

**THE SYNTHESIS AND SOME CHARACTERISTICS OF PARTLY
FLUORINATED ALCOHOLS ON THE BASIS OF
TETRAFLUOROETHYLENE AND HEXAFLUOROPROPYLENE
(REVIEW)**

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In this review the approaches of partly fluorinated alcohols on the tetrafluoroethylene and hexafluoropropylene basis are analyzed. New experimental data regarding telomeric alcohols involvement into reactions with unsaturated compounds and hetero-organic derivatives are discussed. Oxidization processes and reactions in strong acid mediums of telomeric alcohols are analyzed. Also in this review you will find the trends of practical using of telomeric alcohols. The questions regarding partly fluorinated alcohols toxicity and some of their derivatives are discussed here.

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2.4. The oxidization of telomeric alcohols down to polyfluorinated carbone acids and practical use of these acids.

Telomeric alcohols (n = 4, 5) can be used as ingredient of surfactant compositions. Thus the addition of telomeric alcohol (n=4) to sodium laurylsulphate provides 1,5 time surface tension lowering compare to of pure sodium laurylsulphate. It can be used for metal electrolytic refining and ore flotation.

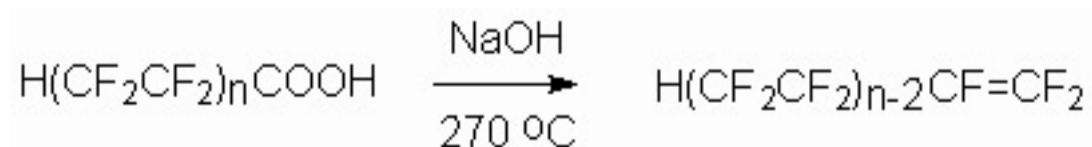
Telomeric alcohols can be oxidized till corresponding α -hydroperfluorocarboxylic acids, which are important for creation of different fluorine materials for practical purpose, among which we'll point out surface active materials, which are used as emulsifiers at polymerization of fluorolefines, as basis for universal foaming agent, work liquids, oil dopes, pesticides, bactericidal and pharmaceutical compositions.

Different salts are obtained on the base of polyfluorinated carboxylic acids. They are used:

- As foaming agents and products for fire extinguishing, as foam stabilizers for production of polyurethane foam,
- In electrometallurgy and electroplating for metals' refining,
- for creation of effective cleansers,
- for materials' treatment and making them water-repellent and oil-repellent ,
- as slushing and insulating metal coatings,
- for creation of new types of extragents and metal dressers,
- for creation of high-temperature lubricants and moving details coatings, they are used as wear-preventive additives for lubricating oils.

