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STEREOSELECTIVITY OF ELECTRON TRANSFER REACTIONS (II); REACTION OF Λ -[Co(EDDS)]⁻ AND Co(II) COMPLEXES WITH STEREOSPECIFIC DIAMINE LIGANDS

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STEREOSELECTIVITY OF ELECTRON TRANSFER REACTIONS (II); REACTION OF Λ -[Co(EDDS)]⁻ AND Co(II) COMPLEXES WITH STEREOSPECIFIC DIAMINE LIGANDS

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Stereoselective electron-transfer between optically active Λ -[Co(EDDS)]⁻ and [Co(diamine)₃]²⁺ (diamine = racemic-1,2-diaminopropane (pn); *R*-1,2-diaminopropane (*R*-pn)) have been investigated in aqueous and in DMSO solutions. In aqueous solution, the reaction between Λ -[Co(EDDS)]⁻ and [Co(pn)₃]²⁺ produces four conformational isomers of the [Co(pn)₃]³⁺ complex which have the Λ -forms in excess. Their optical purities are increased $ob_3 > lelob_2 > lel_2ob > lel_3$. In contrast, the reaction in DMSO results in more Δ isomers than Λ and the sequence of optical purities are in reversed order.

The reaction between Λ -[Co(EDDS)]⁻ and [Co(*R*-pn)₃]²⁺ in aqueous solution produces two conformational isomers of [Co(*R*-pn)₃]³⁺; their absolute configurations are identified as Δ -*lel_2ob* and Λ -*ob_3*. In DMSO, in contrast, the reaction produces only one isomer which is identified as Δ -*lel_2ob*.

KEYWORDS: electron transfer, stereoselectivity, cobalt, diamine

INTRODUCTION

Although cobalt(III) coordinated with EDTA (ethylenediamine tetraacetate) is known to be very stable and is classified as an inert complex, it is reported to react easily with en (ethylenediamine) to produce the tris-ethylenediamine-cobalt

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(III) complex at room temperature.¹ It has also been reported that substitution of Δ -[Co(EDTA)]⁻ or Δ -[Co(*R*-PDTA)]⁻ (where PDTA is propylenediaminetetraacetate) with en produces 33% of Λ -[Co(en)₃]³⁺.³ The mechanism based on the fact that the intermediates in Busch's mechanism are inactive with en.^{2,3} Investigating the kinetic data, Taube, *et al.* suggested that electron-transfer is involved in this substitution reaction.⁴

Lappin, *et al.*, observed that optically active Δ -[Co(EDTA)]⁻ or Δ -[Co(*R*-PDTA)]⁻ reacts with *rac*-[Co(en)₃]²⁺ to give Λ -[Co(en)₃]³⁺ enantiomeric excess of 9% and 8%, respectively.^{5,6} Thus, chiral induction may involve an ionic association between oxidants and reductants. The optically active Λ -[Co(EDDS)]⁻ (EDDS: ethylenediaminedisuccinate) complex, which is equipped with a stereospecific ligand EDDS, reacts with *rac*-[Co(diamine)₃]²⁺ (diamine = en or 1,2-diaminocyclohexane (chxn)) to produce Δ -[Co(en)₃]³⁺ and Δ -[Co(chxn)₃]³⁺ in optical purities of 14% and 22–25%, respectively.⁷ Increased optical purity of [Co(chxn)₃]³⁺ compared to Δ -[Co(en)₃]³⁺ may be due to the rigidity of the inner-sphere ligands.

In this study, the reactions between stereospecifically synthesized Λ -[Co(EDDS)]⁻ and conformationally restricted [Co(diamine)₃]²⁺ (diamine = *rac*-1,2-diaminopropane (pn), *R*-pn) are performed in order to investigate the stereoselectivity of electron transfer reactions. The solvent effect on this reaction is also studied using DMSO.

EXPERIMENT

Synthesis of Ligand and Complexes

The complexes Λ -[Co(EDDS)]⁻, [Co(pn)₃]²⁺, *rac*-[Co(pn)₃]³⁺, and [Co(*R*-pn)₃]²⁺ were synthesized according to published methods.^{8–11} Optical resolution of *R*-pn was accomplished by modification of Dwyer's method.¹² The optical purities of the complexes were determined from their electronic absorption and circular dichroism spectra (CD).

