Influence of Solvents on the Yield of 1-Chlorotetradecane

Ozonisation solvent	*Conversion solvent*	$C_{14}H_{29}Cl$ *% by GLC**
MeOH	MeOH	63
BuOH	MeOH	53
Acetone	Acetone	57
Light petroleum	BuOH	65
Acetic acid	MeOH	20

*Other products to 100%

Results indicated clearly that ozonisation of 1-hexadecene in the lower aliphatic alcohols methanol, ethanol and butanol, or in acetone or light petroleum was to be preferred. The alkoxyhydroperoxides and the ozonides formed then reacted cleanly with hydrated ferric chloride to form 1-chlorotetradecane in yields of 53-65%. Ozonisation in acetic acid led to a yield of only 20% 1-chlorotetradecane subsequently. However, when two different alcohol solvents were used, first in the ozonisation and then in the ferric chloride treatment, some acid-catalysed *trans*-esterification occurred, which complicated the analysis of the pentadecanoate ester(s).

In summary, practicable conditions for the conversion of 1-hydroperoxy-1-methoxypentadecane to 1-chlorotetradecane were found using two or more equivalents of hydrated ferric chloride in methanol. Similar results on a preparative scale were obtained using 1-hexadecene directly in the process without isolating the intermediate alkoxyhydroperoxide. In this generalised way olefinic compounds - which initially may be only sparingly soluble in methanol - may be made soluble at the ozonisation stage (compare methyl erucate[4]).

Frequently, alkyl bromides are preferable to the chlorides in synthetic applications so that conversion of alkyl chlorides, in general cheaper and more readily available, to the bromides offers certain attractions. Methods described recently include halogen exchange with metal bromide salts under phase transfer conditions,[9] and the use of hydrogen bromide catalysed by anhydrous ferric bromide.[10] Although hydrated ferric bromide is not so convenient a reagent as ferric chloride on account of its thermal instability,[11] we obtained 1-bromotetradecane by the same procedure as above in a yield of 57% based on 1-hexadecene.

Other preparative examples of alkyl chloride formation described here are 1,1-dimethoxy-5-chloropentane, 1,1-dimethoxy-11-chlorodecane and ethyl 11-chloro-undecanoate from, respectively, cyclohexene, cyclododecene and 1-ethoxy-1-cyclododecene, the latter being prepared from cyclododecanone.

The results of reactions between methoxyhydroperoxides and transition metal salts which are described in our previous publications[3-5] relate only to aliphatic alkoxyhydroperoxides and ozonides lacking structural variation or complexity adjacent to the reactive group. We have conducted a brief investigation into the reactivity with ferrous sulfate of two α-methoxybenzyl hydroperoxides which were prepared by ozonolysis of stilbene and 4,4′-dimethoxystilbene respectively in methanol.[12] It was considered that analogously to the aliphatic compounds studied earlier these aromatic alkoxyhydroperoxides might undergo rupture of a carbon-carbon bond, with phenyl radical formation and dimerisation leading to biaryls (compare Ref. 13). However, no biaryls were found. Benzaldehyde and methyl benzoate in the one case, and 4-methoxybenzaldehyde together with methyl 4-methoxybenzoate in the other, were the sole products formed from reaction of the alkoxyhydroperoxy derivatives with ferrous sulfate.

The ozonides of stilbene and 4,4′-dimethoxystilbene, prepared in methylene chloride as solvent, were also treated with ferrous sulfate. Unexpectedly, besides aldehyde and carboxylic acids, small amounts of phenol and 4-methoxyphenol were also formed, suggesting a relationship of this side reaction to the Lewis-acid catalysed benzylic hydroperoxide rearrangement to phenols,[14] or to the industrially important acid-catalysed formation of phenol from cumene hydroperoxide.[15]

EXPERIMENTAL SECTION

CAUTION. The hydroperoxy compounds described here are potentially explosive.

Materials. Ozone in a stream of O_2 was generated using a Stage K.G. Ozoniser producing about 0.1 mol O_3 in O_2/h. Transition metal salts listed in Table 2 were commercial products, as were 1-hexadecene (Fluka), cyclohexene (BASF), cyclododecene (Columbian Carbon Co.) and cyclododecanone (Fluka). Alcohols and other solvents were of analytical grade. Light petroleum was a fraction b.p. 40-60°C.

