# Stereoselectivity in Octahedral Complexes. III.[1]
Cobalt(III) Complexes of Nitrogen-Substituted 5-Nitrosalicylaldimines

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Tris(N-R-5-nitrosalicylaldiminato)cobalt(III) complexes have been prepared where R is (S)- or (R)-α-benzylethyl and (R)-α-phenylethyl. Two diastereomers are isolated for each complex by thin layer chromatography of the reaction products. Pmr and CD spectra show these to be the Δ-trans and Λ-trans diastereomers, although no attempt is made to assign configurations to the diastereomers. The ratio of diastereomers in the reaction product is found to be 0.35 for R = α-benzylethyl and 0.48 for R = α-phenylethyl. No isomerization occurs when the separated diastereomers are refluxed in CH_2Cl_2 unless activated charcoal is present. Isomerization occurs in the presence of charcoal, resulting in the predominance of one of the isomers. For ligands of related absolute configuration, the more abundant complexes have very similar CD spectra and presumably have related absolute configurations. The observed stereoselectivity in these complexes is apparently kinetically controlled.

Tris(N-R-salicylaldiminato)cobalt(III) complexes (I) where R is an aryl or n-alkyl substituent have been known for some time.[2-4] Many past attempts to prepare complexes I having an α-*branched* alkyl R substituent by oxidation of the corresponding bis Co(II) complex in the presence of excess ligand have failed, although the Co(II) complexes with n-alkyl R groups are readily oxidized by air to the tris complexes.[5-8] Failure to obtain the tris complexes has been attributed to greater steric strain expected for the octahedral tris complex relative to the tetrahedral bis complex when

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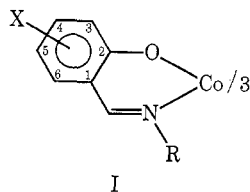
α-branched R substituents are present.[6,8] Recently, however, Yamada, *et al.*,[9] succeeded in preparing tris-(N-isopropyl-5-nitrosalicylaldiminato)cobalt(III) and tris(N-isopropyl-5-bromosalicylaldiminato)cobalt(III) by oxidation of the corresponding bis complexes. Apparently the presence of nitro or bromo substituents at the 5 position of the salicylaldimine ligand stabilizes the Co(III) complex and prevents ready reduction to Co(II), which is often observed even for complexes with n-alkyl substituents.[7]

These observations suggested that complexes I where

R is an α-branched *asymmetric* group would exist when a 5-nitro substituent is present. It was anticipated that the diastereomers of such complexes would be formed stereoselectively as a result of the proximity of asymmetric centers in the tris complex. The present paper reports the preparation and stereochemical investigation of complexes I where X = 5-NO_2 and R = (+)- and (−)-α-benzylethyl and (+)-α-phenylethyl.

The absolute configurations of α-benzylethylamine and α-phenylethylamine have been established by chemical interconversions,[10,11] and that of α-phenylethylamine has been confirmed recently by X-ray studies.[12] The R configuration[13] is found for (+)-α-phenylethylamine, whereas (+)-α-benzylethylamine has the S absolute configuration. Smith, *et al.*,[14] prepared the Schiff bases of these amines with 5-nitrosalicylaldehyde and other salicylaldehyde derivatives. Using amines having the S configuration, they found the Schiff bases to display plain, positive ORD curves in the 450–600-mμ region. These findings were corroborated by Terentev, *et al.*,[15] who examined the Schiff bases of several substituted salicylaldehydes with (−)-α-phenylethylamine and also prepared the Ni(II), Co(II), and Cu(II) complexes.

For all known complexes having structure I only the trans isomers are found. West[7] explained this on steric grounds, pointing out that in the cis isomer there would be severe steric strain due to the presence of three R groups on the same octahedral face. The exclusive, or at least predominant, occurrence of the trans isomers for complexes I having n-alkyl R groups has been demonstrated by dipole moment[16] and nmr studies.[17] More recently the existence of only trans isomers for a number

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(17) A. Chakravorty and R. H. Holm, *ibid.*, **3**, 1521 (1964).

