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RESEARCH ARTICLE

Synthesis, physical properties, and practical applications of perfluorinated alkylmorpholines and alkylpiperidines obtained by electrochemical fluorination

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Abstract

Objectives. The work set out to describe the main physicochemical properties of new perfluorinated compounds (perfluoro-*N*-butylmorpholine, perfluoro-*N*-butylpiperidine, and perfluoro-*N*-ethylpiperidine) developed at the Russian Research Center "Applied Chemistry" (*GIPH*), and to evaluate the potential of their use as perfluorinated technical liquids by studying the temperature dependencies of their density, viscosity, and heat capacity in low temperature regions, as well as their applicability as media for syntheses involving strong Lewis acids using tris(pentafluoroethyl)difluorophosphorane as an example.

Methods. Perfluorinated tertiary amines (perfluorotriethylamine, perfluorotributylamine, perfluoro-*N*-butylmorpholine, perfluoro-*N*-butylpiperidine, perfluoro-*N*-ethylpiperidine) were synthesized by electrochemical fluorination in anhydrous hydrogen fluoride. The composition, purity, and structure of the perfluoroamines were determined by gas–liquid chromatography (Kristall 2000M) and confirmed by nuclear magnetic resonance spectroscopy (Bruker AVANCE III HD 400 MHz) and chromatography–mass spectrometry (Agilent Technologies 7890B/5977A). The thermal characteristics of the samples were determined by differential scanning calorimetry (NETZSCH DSC 214 Polyma). The density and kinematic viscosity of the samples were studied using a Stabinger viscometer (Anton Paar Stabinger Viscometer SVM 3000). Metal tris(pentafluoroethyl)trifluorophosphates were obtained by the fluorination of tris(pentafluoroethyl)difluorophosphorane by fluorides of the alkali metals—lithium, sodium, and potassium.

Results. Electrochemical fluorination produced *tert*-amines of various structures: perfluorotriethylamine, perfluorotributylamine, perfluoro-*N*-butylmorpholine, perfluoro-*N*-butylpiperidine, and perfluoro-*N*-ethylpiperidine with current yields from 29 to 61%. The dependencies of the heat capacity, density, and kinematic viscosity of new perfluorinated alkylmorpholines and alkylpiperidines in comparison with noncyclic perfluoroamines were studied over a wide temperature range. In the media of the obtained perfluoroamines, tris(pentafluoroethyl)difluorophosphorane salts of lithium, sodium, and potassium were synthesized at a yield of 38 to 95%.

Conclusions. The analysis of the physicochemical properties of heterocyclic perfluorinated *tert*-amines obtained in this work (perfluoro-*N*-butylmorpholine, perfluoro-*N*-butylpiperidine, and perfluoro-*N*-ethylpiperidine) indicates that these compounds are not inferior to noncyclic perfluorinated *tert*-amines in a number of characteristics, thus indicating their potential use as perfluorinated technical fluids thanks to their high heat capacity and potentially low conductivity. The key parameters determining their applicability as media for syntheses involving perfluorinated reagents are identified as physicochemical similarity, high density, and low viscosity at subzero temperatures.

Keywords

perfluorinated heat-transfer dielectrics, perfluoro-*N*-butylmorpholine, perfluoro-*N*-alkylpiperidines, tris(pentafluoroethyl)difluorophosphorane, perfluoroalkyl-substituted fluorophosphates, thermal properties, viscosity, density, heat capacity, differential scanning calorimetry, electrochemical fluorination

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НАУЧНАЯ СТАТЬЯ

Синтез, физические свойства и практическое применение перфторированных алкилморфолинов и алкилпиперидинов, получаемых электрохимическим фторированием

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Аннотация

Цели. Описать основные физико-химические свойства новых перфторированных соединений (перфтор-*N*-бутилморфолина, перфтор-*N*-бутилпиперидина, перфтор-*N*-этилпиперидина), разработанных в АО «РНЦ «Прикладная химия (ГИПХ)»; оценить возможность их использования в качестве технических жидкостей путем изучения профилей плотности, вязкости и теплоемкости в зависимости от температуры в областях низких температур, а также их применимость в качестве сред для проведения синтезов с участием сильных кислот Льюиса на примере трис(пентафторэтил)дифторфосфорана.

Методы. Перфторированные третичные амины (перфтортриэтиламин, перфтортрибутиламин, перфтор-*N*-бутилморфолин, перфтор-*N*-бутилпиперидин, перфтор-*N*-этилпиперидин) были синтезированы методом электрохимического фторирования в среде безводного фторида водорода. Состав, чистота и структура перфтораминов анализировались газожидкостной хроматографией (на приборе «Кристалл 2000М»), подтверждались спектроскопией ядерного магнитного резонанса (на приборе Bruker AVANCE III HD с рабочей частотой 400 МГц) и хроматомасс-спектрометрией (на приборе Agilent Technologies 7890B+5977A). Определение термических характеристик образцов проводили с помощью дифференциальной сканирующей калориметрии (на приборе Netzsch DSC 214 Polyma). Плотность и кинематическую вязкость образцов изучали с помощью вискозиметра Штабингера (на приборе Anton Paar Stabinger Viscometer SVM 3000). Трис(пентафторэтил)трифторфосфаты металлов получали реакцией фторирования трис(пентафторэтил)дифторфосфорана фторидами металлов первой группы — лития, натрия, калия.

