

Dielectric Relaxation and Dipole Moment Study of BinaryMixture of 2, 3-Dichloroaniline and 2-Propoxyethanol 1,4-Dioxane Solution Using Microwave Absorption Data

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Abstract

Dielectric constant (ϵ') and dielectric loss (ϵ'') of 2,3-Diachloroaniline (2,3-DCA), 2-propxyethanol (2-PE) and binary mixtures of 2,3-DCA+2-PE in 1,4-dioxane solutions were calculated by using standard standing microwave X-band technique were measured at microwave frequency 10.985 GHz at different temperatures 20^oC, 30^oC, 40^oC and 50^oC using Gopala Krishna's single frequency concentration variation method. The measured values of ϵ' and ϵ'' have been used to evaluate dipole moment (μ) and relaxation time (τ). The dielectric relaxation process of binary mixtures containing 75% mole fraction of 2,3-DCA were calculated at the respective given temperatures. On the basis of observations, it is found that the dielectric relaxation process can be treated as the rate process like the viscous flow process. Non-linear variation of relaxation time with molar concentration of 2,3-DCA in the whole concentration range of the binary mixture indicates the existence of solute-solute and solute–solvent type of molecular association were predicted. Dipole moment (μ) of 2,3-DCA and 2-PE non linearly decreases with rise in temperature. Excess inverse relaxation time (1/ τ)^e calculated at different temperatures are found to be positive and negative.

Keywords: Dielectric relaxation, Binary mixture, 2,3-dichloroaniline,2-Propoxyethanol,Dipole moment, excess inverse relaxation time.

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1. Introduction

The dielectric relaxation measurements in the frequency range of 10 MHz to 30 GHz have been carried out for 2-chloroaniline, 3-chloroaniline, 2-methoxyaniline and 3-methoxyaniline with 1,4-dioxane mixtures over the entire concentration at 25 °C using a picosecond time domain reflectometry technique [1]. The contribution of hydrogen bonds to dielectric properties of 2 and 3-chloroaniline and 2 and 3-methoxyaniline with dioxane mixtures has also been studied. Sharma and Gandhi [2] have analyzed certain binary mixtures of non-rigid molecules in benzene solution at 3 cm microwave and at different temperatures using the principles of Rana and her colleagues [3].

Mishra and his colleagues [4] has studied the dielectric relaxation time of ortho, meta, para tolualdehyde and cuminaldehyde at different temperatures in benzene. They have reported that the molecules relax by molecular as well as intermolecular rotations and the dipole relaxation is a co-operative process. They also have suggested that the solid rotator phase exists on solidification. Undre and Khirade [5] has studied dielectric dispersion in 1, 2-diaminopropane dimethylaminoethanol mixtures and determined the dielectric dispersion ε' and dielectric loss ε'' of binary mixture of 1,2-diaminopropane-dimethylaminoethanol were measured by employing the time domain reflectometry technique over a frequency range from 10 MHz to 20 GHz at 288, 298, 308 and 318 K temperatures. Study of dielectric behavior of binary mixture of diethylene triamine with 2-ethoxyethanol at 10.7 GHz frequency [6]. Becker [7] carried out a systematic study of dielectric relaxation of monohydric alcohol with dihydric alcohols (diols) solutions at 293K over the frequency range of 5 MHz to 72 GHz using the lumped circuit, coaxial and waveguide method.

We therefore make an extensive study further with the available data on the binary polar mixture of 2,3-DCA+2-PE dissolved in 1,4-dioxane in terms of measured dielectric permittivity, dielectric loss, relaxation time, dipole moment and excess inverse relaxation time. The purpose of the present study is also to see the applicability of Debye-Smyth model in the case of the binary polar mixture 2,3-DCA+2-PE in 1,4-dioxane.

The polar-non polar mixture of 2, 3-DCA in 1, 4-dioxene exhibits relaxation time due to rotation of the whole and the flexible parts of molecules in their effective dispersion region [8] of 10.985 GHz electric field. Thus it is worthwhile to study the temperature variation data of 2,3-DCA+2-PE in 1,4-dioxane under 10.985 GHz electric field to know the molecular dynamics of the system.

