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A work of FSUE Russian Scientific Center "Applied Chemistry" in the field of synthesis of new heat-transfer agents and surfactants

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FSUE RSC "Applied chemistry" has invented the technology of synthesis of perfluorinated dielectric-heat transfers (PFDH) with wide range of application, working in different temperature intervals depending on their boiling points, freezing points and other physical and chemical characteristics.
Perfluorinated dielectric-heat transfers (PFDH) are inert, ecologically clean liquids and gases, nonflammable, fire-safe.
They are used for cooling of electronics and different elements of electrical equipment in a wide range of temperatures and thermal loads; as insulating substances in high-voltage electronic equipment; as a medium for the different methods of control in electrotechnics; as float liquids in different gyroscopic devices and for other purposes.
The change of transformer oil for perfluorinated dielectrics reduces volume and mass characteristics of electronic equipments in 10 and more times, decreases energy imputs for providing of heat pick-up in 100-1000 times, increases reliability of equipments.

These compounds obtain their properties such as chemical and biological inertness, thermophysical, dielectrical and other properties due to complete replacement of hydrogen atoms in their molecule for fluorine.
The discussed perfluorinated liquids are produced by means of electrochemical fluorination. This method was offered by Simons in 1949 and introduced at plants of 3M company.

In Russia the technology of electrochemical synthesis of fluorine compounds was developed most widely in FSUE RSC "Applied chemistry". The surfactant "chromine" for chrome plating tubs and inert dielectric liquids (perfluorodibutyl ether, perfluorotriethylamine, perfluorotributylamine) was synthesized and to apply in pilot-industrial plants.

Detailed investigation of main parameters of electrolysis, the development of continuous electrochemical fluorination method (long-term and stable electrolysis) and process implementation preceded the creation of pilot-industrial plants.

The variety of laboratory and industrial electrolyzes is developed, electrolysis parameters, influencing the power output of base compounds are defined.
Scientific research and experimental works carried out allowed us to create the industrial production of perfluorinated compounds, which can be used in a broad temperature range (from 30 to 180 ^{o}C). Their properties are shown in the table 1.
The investigations for development of high-boiling dielectric-heat transfers (perfluorotriamylamine (boiling point ~205^{o}C), perfluorotrihexylamine (boiling point~240^{o}C),

perfluorinated ethers) producing technologies has been started.
The development of surface-active materials on the basis of perfluorooctanesulfofluoride is carried out. Measuring of aqueous solutions (0,20-0,25%) of some samples showed that the coefficient of surface tension was 17-19 mN/m.
The given technology of perfluorinated compounds using electrochemical method can be implemented at any chemical plant, especially there production of other fluorine compounds and appropriate infrastructure exist.

The creation of two surface-active material classes and compositoins on their basis√ flactonits and epilames (efrens) was the result of long term researches, developed in FSUE RSC "Applied chemistry".

Flactonits are used in galvanic processes, fire-fighting, microelectronics, chemical and photographic industries.. The range of operating concentrations of flactonits in different technological processes is 0,5 √ 1000 mg per 1 liter.
Epilames are intended for creation of low-energy sorption layers of surfactants on the surface of hard objects. This allows to decrease coefficient of friction and wear of contacting surfaces and to increase their reliability of operation and working life.

Table 1. *Perfluorinated dielectric-heat-transfers, developed in FSUE RSC "Applied chemistry"*

