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Acoustical and thermodynamic studies of ternary liquid mixtures of primary alkanols in mixed binary solvent at varying temperatures

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Densities (ρ), viscosities (η) and speeds of sound (U) of ternary liquid mixtures of primary alkanols such as 1-pentanol and 1-hexanol, 1-heptanol and 1-octonal with the fixed binary solvent mixtures of N-N dimethylacetamide (DMA) and diethyl ketone (DEK) (ratio 4:2 respectively) were measured over the entire composition range at 303.15, 308.15 and 313.15K and at normal atmospheric pressure. The observed experimental data have been utilized to evaluate some of the thermo dynamical parameters such as adiabatic compressibility (β), intermolecular free length (L_f), free volume (V_f), internal pressure (π_i), viscous relaxation time (τ) and Gibb's energy (ΔG). The variation of these parameter with composition were discussed from the view point of intermolecular interactions in these mixtures. It was observed that the interaction between DMA (proton acceptor) and 1-alkanols is due to hydrogen bonding. The present study observes that there was a dissociation in the associated structure of 1-alkanols when they are further added with binary solvent mixture (DMA + DEK).

Keyword: Adiabatic compressibility, free length, internal pressure, hydrogen bonding, dipole-dipole interactions, molecular interaction

1. Introduction

Ultrasonic velocities, densities, viscosities and derived thermodynamic and acoustical parameters are of considerable interest in understanding the intermolecular interactions in binary as well as ternary liquid mixtures [1-5]. The thermodynamic properties of multi-component liquid mixtures and their analysis are important in an industrial process. Ultrasonic studies can also be used to determine the extent of complexation and to calculate the formation constant values of charge transfer complexes [6-7]. Hydrogen bonded systems are very interesting because hydrogen bonds play a vital role in chemical, physical and biological processes. It is well known that alcohols are highly associated through hydrogen

bonds, thus their structure and properties are determined mainly by quasi-chemical bonds between the molecules which result in the formation of multimers of different size and structure [8]. Measurement of ultrasonic velocity has been adequately employed in understanding the nature of molecular interaction in pure liquid and liquid mixtures. Ultrasonic studies of mixed solvent systems are of importance because of their extensive use in textile, leather and pharmaceutical industries. Organic solvents whose miscibility is unlimited usually form H–bonds in aqueous solutions. Some solvents also present auto-association by hydrogen bonds in pure state but others do not. The practical application of mixed solvents rather than single

solvent in industrial and biological process has been recognized all over the world as they provide a wide choice of solutions with appropriate properties [9].

The present study deals with the thermodynamic study of mixed solvent system at different temperatures. The liquids under investigation have been chosen on the basis of their industrial applications. These applications have greatly stimulated the need for extensive information on the thermodynamic, acoustic and transport properties of these solvents and their mixtures [10-11]. In general, the hydroxyl group makes the alcohol molecule polar. Those groups can form hydrogen bonds to one another and to other compounds. This hydrogen bonding means that alcohols can be used as protic solvents. Two opposing solubility trends in alcohols are: the tendency of the polar OH to promote solubility in water, and the tendency of the carbon chain to resist it. All simple alcohols are miscible in organic solvents. The simple alcohols that are used in the present study are 1-pentanol, 1-hexanol, 1-heptanol and 1-octanol.

It is well known that amides interact with alkanols by dipole–dipole interactions, and form some hydrogen-bonded complexes or hetero-associates [12-13]. On the basis of those studies, it is interesting to note that the thermodynamic results of the addition of the amide group with alkanols to obtain some information on the molecular interactions. On the other hand, N,N-dimethylacetamide is an organic compound abbreviated as DMA, this colourless liquid is miscible with water and the majority of organic liquids. DMA is a common solvent for chemical reactions. Dimethylacetamide is a polar aprotic solvent with a high boiling point, which can be synthesized from methyl formate and dim ethylamine or by reaction of dimethylamine with carbon monoxide. DMA is used in the production of acrylic fibers and plastics. It is also used as a solvent in peptide coupling for pharmaceuticals, in the development and production of pesticides, and in the manufacture of adhesives, synthetic leathers, fibers, films, and surface coatings.