Dielectric study of 2,3-DCA+2-PE binary mixture have not been carried out in the past. As such it is felt that the present study provides most useful information regarding the molecular interaction in the binary mixture of 2,3-DCA+2-PE. 2,3-Dichloroaniline (2,3-DCA) is used starting material for synthesis of bioactive Schiff's bases, azetidinones, thiazolidinones, pyrazolines, acetohydrazides and in coupling reactions. It is used in preparation of poly (2,3-dichloroanline-Co-anline) which control conductivity in broad range from 10^{-9} to 10^{-2} S /cm. Also it is used in preparation in dyes, azo-dyes, isocyanides and in plant protection agent [9]. Studies have demonstrated that mono and dichloroaniline are capable of inducing acute renal failure in vivo and alternating organic ion accumulation by renal cortical slices in vitro [10]. Optimization of separation of mono and dichloroaniline in ion interaction high performance liquid chromatography [11]. 2-Propoxyethanol (2-PE) is used as solvents for

gums, resins, cellulose esters, inks, varnishes and hydraulic fluid. Its fast evaporation, high water solubility and active solvency also make it ideal for a variety of cleaning applications [12-14].

2. Materials

2,3-Diochloroaniline (GC Grade) is obtained from Sigma-Aldrich. 2-Propoxyethanol Sigma Aldrich USA and 1,4-dioxane (AR Grade) are purchased from M/S Sd. Fine chemical, Mumbai, India. Without further purification the two liquids 2,3-DCA+2-PE according to their proportions by volume are mixed well and kept 6h in well stoppered bottles to ensure good thermal equilibrium. These liquids are used as solute and solvent.

3. Experimental Measurements

The X-band microwave bench is used to measure wavelength in the dielectric medium and voltage standing wave ratio (VSWR) using a short-circuiting plunger. The set up is tuned at microwave frequency 10.985 GHz. The experimental techniques of Heston and his colleagues [15] for microwave measurements are used. All the measurements are carried out at temperatures 20°C, 30°C, 40°C and 50°C by circulating ethyleneglycol+water around the dielectric cell and temperature is thermostically controlled with \pm 0.5°C using Nevitech pvt. Ltd. Mumbai India. The whole of the equipment is standardized with the help of standard materials like methanol and ethyleneglycol+water (40:60). Microwave power measured by PM-437 (Attest) power meter, Chennai, India using source of Reflex klystron 2 K 25 (USSR). The densities and viscosities of the pure components and their binary mixtures are measured by using DMA 35 portable vibrating density meter. Anton paar Autria (Europe) having accuracy of density 0.001 gm/cm³, repeatability 0.0005 gm/cm³ and resolution 0.0001 gm/cm³ [16] and viscosity by LVDL, V-pro II Brook field viscometer with an accuracy of \pm 1% (USA) [17]. Rectangular wave guide working TE₁₀ mode, 10 dB, Vidyut Yantra Udyog, India. To hold the liquid sample in the liquid cell, thin mica window whose VSWR and attenuation are neglected is introduced between the cell and rest of microwave bench. The X-band microwave bench is used to measure wavelengths in the dielectric and the voltage standing wave ratio (VSWR).

We determined the dielectric constant (ϵ '), dielectric loss (ϵ "), relaxation time (τ), dipole moment (μ) and excess inverse relaxation time ($1/\tau$)^e of dilute solutions of binary mixture of 2,3-DCA+2-PE in 1,4-dioxane solution, for different mole fraction containing 0,0.2430,0.4906,0.7429,1 of 2,3-DCA+2-PE in 1,4-dioxane solution. The microwave techniques have been used method suggested by Heston *et.al* [15].

All the measurements are carried out at temperatures 20^oC, 30^oC, 40^oC and 50^oC and the temperature is thermostatically controlled within $\pm 0.5^{\circ}$ C.

