Short Communication

Electrolyte Solutions: Measurement and Correlation of the Solubility of Et₄NBF₄ in Different Solvents at Temperatures from (263.15 to 323.15) K

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The efficient organic liquid electrolyte for supercapacitors requires the stable solubility of electrolyte salt and the high permittivity solvents over a wide temperature range. The solubilities of tetraethylammonium tetrafluoroborate (Et₄NBF₄) in propylene carbonate (PC), γ-butyrolactone (GBL), N, N-dimethylformamide (DMF), and acetonitrile (ACN) were determined by a synthetic method at temperatures ranging from (263.15 to 323.15) K under atmospheric pressure. The liquid–solid equilibrium process of these mixture was studied by a laser in-situ observing technique. The experimental solubilities were correlated by the van’t Hoff equation, the modified Apelblat and the λh equations. It is found the calculated solubility data shows good consistency with the experimental values. The thermodynamic properties of the solution process, including the Gibbs energy, enthalpy, and entropy were calculated by the van’t Hoff analysis and the Gibbs equation. These results concerning the solubility of Et₄NBF₄ in different solvents under atmospheric pressure will provide fundamental data in the commercial application of Et₄NBF₄ in supercapacitors.

Keyword: Organic liquid electrolyte, Tetraethylammonium tetrafluoroborate, Solubilities, Different temperatures, Thermodynamic properties, Supercapacitors

1. INTRODUCTION

Electric double-layer capacitors or supercapacitors have attracted extensive attention recently as rechargeable power sources, because they can provide high power density comparable to that for usual
rechargeable chemical batteries [1, 2]. Especially, the high energy density of EDLCs is possible with excellent rechargeability. One of the main ways to increase the energy capacitance of supercapacitors is the development of more nonaqueous organic electrolytes with a operating voltage above 3V. The efficient organic liquid electrolyte for these supercapacitors requires the stable solubility of electrolyte salt and the high permittivity solvents over a wide temperature range.[3]

Tetraethylammonium tetrafluoroborate (Et$_4$NBF$_4$) have been extensively studied as electrolyte salt in non-aqueous supercapacitors, because it is stable, safe to use and provides favorable electrical conductivity[4-6]. Some important physical chemical properties of Et$_4$NBF$_4$ in various high permittivity solvents (γ-butyrolactone[7], propylene carbonate[8], N,N-dimethylformamide[9,10], acetonitrile[11, 12], and so forth) has been investigated. However, a systematic solubility study of Et$_4$NBF$_4$ has not been reported in different permittivity solvents under a wide temperature range.

In this work, the solubilities of Et$_4$NBF$_4$ in four high permittivity solvents, acetonitrile (ACN), γ-butyrolactone (GBL), propylene carbonate (PC), and N,N-dimethylformamide (DMF) at T=(263.15, 273.15, 283.15, 293.15, 303.15, 313.15 and 323.15)K under atmospheric pressure. On this basis, the van’t Hoff equation, the λ h equation and the modified Apelblat equation are used to correlate the obtained experimental data. Some important thermodynamic property parameters were estimated by van’t Hoff analysis including standard molar Gibbs energy, standard molar enthalpy, and standard molar entropy.

2. EXPERIMENTAL

2.1. Materials

Table 1 lists the chemical reagents used in the experiment, including tetraethylammonium tetrafluoroborate (Et$_4$NBF$_4$), acetonitrile (ACN), butyrolactone (γ-GBL), propylene carbonate (PC), and N,N-dimethylformamide (DMF).

<table>
<thead>
<tr>
<th>Materials</th>
<th>Mass fraction purity</th>
<th>Sources</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tetraalkylammonium tetrafluoroborates</td>
<td>≥99.0%</td>
<td>ALDRICH</td>
</tr>
<tr>
<td>γ-butyrolactone (GBL)</td>
<td>≥99.0%</td>
<td>J&amp;K Chemical</td>
</tr>
<tr>
<td>propylene carbonate (PC)</td>
<td>≥99.5%</td>
<td>J&amp;K Chemical</td>
</tr>
<tr>
<td>N,N-dimethylformamide (DMF)</td>
<td>≥99.8%</td>
<td>J&amp;K Chemical</td>
</tr>
<tr>
<td>acetonitrile (ACN)</td>
<td>≥99.9%</td>
<td>J&amp;K Chemical</td>
</tr>
</tbody>
</table>
The water used in all experimental work for solution preparation and dilution was double distilled water (conductivity < 0.1 μs·cm⁻¹) unless otherwise specified.

