exemplifies how an adequate choice of the metal in these polymers would help in tuning the electronic properties of the materials.

The above qualitative conclusion rests on the assumption that the band energies are not substantially affected by replacing the metal by another one. We have performed a calculation on a $(CoPc)_n$ compound of type **1a** to confirm this point.[11] The results of this calculation showed that the $\sigma$ band is higher in energy for this Co polymer as compared with the Cu one (−8.35 eV at the $\Gamma$ point), but the energies of the $\pi$ bands changed by less than 0.05 eV in all cases, thus confirming the above conclusion. Therefore we did not provide further details of the calculation with Co in place of Cu.

### Ladder Polymers

Systems **1** and **2** may be considered as conducting ladder polymers, in analogy to semiconducting ladder polymers of type **4**, which are reported[14] to have conductivities of up to 2 S $cm^{-1}$. We have performed band structure calculations on a model of **4** based on the structure of a monomeric analogue.[16] These calculations showed that, even though there is a half-filled band to support metallic properties, it is rather narrow (bandwidth = 0.33 eV). The corresponding orbitals are extended over the S–Co–S fragment and the phenyl rings, which is consistent with some degree of band conduction for **4**. We expect that **2**, with its broader and more delocalized bands, would support even higher conductivities.

$\pi$–Electron delocalization throughout the extended organic network is responsible for the expected conducting properties of polymers **1b** and **2**. This extended network makes the difference between the title compounds and the $\mu$-oxalato chains mentioned above (**3**): in the latter case the ligand molecules are isolated from each other by Cu atoms and the compound should not be expected to be a conductor.[12a]

### Concluding Remarks

The experimental conductivity data available until now on CuPc polymeric films[9] correspond to phases not very well characterized, making a direct comparison with our calculation infeasible, but indications are that high conductivities (5–8 S $cm^{-1}$) can be reached[9e] for some of these compounds. Smaller conductivity values have been reported for systems prepared under different conditions[9d], but these may be due to the presence of oligomeric or monomeric units[9f] rather than extended polymeric layers in the phases obtained or to the presence of mixtures or impurities. Our prediction is that if a pure phase with a layered structure of type **2** could be prepared, intrinsic metallic behavior should be expected.

One may envision a number of structures that are similar to **1** and **2a** and have intrinsically metallic properties. One such is **2b**, in which the connecting six-membered rings have been eliminated. We have performed EHT energy band calculations also for **2b** and found that there are partially filled broad $\pi$-bands around the Fermi level leading to an intrinsically metallic electronic structure.

Finally, we would like to comment on the problem of electron transport between individual columns of metal phthalocyanines in previously described stacked polymers. We consider that, contrary to the models presented here, the conduction in the equatorial direction (perpendicular to the stack) in those polymers is likely to be poor and would limit the overall conducting properties of the materials. In the case of ZnPcCl,[4] for example, semiconductivity is observed, which cannot be explained by band conduction. This is due to the very small interdimer resonance integral, which we estimate to be around 10 meV using extended Hückel theory (EHT).[10a,7]

Our calculations suggest that one-dimensional CuPc **1b**, but not **1a**, will be a conductor if it can be synthesized, and this illustrates the significance of choosing appropriately the contents of the unit cell in the synthesis of conducting polymers. The two-dimensional polymers **2a** and **2b** are also predicted to be conductors.

**Acknowledgment.** This work has been supported by the Camille and Henry Dreyfus Foundation. Further support by the NSF (Grant DMR-8702148) is gratefully acknowledged. We also thank Professor G. B. Jameson for his support and the Comite Conjunto Hispano-Norteamericano para la Cooperacion Cultural y Educativa for a fellowship awarded to P.G.-R. We thank an anonymous reviewer for suggesting the study of **2b**.

**Registry No.** Poly(copper phthalocyanine), 26893-93-6.

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Contribution from the Department of Chemistry,
State University of New York at Buffalo,
Buffalo, New York 14214

## Reaction of Protons and Molybdenum Dimers in an Ambient-Temperature Molten Salt

Richard T. Carlin and Robert A. Osteryoung*

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We have recently reported the electrochemical interconversions of several molybdenum dimers in the ambient-temperature molten salt $AlCl_3$–1-ethyl-3-methylimidazolium chloride (ImCl).[1] The results of the study performed in a basic melt are summarized in Scheme I.

