from water, and dried at 100°. *Anal.* Calcd for $CoC_{12}H_{20}N_2O_{11}Na$: C, 32.00; H, 4.40; N, 6.22. Found: C, 31.63; H, 4.30; N, 6.30.

Preparation of $K[Co(heida)_2]\cdot H_2O$. Addition of $AgNO_3$ to an aqueous solution of $Na[Co(heida)_2]\cdot H_2O$ caused precepitation of the silver salt, $Ag[Co(heida)_2]$. The solid silver salt was collected, washed with H_2O and EtOH, and dried in air. Trituration of the silver salt with potassium iodide (KI) and extraction into water solution gave the potassium salt, which was obtained by volume reduction of the solution. *Anal.* Calcd for $CoC_{12}H_{20}N_2O_{11}K$: C, 30.90; H, 4.32. Found: C, 31.41; H, 4.21.

Preparation of $[Co(en)(heida)]\cdot H_2O$. The "blue form" of $K[Co(en)(CO_3)_2]\cdot H_2O$ was prepared from $K_3[Co(CO_3)_3]$ and $en\cdot H_2CO_3$ as described by Mori, *et al.*[13] To 100 ml of methanol was added 6 g of the "blue form" of $K[Co(en)(CO_3)_2]\cdot H_2O$ and 3.5 g of 1-hydroxyethyliminodiacetic acid (H_2heida), and the mixture was refluxed until the solids had dissolved (45 min). During the refluxing period, the color of the solution changed from blue to reddish purple. The solution was evaporated to 40 ml on a steam bath in a stream of air. A purple precipitate was filtered from the solution, washed with several portions of methanol, then with ether, and air dried; yield, 3 g, 50%. The compound is soluble in H_2O and in acidified methanol. The complex is unstable in hot H_2O, forming $[Co(en)_3]^{3+}$ and $[Co(heida)_2]^-$. *Anal.* Calcd for $CoC_8N_3H_{16}O_5\cdot H_2O$: Co, 18.94; C, 30.88; H, 5.83; N, 13.50; H_2O, 5.79. Found: Co, 18.86; C, 30.90; H, 5.61; N, 13.24; H_2O, 6.0. Water was determined by weight loss after drying for 12 hr at 110° *in vacuo*. *Anal.* Calcd for $CoC_8N_3H_{16}O_5$: C, 32.78; H, 5.50; N, 14.33. Found: C, 32.59; H, 5.69; N, 14.54.

Preparation of $[Co(Hheida)(H_2O)]^0\cdot H_2O$. A solution of 1 g of $Na[Co(heida)_2]$ in 50 ml of water was passed through a column packed with 10 g of Dowex 50W-X8 cation exchange resin in the acid form, washed through the column, and concentrated to a volume of approximately 10 ml. The purple crystals which precipitated when the solution was cooled in ice were filtered and dried at 100°. *Anal.* Calcd for $CoC_{12}H_{23}N_2O_{12}$: C, 32.29; H, 5.16; N, 6.30; Co, 13.20. Found: C, 32.30; H, 5.31; N, 6.39; Co, 13.26.

Preparation of $K[Co(CH_3COOCH_2CH_2N(CH_2COO)_2)_2]$. The acetyl derivative of $K[Co(heida)_2]$ was prepared by refluxing 50 ml of glacial acetic acid, 75 ml of acetic anhydride, and 1 g of $K[Co(heida)_2]$ for 2.5 hr. The solution turned from purple to brown. This solution was filtered to remove excess starting material and evaporated to dryness. The brown product obtained was washed with methanol three times and dried in air. It was recrystallized from water and dried at room temperature under vacuum. *Anal.* Calcd for $CoC_{16}H_{22}N_2O_{12}K$: C, 36.09; H, 4.16. Found: C, 36.25, 36.36; H, 4.07, 4.10. The compound showed carbonyl bands at 1725 (acetyl carbonyl) and 1640 cm^{-1} (coordinated carbonyl) and no hydroxyl band.

Acknowledgment. The authors are greatly indebted to Dr. James R. Barrante, for running and interpreting the nmr spectra, and to the Mobil Research and Development Co. Inc., for financial aid.

