that of $Tl(TCNQ)_3$, it may be closely related to Tl-Cl bond strength.

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Cobalt(III) Complexes of N_4 and N_2O_2 System Tetradentate Ligands : Amino Acid Cobalt(III) Complexes of 1,3-Diaminopropane-N,N′-Di-α-(β-methyl)-Pentanoic Acid

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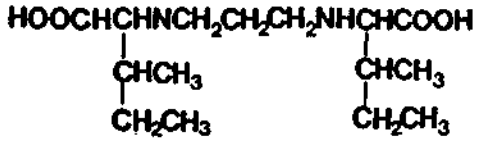
Amino acid cobalt(III) complexes of 1-3-diminopropane-N,N′-di-α-(β-methyl)-pentanoic acid (H_2dpdmp), *uns-cis*-[Co(dpdmp)(aa)] (aa=glycine, S-alanine, R-aspartic acid, sarcosine) have been prepared from the reaction between the *uns-cis*-$[Co(dpdmp)Cl_2]^-$ complex and the corresponding amino acid. In the reaction with the *uns-cis*-$[Co(dpdmp)Cl_2]^-$ complex, glycine and S-alanine have yielded both merridional and facial isomers, while R-aspartic acid and sarcosine, only merridional isomers. The stereospecific substitution reaction of R-aspartic acid to racemic *uns-cis*-$[Co(dpdmp)Cl_2]^-$ complex has yielded two merridional diastereomers; Δ*R-uns-cis*- and Λ*R-uns-cis*-[Co(dpdmp)(R-asp)]. It is of interest to note that this is one of the few $Co^{III}(ONNO)(aa)$ type complex preparations, which gives only one isomer with stereospecificity. On the other hand, two merridional products obtained from the reaction of sarcosine with racemic *uns-cis*-$[Co(dpdmp)Cl_2]^-$ are turned out to be mixtures of optical isomers.

Introduction

1,3-diaminopropane-di-α-(β-methyl)-pentanoic acid (H_2dp-



*Anthor to whom correspondence should be assressed.

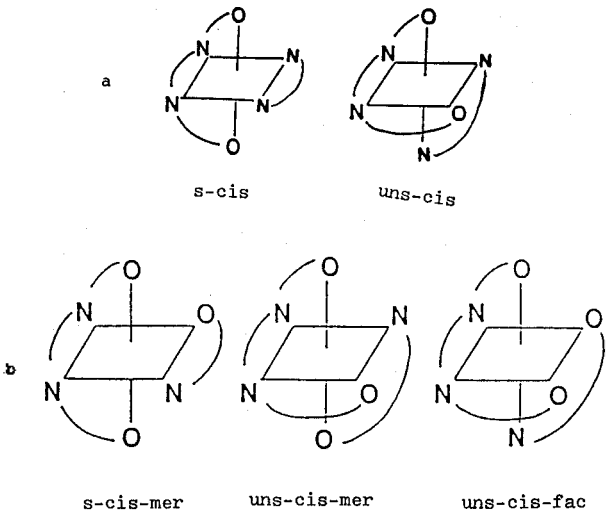


Figure 1. Two possible geometrical isomers in the $[Co(H_2O_4)(en)]^{n+}$ complexes (a), and three possible isomers in the $[Co(N_2O_4)(aa)]^{n+}$ complexes where aa is an amino acid having nitrogen and oxgen donor atoms.

dmp), alinear flexible tetradentate ligand of the type ONNO in the donor atom array, has been synthesized for the first time in this laboratory.[1]