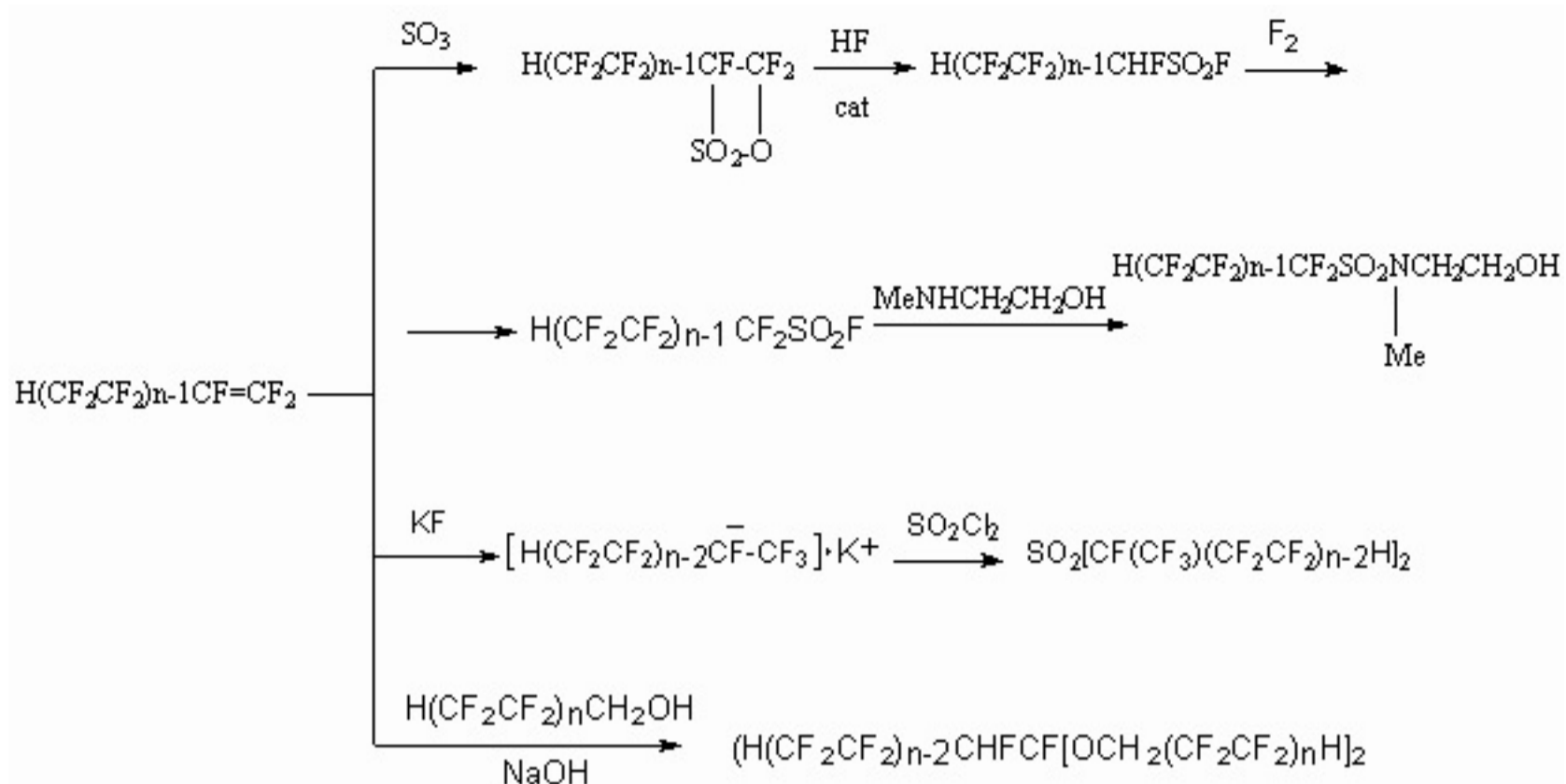
The deterioration of motor oils at 0.2-2% admixture's addition is 2/3 -1/2 of corresponding value for the oil, which doesn't contain the admixture.

Poly-fluorinated carboxylic acids $H(CF_2CF_2)_nCOOH$ (n = 1-5) can be semi-products for obtaining of terminal poly-fluorolefines.



The last ones mentioned can be used for:

- obtaining of polyfluoroalkansulpho acids and obtaining on their basis of surface active materials and electrolytes for lithium batteries and rechargeable accumulators,
- creating of new complex-formers for rare elements' salts,
- production of high-temperature liquid dielectrics, heat-transfers and hydraulic liquids.



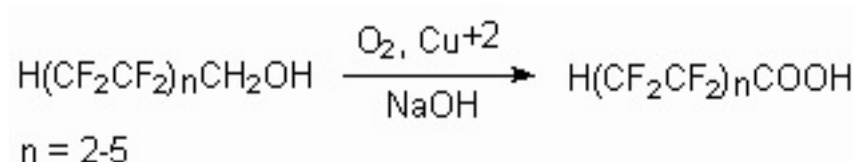
The oxidization of telomeric alcohols is carried out using potassium permanganate action, nitrogen oxides, chrome anhydride, chlorine at 100-140 °C and UV irradiation.

However these methods have essential disadvantages and manufacturing technologies are not developed on their basis. Thereby the oxidization processes of telomeric alcohols are still in the focus of researchers.

