



## Separation of 1,3-dioxolane from its azeotropic aqueous solution by using Good's buffer ionic liquid [TMA][EPPS]



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### ABSTRACT

It was found that the presence of Good's buffer ionic liquid (GBIL), [TMA][EPPS], in 1,3-dioxolane aqueous solution could induce liquid–liquid phase splitting. The influence of this GBIL on the separation of 1,3-dioxolane from its aqueous solution was studied by measuring solid–liquid–liquid equilibrium (SLE) and liquid–liquid equilibrium (LLE) tie-line data for 1,3-dioxolane + water + [TMA][EPPS] at 298.15 K and 101.3 kPa. The LLE phase boundary data were well correlated with an empirical equation and the effective excluded volume (EEV) model, respectively. The LLE tie-line data were also correlated quantitatively with the NRTL model and their consistency was confirmed by correlating these data with the Othmer–Tobias model. The experimental results revealed that [TMA][EPPS] can be used as an auxiliary agent to recover high purity 1,3-dioxolane from its azeotropic aqueous solution. A greener separation process is proposed. In comparison with the conventional inorganic salts, this new GBIL is a biocompatible, non-corrosive, and green chemical compound.

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### 1. Introduction

1,3-Dioxolane, a cyclic ether, is widely used in various industrial sectors, such as textile [1], electroplating [2], lithium batteries [3], polymer [4,5], and gasoline production [6]. It is an aprotic solvent and is employed for the formulation or as a reactant [7]. 1,3-Dioxolane is also suitable for rapidly dissolving a variety of polar polymers, including polyester, epoxide, and urethanes. Due to its small size, 1,3-dioxolane can easily penetrate the polymers and thus enhances the rate of polymer-based coating processes [7]. It also serves as an intermediate in the synthesis of various industrial important polymers and pharmaceutical products [8]. The low boiling point of 1,3-dioxolane (348.6 K) helps in fast drying as well as high throughput [9].

1,3-Dioxolane is miscible with water in all the proportions under ambient conditions. Unfortunately, 1,3-dioxolane forms an azeotropic mixture with water [10,11]. Due to the formation of azeotrope, the separation of 1,3-dioxolane from its aqueous solution became difficult by using simple distillation. Liquid–liquid extraction is one of potential alternative methods for recovery of organic compound from its azeotropic mixture. Many electrolytic salts are

recognized as potential compounds to induce liquid–liquid phase splitting for the aqueous solutions of acetonitrile, acetone, and short-chain alcohols and thus help in the successful recovery of these solvents from their aqueous solutions via extraction technique. Salts are also commonly employed in various processes such as extractive fermentation [12–15], solvent dehydration [16], extractive crystallization [17,18], and purification of biological products, such as protein, nucleic acid, enzyme and others [19–21]. However several disadvantages were found using the inorganic salts in the separation processes. For example, salts are corrosive in nature, and the pH value of the medium may change under high salt concentrations and resulting in the denaturation of biomolecules [22]. To overcome the above mentioned problems, we synthesized a self-buffering Good's buffer ionic liquid (GBIL), [TMA][EPPS], and investigated its applicability for separation of 1,3-dioxolane from its aqueous solutions. This new ionic liquid, [TMA][EPPS], has the cation part derived from *tetra*-methylammonium (TMA) and the anion part from the commonly used Good's buffer, 4-(2-hydroxyethyl)-1-piperazinepropanesulfonic acid (EPPS). Good buffer ionic liquids (GBIL) is the new class of self-buffering and biocompatible ionic liquids derived from the commercially available good's buffer. In comparison to the conventional ionic liquids, this GBIL not only provide the biocompatible medium but also due to their self-buffering nature avoid the use of an external buffering compound and thus can be more

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advantageous in the biological research. [TMA][EPPS] has been confirmed to be self-buffering and biocompatible in nature [23]. The chemical structure of corrosion-inhibitor is noticed to have multiple atoms (nitrogen, sulfur, and oxygen), through which they are absorbed on the metal surface and protect the surface from corrosion [24]. Fortunately, the selected GBIL, [TMA][EPPS], also contains nitrogen, sulfur, and oxygen in their structure and thus may serve as a corrosion-inhibitor. In comparison with using the inorganic salts, the use of the selected GBIL, [TMA][EPPS], as an auxiliary agent for separation maybe more advantageous.

We have measured the liquid–liquid equilibrium (LLE) and solid–liquid–liquid equilibrium (SLLE) tie-line data for the ternary system of 1,3-dioxolane (1) + water (2) + [TMA][EPPS] (3) at 298.15 K and 101.3 kPa. The consistency of these experimental data was checked with Othmer–Tobias model [25]. The experimental LLE phase boundary data were correlated with an empirical model and the effective excluded volume (EEV) model of Guan et al. [26], respectively. In addition, the binary interaction parameters of the NRTL model [27] for the investigated system were also obtained from the LLE tie-line data correlation.