Registry No. $Na[Co(heida)_2]$, 50601-02-0; $K[Co(heida)_2]$, 50601-03-1; $[Co(en)(heida)]$, 50601-04-2; $[Co(Hheida)(heida)(H_2O)]$, 50790-63-1; $K[Co(CH_3COOCH_2CH_2N(CH_2COO)_2)_2]$, 50601-05-3.

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Contribution from the Departments of Chemistry, University of Pittsburgh, Pittsburgh, Pennsylvania 15260, and The Pennsylvania State University, McKeesport, Pennsylvania 15132

Characterization of Two Structural Isomers of a Cobalt(III) Complex with an Ethylenediamine-*N,N',N'*-triacetate-Type Ligand

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Two isomers of the complex $K[Co((R)\text{-}(-)\text{-}PD3A)NO_2]$ (PD3A = 1,2-propanediaminetriacetate) have been isolated and characterized. The red isomer has the same visible absorption spectrum as the nitro complexes formed with several N-substituted ethylenediaminetriacetate ligands, and is designated *cis equatorial*. The purple isomer has been shown by infrared, visible absorption, circular dichroism (CD), and proton magnetic resonance spectra to have *trans equatorial* geometry. The change in the position of the acetate ring attached to the secondary nitrogen in the two isomers results in significant changes in the CD and pmr spectra. The CD spectra of these complexes support previous work which indicates that the asymmetric nitrogen donor atom is the major source of optical activity in complexes of this type.

Introduction

Several cobalt(III) complexes containing pentadentate N-substituted ethylenediamine-*N,N',N'*-triacetate ligands and a nitro group in the sixth position have been prepared and studied thoroughly using circular dichroism and proton magnetic resonance spectroscopy.[1-3] Three structural isomers (Figure 1) of these compounds are possible. These isomers have been designated[3] as *equatorial* if the NO_2 group lies in the plane formed by the metal and the two other coordinated nitrogen atoms or as *polar* if the NO_2 group does not lie in this plane. Two equatorial isomers can form. For one, the two acetate groups on the tertiary nitrogen are coordinated *cis* to one another and in the other, they are coordinated *trans* to one another. Because the *polar* isomer can form only when the acetates are coordinated in *cis* positions, the three possible isomers can be designated *cis equatorial, trans equatorial,* and *cis polar*.

The X-ray structural data[4] for $[Co(EDTA)]^-$ indicated the two acetate rings planar with the ethylenediamine ring, the G rings, are more strained than the out-of-plane acetate rings, the R rings. Thus the favored isomer with an ethylenediamine-*N,N',N'*-triacetate ligand has been assumed to be the

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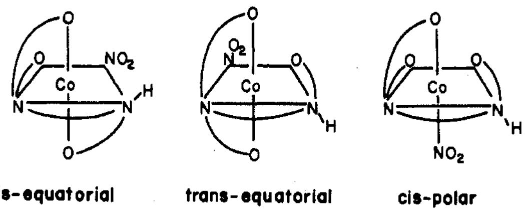

Figure 1. Structural isomers of $[Co(ED3A)NO_2]^-$.

cis equatorial isomer, which would have the same structure as $[Co(EDTA)]^-$ with one G ring opened.

The fact that tetradentate ethylenediamine-*N,N*-diacetic acid (U-EDDA or β-aminoethyliminodiacetic acid) forms a trans complex readily,[5] however, indicates that a trans isomer might be expected with pentadentate ED3A ligands also. The least favored isomer would probably be the cis polar because of the strain in the chelate system with three five-membered rings in the same plane.

For the nitro complex with Co(III) using N-substituted ED3A only one isomer is formed. When Legg, *et al.*,[6] prepared the nitro complex with unsubstituted ED3A, however, two isomers were obtained which were believed by Legg to be linkage isomers resulting in nitro ($Co-NO_2$) and nitrito (Co-ONO) complexes. The "nitrito" complex was reported to convert to the nitro complex within several days even in the solid state. The presence of bands characteristic of the coordinated nitro group in the infrared spectrum of the "nitrito" complex was attributed to this conversion of the "nitrito" to nitro.

