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O.S. Sverdlikovska, M.O. Potapchuk

IONIC CONDUCTIVITY AND GLASS TRANSITION TEMPERATURE OF IONENE-TYPE POLYMERIC IONIC LIQUIDS

Educational and Scientific Institute «Ukrainian State University of Chemical Technology», Ukrainian State University of Science and Technologies, Dnipro, Ukraine

The influence of the polymeric nature of synthesized ionene-type polymeric ionic liquids based on tetrahydro-1,4-oxazine on their ionic conductivity and glass transition temperature has been investigated. Correlations between temperature, chemical structure, ionic conductivity and glass transition temperature of these ionene-type polymeric ionic liquids were established, contributing to the design principles for new polymeric and ionene-type ionic liquids with tailored properties. The identified relationships were confirmed by correlation equations, and their chemical nature was analyzed. The proposed guidelines demonstrate practical value for the development of tetrahydro-1,4-oxazine-based ionene-type polymeric ionic liquids with high ionic conductivity across a wide temperature range.

Keywords: polymeric ionic liquids, ionic liquids, ionic conductivity, glass transition temperature, structure–property relationships.

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Introduction

Current research reveals a number of important challenges that require a systematic approach to implement the strategic direction of development of wide industrial implementation of ionic liquids [1]. Optimization of synthesis methods and development of cost-effective technologies for the production of ionic liquids remain priority tasks for reducing the cost of the final product [2,3].

The unique complex of physicochemical properties of ionic liquids, including the effective combination of high ionic conductivity with thermal stability, low vapor pressure and the ability to predict characteristics, determines the introduction of these materials as indispensable components of modern technological processes [4]. The results of the application of ionic liquids in electrochemical devices are particularly promising, where a significant increase in the efficiency and stability of the systems has been achieved [5,6]. An important aspect is the ability of ionic liquids to ensure the environmental safety of processes while maintaining high productivity, which fully complies with the principles of green chemistry [7].

Analysis of current trends and scientific achievements allows us to identify key areas for future research on ionic liquids [8,9]. The development of new types of functionalized ionic liquids with predicted improved properties and expanded application possibilities remains a priority area of research. The creation of intelligent materials and adaptive systems based on ionic liquids opens up new prospects for innovative industrial implementations [10–12].

Experimental

In this work, new polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine derivatives were prepared using the synthesis method described in ref. [13]. The synthesis of polymer ionic liquids of the ionene type was carried out by the reaction of tertiary diamines based on tetrahydro-1,4-oxazine derivatives with dihalogen derivatives based on difunctional epoxidized compounds in an ethanol–water solvent of variable composition at a temperature of $50^{0}C$ for 20 hours according to the general scheme shown in Fig. 1.

Dihalogen derivatives based on difunctional epoxidized compounds of the grades EX-850 (DH-6), EX-821 (DH-7), EX-830 (DH-8), EX-841 (DH-9), and EX-861 (DH-10) were used as starting monomers for the synthesis of tertiary diamines (TDA-6, TDA-7, TDA-8, TDA-9, TDA-10, respectively) and as monomers for the synthesis of polymer ionic liquids of the ionene type.

The structure of the synthesized polymer ionic liquids of the ionene type was confirmed using IR spectroscopy and elemental analysis (Table 1).

The objective of the research conducted in this study was to formulate recommendations for the development of novel polymeric ionic liquids of the ionene type, based on tetrahydro-1,4-oxazine. These liquids are characterized by high ionic conductivity over a broad temperature range. This is attributable to the judicious selection of structural and chemical characteristics of ionic liquid carriers, ensuring their high efficiency across a range of practical applications. Furthermore, this study enables the determination of the optimal operating modes of ionic liquids within technological systems. To achieve this objective, the following tasks were addressed:

– to determine the influence of the structure of substituents at the quaternary Nitrogen atom, the size of the molecule, the number of charges and the distance between the quaternary Nitrogen atoms in the macromolecular chain of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine on their ionic conductivity and glass transition temperature;

– to present the dependences between the chemical structure of polymer ionic liquids of the ionene type, temperature and their technologically important parameters.

Generalizations of the results of previous works [14] are expedient from a practical point of view and allow a reasonable approach to the choice of the objects of this study - samples of polymeric ionic liquids of the ionene type with chloride anions (having the