Результаты. Синтезированы методом электрохимического фторирования *трет*-амины различной структуры: перфтортриэтиламин, перфтортрибутиламин, перфтор-*N*-бутилморфолин, перфтор-*N*-бутилпиперидин, перфтор-*N*-этилпиперидин с выходами по току от 29 до 61%. В широком диапазоне температур изучены зависимости теплоемкости, плотности и кинематической вязкости новых перфторированных алкилморфолинов и алкилпиперидинов в сравнении с нециклическими перфтораминами. В среде полученных перфтораминов синтезированы литиевые, натриевые и калиевые соли трис(пентафторэтил)дифторфосфорана с выходом по веществу от 38 до 95%.

Выводы. Проведенный анализ физико-химических свойств гетероциклических перфторированных *трет*-аминов — перфтор-*N*-бутилморфолина, перфтор-*N*-бутилпиперидина и перфтор-*N*-этилпиперидина, полученных в рамках данной работы, свидетельствует о том, что эти соединения по ряду показателей не уступают по свойствам нециклическим перфторированным *трет*-аминам и потенциально могут использоваться в качестве технических жидкостей благодаря высокой теплоемкости и потенциально низкой проводимости. Показано, что ключевыми параметрами, определяющими их применимость в качестве сред для синтезов с участием перфторированных реагентов, является их физико-химическое подобие, высокая плотность, а также низкая вязкость при отрицательных температурах.

Ключевые слова

перфторированные диэлектрики-теплоносители, перфтор-*N*-бутилморфолин, перфтор-*N*-алкилпиперидины, трис(пентафторэтил)дифторфосфоран, перфторалкилзамещенные фторфосфаты, термические свойства, вязкость, плотность, теплоемкость, дифференциальная сканирующая калориметрия, метод электрохимического фторирования

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INTRODUCTION

The Russian Research Center "Applied Chemistry" (*GIPH*) is known for having pioneered the development of electrochemical fluorination (ECF) in Russia. Research on ECF of both organic and inorganic compounds, which began over 60 years ago in *GIPH* laboratories, has now been implemented at a small-scale pilot plant [1]. Today, the ECF method, also known as the Simons method, is widely used as a universal method for producing a number of perfluorinated compounds.

A class of such perfluorinated compounds in industrial demand are perfluorinated *tert*-amines: perfluorotriethylamine(MD-3F),perfluorotripropylamine (PFTPA), perfluorotributylamine (BAF-3), and the Fozhalin product. However, the starting compounds (triethylamine, tripropylamine, tributylamine, and triallylamine) for the synthesis of perfluoroamines are currently not produced in Russia. The problem of selecting optimal and readily available starting materials for the synthesis of new perfluorinated compounds by ECF can be solved by using Russian raw materials, such as pyridine and morpholine or their derivatives.

We have developed a method for preparing starting compounds based on pyridine and morpholine—namely, quaternary *N*-alkylpyridinium salts and tertiary *N*-alkylmorpholinium salts—using the quaternization reaction. ECF of these salts in anhydrous hydrogen fluoride gives tertiary heterocyclic perfluorinated amines: perfluoro-*N*-butylmorpholine (*N*-BuMorph), perfluoro-*N*-butylpiperidine (*N*-BuPip), and perfluoro-*N*-ethylpiperidine (*N*-EtPip) [2].

Previously, *GIPH* developed a technology for producing nonflammable, heat- and frost-resistant, and chemically stable fluorinated dielectric fluids operating over a wide temperature range, primarily perfluoroamines of various compositions and perfluoropentane. The technology is based on the synthesis of perfluorinated fluids by ECF of hydrocarbon analogs in a medium of liquid anhydrous hydrogen fluoride [3, 4].

Perfluorinated *tert*-amines, such as MD-3F and BAF-3, are inert transparent liquids that remain stable over a wide temperature range. Due to their inertness, these compounds do not interact with metals, solid dielectrics, or rubbers, and do not dissolve in oils or water. Moreover, temperature changes have virtually no effect on the viscosity of these compounds, which exhibit low compressibility, stable dielectric properties, and low toxicity. These characteristics make perfluoroamines suitable for use as perfluorinated dielectric fluids [5]. Some areas of application of these compounds in technology include their use as hydraulic fluids and working fluids in a wide range of gyroscopic devices [6], as well as solvents, extractants for fluorinated and fluorine-labeled substances, and synthesis media [7–10], and for ultrasound diagnostics and fluorine-19 magnetic resonance imaging (^{19}F MRI) [11]. Since these perfluorinated liquids dissolve oxygen and other gases well, they can be used as gas-carrying media for transporting oxygen to cells in living organisms and for removing carbon dioxide from the blood [12].

Due to their unique heat and frost resistance and thermal stability over a wide range of operating temperatures, perfluoroamines such as MD-3F and BAF-3 are currently the most widely used perfluorinated technical fluids in Russia (Table 1).

Due to economic and geopolitical circumstances, the development of new perfluorinated technical fluids that are not inferior in performance to already in-demand perfluoroamines and can be obtained from commercially available Russian raw materials becomes a pressing research challenge. In this connection, it is of interest to study the influence of the length of perfluorinated substituents in their structure on the physicochemical properties of promising technical fluids, as well as the influence of replacing the perfluorinated methylene fragment with an oxygen ether fragment.

In this study, we investigated the physicochemical properties of new perfluorinated compounds obtained by ECF: perfluoro-*N*-butylmorpholine, perfluoro-*N*-butylpiperidine, and perfluoro-*N*-ethylpiperidine.