Diethyl Ketone (DEK) is an organic compound. This colourless liquid, belonging to a ketone group, which is widely used as a solvent. DEK has quite low solubility in water, making it useful for liquid-liquid extraction. It has a similar polarity to ethyl acetate, but greater stability towards aqueous acid and base. It can be used to extract gold, silver and other precious metals from cyanide solutions, such as those found at gold mines, to determine the levels of those dissolved metals.

Keeping these important aspects in mind, the authors has attempted to measure the ultrasonic velocity, density and viscosity for the following four ternary liquid system.

System –I:	1-pentanol+N-N dimethylactamide + diethylketone
System-II:	1-hexanol+N-N dimethylactamide + diethylketone
System-III:	1-heptanol+N-N dimethylactamide + diethylketone
System-IV:	1-octanol+N-N dimethylactamide + diethylketone

2. Materials and Methods

In the present work, the chemicals, which have been used, are analytical reagent (AR) and spectroscopic reagent (SR) grades of minimum assay of 99.9% obtained from E-Mark, Germany and Sd fine chemical, India. The purities of the above chemicals were checked by the density measurements at 303.15, 308.15 and 313.15K and were compared with available literature values. The ternary liquid mixtures of different known composition were prepared by mole fraction (X) basis. The density, viscosity and ultrasonic velocity were measured as a function of the ternary liquid mixtures of primary alkanols which were added to the fixed binary solvent mixtures of DMA and diethyl ketone (DEK) at 303.15, 308.15 and 313.15K. for this purpose, binary liquid mixture of DMA (X_2) and DEK (X_3) with a fixed ratio X_2/X_3=4:2 respectively was prepared. The primary alkanols (X_1) were added as solutes in the binary solvent at different mole fraction basis. The density was determined using a specific gravity bottle (5ml) by relative measurement method. The weight of the sample was measured using an electronic digital balance with an accuracy of ± 0.1 mg (Model: Shimadzu, Japan Make, Ax-200). An Ostwald's viscometer

(10ml) was used with an accuracy of ± 0.001 Nsm^{-2} for the viscosity measurement. Efflux time was determined by using a digital chronometer to within ± 0.001s. An ultrasonic interferometer having a fixed frequency of 2MHZ (Mittal Enterprises, New Delhi. Model: F-81) has been used for velocity measurements. The overall accuracy in the velocity measurement is ± 2ms^{-1}. An electronically digital operated constant temperature water bath (RAAGA Industries, Chennai) has been used to circulate water through a double walked measuring cell make up of steel containing the experimental solution at the desired temperature. The accuracy in the temperature measurement is ± 0.1K. Extreme care was taken to avoid contamination during mixing. The present work was conducted under normal atmospheric pressure.

3. Results and Discussion

Experimentally determined values of the density (ρ), viscosity(η) and ultrasonic velocity(υ) at 303.15, 308.15, & 313.15 for the systems I& II are reported in Table 1, whereas the same has been furnished in Table 2. The adiabatic compressibility (β), intermolecular free length (L$_f$), free volume (V$_f$), and internal pressure π$_i$ for the systems I, II, and III, IV are depicted in Table 3 and Table 4 respectively. Tables- 5&6 respectively report the values of viscous relaxation time(τ) and Gibb's free energy(ΔG*) for the four ternary liquid systems at 303, 308 and 313K.
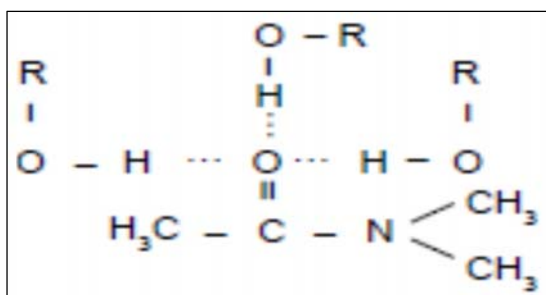
Table 1: The density (ρ), viscosity (η) and ultrasonic velocity (U) of System-I & System-II