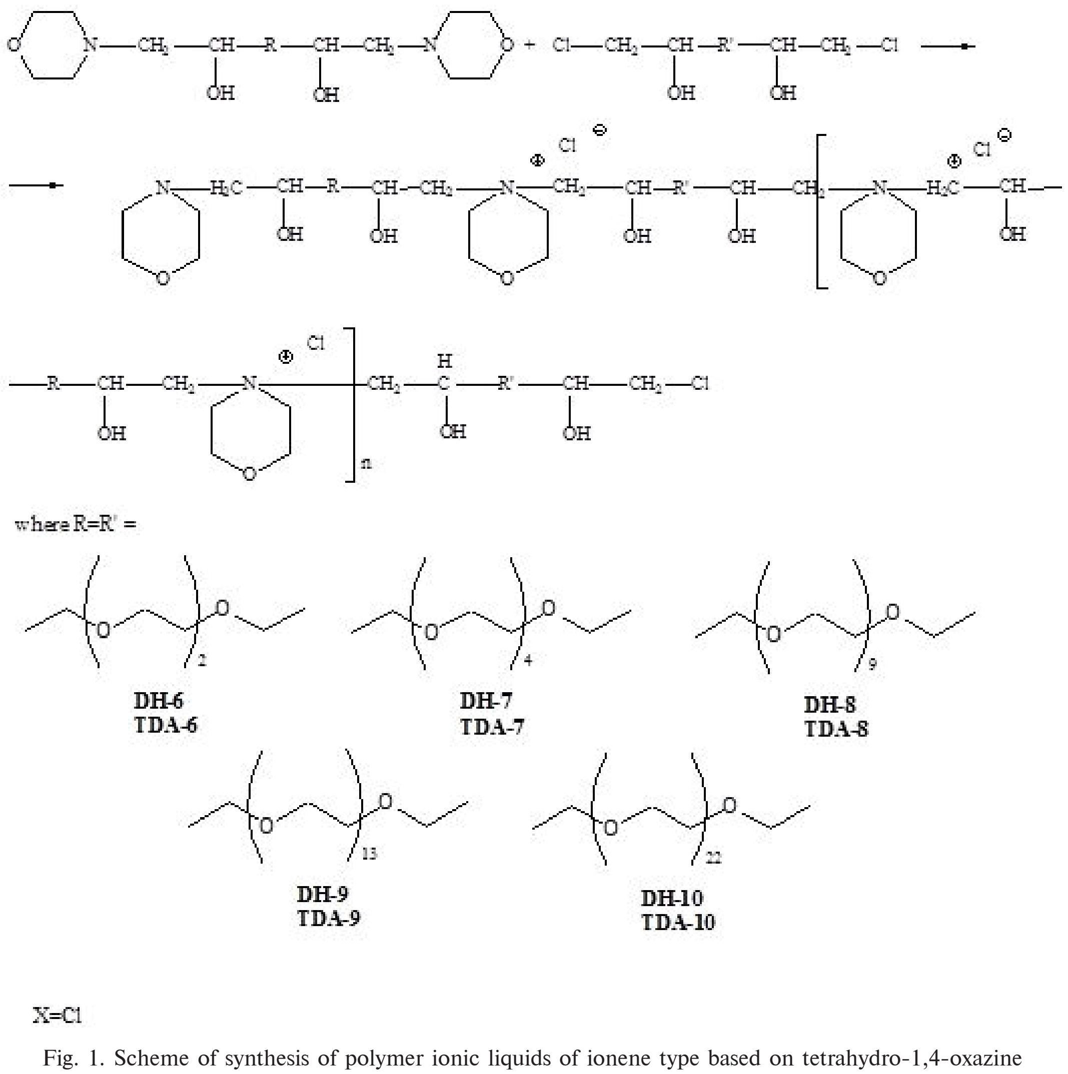
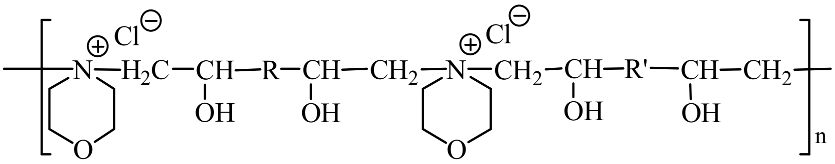


Fig. 1. Scheme of synthesis of polymer ionic liquids of ionene type based on tetrahydro-1,4-oxazine

Table 1

Characteristics of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine of the general formula



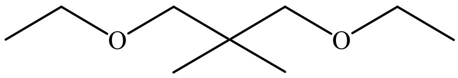
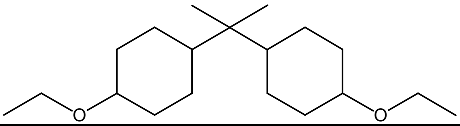
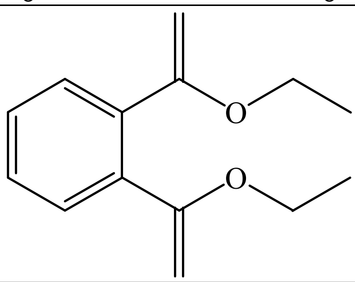
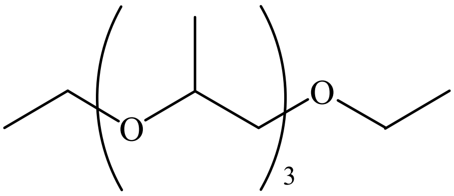
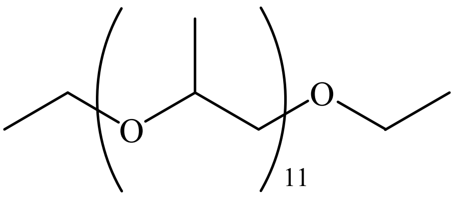
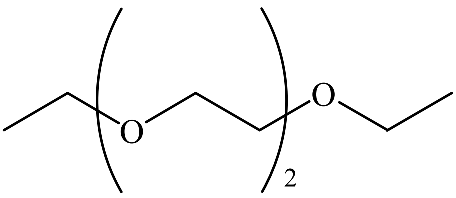
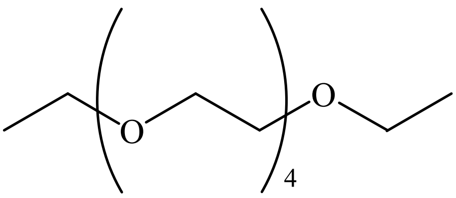
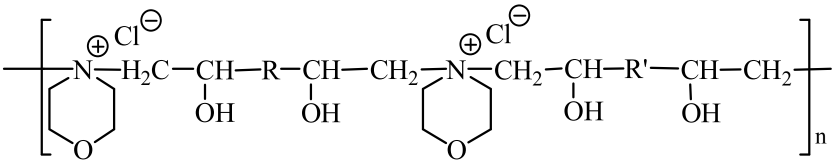
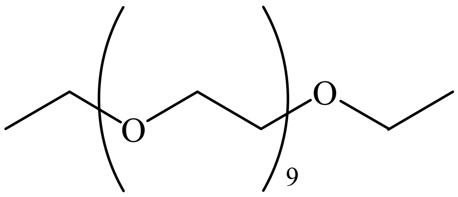
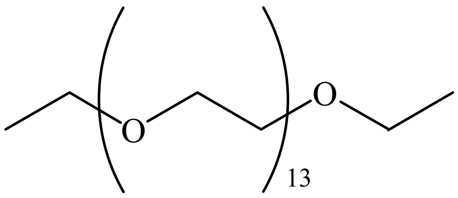
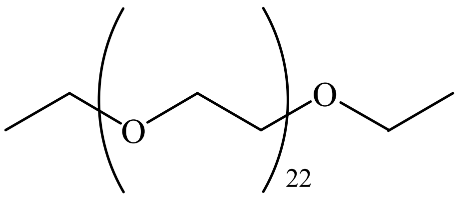
Name	R=R'	n	Nitrogen content, % theor./pract.	Halogen content, % theor./pract.	Refractive index (n_D^{20})
1	2	3	4	5	6
C-1-1		30	0.17/0.17	0.43/0.43	0.805
C-2-2		20	0.20/0.22	0.52/0.54	1.536
C-3-3		10	0.46/0.45	1.17/1.16	1.605
C-4-4		35	0.13/0.13	0.32/0.32	0.895
C-5-5		50	0.05/0.07	1.13/1.15	0.766
C-6-6		42	0.16/0.15	0.41/0.40	0.972
C-7-7		33	0.12/0.15	0.31/0.34	0.854