TABLE I

CHARACTERIZATION OF THE INDIVIDUAL DIASTEREOMERS

Complex[a]	% calcd			% found			δ (J)[b]
	C	H	N	C	H	N	
Co((S)-BzEt-5-NO$_2$sal)$_3$-A	63.4	5.0	9.2	63.6	4.9	9.0	1.32 (7.0), 1.48 (6.7), 1.48 (6.7)
Co((S)-BzEt-5-NO$_2$sal)$_3$-B				63.0	4.8	9.0	1.08 (6.4), 1.15 (6.7), 1.27 (7.2)
Co((R)-BzEt-5-NO$_2$sal)$_3$-A'				63.2	5.2	8.9	1.32 (7.0), 1.48 (6.7), 1.48 (6.7)
Co((R)-BzEt-5-NO$_2$sal)$_3$-B'				63.6	5.1	8.9	1.08 (6.4), 1.15 (6.7), 1.27 (7.2)
Co((R)-PhEt-5-NO$_2$sal)$_3$-C	62.4	4.5	9.7	62.2	4.7	9.6	1.78 (7.0), 1.87 (7.4), 2.02 (7.3)
Co((R)-PhEt-5-NO$_2$sal)$_3$-D				62.9	4.6	9.4	1.20 (7.1), 1.67 (7.0), 1.70 (6.9)

[a] See text for nomenclature. [b] δ is the chemical shift in CH$_2$Cl$_2$ solution relative to TMS, in ppm; J is the coupling constant, in Hz ± 0.5 Hz.

of tris Co(III) complexes of N-alkyl-2-hydroxyaceto-phenimine and N-alkyl-2-hydroxypropiophenimine has been shown by nmr studies.[3,4]

Experimental Section

Preparation of Ligands.—5-Nitrosalicylaldehyde was prepared by treating salicylaldehyde with fuming HNO$_3$ in glacial acetic acid according to the procedure given by Miller.[18] Yields were small in all cases; the purified product melted in the range 123–126° (lit.[18] mp 125–126°). The N-substituted 5-nitrosalicylaldimine ligands were obtained by refluxing 5-nitrosalicylaldehyde and the appropriate amine in ethanol for about 1 hr. The solid products were recrystallized from ethanol and characterized by their pmr spectra. Melting points of purified ligands were 89–91° for N-α-benzylethyl-5-nitrosalicylaldimine (lit.[14] mp 95–97°) and 100–101° for N-α-phenylethyl-5-nitrosalicylaldimine (lit.[14] mp 102–103°). (+)- and (−)-α-benzylethylamine and (+)-α-phenylethylamine were obtained from Aldrich Chemical Co. and used without further purification. For the neat liquids, in the above order, values of [α]^{25}D were found to be +31.7, −32.8, and +36.1°. Reported values are +34.1° (at 25°) and +39.9° (at 23°) for (+)-α-benzylethylamine and (+)-α-phenylethylamine, respectively.[14]

Preparation of Complexes.—Bis(N-substituted 5-nitrosalicylaldiminato)cobalt(II) complexes were prepared by the method of Nishikawa, et al.[5] These products were used as starting material for preparation of the tris Co(III) complexes. The procedure used is a modification of that recently reported by Yamada, et al.[9] In a typical preparation 3.24 g (5.17 mmol) of bis(N-α-benzylethyl-5-nitrosalicylaldiminato)cobalt(II) and 1.47 g (5.17 mmol) of free ligand in chloroform solution were treated dropwise with 3 ml (large molar excess) of 30% H$_2$O$_2$. The mixture was allowed to reflux for 1 hr, during which time the color changed from red-orange to green. The green product was then precipitated by adding n-heptane and was recrystallized from chloroform and n-heptane.

Thin Layer Chromatography.—Initial experiments using microscope slides coated with silica gel demonstrated that the Δ-trans and Λ-trans diastereomers could be separated for all complexes studied. The isomers of tris(N-α-benzylethyl-5-nitrosalicylaldiminato)cobalt(III) are best separated by eluting first with chloroform and then developing several times with a 1:4 toluene–chloroform solvent mixture. A solvent mixture of 1:3 toluene–dichloromethane effects separation of the isomers of tris-(N-(R)-α-phenylethyl-5-nitrosalicylaldiminato)cobalt(III). Preparative-scale separations were carried out using precoated 20 × 20 cm plates with a 2-mm layer of silica gel F-254, obtained from Brinkmann Instruments, Inc. The two principal green bands were 2–3 cm wide and separated by 5–10 mm at the end of several developments. Samples of the isomers were obtained by removing the chromatographic bands from the plates and extracting the mixture of silica gel and complex with acetone. The acetone solutions were filtered; then the acetone was removed by means of a rotary evaporator. The residue was taken up in a small quantity of chloroform and recrystallized by adding

n-heptane to the chloroform solution. The samples were then dried in vacuo. Generally about 50 mg of one diastereomer and more than 100 mg of the other could be obtained from a single chromatographic plate, beginning with 200–300 mg of isomeric mixture. The results of elemental analyses for the individual diastereomers are given in Table I. These analyses were carried out using a Model 185 F & M C, H, and N analyzer. Results for nitrogen were always low.