Stereoselective Ionic Association Between Metal Complexes

Rac-Co[(pn)₃]³⁺ was adsorbed to form a 2 cm band on a Sp-Sephadex C-25 cation exchange resin column (130 × 1.2 cm), and was eluted with 0.2 M Na₃PO₄ solution.¹³ The adsorbed Co(III) complex was separated into four bands which were removed sequentially. The first fraction was adsorbed again as a 2 mm band in the column and eluted with 0.1 M Λ -[Co(EDDS)]⁻ solution containing 0.1 M sodium perchlorate. After the band spread over 4–5 cm, the column was washed by water, then eluted by 0.5 M NaCl. The first and last fraction of the elutant was collected and the stereoselectivity of each fraction was checked using a Jasco J-500-C spectropolarimeter.

Electron-Transfer Measurements

All reactions were performed at room temperature under nitrogen to prevent aerial oxidation of the Co(II) complexes. The mixed solution of Λ -[Co(EDDS)]⁻ (0.04 M)

and $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ (0.04 M) solutions and the pn (0.6 M) solution were purged with deoxygenated nitrogen. After 15 min of purging the solutions were mixed. When the distinct purple-to-yellow color exchange was completed (*ca* 5 min.), the reaction mixture was acidified by adding 20 ml of nitrogen purged 1M HCl, and the solution was diluted with water to 500 mL. This solution was adsorbed onto a column of Dowex 50W-X2 cation exchange resin in the H^+ form. The column was washed with 2 M HCl to remove mono- and dications. Finally the yellow $[\text{Co}(\text{pn})_3]^{3+}$ complex was eluted with 5 M HCl and the elut was evaporated to dryness. The resulting residue was dissolved in water and absorbed onto a column (2.5 \times 10 cm) of Sp-Sephadex C-25 cation exchange resin in the Na^+ form in order to separate isomers in the complex, and then eluted with 0.2 M Na_3PO_4 solution. The resin containing the cobalt(III) complex was divided into four sections as reported.¹³ Each isolated section was again absorbed onto a column of Sp-Sephadex C-25 and washed with water to prevent induced circular dichroism due to phosphate ion, and then eluted with 0.5 M NaCl. The products were characterized by electronic absorption spectra (Shimadzu UV-2201) and circular dichroism spectra.

RESULTS AND DISCUSSION

It was reported that electron-transfer between $\Lambda\text{-}[\text{Co}(\text{EDDS})]^-$ and *rac* $[\text{Co}(\text{en})_3]^{2+}$ produces 14% e.e. $\Delta\text{-}[\text{Co}(\text{en})_3]^{3+}$ due to preferential ionic association between $\Lambda\text{-}[\text{Co}(\text{EDDS})]^-$ and $\Delta\text{-}[\text{Co}(\text{en})_3]^{2+}$ in the course of the reaction.⁷ The reaction of $\Lambda\text{-}[\text{Co}(\text{EDDS})]^-$ and *rac*- $[\text{Co}(\text{chxn})_3]^{2+}$ produces 22–25% e.e. $\Delta\text{-}[\text{Co}(\text{chxn})_3]^{3+}$.⁷ An increase in the optical purity of the latter compared to the former is due to effective ion pairing between the two complexes. Ion pairing is achieved through hydrogen bonds between an oxygen of the C_3 axis in $\Lambda\text{-}[\text{Co}(\text{EDDS})]^-$ and a hydrogen of the diamine of the C_2 axis on the $\Delta\text{-}[\text{Co}(\text{chxn})_3]^{3+}$ complex.