Technical name	Main substance in product	Boiling point,^{o}C	Freezing point,^{o}C	d(20^{o}C) kr/m^3	Specific volume resistance, Om*m(20^{o}C)	Electric strength at frequency 50 Hz, KV/mm
PFDT-30	C_5F_{12}	25-35	-125	1660	$1,5*10^{13}$	30
PFDT-50	$CF_3(C_2F_5)_2N$	45-60	-163	1670	$1,5*10^{13}$	30
PFDT-70	$(C_2F_5)_3N$	65-71	-145	1750	$5*10^{13}$	30
PFDT-100	$(C_4F_9)_2O$	98-102	-70	1730	$4*10^{13}$	46
PFDT-130	$(C_3F_7)_3N$	125-132	-65	1840	$3*10^{14}$	44
PFDT-180	$(C_4F_9)_3N$	178-185	-55	1890	$4,8*10^{14}$	40-50
PFDT-205	$(C_5F_{11})_3N$	195-205	~ -50	~ 1900	-	40-50
PFDT-240	$(C_6F_{13})_3N$	235-245	~ -50	~ 1950	-	-

We will discuss in detail about some perfluorinated organic compounds used in industry which synthesis method was recently researched in the our laboratory.

1.Surfactants.

We have developed the method of obtaining of the following surface active materials: methylethanolamine salt of perfluorooctylsulfo-acid and ethylethanolamine salt of perfluorooctylsulfo-acid. Perfluorooctanesulfofluoride, which is the feed stock for the synthesis of discussed surfactants was synthesized by electrochemical fluorination method in the medium of liquid non-aqueous anhydrous hydrogen fluoride.
These preparations are assigned for extensive use as foaming agents during the process of nickel refining, chromium-plating and others.

2.Perfluoroethanesulfofluoride

Among important practical applications of electrochemical fluorination method there is synthesis of perfluoroalkanesulfofluorides and perfluoroalkanesulfo-acids. Such acids are used as super acids in the organic synthesis and their salts, as intermediates in the synthesis of surfactants. Perfluoroethanesulfofluoride is used during the synthesis of bis(pentafluoroethanesulfonyl)imide ,which there is the perspective material for obtaining electrolytes for lithium-ions batteries.

It was shown that ethanesulfofluoride was the most convenient initial substance for the synthesis of perfluoroethanesulfofluoride . The technology of ethanesulfofluoride producing using halogen exchange at catalyst was developed too.
Obtained perfluoroethanesulfofluoride had the purity 99,9%.
A pilot batches of perfluoroethanesulfofluoride was manufactured at the plant of FSUE RSC "Applied chemistry".

3. Perfluoropropylpentyl ether

Perfluorinated ethers are similar in their properties with perfluorocarbons, but unlike last mentioned they have lower freezing point and lesser viscosity. It greatly expands their field of application in different areas of technics. In Russia perfluorodibutyl ether was commercially produced , as perspective dielectric and heat transfer . However the technology of its obtaining included the addition of n-butylmercaptan as compound for anti-resinification. It using is not permitted now from toughened ecological requirements. The technology of obtaining of perfluoropropylpentyl ether having analogous properties with perfluorodibutyl ether was developed in FSUE RSC "Applied chemistry". This technology excludes using of mercaptan. Moreover, because of the fact, that partly fluorinated ethers are used as basic compound, the yield of final product and efficiency of the process greatly increase.

4. Perfluoromethylcyclohexane

The development of perfluorinated cyclic paraffin's obtaining technology is an important practical task, because fluorocarbons have unusual combination of physical and chemical properties: they have relatively low boiling points, which do not correspond with their molecular weight values, low critical temperatures and pressures, high liquid densities and coefficients of expansion, noticeably lower surface tensions and refraction coefficients than any other liquid has. The condensability of these liquids is higher than the one of most others and therefore the sonic speeds are really low. These compounds have good electric properties. Perfluoroalkanes have unique physical and chemical stability. Fluorocarbons are considered "almost nontoxic" from biochemical point of view, that means the lowest coefficient of toxicity (6).
Due to the peculiarity of technology the base product contains up to 20% of perfluorodimethylcyclopentanes, which form eutectic with the perfluoromethylcyclohexane. This lowers the freezing point up to - 65^{o}C (freezing point of pure perfluoromethylcyclohexane - 35^{o}C).

The development of perspective perfluorinated compounds and new electrolytic additives meeting all modern requirements are being continued.

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