Stereoselectivity Measurements.—The relative abundance of Λ-trans and Δ-trans diastereomers for each complex were obtained by the following gravimetric method. The separated bands were removed as completely as possible from the preparative-layer plates, and the mixtures of silica gel and sample were extracted with acetone and precipitated as described above. The precipitates were collected by filtration and dried in vacuo. Several chromatographic plates for each complex were treated in this manner. The samples from each band were combined and weighed. Results of several determinations are consistent, and the isomer ratios are believed to be accurate within a few per cent.

Isomerization Experiments.—Attempts to determine the conditions required for isomerization of the individual diastereomers of tris(N-R-benzylethyl-5-nitrosalicylaldiminato)cobalt(III) were made in the following manner. One-tenth gram of either isomer in 25 ml of dichloromethane was refluxed for several days. In some experiments activated charcoal was added to the solution. At the end of this time the solutions were filtered and concentrated by evaporation to a volume of about 1 ml. The diastereomers present in the dichloromethane solution could then be identified by pmr, since their resonance signals are not entirely coincident (see Table I). Whenever activated charcoal was added to the solutions, some decomposition or adsorption of the complex occurred, and in several instances it was necessary to record time-averaged pmr spectra due to the low concentration of complex.

Physical Measurements.—Optical rotations at 589 mμ were measured on a Perkin-Elmer Model 141 automatic polarimeter. Circular dichroism and absorption spectra were recorded using a Cary Model 60 spectropolarimeter and a Model 14 spectrophotometer, respectively. Nuclear magnetic resonance spectra were obtained on Varian Models A-60 and HA-100 spectrometers. A Varian Model C-1024 time-average computer was used with the HA-100 instrument when necessary.

Results and Discussion

The four diastereomers possible for tris-bidentate complexes such as I having inequivalent donor atoms and chiral ligands are Δ-cis, Δ-trans, Λ-cis, and Λ-trans.[19] Earlier work has demonstrated that for Co(III) complexes of certain chiral α-amino acids[20] and β-ketoenolates,[1b] these four diastereomers may be separated by

(18) W. v. Miller, Ber., **20**, 1927 (1887).

(19) The symbols Δ and Λ, denoting right- and left-hand helicity about the C_3 or pseudo-C_3 axis, were first proposed by T. S. Piper, J. Amer. Chem. Soc., **83**, 3908 (1961), and more recently recommended by the IUPAC, Inorg. Chem., **9**, 1 (1970). Cis and trans isomers belong to C_3 and C_1 point groups, respectively.

fractional crystallization,[20] column chromatography,[20] or thin layer chromatography[1b] and identified by their pmr and CD spectra. In order to assign Δ and Λ configurations to tris-bidentate complexes of Co(III) from CD spectra, the $^1A_1 \rightarrow {}^1E_a$ and $^1A_1 \rightarrow {}^1A_2$ transitions (assuming D_3 symmetry) must be identified.[21] The relative energies of these transitions seem to be well established for complexes of 1,2-diamines. However, assignments of Δ and Λ configurations to the β-ketoenolate complexes are tentative until these transitions can be identified or until the absolute configuration of at least one complex has been determined by X-ray techniques. This is also true of salicylaldimine complexes, and accordingly no attempt is made in this paper to assign absolute configurations to complexes I.

Tris(N-α-benzylethyl-5-nitrosalicylaldiminato)cobalt-(III).—Preparative-layer chromatography of the complex having R = (S)-α-benzylethyl results in two intense green bands, and the corresponding samples may be extracted from the sorbent and crystallized. These diastereomers are designated hereafter as Co((S)-BzEt-5-NO₂sal)₃-A and Co((S)-BzEt-5-NO₂sal)₃-B (or simply A and B) in order of increasing R_f value. Results of elemental analyses for these separated isomers are given in Table I.