Table 1 (Continued)

Characteristics of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine of the general formula



1	2	3	4	5	6
C-8-8		30	$\frac{0.07}{0.08}$	$\frac{0.17}{0.18}$	0.798
C-9-9		58	$\frac{0.04}{0.05}$	$\frac{0.10}{0.11}$	0.758
C-10-10		80	$\frac{0.02}{0.04}$	$\frac{0.05}{0.07}$	0.655

smallest own radii). Experimental samples of polymeric ionic liquids of the ionic type synthesized in this work and described in ref. [13].

The study of ionic conductivity and glass transition temperature of polymeric ionic liquids of the ionic type was carried out using conductometry and differential scanning calorimetry. A comparative analysis of the influence of the chemical structure of polymeric ionic liquids of the ionene type on their ionic conductivity was carried out based on the results of the study in the temperature range of 15 to 50^{0}C.

Results and discussions

Previous studies [14] have established that it is necessary to study the general correlations between individual parameters and their ionic conductivity separately for each series of ionic liquids, and the established patterns will be inherent only to ionic liquids of a certain structure. From a theoretical point of view, studies aimed at supplementing or confirming the previously established dependences of the ionic conductivity of polymer ionic liquids of the ionene type on their structure and temperature allow predictably forming the processes of creating materials with high ionic conductivity in a wide temperature range. And it is such conclusions that can be considered appropriate from a practical point of view, because they allow a reasonable approach to the definition of a cation-anion pair to ensure the necessary complex of properties of polymer ionic liquids of the ionene type, which determine their effective application in a certain field.

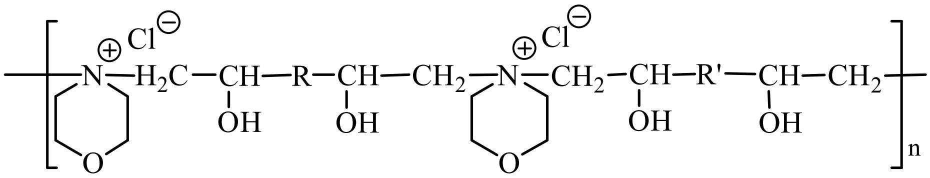
Our work shows (Table 2) that synthesized polymeric ionic liquids based on tetrahydro-1,4-oxazine derivatives with ionic conductivity of about $10^{-1}\div10^{-5}$ S·cm^{-1} in the glass transition temperature range of -143^0C$\div-60^0$C have five orders of magnitude higher ionic conductivity compared to known world analogues based on polymeric ionic liquids ($\sigma=10^{-6}\div10^{-10}$ S·cm^{-1}; $T_m=-8\div80^0$C) [15]. This allows supplementing a number of existing effective polymeric ionic liquids and their low-molecular-weight analogues of the ionene type with compounds with a higher level of ionic conductivity in the range of -143^0C to $+350^0$C.

Experimental studies have established that the nature of the temperature dependence of the ionic conductivity of the synthesized polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine is the same (Fig. 2).