In this work, reactions between *rac*- $[\text{Co}(\text{pn})_3]^{2+}$ or $[\text{Co}(\text{R-pn})_3]^{2+}$ and $\Lambda\text{-}[\text{Co}(\text{EDDS})]^-$ are performed in order to investigated the inner-sphere effect in stereoselective electron-transfer reactions. The two reactants *rac*- $[\text{Co}(\text{pn})_3]^{2+}$ or $[\text{Co}(\text{R-pn})_3]^{2+}$ are known to be rigid because of the methyl group. Electron-transfer between $\Lambda\text{-}[\text{Co}(\text{EDDS})]^-$ and *rac*- $[\text{Co}(\text{pn})_3]^{2+}$ in aqueous solution was accomplished under a nitrogen atmosphere. The products were identified as $[\text{Co}(\text{pn})_3]^{3+}$ by electronic absorption spectra. Since there are various conformational isomers of the chelate ring, the product was absorbed onto a Sp-Sephadex C-25 cation exchange resin column in order to separate isomers and then eluted with Na_3PO_4 solution. The product is separated into four sections as *lel*₃, *lel*₂*ob*, *lelob*₂, *ob*₃ in the sequence identical to the previous report.¹³ From the area under the elution curves, the percentages of ionic distribution for each isomer can be calculated. The ratio of *lel*₃:*lel*₂*ob*:*lelob*₂:*ob*₃ is estimated to be 24:48:24:4. This ratio is different from that obtained previously 35:41:18:4.^{13a} The percent ionic distribution for formation of each isomer shows a similar pattern to that obtained from electron transfer reactions and general synthetic methods. The percent of *lel*₃ isomer reduced and that of *lel*₂*ob* is increased a little. The reason for variation in percent ionic distribution observed may be due to ionic association of the pn complex with $\Lambda\text{-}[\text{Co}(\text{EDDS})]^-$. Other possible reasons for this variation could be the relative

reactivity between the pn complex and Λ -[Co(EDDS)]⁻ and/or the relative stability of isomers which originate from the different conformations.

The CD spectra of the isomers are shown in Fig. 1. All isomers exhibit the positive Cotton effect in the first absorption region. The positive Cotton effect implies that the resultant [Co(pn)₃]³⁺ complexes are Λ . The optical purity of the isomers are measured from the CD spectra to be 2.25% e.e. ($\Delta\epsilon = +0.06$), 8.88% e.e. ($\Delta\epsilon = +0.2$), 16.24% e.e. ($\Delta\epsilon = +0.38$), and 34.95% e.e. ($\Delta\epsilon = +0.83$), respectively for *lel*₃, *lel*_{2ob}, *lelob*₂, and *ob*₃. Thus chiral induction in this set of reactions is due to preferential ionic association between Λ -[Co(pn)₃]²⁺ in its racemic mixture and the Λ -[Co(EDDS)]⁻ complex in the electron transfer reaction.

We have investigated ionic association between Λ -[Co(EDDS)]⁻ and *rac*-[Co(pn)₃]³⁺ in order to interpret the stereoselectivity. When *rac*-[Co(pn)₃]³⁺ adsorbed on the Sp-Sephadex C-25 cation resin is eluted with Λ -[Co(EDDS)]⁻