Thus, telomeric alcohols (C1-16) are oxidized using nitric acid in the presence of catalysts, which are oxides and salts of ferrum, nickel, copper and vanadium [109]. At oxidization of $\text{H}(\text{CF}_2)_8\text{CH}_2\text{OH}$ by 55 %-nitric acid and simultaneous oxygen supply at 125°C and $P=0,8$ MPa in the presence of $\text{FeCl}_2 \cdot n\text{H}_2\text{O}$ after 7 hours at 100 % conversion of alcohol the corresponding acid $\text{H}(\text{CF}_2)_8\text{COOH}$ is obtained with 99.6 % yield [109].

Higher carbon acids are obtained using electrochemical fluorination of telomeric alcohols $\text{H}(\text{CF}_2\text{CF}_2)_n\text{CH}_2\text{OH}$, ($n = 2-5$) in anhydrous hydrogen fluoride [27]. The last have found their application at emulsion polymerization of tetrafluoroethylene.

Catalytic system, containing bivalent copper complexes, catalyzes selective oxidization of telomeric alcohols till perfluorocarboxylic acids [110]. This method is especially effective for alcohols with long hydrocarbon chain (C8-14). The process passes in alkaline medium, which is necessary for alcohol conversion into anion form, that essentially facilitates the molecular oxygen activation at low temperatures.

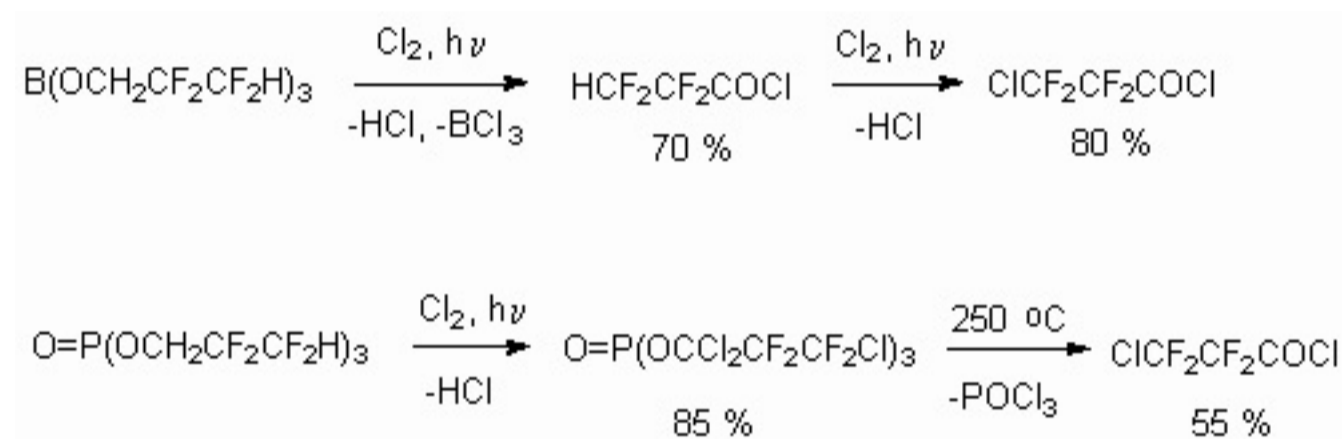


The participation of copper complexes in the transfer of electrons from alcoholates ions to oxygen obviously allows the reaction to pass according to

thermodynamically efficient di-electrone mechanism, that determines for abnormally high oxidization rates. The rate of reaction doesn't depend on molecular mass of oxidized alcohols in the interval $n = 1-6$. Variation of reaction carrying out conditions (temperature, solvent, ligand, alkali and catalyst concentration and so on) allows oxidizing of telomeric alcohols selectively (100%). For example, $\text{HCF}_2\text{CF}_2\text{CH}_2\text{OH}$ is oxidized by molecular oxygen in the presence of copper catalyst and alkali agent (in the presence of $5 \cdot 10^3$ M solution CuCl_2 and $1 \cdot 10^{-2}$ M solution of ortho-phenanthroline in iso-butyl alcohol and NaOH at 45°C , 6.5 hours) with the yield of 73.8 % to $\text{HCF}_2\text{CF}_2\text{COOH}$ (conversion = 50.5 %).