Acidic melts are defined as melts where the $AlCl_3$:ImCl molar ratio is greater than one, and basic melts are melts where this ratio is less than one.[2] These previous studies were hampered in part by the presence of protonic impurities in the melt. We have since found that the addition of $EtAlCl_2$ to $AlCl_3$–ImCl melts effectively removes these protonic impurities, forming $AlCl_3$, a component of the melt.[3] We have applied this purification method to melts containing molybdenum dimer and wish to report several interesting reactions involving the addition and removal of hydrogen to the Mo–Mo quadruple bond in $Mo_2Cl_8^{4-}$.[4]

### Experimental Section

ImCl was synthesized as previously described.[5] Melts were prepared by gradual addition of sublimed $AlCl_3$ (Fluka) to ImCl with stirring. $Mo_2(OAc)_4$[6] and $ImHCl_2$[7] were prepared by using literature procedures.

The electrochemical cell consisted of a glass vessel. The working electrode was a Bioanalytical Systems Pt electrode consisting of a 0.08-cm-radius Pt disk sealed in Kel-F. The counter and reference electrodes consisted of Al wire immersed in 1.5:1.0 $AlCl_3$:ImCl melts contained in separate glass-fritted compartments. Cyclic voltammetry was performed on an EG&G PARC Model 173 potentiostat/galvanostat with a EG&G PARC Model 175 wave form generator and a Hewlett Packard Model 2000 XY recorder. Normal-pulse voltammetry was performed with an EG&G PARC Model 273 potentiostat/galvanostat, which was controlled by a computer system similar to that described elsewhere.[8] All electrochemical experiments were performed at 27 °C under a purified He atmosphere in a Vacuum Atmosphere drybox with an HE 493 Dri Train.

Visible spectra were obtained on a Tracor Northern 6500 spectrometer equipped with a photodiode array. All spectra were measured by using 1 mm quartz cells.

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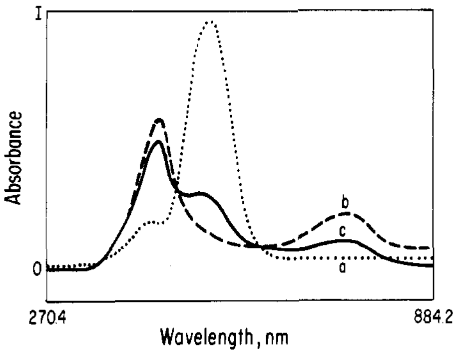
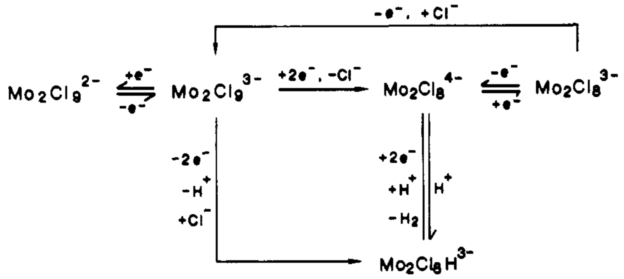


**Figure 1.** Visible absorption spectra for a basic $AlCl_3$–ImCl melt containing (a) 11 mM $Mo_2Cl_8^{4-}$, (b) 11 mM $Mo_2Cl_8H^{3-}$, or (c) a mixture of $Mo_2Cl_8^{4-}$ and $Mo_2Cl_8H^{3-}$ produced by addition of $EtAlCl_2$ to 11 mM $Mo_2Cl_8H^{3-}$.

