CHEMISTRY
AN ASIAN JOURNAL

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## Accepted Article

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**To be cited as:** *Chem. Asian J.* 10.1002/asia.201900956

**Link to VoR:** http://dx.doi.org/10.1002/asia.201900956

**A Journal of**

ACES
Asian Chemical
Editorial Society

A sister journal of **Angewandte Chemie** and **Chemistry – A European Journal**

WILEY-VCH

FULL PAPER WILEY-VCH

# Modulation of electronic mobility of a one-dimensional coordination polymeric molecular wire with light

Monochura Saha, Sheelbhadra Chatterjee, Munshi Sahid Hossain, Arijeet Ghude and Subhajit Bandyopadhyay*[a]

*Dedicated to Professor Vinod K. Singh on the occasion of his 60th birthday*

**Abstract:** Metal ions often influence the photoswitching efficiency of a photochromic system. This article reports a one-dimensional polymer having cyclic azobenzenes coordinated to silver ions that are bridged by nitrates. The coordination polymer (CP-**2**) displays a photoresponsive behavior. The switching ability in the polymer form was faster compared to the parent azobenzene ligand without the metal ions. Azobenzenes are reported to be poorly conducting. Here, although the azobenzene ligand does not show significant electronic mobility, the coordination polymer (CP-**2**) displays a modest conductivity. The conductance in the *cis* form of the polymer is significantly higher compared to the *trans* form. Upon exposure to visible light, the *cis* form undergoes photoisomerization to the *trans* form with drastic drop in the electronic mobility. The *trans* form can be reverted to the *cis* form thermally or by using UV light. Thus this system offers a reversible control of the conductivity using light.

## Introduction

Smart or stimuli-responsive materials have been a focus of research over the last few decades.[1]-[12] These materials respond to small changes of stimuli (pH, magnetic fields, temperature, light, etc.) with a change in their physical or chemical properties, often reversibly.[13]-[22] Photochromic materials, in particular, are of considerable interest as the regulation of their activity using light allows both spatial and temporal control without any physical contact. Taking advantage of these unique properties, researchers are actively looking at the applications of photochromic materials in consumer devices, biomedical devices, electronics, and energy storage. [23]-[28]

The integration of photochromic units in polymeric systems renders tunability of the polymers using light. Coordination polymers consist of metal cations linked by organic ligands, forming extensive repeating networks in one, two, or three-dimensions. Coordination polymers have been of recent interest due to their rich structural chemistry arising from the fact that their physical and chemical properties are highly dependent on the choice of the coordinating ligands. [29]-[34] In some varieties of coordination polymers, inorganic bridges and conjugated organic bridges allow the conduction of electrons along the network, through the interaction of the metal *d*-electrons with the ligand π-electronic framework. Conducting polymers hold a significant advantage over conventional metal- or metalloid-based conductors as they provide better control during the synthesis, allowing fine-tuning of the activities for their specific purpose. Commercial applications of conducting polymers include organic LEDs, already available to consumers in screens in modern display devices, among others. Introducing the ability to switch the conductance of the system after using light is a fascinating prospect for external control over molecular electronics. [35]-[41]

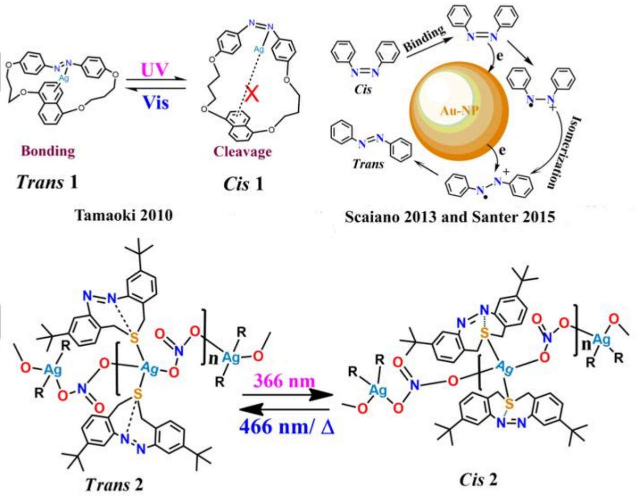

**Figure 1**: Schematic representation of previously reported work (top) and our work (bottom).

Azobenzene is a well studied reversible photochromic system – where the switching from the thermodynamically favored *trans* isomer to the *cis* isomer is carried out upon exposure to UV-light. The reverse reaction occurs efficiently with visible light or heat even at room temperature. This transformation is highly dependent on the structure of the azobenzene system and the local environments such as the solvents or the matrix, and thus the tuning of this process is an interesting problem to tackle. Recent reports have suggested that gold nanoparticles can enhance the isomerization process in acyclic azobenzenes through gold-mediated electron transfer, increasing the rate of the photoswitching (Figure 1).[42-44] Azobenzene can also be functionalized and used as a ligand for coordination polymers (CP), including conducting the CPs.[45] Recent reports have