Oxidization of telomeric alcohols $\text{H}(\text{CF}_2)_n\text{CH}_2\text{OH}$ ($n = 2, 4, 6$) in the liquid phase by air oxygen at 350°C in the presence of V_2O_5 results in formation of $\text{H}(\text{CF}_2)_n\text{CH}(\text{OH})_2$ with the yield 74-93 % [111].

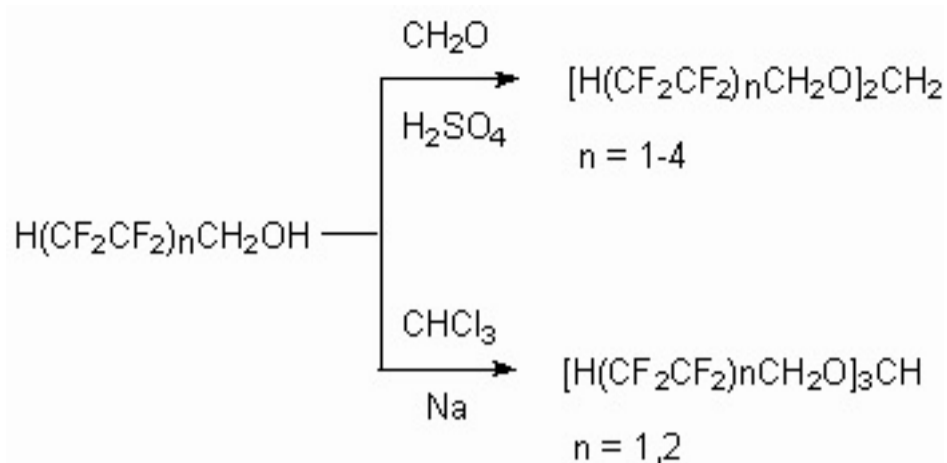
The authors [112] worked out the approach to 3- chlorotetrafluoropropionic acid synthesis using photochemical chlorination of telomeric alcohol $\text{HCF}_2\text{CF}_2\text{CH}_2\text{OH}$ borate and phosphate esters.



The chlorine action is processed at UV-radiation (lamp of 100-400 W) at $25-110^\circ\text{C}$, at that consecutive replacement α - and ω - hydrogen atoms of alcohol can occur. Decomposition of chlorinated borate passes now at conditions of reaction till acid chlorine anhydride is formed, while chlorinated phosphates require high temperatures ($180-250^\circ\text{C}$). At the same time the telomeric alcohols' effect of chlorine in the presence of catalyst results in formation of 1-chloroperfluoroalkanes [113].

2.5. Dielectric heat transfers synthesis based on partly fluorinated alcohols.

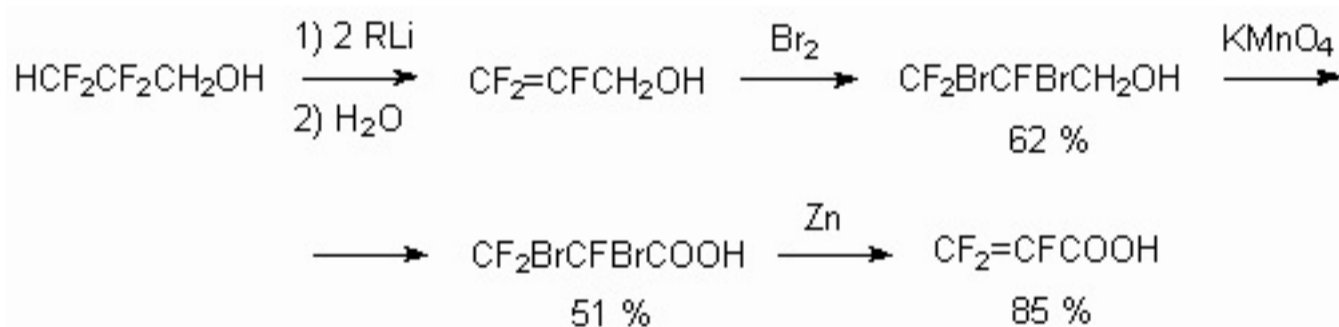
Telomeric alcohols can act as intermediate products to obtain high-temperature and high effective heat transfers [114], dielectrics [115], and they can act as effective lubricating oils and compositions, for example at modification of tosyl ethers based on phenols and telomeric alcohols [116]. It is stated, that the formation of corresponding acetals [117,118] occurs during reaction of telomeric alcohols with formaldehyde in concentrated sulfuric acid.



