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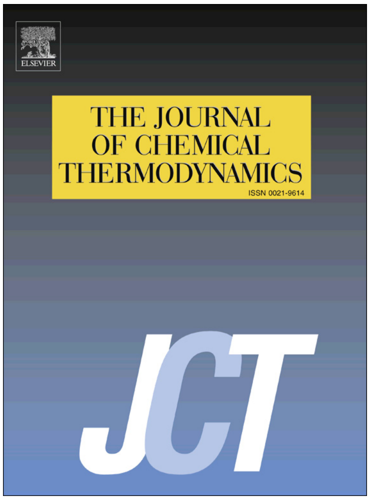

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Consistency of experimental data in SLLV equilibrium of ternary systems with electrolyte. Application to the water + NaCl + 2-propanol system at 101.3 kPa

Jorge Garcia-Cano, Alejandro Gomis, Alicia Font, Maria Dolores Saquete, Vicente Gomis*

University of Alicante, PO Box 99, E-03080 Alicante, Spain

Highlights

- Thermodynamic consistency for the phase equilibrium of water + NaCl + 2-propanol ternary system.
- Experimental determinations at 101.3 kPa of the liquid-liquid-vapor, solid -liquid- vapor and solid -liquid-liquid- vapor equilibrium for the water + NaCl + 2-propanol ternary system.
- The influence of salt and temperature on water + 2-propanol equilibria.
- Influence of propanol type on the water + NaCl + 1-propanol or 2-propanol equilibrium phase.

Keywords

Consistency, Water; 1-Propanol; 2-Propanol; NaCl; Isobaric; Vapor-liquid-liquid-solid; Phase diagram

Abstract

The SLLV phase equilibria of the water + NaCl + 2-propanol mixture have been determined experimentally at 101.3 kPa by means of a modified recirculating still. The results obtained allow us to study the shape of the phase diagram of the system, to analyze the evolution with temperature of this equilibrium diagram and to show the differences with a similar system such as water + NaCl + 1-propanol.

Moreover, the experimental data obtained have been compared with previously published data showing their important inconsistencies and presenting the rules that must be met by the experimental equilibrium data of water + non-volatile salt + organic solvent type systems in each one of the different SLLV, LLV and SLV equilibrium regions.

1. Introduction

The knowledge of the equilibrium phase diagram of mixed solvent electrolyte systems is important because this type of mixture is found in many processes. These processes include regeneration of solvents, liquid-liquid extraction of mixtures containing salts and extractive distillation and crystallization. For example, the salt effect distillation is used commercially in the concentration of aqueous nitric acid, using the salt magnesium nitrate as the separating

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*Corresponding author. Tel.: +34 965903365.
E-mail address: vgomis@ua.es (V. Gomis).

agent. Other commercial applications include acetone-methanol separation using calcium chloride and isopropanol-water separation using the same salt [1].

However, the literature available on the mixed solvent electrolyte systems is scarce. Moreover, the relatively small amount of existing experimental data is incomplete and some of them containing important inconsistencies as shown in a previous paper [2].

Finally, the thermodynamic models used to represent these systems, such as the extended UNIQUAC model for electrolytes [3], or the electrolyte NRTL model [4] all need the equilibrium data to calculate the model parameters.

In two previous papers [2, 5], we studied the equilibrium diagram of the water + NaCl + 1-butanol and water + NaCl + 1-propanol systems at 101.3 kPa in order to examine the shapes of the various equilibrium surfaces and regions that occur in them. To do this, a detailed analysis of the evolution with temperature of the different equilibrium regions of these systems was carried out. The influence of salt on the equilibrium was investigated demonstrating that many of the previously published data were inconsistent and inaccurate. It is really important to have reliable experimental data suitable for the development of new thermodynamic models or obtaining new binary interaction parameters with the existing models.

In the previously studied systems, the electrolyte was NaCl although there was an important difference between them: the 1-butanol is partly miscible with water while 1-propanol is completely miscible. The objective of the present paper is to extend those studies with NaCl to another system involving a completely miscible solvent in order to verify the inconsistency of previously published data and to analyze once again the evolution with temperature of the different equilibrium regions. The chosen system is water + NaCl + 2-propanol at 101.3 kPa whose equilibrium phase diagram could be in principle similar to that with 1-propanol since both systems contain a binary water + propanol with a homogeneous minimum boiling azeotrope. However, as shown in Figure 1, the azeotropic composition of the two systems is quite different: 0.69 mole fraction in 2-propanol [6, 7] for one of them and 0.43 in 1-propanol [8, 9] for the other one. This fact could make the phase diagrams of both systems and their evolution with the temperature very different.

In this work, LV (liquid-vapor), LLV (liquid-liquid-vapor), SLV (solid-liquid-vapor) and SLLV (solid-liquid-liquid-vapor) equilibrium data of the water + NaCl + 2-propanol system at 101.3 kPa have been determined experimentally. The results obtained permit us to carry out a study of the shape of the phase diagram of the system, to show the inconsistency of previous results, to analyze the evolution with temperature of the phase diagram and to compare it with that of a similar system as water + NaCl + 1-propanol.