In the present study the Co(III) complex with a nitro group and (*R*)-(−)-1,2-propanediamine-*N,N',N'*-triacetic acid was prepared. Two stable isomers were obtained which corresponded to the nitro and "nitrito" complexes obtained by Legg, *et al.*,[6] with ED3A. The stability and optical activity of the complexes with (*R*)-(−)-PD3A allowed a more complete characterization of the isomers formed, and it has been concluded that the two isomers obtained do not involve linkage isomerism of the nitro group. Instead, the so-called "nitrito" complex is really the trans equatorial nitro complex.

Experimental Section

Preparation of (*R*)-(−)-1,2-propanediamine-*N,N',N'*-triacetic Acid. Racemic 1,2-propanediamine was resolved by the method of Dwyer, *et al.*,[7] and stored as the diastereoisomer. Aqueous (*R*)-(−)-1,2-propanediamine was prepared as needed by the method of Liu, *et al.*[8] To an aqueous solution containing 0.1 mol of the active diamine in 100 ml of solution was added 36 g (0.3 mol) of sodium chloroacetate and 12 g (0.3 mol) of NaOH. The mixture was stirred until the salts had dissolved and then left standing at room temperature overnight. The solution was acidified to pH 6 with concentrated HCl and divided in half for use in preparing the complexes.

Preparation of the Cis Equatorial Isomer of Potassium Nitro((*R*)-(−)-1,2-propanediaminetriacetate)cobaltate(III) Hydrate, K-*cis-eq*-$[Co((R)\text{-}(-)\text{-}PD3A)NO_2]\cdot H_2O$. To half of the (*R*)-(−)-PD3A solution prepared as described above was added 0.2 g (0.05 mol) of cobalt(II) chloride hexahydrate in 150 ml of H_2O, 4.25 g (0.05 mol) of potassium nitrite, and 1.5 g of activated charcoal. The mixture was air-oxidized for about 60 hr and then filtered. The volume of the filtrate was decreased to about 75–100 ml and allowed to stand uncovered for 2 days. White solid which had separated was removed and the filtrate was diluted and placed on a large Dowex 1-X8 anion-exchange column (100–200 mesh, Cl form) half at a time. The elution and isolation procedures were similar to those used by Legg, *et al.*,[6] for the corresponding ED3A complex. The first material to be eluted from the column was $[Co((R)\text{-}(-)\text{-}PDTA)]^-$ followed by the brick red cis equatorial isomer of $[Co((R)\text{-}(-)\text{-}PD3A)NO_2]^-$. Two small bands, the lower purple and the upper orange, were also present after the major bands had been removed, but no material was isolated from them. The purple band was presumed to be due to the trans equatorial isomer of $[Co((R)\text{-}(-)\text{-}PD3A)NO_2]^-$, but it converted to the red cis equatorial isomer after several days on the column. *Anal.* Calcd for the cis equatorial isomer of $K[Co(PD3A)NO_2]\cdot H_2O$: C, 26.5; H, 3.70; N, 10.3. Found: C, 26.9; H, 3.71; N, 10.4; $[\alpha]_{546}$ −645° (0.07 g/100 ml in water).

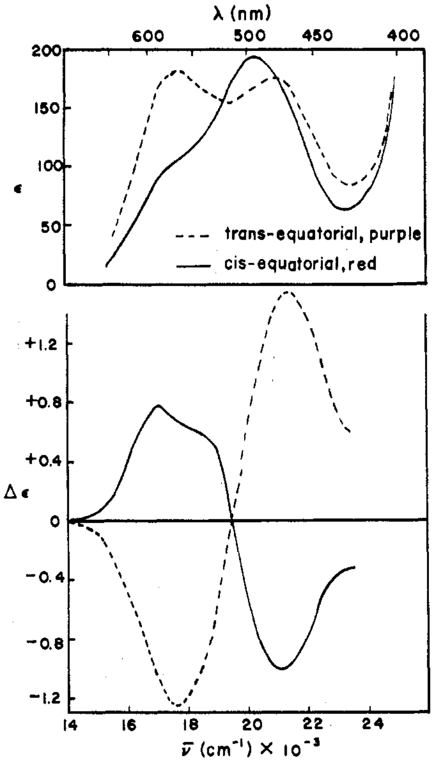

Figure 2. Absorption and circular dichroism spectra of the red, cis equatorial, and the purple, trans equatorial, isomers of $K[Co((R)\text{-}(-)\text{-}PD3A)NO_2]$.