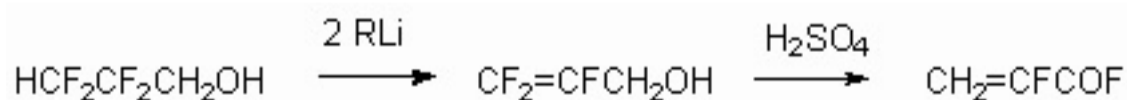
Fluoroalkyl ortho ethers [119] are obtained at interaction with chloroform in the presence of alkali metals. These ethers are thermal resistant and their liquid boiling points are high, so they can find their application as heat transfers, while their additional fluorination using element fluorine results in formation of perfluorinated ethers, which be of interest of as liquid dielectrics.

2.6. The processes with telomeric alcohols.

Telomeric alcohols have two reaction centers, according to which the reactions can be carried out. Above we had discussed the telomeric alcohols reactivity in reactions, passed affecting alcohol group. At the same time the presence of CHF_2 fragment at the end of carbon chain allows to expect the turnings with its active participation. First of all, the presence of fluorine atoms and the influence of CF_2 fragment being alongside results in acidity increasing and thereafter this makes carbanion simulated emission possible. Indeed, the action of two moles of alkyl lithium leads to formation of terminal multiply bond [26]. This, for example, allows to work out the trifluoroacrylic acid obtaining method using 2,2,3,3-tetrafluoropropyl alcohol as raw product.

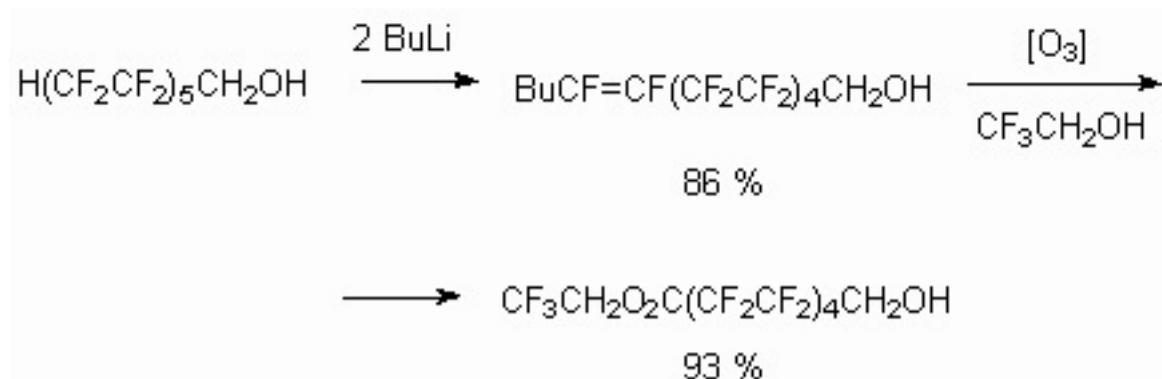


If you affect allyl alcohol with concentrated sulphuric acid, the α -fluoroacrylic acid fluoroanhydride will be formed [120].



R = Me, Bu

The ozone oxidized unsaturated alcohol (Z+E) [121] in $\text{CF}_3\text{CH}_2\text{OH}$ medium till corresponding acid is obtained analogously by BuLi action out of $\text{H}(\text{CF}_2\text{CF}_2)_5\text{CH}_2\text{OH}$.