To calculate dielectric constant (ϵ '), dielectric loss (ϵ ") by the using equations 1 and 2

$$\mathcal{E}' = \left(\frac{\lambda_0}{\lambda_c}\right)^2 + \left(\frac{\lambda_0}{\lambda_d}\right)^2 \tag{1}$$

$$\varepsilon'' = \frac{2}{\pi} \left(\frac{\lambda_0}{\lambda_d} \right)^2 \cdot \frac{\lambda_s}{\lambda_d} \left(\frac{d\rho}{dn} \right)$$
(2)

Where λ_0 , λ_c , λ_g and λ_d are the free space wavelengths, the cut-off wavelength, the waveguide wavelength and the wavelength in the waveguide filled with solution in centimeter respectively. ρ is the inverse of voltage standing wave ratio (VSWR) and $d\rho/dn$ is the slope of ρ versus n, where n=1,2,3..... Such that $(n\lambda_d/2)$ represents the length of the dielectric filled waveguide. The ε' and ε'' values are estimated to be reproducible within $\pm 0.5\%$ and $\pm 1\%$ respectively. The relaxation times (τ) and the dipole moment (μ) have been calculated by using equation 5 and 6 by the single frequency concentration variational method of Gopala Krishna [18].

$$X = \frac{\varepsilon'^{2} + \varepsilon''^{2} - 2}{(\varepsilon' + 2)^{2} + \varepsilon''^{2}}$$
(3)

$$Y = \frac{3\varepsilon''}{(\varepsilon'+2)^2 + \varepsilon''^2}$$
(4)

$$\tau = \frac{\lambda_0}{2\pi c} \left(\frac{dY}{dX}\right) \tag{5}$$

$$\mu^{2} = \frac{9kTM}{4\pi Nd_{0}} \left[1 + \left(\frac{dY}{dX}\right)^{2} \right] \frac{dX}{dW}$$
(6)

Where c is the velocity of electromagnetic waves in vacuum, K is the Boltzmann's constant, N is the Avogadro number, M is the molecular weight of polar substance in gm/mol, W is the weight fraction, T is absolute temperature and d_0 is the density of solution in gm/cm³. The slope of the line drawn between x and y used for determine the value of relaxation time (τ) and the slope of line x and w used for calculating the dipole moment (μ).

The excess inverse relaxation time $(1/\tau)^e$ may be evaluated for binary system in non-polar solvent using the relation [19].

$$(1/\tau)^{E} = (1/\tau)_{m} - [X_{1}(1/\tau_{1}) + X_{2}(1/\tau_{2})]$$
⁽⁷⁾

The symbol m, 1 and 2 are related to the mixture, liquid 1 and liquid 2 respectively. Information regarding the dynamics of a binary system interaction from this excess property is as follows.

Where $(1/\tau)^{e}$ is the excess inverse relaxation time which represents the average broadening of dielectric spectra. The inverse relaxation time analogy is taken from spectral line broadening which is the inverse of the relaxation time from resonant spectroscopy [20]. The information regarding the dynamics of solute-solvent interaction from this excess property is as follows:

- (i) $(1/\tau)^e = 0$: There is no change in the dynamics of solute-solvent interaction.
- (ii) $(1/\tau)^e < 0$: The solute-solvent interaction produces a field such that the effective dipoles rotate slowly.
- (iii) $(1/\tau)^e > 0$: The solute-solvent interaction produces a field such that the effective dipoles rotate quickly, i.e. the filed co-operate in the ratio of the dipoles.

The experimental techniques for the measurements of dielectric constant (ϵ '), dielectric loss (ϵ "), relaxation time (τ), dipole moment (μ) and excess inverse relaxation time ($1/\tau$)^e. All measurements are carried out at temperatures 20^oC, 30^oC, 40^oC and 50^oC in a dielectric cell by circulating thermo stated ethyleneglycol+water. The dielectric constant (ϵ '), dielectric loss (ϵ ") for 2,3-DCA+2-PE and their binary mixture containing mole fraction (X) 0,0.2430,0.4906,0.7429,1 in 1,4-dioxane solution. The values of wavelength in dielectrics (λ_d) and (d ρ /dn) along with weight fraction of solute in 1,4-dioxane at different temperatures are listed in Tables1-4.

4. Results and Discussion

The values of ε' and ε'' for the binary mixture 2,3-DCA+2-PE in the 1,4-dioxane solution have been calculated using the short-circuited waveguide method of Heston [15]. This method is highly accurate for the measurement of ε' and ε'' of polar mixtures in dilute solutions of non-polar solvent at very low concentrations. The accuracy in measurements dielectric permittivity (ε') and dielectric loss (ε'') values is $\pm 1\%$ and $\pm 2\%$ respectively. From observations Table 1-4 the variation of ε' and ε'' with weight fraction of solute in 1,4-dioxane for all binary mixtures is found to be linear. This shows that there is no change in the nature of the rotating molecular entities in the 1, 4-dioxane solution. This ensures the applicability of the Debye theory [21] and that of Gopala Krishna's method in the studied concentration range of the binary mixtures in the 1,4-dioxane solutions. The variation of relaxation time (τ) versus mole fraction $X_{2,3-DCA}$ at different temperatures is shown in (fig 1).