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Supporting information for this article is given via a link at the end of the document. CCDC 1918660

10.1002/asia.201900956

shown that silver-based CPs exhibit electrical conductance, which begets the question of whether it is possible for such a polymer to exhibit differential electronic mobility upon switching with light (Figure 1). In this work we report the synthesis of a one-dimensional (1D) CP based on a cyclic-azobenzene coordinated to a $Ag^+$-ion. It was observed that the switching of the azobenzene system accelerated upon the coordination to the $Ag^+$-ion for which the single crystal x-ray diffraction revealed the formation of a CP system. The 1D coordination polymers, interestingly, exhibits a variable conductance upon exposure to light. This is ascribed to the change in the geometry of the ligand and thus the electronic environment around the metal ion. This change is photonically reversible. The metal ion accelerates photoswitching as well, allowing faster changes in the conductance of the polymer.

## Results and Discussion

The nine-membered cyclic azobenzene **1** was synthesized by the reduction of the *bis*(nitrobenzyl) sulfane precursor **3** using glucose/NaOH as the reducing partner in the ethanol and water mixture.[46]-[47] The synthesis of the precursor **3** was accomplished by the nucleophilic substitution of the corresponding bromo-benzyl compound **4** with sodium sulfide in methanol at room temperature (Scheme S1). The reaction mixture was purified by preparative TLC to obtain **1** in 20 % yield, and characterized by several spectroscopic tools including $^1$H, $^{13}$C NMR, IR spectroscopy and ESI mass spectrometry (Figure S1-S5). On crystallization in acetonitrile at room temperature, the red plate shape crystals of compound **1** were obtained which is belongs to the monoclinic $P2_1/c$ symmetric space group (Figure S6).

The photoisomerization behavior of compound **1** was studied primarily through UV/Vis spectroscopy. On exposure of the sample (acetonitrile, 30µM) to UV light (λ=366 nm, irradiation power of 2.3 mW cm$^{-2}$), the thermodynamically more stable *trans* form gradually underwent isomerization into the *cis* form and a photostationary state (PSS) consisting of a 70:30 ratio of *cis*:*trans* of the isomers (Figure 4A-4C, S7A-S7B), was obtained from the UV-vis spectroscopy. This was also confirmed by the $^1$H NMR spectroscopy. During the photoisomerization process, the UV-absorption bands at $\lambda_{max}$ = 318 nm (ε = 13800 M$^{-1}$.cm$^{-1}$) and at $\lambda_{max}$ = 444 nm (ε = 286 M$^{-1}$·cm$^{-1}$), corresponding to the π–π* and the symmetry forbidden n–π* transition respectively, were observed to diminish in intensity and were accompanied by the simultaneous formation and growth of a new peak at $\lambda_{max}$ = 406 nm (ε =370 M$^{-1}$.cm$^{-1}$). Exposure of the sample to blue LED light (λ = 466 nm, irradiation power = 0.5 mW cm$^{-2}$) led to the reversal of the PSS.

The $^1$H NMR studies of the photoisomerization behavior of **1** were also performed in $CD_3CN$ at 298K (Figure S8). The peaks at δ 7.83, 7.34 and 7.20 corresponding to the aromatic protons of the azobenzene unit, gradually decreased in the presence of 366 nm UV light, and were accompanied by upfield shifts with the appearance of three new peaks at δ 7.08, 7.02, and 6.60, corresponding to the same set of aromatic protons. This behavior is consistent with the expected behavior for *trans* to *cis* conversion of the azobenzene systems.[48]-[49]

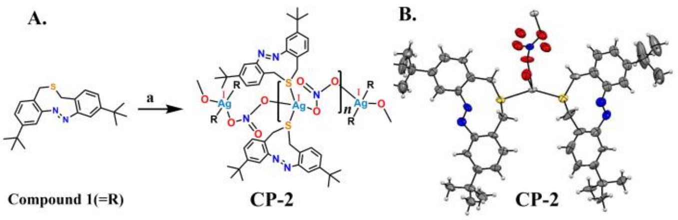

**Figure 2**: (A). Synthesis of CP-**2** Conditions: (a). $AgNO_3$ in MeOH, 48h at r.t in dark conditions, -18 °C, 5d ; (B) Single crystal structure of one unit of the coordination polymer 2 with a 50% ellipsoid probability. The structures with the repeated units are presented in Figures 5A and S17.