The pmr spectrum of each isomer shows three resonances for the protons of the methyl group bound to the asymmetric carbon; each resonance is further split by spin coupling with the single proton on the asymmetric carbon. Spectra recorded at 100 MHz are shown in Figure 1. It is apparent that some signals are coinci-
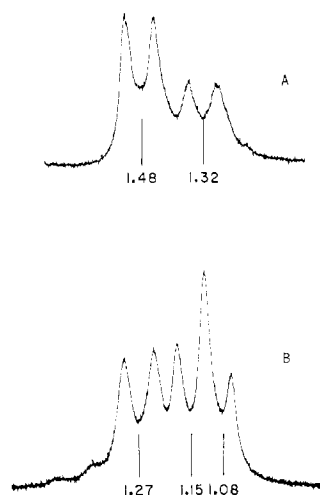

Figure 1.—Proton resonance spectra in the methyl region of diastereomers at 100 MHz: A, Co((S)-BzEt-5-N-O₂sal)₃-A or Co((R)-BzEt-5-NO₂sal)₃-A'; B, Co((S)-BzEt-5-NO₂sal)₃-B or Co((R)-BzEt-5-NO₂sal)₃-B'.

dent at 100 MHz; others overlap in the 60-MHz spectrum. The chemical shift values and coupling constants given in Table I were determined by comparing

(20) (a) J. H. Dunlop and R. D. Gillard, *J. Chem. Soc.*, 6531 (1965); (b) B. E. Douglas and S. Yamada, *Inorg. Chem.*, **4**, 1561 (1965); (c) R. G. Denning and T. S. Piper, *ibid.*, **5**, 1056 (1966); (d) E. Larsen and S. F. Mason, *J. Chem. Soc. A*, 313 (1966); (e) R. D. Gillard and N. C. Payne, *ibid.*, 1197 (1969).

(21) A. J. McCaffery, S. F. Mason, and R. E. Ballard, *ibid.*, 2883 (1965).

spectra at 60 and 100 MHz. The occurrence of three equally intense methyl proton signals clearly demonstrates that both isomers have trans geometry (C_1 point group).

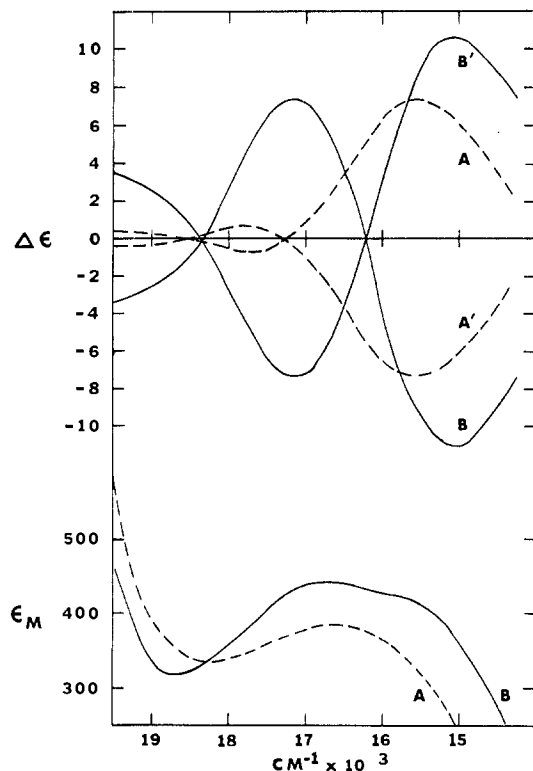Absorption and CD spectra in the visible region for both diastereomers are shown in Figure 2. The ex-

Figure 2.—Absorption and circular dichroism spectra of tris(N-α-benzylethyl-5-nitrosalicylaldiminato)cobalt(III) diastereomers in dichloromethane solution. Letters in the figure correspond to those in Table I.

pected splitting of the $^1A_{1g} \rightarrow {}^1T_{1g}$ (O_h) transition as a result of lowering the symmetry to C_1 is more evident in the absorption spectrum of isomer B than in that of isomer A. Circular dichroism spectra of the two isomers differ markedly in the region of this transition. Co((S)-BzEt-5-NO₂sal)₃-B displays a negative Cotton effect followed by a positive Cotton effect as one proceeds toward higher energy, whereas isomer A shows a dominant positive Cotton effect in the low-energy region. These results indicate the two isomers have opposite helicities when viewed down the pseudo-C_3 axis; one is Δ-trans, and the other is Λ-trans. The CD curves are not exactly enantiomeric since A and B are diastereomers rather than enantiomers.

Chromatography of the complex having the (R)-α-benzylethyl substituent also produces two intense green bands, and pmr spectra of the samples isolated are identical with those presented in Figure 1. These diastereomers are designated Co((R)-BzEt-5-NO₂sal)₃-A' and Co((R)-BzEt-5-NO₂sal)₃-B' (or A' and B') in order of increasing R_f values on the thin layer plates. Sample B' exhibits a CD spectrum which is enantiomeric in all respects with that of Co((S)-BzEt-5-NO₂sal)₃-B. This

demonstrates that the two complexes are true enantiomers, differing in configuration about Co as well as about the three ligand asymmetric centers. For the same reason the CD curves of complexes A and A' are enantiomeric.