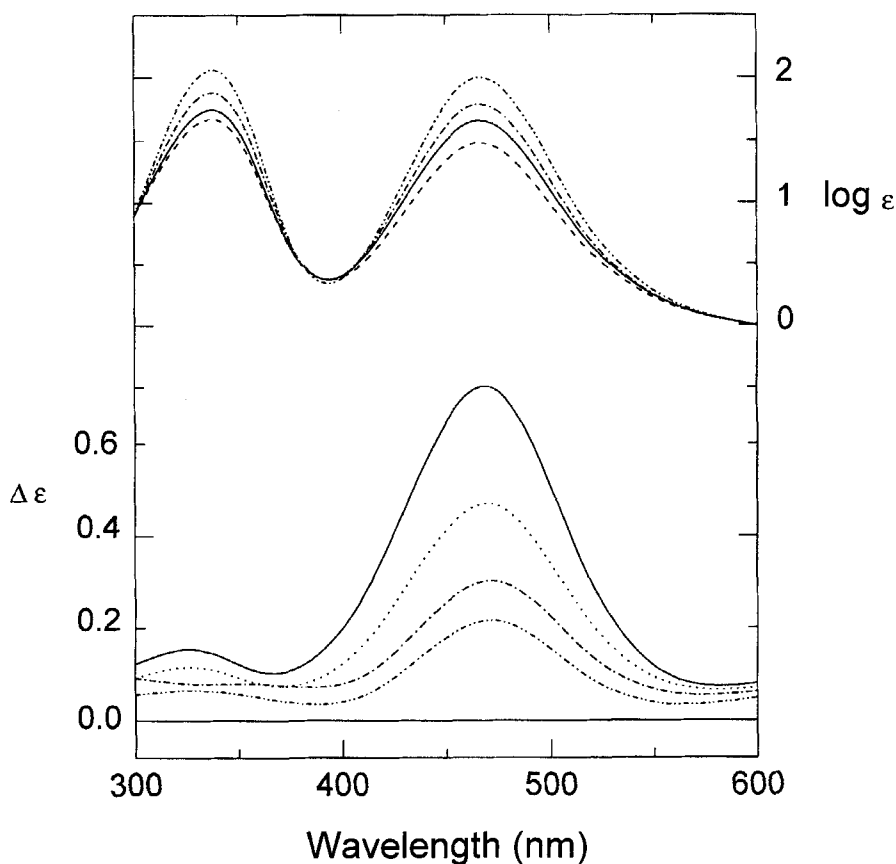


Figure 1 The electronic absorption and CD spectra of conformational isomers of Λ -[Co(\pm pn)₃]³⁺ in aqueous solution; *lel*₃ (---), *lel*_{2ob} (-.-), *lelob*₂ (.....), *ob*₃ (—).

solution, the first eluted fraction is shown to give a positive Cotton effect and last fraction gives a negative Cotton effect (Fig. 2 (a) and (b), respectively). This observation suggests that ionic association between Λ -[Co(EDDS)]⁻ and Λ -[Co(pn)₃]³⁺ is preferred. This preferential stereoselectivity is in agreement with the stereoselectivity for electron transfer: We have considered the chirality of each complex in order to interpret the ion pairing mechanism based on this result. In Λ -[Co(pn)₃]³⁺, the helicity is composed of M(C₃)P(C₂){M(C₃) = the helicity is minus along the C₃ axis, P(C₂) = plus along the C₂ axis}. Whereas Λ -[Co(EDDS)]⁻ is designated M(C₂)P(C₃). Since homochiral pairwise interactions (PP or MM) are believed to be favored,¹⁴ the pair is effective in the C₂-C₃ interaction. The ionic association model is displayed in Fig. 3. This result is identical with that previously reported for Λ -[Co(EDDS)]⁻ and Δ -[Co(en)₃]³⁺.⁷ The pairing is from effective hydrogen bonding through the C₂ axis of Δ -[Co(en)₃]³⁺ and the C₃ axis of Λ -[Co(EDDS)]⁻. Despite the same helicity, there is some difficulty for ionic