Preparation of the Trans Equatorial Isomer of Potassium Nitro((*R*)-(−)-1,2-propanediaminetriacetato)cobaltate(III) Hydrate, K-*trans-eq*-$[Co((R)\text{-}(-)\text{-}PD3A)NO_2]\cdot H_2O$. To the other half of the (*R*)-(−)-PD3A solution was added 14.6 g (0.05 ml) of cobalt(II) nitrate hexahydrate, 5 ml of 30% H_2O_2, and 3 g of activated charcoal. The mixture was air-oxidized for 2 days and then filtered. The neutral PD3A-aquo complex was separated from charged complexes by passing a dilute solution through an anion-exchange column (Cl form) and a cation-exchange column (K^+ form) as described by Legg, *et al.*,[6] for the ED3A complex. The resulting solution was evaporated to 150 ml and 6.0 g (0.07 mol) of potassium nitrite and 28 ml of glacial acetic acid were added. The solution was left standing overnight. The next day the solution volume was decreased on the rotary evaporator until solid started to separate. The mixture was cooled in ice and a fraction of white solid removed. A little 95% ethanol was added to the filtrate. The purple trans equatorial isomer separated and was recrystallized from warm water; yield 3.5 g. This complex is stable in the solid state and in an aqueous solution (circular dichroism of solutions are unchanged after standing several weeks). It converts to the cis equatorial isomer after several days on the Dowex 1-X8 anion exchange column, however. It is unstable in acidic solutions apparently losing the NO_2 group. *Anal.* Calcd for the trans equatorial isomer of $K[Co(PD3A)NO_2]\cdot H_2O$: C, 26.5; H, 3.70; N, 10.3. Found: C, 26.78; H, 3.83; N, 10.57; $[\alpha]_{546}$ +735° (0.04 g/100 ml in water).

Physical Measurements. Absorption spectra were obtained using a Cary 14 recording spectrophotometer with a tungsten lamp. Circular dichroism spectra were recorded on a Roussel-Jouan Dichrograph with a tungsten source. Optical rotations were measured with a Rudolph spectropolarimeter with a xenon lamp. A Beckman IR-12 spectrophotometer was used to record infrared spectra of the compounds in KBr disks.

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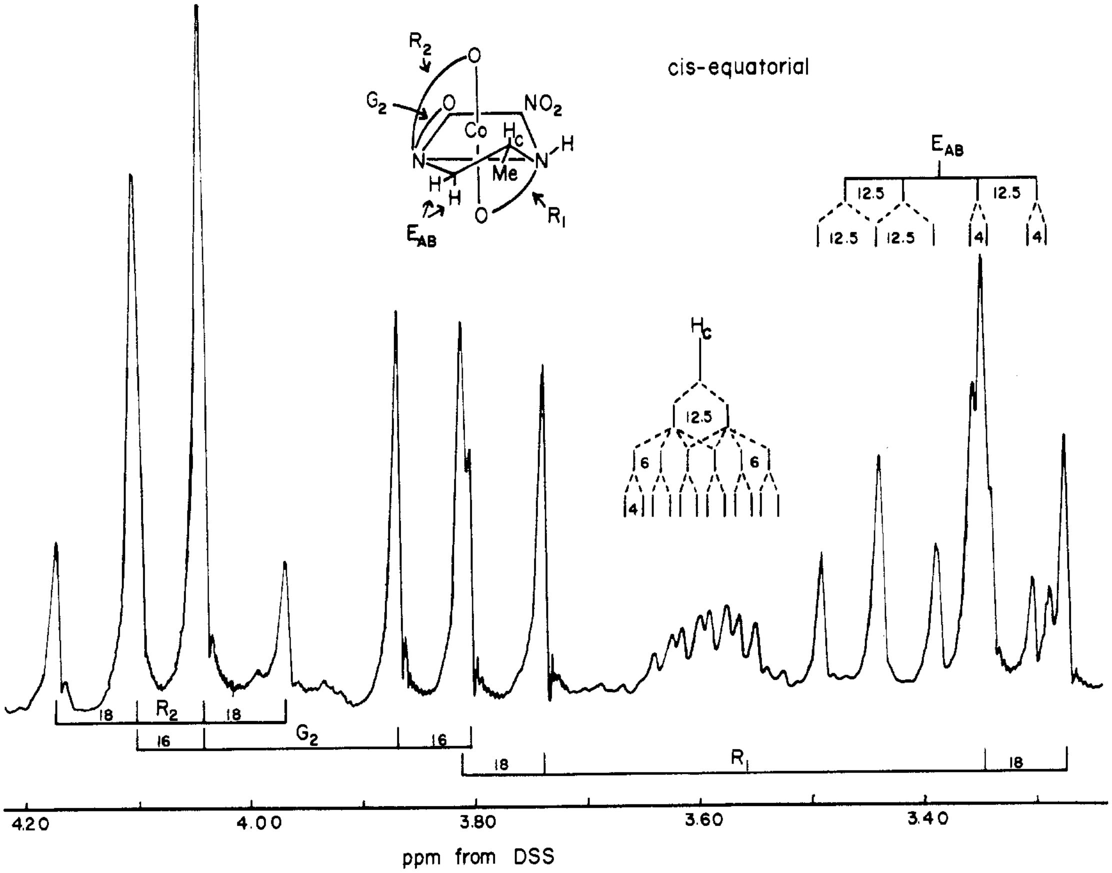