Table 1. Rated physicochemical properties of perfluorinated technical fluids produced by *GIPH*

Parameters	Trade name of perfluorinated liquid (perfluorinated heat-transfer dielectric)							
	PFDT-30 (PFP)	PFDT-50 (MD-46)	PFDT-70 (MD-3F)	PFDT-100 (DEF)	PFDT-130 (PFTPA)	Fozhalin	PFDT-180	
							BAF-3	BAF-85
	Main substance in product							
	Perfluoro-pentane	Perfluoro-methyldiethylamine	Perfluoro-triethylamine	Perfluoro-dibutyl ether	Perfluoro-tripropylamine	Fozhalin	Perfluoro-tributylamine	
Distillation range, °C	25–35	45–50	65–71	98–102	125–132	125–132	178–185	150–185
Solidification temperature, °C	−125	−163	−145	−110	−60	−110	−50	−55
Density (20°C), kg/m^3	1660	1670	1750	1730	1800–1850	1830–1850	1890	
Kinematic viscosity (20°C), mm^2/s, max	0.4	0.5	0.6	0.76	0.93	1.1	3.3	

MATERIALS AND METHODS

The perfluorinated *tert*-amines *N*-BuMorph, *N*-BuPip, and *N*-EtPip were synthesized during the course of the present work. The purity, composition, and structure of the samples were studied by gas–liquid chromatography with a Kristall 2000M chromatograph (*Khromatek*, Russia), nuclear magnetic resonance spectroscopy (NMR) with an AVANCE III HD 400 MHz NMR spectrometer (*Bruker*, USA), and chromatography–mass spectrometry with a 7890B/5977A system (*Agilent Technologies*, USA). The thermal studies were carried out with a DSC 214 Polyma differential scanning calorimetry (DSC) instrument (*NETZSCH*, Germany) in the temperature range from −150 to +110°C in a sealed crucible (isochoric conditions). The temperature dependence of the heat capacity of perfluorinated *tert*-amines was calculated using a comparison with a sapphire standard over the temperature range from the highest glass transition temperature (T_g) to 110°C, ignoring exothermic events of phase transitions (crystal lattice changes and melting). The density and kinematic viscosity were measured with a Stabinger Viscometer SVM 3000 (*Anton Paar*, Austria) over the temperature range from −35 to +45°C.

Bromides of *N*-butylmorpholinium, *N*-butylpyridinium, and *N*-ethylpyridinium were prepared by the quaternization of morpholine (CAS 110-91-8) and pyridine (CAS 110-86-1) with the alkyl halide 1-bromobutane (CAS 109-65-9) or 1-bromoethane (CAS 74-96-4) in dimethylformamide (CAS 68-12-2) according to a previously published procedure [2]. The starting compounds triethylamine (CAS 121-44-8), tributylamine (CAS 102-82-9), and anhydrous hydrogen fluoride (CAS 7664-39-3) are commercially available reagents.

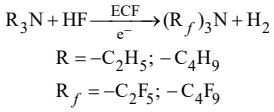
Lithium, sodium, and potassium tris(pentafluoroethyl)-trifluorophosphates were obtained by fluorinating tris(pentafluoroethyl)difluorophosphorane (CAS 91543-32-7) with Group I metal fluorides—LiF (CAS 7789-24-4), NaF (CAS 7681-49-4), and KF (CAS 7789-23-3)—in a perfluorinated *tert*-amine medium.

Synthesis of perfluorinated tertiary amines

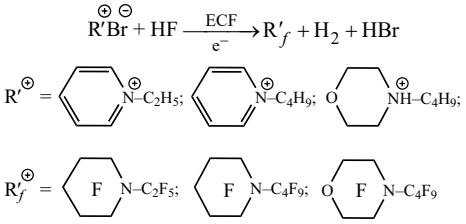
Samples of perfluorinated *tert*-amines with sufficiently high current yields were obtained by ECF (Table 2). The fractions of the target products isolated by distillation were subjected to a series of studies to determine their main physicochemical properties.

ECF was carried out under standard conditions (direct current, current density 0.02 A/cm^2, voltage 4.0–7.0 V, electrolyzer temperature from 15 to 20°C, organic matter concentration in the electrolyte from 10 to 15%) in a 0.75-L Simons-type steel electrolytic cell with nickel electrodes using the procedure described in the literature [2, 13].

Schemes 1 and 2 present the overall reactions of the ECF synthesis of a series of perfluorinated *tert*-amines: triethylamine, tributylamine, *N*-ethylpyridinium bromide, *N*-butylpyridinium bromide, and *N*-butylmorpholinium bromide.



Scheme 1. ECF synthesis of MD-3F and BAF-3

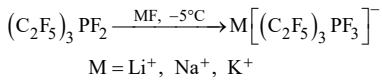


Scheme 2. ECF synthesis of *N*-EtPip, *N*-BuPip, and *N*-BuMorph

The reactions shown in Schemes 1 and 2 produce not only target perfluorinated compounds, but also byproducts from degradation, isomerization, and partial fluorination of the starting molecule, as well as nitrogen trifluoride NF_3 and perfluorocarbons with carbon chain lengths of C_1–C_5. The perfluoroamines obtained in the ECF stage were isolated from the crude products by distillation (>97.5 wt %, gas chromatography–mass spectrometry).

Synthesis of metal tris(pentafluoroethyl)-trifluorophosphates

Lithium, sodium, and potassium tris(pentafluoroethyl)-trifluorophosphates (LiFAP, NaFAP, and KFAP, respectively) were synthesized in a medium of perfluorinated *tert*-amines obtained in the ECF stage: BAF-3, MD-3F, *N*-BuMorph, *N*-BuPip, and *N*-EtPip (Scheme 3).