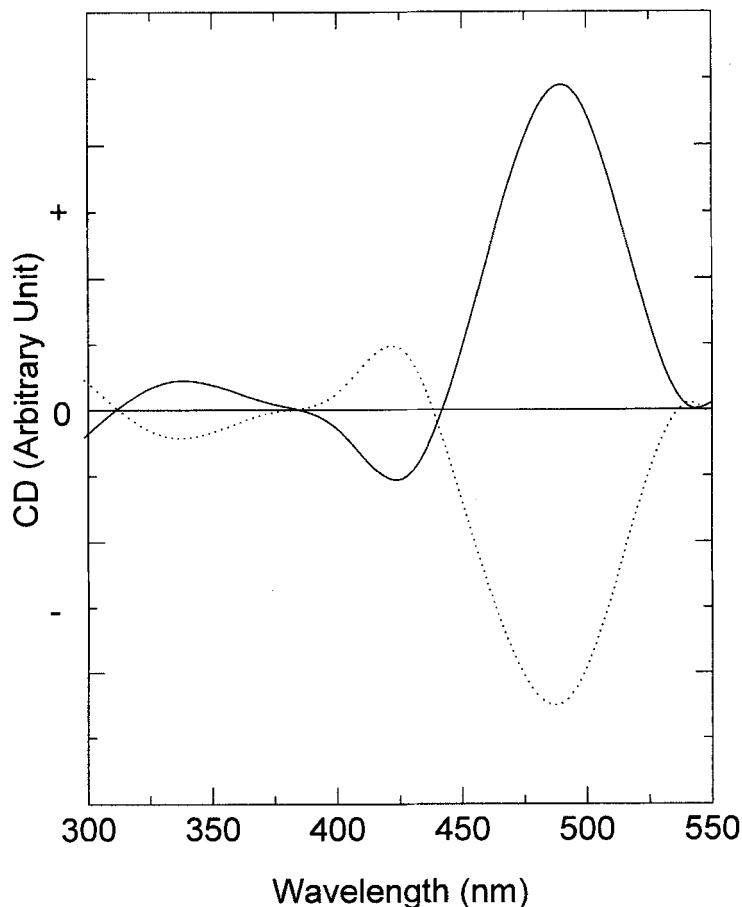


Figure 2 The CD spectra of [Co(±pn)₃]³⁺ according to elution order. The first elution (—), last elution (----).

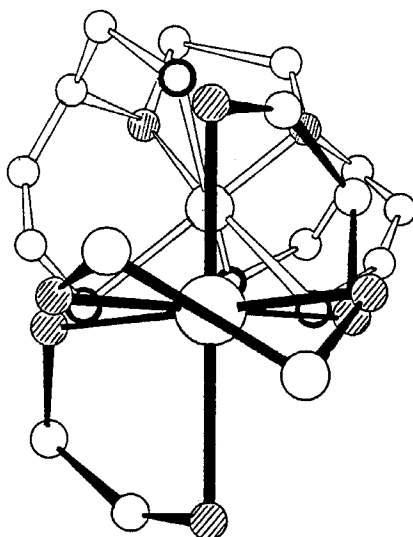


Figure 3 The model of ionic association for Λ -[Co(EDDS)]⁻ with Λ -[Co(pn)₃]³⁺. Carbon (○), nitrogen (⊗), and oxygen (●).

association between Λ -[Co(EDDS)]⁻ and Λ -[Co(pn)₃]³⁺ because the methyl group in the ethylenediamine backbone hinders effective approach of Λ -[Co(EDDS)]⁻. That the optical purity of the produced [Co(pn)₃]³⁺ in the electron-transfer reaction is less than that of [Co(en)₃]³⁺ supports the homochiral pairwise interaction model.

The [Co(R-pn)₃]²⁺ complex has been reacted with the Λ -[Co(EDDS)]⁻ complex in aqueous solution to investigate the stereoselectivity of electron transfer. Since conformational isomers can coexist in the product, we have isolated each isomer utilizing the same method as *rac*-[Co(pn)₃]²⁺. The product is divided into two fractions in the column which are identified as *lel*₃ and *ob*₃ respectively. The percent isomer distributions for formation from the elution curve are 93% *lel*₃ and 7% *ob*₃ with the *lel*₃ isomer the first eluted one and the *ob*₃ isomer last one. The absolute configuration of these isomers were Δ -*lel*₃(A) and Λ -*ob*₃(B) by the Cotton effect as shown in Fig. 4. These results indicate the importance of the stabilities of an oriented methyl group in stereospecific 1,2-diaminopropane rather than electron-transfer between the complexes. Our results on formation of the two isomers agrees with an earlier report¹⁵ for [Co(R-pn)₃]³⁺ but the distribution of each isomer is somewhat different; Δ -*lel*₃ 93% and *ob*₃ 7% and their optical purities are 100% e.e. ($\Delta\epsilon = -2.3$) and 74% e.e. ($\Delta\epsilon = +1.83$), respectively.