With decreasing temperature, the ionic

Table 2

Glass transition temperature, ionic conductivity of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine of the general formula



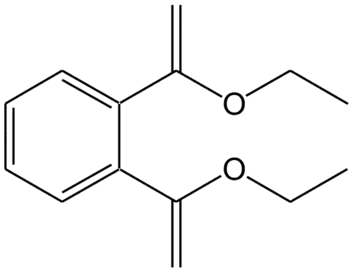
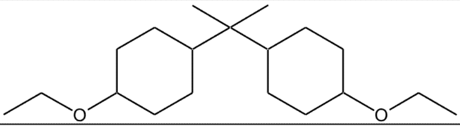
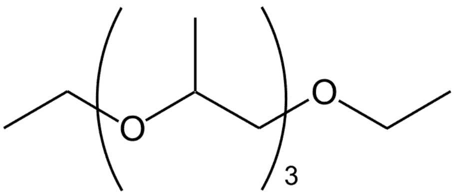
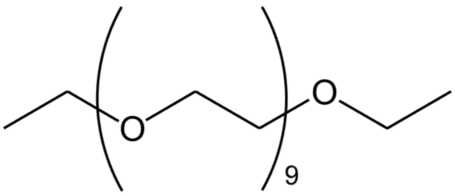
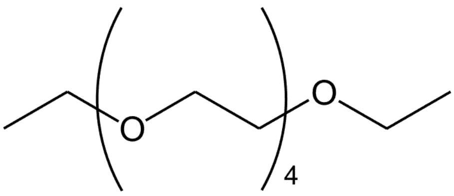
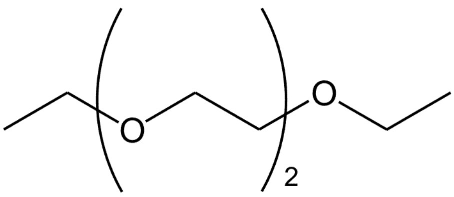
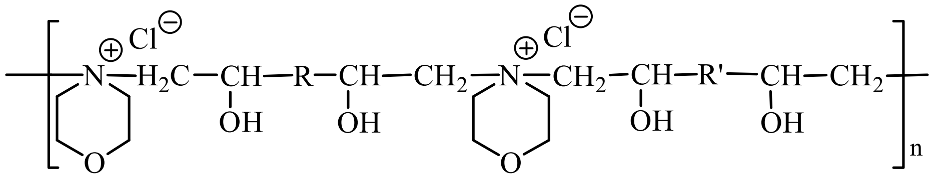
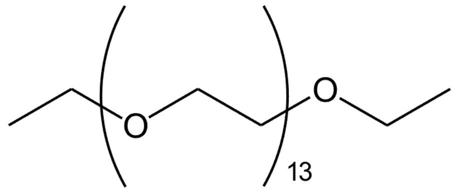
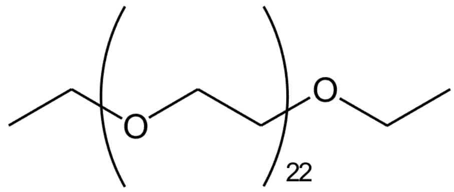
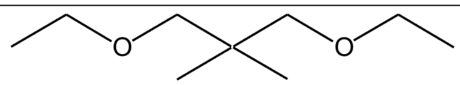
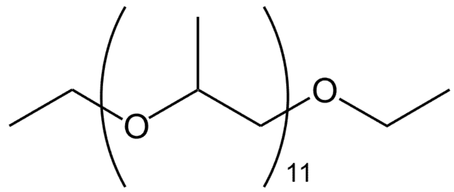
Name	R=R'	n	η_{red}/C, dL/g	Specific ionic conductivity (σ), $S \cdot cm^{-1}$ at 20^0C	Glass transition temperature, ^0C
1	2	3	4	5	6
C-3-3		10	1.08	$0.452 \cdot 10^{-1}$	–143
C-2-2		20	4.08	$0.322 \cdot 10^{-1}$	–126
C-4-4		35	6.32	$0.298 \cdot 10^{-2}$	–79
C-8-8		30	4.73	$0.296 \cdot 10^{-2}$	–73
C-7-7		33	5.87	$0.255 \cdot 10^{-2}$	–71
C-6-6		42	8.09	$0.223 \cdot 10^{-2}$	–69

Table 2 (Continued)

Glass transition temperature, ionic conductivity of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine of the general formula



1	2	3	4	5	6
C-9-9		58	8.28	$0.812 \cdot 10^{-3}$	–67
C-10-10		80	9.14	$0.054 \cdot 10^{-4}$	–63
C-1-1		30	9.65	$0.813 \cdot 10^{-5}$	–61
C-5-5		50	4.05	$0.676 \cdot 10^{-5}$	–60

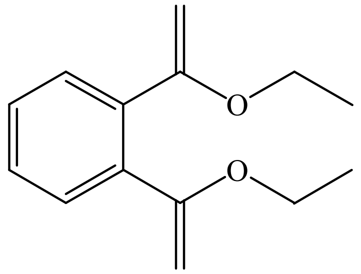
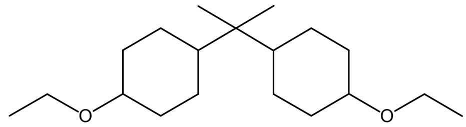
conductivity of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine decreases. This correlates well with the viscosity of solutions of the corresponding polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine (Fig. 3): with increasing viscosity, the ionic conductivity decreases. This dependence is explained by the complex of intra- and intermolecular interactions of polymer macromolecules.