2.2. Apparatus and Procedure.

In this work, the solubility of \( \text{Et}_4\text{NBF}_4 \) in four organic solvents was based on the fact that the light intensity penetrating through the solution increases with the dissolution of \( \text{Et}_4\text{NBF}_4 \) at the temperature ranges from (263.15 to 323.15) K under atmospheric pressure of 101.325 kPa. A synthetic method was used to measure the solubilities of \( \text{Et}_4\text{NBF}_4 \) [13]. The measurements were operated in a jacketed glass vessel (~50 mL) with a magnetic stirrer. At the beginning of each measurement, certain masses of \( \text{Et}_4\text{NBF}_4 \) and organic solvents were weighed by a precision Sartorius autobalance (an uncertainty of ± 0.0001 g) and then transferred to the glass vessel at a known temperature. A laser beam was utilized to monitor the dissolution of the solid + liquid mixture. The intensity of the penetrated light decreased with the increase of the amount of solute in the vessel, and the penetrated light intensity reached its saturation value when the last portion of the solid solute just disappeared. The total consumption of the solute and the mass of the total solvent were recorded.

The mole fraction solubility (\( x_1 \)) of \( \text{Et}_4\text{NBF}_4 \) could be estimated in different pure organic solvents:

\[
x_1 = \frac{m_1}{m_1/M_1 + m_2/M_2}
\]

(1)

Where \( m_1 \) and \( m_2 \) represent the mass of \( \text{Et}_4\text{NBF}_4 \) respectively, \( M_1 \) and \( M_2 \) are the corresponding molecular weights, respectively.

All of the experiments were investigated at least three times, and the relative uncertainties of the experimental data were within 3 %, obtained from the mass ratio of the additional solute to the dissolved solute.

3. RESULTS AND DISCUSSION

3.1. Solubility data and correlation models

Before the experiment, the accuracy about the equipment and method was confirmed by comparing our solubility data of potassium chloride in water (26.4wt%, 298.15 K) with those in literature [14]. Our experimental data was in agreement with literature values and thus demonstrated that our experimental method was accurate and reliable.

The solubilities of tetraethylammonium tetrafluoroborate (\( \text{Et}_4\text{NBF}_4 \)) in pure propylene carbonate (PC), \( \gamma \)-butyrolactone (GBL), \( N,N \)-dimethylfomamide (DMF), and acetonitrile (ACN) at different temperature are listed in table 2. The nonlinear van’t Hoff equation, \( \lambda h \) equation and modified Apelblat equation have been use to described the relationship between mole fraction solubility and temperature in different solvents.
Table 2. Solubilities of tetraethylammonium tetrafluoroborate in different solvent at (263.15 to 323.15) K at 101.325kPa

<table>
<thead>
<tr>
<th>T/K</th>
<th>$10^2 x_1$</th>
<th>$10^2 x_1^v$</th>
<th>$10^2 RD^v$</th>
<th>$10^2 x_1^A$</th>
<th>$10^2 RD^A$</th>
<th>$10^2 X_1^{zh}$</th>
<th>$10^2 RD^{zh}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GBL</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>263.15</td>
<td>5.984</td>
<td>5.786</td>
<td>3.30</td>
<td>5.945</td>
<td>0.65</td>
<td>5.739</td>
<td>4.10</td>
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<td>8.440</td>
<td>0.65</td>
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<td>10.019</td>
<td>2.19</td>
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<tr>
<td>313.15</td>
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<td>13.617</td>
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<tr>
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<td>15.710</td>
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<td>15.784</td>
<td>1.71</td>
<td>15.623</td>
<td>2.71</td>
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<tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
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<td>4.869</td>
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<td>4.963</td>
<td>-1.92</td>
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<td>4.93</td>
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<td>10.991</td>
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<tr>
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<td>14.724</td>
<td>0.43</td>
<td>14.334</td>
<td>3.07</td>
<td>14.574</td>
<td>1.44</td>
</tr>
<tr>
<td>DMF</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>263.15</td>
<td>6.554</td>
<td>6.113</td>
<td>6.73</td>
<td>6.497</td>
<td>0.88</td>
<td>6.1271</td>
<td>6.52</td>
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<tr>
<td>273.15</td>
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<td>7.273</td>
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</tr>
<tr>
<td>283.15</td>
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<tr>
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<td>13.040</td>
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<td>12.923</td>
<td>-0.34</td>
<td>13.025</td>
<td>-1.13</td>
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<td>323.15</td>
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<td>14.753</td>
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<td>15.072</td>
<td>-0.46</td>
<td>14.722</td>
<td>1.88</td>
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<td>ACN</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>263.15</td>
<td>6.774</td>
<td>6.461</td>
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<td>6.619</td>
<td>2.29</td>
<td>6.418</td>
<td>5.26</td>
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<tr>
<td>273.15</td>
<td>7.839</td>
<td>7.773</td>
<td>0.85</td>
<td>7.690</td>
<td>1.90</td>
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<tr>
<td>283.15</td>
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<td>8.945</td>
<td>1.40</td>
<td>9.210</td>
<td>-1.52</td>
</tr>
<tr>
<td>293.15</td>
<td>10.590</td>
<td>10.832</td>
<td>-2.28</td>
<td>10.412</td>
<td>1.68</td>
<td>10.816</td>
<td>-2.14</td>
</tr>
</tbody>
</table>
The nonlinear van’t Hoff equation [15-16] was proposed to correlate the experimental data as follows:

$$\ln \left( x_1 \right) = A + \frac{B}{T}$$

(2)

Where $x_1$ and $T$ are the mole fraction of the solute and absolute temperature, respectively, and $A$ and $B$ are the parameters of the equation. The parameter values of $A$ and $B$ are given in table 3.

**Table 3.** Parameters of the van’t hoff equation for tetraethylammonium tetrafluoroborate in the selected organic solvents

<table>
<thead>
<tr>
<th>Solvent</th>
<th>A</th>
<th>B</th>
<th>$10^3$rmsd</th>
</tr>
</thead>
<tbody>
<tr>
<td>GBL</td>
<td>2.53</td>
<td>-1415.67</td>
<td>2.58</td>
</tr>
<tr>
<td>PC</td>
<td>2.63</td>
<td>-1468.94</td>
<td>2.46</td>
</tr>
<tr>
<td>DMF</td>
<td>1.95</td>
<td>-1248.57</td>
<td>2.59</td>
</tr>
<tr>
<td>ACN</td>
<td>2.31</td>
<td>-1328.76</td>
<td>1.78</td>
</tr>
</tbody>
</table>

The absolute temperature $T$ dependence of the experimental solubility ($x^e$) of $\text{Et}_4\text{NBF}_4$ in selected solvents can be well-correlated by the modified Apelblat equation derived from the Williamson equation [17, 18].

$$\ln \left( x_1 \right) = A + \frac{B}{T} + C \ln T$$

(3)

Where $x_1$ is the mole fraction solubility of $\text{Et}_4\text{NBF}_4$, $T$ is temperature in K, and $A$, $B$ and $C$ are regression curve parameters which are summarized in table 4.

**Table 4.** Parameters for tetraethylammonium tetrafluoroborate in the different solvents obtained by the modified apelblat equation

<table>
<thead>
<tr>
<th>Solvent</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>$10^3$rmsd</th>
</tr>
</thead>
<tbody>
<tr>
<td>GBL</td>
<td>-33.63</td>
<td>188.07</td>
<td>5.40</td>
<td>2.17</td>
</tr>
<tr>
<td>PC</td>
<td>64.69</td>
<td>-4204.71</td>
<td>-9.28</td>
<td>2.74</td>
</tr>
<tr>
<td>DMF</td>
<td>-88.30</td>
<td>2734.14</td>
<td>13.49</td>
<td>1.08</td>
</tr>
<tr>
<td>ACN</td>
<td>-58.60</td>
<td>1361.26</td>
<td>9.10</td>
<td>2.44</td>
</tr>
</tbody>
</table>
The form of the \( \lambda h \) equation has been shown as follows [19-21] eq (4):

\[
\ln \left[ 1 + \frac{\lambda h (1-x_i)}{x_i} \right] = \lambda h \left[ \frac{1}{T / K} - \frac{1}{T_m / K} \right]
\]

(4)

Where \( x_i \) is the mole fraction solubility of Et\(_4\)NBF\(_4\), \( T \) is the experimental temperature of Et\(_4\)NBF\(_4\) in K, \( T_m \) is the normal melting temperature of Et\(_4\)NBF\(_4\) and \( \lambda \) and \( h \) are two constants of the \( \lambda h \) model. \( \lambda \) is identified as the association number of solute molecules in associating solution, and its value is a reflection of the nonideality of the solution. \( h \) equals the ratio of the enthalpy of solution to the gas constant \( R \), so it can be used to estimate the enthalpy of solution. The \( \lambda \) and \( h \) are the model parameters which are listed in table 5.