The faster moving chromatographic band for each complex contains considerably more sample than the band with smaller R_f value. An estimate of the relative abundances of the two trans isomers in each case was made by weighing samples obtained from several chromatographic plates (see Experimental Section). The ratios found are A/B = 0.34 and A'/B' = 0.36. In the absence of stereoselective effects the Δ-trans and Λ-trans isomers are expected to be present in equal amounts. Clearly there is an appreciable stereoselective effect which is either thermodynamic or kinetic in origin. In labile complexes stereoselectivity is thermodynamically controlled, and the relative abundance of a given isomer depends on its relative free energy, since a rapid equilibrium presumably exists among the diastereomers. Inert complexes, on the other hand, retain their initial configurations, and the relative abundances of the diastereomers depend upon their relative rates of formation.

The diastereomers A, B, A', and B' are obviously inert under the conditions used to separate them. Furthermore it was demonstrated by pmr (see Experimental Section) that refluxing samples of A' or B' in dichloromethane for at least 3 days in the absence of activated charcoal does not cause detectable isomerization. In the presence of charcoal, however, isomerization does occur. If *either* isomer A' or B' is refluxed in dichloromethane in the presence of activated charcoal, the pmr spectrum of the resulting solution shows the pattern of resonances attributable to isomer B' in addition to two signals further downfield which are due to free ligand. Addition of a small amount of A' to the pmr solution results in the appearance of new signals at the frequencies expected for A'. Assuming an equilibrium is set up between the diastereomers under these conditions,[22] it must be concluded that the equilibrium is strongly displaced to one side, favoring isomer B'. Thus isomer B' must be the thermodynamically more stable isomer. Since both isomers are formed in appreciable amounts during preparation of the complex, it appears that equilibrium conditions are not maintained and that the observed stereoselectivity is kinetically controlled.

In addition to the two intense chromatographic bands discussed above for these complexes, several faint bands are observed. Samples of these compounds were obtained from several plates in order to ascertain whether any could be the cis isomers. Elemental analyses of two of the compounds gave values close to those calculated for $Co((R)$- or (S)-BzEt-5-NO$_2$sal)$_3$. Pmr spectra, however, show three doublets as expected for trans isomers. It seems likely that these two are "mixed-ligand" complexes having two S and one R ligands, or *vice versa*, resulting from a ~4–7% optical impurity in

(22) See ref 20e for a discussion of charcoal equilibrations.

the amines used to prepare the ligands (see Experimental Section). These mixed-ligand complexes would be formed in small proportions relative to the "pure" ligand species, and each could exist in Δ and Λ configurations. CD and absorption spectra were recorded on samples from the chromatographic plates of $Co((R)$-BzEt-5-NO$_2$sal)$_3$. Both samples showed absorption maxima at *ca.* 15,800 cm^{-1}. In the region of this absorption one complex had a positive CD; the other showed a negative CD.

Tris(N-(R)-α-phenylethyl-5-nitrosalicylaldiminato)-cobalt(III).—Two intense green bands separate on the thin layer chromatograms of this complex. The sample obtained from the faster moving band, designated hereafter as $Co((R)$-PhEt-5-NO$_2$sal)$_3$-D, exhibits a positive and then a negative Cotton effect on going toward higher energy in the region of the ligand field transition (see Figure 3). The other sample, $Co((R)$-
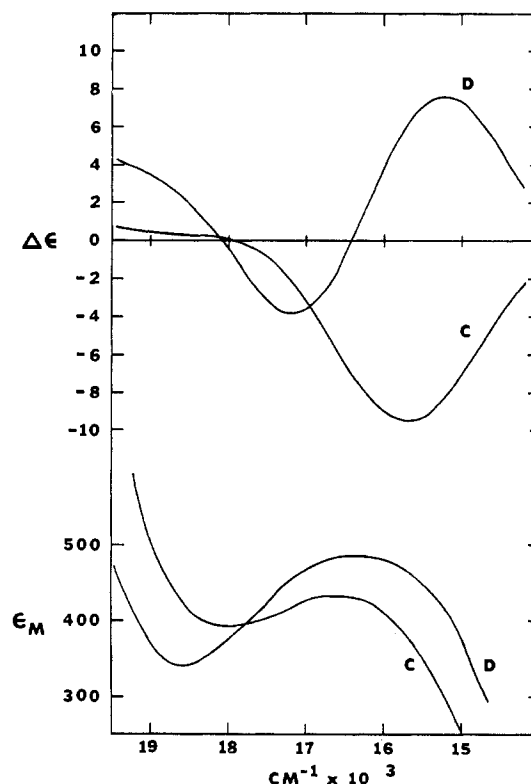