It was reported that the eight-membered diazocine systems are thermodynamically stable in the *cis* conformation. [50]-[51] We have demonstrated that the effect of ring strain responsible for the stability of *cis* form does not extend to the 9-membered ring system, as our system was found to be more stable in the *trans* form.[52]

The effect of metal ions on the photoisomerization of azobenzene systems were studied in a Ag (I)-complex with a 24-membered cyclic azobenzene by Tamaoki (Figure 1A)[43] where the *trans*-complex was found to be more stable. Scaiano and Santer's groups have reported the enhanced photoswitching rate of acyclic azobenzene systems in presence of AuNPs (Figure 1B).[53]-[54] These observations prompted us to prepare and explore the properties of a coordination polymer with with the cyclic azobenzene **1** and $Ag^+$ ions. The [$Ag^+$-**1**] CP-**2** was prepared by the addition of silver nitrate ($AgNO_3$) salt dissolved in methanol to a solution of the **1** in acetonitrile, followed by continuous stirring of the mixture at room temperature for 48 hours in dark conditions(Figure 2A). Crystallization via slow evaporation in the freezer at -18 °C for 7 days yielded red, needle shaped crystals of **2**, which were isolated and characterized by UV-Vis (Figure 3A) and fluorescence spectroscopy (Figure 3B), FTIR (Figure S10), $^1$H NMR spectroscopy (Figure S9), mass spectrometry (Figure S11), and single crystal XRD (Figure 2B).

In comparison with **1**, the Ag-"complex", **2** displayed a blue shift in the UV-Vis spectroscopy for the peak corresponding to the π-π* transition as the absorption band at 318 nm shifted to 308nm on increasing the salt concentration from 0 to 2 equivalents gradually (Figure 3A). This shift was accompanied by a decrease in the intensity of both the 318nm and the 444nm bands. In fluorescence studies of **1** (30µM, acetonitrile, $\lambda_{ex}$ 320nm at 298K), the peaks at 366nm and 396nm were observed to increase in intensity on increasing metal salt concentration (Figure 3B). In the $^1$H NMR studies performed in $CD_3CN$, the addition of silver nitrate caused the peaks associated with the aliphatic and aromatic protons at δ 7.76, 7.43, 7.28, and 4.14 to shift slightly downfield to 7.77, 7.44, 7.28 and 4.15 respectively, indicating the interaction between the $Ag^+$ and compound **1** (Figure 3C, S13).[55] The Job's plot indicated a 2:1 binding stoichiometry between the compound **1** and the $Ag^+$ ions, which was unexpected and somewhat baffling result since the number of coordination sites in the molecule was very limited (Figure S14). Finally, the single crystal XRD analysis of the crystals of the "complex" clearly revealed the formation of a silver(I)-azobenzene coordination polymer with bridging nitrate groups, crystallizing in the monoclinic P21 space group (Figure 2B). The crystallographic data and selected bond distances and angles are listed in the supporting information (Tables S2-S4). The silver nitrate forms the main backbone of the chain, with two azobenzene units coordinated to the silver(I) through the sulfur atom of **1** (Figure S17). The growth of this one-dimensional coordination polymer was investigated through dynamic light scattering (DLS) studies in acetonitrile (Figure 3E, S18). It was found that over the course of 5 days, the hydrodynamic volume of the particles increased from 100 (±10) nm to 500 (±10) nm. Scanning electron microscopy (SEM) confirmed a rod-shaped morphology of the dried sample of the coordination polymer (Figure 3F, S19).