<table>
<thead>
<tr>
<th>Solvent</th>
<th>( \lambda )</th>
<th>( h )</th>
<th>10(^3)rmsd</th>
</tr>
</thead>
<tbody>
<tr>
<td>GBL</td>
<td>1.70</td>
<td>870.46</td>
<td>2.72</td>
</tr>
<tr>
<td>PC</td>
<td>1.73</td>
<td>888.54</td>
<td>2.45</td>
</tr>
<tr>
<td>DMF</td>
<td>1.05</td>
<td>1188.86</td>
<td>2.60</td>
</tr>
<tr>
<td>ACN</td>
<td>1.52</td>
<td>909.30</td>
<td>1.98</td>
</tr>
</tbody>
</table>

Table 5. Parameters for tetraethylammonium tetrafluoroborate in different solvents obtained by the \( \lambda h \) equation

The values of model parameters for van’t Hoff equation, modified Apelblat and \( \lambda h \) equations were obtained through regression of the experimental solubility data and are listed respectively in table 3–5 together with root-mean-square deviations (rmsd), described as:

\[
rmsd = \left( \frac{1}{N} \sum_{i=1}^{N} (x'_i - x''_i)^2 \right)^{1/2}
\]

(5)

Where \( N \) denotes the number of solubility data points measured in each solvent; \( x'_i \) and \( x''_i \) refer to the calculated solubility and experimental solubility, respectively.

The relative deviations (RD) between the calculated solubility and the experimental solubility are given in table 2.

\[
RD = \frac{x'_i - x''_i}{x''_i}
\]

(6)

As we can see from table 2, the calculated data of Et\(_4\)NBF\(_4\) in the four pure organic solvents show good agreement with the experimental data from small rmsds. The solubility data could be successfully correlated using the van’t Hoff equation, the modified Apelblat equation and the \( \lambda h \) equation. Meanwhile, the solubilities data of Et\(_4\)NBF\(_4\) in different solvents at the temperature range of (263.15 to 323.15) K are shown in Fig. 1.
Figure 1. Mole fraction solubility ($x_1$) of tetraethylammonium tetrafluoroborate versus temperature ($T$) in the selected pure solvents: ■, GBL; ▲, PC; ●, DMF; ◆, ACN. Solid lines, calculated from equation 2.

From table 2 and figure 1, we can see that the solubility of Et$_4$NBF$_4$ in the four organic solvents are dependent on the temperature, and the solubility data of Et$_4$NBF$_4$ are increased with the increase of temperature, but the increment of solubility with temperatures different in different pure solvents.

3.2. Thermodynamic parameter for the solution properties

Figure 2. A van’t hoff plot of the mole fraction solubility ($\ln(x_1)$) of tetraethylammonium tetrafluoroborate in the selected pure solvents against $T^{-1}$ with a straight line to correlate the data: ■, GBL; ▲, PC; ●, DMF; ◆, ACN.
The dependence of the solubilities to temperature can find by some related equations. According to the van’t Hoff equation, the standard molar enthalpy of solution ($\Delta H_{\text{soln}}^0$)[22-24] of Et$_4$NBF$_4$ in the solvents are estimated from eq (7): 

$$\frac{\Delta H_{\text{soln}}^0}{R} = -\left(\frac{\partial \ln x_i}{\partial (1/T)}\right)$$  \(\text{(7)}\)

Where $x_i$ is the mole fraction solubility, $R$ is the universal gas constant (8.314 J·mol$^{-1}$·K$^{-1}$) and $T$ is the corresponding temperature in K. From eq (2), eq (7) can be simplified to 

$$\Delta H_{\text{soln}}^0 = -BR$$  \(\text{(8)}\)

Over a limited temperature interval, the standard molar enthalpy of solution $\Delta H_{\text{soln}}^0$ would be valid for the mean temperature [25, 26]. Equation (9) can be written as: 

$$\frac{\Delta H_{\text{soln}}^0}{R} = -\left(\frac{\partial \ln x_i}{\partial (1/T - 1/T_{\text{mean}})}\right)$$  \(\text{(9)}\)

For clarity, $\Delta H_{\text{soln}}^0$ can also be obtained from the slope of the solubility curve where $\ln (x_i)$ is plotted versus $(1/T - 1/T_{\text{mean}}).$ Figure 3 shows the linear.