From (fig 1). The relaxation time (τ) gradually increases with increase mole fraction of 2, 3-DCA in 2, 3-DCA+2-PE binary mixture and attained a maximum value at X_{2, 3-DCA}=0.75 of 2, 3-DCA in the binary mixture. Then further increase in the mole fraction of 2, 3-DCA in binary mixture, increases the relaxation time and reach towards the value of pure 2, 3-DCA in 1,4-dioxane solution. This behavior indicates solute-solute molecular association between type of 2, 3-DCA and 2-PE. In its whole concentration range, the relaxation time of 2, 3-DCA+2-PE binary mixture remains longer than that of pure 2, 3-DCA. Therefore, the solute-solute type molecular association between 2, 3-DCA and 2-PE is indicated in its entire concentration range. In view of above results, it is proposed that in the binary mixtures of 2, 3-DCA and 2-PE, 2, 3-DCA exists in the dimer structure resulting because of H-bonding and dimer structure of 2,3-DCA binary mixture. This type of molecular so as to give the maximum values of relaxation time at 75mol% 2,3-DCA binary mixture. This type of molecular

associations have been proposed in (fig 2). The value of dipole moment 2,3-DCA binary mixture with 100% mole fraction of 2,3-DCA in the binary mixture depends on the temperature. This indicates the presence of solute-solvent molecular association of pure 2,3-DCA in 1, 4-dioxane solution. The value of dipole moment of 2-PE binary mixture with 0.00 mole fraction of 2,3-DCA in binary mixture Table 1-4 is found to be slightly changed with temperature. This could be explained on the basis of the solvent effects [22]. The change in dipole moment with temperature may be due to the stretching of the bond moment and due to the change in the bond angle. The dipole moment value of 2,3-DCA slightly non-linearly increases with the rise in temperature in 1, 4-dioxane solution [23-24].



Figure (1):- Relaxation time (τ) versus mole fraction of 2, 3-DCA in 2,3-DCA+2-PE binary mixture in 1,4dioxane solution at different temperatures.

This predicts the solute-solvent type of molecular association for 2, 3-DCA in the 1, 4-dioxane solution. Solutesolute association can be interpreted because of the molecule association arising due to hydrogen bonding between 2-propoxyethanol and 2, 3-dichloroaniline. The hydrogen bonding δ^+ on hydrogen of hydroxyl group of 2-propoxyethanol that form hydrogen bonding with nitrogen of 2, 3-dichloroaniline as shown in (fig 2). It may be explained on the basis that the dielectric relaxation process involves the rotation of molecular entities.

Solute- solvent association can be interpreted because of the molecule association arising due fractional positive charge on hydrogen of 2-propoxyethanol and lone pair electron present on oxygen of 1,4-dioxane is shown in (fig 3).

The excess inverse relaxation time $(1/\tau)^{e}$ for the binary mixture of system 2,3-DCA+2-PE for different mole fraction of 2,3-DCA and different temperatures are calculated using equation 7 and reported in Table 1-4. From (fig 4). The excess inverse relaxation time $(1/\tau)^{e}$ is found to be positive at 20^oC temperatures through the entire range of concentration of mole fraction of 2, 3-DCA. This suggests that the solute-solute interaction provides a field such that the effective dipoles rotates faster i.e. the field facilitates rotation of dipoles, whereas at the temperatures 30^{0} , 40^{0} and 50^{0} C, the excess inverse relaxation time $(1/\tau)^{e}$ has negative values. The negative peak

is observed at around X=0.243 mole fraction of 2, 3-DCA concentration in 2, 3-DCA+2-PE binary liquid mixture. This suggests that the slower rotation of dipoles, this indicates the formation of linear structure probably dimmeric and these rotations slowly under the influence of an external varying field. Similar results have been already predicated by Pande [25].

$$\begin{array}{c}
\delta + \delta - \\
H - O - CH_2 - CH_2 - O - CH_2 - CH_2 - CH_3 \\
\ddot{N}H_2 \\
\hline Cl \\
\hline Cl
\end{array}$$

Figure (2):- Solute-solute molecular association between 2,3-DCA and 2-PE



Figure (3):- Solute-solvent molecular association of 2-PE in 1,4-dioxane solution



Figure (4):- Excess inverse relaxation time $(1/\tau)^e$ versus mole fraction(X) of 2,3-DCA in 2,3-DCA+2-PE binary mixture in 1,4-dioxane solution at different temperatures.