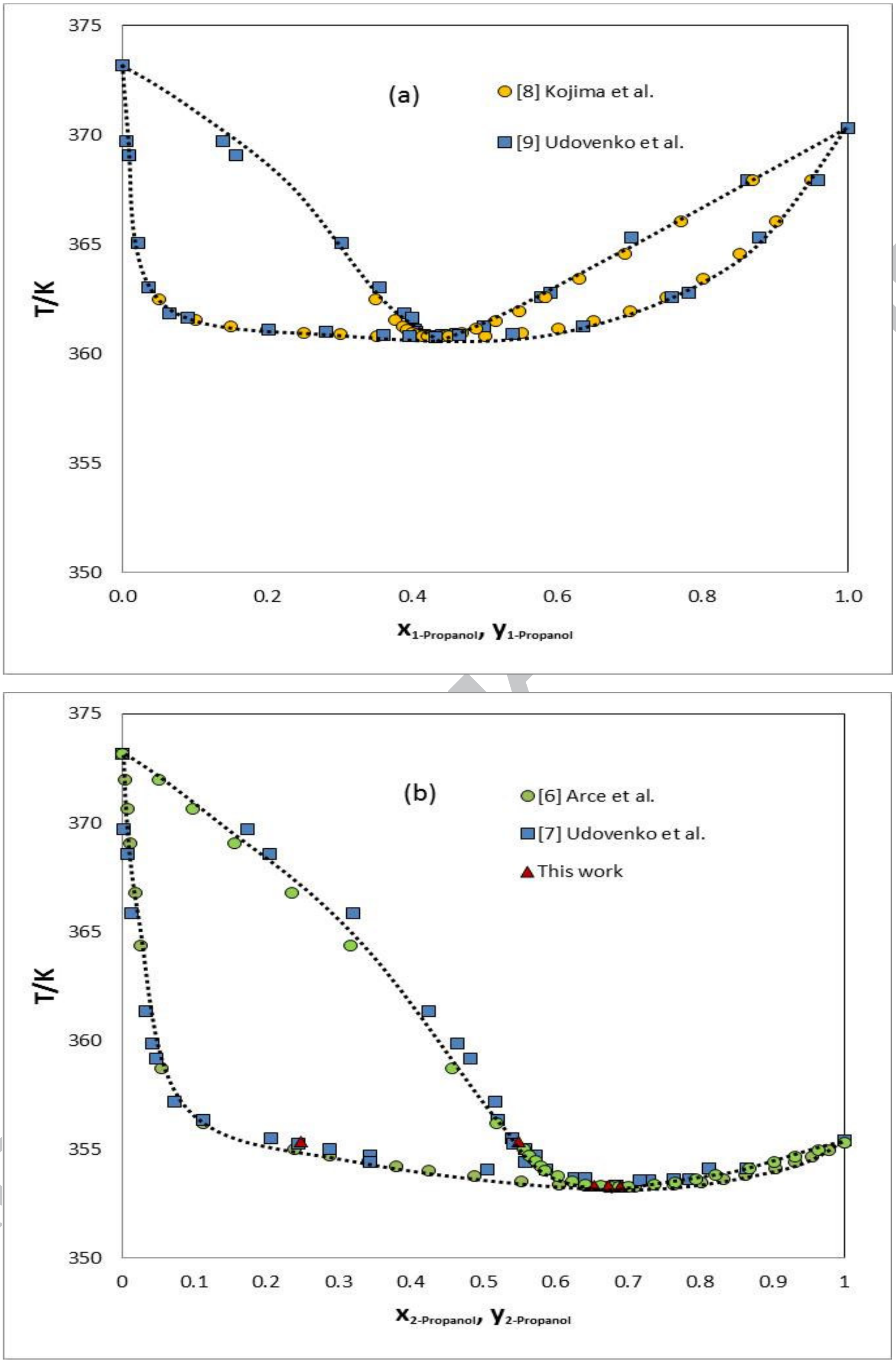

Fig. 1. Binary equilibrium diagrams. a). Txy of water + 1-propanol at 101.3 kPa. b). Txy of water + 2-propanol at 101.3 kPa. Compositions in mole fraction.

2. Previous experimental equilibrium studies of the system and inconsistencies.

The water + NaCl + 2-propanol system at 101.3 kPa includes the water + 2-propanol binary system with many experimental LV equilibrium data in bibliography [for example: 10, 11] and two SLV binaries 2-propanol+ NaCl and water + NaCl [9, 10]. It also includes the SLLE and LLE ternary equilibrium diagram at a temperature below boiling conditions which has been determined by several authors [12-16]. Figure 2 shows the equilibrium data at 298.15K, showing the different regions of the system at this temperature: a region with one liquid phase (L), two SL regions, one LL region and one SLL region. As shown in the Figure, water and 2-propanol are completely miscible but the presence of NaCl can split the mixture into two liquid phases in equilibrium.

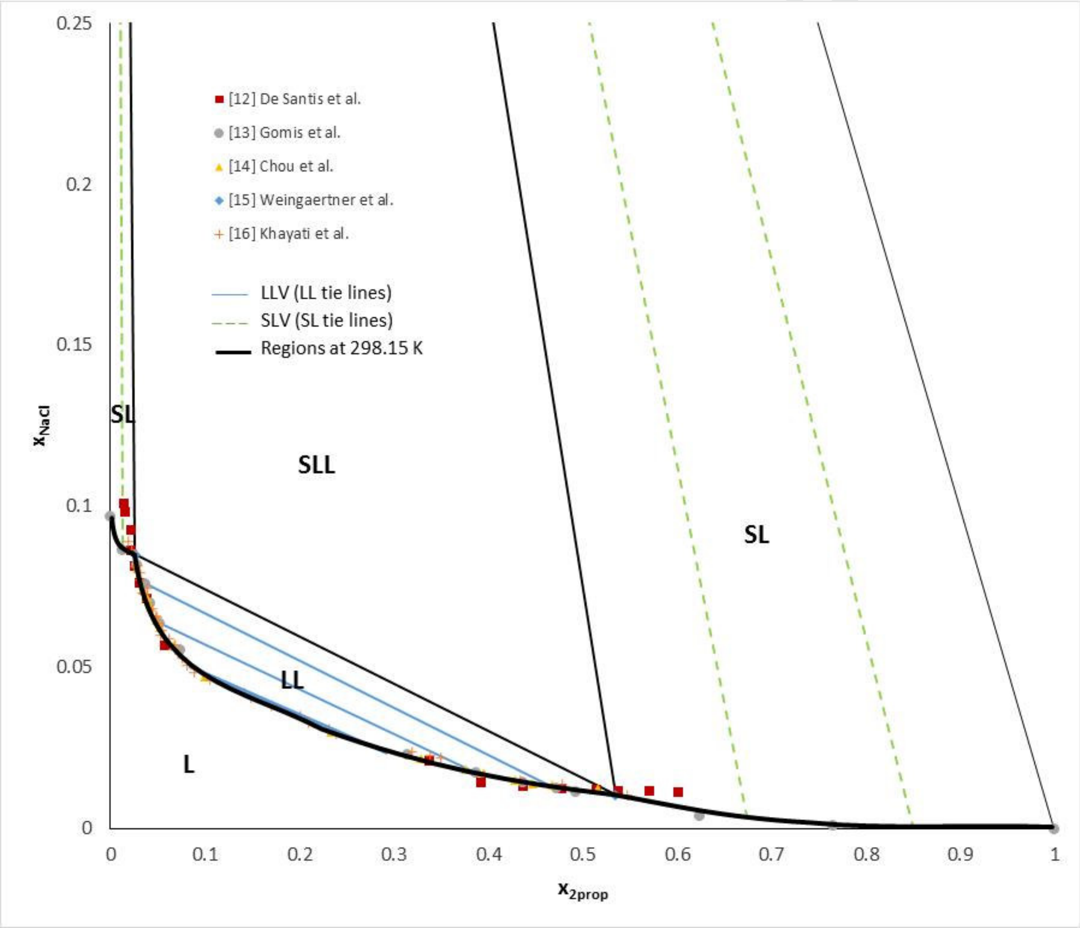

Fig. 2. Equilibrium diagram (mole fraction) of the water + NaCl + 2-propanol system at 298.15 K.