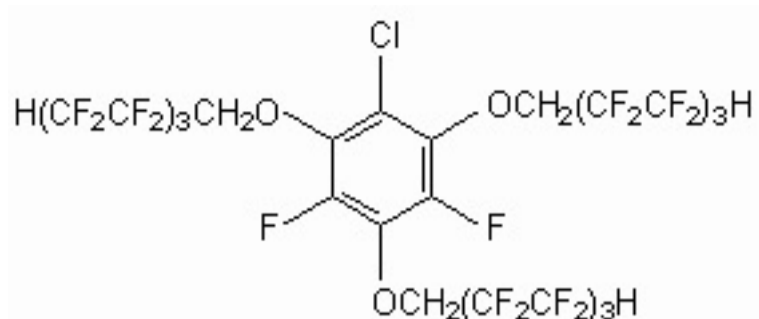
The possibility of terminal multiply bond simulated emission can be used for other purposes also.

3. Toxicity of Partly fluorinated alcohols and some of its derivatives.

There had been carried out the researches of muscles regarding studying of toxicological parameters [122,123] for partly fluorinated dialkyl ethers telomeric alcohols and partly fluorinated carbon acid esters. The results are listed in table 3. Judging by data of this table 3 we can make a conclusion, that studied compounds are referred to III or IV class (Russian classification) of medium or low toxic compounds. It is stated, that dialkyl ethers do not locally affect skin and eye mucous membrane. Telomeric alcohols in sub-toxic dose (1000 mg/kg) bring on the suppression of respiratory center and also central nervous system, appearing as strong sedative effect and movement disorder of mice. These neurotoxic evidences are restored to physiological norm in 24 hours. When you rate the danger level of these compounds for a human being it is necessary to take into account their possible influence on myocardium function, thyroid gland, and also their gonado-and- genotoxic effect. This data allow using these compounds for production of fluoromaterials and for their production technology successfully.

Table 3. Some toxicological parameters of fluorine containing compounds[122,123].

Compound	LD₅₀ mg/kg	LD₁₀₀ mg/kg	Dangerous class
CF ₃ CHF ₂ CF ₂ OCH ₂ CF ₂ CHF ₂	5000		IV
HCF ₂ CF ₂ CH ₂ OCH ₂ CH ₃	3420	6000	III
HCF ₂ CF ₂ CH ₂ OH	2320	3000	III
HCF ₂ CF ₂ CF ₂ CF ₂ CH ₂ OH	1180	2000	III
CF ₃ CHFC(O)OC ₂ H ₅	5000		IV
HCF ₂ CF ₂ CH ₂ OCHF ₂	5000		IV
CH ₂ [OCH ₂ (CF ₂ CF ₂) ₂ H] ₂	5000		IV
CH ₂ [OCH(CH ₃)CF ₂ CF ₂ H] ₂	1917	5000	III
HCF ₂ CF ₂ C(CH ₃)=CH ₂	1334	3000	III
CF ₃ CHF ₂ CF ₂ CH ₂ OH	640	1000	III



Conclusion

The above material allows to establish the growing researchers' interest in working out of approaches of poly-fluorinated fragments application into organic molecules and approaches of simple substitutes transformation into complicated functional alignments. In this regard the use of partly fluorinated alcohols, obtained on the basis of polyfluoroolefines, opens up possibilities for new fluorine-containing materials synthesis. Much success, lately achieved in working out of partly fluorinated alcohols synthesis method, using which (p. f. Alcohols) you are able to carry out the fluorine- containing fragments application into organic molecules, displays that they in a few cases can be an alternative for classical and well-known methods. A number of processes have obvious advantages and real application opportunities in industrial technologies. There is no doubt, that issues, connected with ideas and processes realization, are of interest of both chemists, working in the field of fluoroorganic synthesis and specialists in the field of practical use of fluoromaterials on the base of fluorine organic compounds. In this review the author did his best to demonstrate new approaches and synthetic possibilities of new semi-products basically by example of telomeric alcohols, he also tried to display tendencies and main directions of researches in the field of synthesis of poly-fluoroorganic compounds, which contain different molecular frames and functional groups. Thus, commercialized by industry process of telomeric alcohols obtaining gave a stimulus of their wide and thorough research and synthesis of new fluorine materials, since such processes are rather simple and can be implemented on industrial scale/basis. We can hope, that later there will be revealed other reactions, resulting in fluorine-containing materials.

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