Scheme 3. Synthesis of metal tris(pentafluoroethyl)-trifluorophosphates

The synthesis was carried out at −5°C in a Peltier-cooled thermostat with stirring. The synthesis process involved two stages: first, the fluorides were thermostatted and dispersed ultrasonically in the corresponding perfluoroamine at −5°C for 1 h; second, perfluoroethylphosphorane was added to the suspension, and the mixture was kept cool and stirred for additional 3 h. The solution became clear and homogeneous, indicating the mutual solubility of the new perfluorinated liquids, reagents, and products. The solvent and residual phosphorane were then removed by vacuum distillation. The residue was weighed, and the conversion was calculated, taking into account unreacted metal fluorides.

RESULTS AND DISCUSSION

Both noncyclic and heterocyclic perfluorinated *tert*-amines were synthesized by ECF with current yields from 29 to 61% (Table 2).

Table 2. Current yields of ECF of *tert*-amines

Initial compound	Product	Current yield, %
$(C_2H_5)_3N$	MD-3F	61
$(C_4H_9)_3N$	BAF-3	45
$[C_5H_5N^+C_2H_5]Br^-$	*N*-BuMorph	54
$[C_5H_5N^+C_4H_9]Br^-$	*N*-BuPip	42
$[C_4H_9ON^+C_4H_9]Br^-$	*N*-EtPip	29

Our results on current yields of fluorination of tertiary amines of both linear and cyclic structures demonstrate that the current yield depends significantly on the structure of the starting compound. For example, the highest current yield is observed for the fluorination of triethylamine (61%), where side processes of degradation and isomerization are minimal. Increasing the hydrocarbon chain length and the presence of an oxygen bridge (–O–) in the structure of the starting compound complicate the complete fluorination process and, as a rule, increase the susceptibility to side processes of degradation under ECF conditions, which, in turn, leads to a decrease in the yield of the target products.

The fluorination of heterocyclic structures occurs with moderate current yields (29–54%), comparable to that of the noncyclic analog BAF-3 (45%). This indicates the fundamental possibility of using pyridinium and morpholinium salts as readily available in Russia raw materials for the synthesis of perfluorinated *tert*-amines. The structure of the obtained perfluorinated *tert*-amines was established by NMR spectroscopy and confirmed by mass spectroscopy (Fig. 1).

Perfluoro-*N*-ethylpiperidine. ^{13}C NMR (101 MHz, DMSO-d_6), δ, ppm: 102.4–114.6 (m, $\underline{C}F_2$), 116.0 (qt, J = 284.6, 42.8 Hz, $\underline{C}F_3$). ^{19}F NMR (377 MHz, DMSO-d_6), δ, ppm: −89.36 (3F, p, J = 8.6 Hz, $C\underline{F}_3$), −93.58 (4F, m, $C^{2,6}_{Pip}F_2$), −97.39 (2F, ddd, J = 42.2, 23.4, 18.7 Hz, $CF_3C\underline{F}_2$), −134.69 (4F, m, $C^{3,5}_{Pip}F_2$), −137.07 (2F, m, $C^{4}_{Pip}F_2$).

Perfluoro-*N*-butylpiperidine. ^{13}C NMR (101 MHz, DMSO-d_6), δ, ppm: 102.58–117.4 (m $\underline{C}F_2$), 116.62 (qt, J = 286.8, 33.1 Hz, $\underline{C}F_3$). ^{19}F NMR (376 MHz, DMSO-d_6), δ, ppm: −84.00 (3F, t, J = 10.5 Hz, $C\underline{F}_3$),

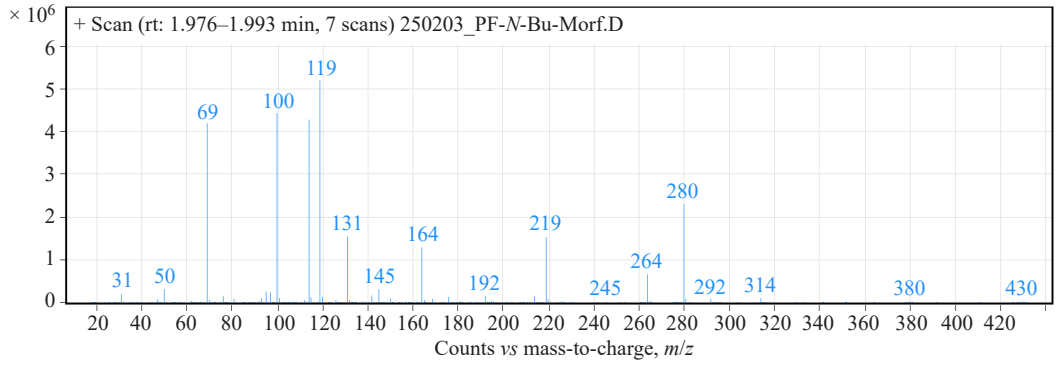
−91.17 (2F, m, α-CF_2), −93.01 (4F, m, $C^{2.6}_{Pip}F_2$), −125.76 (2F, m, β-CF_2), −128.86 (2F, m, γ-CF_2), −134.48 (4F, m, $C^{3.5}_{Pip}F_2$), −136.90 (2F, m, $C^{4}_{Pip}F_2$).

Perfluoro-*N*-butylmorpholine. ^{13}C NMR (101 MHz, DMSO-d_6), δ, ppm: 104.6–114.5 (m, $\underline{C}F_2$), 112.12 (tt, J = 273.6, 32.1 Hz), 113.77 (tt, J = 286.8, 33.3 Hz), 116.56 (qt, J = 285.4, 32.3 Hz). ^{19}F NMR (376 MHz, DMSO-d_6), δ, ppm: −84.00 (3F, tt, J = 10.5, 2.4 Hz, $C\underline{F}_3$), −89.82 (4F, m, $C^{2.6}_{Morph}$), −92.30 (2F, m, α-CF_2), −94.33 (4F, m, $C^{3.5}_{Morph}$), −125.95 (2F, m, β-CF_2), −128.85 (2F, m, γ-CF_2).