The reaction between Λ -[Co(EDDS)]⁻ and *rac*-[Co(pn)₃]²⁺ was performed using the identical method in DMSO instead of water to investigate the solvent effect. Four isomers are observed as seen in aqueous solution. The percent isomer distribution is shown to be *lel*₃ 36%, *lel*₂*ob* 30%, *lelob*₂ 24%, and *ob*₃ 10% from the elution curve. The absolute configuration of each isomer was in excess Δ form. Their optical purities were *lel*₃ 19% ($\Delta\epsilon = -0.5$), *lel*₂*ob* 28% ($\Delta\epsilon = -0.64$), *lelob*₂ 27% ($\Delta\epsilon = -0.62$), *ob*₃ 29.72% e.e. ($\Delta\epsilon = -0.74$). The absolute configurations of the products are inverse compared to those obtained in water. In order to explain

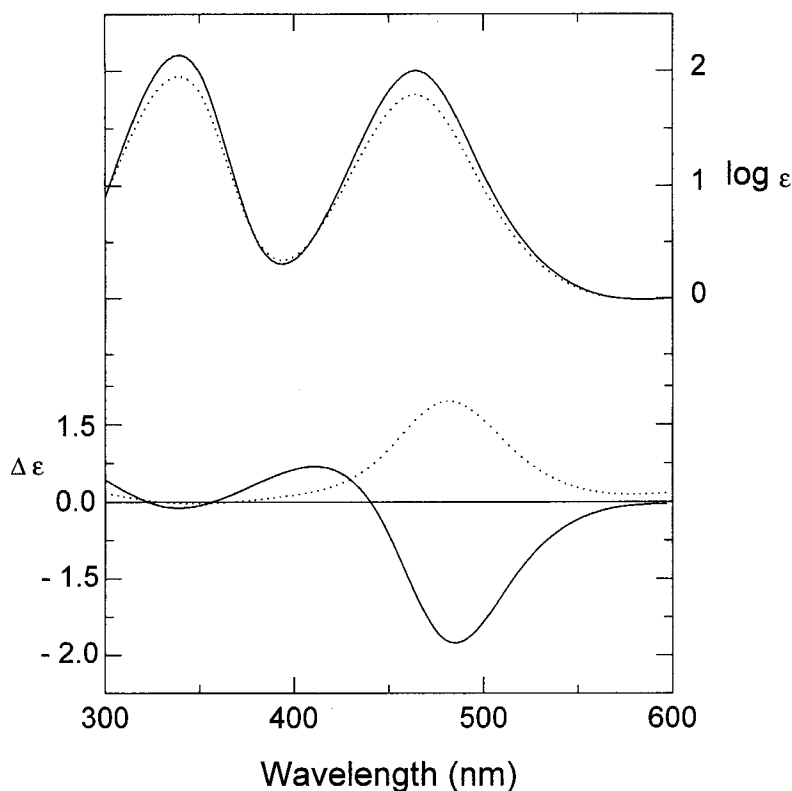


Figure 4 The electronic absorption and CD spectra of conformational isomers of $[\text{Co}(\text{R-pn})_3]^{3+}$ in aqueous solution; lel_2ob (—), ob_3 (....).

this difference, we investigated ion pairing between Λ - $[\text{Co}(\text{EDDS})]^-$ and rac - $[\text{Co}(\text{pn})_3]^{3+}$ in DMSO. Although we have not obtained clear evidence, we propose an ionic association model based on the results of electron-transfer reactions (Fig. 5). As previously discussed, we have estimated the helicity on each complex. The helicity of Δ - $[\text{Co}(\text{pn})_3]^{3+}$ is designed as $P(C_3)M(C_2)$ and the helicity of Λ - $[\text{Co}(\text{EDDS})]^-$ is $P(C_3)M(C_2)$. This pair prefers to have the C_3 - C_3 association because the C_3 - C_3 interaction is more selective than a C_2 - C_2 interaction; particularly Λ - $[\text{Co}(\text{EDDS})]^-$ having a stereoselective C_3 site is much more selective. The ion-pairing model that is shown in Fig. 6 is not obstructed by the methyl group of the pn ligand and the complexes are very effective in approaching each other. The inversion of absolute configuration in DMSO can be explained by this model. In DMSO, ionic association is very effective, in fact there is no obstacle to association along the C_3 - C_3 axis; in aqueous solution the conformation change of methyl group of chelate in approach of $[\text{Co}(\text{pn})_3]^{3+}$ and Λ - $[\text{Co}(\text{EDDS})]^-$ complex creates an obstacle along the C_3 - C_3 axis.

