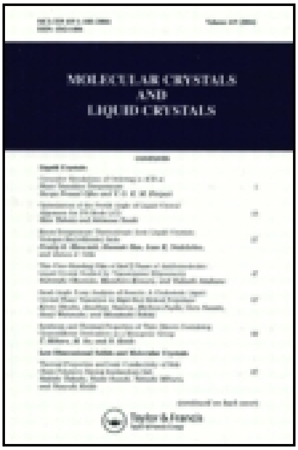

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Modification of High Tilted Antiferroelectric Mixture for Display Applications

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In search of the long pitch, orthoconic antiferroelectric liquid crystalline mixture (OAFLC), the high tilted host mixture with antiferroelectric smectic phase (AFLC) was formulated. The influence of the molecular dipole moment magnitude and orientation on physical properties of the AFLC was tested by doping of the host by three different dopants. The extensive study of the host material and obtained admixtures was done. Results are discussed in the context of the influence of the molecular dipole moment orientation and magnitude of the dopant on the tilt, helical pitch and spontaneous polarization of the SmC_A^* *phase of obtained mixtures.*

Keywords: helical pitch; high tilted smectic; molecular dipole moment; orthoconic antiferroelectric smectic liquid crystals

INTRODUCTION

Smectic Antiferroelectric Liquid Crystals (AFLC) offer less complicated solutions for high demanding applications than commonly known Ferroelectric Liquid Crystals (FLCs) [1–4] do. Among all known AFLCs those exhibiting extremely high molecular tilt, reaching

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45°, called Orthoconic AFLCs (OAFLCs) seems to be most promising for special display and photonic applications.

OAFLCs are unique materials which show a peculiar electrooptical behavior when the surface stabilized structure (SSOAFLCs) is used [3–6]. Less ordered structures provide interesting properties also (see Ref. [6–8]). SSOAFLCs is an optically uniaxial and optically negative medium, with the optical axis perpendicular to the substrate planes when the normally incident light beam is considered. Due to this property, the SSOAFLCs placed between crossed polarizers provide excellent dark state, hence an extremely high contrast, regardless of the structure defects. It helps to overcome a common problem of practical fabrication of optically uniform smectic structures [9].

Upon driving with electric field the SSOAFLCs exhibit threestable switching and provide inherent gray scale capability with hemispheric viewing angle as well as remarkable mechanical durability. Due to this SSOAFLCs are candidates for future passive driving displays with symmetric driving schemes and easy gray scale generation. In spite of all those attractive properties the mass scale fabrication with using of OAFLCs has not been started yet due to some problems of very basic physical nature and technological obstacles [1].

The main problem at practical fabrication of displays when the OAFLCs are used is the light leakage [10]. The reason of this parasitic effect is the presence of the remainder of the helical structure which is not completely unwound while the SSOALFC structure is formed within the cell where the cell gap is larger than the helical pitch. Besides of understanding and the control of the surface-stabilization mechanisms of OAFLCs the search for a long peach materials stands out as a major task. Due to this, the synthesis efforts are oriented to the obtaining new materials or mixtures characterized by orthoconic property and the helical pitch longer than the cell gap (which is usually around 1.5 μm).

It was proved, that the fluorine substitution is a very efficient tool for AFLC materials engineering [2,11–15], especially for induction of an anticlinic ordering and the high molecular tilt [2,15–19]. High tilted materials without pretransitional effects are possible to create within the structures of biphenyl benzoates. Systematical investigations of an influence of the molecular building units on the phase sequence, optical tilt angle, spontaneous polarizations, helical pitch, driving voltages and electrooptical response time were done [13,20–26].

As a continuation of these studies we formulated a new high tilted, near orthoconic mixture. It is an equimolar binary mixture W236 of two close structural analogues [27]. This high tilted, long pitch AFLC working mixture was taken as an reference material. The induction of an orthoconic property simultaneously with the long helical pitch was searched by doping with three different structural analogs. The molecular dipole moment value and its orientation on physical properties of near orthoconic mixture was tested by introduction of mentioned dopants into three tested admixtures. An extensive investigation of the material parameters of prepared admixtures were done. Results are presented and discussed in the context of the influence of molecular polarity of dopants on such parameters like molecular tilt, helical pitch, spontaneous polarization, rotational viscosity.