However, this feature of the splitting of phases in the presence of NaCl is not mentioned or considered in reference [17] when the authors present the results of their detailed study of the LV equilibrium of the system. Figure 3 represents the compositions of liquid phases whose boiling temperatures and vapor compositions were determined by the authors. Assuming that the size and position of the regions shown in Figure 2 do not change too much with the temperature (as will be shown later), the represented points of Figure 3 must belong to similar regions: one LV, two SLV regions, one LLV region and one SLLV region.

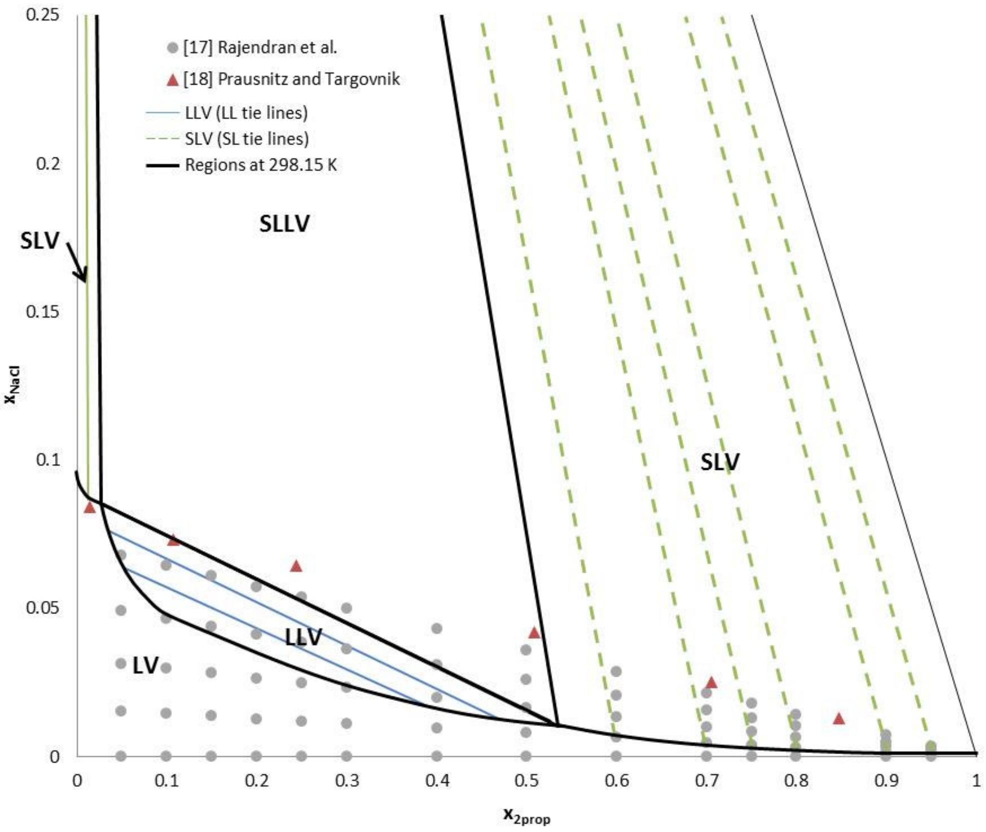

Fig. 3. Equilibrium diagram (mole fraction) of the water + NaCl + 2-propanol system with the points representative of the compositions of the liquids of the LVE determined by [17] and [18].

In a three-component system, according to the phases rule, the SLLV region is invariant for a determined pressure. Any mixture belonging to this SLLV region splits into two liquid phases and one solid which boil at a sole temperature and give a single composition of the vapor in equilibrium. However, the 7 points determined by [17] for this SLLV region are inconsistent since there are important differences in their boiling temperatures and in their vapor composition with errors of 2 K and 0.034 mole fraction respectively.

On the same Figure 3, the experimental data of [18] have been plotted. In this case, there are only two mixtures belonging to the SLLV region. The results given for them are consistent since the compositions of the vapor in equilibrium for these two points are identical (0.64 mole fraction in 2-propanol). Unfortunately, the authors do not give the boiling temperature to check that they should boil at the same temperature.

The inconsistencies in the SLV region (organic side) in [17] are also notorious. In this case, all the points of this SLV region placed in the same straight line passing through the vertex corresponding to NaCl split into one liquid and a solid which boil at a specific temperature and give the same vapor composition. Figure 3 shows 6 different straight lines of this type. The results given by the authors are again inconsistent with differences in temperatures of 0.4 K and 0.024 mole fraction in the composition of vapor between mixtures located on almost the same straight line.

The LLV region has also to meet requirements: all the points placed in the same tie line of this LLV region split into the same two liquid phases which boil at a determined temperature and give the same composition of vapor. As the size of the LLV region is small and the authors do not give experimental data of the tie lines of the region but only several points of that region which probably belong to different tie lines, it is difficult to check whether the results for the given points are consistent.

Finally, the authors [17] give boiling temperatures lower than the azeotropic temperature of the water+2-propanol binary pair for some ternary mixtures. For that, the existence of a ternary minimum boiling azeotrope would be necessary which is not possible when one of the components (the salt) is not present in the vapor in equilibrium since the composition of any ternary liquid mixture cannot be equal to that of its vapor.

In conclusion, the errors in the given results are in a gap of 2 K in boiling temperatures and 0.034 mole fraction in vapor compositions. These errors are much greater that the uncertainty of the measurements given by the authors and it is therefore necessary to determine new experimental consistent data of the system.