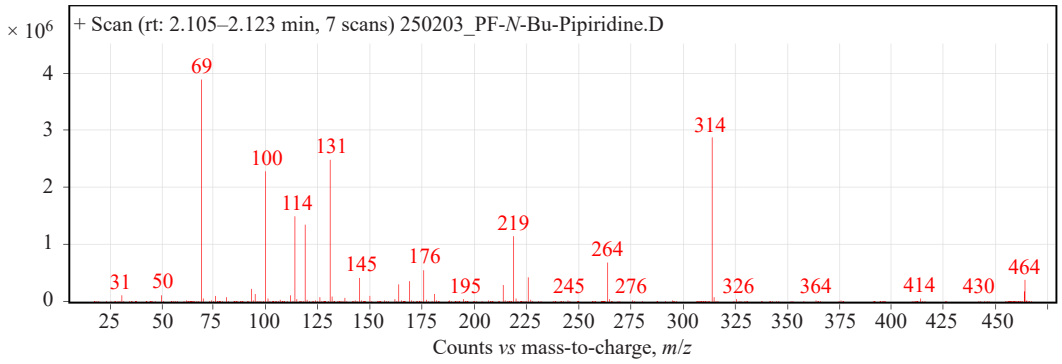
Note that the range of applications of perfluorinated heat-transfer dielectrics is exceptionally broad. However, their application range in various temperature ranges depends on their boiling and freezing points, and other physicochemical properties (Table 1).

DSC analysis of the samples (Fig. 2, Table 3) showed that, at extremely low temperatures, the samples behave like semicrystalline polymers—the glass transition temperatures of *N*-BuMorph, *N*-BuPip, and *N*-EtPip are below −120°C.

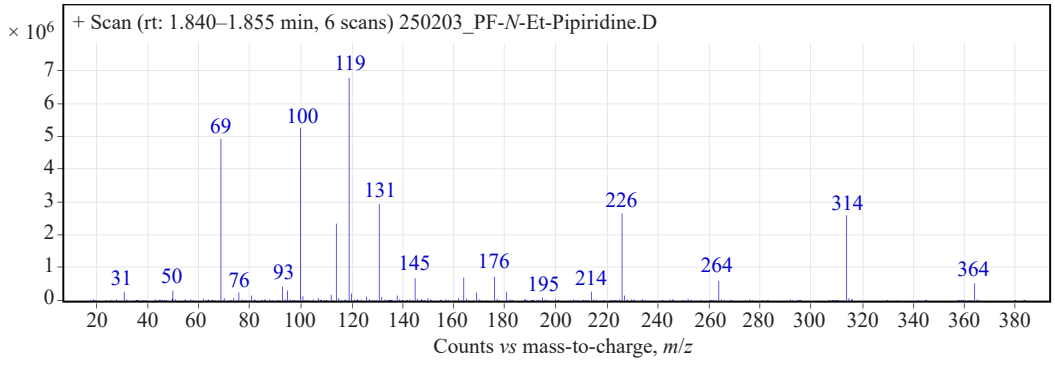
Above the glass transition temperatures, the structures of the described compounds undergo structural rearrangements with the corresponding exothermic effects, leading to increased microcrystallinity and, with a further increase in temperature, to melting. The detected thermal effects corresponding to melting are higher than the exothermic effects of crystallization,



(a)



(b)



(c)

Fig. 1. Mass spectra of (a) *N*-BuMorph, (b) *N*-BuPip, and (c) *N*-EtPip samples

which also supports the assumption of the semicrystalline state of the polymer in the glassy phase. Moreover, *N*-BuMorph exhibits multiple crystallization regions, while perfluoro-*N*-alkylpiperidines exhibit only one. This may be explained by the contribution of oxygen to the coordination of the forming chains. Moreover, for the substituted morpholine, the phase transition enthalpies are significantly higher, further supporting the assumption of the significant influence of the heteroatom within the molecule on coordination. It should be noted that the comparison samples (MD-3F, BAF-3) demonstrate the corresponding phase transitions prior to melting of the crystalline phase. However, the endothermic and exothermic effects for them are expressed weakly, which indicates a lower degree of their coordination due to steric hindrance caused by the three bulky perfluorinated substituents. Increasing the length of the substituent at the nitrogen atom predictably increases the boiling, melting, and glass transition temperatures.

