Supporting Information

The path towards type V deep eutectic solvents: inductive effect and steric hindrance in the system tert-butanol + perfluoro tert-butanol

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Sample preparation

Binary mixtures of different molar fraction of tert-butanol with perfluoro-tert-butanol [TBF + TBH] were prepared gravimetrically using an analytical balance, Mettler Toledo, model AG245, with a mass resolution of ± 0.01 mg. All mixtures were prepared and handle for the calorimetric measurements in a glove box (Captair pyramid 2200) under nitrogen atmosphere and kept in sealed glass bottles under nitrogen. The composition uncertainty was estimated as better than ±0.01 mole fraction.

Solid-liquid phase diagram of [TBF + TBH]

The following thermal procedure was done for the study of the solid-liquid equilibrium of the [TBF +TBH] binary mixtures: first an isothermal step of 1 min at 298 K, followed by a fast cooling (scanning rate 50 K·min⁻¹) until 178 K·min⁻¹ and it was done an isothermal step of 15 min to to insure complete crystallization; a heating step of 5 K·min⁻¹ was performed until total isotropization. The eutectic temperatures, \( T_{\text{eutectic}} \), solid-solid transition temperatures, \( T_{\text{ss}} \), melting temperatures, \( T_{\text{melting}} \), for the different compositions (molar fraction of TBF, \( x_{\text{TBF}} \)) of the binary mixtures TBF+ TBH are given in Table SI.1.
- Excess Enthalpies

The partial excess molar enthalpies of TBF in TBH, $\overline{H}_2^E$ (TBH being defined as the component 1 and TBF component 2) at atmospheric pressure and at $T = 298.15 \pm 0.01$ K were experimentally obtained by Isothermal Titration Calorimetry (ITC). The calorimetric experiments consisted in subsequent injections of 10-40 µL of TBF to a sample of TBH or TBH-TBF binary solutions.

Each injection generated a heat flow signal, which was allowed to return to baseline before each new injection (see a typical experiment in figure SI.1). The area of the peak generated is proportional to the heat involved in the mixing experiment, which is directly related with the partial excess molar enthalpy as shown in the equation 1.

$$\overline{H}_2^E = \left( \frac{Q_1}{\partial n_2} \right)_{p,T,n_1}$$

The experimental data, $\overline{H}_2^E$, summarized in table SI.1, was fitted using the derivative of the Redlich–Kister fit (The values of the partial excess molar enthalpies obtained from the fitting are also presented in table SI.1 and are represented in figure SI.2).

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**Figure SI.1.** Plot of the typical results obtained from ITC for the injection of 12 µL of TBF in a solution of $x_2 = 0.8$ (initial concentration).
Table SI.1. Different compositions (molar fraction of TBF, $x_{\text{TBF}}$) of the binary mixtures TBF + TBH, eutectic temperatures, $T_{\text{eutectic}}$, solid-solid transition temperatures, $T_{\text{ss}}$, and melting temperatures, $T_{\text{melting}}$.

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<th>$x_{\text{TBF}}$</th>
<th>$m_{\text{sample/mg}}$</th>
<th>$T_{\text{eutectic I}}$ / K</th>
<th>$T_{\text{eutectic II}}$ / K</th>
<th>$T_{\text{eutectic III}}$ / K</th>
<th>$T_{\text{ss}}$ / K</th>
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*aThe uncertainty of the experimental results $\sigma$ (temperature) was estimated as ± 1.0 K for the temperature.*
Table SI.2. Experimental and fitted (Redlich–Kister equation, n=4) results of the partial excess molar enthalpy, $H^E_2$.

<table>
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<tr>
<th>$x_{TBF}$</th>
<th>$H^E_2$ experimental data (kJ·mol$^{-1}$)</th>
<th>$H^E_2$ fitted (fitted n = 4) (kJ·mol$^{-1}$)</th>
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Figure S1.2. Partial excess molar enthalpy, $H^E_2$ (× experimental results and — results obtained by the Redlich–Kister fit, $n = 4$) versus molar fraction of TBF, $x_{\text{TBF}}$.

Table S1.3. Derivative Redlich–Kister equation coefficients $A_i$ ($n = 4$). Binary mixtures: TBH ($x_1$) + TBF ($x_2$).

<table>
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<tr>
<th>$A_i$</th>
<th>Values / kJ·mol$^{-1}$</th>
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Table SI.4. Excess molar enthalpy of mixing, $H^E$ derivative from the Redlich–Kister equation and from the parameters $A_i$ ($n=4$).

<table>
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<tr>
<th>$\alpha_{TB}$</th>
<th>$H^E$ / kJ·mol$^{-1}$</th>
<th>$H_2^E$ / kJ·mol$^{-1}$</th>
<th>$H_1^E$ / kJ·mol$^{-1}$</th>
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</thead>
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<td>-0.17</td>
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Figure SI.3. Excess molar enthalpy of mixing, $H^E$ derivative from the Redlich–Kister equation and from the parameters $A_i \, (n = 4)$.

Due to experimental difficulties, it was not possible to measure experimentally the partial excess molar enthalpy of TBH. Therefore, the fitting parameters obtained before were used to estimate the $\bar{H}_1^E$, as well as, the excess molar enthalpies of mixing. Using the experimental values of $\bar{H}_2^E$ and the estimated $\bar{H}_1^E$ values, it was calculated the “experimental” results of excess enthalpy of mixing. In figure SI.3 is represented the excess molar enthalpies of mixing obtained directly from the fitting parameters and from the experimental results. This figure acts as an indication of the uncertainty existent in the central area of the diagram, which is due to both lower absolute values of the $\bar{H}_2^E$ and having only experimental values of one of the components.
Table SI.5. Experimental excess molar enthalpy of mixing, \( H^E \) derived by combing the experimental data set of \( H^E_2 \) with the Redlich–Kister equation \((n=4)\).

<table>
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<tr>
<th>( x_{TBF} )</th>
<th>( H^E_2 ) / kJ·mol(^{-1}) (experimental)</th>
<th>( H^E_1 ) / kJ·mol(^{-1}) (derived from RK fitting)</th>
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Figure SI.4. Derived experimental results of excess enthalpies of mixing, $H^E$, of TBF + TBH at 298.15 K (o) and Redlich–Kister fitting (---).
Table S1.6. Experimental densities and excess volumes for the TBH+TBF system at 298.15K as a function of composition.

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<th>α(TBF)</th>
<th>$V^E$ / g.cm$^{-3}$</th>
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<td>1 (ρ = 1.68145 g.cm$^{-3}$)</td>
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