3. Experimental

3.1. Chemicals

Ultrapure water obtained by means of a MiliQPlus system was used. Its conductivity was less than 1 µS/cm. With respect to the rest of chemicals, Table 1 presents their description and includes ethanol since it was used as an internal standard for quantitative chromatographic analysis. The water content of the organic compounds presented in the table was determined by the Karl Fischer technique.

3.2. Apparatus and procedures

A modified vapor-liquid Fischer Labodest unit (*Fischer Labor und Verfahrenstechnik*) was used in the determinations. A previous paper [19] describes the operation of the equipment. It was also coupled to an ultrasonic transducer to enhance the mixing and dispersion of the phases and consequently the mass transfer between them, especially between the solid and the two liquid phases in the boiling flask. In this way, the original commercial equipment can be used in the determination of SLLV equilibrium.

To carry out an experiment, a mixture of water, 2-propanol and NaCl is placed inside the boiling flask where it is heated at a pressure of 101.3 kPa that is maintained by a Fisher M101 phase equilibrium control system. The boiling temperature is measured using a Pt100 sensor coupled to a digital thermometer (Hart Scientific 5615).

When equilibrium is reached, samples of the vapor and liquid phases are taken and analyzed. The methodology was described previously [19] and is summarized below. Vapor phases are taken for subsequent analysis by chromatography with a Thermal Conductivity Detector (TCD). Liquid phases (one or two) with or without solid in suspension are also taken from the equipment and separated at the boiling temperature. A small sample of these liquids is introduced into a vial with a known amount of ethanol, which serves as an internal standard. This sample is subsequently analyzed by gas chromatography with TCD to determine the water and 2-propanol. Another aliquot of the liquid phase is collected and the amount of salt it contains determined by a gravimetric method.

A Shimadzu GC14B gas chromatograph with a 2 m x 3 mm 80/100 Porapack Q packed column is employed to perform the analysis. When the liquid samples are analyzed, glass wool is put inside the glass insert to prevent NaCl from entering the column. Helium is used as carrier gas at a flow rate of 25 mL/min. The temperature of the oven is set to 463.15 K while the injector and the detector are set to 483.15K. A 100 mA current is used.

The equipment and methodology were verified by analysis of several LV equilibrium points of the water + 2-propanol binary system. The results are presented in table 6 together with the data of the ternary system. Moreover, they are plotted in Figure 1b and show the perfect accordance with the experimental LV equilibrium data in bibliography

The uncertainties were obtained from a statistical calculation of repeated measurements. The temperature standard uncertainty (u) is 0.06 K. The composition relative uncertainty ($u_r = \frac{u}{x}$) is 2% for all components except for the NaCl in the organic phase where the relative uncertainty is 5%.

4. Results

Experimental results. Equilibrium data and boiling temperature of different mixtures are shown in Tables 2, 3, 4, 5 and 6 corresponding to the different regions of the equilibrium diagram. Figure 4 shows some of these data: the LLVE region is represented by continuous tie lines joining the two liquid phases in equilibrium, and dashed lines joining the organic liquids with the point that is representative of the vapor phase. In the same way, the organic SLVE region is represented by continuous lines joining solid and liquid and dashed lines joining the liquids with the vapor. Lastly, the SLLVE region is represented by a triangle whose three vertices are the solid salt and the compositions of the two liquid phases that are in equilibrium with each other. It can be observed that the shape of this phase diagram at boiling temperatures is very similar to that of the water + NaCl + 1-propanol system [2].