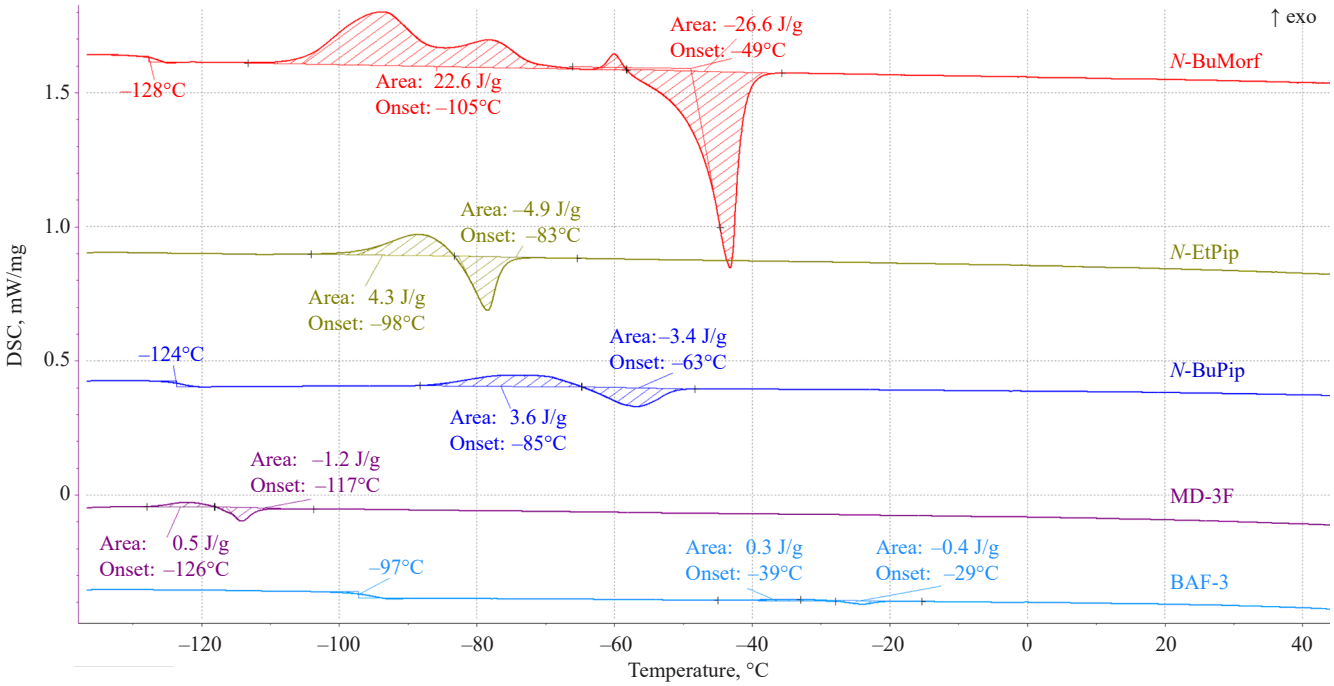


Fig. 2. DSC curves for *N*-BuMorph, *N*-EtPip, *N*-BuPip, MD-3F, and BAF-3 samples

Table 3. Results of thermal analysis of BAF-3, MD-3F, *N*-BuMorph, *N*-BuPip, and *N*-EtPip samples

Sample	Molar mass, g/mol	Highest found glass transition temperature, °C (Change in heat capacity in glass transition ΔC_p, J/g·K)	Phase transition temperature, °C	Melting point, °C (Enthalpy of melting, J/g)
BAF-3	670.96	−97.2 (0.135)	−38.9	−28.5 (0.44)
MD-3F	370.98	−148.1 (0.17)	−126.4	−116.5 (1.15)
N-BuMorph	448.97	−127.7 (0.15)	−105.4	−48.9 (26.58)
N-BuPip	482.97	−123.7 (0.131)	−85.2	−62.7 (3.41)
N-EtPip	382.98	−147.3 (0.175)	−97.5	−82.7 (4.90)

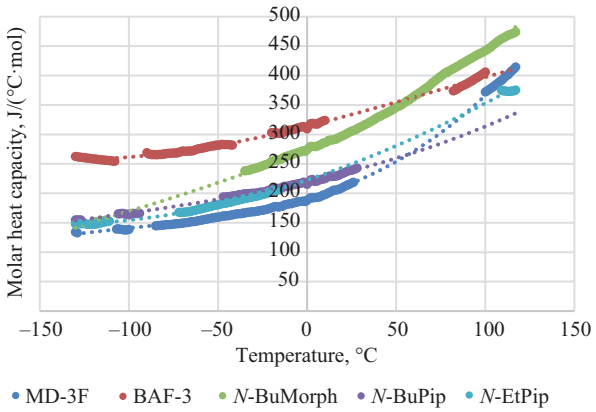


Fig. 3. Temperature dependencies of the molar heat capacity of MD-3F, BAF-3, *N*-BuMorph, *N*-BuPip, and *N*-EtPip samples

The calculated DSC dependencies of heat capacity on temperature (Fig. 3, Table 4) are within the expected limits described previously for perfluorinated compounds [5]. BAF-3, which has a high heat capacity at low temperatures, and is characterized by a moderate increase in heat capacity with increasing temperature. For MD-3F, conversely, the heat capacity increases more rapidly with increasing temperature. A similar dependence is observed when comparing *N*-perfluoroalkyl-substituted perfluoropiperidines. Thus, the length of the perfluorinated substituent directly affects the value of the molar heat capacity, but with increasing temperature, other factors begin to have a stronger influence on the increase in heat capacity. Among such factors are, for example, the conformational flexibility of the molecular chain and the

Table 4. Coefficients of the cubic equations for the temperature dependencies of the heat capacity of BAF-3, MD-3F, *N*-BuMorph, *N*-BuPip, and *N*-EtPip samples