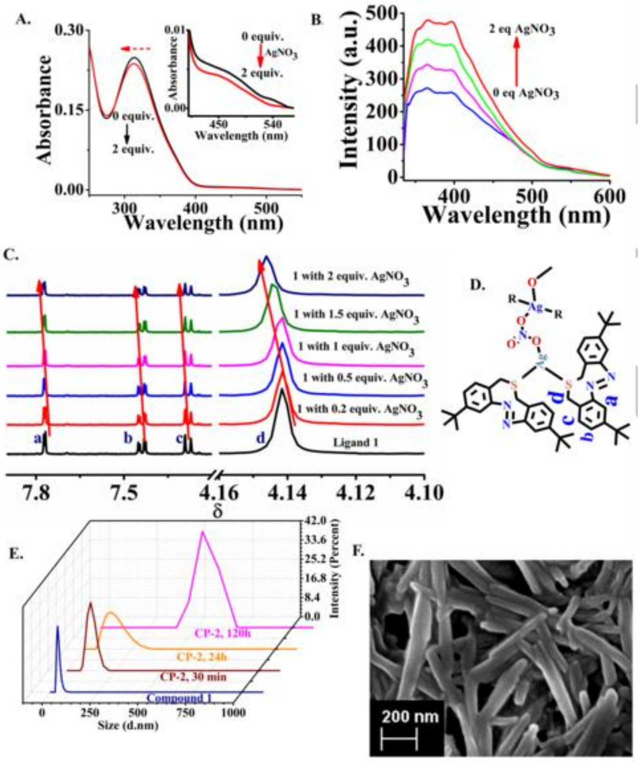

**Figure 3**: (A) UV-Vis spectral changes of **1** (30 μM) upon complextation with $AgNO_3$ (0-60 μM), (B). Changes in the emission spectra of **1** (30 μM) upon addition of $AgNO_3$, $\lambda_{ex}$ 320 nm at 298K in acetonitrile-methanol solvent (3:2 v/v), (C) $^1H$ NMR titration of compound **1** upon increasing concentration of $AgNO_3$. (E). dynamic light scattering data of CP- **2**; (F). SEM images of CP **2.**

UV-Vis spectroscopy observations of **2** (30 μM in acetonitrile) were similar to those of **1**. On exposure to UV light of 366nm (irradiation power of 2.3 mW $cm^{-2}$), the *trans* form of CP-**2** gradually underwent isomerization into the *cis* isomer, forming a PSS with a 70:30 ratio of *cis*:*trans* isomers, the same as the composition of the two isomers of **1** (Figure 4D-4E and S15). During the isomerization from the *trans* to the *cis* form of the CP-**2**, the absorption peaks at 308 nm (ε = 7700 $M^{-1}.cm^{-1}$) and 444 nm (200 $M^{-1}.cm^{-1}$) gradually diminished and a new peak was observed at 406 nm (ε =230 $M^{-1}.cm^{-1}$).

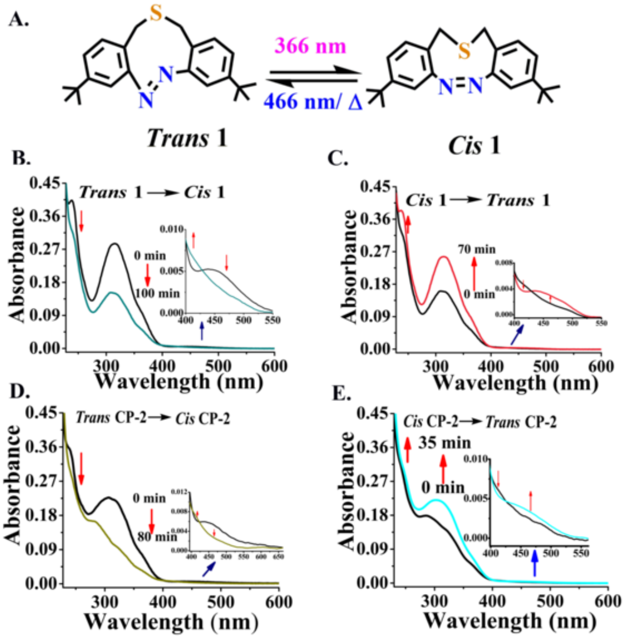

**Figure 4:** (A) Schematic representation of compound **1.** Photoisomerization of compound **1** and CP-**2.** (B) and (C) for the compound **1**; (D) and (E) for the CP- **2** all the cases concentration was 30 μM in acetonitrile solvent at ~ 273 K (ice-cool) temperature under 366 nm UV light and 466 nm Blue light.