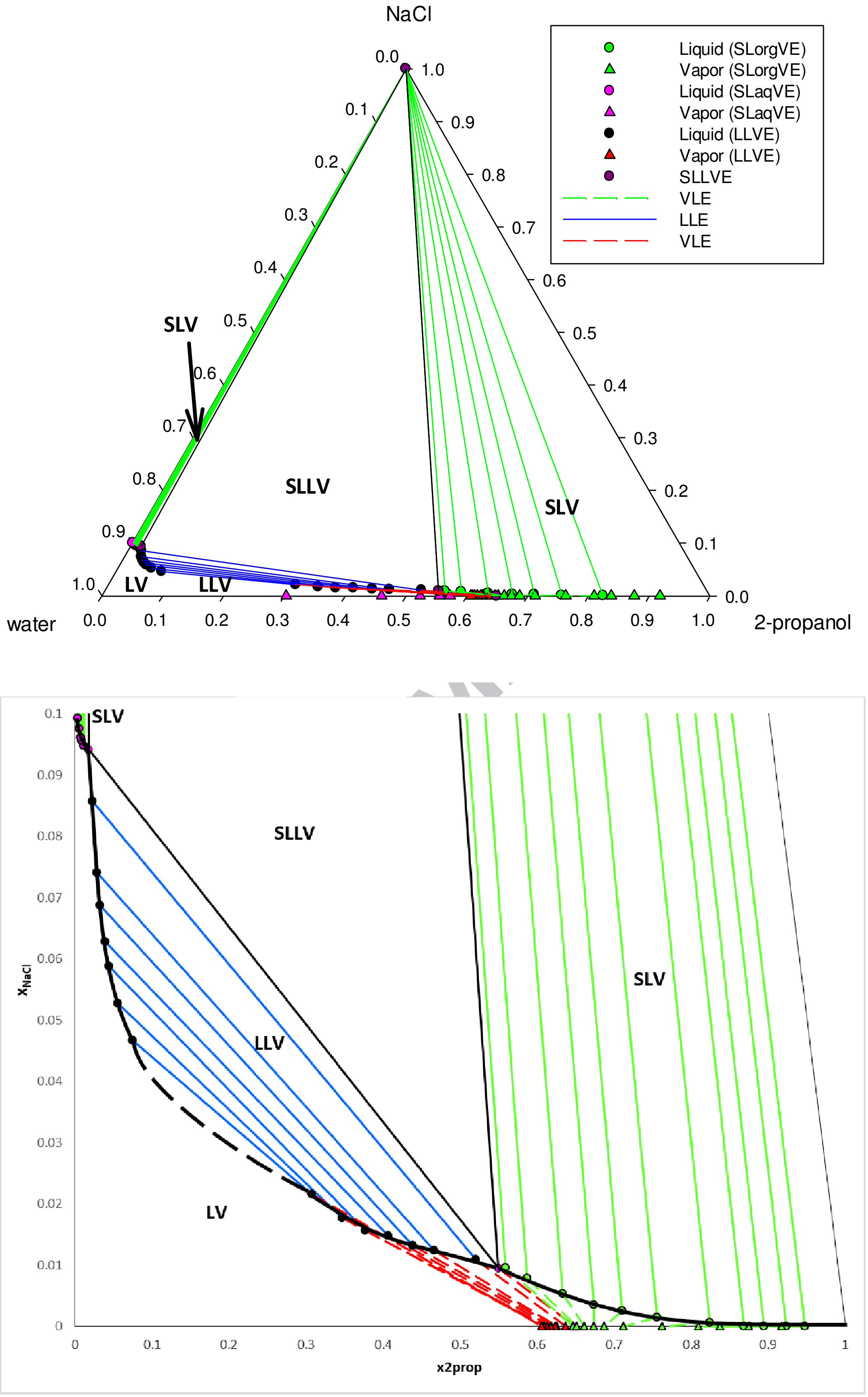

Fig. 4. a) Equilibrium diagram (mole fraction) for the water + NaCl + 2-propanol system at 101.3 kPa. b) Enlarged region.

Comparison with previous bibliography results. As mentioned previously, various authors [17, 18] have published experimental equilibrium data on the present system. Although we have demonstrated that they are not consistent, Figure 5 compares some of their results with ours. The comparison is only done for mixtures saturated with salt belonging therefore to the regions SLV and SLLV since the previous works do not consider the existence of the LLV region. For comparison, in the same Figure, the points determined by [6] have been drawn for the water + 2-propanol without salt binary system.

All the points of the system with salt of this Figure correspond to saturated mixtures of the ternary system and should be on the same line which must be a horizontal straight line for the mixtures belonging to the SLLV region. The experimental points of our work verify this condition. However, there is a great dispersion and inconsistency of the points determined previously in [17].

Finally, the two points determined in [18] for the SLLV are consistent and similar to our results but the values given for the mole fraction of 2-propanol in the vapor for the two mixtures of the SLV region are very small with respect to those of the binary system without salt.

In spite of these inconsistencies, as these were the only published data available for this system they were used in the studies with thermodynamic models involving electrolytes.

Phase diagram and evolution with temperature. By interpolation in Tables 2-6 the diagrams of boiling temperature isotherms and iso composition of phase vapor have been drawn in Figures 6 and 7. As shown previously, the SLLV region remains invariant and the isotherms and the vapor iso composition lines of the SL regions are straight lines

Notice the very small differences in the boiling temperatures of some of the characteristic points of the system such as the binary azeotrope water + 2-propanol (353.31 K), the plait point of the LLV region (354.1 K approximately) and the invariant SLLV mixture (353.60 K). This feature causes a great part of the mixtures in the system to boil within a very narrow range of temperatures of only 1 K.

Another characteristic that could be extracted from these Figures and considered as an anomaly is the slight decreases in the boiling point of many mixtures when salt is added. For example, consider a water + 2-propanol binary mixture with 0.2 mole fraction in 2-propanol. The boiling point of this mixture is 355.3 K according to Figure 6. As salt is added to it, the liquid mixture remains homogeneous and its boiling point decreases slightly until 354.1 K. If more salt is added, the liquid mixture splits into two liquid phases and the boiling point continues to decreases until 353.6 K. If more salt is added, it does not dissolve and the boiling point of the SLL mixture remains invariant at 353.6 K. All the mixtures in the 0.05-0.55 range in 2-propanol present this characteristic of decreases in the boiling point when salt is added.

The curves of Figures 6 and 7 allow us to draw the Figure 8 to analyze the evolution with the temperature of the different equilibrium regions of the system and compare them with those of the system with 1-propanol [2]. In spite of the similarity of both systems, the evolution is quite different. When the temperature increases the first mixture to reach the boiling point in both systems is the binary azeotrope. However, as the concentration of propanol in the azeotrope is considerably higher in the system with 2-propanol and it is placed far from the LL region the evolution is very different. In the case of 2-propanol, the SLV (organic branch) region appears first (Figures 8 b, c), the SLL region boils at less temperature than the samples with only LL. Consequently, the LLV triangle (Figures 8 d, e, f) moves down differently than the system with 1-propanol which moves up since the binary azeotrope water + 1-propanol is placed close to the LL region.

At higher temperatures (Figures 8 g, h) the L region (aqueous branch) shrinks in size and the LV regions increases in a similar way as in the system with 1-propanol.