Figure 3. The 250-MHz pmr spectrum of the cis equatorial isomer of K[Co((*R*)-(–)-PD3A)NO_2] in neutral D_2O.

Proton magnetic resonance spectra were obtained with the 250-MHz nmr spectrophotometer at Carnegie-Mellon University. The field frequency ratio was stabilized by locking to water. Deuterium oxide was used as the solvent with sodium 2,2-dimethyl-2-silapentane-5-sulfonate (DSS) as an internal reference. Acidic solutions were prepared by addition of 1 drop of 1 *M* nitric acid to 1 ml of deuterium oxide before dissolving the complex.

Elemental analyses were preformed by Alfred Bernhardt, Elbach, West Germany.

Results and Discussion

The combined results of application of four spectroscopic techniques to the complexes with the formula K[Co((*R*)-(–)-PD3A)NO_2]·H_2O indicate that the compounds are both nitro complexes which vary in the geometry of the coordinated pentadentate ligand. The red complex is designated cis equatorial and is the isomer which is commonly obtained with pentadentate ligands of this type.[3] The purple isomer is designated trans equatorial and has been completely characterized for the first time in this study. The trans equatorial isomer corresponds to the complex which Legg, *et al.*,[6] believed to be a nitrito complex, [Co(ED3A)ONO].

The red isomer has infrared bands at 1410 (m), 1335 (s), and 832 cm^{-1} (w) which are considered typcial for the coordinated nitro group.[6] The purple isomer shows the same characteristic bands at 1412 (m), 1330 (s), and 820 cm^{-1} (w). Bands at 1060 and 1455 cm^{-1} are also present, but they are both too weak to support the presence of a coordinated nitrito group. Thus, the infrared spectra give no evidence of linkage isomerism of the nitro group.

The visible absorption spectra of the two isomers are shown in Figure 2. The red isomer has a shoulder at about 17,200 cm^{-1} (ϵ 93) and a maximum at 20,300 cm^{-1} (ϵ 191). The purple isomer has two low-energy maxima occurring at 17,800 cm^{-1} (ϵ 181) and 20,900 cm^{-1} (ϵ 174). The two spectra are very similar except the difference in intensities of the low-energy bands. The positions of the CD maxima also support the view that the splitting pattern is very similar for the two isomers (Figure 2). This similarity supports the assignment of trans equatorial and cis equatorial geometries to the two isomers because both of these isomers have the same microsymmetry about the metal ion. On the other hand, either a cis polar isomer or a nitrito complex would have a different effective crystal field, and in either case the crystal field splitting would be expected to be smaller.[9]