The reverse reaction upon exposure to 466 nm light (blue LED, irradiation power of 0.5 mW $cm^{-2}$) proceeded similarly. The rate of the *trans* to *cis* conversion of the CP-**2** was similar to that of the compound **1**. However, the reverse reactions under the 466 nm light was 2.5 times faster for the polymer **2** at 273K compared to the compound **1**. Photoisomerization studies of **2** monitored by $^1H$ NMR showed that upon exposure to 366nm UV light the $^1H$ NMR signals at δ 7.76, 7.43 and 7.28 underwent a upfield shift to δ 7.07, 6.86 and 6.65 respectively (Figure S16)**.** These shifts on photoisomerization were higher for the $Ag^+$-complex than for the compound **1**.

In order to check the stability of the *cis* complex, kinetics studies were performed and various thermodynamic parameters, including $\Delta E_{act}$, $\Delta H^{\#}$, $\Delta S^{\#}$ and $t_{1/2}$ were calculated from the variable temperature kinetics data (Table 1). The thermal reversal of **1** and **2** were studied at various temperatures. The Arrhenius plot thus obtained from the kinetics data at 25, 35, 45 and 55 °C provided the activation energy for the *cis* to *trans* thermal switching process (Figure S20-S21). The activation energy $\Delta E_{act}$ for CP-**2** was found to be 20 kcal/mol, significantly (6 kcal/mol) lower than that obtained for the compound **1**, for which the value was found to be 26 kcal/mol. The $t_{1/2}$ values for **2** and **1** of 75 min and 300 min respectively followed the expected trend.

The probable reason of this decrease in the activation energy for the thermal *cis*→ *trans* reversal process can be ascribed to the complextation of the azobenzene ligand with the $Ag^+$ ions. [53]-[54] In the absence of the metal ion, the double bond character of the N=N double bond restricts the N-N rotation that slows down the thermal switching process. The $Ag^+$ ion, upon coordination to **1**, imparts a partial positive charge to the sulfur atom. Thus, the nitrogen lone pair in the *cis* form, which is in close proximity of the S-atom, gets polarized. This reduces the double bond character of the N=N bond. The reduction of the bond order consequently reduces the energy barrier of the switching process.

**Table 1.** Kinetics data for compound **1** and CP-**2**

| Species | *Cis* → *trans* activation[a] energy ($E_a$); *kcal/mol* | Enthalpy[b] of activation ($\Delta H^{\#}$) for *cis* to *trans* conversion; *kcal/mol* | Half life of the *cis* form $t_{1/2}$ *(min) at 298* K | Enthalpy[b] ($\Delta S^{\#}$) for *cis* to *trans* conversion (Cal/mol.K) |
|---|---|---|---|---|
| **1** | 26 | 25.5 | 300 | 9 |
| **CP-2** | 20 | 20 | 75 | 5 |

[a] Arrhenius equation (Figure S20-S20). [b] Eyring equation (Figure S22).

We also wondered if the electrical conductance of the CP-**2** can be modulated with light. It is known that azobenzene systems by themselves cannot switch conductance upon photoisomerization.[56] However incorporation of azobenzene into covalent and non-covalent carbon nanomaterials display their ability to modulate conductance in the assemblies.[57] Therefore, the switching of the electronic mobility upon the *cis-trans* isomerization of our silver-cyclic azobenzene CP-**2** was investigated. The reversible switching of the conductance was studied in the solid state in a thin-film on an indium tin oxide (ITO) surface. Fabrication of the device for the investigation of the conductance was done by wet etching of the CP-**2** on the ITO glass substrate with a layer width of ~200 nm with an Al-layer as the top contact (Figure 5A, see supporting information for details of fabrication).

The current-voltage (*I*-*V*) characteristic studies shown in Figure 5B displayed a linear current−voltage (*I*−*V*) plot of the hybrid at a voltage ranging from 0 to +2 V. The CP-**2** in the *cis* form displayed a strong I−*V* response whereas, with the *trans* form the response was much weaker. Therefore to study the effect of light upon conductance, the *trans*-isomer of CP-**2** was photo-irradiated with 366 nm light and converted to the *cis* CP-**2** and the conductance was measured. The response ratio between the two isomers, defined by $I_{cis}(V)/I_{trans}(V)$ at a voltage of 1 V was found to be 16. The ratio of $\delta I/\delta V$ of the *cis* CP-**2** and the *trans* CP-**2** obtained from the slopes of the two straight lines was 14.3. It is to be noted that the switching efficiency in the solid state was much slower compared to the efficiency in the solution phase.