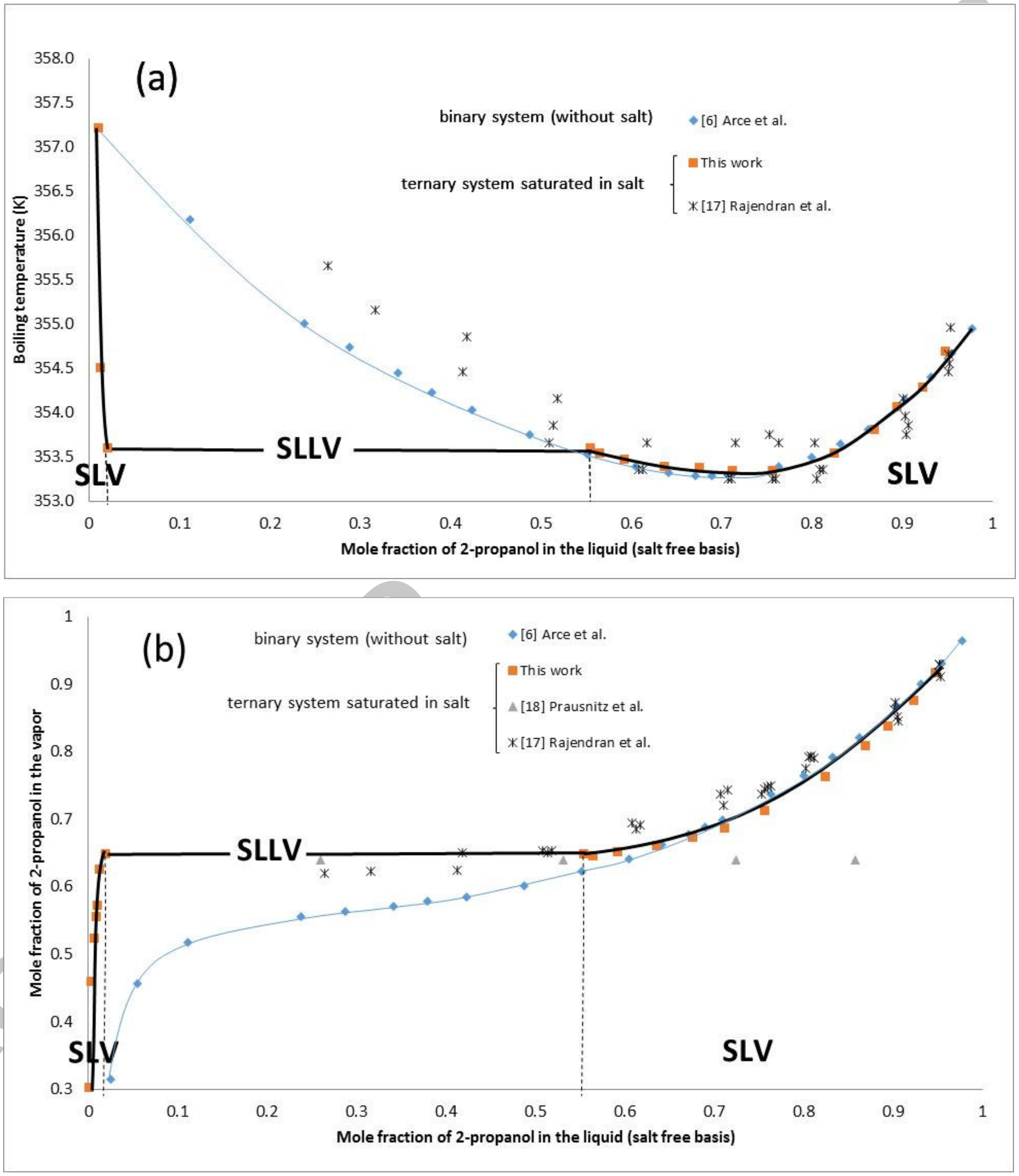

Fig. 5. Boiling temperature (a) and composition of the vapor (b) for samples saturated with salt (SLV and SLLV).

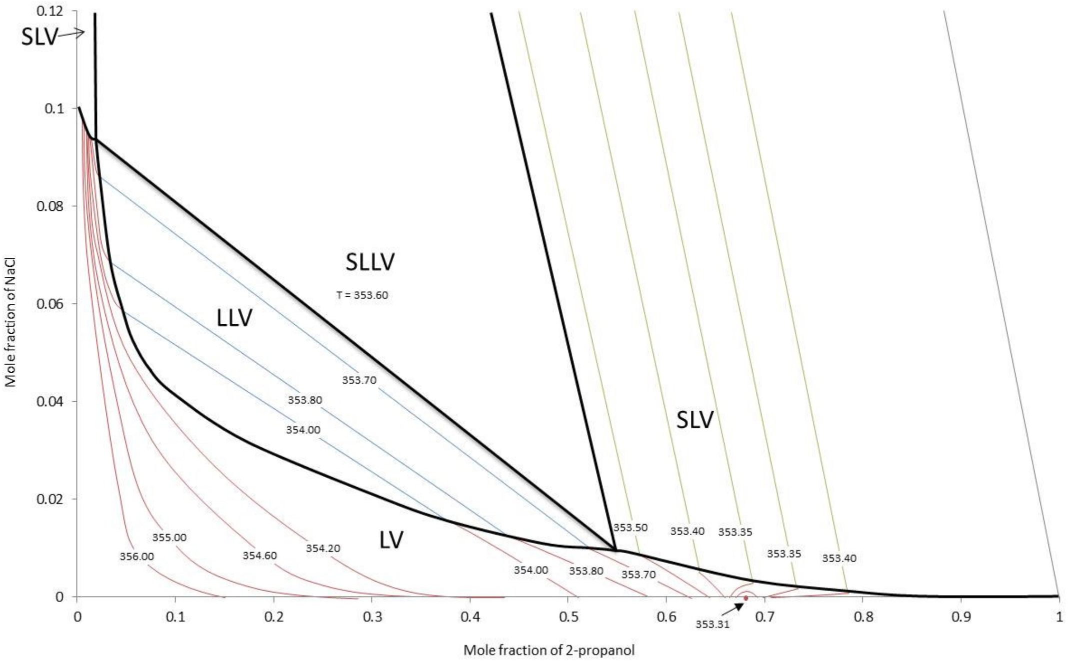

Fig. 6. LLV, SLV and LV isotherms for the ternary system water + NaCl + 2-propanol at 101.3 kPa. Parameter of the curves = boiling temperature (K).

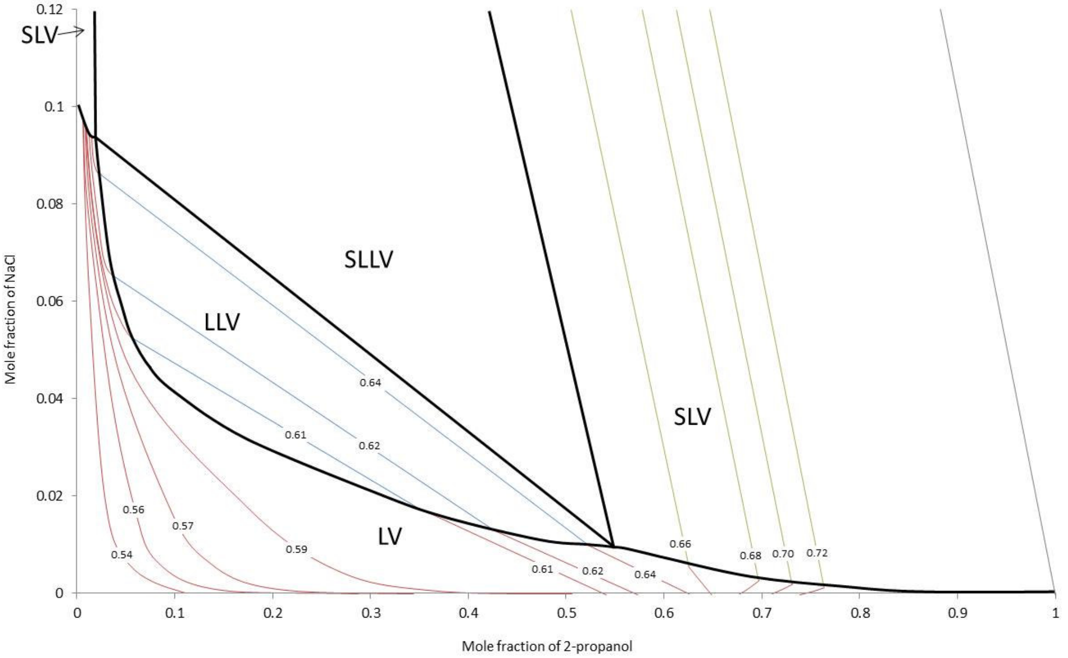

Fig. 7. Curves of iso concentration of 2-propanol in the vapor phase for the system water + NaCl + 2-propanol at 101.3 kPa. Parameter of the curves = mole fraction of 2-propanol in the vapor.