The dpdmp anion is a tetradentate ligand having two nitrogen and two oxygen donor atoms, and has been found to yield exclusively the *uns-cis* (unsymmetric *cis*) geometric isomer in or out of the three possible isomers (Figure 1a) in the preparation of the dichloro cobalt(III) complex, $[Co(dpdmp)Cl_2]^-$, as well as the dinitro $[Co(dpdmp)(NO_2)_2]^-$ and carbonato $[Co(dpdmp)CO_3]^-$ complexes. The geometrical isomerism in the cobalt(III) complexes of the N_2O_2-type tetradentate ligands has been studied extensively.[2~8] When L is a symmetrical bidentate ligand such as ethylenediamine in the $[Co(N_2O_2)L]^{n+}$ complexes, there are two possible geometrical isomers : s-*cis* and *uns-cis* (Figure 1a). When L is an unsymmetrical bidentate ligand such as an amino acid, however, an additional isomerism is possible as shown in Figure 1b : s-*cis-mer* (symmetrical-*cis*-merridional), *uns-cis-mer*, and *uns-cis-fac* (unsymmetric-*cis*-facial).

As a part of our continuing study of the cobalt(III) complexes of dpdmp, we have been interested in the amino acid complexes of [Co(dpdmp)(aa)] ((aa=glycine, S-alanine, R-aspartic acid, sarcosine). Earlier studies on the other complexes of the type $[Co(N_2O_2)(aa)]$ have shown that, while both meridional and facial isomers are yield ed in some complexes, preference for either merridional or facial isomer is shown by other complexes.[2,3] It is of interest to observe which isomers would be obtained from the reaction between $[Co(dpdmp)Cl_2]^-$ and amino acid.

Experimental

Glycine, S-alanine, R-aspartic acid, and sarcocine were purchased from Aldrich Chemical Co. and used without further purification. Dowex 50W-X4 cation exchange resin (200-400 mesh, H^+ form) and Dowex 1-80X anion exchange resin (200-400 mesh, Cl^- form) were used after repeated purifications.

Electronic absorption and infrared spectra were recorded on a Shimadzu UV-240 double Beam Spectrometer and a Shimadzu IR 435 Spectrometer, respectively. Pmr spectra were measured with a 270 MHz JEOL GSX-270 Spectrometer and a 80 MHz Varian FT-80A spectrometer. Elemental analyses were performed by Micro-Tech Analytical Lab., Skokie, Illinois, USA.

Preparation of 1-3-diminopropane-N,N'-di-α-(β-methyl)-pentanoic acid (H_2dpdmp) and *uns-cis*-H[Co(dpdmp)Cl_2]$^-$. The ligand dpdmp and the dichloro cobalt (III) complex of dpdmp have been described elsewhere.[1]

Preparation of *uns-cis-mer*- and *uns-cis-fac*-1-3-diminopropane-N,N'-di-α-(β-methyl)-pentanatoglycinatocobalt(III), *uns-cis-fac*- and *uns-cis-mer*-[Co(dpdmp)(gly)]. 0.65 g (1.5 mmol) of *uns-cis*-H[Co(dpdmp)Cl_2] was dissolved in 30 mL of water. The solution was heated to 60 ℃, and stirred for 30 min. 0.11 g (1.5 mmol) of glycine was added and the pH of the solution was sdjusted to 8.0 with 1.0 N NaOH. 0.1 g of active carbon was added and the solution was filtered. The filtrate was concentrated to 5 mL, which was admitted to a column packed with Dowex 50W-X4 cation exchage resin. Two bands were detected. Each of the two collected fractions was concentrated to isolate the violet *uns-cis*-merridional and pink *uns-cis*-facial isomers. Yield: *uns-cis-mer* : 0.2 g (31%); *uns-cis-fac*, 0.04 g (6%) Anal. Calcd. for $CoC_{17}H_{28}N_3O_6$: C, 47.33; H, 6.41; N, 9.69. Found: *uns-cis-mer* : C, 47.24; H, 6.41; N, 9.69. *uns-cis-fac*: C, 47.10; H, 6.65; N, 9.60