Function of specific heat capacity after glass transition (excluding phase transitions)					
Sample	*a* (x^3)	*b* (x^2)	*c* (x)	*d* (const)	R^2
BAF-3	−3.00E-09	3.00E-06	0.0003	0.1999	0.9967
MD-3F	5.00E-08	−3.00E-06	0.0008	0.3525	0.9986
N-BuMorph	2.00E-08	−1.00E-06	0.0021	0.313	0.9985
N-BuPip	1.00E-08	4.00E-08	0.0009	0.317	0.9964
N-EtPip	−5.00E-09	1.00E-05	0.0003	0.3854	0.9994
Function of molar heat capacity after glass transition (excluding phase transitions)					
Sample	*a* (x^3)	*b* (x^2)	*c* (x)	*d* (const)	R^2
BAF-3	−8.00E-06	0.0015	0.7558	314.12	0.9964
MD-3F	2.00E-05	0.0063	0.8995	190.26	0.9996
N-BuMorph	9.00E-06	0.0031	1.2769	275.09	0.9985
N-BuPip	5.00E-06	0.002	0.6858	219.57	0.9964
N-EtPip	−2.00E-06	0.0031	1.0118	222.56	0.9994

activation of low-frequency vibrational modes, which make an increasing contribution to the total energy of the system. *N*-BuMorph, in turn, significantly changes its molecular dynamics with increasing temperature due to its peculiar "switching on" of degrees of freedom and more flexible carbon skeleton. The relationships found in our studies ($y = ax^3 + bx^2 + cx + d$) are characterized by the lowest extrapolated heat capacity at 0 K (the smallest constant term d (const)) for MD-3F and the smallest minimum increase in the linear coefficient c for *N*-BuPip.

In terms of specific heat capacity, MD-3F and *N*-EtPip exhibit similar thermophysical properties, while the industrial use of *N*-BuMorph may be complicated by significant changes in properties (the equation for *N*-BuMorph has the largest linear coefficient c).

The obtained density and viscosity values are directly related to the molecular weight and more compact molecular packing (Fig. 4). The linear coefficient shows the thermal expansion values: the highest thermal expansion value was found for MD-3F; and the lowest, for BAF-3 and *N*-BuPip. A linear dependence ($y = kx + b$) is observed for density values (the coefficients are given in Table 5), while, for molar heat capacity, a power-law dependence is observed (the coefficients are given in Table 6).

Table 5. Coefficients of linear dependencies of density on temperature for BAF-3, MD-3F, *N*-BuMorph, *N*-BuPip, and *N*-EtPip samples

Sample, atm. pressure, kPa	k (x)	b	R^2
BAF-3, 101.0 ± 0.1	−0.0021	1.9270	0.9998
MD-3F, 100.0 ± 0.1	−0.0028	1.8074	0.9994
N-BuMorph, 100.3 ± 0.1	−0.0023	1.8608	0.9998
N-BuPip, 99.8 ± 0.1	−0.0021	1.8739	0.9987
N-EtPip, 100.0 ± 0.1	−0.0026	1.8312	0.9998

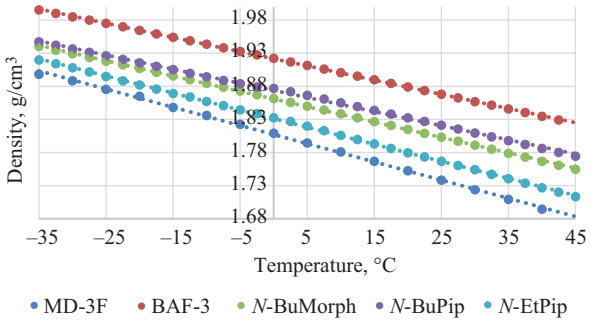


Fig. 4. Temperature dependencies of the density of MD-3F, BAF-3, *N*-BuMorph, *N*-BuPip, and *N*-EtPip samples

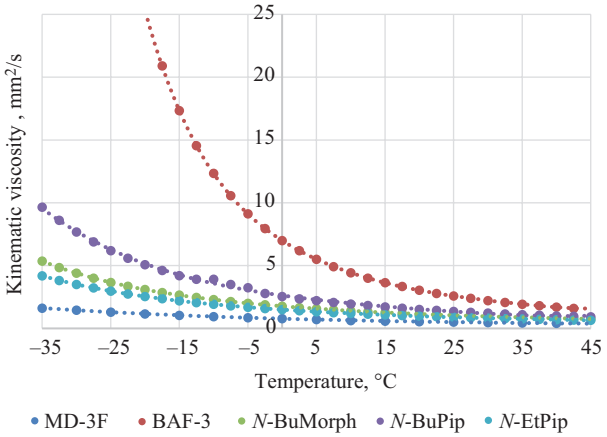


Fig. 5. Temperature dependencies of the kinematic viscosity of MD-3F, BAF-3, *N*-BuMorph, *N*-BuPip, and *N*-EtPip samples

For the studied perfluorinated liquids (in pairs MD-3F/BAF-3 and *N*-EtPip/*N*-BuPip), a clear correlation was established between the length of the molecular chain of the substituent and their macroscopic properties: an increase in the chain length naturally leads to a simultaneous increase in the structural characteristics (density and viscosity) (Figs. 4 and 5; Tables 5 and 6) and energy capacity (molar heat capacity) (Fig. 3, Table 4).

The introduction of an oxygen bridge (–O–) in place of a methylene group (–CH_2–) into the structure of a perfluorinated compound (in the *N*-BuMorph/*N*-BuPip pair) leads to a significant reduction in density, molar heat capacity (at low temperatures), and viscosity. The ether bond increases the average distance between molecules, reduces the total number of low-frequency vibrational modes, and creates more rigid and directional intermolecular interactions; however, the total energy is lower than the combination of dispersion forces in the hydrocarbon analog. Furthermore, the oxygen bridge lowers the energy barrier to the internal motion of molecular parts around the C–O bonds. At low temperatures, these rotations are "frozen." As the temperature increases, they "turn on" and begin to significantly contribute to the heat capacity, accelerating the increase in heat capacity with increasing temperature.

Perfluorinated compounds can be used as media for organic synthesis. To study the basicity properties of *N*-BuMorph, *N*-BuPip, and *N*-EtPip, a series of additional syntheses were performed using the obtained heavy perfluorinated liquids.