**Scheme I**



## Results and Discussion

**Proton/Dimer Reactivity.** Addition of $Mo_2(OAc)_4$ to a melt with a $AlCl_3$:ImCl molar ratio of 0.8:1.0, $[Cl^-] = 1.0$ M, resulted in the initial formation of an orange solution, which, after approximately 1 h, took on a dark red color. The initial orange color is consistent with formation of mixed acetato–chloro molybdenum(II) dimers, such as $[Mo_2(OAc)_2Cl_4]^{2-}$, and the dark red color is indicative of the octachloro dimer, $Mo_2Cl_8^{4-}$.[4] We have previously shown that $Mo_2Cl_8^{4-}$ is slowly converted to the Mo(III) bridging-hydrido dimer $Mo_2Cl_8H^{3-}$ by reaction with protonic impurities in a basic melt.[1] To hasten this conversion, $ImHCl_2$, a proton (or HCl) source, was added to a concentration of 0.3 M.[7] After several hours, the red Mo(II) dimer, $Mo_2Cl_8^{4-}$, was completely converted to the yellow-green Mo(III) dimer, $Mo_2Cl_8H^{3-}$. The visible absorption spectra for the two melts are shown in parts a and b of Figure 1, respectively. This reaction is conventionally viewed as an oxidative addition to a quadruple bond.[9,10] The proton is formally considered a hydride ligand, and the two molybdenums are, therefore, considered to be formally oxidized from Mo(II) to Mo(III). The reaction may also be viewed as a Lewis acid–base reaction with the Mo–Mo quadruple bond in $Mo_2Cl_8^{4-}$ acting as the Lewis base. The only difference between the two views of the reaction is whether or not electrons in the quadruple bond have been formally transferred to the hydrogen atom. Viewing this reaction as a protonation is consistent with the fact that the reaction is facile in an acidic melt, i.e. a melt containing $AlCl_3$ in excess over ImCl.[1] It has been demonstrated that HCl in acidic $AlCl_3$–ImCl (excess $AlCl_3$) melts behaves as a strong acid comparable in strength to such superacids as liquid HF.[11]

Upon addition of excess $EtAlCl_2$ (0.8 M) to the basic melt containing 0.3 M $H^+$ (or HCl), a slow, but visible, reaction occurred accompanied by gas bubble formation. The visible spectrum of the resulting melt, which was 0.5 M in $EtAlCl_2$, 0.5 M in $Cl^-$, and 11 mM in Mo dimer, is shown in Figure 1c. Only a small quantity of $Mo_2Cl_8H^{3-}$ was converted back to $Mo_2Cl_8^{4-}$ despite the large excess of $EtAlCl_2$. Stirring the solution overnight showed no further reaction of $EtAlCl_2$ with $Mo_2Cl_8H^{3-}$. It is noteworthy that two isosbestic points are seen in Figure 1 at 475 and 607 nm. One possible explanation for this partial conversion is that when the $EtAlCl_2$ is added to the melt, it can react immediately with $Mo_2Cl_8H^{3-}$, as in eq 1, or it can react with $Cl^-$

$$Mo_2Cl_8H^{3-} + EtAlCl_2 + Cl^- \rightarrow Mo_2Cl_8^{4-} + EtH + AlCl_3 \qquad (1)$$

to form $EtAlCl_3^-$, which then does not react with the hydrido dimer. To confirm this hypothesis, $AlCl_3$ was added to the melt in sufficient quantity to produce an acidic melt. A brown precipitate formed, which is probably a form of $\beta$-$MoCl_2$ believed to consist of Mo(II) tetrameric analogues to cyclobutadiynes, which are themselves joined through chloride bridges forming metal cluster sheets.[12] Addition of ImCl, converting this acid melt back to a basic melt, resulted in dissolution of the brown precipitate and gave a dark red solution. The visible spectrum of this basic melt showed that $Mo_2Cl_8H^{3-}$ had been quantitatively converted back to $Mo_2Cl_8^{4-}$. It would appear that in the acidic melts where $EtAlCl_2$ may exist as $EtAl_2Cl_6^-$, the conversion of the hydrido Mo(III) dimer back to the Mo(II) dimer is facile. This may be explained by viewing the hydrido ligand bridging the Mo–Mo bond as a Lewis base and assuming the reaction proceeds by initial attack of $EtAl_2Cl_6^-$, a strong Lewis acid, at the hydrido ligand. In a basic melt, the weakly acidic $EtAlCl_3^-$ species is not capable of reacting with the hydrido ligand. In an acidic melt, however, the strongly acidic $EtAl_2Cl_6^-$ does react with the hydrido dimer as in eq 2. It should be noted that eq 1 and

$$Mo_2Cl_8H^{3-} + EtAl_2Cl_6^- \rightarrow Mo_2Cl_8^{4-} + EtH + Al_2Cl_6 \qquad (2)$$

2 differ only in their conception of how the Lewis acidic chloroethylaluminate species exist in the melt; both involve reaction of a strong Lewis acidic chloroethylaluminate species with the hydrido dimer.