Preparation of *uns-cis-mer*- and *uns-cis-fac*-S-alaninato-1-3-diminopropane-N,N'-di-α-(β-methyl)-pentanatocobalt(III), *uns-cis-fac*- and *uns-cis-mer*-[Co(dpdmp)(S-ala)]. These were prepared from the same method as that used to prepare [Co(dpdmp)(gly)] using 1.5 mmol of S-alanine in place of glycine. Two bands were detected from the cationic exchange resin column. Each of the two collected fractions was concentrated to isolate the pink *uns-cis*-facial and the violet *uns-cis*-merridional isomers, which were recrystallized from ethanol and ether, and vacuum dried. Yield: *uns-cis-mer:* 0.1 g (15%); *uns-cis-fac*, 0.03 (5%) Anal. Calcd. for $CoC_{18}H_{32}N_3O_6$: C, 48.54; H, 7.19; N, 9.43. Found: *uns-cis-mer* : C, 49.21; H, 7.32; N, 9.28. *uns-cis-fac*: C, 49.10; H, 6.95; N, 9.60

Preparation of *uns-cis-mer*(1)-and *uns-cis-mer*(2)-1-3-diminopropane-N,N'-di-α-(β-methly)-pentanatosarcosinatocobalt(III), *uns-cis-mer*(1)- and *uns-cis-mer*(2)-[Co(dpdmp)(sar)]. These were prepared according to the same method as that used to prepare [Co(dpdmp)(gly)] using 0.9 g (1.0 mmol) of sarcosine in place of glycine. Two band fractions were collected and both fractions were turned out to be the merridional isomers. Yield: *uns-cis-mer*(1) (The first band): 0.02 g (2.2%); *uns-cis-mer*(2) (The second band), 0.3 g (33%) Anal Calcd for $CoC_{18}H_{32}N_3O_6$: C, 48.54; H, 7.19; N, 9.84. Found: *uns-cis-mer*(1): C, 48.13; H, 6.96; N, 10.10. *uns-cis-mer*(2): C, 48.10; H, 7.21; N, 9.76.

Preparation of *uns-cis-mer*(1)- and *uns-cis-mer*(2)-1-3-diminopropane-N,N'-di-α-(β-methyl)-pentanato-R-aspartatocobalt(III), *uns-cis-mer*(1)- and *uns-cis-mer*(2)-[Co(dpdmp)(R-asp)]. These were prepared according to the same method as that used to prepare [Co(dpdmp)

(gly)] using R-aspartic acid (0.7 g, 5 mmol) in place of glycine. Two fractions were collected from the cationic exchange resin column. The solid product from each fraction was recrystallized from ethanol and ether and vacuum dried. Yield: *uns-cis-mer*(1) : 1.3 g (54%); *uns-cis-mer*(2): 0.09 g (4%) Anal. Calcd. for $CoC_{19}H_{32}N_3O_8$: C, 46.63; H, 6.54; N, 8.59. Found: *uns-cis-mer*(1): C, 47.33; H, 6.81; N, 8.38; *uns-cis-mer*(2): C, 46.98; H, 6.79; N, 8.34.

Results and Discussion

The substituton reaction of the *uns-cis*-$[Co(dpdmp)Cl_2]^-$ complex with glycine, S-alanine, R-aspartic acid, and sarcosine has resulted in the isolation of the corresponding amino acid cobalt(III) complexes of dpdmp. Both meridional and facial geometric isomers have been obtained for the dpdmp cobalt(III) complexes of glycine and S-alanine. While no facial isomers has been formed, two merridional isomers, *uns-cis-mer*(1) and *uns-cis-mer*(2), have been produced for the dpdmp cobalt(III) complexes of sarcosine and R-aspartic acid.

Table 1 shows the-COO stretching frequencies for the dpdmp ligand and the complexes prepared in this work. The glycine complexes, *uns-cis*-[Co(dpdmp)(gly)], show the CO_2 vibration at 1630 cm^{-1} for the merridional iosmer and 1640 cm^{-1} for the facial isomer, indicating the bond formation between the metal ion and the ligand carboxylate oxgen donor atoms.