EXPERIMENT

Reference Mixture and Admixtures Formulation

The synthesis and basic properties of used compounds are presented elsewhere (for component used for the composition of the reference binary mixture W236 see Ref. [28–31], for dopant A see Ref. [28], for dopant B see Ref. [32] and Ref. [22,33–39] for dopant C which is well known compound MHPOBC). Recently synthesized compounds A and B exhibit the orthoconic property while the saturated value of the molecular tilt of compound C (MHPOBC) is 23°. The helical pitch of compounds A and B at the synclinic SmC* phase was a little below 1 μm, but it was too short to measure by spectroscopic method at the

TABLE 1 Phase Transition Temperatures T_D and Enthalpies ΔH Obtained for Compounds A, B, C and Mixtures W-236, W-236A, W-236B, W-236C by DSC. Smectic Phases were Recognized by Textures Observations Under Polarizing Microscope. For the C Dopant the Smectic Subphases are Omitted

Mixtures	SmC_A^*		SmC^*		SmA^*		Iso	
A	●	85.50	●			128.30	●	T_D [C deg]
		0.01				7.75		ΔH [J/g]
B	●	86.00	●			104.0	●	T_D [C deg]
		0.10				10.9		ΔH [J/g]
C (*MHPOBC*)	●	118.8	●	122.6	●	147.7	●	T_D [C deg]
W-236	●	95.16	●			103.12	●	T_D [C deg]
		0.10				7.25		ΔH [J/g]
W-236A	●	85.67	●			107.06	●	T_D [C deg]
		0.06				7.72		ΔH [J/g]
W-236B	●	88.77	●			103.67	●	T_D [C deg]
		0.06				7.43		ΔH [J/g]
W-236C	●	97.99	●	107.15	●	110.19	●	T_D [C deg]
		0.09		1.50		4.79		ΔH [J/g]

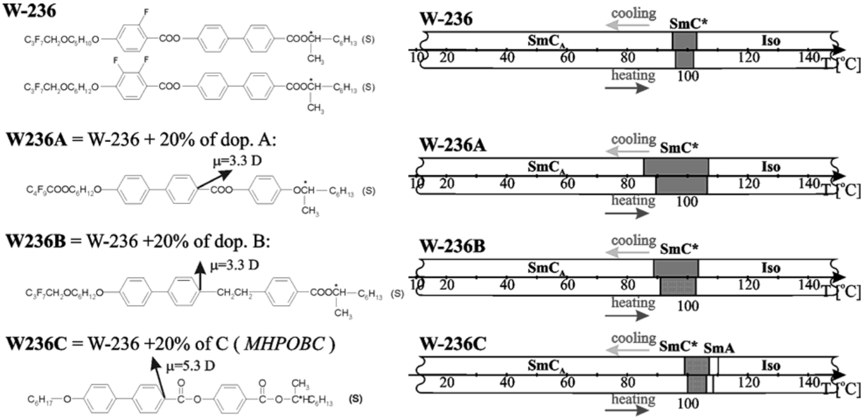


FIGURE 1 To the left, molecular structures of used compounds. Arrows denotes the overall molecular dipole moment orientation calculated by computer modeling (see also Tables 1 and 2). To the right, the graph of materials polymorphism evaluated upon DSC and polarizing microscopy combined studies.

anticlinic SmC_A^* phase. The helical pitch of the C compound at SmC_A^* phase was approximated after Ref. [38] as $\lambda = 600$ [nm].

The reference material, which is an equimolar bicomponent mixture W236, is room temperature, mixture as expected according to properties of used components (see Table 1). Three tested admixtures were obtained by doping of the reference mixture W236 with 20% (mole fraction) of dopants A, B, and C. The DSC study (SETARAM 141 instrument was used) combined with the microscopic observations (PZO, Biolar PI polarizing microscope with Lincam 660 hot stage, controlled by the TMS 93 unit) were done for investigation of the mesogenic behavior. Results are presented in the Figure 1 and in the Table 1. Obtained mixtures called W236A, W236B, and W236C exhibit similar polymorphism without significant change of phase transitions temperatures regarding to the reference one, however the W236C admixture exhibits the additional SmA* phase.