Fluorination reactions of the strong Lewis acid tris(pentafluoroethyl)difluorophosphorane (fluoroalkyl phosphate (FAP) anion) with Group I metal fluorides (lithium, sodium, and potassium) were selected as model reactions for optimizing the previously developed technology framework [14]. It has previously been shown that lowering the synthesis temperature significantly increases the product yield; however, the synthesis media used either tend to form complexes with phosphorane or dissolve it only sparingly [15, 16]. We found that perfluoroethylphosphorane is virtually completely miscible with all the studied perfluorinated liquids, as well as exhibiting sufficient power to dissolve the target metal perfluorophosphates. An important property explaining the selection of these substances as synthesis media is their low viscosity at subzero temperatures, low volatility, and their ability to be easily regenerated. The obtained results can be used to develop effective methods for producing salts based on the FAP anion suitable for practical application in electrochemical devices, as phase-transfer catalysts, and as dispersants for polyfluorinated compounds in polar solvents.

The yields of reactions according to Scheme 3 (Table 7) were calculated based on the increase (change) in the test tube weight after complete removal of the liquid phase by evacuation without further purification of the resulting products.

The highest product yield was observed in MD-3F, while, among the new perfluorinated liquids studied, the highest yield was observed in *N*-BuMorph, which, as shown above, according to the DSC results, has potential for coordination interactions that facilitate the reaction under study. In this work, it is established that the tertiary nitrogen atom in perfluorinated compounds did not form salts with the strong Lewis acid used; the presence of an additional potential oxygen Lewis base in the solvent had a beneficial effect on the product yields

Table 6. Coefficients of temperature dependencies of density for BAF-3, MD-3F, *N*-BuMorph, *N*-BuPip, and *N*-EtPip samples

Kinematic viscosity function, mm^2/s						
Sample	*a* (x^4)	*b* (x^3)	*c* (x^2)	*d* (x)	*e* (const)	R^2
BAF-3	−2.74E-06	2.72E-04	2.82E-03	−3.60E-01	7.057	0.9999
MD-3F	9.00E-09	−2.00E-06	2.00E-04	−1.48E-02	0.7755	0.9997
N-BuMorph	2.00E-07	−2.00E-05	8.00E-04	−4.14E-02	1.7615	0.9999
N-BuPip	5.00E-07	−4.00E-05	1.60E-03	−7.54E-02	2.6259	0.9984
N-EtPip	1.00E-07	−1.00E-05	6.00E-04	−3.43E-02	1.4926	0.9999

Table 7. Yields of the target products depending on the solvent

Solvent	Product yield, %		
	LiFAP	NaFAP	KFAP
BAF-3	38	58	51
MD-3F	71	92	95
N-BuMorph	68	90	89
N-BuPip	56	87	81
N-EtPip	45	91	77

in comparison with the solvent that did not contain it, which is fully consistent with the literature data on the basicity of perfluoroamines and perfluoropolyethers [8]. The low yields of the lithium salt are also explained by its intrinsic properties. According to our observations, the instability constant of this complex salt is significantly lower than those of salts of another Group I metals. In addition, the product yield is expectedly affected by the ease of further purification. The advantage of using MD-3F as a synthesis medium is explained by its lowest boiling point, by its lowest viscosity under the described conditions, which ensures good mass transfer, and by its moderate solvating capacity, which does not suppress the reactivity of ionized intermediates formed by the Lewis acid.

CONCLUSIONS

For new perfluorinated tertiary amines (*N*-BuMorph, *N*-BuPip, and *N*-EtPip) obtained by electrochemical fluorination from readily available Russian raw materials, the key thermal characteristics (melting point, glass transition temperature, and phase transition temperature) were determined to characterize the temperature dependencies of density and kinematic viscosity. The key properties of perfluoroamines in tris(pentafluoroethyl)-difluorophosphorane fluorination reactions were studied.

The proposed methods for studying the physicochemical properties demonstrated the theoretical feasibility of using the new perfluorinated compounds as coolants and other fluids for technical devices that are capable of operating across various temperature ranges. The heterocyclic perfluorinated *tert*-amines are not inferior in a number of properties to known noncyclic perfluorinated *tert*-amines.

The results of the study suggest that the perfluorinated *tert*-amines *N*-BuMorph, *N*-BuPip, and *N*-EtPip obtained in *GIPH* can be used as perfluorinated technical fluids and synthesis media and possess properties similar to well-studied tertiary perfluorinated alkylamines, which supports the advisability of further substantiating their use as dielectrics.

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Authors' contributions

E.V. Litvinenko—conducting experimental research on ECF, collecting and processing material, analysis of literary sources, writing and editing the text of the article.

N.B. Lesnevskaya—developing a methodology and conducting experimental studies on ECF of tertiary amines.

A.A. Ludikainen—conducting experimental studies, developing a methodology for obtaining starting compounds for synthesis by ECF.

V.A. Matalin—developing a concept of scientific work and methodology for ECF of tertiary amines, editing and critical revising the article.

I.A. Kulik—conducting experimental studies, synthesis of starting compounds for ECF.

I.G. Mokrushin—conducting experimental and analytical studies, analyzing literary sources, developing conditions for the synthesis of perfluoroalkyl substituted metal fluorophosphates, processing and interpreting the results, writing and editing the text of the article.

The authors declare no conflicts of interest.

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[Original Russian Text: Matalin V.A., Kaurova G.I., Berenblit V.V., Gribel' V.I. Electrochemical fluorination of toluene and benzotrifluoride in the presence of triallylamine. *Zhurnal prikladnoi khimii.* 2007;80(12):2012–2014 (in Russ.). https://www.elibrary.ru/iirwmh]
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