The proton nmr of the red isomer is shown in Figure 3. The subscripts 1 and 2 on the G and R indicate that the acetate ring is on a nitrogen atom with one acetate ring attached or with two acetate rings, respectively. The methyl doublet centered at 1.37 ppm coupled to H_C with $J = 6$ Hz is not shown. The spectrum is consistent with those of similar pentadentate nitro complexes[3] which have been assigned the cis equatorial geometry. In particular J_{AB} for the R-type out-of-plane rings is 18 Hz, while J_{AB} for the G-type in plane is 16 Hz, as found by Blackmer and Sudmeier for similar compounds.[3] In acidic solution (Figure 4) the low-field portion of the R_1 acetate AB pattern is split by coupling to the amine proton with $J = 8$ Hz. The high-field portion of this AB pattern and the resonance of H_C are both broadened. The observed splitting of the acetate protons by the amine proton is similar to that shown previously[3,6] for cis equatorial isomers and would be expected from consideration of the dihedral angles involved.[10] The broadening of the H_C resonance in acidic solution indicates that it and thus the methyl group are on the carbon adjacent to the secondary nitrogen.

The other two protons on the diamine ring give rise to the pattern E_{AB}. They are both coupled to H_C which should

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(9) H. Yamatera, *Bull. Chem. Soc. Jap.*, 31, 101 (1958).
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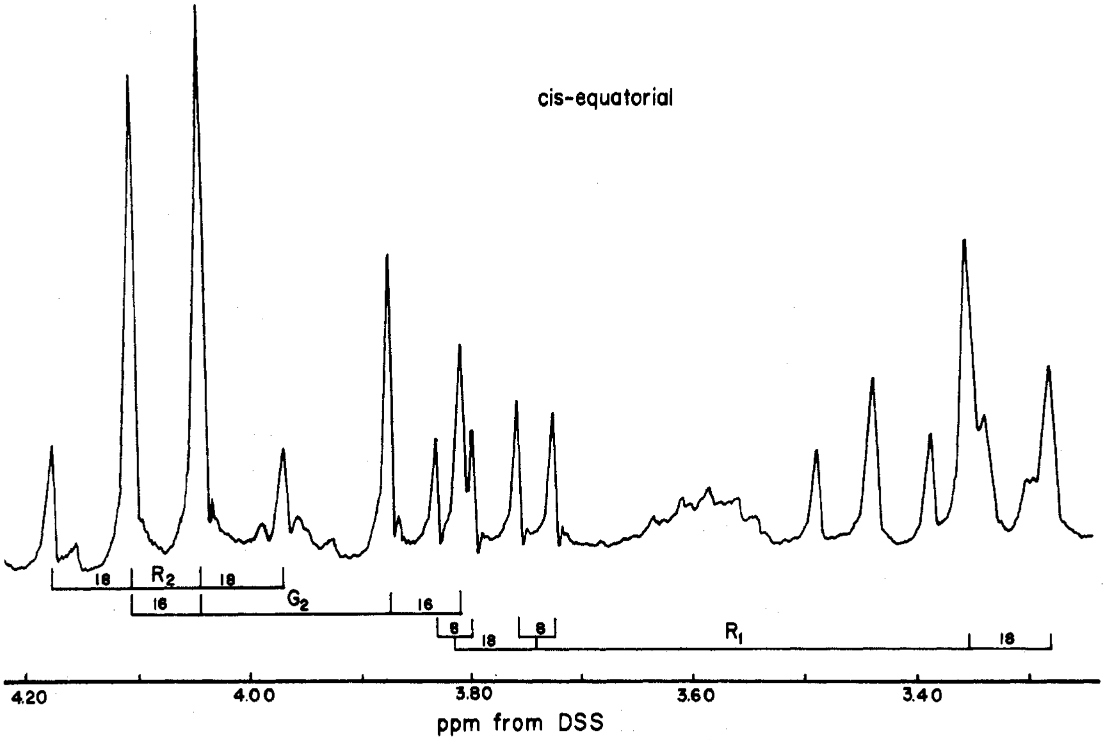

Figure 4. The 250-MHz pmr spectrum of cis equatorial [Co((*R*)-(−)-PD3A)NO_2] in dilute HNO_3 in D_2O.

be in an axial position due to the preference of the bulkier methyl group for the equatorial position. The low-field resonance of the E_{AB} pattern should be due to the other axial proton on the diamine ring and the high-field resonance to the equatorial proton,[11] coupled to the axial proton H_C with $J =$ 12.5 and 4 Hz, respectively.