## 2. Experimental

### 2.1. Materials

1,3-Dioxolane (mass fraction purity > 0.995) was supplied by Sigma Chemical Co. (USA). The buffer EPPS (mass fraction purity 0.998) and *tetra*-methylammonium hydroxide (25 wt % in water) were purchased from Sigma Chemical Co. (USA). The double de-ionized highly pure water was used in the sample preparation and was taken from a Nano-pure-Ultra pure water purifying system with resistivity of 18.3 MΩ cm. The description of the materials used is given in Table 1. The experimental mixtures were prepared gravimetrically by using an electronic balance (Model GR-200, R&D, Japan) with an accuracy of ±0.1 mg.

### 2.2. Synthesis of ionic liquid

The ionic liquid [TMA][EPPS] was synthesized in our lab by using the similar procedure as explained elsewhere [23,28]. The aqueous ammonium-hydroxide solution of *tetra*-methylammonium (TMA) was neutralized by a slightly excess of an equimolar aqueous buffer solution in a flask equipped with a water condenser. The reaction mixture was stirred vigorously for about 10–12 h to ensure the completion of the reaction. The mixture was then dried at reduced pressure and at 50–60 °C. A mixture of ethanol and acetonitrile (1:1) was added to the obtained viscous liquid, to remove the unreacted material and any other impurity. The mixture was then filtered and the filtrate was dried under reduced pressure to get the desired GBIL with low water content. The purity and the water content of the synthesized ionic liquid were confirmed with  $H^1$

NMR and Karl-Fischer titration, respectively.

### 2.3. Experimental procedure

The experimental tie-line data of the investigated system have been determined experimentally at 298.15 K and 101.3 kPa by using a tightly closed and jacketed equilibrium cell made up of Pyrex glass. The details of the experimental set-up and the measurement procedure were given in our previous articles [29,30]. The total volume of the equilibrium cell is about 50 cm<sup>3</sup>. During the course of the experiment, the temperature of equilibrium cell was maintained at 298.15 K by circulating constant temperature water through the jacket of the equilibrium cell with the help of water circulating thermostat (Model-B402L, Firstec Scientific Co. Ltd., Taiwan). The temperature inside the equilibrium cell was measured using a precise digital thermometer (Model-1560, Hart Scientific Co., USA) to an uncertainty of ±0.1 K. In each experimental run, a mixture of 1, 3-dioxolane + water + [TMA][EPPS] with a pre-specified composition was loaded into the equilibrium cell. The mixture was agitated with a magnetic stirrer bar for 12 h. After the mixing period, the stirrer was turned off and the mixture was allowed to settle into two liquid phases at least 8 h. The required time intervals for the mixing period and the settled period were estimated by taking samples at different intervals of time. The samples from the upper phase and the lower phase were collected for the composition analysis. The analysis samples were collected with an aid of air-tight glass syringe from the top and the bottom sampling ports of the cell, respectively. A series of liquid–liquid equilibrium (LLE) and solid–liquid–liquid equilibrium (SLLE) data for this investigated ternary system were measured by changing the feed composition of the loaded mixture.

### 2.4. Sample analysis

The compositions of 1,3-dioxolane and water in the collected samples from the upper phase and the lower phase were analyzed with a gas chromatograph (GC, Model 9800, China Chromatography Co., Taiwan). This GC was equipped with a thermal conductivity detector (TCD). A stainless steel packed column (Porapak QS, 80/100, and 2 m of length) was used for the analysis. To protect the detector and the column from blocking by the GBIL, a stainless steel tube packed with glass wool, as a trapper for GBIL, was installed in between the injector section and the column of the GC. The trapper was replaced when significant amount of GBIL was trapped. Helium with mass fraction purity >0.9995 was used as a carrier gas. About 0.001 cm<sup>3</sup> of the analysis sample was injected in the GC by using air-tight glass syringe. The peak-ABC chromatograph data handling system was employed to analyze the response of gas chromatography for the injected sample.

Prior to the analysis of the collected samples, the GC was calibrated by using a series of standard samples over the entire

**Table 1**  
Description of the materials used.

Compound	Source	Initial mass fraction	Purification method	Final mass fraction	Analysis method
1,3-Dioxolane	Sigma Chemical Co. (USA)	0.995	None	0.995	None
EPPS <sup>a</sup>	Sigma Chemical Co. (USA)	0.998	None	0.998	None
<i>Tetra</i> -methylammonium hydroxide	Sigma Chemical Co. (USA)	0.250	None	0.250	None
Ethanol	Sigma Chemical Co. (USA)	0.998	None	0.998	None
Acetonitrile	Sigma Chemical Co. (USA)	0.998	None	0.998	None
De-ionized water	Our lab.	–	None	–	None
[TMA][EPPS]	Synthesize	–	Recrystallization	0.998	$H^1$ NMR <sup>b</sup> and KF <sup>c</sup>

<sup>a</sup> EPPS = 4-(2-hydroxyethyl)-1-piperazinepropanesulfonic acid.

<sup>b</sup>  $H^1$ NMR = Proton nuclear magnetic resonance spectra.

<sup>c</sup> KF = Karl-Fischer titration.

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