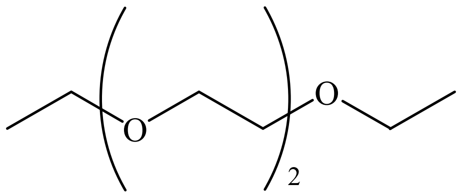
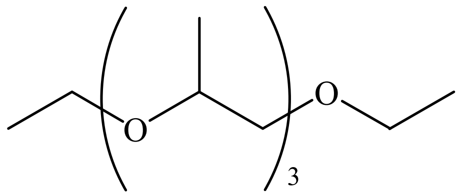
Generalization of the results of the study of the glass transition temperature and ionic conductivity of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine (Table 2) allows us to state the presence of an interesting pattern: a simultaneous increase in ionic conductivity and a decrease in the glass transition temperature of polymers, due to an increase in the entropy factor, caused by a decrease in ordering and an increase in the mobility of the segments of the polymer macromolecule chain with ionic groups. In particular, the correlation equation confirming this dependence for C-6-6÷C-10-10 has the following form:

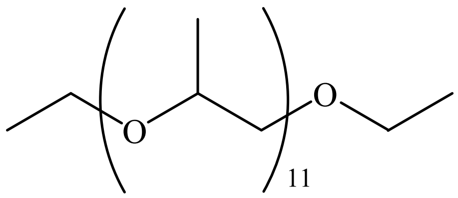
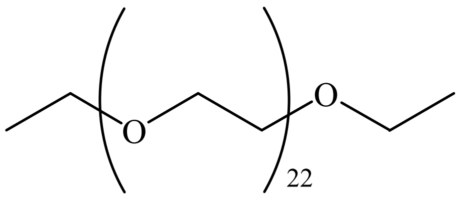
$$\sigma = -0.031 \cdot 10^{-2} \cdot T_m - 1.9;\ r = -0.985.$$

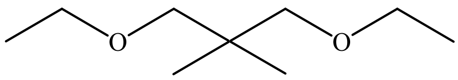
When determining the effectiveness of practical application of ionic liquids on the processes of their operation in technological systems, it follows from the results obtained that the ionic conductivity of polymeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine increases with a decrease in the glass transition temperature of polymers and the viscosity of their solutions and an increase in the temperature.

Analysis of the results (Table 3) of the study of the influence of substituents near the quaternary Nitrogen atom of synthesized polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine on their ionic conductivity showed a dependence that can be built into the following series:

 >  >

 >  >

 >  >

, in which the ionic conductivity decreases.

Previous studies [14] have established that the introduction of allyl groups to the quaternary nitrogen atom, the presence of a dibenzyl fragment and an alkylaromatic radical at the cationic center of polymeric ionic liquids and ionene-type ionic liquids causes an increase in ionic conductivity at a low glass transition temperature, which is confirmed by the correlation dependences between these parameters for each ionic liquid. The theoretical and practical results of systematic studies of this issue are the basis for studying the possible more effective adjustment of the nature of substituents at the cationic center of the polymer macromolecule to ensure a high ionic conductivity while maintaining properties up to very low temperatures.

This work shows that the introduction of allyl and oxyethylene groups to benzyl and dibenzyl fragments at the cationic center of the polymer macromolecule (the chain of the polymer macromolecule of this structure is more extended, which provides greater rigidity) causes an increase in the ionic conductivity of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine at a low glass transition temperature.

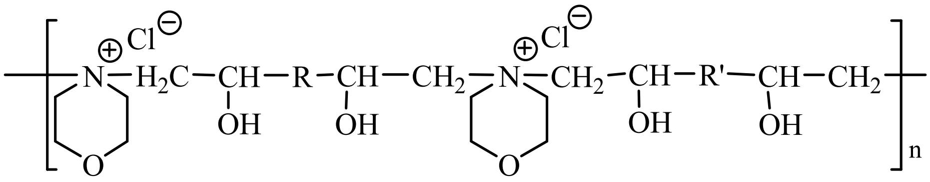
It should be noted that the replacement of the $-O-CH_2-CH_2-$ group with $-O-CH(CH)_3-CH_2-$ leads to a decrease in ionic conductivity, probably due to an increase in spatial complications. However, no physicochemical calculations are presented in this paper to confirm this hypothesis, which may be the basis for further research.

A comparative analysis of polymeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine with an aliphatic substituent and polymeric ionic liquids of the ionene type based on tetrahydro-1,4-oxazine with an alkylaromatic substituent at the nitrogen atom showed that the ionic conductivity of the latter is higher. Obviously, this is due to the «loosening» of the packing of the polymer chain with an alkylaromatic substituent due to the greater stiffness of the polymer macromolecule, which is more extended compared to the polymer containing aliphatic substituents. It should be noted that this does not contradict the practical data of previous works [14].

Comparison of the ionic conductivity of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine with different distances between quaternized nitrogen atoms in the polymer macromolecule indicates an increase in the ionic conductivity of the polymer with a decrease in the

Table 3

Ionic conductivity of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine of the general formula