These experiments demonstrate the ability to control the reactivity of protons in this unique ionic liquid. By using $ImHCl_2$ as a proton source and $EtAlCl_2$ as a reagent to remove protons, one can effectively perform hydrido/proton interconversion chemistry at a metal–metal bond.

**$Mo_2Cl_8^{4-}$ Voltammetry.** To demonstrate that excess $EtAlCl_2$ in a basic melt did not interfere with the electrochemistry of $Mo_2Cl_8^{4-}$ and that such an excess would continue to remove protons with equal efficacy in aged melts, a 0.8:1.0 $AlCl_3$:ImCl melt containing 0.4 M $EtAlCl_2$ was prepared. To this proton-free, $EtAlCl_2$-rich melt was added 16 mM $Mo_2(OAc)_4$. After the melt was stirred for several hours, the visible spectrum of the resulting melt showed only $Mo_2Cl_8^{4-}$ to be present. Even after exposure to the drybox atmosphere (which contains low levels of $H_2O$) for several days, no hydrido dimer was produced. Using $EtAlCl_2$ is beneficial since previously it was necessary to electrochemically generate $Mo_2Cl_8^{4-}$ in a 0.8:1.0 $AlCl_3$:ImCl melt after first electrochemically reducing the concentration of protonic impurities.[1]

Cyclic voltammetry using a Pt electrode was performed on this proton-free 16 mM $Mo_2Cl_8^{4-}$ melt. As seen in Scheme I, $Mo_2Cl_8^{4-}$ undergoes two one-electron oxidations. Previously, the $Mo_2Cl_8^{4-}$ oxidations were examined at a glassy-carbon working electrode where the first one-electron oxidation of $Mo_2Cl_8^{4-}$ to $Mo_2Cl_8^{3-}$ was determined to be quasi-reversible, and the second one-electron oxidation was believed to involve at least one chemical step. At the Pt electrode employed here, the peak potential separation, $\Delta E_p$, for the first oxidation at $E_0 = -0.16$ V is only 65 mV at 20 mV/s, indicating it to be essentially reversible at this scan rate, $\nu$. At 500 mV/s, $\Delta E_p$ increases to 80 mV/s; however, this increase is probably due to uncompensated resistance. The ratio of the cathodic peak current, $I_p^c$ to the anodic peak current, $I_p^a$ is approximately one. It is apparent from the above analyses that this first one-electron oxidation is well-behaved at a Pt electrode even

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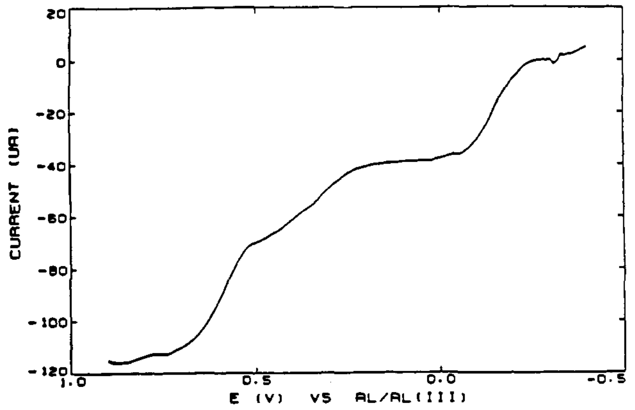

**Figure 2.** Normal-pulse voltammogram at Pt for oxidation of 16 mM $Mo_2Cl_8^{4-}$ ($t_p$ = 50 ms). Melt is basic $AlCl_3$–ImCl containing 0.4 M $EtAlCl_2$. Voltage is scanned from the open circuit potential in anodic direction.

in the presence of a large excess of $EtAlCl_2$.