		٤′ ^с	ε″ ^d	τ	μ (D) ^f	g
MF ^a	$\mathbf{WF}^{\mathbf{b}}$	(±0.5%)	(±1%)	(P.Sec)	(Debye)	$(1/\tau)^{\rm e}$
	(W)					
	0.0577	1.967	0.090			
0	0.0984	2.184	0.110	2.53	1.49	0
	0.1407	2.221	0.134			
	0.1792	2.298	0.169			
	0.0588	1.970	0.061			
	0.1112	2.278	0.126	2.74	1.61	0.0114
0.2430	0.1580	2.280	0.140			
	0.2001	2.359	0.155			
	0.0651	1.940	0.058			
	0.1222	2.278	0.124	2.98	1.52	0.0252
0.4906	0.1728	2.298	0.147			
	0.2179	2.592	0.148			
	0.0709	2.131	0.117			
	0.1325	2.318	0.164			
0.7429	0.1864	2.338	0.176	3.40	1.14	0.0274
	0.2341	2.878	0.181			
	0.0769	2.245	0.124			
	0.1428	2.380	0.188	4.50	1.02	0
1	0.1999	2.420	0.193			
	0.2499	2.422	0.195			

Table 1: Dielectric constant (ϵ') dielectric loss (ϵ''), relaxation time (τ) ,dipole moment (μ) and excess inverse relaxation time ($1/\tau$)^e of different mole fraction of 2,3-DCA in (2,3-DCA+2-PE) binary mixture in 1,4-dioxane at 20^oC.

a Mole Fraction.

b Weight Fraction (W).

c Dielectric Permittivity (ε').

d Dielectric Loss (ϵ ").

e Relaxation Time (τ).

f Dipole Moment μ (D).

Table 2: Dielectric constant (ϵ') dielectric loss (ϵ''), relaxation time (τ) ,dipole moment (μ) and excess inverse relaxation time ($1/\tau$)^e of different mole fraction of 2,3-DCA in (2,3-DCA+2-PE) binary mixture in 1,4-dioxane at 30^{0} C.

		٤′ ^с	ε″ ^d	τ	μ (D) ^f	g
$\mathbf{MF}^{\mathbf{a}}$	$\mathbf{WF}^{\mathbf{b}}$	(±0.5%)	(±1%)	(P.Sec)	(Debye)	(1/τ) ^e
	(W)					
	0.0577	2.080	0.098			
	0.0984	2.259	0.119			
0	0.1407	2.380	0.166	2.60	1.61	0
	0.1792	2.467	0.180			
	0.0588	2.192	0.098			
	0.1112	2.368	0.148			
0.2430	0.1580	2.500	0.158	3.08	1.56	-0.0276
	0.2001	2.564	0.159			
	0.0651	2.148	0.078			
	0.1222	2.259	0.115			
0.4906	0.1728	2.467	0.156	3.18	1.88	-0.0050
	0.2179	2.710	0.202			
	0.0709	2.131	0.123			
	0.1325	2.221	0.161	3.87	1.39	-0.0276
0.7429	0.1864	2.401	0.181			
	0.2341	2.422	0.190			
	0.0769	2.148	0.106			
	0.1428	2.298	0.164			
1	0.1999	2.480	0.185	4.60	1.49	0
	0.2499	2.512	0.194			

a Mole Fraction.

b Weight Fraction (W).

c Dielectric Permittivity (ϵ ').

d Dielectric Loss (ϵ ").

e Relaxation Time ($\boldsymbol{\tau}).$

f Dipole Moment $\boldsymbol{\mu}$ (D).