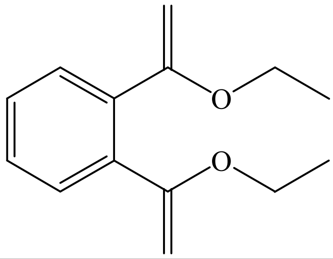
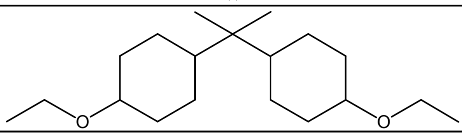
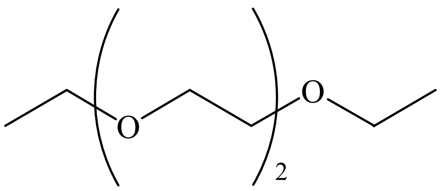
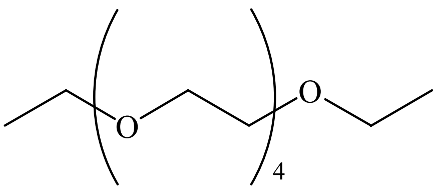
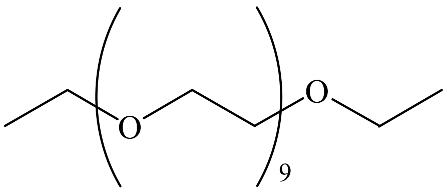
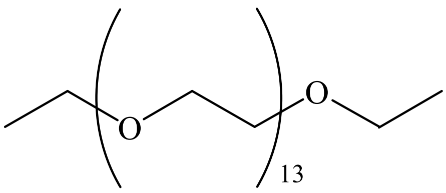
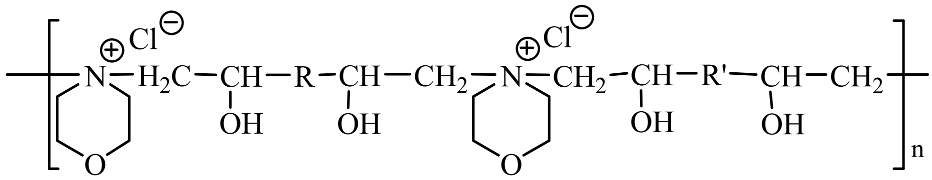
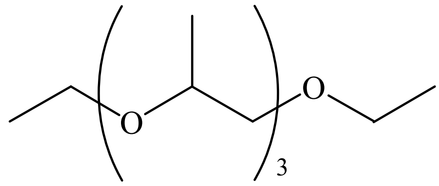
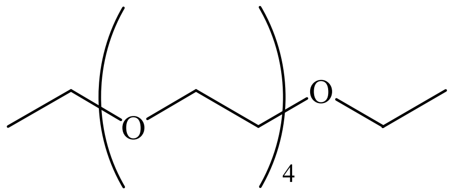
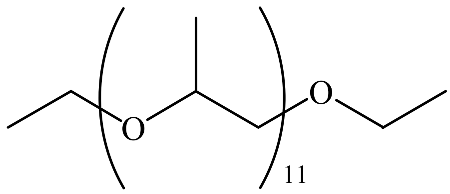
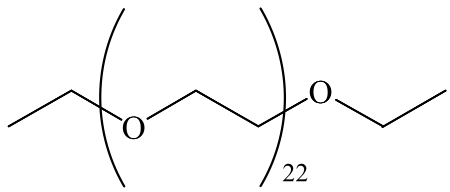
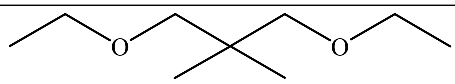
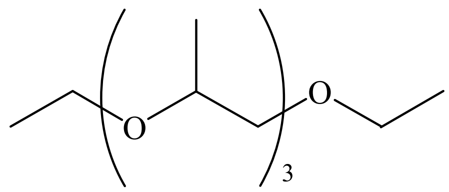
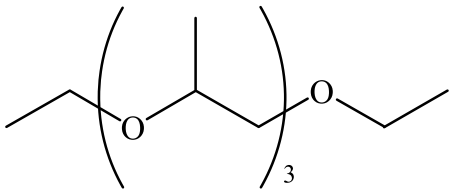
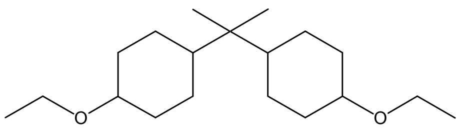
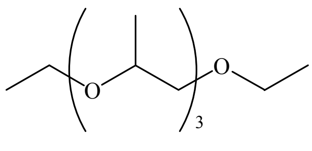
Title	R'	n	$\overline{M}_n$	η_{red}/C, dL/g	σ, $S \cdot cm^{-1}$
1	2	3	4	5	6
C-2-3*		15	10725	2.52	$0.426 \cdot 10^{-1}$
C-2-2*		20	10780	4.08	$0.322 \cdot 10^{-1}$
C-2-6*		10	10710	2.84	$0.158 \cdot 10^{-1}$
C-2-7*		11	10065	1.97	$0.976 \cdot 10^{-1}$
C-2-8*		11	10725	2.04	$2.742 \cdot 10^{-2}$
C-2-9*		5	9515	0.92	$0.857 \cdot 10^{-2}$

Table 3 (Continued)

Ionic conductivity of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine of the general formula



1	2	3	4	5	6
C-2-4*		13	10023	4.56	$0.624\cdot10^{-2}$
C-4-7**		10	10270	3.25	$0.524\cdot10^{-2}$
C-2-5*		13	10387	1.02	$1.524\cdot10^{-3}$
C-2-10*		12	10308	1.87	$0.253\cdot10^{-3}$
C-2-1*		17	10455	5.85	$0.626\cdot10^{-4}$
C-4-4**		16	10416	3.98	$0.483\cdot10^{-2}$
C-4-4**		35	22785	6.32	$0.298\cdot10^{-2}$

Notes: * – R=  ; ** – R=  .