Mole fraction (X$_1$)	Density ρ/(kg/m^3)			Viscosity η/(× 10^{-3}NSm^{-2})			Ultrasonic Velocity U/(m/s)		
	Temperature (K)								
	303	308	313	303	308	313	303	308	313
System I 1-pentanol+N - N dimethylactamide + diethylketone									
0.1000	883.085	880.677	876.046	0.7754	0.6957	0.6723	1328	1325	1320
0.3327	875.202	865.584	858.906	1.1893	0.7257	0.6957	1309	1280	1262
0.5551	850.351	847.150	843.097	1.4275	0.8972	0.7256	1294	1276	1252
0.6993	841.437	835.629	829.380	1.4634	1.0872	0.7951	1282	1270	1269
0.9089	831.749	809.104	804.704	1.4939	1.2356	0.8325	1272	1265	1257
System II 1-hexanol+ N - N dimethylactamide + diethylketone									
0.0997	879.671	872.267	866.119	0.7824	0.7372	0.6888	1340	1326	1311
0.3353	858.671	852.697	845.977	1.0183	0.9861	0.8978	1337	1323	1308
0.5532	846.472	837.447	825.487	1.5677	1.2718	1.0178	1332	1316	1301
0.6983	837.255	826.380	815.250	1.6872	1.3913	1.2531	1327	1310	1295
0.9089	819.825	813.998	807.428	1.7276	1.4824	1.4674	1320	1306	1284

Table 2: The density (ρ), viscosity (η) and ultrasonic velocity (U) of System-III & System-IV

Mole fraction (X_1)	Density $\rho/(kg/m^3)$			Viscosity $\eta/(\times 10^{-3} NSm^{-2})$			Ultrasonic Velocity $U/(m/s^{-1})$		
	Temperature (K)								
	303	308	313	303	308	313	303	308	313
System III 1- heptanol + N - N dimethylactamide + diethylketone									
0.1002	881.415	872.537	863.215	0.8115	0.7751	0.7297	1341	1332	1324
0.3326	861.789	856.881	850.998	1.1526	1.0967	1.0219	1336	1326	1312
0.5557	854.403	848.641	843.655	1.7657	1.6332	1.5120	1331	1320	1301
0.6997	841.572	829.079	818.186	2.0831	2.0759	1.8475	1294	1287	1275
0.9059	834.808	809.198	805.784	2.3901	2.1759	2.0475	1289	1279	1263
System –IV 1-octanol+ N - N dimethylactamide + diethylketone									
0.1009	882.495	878.695	873.071	0.8215	0.7763	0.7289	1345	1328	1311
0.3333	860.615	856.551	851.881	1.3678	1.2678	1.1993	1334	1321	1304
0.5550	849.675	845.165	841.759	1.6341	1.41567	1.3517	1316	1303	1290
0.6980	840.761	836.779	831.238	2.1578	2.0104	1.7271	1301	1292	1284
0.96807	830.023	809.104	804.354	2.9645	2.6855	2.3408	1297	1281	1274

In all the four liquid systems, the ultrasonic velocity (U) decreases with increasing molar concentration of 1-alkanols as well as rise of temperature. It is observed that as the number of hydrocarbon group increases, which lead to decrease in ultrasonic velocity. This behaviour at such concentrations which is different from the ideal mixture behaviour can be attributed to intermolecular interactions in the systems studied [14].

Fig 1: Interaction between DMA-Alkanols

From Tables 3 & 4 one can notice that the values of adiabatic compressibility and intermolecular free length increase with increase of mole fraction of alkanols concentrations as well as rise of temperature. Such a continuous increase in

adiabatic compressibility with respect to the solute concentration has been qualitatively ascribed to the effect of hydrogen bonding or dipole-dipole interactions [15].

N-N dimethylacetamide (DMA) is normally belonging to the amide group due to its wide range applicability as solvent in chemical and biological processes. It is highly polar, aprotic solvent and its molecules are associated through dipolar interactions [16]. On the other hand, alkanols are polar liquids, strongly self-associated by hydrogen bonding to extents of polymerization that may differ depending upon temperature, chain length and position of the OH group. Alkanol size, chain length etc., (which is related through density and refractive index) are important parameters that must be taken into account to explain the behaviour of the DMA, diethyl ketone (DEK) with 1-alkanols mixtures.

Alkanols are liquids which are associated through the hydrogen bonding and in the pure state, they exhibit an equilibrium between the monomer and multimer species. Also, they can be associated with any other groups having some degree of polar attractions [16]. Due to polar nature of DMA and alkanols, the dipole-dipole interactions

prevail in these mixtures. When the compounds are mixed, the changes that occur in association equilibria are evidently rupture of the hydrogen bonds [17] in alkanols and resulting in DMA-DMA dipole-dipole interactions and the formation of O-H. C=O and perhaps even O-H. $N(CH_3)_2$ hydrogen bonds between the components.