Analyses. GLC was carried out on silicone elastomer SE30 and on PEGA 5% (support Diatoport S 80-100). Identity of compounds was confirmed by comparison in some cases with authentic samples and by spectral analyses (IR, NMR, MS). Combined GLC/MS was performed using a 10 m column of CP SIL 5 and an MS 5970 A (Hewlett Packard) spectrometer. Combustion analyses were carried out at Microanalytical Laboratory, Max Planck Institute for Coal Research, Mülheim, Federal Republic of Germany.

1-Methoxy-1-hydroperoxypentadecane. A stream of ozone in oxygen was passed into a solution of 30.0 g (0.13 mole) 1-hexadecene in 1 l methanol at 20°C. After completion of the reaction the solvent was distilled off on a Rotavap at 20°C/2 kPa, the residue (36.5 g) was dissolved in 200 ml light petroleum and cooled in ice. The crystals which formed (18.8 g, 52% of theory) were recrystallised from 100 ml light petroleum; yield 7.3 g (20% of theory), m.p. 42-43°C. (Found: C, 69.9; H, 12.4; 0, 17.5. $C_{16}H_{34}O_3$ requires: C, 70.13; H, 12.14; 0, 17.52). The hydroperoxide content by iodometric titration was 99-101.5%.

Thermal Decomposition. MS analysis indicated decomposition of the compound to methyl pentadecanoate. This was in agreement with results of thermal decomposition experiments carried out separately on the crystalline solid which was heated slowly to 130°C (**CAUTION!** *For description of an explosion under similar reaction conditions see Ref. 12, p. 1540*). The products in different experiments were mixtures of the methyl ester (75-90%) and the free carboxylic acid (10-25%), determined by GLC before and after methylation with diazomethane (compare Ref. 7). Pure pentadecanoic acid was obtained by saponification and crystallisation, m.p. 51°C. Lit.[16] m.p. 51°C.

Reaction of 1-Methoxy-1-hydroperoxypentadecane with Transition Metal Chlorides

(a) *Comparative Experiments*. 30 ml aliquots of methanolic solutions 1 molar in the appropriate transition metal chloride (as hydrate, or anhydrous) were heated to b.p., and 2.7 g (10 mmol) quantities of pure crystalline 1-methoxy-1-hydroperoxypentadecane were added all at once. Boiling was continued for 15 min, the mixtures were cooled, diluted with water, extracted with light petroleum, the extracts dried (Na_2SO_4) and the products analyzed directly by GLC. Results are contained in Table 1.

(b) *Preparative experiments*.

1-Chlorotetradecane. (i) A solution of 40.0 g 1-hexadecene (0.18 mole) in 800 ml methanol was treated with an O_3/O_2 mixture as above. The residue obtained on evaporation weighed 53 g, and 49.8 g of this was added to 400 ml 1 M $FeCl_3.6H_2O$ solution in methanol at the b.p. in 7 min. The mixture boiled spontaneously without further heating and then heating was continued for a further 15 min. The mixture was cooled, concentrated on a Rotavap and the residue was extracted with light petroleum. The organic solvent was removed and the residue (42.8 g) was fractionally distilled, giving 24.8 g 1-chlorotetradecane, b.p. 114°C/53 Pa, n_D^{20} 1.4469; (lit.[17] b.p. 139-142°C/530 Pa, n_D^{20} 1.4450); yield 63% of theory (Found: C, 72.2; H, 12.5; Cl, 15.2. $C_{14}H_{29}Cl$ requires: C, 72.63; H, 12.63; Cl, 15.31). IR, NMR and MS were in agreement. MS parent peak 232. IR 655 cm^{-1} (C-Cl). - (ii) When the ozonisation was carried out in butanol (500 ml) the same procedure gave 64.5 g crude 1-butoxy-1-hydroperoxypentadecane, of which 61.5 g was converted to 1-chlorotetradecane with 400 ml 1 M $FeCl_3.6H_2O$

in methanol. The reaction product (53.7 g) was fractionally distilled, giving 21.4 g 1-chlorotetradecane; yield 53% of theory.

Influence of Solvents. (i) A solution of 60.0 g (0.27 mol) 1-hexadecene in 600 ml acetone was treated with O_3/O_2 at 5°C until reaction was complete. The solution was added within 5 min to a boiling solution of 162 g (0.6 mol) $FeCl_3.6H_2O$ in 600 ml acetone. After 15 min (peroxide reaction negative) 600 ml acetone was distilled off, the mixture was diluted with 250 ml water and extracted with 3 x 300 ml light petroleum. The dried extracts (Na_2SO_4) were evaporated, yielding 61.3 g product which was fractionally distilled; 28.8 g 1-chlorotetradecane b.p. 114-115°C/60 Pa was obtained. Yield 46% of theory. - (ii) Similarly a solution of 56.0 g (0.25 mol) 1-hexadecene was ozonised in 1 l light petroleum at 0°C. The solution was added gradually to a solution of 135 g (0.5 mol) $FeCl_3.6H_2O$ in 500 ml butanol at 70-75°C, during which petroleum was distilled off. The reaction mixture was cooled, extracted with 750 ml water and the butanol layer evaporated at 70°C/2 kPa. The residue (75 g) was fractionally distilled, giving 38.0 g 1-chlorotetradecane, b.p. 119°C/270 Pa. Yield 65% of theory.