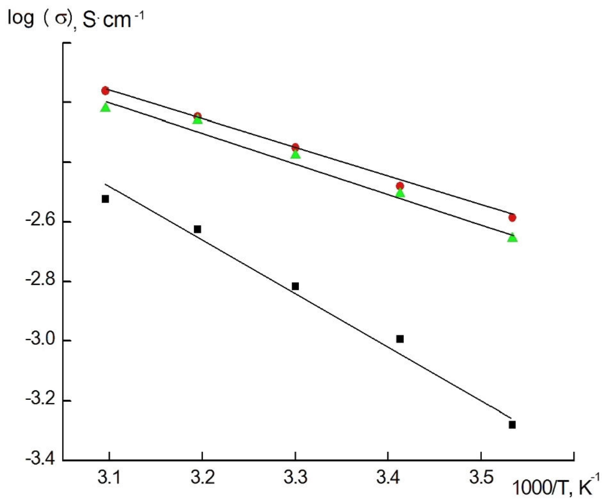


Fig. 2. Temperature (1000/T) dependence of specific ionic conductivity (logσ) of polymer ionic liquids of ionene type based on tetrahydro-1,4-oxazine:
1 – C-8-8; 2 – C-4-4; and 3 – C-1-1

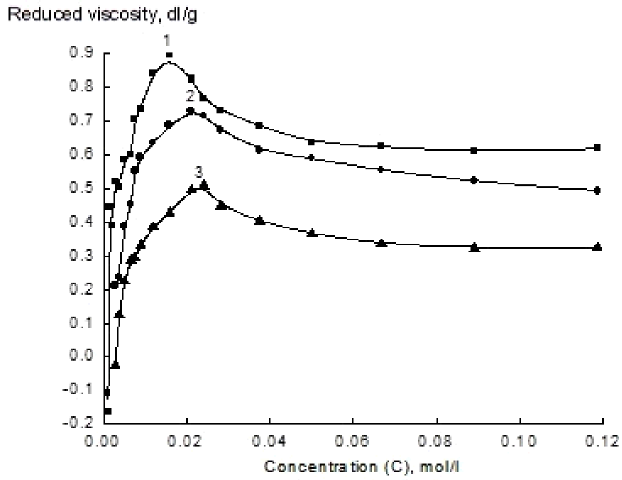
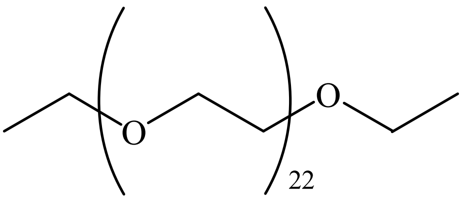


Fig. 3. Concentration (C) dependence of the reduced viscosity (η_{red}) of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine in water:
1 – C-1-1; 2 – C-8-8; and 3 – C-4-4

distance between the quaternary nitrogen atoms in this macromolecule, which is due to an increase in the number of charges in the polymer macromolecule chain. Analysis of the results of the study of this dependence of the ionic conductivity of polymer ionic liquids of the ionene type with a radical at the cationic center, which has a dibenzyl fragment group in its structure, with approximately the same molecular weight, given in Table 3, can be presented in the form of the following series:



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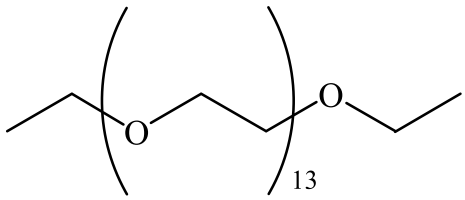

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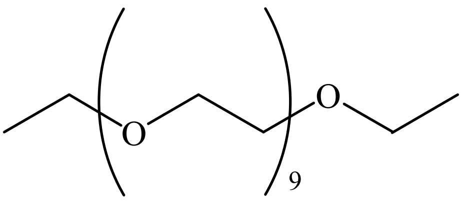

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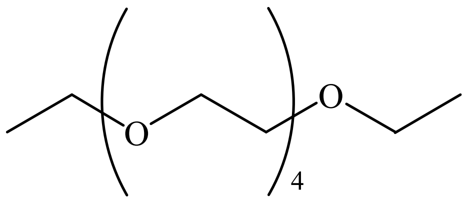

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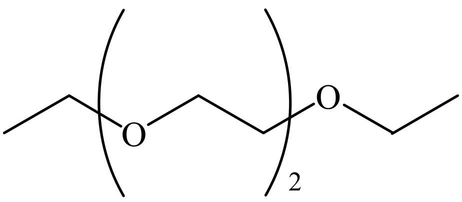

.

The correlation equation confirming this dependence for C-2-6÷C-2-10 has the following form:

$$\sigma=-0.19\cdot10^{-3}T_m-1.63,\ r=-0.802.$$

The main regulator affecting the level of ionic conductivity of polymer ionic liquids of the ionene type is also the size of the polymer macromolecule, which is confirmed by the results of the study of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine with the same structure, but with different degrees of polymerization (Table 3). It is shown that an increase in the chain length of the polymer macromolecule leads to a decrease in their ionic conductivity and an increase in the glass transition temperature. This dependence of the ionic conductivity on the molecular weight of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine is confirmed by the following correlation equation:

$$\sigma=-0.015\cdot10^{-5}\cdot M+638.8,\ r=0.998.$$

This is due to a decrease in the linearity of the polymer macromolecule due to an increase in the number of charges in the chain and spatial obstacles, which leads to a decrease in the mobility of charge carriers.

The obtained data do not differ from the practical data in ref. [14] and allow us to confirm the statement that the determining factors of the high ionic conductivity of the studied polymer ionic liquids of the ionene type are both the distance between the quaternary nitrogen atoms in this macromolecule and the size of the polymer macromolecule.