Molecular Modeling

The molecular modeling of molecules chosen for dopants was done using HyperChem 7.52 molecular modeling system. The aim of this task was to obtain the molecular dipole moment and the dipole moments components. Frames of coordinates were equally oriented for all considered molecules. The Ox axis was oriented along the molecular main axis of inertia, the Oy axis was in plane of the aromatic ring closest to the chiral center. The semi-empirical MNDO method [40] with Polak-Ribiere algorithm of molecular modeling was applied. Subsequently results obtained from the MNDO method were tested using force-field MM+ and Amber methods. For all studied molecules and for all mentioned modeling methods used a single, molecular conformation characterized by the deep minimum energy was obtained. Results are presented in the Table 2.

Cell Preparation and Electrooptical Performance

The tilt angle θ and the spontaneous polarizations P_s, were investigated by standard optical methods [41] using cells intentionally prepared in our laboratory. Cells gap was 1.6 μm. Cells were filled by capillary action with material under study in the isotropic phase. Both surfaces of cell were covered with ITO conducting layers and spincoated with the Nylon 6/6 polyamide (the solution of 0.3% by wt. in 2,2,2-Trifluoroethanol) and cured. Next the suitable antiparallel rubbing was applied on both cells surfaces. The uniform quasi-bookshelf structures of ALFCs were obtained during several slow melting-cooling cycles (0.05 K/min) in the presence of the electric field ($E = 12\,V/\mu m$, triangle pulse at 15 [Hz]). The routine microscope inspection of all cells proved high optical uniformity of the SSOAFLC structure with low number of visible defects (see Fig. 2) for the area where the electric field was applied.

The dynamic properties of the mixtures were investigated by applying of a pulse of triangle wave of the electric field at the frequency of

TABLE 2 Results of the Molecular Dipole Moment Calculations for Used Dopants

	Molecular dipole moment and its components [D]							
Dopant	μ_x	μ_y	μ_z	μ	$\mu_\perp$	α [deg]	β [deg]	γ [deg]
A	2.75	−1.84	−0.45	3.34	1.89	34.49	123.37	97.73
B	1.51	−3.01	−0.38	3.39	3.04	63.63	152.71	96.51
C	−1.47	−0.05	−5.11	5.31	5.11	106.05	90.56	163.94

where: μ – the magnitude of the molecular dipole moment.

μ_x, μ_y, μ_z – the *x, y, z* components of the molecular dipole moment.

$\mu_\perp$ – the component of the molecular dipole moment perpendicular to the molecular main axis of inertia.

α – the angle between the dipole moment and Ox molecular axis.

β – the angle between the dipole moment and short Oy molecular axis.

γ – the angle between the dipole moment and short Oz molecular axis.

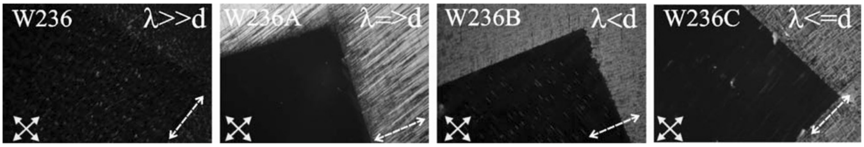


FIGURE 2 Textures of antiferroelectric phase for W-236, W-236A, W-236B, W-236C mixtures respectively at the room temperature at the zero electric field applied. Between ITO electrodes is a visible near black area in the middle of the cell. For the mixture W 236A in the area out of electrodes the synclinic state with in-plane orientation of the optical axis was induced. λ and d stand for the molecular pitch and the cell gap respectively. Crossed arrows indicate orientations of polarizers, the dashed line arrow indicate the antiparallel rubbing direction.

50 [mHz]. Switching loops of the light transmitted by the cell placed between crossed polarizers are presented in the Figure 3.

As to compare the electrooptical performance of the reference mixture and three admixtures a number of parameters defined in the Figure 4 were evaluated and results of those measurements are collected in the Table 3.