Ethyl 11-chloro-undecanoate. 5.0 g (20 mmol) 1-ethoxy-1-cyclododecene, prepared from cyclododecanone,[18] was dissolved in 50 ml ethanol and added dropwise in 20 min to 100 ml ethanol at about 0°C (ice bath) through which a current of O_3/O_2 was passed. Ethanol was removed on the Rotavap and the residue was added to 50 ml 1 M $FeCl_3.6H_2O$ in ethanol under reflux. The crude product (5.4 g), obtained by extraction with light petroleum as above, was percolated through (15 g) Al_2O_3 column eluting with light petroleum/ether (95:5 v/v). The mixture obtained was shaken with 200 ml 4% 2,4-dinitrophenylhydrazine solution in 4 M HCl for 15 min to remove aldehydic products as their 2,4-DNPs. The light petroleum/ether layer was dried (Na_2SO_4) and percolated through a silicagel column. The ester, ethyl 11-chloro-undecanoate, was recovered pure from the eluate by evaporation and distilled in a small-bulb apparatus; yield: 2.5 g (42% of theory), n_D^{20} 1.4483 (lit.[19] 144°/530 Pa, n_D^{20} 1.4480) (Found: C, 62.8; H, 10.1; O, 12.9. $C_{13}H_{25}O_2Cl$ requires: C, 62.70; H, 10.12; O, 12.85).

1,1-Dimethoxy-11-chloro-undecane. A stream of ozone in oxygen was passed into a solution of 29.6 g (0.18 mol) cyclododecene in 500 ml methanol at 20°C. When no more starting material remained the solution was evaporated (Rotavap) at 20°C/2 kPa. The residual oil weighed 47.0 g, and 40.3 g of this was added in 6 min to a boiling solution of 135 g $FeCl_3.6H_2O$ in 500 ml methanol. When spontaneous boiling ceased, heating was applied up to b.p. for another 15 min. The mixture was cooled and extracted with 3 x 250 ml light petroleum. The combined extracts were dried (Na_2SO_4) and evaporated. The residue (32 g) was distilled, giving 19.2 g 1,1-dimethoxy-11-chloro-undecane, b.p. 114-115°C/4 Pa, n_D^{26} 1.4460. Yield 48% of theory. (Found: C, 62.3; H, 10.7; Cl, 14.0; O, 12.9. $C_{13}H_{27}ClO_2$ requires: C, 62.32; H, 10.86; Cl, 14.15; O, 12.77). MS parent peak 250. IR: 1130, 1080, 1050 cm^{-1} (acetal); 655 cm^{-1} (C-Cl).

1,1-Dimethoxy-7-chloroheptane. This was prepared similarly by ozonising 47.4 g (0.44 mol) cyclooctene in 800 ml methanol and adding the product after evaporation to a boiling solution of 243 g (0.9 mol) $FeCl_3.6H_2O$ in 900 ml methanol. Distillation of the product, isolated as above, gave 37.5 g 1,1-dimethoxy-7-chloroheptane b.p. 84-85°C/0.4 kPa, n_D^{25} 1.4380.

Yield 44% of theory. (Found: C, 55.4; H. 9.7; Cl, 18.1; O, 16.6. $C_9H_{19}ClO_2$ requires: C, 55.57; H. 9.85; Cl, 18.23; O, 16.45). MS parent peak 194.