Table 3: The adiabatic compressibility (β) free length, (L_f) free volume (V_f) and internal pressure ($π_i$) of System-I & II.

Mole fraction (X_1)	Adiabatic compressibility $β/(×10^{-10}m^2N^{-1})$			Free length $L_f/×10^{-10}m$			Free volume $V_f/×10^{-7} m^3 mol^{-1}$			Internal pressure $π_i/10^6Nm^{-2}$		
	Temperature (K)											
	303	308	313	303	308	313	303	308	313	303	308	313
System-I 1-pentanol+N - N dimethylactamide + diethylketone												
0.1000	6.4154	6.4681	6.5512	5.0538	5.1149	5.1963	2.0514	2.4056	2.5180	376.850	362.416	361.478
0.3327	6.6682	7.0513	7.3103	5.1524	5.3405	5.4891	1.0625	2.1554	2.4812	465.175	370.812	369.689
0.5551	7.0231	7.2500	7.5668	5.2877	5.4153	5.5845	0.7987	1.5697	2.0977	500.612	405.253	372.730
0.6993	7.2310	7.3499	7.4872	5.3654	5.4525	5.5551	0.7377	1.1799	1.8712	504.709	442.262	382.558
0.9089	7.5951	7.7235	7.8649	5.4988	5.5893	5.6935	0.7326	0.9660	1.7301	506.086	460.671	384.013
System –II 1-hexanol+ N - N dimethylactamide + diethylketone												
0.0997	6.3309	6.5202	6.7176	5.0204	5.1355	5.2618	2.1000	2.2608	2.4609	368.853	363.830	357.758
0.3353	6.5149	6.7001	6.9091	5.0928	5.2059	5.3363	1.5014	1.5509	1.7549	394.957	381.336	380.527
0.5532	6.6585	6.8949	7.1570	5.1487	5.2810	5.4312	0.8232	1.1064	1.5192	467.096	427.285	387.042
0.6983	6.7826	7.0514	7.3142	5.1964	5.3406	5.4905	0.5935	0.6763	0.7542	469.505	432.462	415.736
0.9089	7.0005	7.2026	7.5121	5.2792	5.3975	5.5643	0.5156	0.6223	0.7447	477.265	453.952	432.237

Table 4: The adiabatic compressibility (β) free length, (L_f) free volume (V_f) and internal pressure ($π_i$) of System-III & IV

Mole fraction (X_1)	Adiabatic compressibility $β/(×10^{-10}m^2N^{-1})$			Free length $L_f/×10^{-10}m$			Free volume $V_f/×10^{-7} m^3 mol^{-1}$			Internal pressure $π_i/10^6Nm^{-2}$		
	Temperature (K)											
	303	308	313	303	308	313	303	308	313	303	308	313
System III 1- heptanol + N - N dimethylactamide + diethylketone												
0.1002	6.3090	6.4596	6.6085	5.01174	5.1116	5.2189	2.0393	2.1627	2.3464	369.063	365.430	358.891
0.3326	6.50108	6.6373	6.8265	5.0874	5.1814	5.3041	1.3373	1.4247	1.5589	398.779	395.402	388.171
0.5557	6.6066	6.7628	7.0029	5.1285	5.2302	5.3460	0.7744	0.8598	0.9444	455.430	455.096	436.680
0.6997	7.0964	7.2819	7.5184	5.3152	5.4272	5.5392	0.5020	0.5714	0.6172	507.454	489.193	480.297
0.9059	7.3865	7.5544	7.7799	5.4228	5.5278	5.6347	0.4168	0.4995	0.5537	510.531	495.112	485.316
System –IV 1-octanol+ N - N dimethylactamide + diethylketone												
0.1009	6.2638	6.4530	6.6641	4.9937	5.1090	5.2150	2.0590	2.1168	2.2316	364.392	361.610	356.865
0.3333	6.5294	6.6902	6.9034	5.0985	5.2020	5.3078	1.2404	1.3470	1.4920	384.870	377.316	374.010
0.5550	6.7957	6.9689	7.1389	5.2014	5.3093	5.3976	0.9526	1.1890	1.6702	403.819	382.228	380.838
0.6980	7.0270	7.1591	7.2970	5.2892	5.3812	5.4571	0.4896	0.5717	0.6898	435.255	427.207	401.878
0.96807	7.2937	7.5317	7.6597	5.3886	5.5195	5.5910	0.3316	0.3794	0.5020	483.496	434.108	411.399