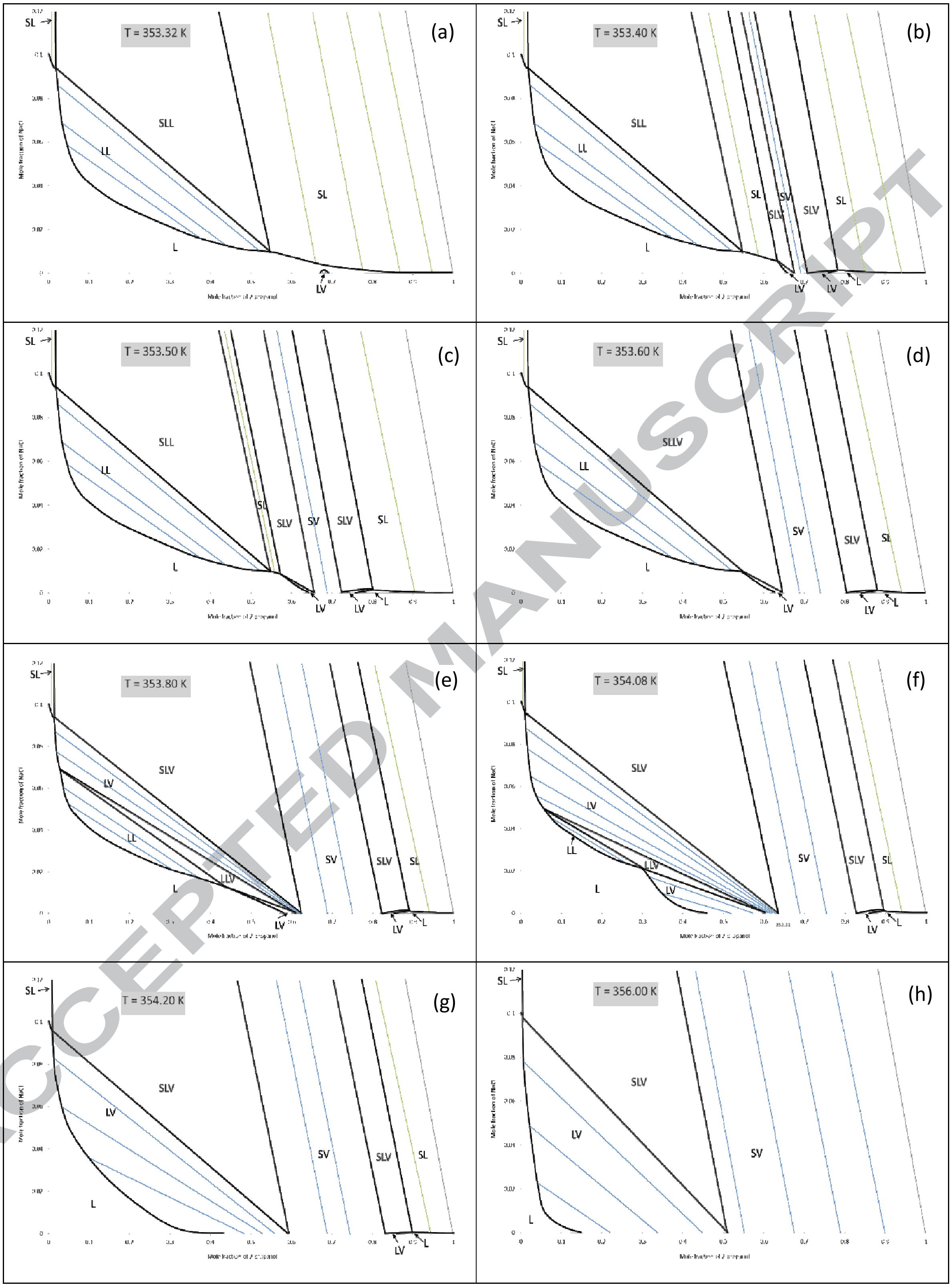

Fig. 8 (a-h). Evolution with temperature (increasing T) of the different equilibrium regions of the water + NaCl + 2-propanol system at 101.3 kPa.

4. Conclusions

The SLLV phase equilibria of the water + NaCl + 2-propanol mixture have been studied at 101.3 kPa and the influence analyzed of the salt on the vapor-liquid-liquid-solid equilibrium. The shape of the phase diagram at boiling temperatures is very similar to that of the water + NaCl + 1-propanol system but the evolution with temperature of this equilibrium diagram is very different.

Previously published experimental data have been compared with the data obtained in this work and it has been demonstrated that the previously published data were inconsistent: in addition to the usual considerations for consistency of LL and LV equilibrium data, the water + non volatile salt + organic solvent type systems must meet the following consistency rules in the different regions that can be checked drawing the equilibrium diagrams with the boiling isotherms and isocomposition of the vapor in equilibrium:

- For the whole system: the boiling temperature of any ternary mixture is always higher than the minimum boiling temperature of the pure compounds or their binary mixtures since the existence of a ternary minimum boiling azeotrope is not possible.
- For the SLLV region: the SLLV region is invariant for a determined pressure. Any mixture belonging to this SLLV region splits into two liquid phases and one solid which boil at a sole temperature and give a single composition of the vapor in equilibrium.
- For the SLV regions: all the points of an SLV region placed in the same straight line passing through the vertex corresponding to the salt split into one liquid and a solid which boil at the same temperature and give the same vapor composition.
- For the LLV region: all the points placed in the same tie line of the LLV region split into the same two liquid phases which boil at the same temperature and give the same composition of vapor.