Two isomers have been isolated during the preparation of the dpdmp cobalt(III) complexes of glycine, one violet and the other pink. The electronic absorption spectrum is particullary helpful in distinguishing whether a geometric isomer is a meridional one or a facial one. In the visible spectrum of the CoN_3O_3 system, the band at the longer wavelength is due to the transition $A_{1g} \rightarrow {}^1T_{1g}$ (O_h) and the other one at the shorter wavelength to the transition $A_{1g} \rightarrow {}^1T_{2g}$ (O_h). The meridional geometry is lower in symmetry than the facial isomer. The loss of symmetry from the facial(holohedrized cubic symmetry, X=Y=Z) to meridional geometry (holohedrized rhombic symmetry) is expected to cause a splittiong or at least broadening of the $A_{1g} \rightarrow {}^1T_{1g}$ first band absorption for the merridional isomer.[9~11] Thus, the visible absorption peaks of the facial isomers are generally sharp and symmetrical, while those of the merridional isomers are broadened.[12~14] The electronic absorption spectra of the geometric isomers of the *uns-cis*-[Co(dpdmp)(gly)] (Figure 2) shows a sharp and symmetrical band in the visible region for the facial isomer pink in color an a broadened band in the same region for the violet meridional isomer. While the pmr spectra are not particularly helpful in distinguishing the merridional and facial isomer, the pmr spectrum of the *uns-cis*-merridional [Co(dpdmp)(gly)] complex (Figure 3) does show the α-methylene protons of the dpdmp at 3.5 ppm as two doublets, indicating the fact that the dpdmp ligand is coordinated to the cobalt(III) ion in the *uns-cis* geometry.

In the S-alanine complexes of *uns-cis*-[Co(dpdmp)(D-ala)], two geometrical isomers obtained in this work show the coordinated-COO vibration at 1630 and 1626 cm^{-1} (Table 1). The visible spectra of these complexes (Figure 4) show a sharp and nearly Gaussian band for the facial isomer in the visible region and a broadened band in the same region for the merridional isomer. Thus, the pink product is the facial isomer and the violet product the merridional isomer.

The R-aspartic acid has two carboxylate groups. The ir spectra of the R-aspartic acid complexes prepared in this

Table 1. The-COO Antisymmetric Stretching Frequencies of the *uns-cis*-[Co(dpdmp)(amino acid)] Complexes

Compound	as COO (cm^{-1})
dpdmp	1590
uns-cis-mer-[Co(dpdmp)(gly)]	1630
uns-cis-fac-[Co(dpdmp)(gly)]	1640
uns-cis-mer-[CO(dpdmp)(S-ala)]	1630
uns-cis-fac-[Co(dpdmp)(S-ala)]	1626
uns-cis-mer-[Co(dpdmp)(R-asp)]	1635, 1720[a]
uns-cis-fac-[Co(dpdmp)(R-ap)]	1650, 1720[a]
uns-cis-mer(1)-[Co(dpdmp)(sar)]	1620
uns-cis-mer(2)-[Co(dpdmp)(sar)]	1620

[a]Uncoordinated COO stretching band.

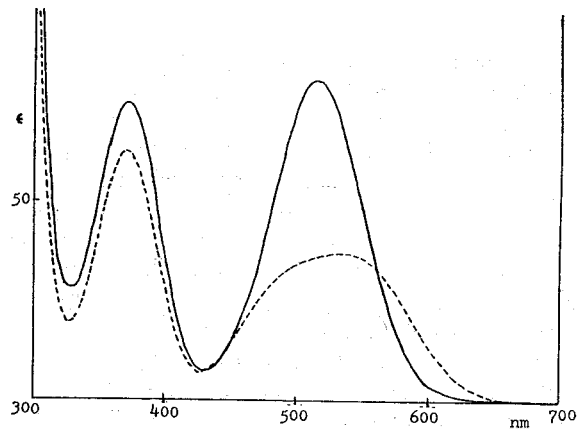


Figure 2. Electronic absorption spectra for the isomers of *uns-cis*-[Co(tmdmp)(gly)], *mer* (---), *fac* (−).