		٤ ^{, c}	ε″ ^d	τ	μ (D) ^f	g
MF ^a	$\mathbf{WF}^{\mathbf{b}}$	$(\pm 0.5\%)$	$(\pm 1\%)$	(P.Sec)	(Debye)	$(1/\tau)^{e}$
	(W)					
	0.0277	2.120	0.087			
	0.0577	2.121	0.103			
	0.0984	2.299	0.130	1.80	1.51	0
0	0.1407	2.455	0.148			
	0.1792					
	0.0588	2.166	0.116			
	0.1112	2.359	0.164	2.25	1.55	-0.0301
0.2430	0.1580	2.512	0.192			
	0.2001	2.521	0.197			
	0.0651	2.080	0.095			
	0.1222	2.175	0.142			
0 4906	0.1728	2.422	0.188	2 48	1.67	-0.0113
0.4900	0.2179	2.489	0.193	2.40	1.07	
	0.0700	2.064	0.066			
	0.0709	2.166	0.094			
0.7420	0.1325	2.422	0.141	2.78	1.60	-0.0518
0.7429	0.1864	2.444	0.159			
	0.2341	0.1.55	0.120			
	0.0769	2.166	0.129			
	0.1428	2.278	0.178	3.70	1.15	0
1	0.1999	2.422	0.194			
	0.2499	2.369	0.197			

Table 3: Dielectric constant (ϵ') dielectric loss (ϵ''), relaxation time (τ) ,dipole moment (μ) and excess inverse relaxation time ($1/\tau$)^e of different mole fraction of 2,3-DCA in (2,3-DCA+2-PE) binary mixture in 1,4-dioxane at 40^oC.

a Mole Fraction.

b Weight Fraction (W).

c Dielectric Permittivity (ϵ ').

d Dielectric Loss (ε").

e Relaxation Time (τ).

f Dipole Moment $\boldsymbol{\mu}$ (D).

Table 4: Dielectric constant (ϵ') dielectric loss (ϵ''), relaxation time (τ) ,dipole moment (μ) and excess inverse
relaxation time $(1/\tau)^{e}$ of different mole fraction of 2,3-DCA in (2,3-DCA+2-PE) binary mixture in 1,4-dioxane
at 50° C.

		٤ ^{, c}	ε″ ^d	τ	μ (D) ^f	g
MF ^a	$\mathbf{WF}^{\mathbf{b}}$	(±0.5%)	(±1%)	(P.Sec)	(Debye)	$(1/\tau)^{e}$
	(W)					
0	0.0577 0.0984 0.1407 0.1792 0.0588 0.1112 0.1580 0.2001	1.990 2.173 2.307 2.478 2.110 2.330 2.435 2.449	0.072 0.093 0.102 0.138 0.112 0.144 0.163 0.180	1.25	1.86	0 -0.1562
0.4906	0.0651 0.1222 0.1728 0.2179	2.087 2.202 2.422 2.512	0.099 0.139 0.181 0.195	2.23	1.72	-0.1033
0.7429	0.0709).1325 0.1864 0.2341	2.080 2.175 2.422 2.489	0.095 0.142 0.188 0.193	2.48	1.66	-0.0210
1	0.0769 0.1428 0.1999 0.2499	2.131 2.318 2.338 2.870	0.117 0.164 0.176 0.181	3.40	1.18	0

a Mole Fraction.

b Weight Fraction (W).

c Dielectric Permittivity (ε').

d Dielectric Loss (ε").

e Relaxation Time (τ).

f Dipole Moment μ (D).

5. Conclusion:

The molecular association between 2,3-DCA+2-PE is maximum nearly at a 25:75 mol% ratio respectively and then increases at higher mol% of 2,3-DCA in binary mixtures of 2,3-DCA+2-PE exists in the dimer structure resulting because of H-bonding and dimer structure of 2,3-DCA interact with the 2-PE molecules so as to give the maximum values of relaxation time at 75 mol%, of 2,3-DCA binary mixture respectively. Relaxation time increases due to addition of $-CH_2$ group in the binary mixtures. This confirms that the relaxation time (τ) increases due to increase in carbon atoms in binary mixtures. The change in dipole moment with temperature may be due to the stretching of bond moment and due to the change in bond angle. The excess inverse relaxation time ($1/\tau$)^e are found to positive and negative.

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