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TABLES

Table 1.

Specifications of chemical compounds.

Chemical	Provider	Initial purity (mass %)	Water content KF[a] (mass %)	Purification method	Analysis method
NaCl	VWR	> 99.9		none	
2-Propanol	Merck	> 99.5	0.09	none	GC [b]
Ethanol	VWR	> 99.8	0.02	none	GC [b]

[a] KF = Karl Fischer Technique

[b] GC = Gas chromatography

Table 2.

Equilibrium data (mole fraction) corresponding to the three phase region ($1S + 1L_{org} + 1V$) of the water + NaCl + 2-propanol system at 101.3 kPa. Solid composition is pure NaCl.

	Liquid			Vapor	
T/K	Water[1]	NaCl[2]	2-Propanol[1]	Water[1]	2-Propanol[1]
353.55	0.431	0.0095	0.559	0.354	0.646
353.48	0.405	0.0079	0.587	0.348	0.652
353.40	0.362	0.0053	0.633	0.339	0.661
353.38	0.323	0.0035	0.673	0.326	0.674
353.35	0.288	0.0025	0.710	0.313	0.687
353.35	0.243	0.0014	0.756	0.288	0.712
353.55	0.176	0.0006	0.823	0.237	0.763
353.82	0.131	< 0.0005	0.869	0.191	0.809
354.07	0.106	< 0.0005	0.894	0.163	0.837
354.29	0.077	< 0.0005	0.923	0.125	0.875
354.70	0.052	< 0.0005	0.947	0.082	0.918

T is in K with a standard uncertainty of 0.06 K, the pressure standard uncertainty is 0.1 kPa and composition relative standard uncertainty is $u_r = \frac{u}{x}$ is 2% except for the NaCl where the relative standard uncertainty is 5%.

[1] obtained by GC-TCD

[2] obtained by gravimetric analysis

Table 3.

Equilibrium data (mole fraction) corresponding to the three phase region (1S + 1L_{aq} + 1 V) of the water + NaCl + 2-propanol system at 101.3 kPa. Solid composition is pure NaCl.

	Liquid			Vapor	
T/K	Water[1]	NaCl[2]	2-Propanol[1]	Water[1]	2-Propanol[1]
354.51	0.894	0.0948	0.011	0.374	0.626
357.23	0.895	0.0956	0.009	0.428	0.572
358.65	0.896	0.0961	0.008	0.445	0.555
361.57	0.897	0.0975	0.006	0.477	0.523
367.10	0.898	0.0992	0.003	0.540	0.460
372.71	0.898	0.1005	0.001	0.697	0.303

T is in K with a standard uncertainty of 0.06 K, the pressure standard uncertainty is 0.1 kPa and composition relative standard uncertainty is $u_r = \frac{u}{x}$ is 2%

[1] obtained by GC-TCD

[2] obtained by gravimetric analysis

Table 4.

Equilibrium data (mole fraction) corresponding to the three phase region (1 L_{aq} + 1L_{org} + 1 V) of the water + NaCl + 2-propanol system at 101.3 kPa.

	Aqueous Liquid			Organic Liquid			Vapor	
T/K	Water[1]	NaCl[2]	2-Propanol[1]	Water[1]	NaCl[2]	2-Propanol[1]	Water[1]	2-Propanol[1]
353.63	0.891	0.086	0.023	0.469	0.011	0.520	0.363	0.637
353.74	0.897	0.074	0.029	0.521	0.012	0.467	0.374	0.626
353.80	0.898	0.069	0.033	0.549	0.013	0.438	0.377	0.623
353.88	0.898	0.063	0.039	0.579	0.015	0.406	0.382	0.618
353.95	0.897	0.059	0.044	0.607	0.016	0.377	0.387	0.613
354.01	0.891	0.053	0.056	0.635	0.018	0.347	0.391	0.609
354.08	0.878	0.047	0.075	0.670	0.022	0.308	0.394	0.606

T is in K with a standard uncertainty of 0.06 K, the pressure standard uncertainty is 0.1 kPa and composition relative standard uncertainty is $u_r = \frac{u}{x}$ is 2% except for the NaCl in the organic phase where the relative standard uncertainty is 5%.

[1] obtained by GC-TCD

[2] obtained by gravimetric analysis

Table 5.

Equilibrium data (mole fraction) corresponding to the four phase region (1S + 1 L_{aq} + 1 L_{org} + 1 V) of the water + NaCl +2-propanol system at 101.3 kPa.

Phase	Water[1]	NaCl[2]	2-Propanol[1]
Solid	0.000	1.000	0.000
Organic Liquid	0.441	0.0095	0.549
Aqueous Liquid	0.888	0.094	0.018
Vapor	0.351	0.000	0.649
Temperature (K)	353.60		

T is in K with a standard uncertainty of 0.06 K, the pressure standard uncertainty is 0.1 kPa and composition relative standard uncertainty is $u_r = \frac{u}{x}$ is 2% except for the NaCl in the organic phase where the relative standard uncertainty is 5%.

[1] obtained by GC-TCD

[2] obtained by gravimetric analysis

Table 6.

Equilibrium data (mole fraction) corresponding to the liquid-vapor region of the water + NaCl +2-propanol system at 101.3 kPa.

	Liquid			Vapor	
T(K)	Water[1]	NaCl[2]	2-Propanol[1]	Water[1]	2-Propanol[1]
355.33	0.753		0.247	0.451	0.549
353.32	0.347		0.653	0.327	0.673
353.30	0.311		0.689	0.312	0.688
354.13	0.631	0.0007	0.368	0.413	0.587
354.47	0.736	0.0032	0.261	0.421	0.579
354.66	0.791	0.0020	0.207	0.428	0.572
354.86	0.856	0.0078	0.136	0.431	0.569
355.09	0.910	0.0083	0.082	0.438	0.562
356.00	0.935	0.0129	0.052	0.448	0.552
360.86	0.966	0.0123	0.021	0.548	0.452

T is in K with a standard uncertainty of 0.06 K, the pressure standard uncertainty is 0.1 kPa and composition relative standard uncertainty is $u_r = \frac{u}{x}$ is 2% except for the NaCl in the liquids where the relative standard uncertainty is 5%.

[1] obtained by GC-TCD

[2] obtained by gravimetric analysis