The second one-electron-oxidation wave at the Pt electrode is relatively broad and shifts from 0.3 to 0.4 V as $\nu$ is increased from 10 to 500 mV/s. Also, at the higher scan rates, the ratio $I_p^a/\nu^{1/2}$ decreases. These observations are indicative of an electrochemical process coupled with a homogeneous chemical reaction.[13] This is expected since the final product of the second oxidation has been shown to be $Mo_2Cl_9^{3-}$, the formation of which requires a degree of structural rearrangement of $Mo_2Cl_8^{4-}$.[1]

The $Mo_2Cl_8^{4-}$ oxidations were also examined by using normal-pulse voltammetry at the Pt electrode. Pulse widths, $t_p$, were varied from 50 to 1000 ms. After each analysis pulse, the melt was stirred for 2 s and then left unstirred for 5 s before applying the next more anodic pulse. A normal-pulse voltammogram is shown in Figure 2. Three distinct waves are apparent corresponding to first, a one-electron oxidation of $Mo_2Cl_8^{4-}$ to $Mo_2Cl_8^{3-}$, second, a one-electron oxidation of $Mo_2Cl_8^{3-}$ to $Mo_2Cl_9^{3-}$, and finally, a one-electron oxidation of $Mo_2Cl_9^{3-}$ to $Mo_2Cl_9^{2-}$. The position and shapes of the first and third oxidation waves remained essentially invariant with changing pulse width as expected for reversible processes.[14] The limiting current plateau, $i_{lim}$, for the second oxidation, however, was not easily discernible especially at the shorter pulse widths. Despite the less than ideal second wave, the ratios of limiting currents for the three oxidations were approximately 1:1:1 as expected for electrochemical processes under diffusion control. A plot of $i_{lim}$ vs. $t_p^{1/2}$ for the first oxidation wave is linear and passes through the origin. From the slope of this plot, a $D$ value of $(2.61 \pm 0.03) \times 10^{-7}$ cm²/s for $Mo_2Cl_8^{4-}$ was calculated.[15] This value is consistent with previously determined dimer diffusion coefficients.[1]

## Conclusions

In the acidic $AlCl_3$–ImCl molten salt, the molybdenum dimers, $Mo_2Cl_8^{4-}$ and $Mo_2Cl_8H^{3-}$, are easily interconverted by using $ImHCl_2$ as a proton source and $EtAlCl_2$ as a proton-removing reagent. The presence of excess $EtAlCl_2$ in the melt does not alter the electrochemistry of $Mo_2Cl_8^{4-}$. These two reagents should prove useful in examining the interaction of hydrogen with other metal clusters in these unique solvent systems.

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**Registry No.** $Mo_2(OAc)_4$, 14221-06-8; $AlCl_3$, 7446-70-0; ImCl, 65039-09-0; $Mo_2Cl_8^{4-}$, 34767-26-5; $ImHCl_2$, 116210-73-2; $Mo_2Cl_8H^{3-}$, 66496-30-8; $EtAlCl_2$, 563-43-9; $Mo_2Cl_8^{3-}$, 64477-04-9; $Mo_2Cl_9^{3-}$, 52409-23-1; $Mo_2Cl_9^{2-}$, 51059-87-1.

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Contribution from the Centre d'Etudes Nucleaires
de Grenoble, DRF-G/Service de Physique/Groupe SCPM,
85X 38041 Grenoble Cedex, France

# A Model for the Spin States of High-Potential $[Fe_4S_4]^{3+}$ Proteins

Louis Noodleman*

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The complex and highly variable spin-state structures of $[Fe_3S_4]$ and $[Fe_4S_4]$ proteins and synthetic analogues are now well appreciated[1-4] but not well understood. Here, we propose and solve a simple Hamiltonian for the spin states of oxidized high-potential $[Fe_4S_4]^{3+}$ (HP) clusters. The relative energies of the low-lying spin states are analyzed including conditions for state crossings. We then use the vector model originally developed by Middleton[5] for reduced $[Fe_4S_4]^+$ and utilized by him also for $[Fe_4S_4]^{3+}$ to approximately evaluate effective isotropic $g$ and $A$ values. Important early experimental work on this system is due to Antanaitas and Moss.[5c] Middleton's vector model[5b] for $[Fe_4S_4]^{3+}$ is essentially correct, but one must also show how to generate the correct spin-state energies from the basic physics of the problem. Recent developments[4,6] in the spin coupling theory of mixed-valence antiferromagnetic (AF) complexes combined with quantitative calculations[7-9] make further progress possible.