Other factors should be considered for the medium effect. DMSO is bulkier than water due to differences in solvation. Water is less abundant at the C_2 site compared to the C_3 site because there is some difficulty of solvation at the C_2 face by the

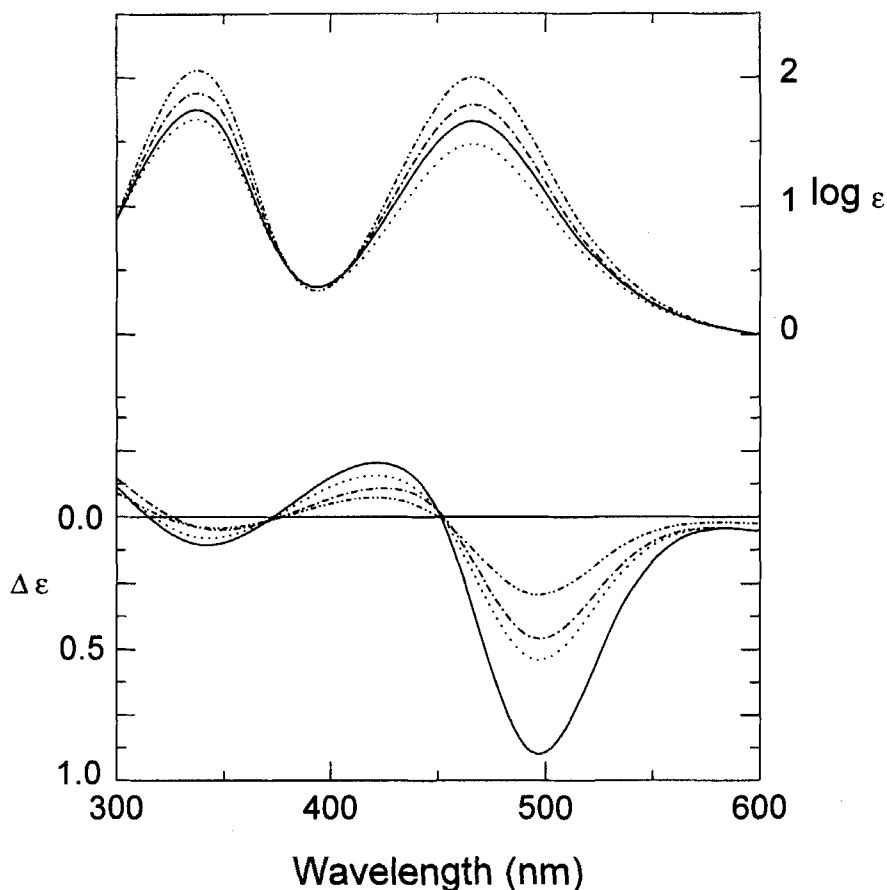


Figure 5 The electronic absorption and CD spectra of conformational isomers of Δ -[Co(\pm pn) $_3$] $^{3+}$ in DMSO; ob_3 (—), $lelob_2$ (....), $lel_2 ob$ (-.-), lel_3 (- - -).

conformational change of the methyl group on the pn ligand. The C_2 site is less shielded by water than the C_3 site. Therefore, the orientation of Λ -[Co(EDDS)] $^-$ in the C_2 direction is more effective than in the C_3 direction without hinderance by water. Water acts as an inhibitor to ionic association. Likewise, added salt to control the ionic strength causes a reduction in optical purity. Particularly, considering the couples interacting as C_3 - C_2 , it is reasonable that the tris diamine complex is oriented in the C_2 direction e.g. Δ -[Co(edta)] $^- \sim \Lambda$ -[Co(en) $_3$] $^{3+}$, Λ -[Co(EDDS)] $^- \sim \Delta$ -[Co(en) $_3$] $^{3+}$. The inversion of absolute configuration in DMSO can be understood by the bulkiness of DMSO. The bulkiness does not allow selective solvation like water and ionic association is not hindered by the methyl group on the pn ligand for conformational change. Furthermore, considering the proposed ion-pairing model, that the C_3 - C_3 interaction is achieved in effective association. The relatively good association between Λ -[Co(EDDS)] $^-$ and Δ -[Co(pn) $_3$] $^{2+}$ in DMSO can be achieved without the obstacles of hydrogen bonding. Optical purities are also affected. The proposed model is also