Thus, the generalization of the established results of these studies can be formulated as the following thesis:

1. With a decrease in the viscosity of polymer solutions, their glass transition temperature, and an increase in temperature, the ionic conductivity of polymer ionic liquids of the ionene type increases.

2. The introduction of allyl and oxyethylene groups to benzyl and dibenzyl fragments at the functional center of the macromolecule of polymer ionic liquids of the ionene type leads to an increase in ionic conductivity.

3. Substitution of $-O-CH_2-CH_2-$ bridge by the group $-O-CH(CH)_3-CH_2-$ in the radical of the cationic part of polymeric ionic liquids of ionene type causes a decrease in ionic conductivity.

4. The introduction of an alkylaromatic radical into the main chain of the macromolecule of polymer ionic liquids of the ionene type increases its ionic conductivity compared to a polymer of aliphatic structure.

5. With a decrease in the distance between quaternized nitrogen atoms in the polymer macromolecule, the ionic conductivity of polymer ionic liquids of the ionene type increases.

6. With the increase of the polymer macromolecule size, the ionic conductivity of polymeric ionic liquids of the ionene type decreases.

Based on the results of the research obtained in this work, further development of scientific ideas about polymeric ionic liquids of the ionene type was achieved due to the established dependences of the ionic conductivity of these compounds on temperature, the nature of the substituents at the quaternary nitrogen atom, the number of quaternary ammonium groups in the cation radical, and the length of the macromolecule chain, which were confirmed by correlation equations. Thanks to the established analytical dependences, it becomes possible to generalize the theories of existing knowledge of the justified choice of structural and chemical characteristics of carriers to solve the scientific and applied problem of developing polymeric ionic liquids and ionene-type ionic liquids based on tetrahydro-1,4-oxazine with high ionic conductivity while maintaining their liquid state in a wide range of temperatures.

Conclusions

1. For the first time, new polymeric ionic liquids based on tetrahydro-1,4-oxazine with high-level ionic conductivity ($10^{-1}\div10^{-5}$ $S\cdot cm^{-1}$) have been synthesized when stored at temperatures of $-60\div-143^{0}$C.

2. For the first time, correlations have been established between the chemical structure of polymeric ionic liquids based on tetrahydro-1,4-oxazine and their physicochemical properties, which are the basis for the synthesis of new compounds with specified characteristics: ionic conductivity increases with a decrease in the viscosity of polymer solutions, glass transition temperature and molecular weight of polymeric ionic liquids and an increase in temperature.

3. It has been established that the ionic conductivity of polymer ionic liquids of the ionene type based on tetrahydro-1,4-oxazine increases with a decrease in the length and number of charges in the macromolecule chain, the introduction of an alkylaromatic radical into the main chain of the polymer macromolecule, the substitution of the bridge $-O-CH(CH)_3-CH_2-$ by the group $-O-CH_2-CH_2-$ in the radical of the cationic part of the polymer, allyl and oxyethylene groups to benzyl and dibenzyl fragments at the functional center of the polymer macromolecule.

4. The dependences found, for which an explanation of the chemical nature is provided, are confirmed by correlation equations, which is extremely important for the further synthesis of polymeric ionic ionene type with the predicted defining parameters of ionic liquids, glass transition temperature and ionic conductivity.

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ІОННА ПРОВІДНІСТЬ І ТЕМПЕРАТУРА СКЛУВАННЯ ПОЛІМЕРНИХ ІОННИХ РІДИН ІОНЕНОВОГО ТИПУ

О.С. Свердліковська, М.О. Потапчук

Визначено особливості впливу полімерної природи синтезованих полімерних іонних рідин іоненового типу на основі тетрагідро-1,4-оксазину на їх іонну провідність і температуру склування. Встановлено кореляційні залежності між температурою, хімічною будовою полімерних іонних рідин іоненового типу на основі тетрагідро-1,4-оксазину та їх іонною провідністю і температурою склування, що дозволило доповнити основи синтезу нових полімерних іонних рідин та іонних рідин іоненового типу із заданими властивостями. Знайдені залежності підтверджено кореляційними рівняннями і пояснено їх хімічну природу. Доведено практичну привабливість запропонованих рекомендацій для розробки полімерних іонних рідин іоненового типу на основі тетрагідро-1,4-оксазину з високою іонною провідністю у широкому діапазоні температур.

Ключові слова: полімерні іонні рідини, іонні рідини, іонна провідність, температура склування, взаємозв'язки «структура–властивості».

IONIC CONDUCTIVITY AND GLASS TRANSITION TEMPERATURE OF IONENE-TYPE POLYMERIC IONIC LIQUIDS

O.S. Sverdlikovska *, M.O. Potapchuk

Ukrainian State University of Science and Technologies, Dnipro, Ukraine

*** e-mail: o.sverdlikovska@gmail.com**

The influence of the polymeric nature of synthesized ionene-type polymeric ionic liquids based on tetrahydro-1,4-oxazine on their ionic conductivity and glass transition temperature has been investigated. Correlations between temperature, chemical structure, ionic conductivity and glass transition temperature of these ionene-type polymeric ionic liquids were established, contributing to the design principles for new polymeric and ionene-type ionic liquids with tailored properties. The identified relationships were confirmed by correlation equations, and their chemical nature was analyzed. The proposed guidelines demonstrate practical value for the development of tetrahydro-1,4-oxazine-based ionene-type polymeric ionic liquids with high ionic conductivity across a wide temperature range.

Keywords: polymeric ionic liquids; ionic liquids; ionic conductivity; glass transition temperature; structure–property relationships.

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