Although the CP-**2** exhibits a remarkable increase in the current for the *cis* form compared to the *trans* form, the silver nitrate backbone by itself exhibits no increase in the current even after UV illumination, confirming the role and importance of the azobenzene ligand. This result was in well agreement with a report by Feng *et al.* where a conductance switch of covalently linked carbon nanotubes and azobenzene was studied. [56]

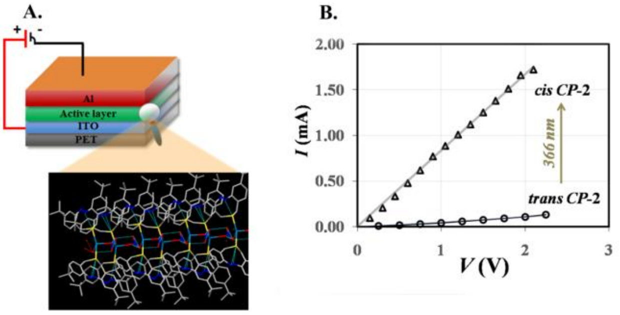

Figure 5: (A). Schematic representation of the device set-up for the conductance studies in the solid state on a thin-film; (B). I-V characteristics graph of the two photoisomers of CP- **2** obtained by photo-irradiation as indicated in the figure.

A plausible explanation for this current increase relies on the difference in dipole moment between the two geometrical isomers. The structure of the *cis* isomer has the two lone pairs of the nitrogens in the double bond oriented in the direction of the sulfur, and thus are able to donate more of the electron density to the sulfur (and in turn the silver) as compared to the *trans* isomer, in which only one of the lone pairs of the nitrogens is pointing towards the sulfur. In case of carbon nanotube-azobenzene systems, a similar geometry change induced mechanism for conductance modulation is reported.[57] The switching of conductance is illustrated in Figure 5B – the increase in the observed current is due to significant *trans-cis* isomerization, and the fall in current under visible light is due to the thermal *cis*−*trans* back isomerization.

## Conclusions

Coordination polymers having photoswitching ability offer photocontrolled tunable properties. Although, a few reports of linear azobenzene based CP exist, coordination polymers with cyclic azobenzene systems have not been reported earlier. This work reports a cyclic azobenzene based photoswitchable ligand that coordinates to silver ions and forms a polymeric network through nitrate bridges. The structure of the polymer is characterized by single crystal X-ray crystallography which shows that each monomeric unit consists of two S-atoms from the two azobenzene-ligands that are coordinated to a silver ion. The monomeric units are linked by bridging nitrates which impart its linear structure. The polymer displays superior switching speed with light compared to the ligand itself. The thermal conversion from the *cis* to the *trans* isomer was studied by variable temperature kinetics. The VT kinetics revealed the thermodynamic parameters for the isomerization process that explained the faster switching of the polymer is due to the fact that the energy of activation for the isomerization is lower in the polymer compared to the ligand itself by 6 kcal/mol. The coordination polymer was spin-coated on an ITO surface where the *trans* form display a modest conductance. However, the *cis* form obtained under the UV exposure displayed a drastic enhancement of the conductance. This work demonstrates the photo modulation of electronic mobility of a coordination polymer.

## Acknowledgements

The authors thank DST-SERB for financial support (Grant number EMR/2017/003720). MS and SC are supported by INSPIRE doctoral fellowship by DST-India. MSH is funded by a CSIR-JRF. AG is supported by a INSPIRE undergraduate scholarship.

**Keywords:** azo compounds • photochromism • silver • molecular electronics • electronic mobility

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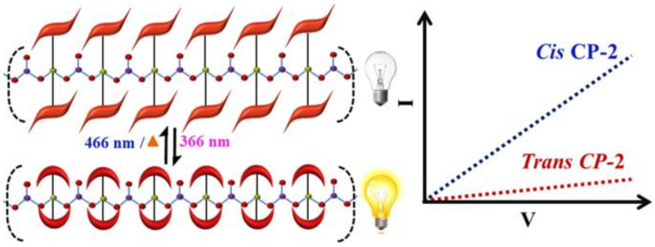

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***Modulation of electronic mobility of a one-dimensional coordination polymeric molecular wire with light***