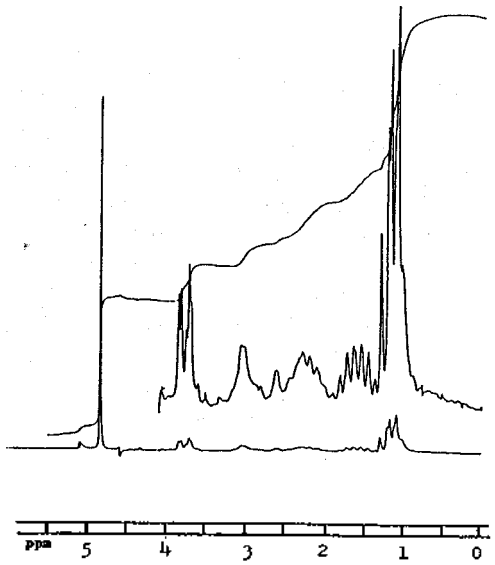


Figure 3. PMR Speotrum of *uns-cis-mer*-[Co(tmdmp)(gly)].

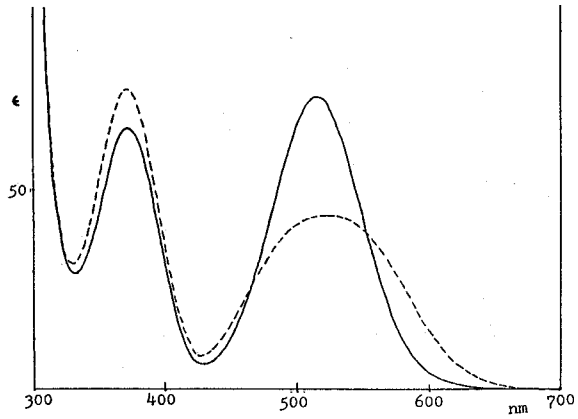


Figure 4. Electronic absorption spectra for the isomers of *uns-cis*-[Co(tmdmp)(S-ala)], *mer* (---), *fac* (—).

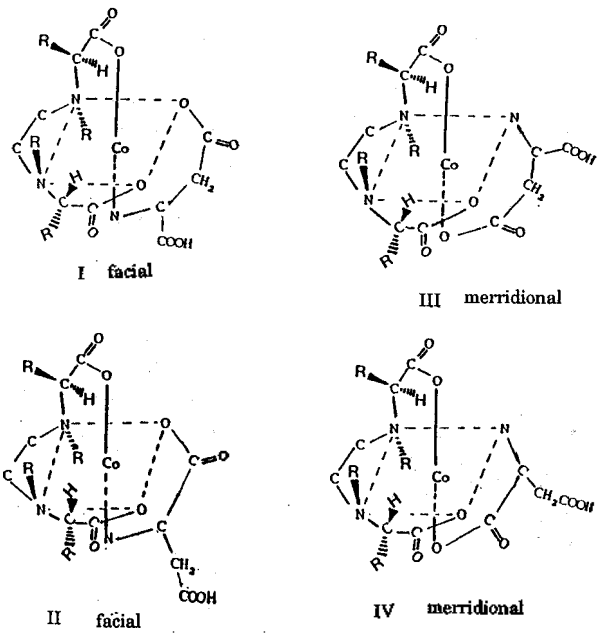


Figure 5. Four possible modes of N, O chelation in the *uns-cis*-[Co(dpdmp)(R-asp)]: six-membered chelate ring *via* the carboxylate group on the β-carbon of R-aspartic acid (structures I and III) and five-membered chelate ring *via* the carboxylate group on the α-carbon of R-aspartic acid (structures II and IV).