Figure 3.—Absorption and circular dichroism spectra of tris(N-α-phenylethyl-5-nitrosalicylaldiminato)cobalt(III) diastereomers in dichloromethane solution. Letters in the figure correspond to those in Table I.

PhEt-5-NO$_2$sal)$_3$-C, shows a dominant negative CD in this region. Pmr spectra of both isomers in the region of the methyl resonance show the pattern of three doublets expected for trans diastereomers (see Table I). It is clear that these are the Δ-trans and Λ-trans diastereomers. In addition to these isomers, a very small amount of another green complex was obtained from the chromatograms. This complex gave satisfactory elemental analyses, but its pmr spectrum shows a multiplicity of signals in the methyl region, thus excluding the cis structure. The sample is probably a mixed-

ligand complex, as discussed above, having trans geometry.

$Co((R)$-PhEt-5-NO$_2$sal)$_3$-D is the more abundant diastereomer. The ratio of abundances of C and D obtained from two independent chromatograms agrees well; the average ratio is C/D = 0.48. Diastereomers C and D fail to isomerize upon refluxing in dichloromethane in the absence of activated charcoal. Also the pmr spectrum of a solution of isomer D shows no change after refluxing for 3 days in the presence of charcoal. However, under identical conditions isomer C is converted over to isomer D, indicating that D is the thermodynamically more stable isomer. Thus the ratio of diastereomers obtained during synthesis must be determined largely by kinetic factors.

Intramolecular Steric Interactions.—Results of isomerization experiments on $Co((R)$-BzEt-5-NO$_2$sal)$_3$ indicate that isomer B' is thermodynamically more stable than isomer A'. Space-filling molecular models were constructed for the two isomers in hopes that they would reveal differences in intramolecular steric interactions between the Δ and Λ forms of the complex, which would explain qualitatively why one isomer should be favored. It was assumed that such interactions would involve the two N substituents on the same triangular face of the octahedron. Evaluation of the steric interactions is complicated by the number of conformations possible resulting from rotation about the C–N and C–C bonds; also slight distortions of the chelate rings significantly alter the interactions. Thus the differences in the Δ and Λ configurations as far as bond strain (enthalpy) and R-group rotational freedom (entropy) are concerned are difficult to assess from models. Similar difficulties are encountered with models of Δ- and Λ-$Co((R)$-PhEt-5-NO$_2$sal)$_3$; however it appears that the intramolecular steric interactions are more severe in the Δ diastereomer. This would imply that isomer D has the Λ configuration.

Since (R)-$(+)$-α-phenylethylamine and (R)-$(-)$-α-benzylethylamine have related absolute configurations, the complexes having these R substituents should experience similar intramolecular steric interactions both during and after formation. Thus regardless of whether stereoselectivity is kinetically or thermodynamically controlled, the more abundant isomers should have the same helicity. With this in mind, it is significant to note that the CD spectra of $Co((R)$-PhEt-5-NO$_2$sal)$_3$-D and $Co((R)$-BzEt-5-NO$_2$sal)$_3$-B' are very similar. The above results indicate these isomers are favored over C and A' both kinetically and thermodynamically. CD spectra of the lesser abundant isomers, C and A', are also very similar.

Finally, some comment should be made regarding the stability of these tris Co(III) complexes having α-branched R groups. If the observed tendency[7] of Co(III) in salicylaldimine complexes to be reduced to Co(II) involves oxidation of the ligand, this tendency is expected to be less where a 5-nitro substituent is present, due to the electron-withdrawing power of the substituent. The effect is illustrated by resonance hybrids II and III. Other electronegative substituents at the
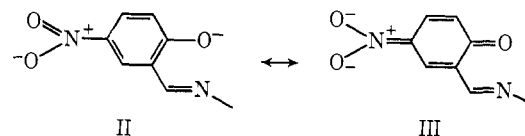
5 position may also be expected to stabilize Co(III) complexes of salicylaldimine ligands. Thus tris(N-isopropyl-5-bromosalicylaldiminato)cobalt(III) has been prepared.[9]