The Tilt Angle, Spontaneous Polarization, Rotational Viscosity and Helical Pitch Studies

The tilt angle was measured using cells placed in the Instec hot stage mounted on the rotating table of the polarizing microscope between crossed polarizers. Cells were driven using square shape driving pulse. The electrooptical effect was observed using PIN 20 linear photodiode (FLC Electronics, Sweden) connected to the digital oscilloscope HP 54601B. Two orientations of the optical axis induced by opposite polarizations of the driving electric field were recorded. The tilt was calculated as a half of the angle between recorded orientations of optical

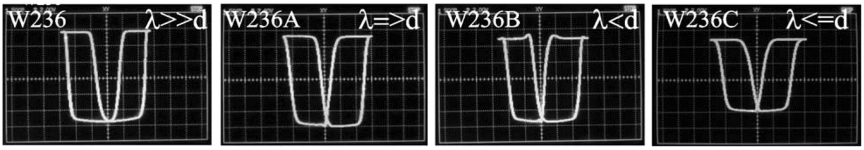


FIGURE 3 Optical hysteresis of the cells in the birefractive set-up upon application of symmetric triangular wave for host mixture W-236 and doped admixtures W-236A, W-236B, and W-236C respectively at the frequency of 0.1 [Hz]. λ denotes the molecular pitch, d stands for the cell gap.

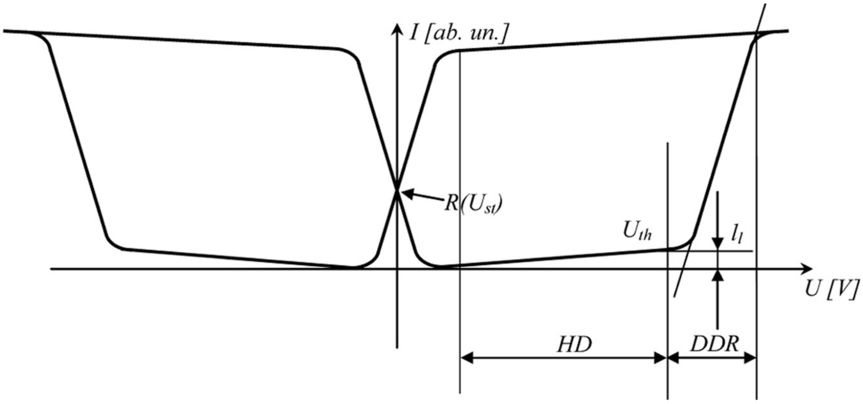


FIGURE 4 The schematic view of the switching loops of the threestable switching of the cell with W236 series mixtures.

axis. The results of the tilt angle measurement are presented in the Figure 5b.

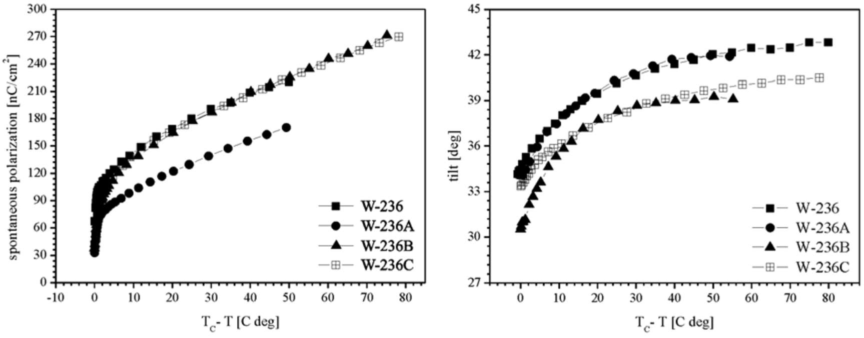
The spontaneous polarization P_s was studied using the same cells driven by triangle driving pulse at frequency of 50 [Hz]. The depolarization current was registered and integrated in the time domain as to obtain the value of the polarization charge. The spontaneous polarization P_s was calculated using the custom made software. Results of measurements of spontaneous polarization P_s are presented in the Figure 5a.