The proton nmr of the purple isomer is shown in Figure 5. The methyl doublet centered at 1.44 ppm and coupled to H_C with $J = 6$ Hz is not shown. The diamine backbone should have the same geometry as in the red isomer. The coupling of H_C to the E_{AB} protons is similar to the red isomer, but the proton to which it is coupled more strongly ($J = 13$ Hz), presumably the axial proton, is now on the high-field side of the equatorial proton for which $J = 4$ Hz. Also the AB patterns for the three acetate rings are very different from those for the red isomer and other nitro compounds with the cis equatorial geometry (Table I).

In acidic solution (Figure 6) all four lines of the AB pattern labeled G_1 are split into doublets. The low-field proton shows $J = 6$ Hz, while $J = 10$ Hz for the high-field proton. The splitting of both acetate protons by the amine proton[3] and the small chemical shift difference between the two protons[6] indicate that the acetate ring on the secondary nitrogen in this isomer is a G-type in-plane ring. The AB coupling constant of 16 Hz is the expected value for a G ring.[3]

Because the visible absorption spectrum indicates that the nitro group is in an equatorial in-plane position, the other two acetate rings are necessarily both of the R type. The overall geometry of the purple isomer is thus trans equatorial. Not surprisingly, the R-type rings in this geometry show different AB patterns than R-type rings in the cis equatorial geometry. Also the repositioning of the G_2 ring of the cis equatorial isomer to an R_2 ring in the trans equatorial isomer might be expected to influence the chemical shift of the axial proton on the adjacent backbone carbon atom. As noted above, this proton has a chemical shift difference of about 0.4 ppm between the red and purple isomers.

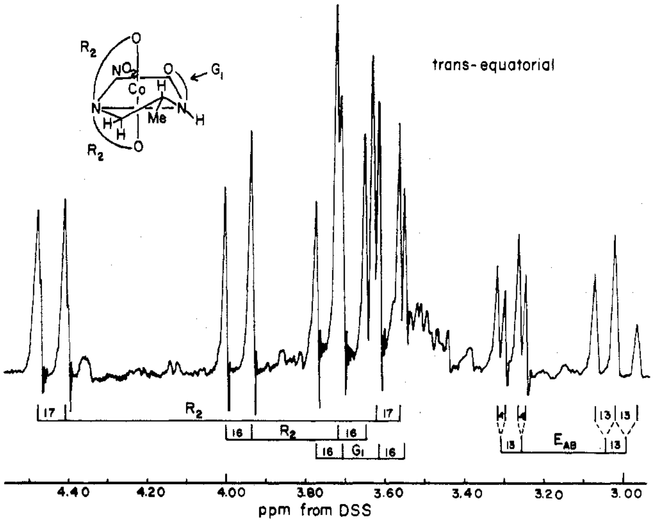

Figure 5. The 250-MHz pmr spectrum of the trans equatorial isomer of K[Co((*R*)-(−)-PD3A)NO_2] in neutral D_2O.

The CD spectra of the two isomers are shown in Figure 2. The preference of the methyl group in (*R*)-(−)-PD3A for an equatorial position results in stereospecific coordination with cobalt[12] to give complexes with the structures shown. Previous work[2] with ethylenediaminetriacetate-type ligands indicates that the asymmetric nitrogen is the major source of asymmetry in these complexes. Because the asymmetric nitrogen in the cis equatorial isomer is necessarily in an *R* configuration[13] and that in the trans equatorial is necessarily in an *S* configuration, one would expect the two CD curves to have opposite signs. The curves are not exact mirror

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Table I. Chemical Shifts[a] for Acetate Protons in Cobalt(III) Complexes with ED3A-Type Ligands

	Type of ring		
Complex	R_1 (18 Hz)[b]	R_2 (18 Hz)	G_2 (16 Hz)
$[Co(ED3A)NO_2]^-$ [c]	4.15 3.10 (1.05)[d]	4.05	4.08 3.87 (0.21)
$[Co(DMP3A)NO_2]^-$ [e]	3.63 3.27 (0.36)	4.31 4.02 (0.29)	4.14 3.89 (0.25)
$[Co((R)\text{-}(-)\text{-}PD3A)NO_2]^-$ red isomer	3.77 3.31 (0.46)	4.13 4.01 (0.11)	4.07 3.85 (0.22)