Table 5: The viscous relaxation time (τ) and Gibb's free energy (ΔG*) of System I & II

Mole fraction (X₁)	Viscous relaxation time τ /(× 10⁻¹²S)			Gibb's free energy ΔG*/(× 10⁻²⁰ kJ mol⁻¹)		
	Temperature (K)					
	303	308	313	303	308	313
System – I 1-pentanol+N - N dimethylactamide + diethylketone						
0.1000	0.6616	0.5984	0.5878	0.5976	0.5718	0.5603
0.3327	1.0547	0.6805	0.6764	0.7927	0.6248	0.6009
0.5551	1.3333	0.8651	0.7302	0.8906	0.7285	0.6740
0.6993	1.4073	0.0627	0.7917	0.9133	0.8159	0.7089
0.9089	1.5090	1.2692	0.8708	0.9452	0.8914	0.7501
System – II 1-hexanol+ N - N dimethylactamide + diethylketone						
0.0997	0.6587	0.6292	0.6024	0.5958	0.5931	0.5909
0.3353	0.9823	0.8787	0.8249	0.7629	0.7351	0.7267
0.5532	1.3883	1.1662	0.9688	0.9076	0.8554	0.7962
0.6983	1.5219	1.3048	1.2190	0.9460	0.9032	0.8954
0.9089	1.6885	1.5829	1.4660	0.9963	0.9853	0.9751

Table 6: The viscous relaxation time (τ) and Gibb's free energy (ΔG*) of System-III & IV

Mole fraction(X₁)	Viscous relaxation time τ /(× 10⁻¹²S)			Gibb's free energy ΔG*/(× 10⁻²⁰ kJ mol⁻¹)		
	Temperature (K)					
	303	308	313	303	308	313
System-III 1- heptanol + N - N dimethylactamide + diethylketone						
0.1002	0.6909	0.6559	0.6303	0.61580	0.6108	0.6105
0.3326	1.2965	1.1681	1.0927	0.8790	0.8561	0.8482
0.5557	1.5514	1.4689	1.3082	0.9541	0.9535	0.9577
0.6997	2.2509	2.1073	1.9473	1.1097	1.1070	1.0978
0.9059	2.4480	2.2122	2.05756	1.1448	1.1276	1.1216
System – IV 1-octanol+ N - N dimethylactamide + diethylketone						
0.1009	0.6843	0.6625	0.6460	0.6178	0.6150	0.6143
0.3333	1.1878	1.1273	1.0311	0.8424	0.8410	0.8231
0.5550	1.4769	1.3121	1.2434	0.9335	0.9055	0.9040
0.6980	2.0166	1.9032	1.6761	1.0637	1.0636	1.0330
0.9680	2.7379	2.5569	2.2663	1.1916	1.1892	1.1633

Mixing of DMA with alkanols would release the dipoles of alkanols due to the breaking of hydrogen bonds. As a result, the free dipoles released from the alkanols in association with DMA molecules forming strong hydrogen bonds, hence stronger molecular association existing between the DMA and alkanols molecules through hydrogen bonding [18]. Further, mixing of 1-alkanols with binary solvent mixture causes dissociation of hydrogen bonded structure of 1-alkanols and subsequent formation of (new) H-bond (C=O –H….O) between proton acceptor oxygen atom (with lone pair of electrons) of C=O group of DMA and proton of OH- group of 1-alkanols [18].
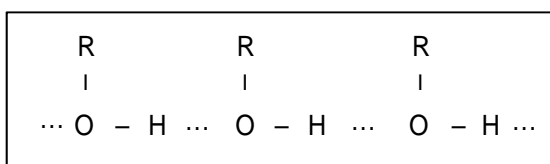
The 1–alkanols are associated through hydrogen bonding,