The standard molar Gibbs energy of solution ($\Delta G_{\text{soln}}^0$), the standard molar entropy of solution ($\Delta S_{\text{soln}}^0$) can be calculated according to [27, 28]:

$$\Delta G_{\text{soln}}^0 = -RT_{\text{mean}} \times \text{intercept}$$  \(\text{(10)}\)

$$\Delta S_{\text{soln}}^0 = \frac{\Delta H_{\text{soln}}^0 - \Delta G_{\text{soln}}^0}{T_{\text{mean}}}$$  \(\text{(11)}\)

The standard thermodynamic parameters for the solution process in the selected solvents are listed in table 6. As shown in table 6, the positive values of $\Delta H_{\text{soln}}^0$ for all cases suggest that the dissolution process of Et$_4$NBF$_4$ in different solvent systems is endothermic. The standard molar Gibbs energy ($\Delta G_{\text{soln}}^0$) values for all cases are positive, which demonstrates that the process is nonspontaneous.

**Figure 3.** The solubility ($\ln(x_i)$) of tetraethylammonium tetrafluoroborate in different pure solvent systems vs. $(1/t - 1/t_{\text{mean}})$ to correlate the data by a straight line: ■, gbl; ▲, pc; ●, dmf; ◆, acn.
Table 6. Thermodynamic properties related to the dissolution process of tetraethylammonium tetrafluoroborate in different solvent systems

<table>
<thead>
<tr>
<th>Solvent</th>
<th>$\Delta G_{\text{solv}}^0$ / KJ.mol$^{-1}$</th>
<th>$\Delta H_{\text{solv}}^0$ / KJ.mol$^{-1}$</th>
<th>$\Delta S_{\text{solv}}^0$ / J.mol$^{-1}$.K$^{-1}$</th>
</tr>
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<tbody>
<tr>
<td>GBL</td>
<td>5.61</td>
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<td>20.12</td>
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<tr>
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<td>22.79</td>
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<td>DMF</td>
<td>5.63</td>
<td>9.91</td>
<td>14.60</td>
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<td>5.41</td>
<td>10.71</td>
<td>18.08</td>
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</tbody>
</table>

The values of $\Delta S_{\text{solv}}^0$ is positive in all the solvents, which shows the dissolution process was entropically favorable.

Therefore, the obtained thermodynamic properties for the dissolution process of Et$_4$NBF$_4$ in such four organic solvents including Gibbs energy, enthalpy and the entropy suggests the dissolution of Et$_4$NBF$_4$ are dependent on the temperature, and the solubilities of Et$_4$NBF$_4$ are increased with the increase of temperature. The experimental data is in good agreement with the van’t Hoff equation and the Gibbs equation. These results will help to deeply understand the dissolution behaviors of Et$_4$NBF$_4$ in different solvents and provide fundamental data for the commercial application of Et$_4$NBF$_4$.

4. CONCLUSION

The solubilities of tetraethylammonium tetrafluoroborate (Et$_4$NBF$_4$) in the four organic solvents were determined at temperatures ranging from (263.15 to 323.15) K under atmospheric pressure by employing the synthetic method. The following conclusions can be drawn: (1) the solubilities of Et$_4$NBF$_4$ in the four organic solvents are dependent on the temperature, and the solubility data of Et$_4$NBF$_4$ are increased with the increase of temperature, but the increment of solubility with temperature is different in different pure solvents; (2) the experimental data is in good agreement with the van’t Hoff equation, the modified Apelblat equation and the $\lambda h$ equation; (3) the thermodynamic properties for the solution process including Gibbs energy, enthalpy and the entropy were obtained by the van’t Hoff analysis and the Gibbs equation. These results concerning the solubility of Et$_4$NBF$_4$ in different solvents will provide fundamental data for the commercial application of Et$_4$NBF$_4$.

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