	Type of ring		
Complex	G_1 (16 Hz)	R_2 (16 Hz)	R_2 (17 Hz)
$[Co((R)\text{-}(-)\text{-}PD3A)NO_2]^-$ purple isomer	3.74 3.79 (0.15)	3.97 3.69 (0.28)	4.45 3.59 (0.86)

[a] Values in ppm from DSS. [b] $|J|_{AB}$. [c] ED3A = ethylenediaminetriacetate.[3] [d] Difference in chemical shift values for the two protons, δ_{AB}, in ppm. [e] DMP3A = 1,2-diamino-2-methylpropanetriacetate.[3]

images, however, because the diamine backbone and the asymmetric carbon make the same contribution to both. Furthermore the complexes contain different arrangements of acetate rings which might contribute slightly to the optical activity.

The CD curves for these complexes provided the first evidence that they were not linkage isomers of the nitro group. The curves are readily explained by the opposite configurations of the asymmetric nitrogens resulting from the two different arrangements of the coordinated pentadentate ligand. The infrared, visible absorption, and especially the proton magnetic resonance spectral data all support this interpretation of the structure of the two complexes. This is the first time that a stable trans equatorial isomer of an ethylenediamine-*N,N',N'*-triacetate type ligand has been isolated and characterized. It is possible that this isomer does not form with N-substituted ED3A-type ligands because of interaction between the N-substituent and the group in the adjacent out-of-plane position. Furthermore, although the trans equatorial isomer is formed with unsubstituted ED3A, it readily converts to the cis equatorial form.[6] This conversion to the cis equatorial from the trans equatorial form may require a flip in the conformation of the puckered backbone ring which is hindered in (*R*)-(–)-PD3A by the preference of the methyl group for an equatorial position. In general for N-substituted ED3A ligands or for ED3A-type ligands without bulky backbone groups, one would expect to obtain cis equatorial as the only stable isomer.

The mechanism for interconversion of the two isomers could be tested by resolving the trans equatorial isomer of $[Co(ED3A)NO_2]^-$. If this isomer converts to the cis equatorial by a C_3-twist mechanism with a simultaneous flip in the conformation of the backbone ring, the configuration of the asymmetric nitrogen would be unchanged. Thus no change in the sign of the CD bands would accompany the conversion. On the other hand, if the conversion involves breakage of the N-H bond and inversion of the configuration of the asymmetric nitrogen, it would be accompanied by a change in the sign of the CD bands.

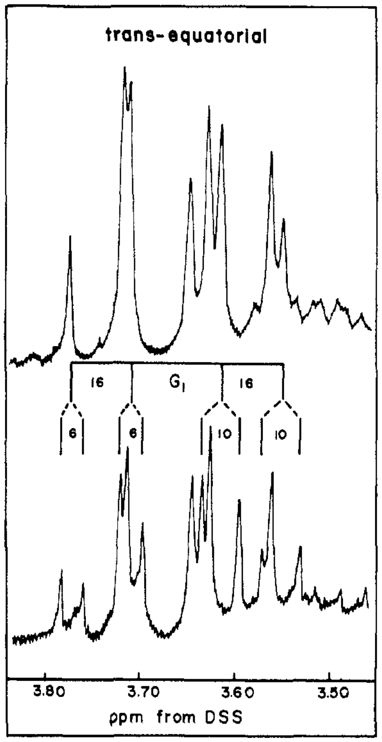

Figure 6. A portion of the 250-MHz pmr spectrum of trans equatorial $[Co((R)\text{-}(-)\text{-}PD3A)NO_2]^-$ in neutral D_2O (top) and in dilute HNO_3 in D_2O (bottom).

Acknowledgment. Financial support for this research was provided by the University Science Development Program (GU 3184) to C. W. M. and The Pennsylvania State University to C. M. The authors thank Mr. B. Aufderheide for his assistance in obtaining the 250-MHz pmr spectra and Dr. P. Krause for help with the ir spectra.

Registry No. K-*cis-eq*-$[Co((R)\text{-}(-)\text{-}PD3A)NO_2]$, 50790-33-5; K-*trans-eq*-$[Co((R)\text{-}(-)\text{-}PD3A)NO_2]$, 50790-34-6; (*R*)-(–)-PD3A, 50790-35-7; (*R*)-(–)-1,2-propanediamine, 6852-78-4; sodium chloroacetate, 3926-62-3.