R   R   R

| | |

···O – H ··· O – H ··· O – H ···

Fig 2: structure of Alkanol

Moreover, N-N dimethylacetamide (DMA) is an eminently suitable solvent for salts or compounds with a high molecular weight owing to the combined action of its small molecule, and its ability to form complexes.   The interaction between the diethyl ketone (DEK) with polar group of amides, such as DMA play a crucial role in the structural effects. Since, ketones are also polar and so the dipole-dipole interactions are strong, naturally they form weaker hydrogen bonds from the alkanols. Since ketones lack hydroxyl groups, there are incapable to create intermolecular hydrogen bonds, but due to the presence of oxygen, they come form hydrogen bonds with acetones which lead to the complete solubility in the solvent. The carbonyl group of ketone is polar since oxygen is more electro negative than carbon and forms a positively charged dipole.   DMA with acetone as mixed solvents are interesting liquid systems for studying molecular interactions, since amides and acetones are the most common solvents used in chemical reactions and in many industrial processes. DMA can interact with an alkanols by virtue of better hydrogen bond acceptor ability of it oxygen atom resulting in structural and packing effects. Due to high polar nature of DMA, which may be induced polarity in DEK and 1-alkanols and so there may be induced dipole-dipole interactions. When the compounds are mixed, the changes that occur in association equilibria are evidently rupture of hydrogen bonds in pure compounds and the formation of O-H…C=O hydrogen bonds between the components.

As discussed already, alkanols are associated through hydrogen bonding, DEK and 1-alkanol interactions are due to hydrogen bonding between the oxygen of ketone (diethyl ketone) and proton of hydroxyl group of alkanols. Further the addition of alkanols with the binary solvent mixture (DMA + DEK) causes dissociation of hydrogen bonded structure of the same which lead to weakening of molecular interactions between Ketone (DEK) and alkanols The subsequent formation of new hydrogen bonding between proton acceptor of oxygen atom of C=O group of DMA and proton of –OH group of 1-alkanols. Further, one can notice from the Tables 3&4 that a decrease in free volume and an increase in internal pressure with increase in concentrations of 1-alkanols is observed in all the four liquid systems studied. As discussed earlier, the dissociation of hydrogen bonded structure of 1-alkanols due to the mixing of DMA with it and subsequent formation of a new bond, such a dissociation effect may lead to a compression in volume resulting in decrease of free volume in the present study.   Also, the corresponding increase in internal pressure generally indicates association through hydrogen bonding [19] and hence supports the present investigation. Similar results were observed by earlier workers in which an increase in internal pressure generally indicates association through hydrogen bonding. Further, the increase in temperature however, makes the weakening of molecular association due to the thermal expansion of liquids.

From Tables 5&6, it is observed that the relaxation time (τ) increases in all the four liquid systems and decrease with rise of temperature. The relaxation time which is the order of 10^{-12} seconds is due to structural relaxation process [20] and such situation suggests that the molecules get rearranged due to cooperation process[21]. The Gibbs free enegy (ΔG^*) from Table 5&6 increases with increase of molar concentration of alkanols and however almost exhibiting decreasing trend over the elevation of temperature. The increasing positive values of Gibbs free energy suggest that the closer approach of unlike molecules is due to hydrogen bonding [22]. The decreasing trend of Gibb's free energy over the elevation of temperature clearly establishes the weakening of molecular association in the solution.

4. Conclusion

Hence, our present investigation can be briefly summarized as

1. The interactions between DMA and 1-alkanols is through hydrogen bonding and with DEK through induced dipole-dipole interactions.
2. DMA interact with alkanols by virtue of hydrogen bond acceptor ability of its oxygen atom, resulting in structural and packing effects.
3. The further addition of alkanols with the binary solvent mixture causes the dissociation of structure of 1-alkanols leading to weakening of molecular interaction (rupturing of hydrogen bonds) between ketone (DEK) and 1-alkanols.
4. Interestingly, the elevation of temperature may also be attributed for rupturing of hydrogen bonds resulting in weakening of molecular association in the liquid mixtures.
5. Hence, it is very obvious that existence of weak molecular interactions prevail in the present systems of liquid mixtures.
6. Hence, Ultrasonic study is a powerful probe for charactering the physic-chemical properties and existence of molecular interactions in the liquid mixtures.

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