To obtain the proper spin-state ordering for $[Fe_4S_4]^{3+}$ clusters requires at least two Heisenberg AF coupling parameters $J$ and also one resonance delocalization parameter $B$ for the mixed-valence pair $Fe^{3+}$–$Fe^{2+}$. In HP complexes, Mossbauer spectroscopy shows that four Fe sites occur in two internally equivalent pairs[1,5] designated $\beta$ for the $Fe^{3+}$–$Fe^{3+}$ and $\alpha$ for the $Fe^{3+}$–$Fe^{2+}$ mixed valence (equivalently $Fe^{2.5+}$). Quantitative X$\alpha$–VB calculations[7-9] on $[Fe_4S_4]^{3+,2+}$ models in $C_{2v}$ symmetry show that the electron undergoing delocalization resides in an orbital largely confined (≥85%)[7b] to one $Fe_2S_2$ layer with bonding along the Fe–Fe axis. It appears, therefore, that one $B$ parameter confined to the mixed-valence pair is sufficient to describe resonance or double exchange in HP. The Heisenberg AF coupling constant should be largest in the $Fe^{3+}$–$Fe^{3+}$ pair and decrease in the order $Fe^{3+}$–$Fe^{3+}$ > $Fe^{3+}$–$Fe^{2.5+}$ > $Fe^{3+}$–$Fe^{2+}$; the second term represents an interlayer pair, and the third, the mixed-valence pair. (In the notation above, $Fe^{3+}$–$Fe^{2.5+}$ is used, since the relevant Heisenberg coupling links a ferric center with a mixed-valence center.) This order is verfied by our X$\alpha$–VB calculations on $[Fe_3S_4]^{+,0}$ clusters[8,9] and has been assumed in fitting $[Fe_4S_4]$ susceptibility data by others.[10]

The Mossbauer hyperfine structure of $[Fe_4S_4]^{3+}$ gives the surprising result that the effective $A$ values of the $Fe^{3+}$–$Fe^{3+}$ pair are positive,[1,9] so that the ferric pair spins are aligned oppositely to the net system spin $S = 1/2$ in a magnetic field. (All $A$ values are with reference to the ground-state magnetic moment of the $^{57}$Fe nucleus.) Münck and Papaefthymiou[11] have proposed that $S_{12}' = 4$ is then required for the ferric pair spin. But how and why does this occur?

Consider a system spin Hamiltonian:

$$H = J(\vec{S}_1\cdot\vec{S}_2 + \vec{S}_1\cdot\vec{S}_3 + \vec{S}_1\cdot\vec{S}_4 + \vec{S}_2\cdot\vec{S}_3 + \vec{S}_2\cdot\vec{S}_4 + \vec{S}_3\cdot\vec{S}_4) \pm B(S_{34}' + 1/2) + \Delta J_{12}(\vec{S}_1\cdot\vec{S}_2) + \Delta J_{34}(\vec{S}_3\cdot\vec{S}_4) \quad (1)$$

$S_1$ and $S_2$ are site spins for $Fe^{3+}$–$Fe^{3+}$, and $S_3$ and $S_4$, site spins for $Fe^{3+}$–$Fe^{2+}$, so $S_1 = S_2 = 5/2$, $S_3 = 5/2$, and $S_4 = 2$. (The last two spin values can be interchanged without affecting the argument.) The interlayer coupling constant linking one site of pair $\alpha$ with one site of pair $\beta$ is $J$ describing four equal interlayer site couplings, while the ferric ($\beta$) pair and mixed-valence ($\alpha$) pair couplings are $J_{12} = J + \Delta J_{12}$ and $J_{34} = J + \Delta J_{34}$, respectively. In a more general context, the $B$ resonance term can be derived

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*Address correspondence to Department of Molecular Biology MB1, Research Institute of Scripps Clinic, La Jolla, CA 92037.

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