work show one uncoordinated carboxylate group at 1720 cm^{-1} (Table 1) and indicate the fact that R-aspartic acid is coordinated wia the amino group and one carboxylate group (N-O chelation). There are two possible modes of N-O chelation in the *uns-cis*-[Co(dpdmp)(R-asp)] for each of the meridional and facial isomers (Figure 5): six-membered chelate ring *via* the carboxylate group on the β-carbon (Structure I and III) and five-membered chelate ring *via* the carboxylate group on the α-carbon (Structure II and IV). The five-membered chelate ring is more stable than the six-membered chelate ring and the aspartic acid is generally known to coordinate *via* the amino group and the carboxylate group on the α-carbon to forma five-membered chelate.[13,15] Therefore, it is belived that in the *uns-cis*-[Co(dpdmp)(R-asp)] co-

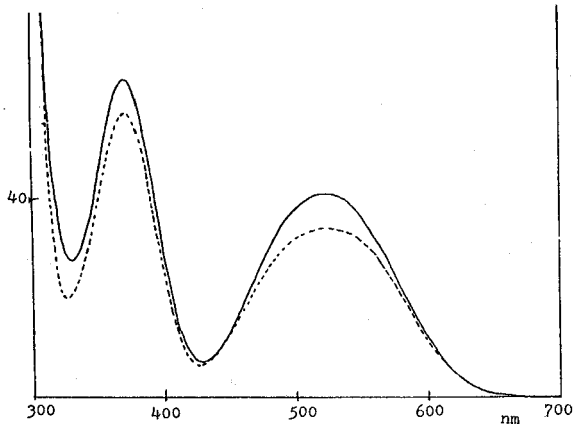


Figure 6. Electronic absorption spectra spectra for the isomer of *uns-cis*-[CO(tmdmp)(R-asp)], *mer* (---), *fac* (—).

Table 2. λ_{max} Values in the Visible Region of the *uns-cis*-[Co(dpdmp)(amino acid)] Complexes

Compound	λ_{max} (Band I)
uns-cis-mer-[Co(dpdmp)(gly)]	535
uns-cis-fac-[Co(dpdmp)(gly)]	515
uns-cis-mer-[Co(dpdmp)(S-ala)]	525
uns-cis-fac-[Co(dpdmp)(S-ala)]	515
uns-cis-mer(1)-[Co(dpdmp)(R-asp)]	525
uns-cis-mer(2)-[Co(dpdmp)(R-asp)]	523
uns-cis-mer(1)-[Co(dpdmp)(sar)]	518
uns-cis-mer(2)-[Co(dpdmp)(sar)]	520

mplexes prepared in this work, the aspartic acid is coordinated *via* a five-membered N-O chelation (Structure II or IV). During the preparation of the *uns-cis*-[Co(dpdmp)(R-asp)] complexes, two isomers have been obtained. Both isomers have essentially the same violet color an are belived to be the meridional isomers.

The visible spectra of the two *uns-cis*-[Co(dpdmp)(R-asp)] isomers (Figure 6) show broad bands along with the same λ_{max} (Table 2) and confirm our observation that both of these isomers are the meridional isomers, which in turn means that the facial isomers could not be formed in the *uns-cis* geometry. If the aspartic acid takes the facial isomer (Structure II, Figure 5), the non-bonded interactions or repulsions between the outside chelate ring coplanar with the middle chelate ring of dpdmp and the carboxymethyl group on the α-carbon of the aspartic acid would be too great to form a complex under the *uns-cis* geometry, while such interactions will almost be non-existent if the aspartic acid coordinates to the cobalt(III) ion to make a meridional isomer (Structure IV, Figure 5). Therefore, the aspartic acid should coordinate to give a meridional isomer in the [Co(dpdmp)(R-asp)] complex.