1,1-Dimethoxy-5-chloropentane. Cyclohexene (56.7 g, 0.7 mol) in 1 l methanol was ozonised as above and after evaporation the residue (133 g) was added to 326 g (1.2 mol) $FeCl_3.6H_2O$ in 1.2 l methanol at the b.p. After 15 min the mixture was cooled, concentrated and extracted with 800 ml CH_2Cl_2. The dried extract (Na_2SO_4) was fractionally distilled giving 54.0 g 1,1-dimethoxy-5-chloropentane, b.p. 85-88°C/1.6 kPa; yield 47%. The acetal was identified by IR and NMR, and, through conversion to the 2,4-DNP of 5-chloropentanal, by comparison with 1,1-diethoxy-5-chloropentane.[20]

1-Bromotetradecane. To 1 l methanol solution containing 1 mol $FeCl_3.6H_2O$ conc. ammonia solution was added in slight excess with stirring. The precipitate was washed with methanol/ /water (3:1 v/v) until chloride-free and was then dissolved in concentrated (48%) HBr solution in water. Water was distilled off at 50°C/2 kPa till the weight of the residue (400 g) corresponded approximately with the composition $FeBr_3.6H_2O$. A solution of 56.0 g hexadecene (0.25 mol) in 1 l methanol was ozonised as above at 0°C. Methanol was partly evaporated at 20°C/2 kPa and the residue (300 ml) was added to a boiling solution of 283 g $FeBr_3.6H_2O$ (0.7 mol) in 600 ml methanol. After 15 min boiling the mixture was concentrated (CAUTION: free bromine) and the residue extracted with 1 l CCl_4. This extract was washed with 100 ml 2% sodium thiosulfate solution and dried (Na_2SO_4). The solvent was removed and the residue (65 g) fractionally distilled; 47.7 g 1-bromotetradecane was obtained, b.p. 107°C/7 Pa, n_D^{20} 1.4608, yield 57%. MS parent peak 276. IR 650 and 560 cm^{-1} (in KBr prism). Lit.[21] b.p. 167- -169°C/2.4 kPa, n_D^{20} 1.4575.

1-Chloro-2-phenylethane. An O_3/O_2 mixture was passed into a solution of 56.3 g (0.43 mol) 4-phenyl-1-butene in 600 ml methanol. The solvent was evaporated at 20°C/2 kPa, and 90.5 g of the residue (91.4 g) was added to a boiling solution of 222 g (0.8 mol) $FeCl_3.6H_2O$ in 800 ml methanol during 12 min. When spontaneous boiling ceased, heating was continued for 15 min, the mixture was cooled, concentrated, and the residue extracted with light petroleum. The dried extract (Na_2SO_4) was evaporated and the residual oil distilled giving 23.0 g (38% of theory) 1-chloro-2-phenylethane, b.p. 50-53°C/ 53 Pa, n_D^{20} 1.5299 (lit.[22] b.p. 34°C/ /133 Pa, n_D^{23} 1.5285) (Found: C, 68.4; H, 6.5; Cl, 25.0. C_8H_9Cl requires: C, 68.39; H, 6.46; Cl, 25.24).

Reaction of α-Methoxybenzyl Hydroperoxides with Ferrous Sulphate. - (i) Stilbene and 4,4'-dimethoxystilbene (1.0 g quantities of each) were dissolved in 25 ml aliquots of methanol and ozone in oxygen was passed in until reaction was complete (TLC). Excess hydrated ferrous sulfate (5 g) was added to each solution of methoxyhydroperoxide and the mixtures were then shaken at room temperature for 10 min, and poured out into light petroleum (50 ml). The organic layers were separated, dried (Na_2SO_4) and evaporated. The residues were analysed by combined GLC/MS. The sole products detected in typical experiments were benzaldehyde (23%) and methyl benzoate (77%) from stilbene, and 4-methoxybenzaldehyde (35%) and methyl 4-methoxybenzoate (65%) from 4,4'-dimethoxystilbene. - (ii) Stilbene and 4,4-dimethoxystilbene (each 1.0 g) were ozonised similarly in CH_2Cl_2 as solvent. After evaporation the ozonides were treated with 2.5 g quantities of hydrated ferrous sulfate in 50 ml methanol.

Work-up and analysis as above showed benzaldehyde (38%), phenol (2%) and benzoic acid (60%) were formed from stilbene. In the case of 4,4-dimethoxystilbene GLC/MS revealed 4-methoxybenzaldehyde (90%) and 4-methoxyphenol (10%), but 4-methoxybenzoic acid was not eluted from the column under the same conditions. The mixture of products from 4,4-dimethoxystilbene was crystallised from acetone, giving 0.12 g 4-methoxybenzoic acid, m.p. 180 °C (lit.[23] mp. 184 °C), identified by IR.

The presence of 4-methoxyphenol among the reaction products was confirmed by comparison on TLC with an authentic sample. Rapid development of an intense blue spot on spraying with phosphomolybdic acid reagent and heating clearly distinguished 4-methoxyphenol from the other products formed, and may be attributed to its ready oxidisability.

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