The rotational viscosity γ_φ was evaluated from the measurements of the switching time ON (τ_{10-90}) by square driving pulse at saturated voltage, using practical formula [42]:

$$\gamma_\varphi = \frac{1}{1.8} P_s E \tau_{10-90.} \tag{1}$$

TABLE 3 Parameters of Switching Loops Measured at the Switching Pulse Frequency f = 50 [mHz] Defined in the Figure 4

	Mixtures			
	W-236	W-236A	W-236B	W-236C
U [V]	14.0	14.5	12.5	12.0
U_{th} [V]	8.9	10.10	8.55	6.90
l_l [V]	0	4.38	7.50	4.37
DDR [V]	3.55	3.35	2.30	2.75
HD [V]	4.00	9.10	7.95	5.20
$R(U_{st})$ [%]	5.90	21.53	63.37	9.99



(a) (b)

FIGURE 5 (a) The spontaneous polarization P_S of the W-236, W-236A, W-236B, and W-236C mixtures obtained by triangle switching pulse method. (b) The tilt angle of W-236, W-236A, W-236B, and W-236C mixtures obtained by optical switching.

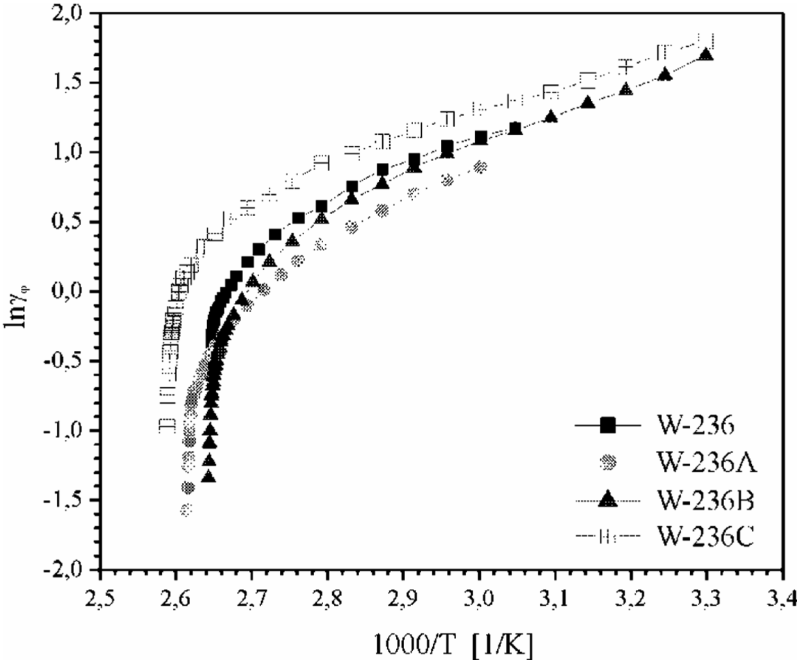


FIGURE 6 The Arhenius plot of ln γ vs. 1000/T [1/K] for studied mixtures.

TABLE 4 Results of Fitting of Parameters for Eq. (2)

	Mixtures			
	W-236	W-236A	W-236B	W-236C
ln A	−4.67	−8.47	−5.23	−4.36
Activation energy F [eV]	1.93	3.07	2.09	1.86

The Arhenius plot is presented in the Figure 6. The activation energy F was calculated using linear approximation method according to the formula (2):

$$-\ln\gamma = \ln A + \frac{F}{T_R} \tag{2}$$

where A – is a constant, T_R – is a reduced temperature, F – stands for an activation energy. Results are presented in Table 4.

The wavelength of selective reflection λ values was measured for the samples placed on perfectly cleaned bar glass. The homeotropic structure was obtained. The helical pitch λ was evaluated as a selective reflection wavelength λ_s divided by the materials refractive index n obtained from refractometric measurements. For the SmC_A^* phase this result was multiplied by 2. Results are presented in the Figure 6b.

For the further evaluation of the influence of the material doping on physical parameters of obtained phases, the numerical fitting of the characteristics of the spontaneous polarization, tilt angle and the rotational viscosity, according to power Eqs. (3) and (4) was done [43] (see also Table 5).

$$P_S = P_0(T_C - T)^{\beta} \tag{3}$$

$$\theta_S = \theta_0(T_C - T)^{\beta} \tag{4}$$

TABLE 5 The β Factor Calculated for Eqs. (3) and (4)

	Mixture			
	W236	W236A	W236B	W236C
$\beta \rightarrow P_s$	0.188	0.233	0.287	0.276
$\beta \rightarrow \theta_s$	0.049	0.051	0.055	0.042