The *uns-cis*-···Co(dpdmp)Cl_2]$^-$ complex is a racemic mixture having the Λ and Δ enantiomers. The absolute configuration of the asymetric carbon in the R-aspartic acid is R and should take the λ chelate ring conformation upon coordination to a metal ion for the carboxymethyl group to be in an equatorial position. Therefore, the R-aspartic acid coor-

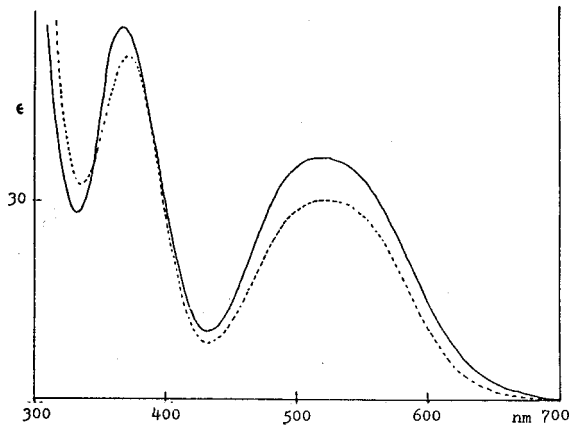


Figure 7. Electronic absorption spectra for the isomers of *uns-cis-mer*-[Co(tmdmp)(sar)], (Λ-S, Δ-R; —), (Δ-S, Λ-R; ---).

dinates stereospecifically to a metal ion to give the λ chelate ring conformation with the R absolute configuration of the asymmetric carbon, and the two meridional isomers are resulted from the following stereospecific reaction of R-aspartic acid to give the diastereomers:

Λ, Δ-*uns-cis*-$[Co(dpdmp)Cl_2]^-$ + R-asp →

R-*uns-cis-mer*(1)-[Co(dpdmp)(R-asp)] (1)

R-*uns-cis-mer*(2)-[Co(dpemp)(R-asp)] (2)

It is very interesting to note here that only the meridional isomers are formed in the *uns-cis*-[Co(dpdmp)(R-asp)] complexes due to the diastereoisomers mentioned above.

The substitution reaction of the *uns-cis*-$[Co(dpdmp)Cl_2]^-$ with sarcosine has yielded two isomers, which have the same violet color and are believed to be the meridional isomers. The visible absorption spectra of these products (Figure 7) indicate that they are the meriodional isomers. As is the case for the R-aspartic acid, the sarcosine should coordinate to a metal ion to give a meriodional isomer for the nonbonded interactions between the outside chelate ring coplanar with the middle chelate ring of dpdmp and the N-methyl group of sarcosine in the *uns-cis*-[Co(dpdmp)(sar)] complex are too great to form a facial isomer. Therefore, only the meridional isomers are obtained in the preparation of the *uns-cis*-[Co(dpdmp)(sar)] complex. The nitrogen donor atom of sarcosine becomes asymmetric upon coordination to a metal ion to have the R or S absolute configuration, and the two meridional isomers obtained in this work are resulted from the following substitution reaction to give four optical isomers (Δ-R, Δ-S, Λ-R, Λ-s):

Λ, Δ-*uns-cis*-$[Co(dpdmp)Cl_2]^-$ + sarcosine→

ΔR- and Λ-S-*uns-cis-mer*(1)-[Co(dpdmp)(sar)]

ΔS- and Λ-R-*uns-cis-mer*(2)-[Co(dpdmp)(sar)]

An examination of molecular model suggests that the Δ-R and Λ-S isomers could be less stable than the Δ-S and Λ-R isomers. When two bands appeared in the ion exchange column during the preparation of the sarcosine complexes, the yield from the first band fraction was much smaller than the yield from the second band fraction. Therefore, the product from the first band fraction is believed to be a mixture of Δ-R and Λ-S isomers (λ_{max}=518 nm, *uns-cis-mer*(1)) and the product from the second band fraction a mixture of Δ-S and Λ-R isomers (λ_{max}=520 nm, *uns-cis-mer*(2)). It is interesting to observe that only merridional isomers have been formed in our preparation of the *uns-cis*-[Co(dpdmp)(sar)] complexes.

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