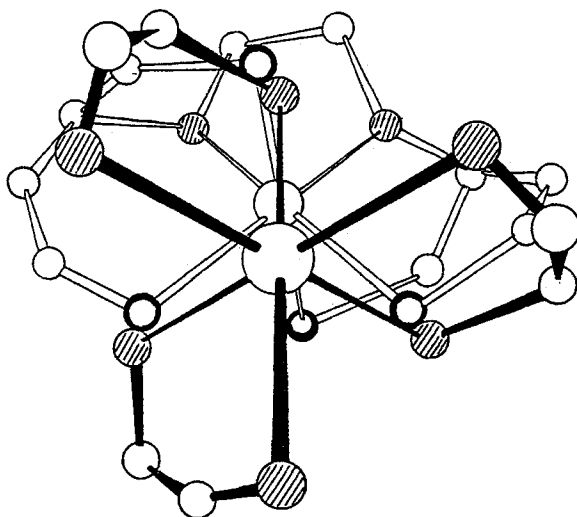


Figure 6 The model of ionic association between Λ -[Co(EDDS)]⁻ and Δ -[Co(pn)₃]³⁺. Carbon (○), nitrogen (⊗), and oxygen (○).

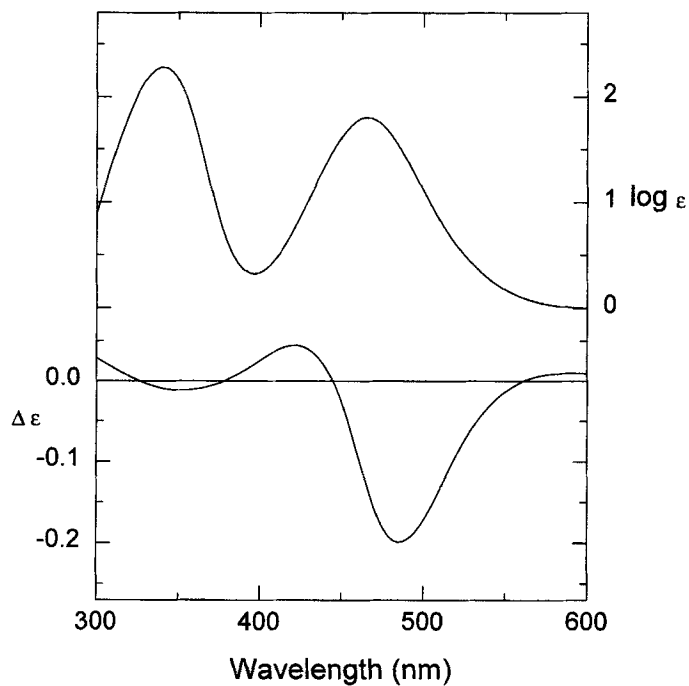


Figure 7 The electronic absorption and CD spectra of conformational isomers of Δ -ob-[Co(R-pn)₃]³⁺.

supported by comparison of the product chirality. That obtained in DMSO is greater than in aqueous solution (Δ -[Co(EDTA)]⁻ ~ Λ -[Co(en)₃]³⁺, Λ -[Co(EDDS)]⁻ ~ Δ -[Co(en)₃]³⁺).

On the other hand, the electron transfer reaction between Λ -[Co(EDDS)]⁻ and [Co(R-pn)₃]²⁺ in DMSO produces only the *lel*₃-[Co(R-pn)₃]³⁺ isomer. Its absolute configuration and optical purity are identified as Δ -*lel*₃-[Co(R-pn)₃]³⁺ by CD spectra (Fig.7). The dominantly formed isomer has a high probability of association with Λ -[Co(EDDS)]⁻ during the electron transfer reaction.

Acknowledgment

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