DISCUSSION AND CONCLUSIONS

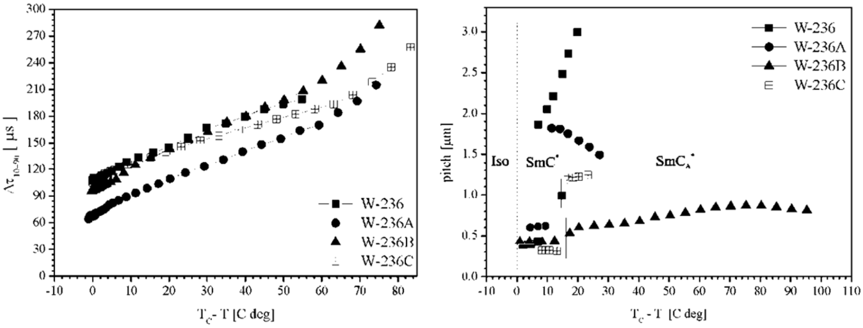
The investigation of the spontaneous polarization of the reference mixtures and doped admixtures reveal that the spontaneous polarization value of the reference mixture is not affected by the doping with B and C compounds, while the compound A causes a significant drop of P_s value. This suggests that the introduction of the compound with the tilted value of the molecular dipole moment as well as the carboxylic polar group at the fluorinated aliphatic chain affects the hindered rotation degree, which is a main factor of the spontaneous polarization induction within the smectic phase [44]. This effect does not influence on the molecular tilt angle. Upon doping the β factor of the exponential Eq. (3) increases noticeably.

The tilt investigation revealed that the orthoconic property was not induced upon doping with chosen dopants unfortunately as was expected on the basis of earlier studies [33]. An observation of the tilt angle behavior shows that the doping of the reference mixture with the protonated compound C and compound B with the different linking bridge within the molecular core induces a drop of the tilt angle. Both compounds posses molecular dipole moment near orthogonal to the molecular main axis of inertia while in the case of the reference mixture components and dopant A the molecular dipole moment is tilted at the reasonable angle.

As far as the rotational viscosity is concerned, the doping with the compound A is effective. At this case, the drop of the switching time is approximately 25% at room temperature. This effect observed together with the simultaneous drop of the spontaneous polarization support the hypothesis that in this case the molecular movement is more free than in case of pure reference mixture.

The doping of the reference mixture W236 with the dopant A induces a similar value of the pitch in the SmC_A^* phase at the temperature close to the phase transitions. The doping of W236 with compounds B and C also weakly affect the pitch value of the synclinic phase, but they induce a significant decrease of the helical pitch value in the SmC_A^* anticlinic phase (Fig. 7). This suggest the different influence of the dopant A and dopants B and C on the interlayer interactions due to their polar properties. The doping of the host mixture with dopants presented here seems to slightly affect the factor β from the exponential Eq. (4) (see Table 5).

The very intriguing observation concerns the helical pitch of W236 mixture, which becomes longer for the anticlinic SmC_A^* phase than in the SmC^* phase. Moreover, the pitch increases in this phase with temperature decreasing. The increase of the pitch upon SmC^* – SmC_A^*



(a) (b)

FIGURE 7 (a) The switching time ON – τ_{10-90} in ferro- and antiferroelectric phases vs. temperature for W236, W236A, W236B and W236C mixtures measured for the constant amplitude of the electric field 8 [V/μm], square pulse. (b) The pitch λ vs. reduced temperature T_c-T evaluated by using method of the selective reflection of light. Short vertical lines indicate the SmC* – SmC$_A^*$ phase transition.

phase transition is observed upon doping with compound A, however in opposite to the reference mixture the pitch drops with the temperature decrease, which is considered as a regular behavior. An irregular behavior of the molecular pitch vs. temperature in SmC$_A^*$ phase was previously observed and discussed [45,46].

As far as the case of the mixture W-236B is concerned, one can see a reasonable drop of the pitch in AFLC phase.

In conclusion one can say that the introduction of the dopant with alternating dipole moment strongly affects physical parameters of the SmC* as well as SmC$_A^*$ phases. One can distinguish an influence on the inter- and intralayer molecular interactions which govern physical parameters of the phase separately. In this study the influence of the fluorinated and protonated terminal chains as well as the molecular polarizability changes should be also considered. From this point of view the further investigations of those factors should be done in careful investigations.

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