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Preparative Study on Chiral Orthotelluratobis(diamine)cobalt(III) Complexes and Their Condensation Products

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Five chiral cobalt(III) complexes coordinating an orthotellurate or a ditellurate ligand have been prepared and their optically active isomers isolated. They belong to one of the three following types: first a "mononuclear" $[Co(TeO_6H_4)(diamine)_2]^+$ type (diamine=ethylenediamine (en) and (*R*)-(−)-1,2-propanediamine); second a "dinuclear" $[Co(diamine)_2(Te_2O_{10}H_4)Co(diamine)_2]^{2+}$ one (diamine=en and (*R*,*R*)-(−)-*trans*-1,2-cyclohexanediamine); and lastly a "tetranuclear" $[\{Co(diamine)_2\}_2(Te_2O_{10})\{Co(diamine)_2\}_2]^{4+}$ one (diamine=en). The structures of these complexes have been discussed from the visible to ultraviolet absorption and circular dichroism spectra.

Orthotellurate anion $[TeO_6H_{6-n}]^{n-}$ is capable of coordinating with a cobalt(III) center as a bidentate ligand, as was exemplified by the isolation of $K_3[Co(TeO_6H_3)_2(H_2O)_2]$.[1] Also the anion has a tendency to condense easily in solution. One of the condensation products, tetrahydrogenditellurate anion, $Te_2O_{10}H_4^{4-}$, was proved to occur in its potassium salt, in which each tellurium atom is surrounded by six oxygen atoms in an octahedral arrangement, two such octahedra having one edge in common.[2] Thus the anion is a promising ligand for the synthesis of new polynuclear compounds of a hybrid type between metal chelate and heteropoly oxoanion, such as $H_3[Co_4I_3O_{18}(en)_3]\cdot 5H_2O$[3] and $Na_5[Co(Nb_6O_{19})(dien)]$[4]. That is, the mononuclear cobalt(III) complex with the orthotellurate ligand will be able to polymerize into some polynuclear complexes by the dehydration condensation of the coordinated orthotellurate moiety. The present study is concerned with the preparation of such new polynuclear telluratocobalt(III) complexes with configurational and/or vicinal chirality and of their starting mononuclear complex, $[Co(TeO_6H_4)(diamine)_2]^+$, along with electronic absorption and circular dichroism (CD) investigations. The bidentate diamine used are ethylenediamine (en), (*R*)-(−)-1,2-propanediamine (*l*-pn), and (*R*,*R*)-(−)-*trans*-1,2-cyclohexanediamine (*l*-chxn). This study has been partly reported in a preliminary letter.[5]

Experimental

Preparation, Separation, and Optical Resolution of Complexes.

(1) $[Co(TeO_6H_4)(en)_2]_2SO_4\cdot H_2O$: A solution of 2.3 g (10 mmol) of $Te(OH)_6$ in 25 cm³ of water was added to a solution of 2.9 g (5 mmol) of $[Co(CO_3)(en)_2]_2SO_4$ in 20 cm³ of water. The mixture was stirred mechanically at about 55 °C for an hour, from which a light red product deposited. After the solution had been kept in a refrigerator for a few hours, the product was separated by filtration, washed several times with water, methanol and then acetone, and dried in a desiccator over $CaCl_2$. Found: C, 10.64; H, 4.44; N, 11.99; Co, 12.9; Te, 27.6; SO_4^{2-}, 10.9%. Calcd for $[Co(TeO_6H_4)(en)_2]_2SO_4\cdot H_2O$: C, 10.35; H, 4.57; N, 12.08; Co, 12.7; Te, 27.5; SO_4^{2-}, 10.4%. Cobalt was weighed as Co_3O_4, tellurium as the metal, and sulfate ion as AgCl after being substituted to chloride ion with anion exchange resin (Dowex 1-X8, Cl^- form).

(2) $[Co(TeO_6H_4)(en)_2]Cl\cdot 3H_2O$: A solution of 1.15 g (5 mmol) of $Te(OH)_6$ in 15 cm³ of water was added to a solution of 1.43 g (5 mmol) of *cis*-$[CoCl_2(en)_2]Cl$ in 10 cm³ of water, and to the mixture was added 0.24 g (10 mmol) of LiOH. After stirring mechanically at about 35 °C for an hour, the solution was cooled to room temperature and an appropriate amount of ethanol was gradually added to it. After the solution had been kept in a refrigerator overnight, the light red precipitate deposited was separated by a centrifuge. This was dissolved in a small amount of water and the solution was poured into a column (3 cm × 60 cm) of strong acid cation exchanger (SP Sephadex C-25, Li^+ form). The adsorbed band was eluted with 0.25 M LiCl solution, which was adjusted in advance to pH 10 by 1 M LiOH solution. During the elution the column was cooled by flushing ice-water. Two red bands, E_1 and E_2, were eluted in this order. The later eluate (E_2) was treated as in (3) to obtain another complex. The earlier eluate (E_1) was concentrated by freeze-drying method. The product obtained was recrystallized from water (pH 10 by LiOH) at 0 °C by adding ethanol, and washed repeatedly with methanol and acetone. Found: C, 9.49; H, 4.76; N, 11.26%. Calcd for $[Co(TeO_6H_4)(en)_2]Cl\cdot 3H_2O$: C, 9.68; H, 5.29; N, 11.29%. This complex was also obtained predominantly by using DMSO as solvent as follows: A solution of 2.86 g (10 mmol) of *cis*-$[CoCl_2(en)_2]Cl$ in 30 cm³ of DMSO was added with stirring to a solution of 2.30 g (10 mmol) of $Te(OH)_6$ in 40 cm³ of DMSO, and to the mixture was added 0.5 g (20 mmol) of LiOH in 10 cm³ of water. The solution was stirred mechanically at about 35 °C for a few hours. The light red precipitate deposited was separated by filtration. The chromatography of the product showed only one red band (E_1). The absorption spectrum of the eluate was the same as that of the E_1 complex prepared in water.

(3) $[Co(en)_2(Te_2O_{10}H_4)Co(en)_2]Cl_2\cdot CH_3OH\cdot 4H_2O$: The later eluate ($E_2$) from the chromatography in (2) was concentrated at 15 °C in a rotary evaporator, and a large volume of ethanol was added to the concentrated solution. After the solution had been kept in a refrigerator overnight, the red product deposited was collected by a centrifuge. The product was recrystallized from an aqueous LiOH solution of pH 10 at 0 °C by adding acetone–methanol mixture and washed repeatedly with ethanol and acetone. Found: C, 11.28; H, 4.74; N, 11.55%; Co/Te (molar ratio)=1.1. Calcd for $[Co(en)_2(Te_2O_{10}H_4)Co(en)_2]Cl_2\cdot CH_3OH\cdot 4H_2O$: C, 11.34; H, 5.09; N, 11.77%; Co/Te=1.0. Cobalt and tellurium were determined for the eluates from the column. The E_2 complex was also obtained predominantly by raising the reaction temperature to *ca.* 60 °C.

(4) $[\{Co(en)_2\}_2(Te_2O_{10})\{Co(en)_2\}_2]Cl_4\cdot CH_3OH\cdot 12H_2O$: The similar procedure to that for E_1 and E_2 complexes was adopted in water using 1.15 g (5 mmol) of $Te(OH)_6$, 2.86 g

(10 mmol) of *cis*-$[CoCl_2(en)_2]Cl$, and 0.48 g (20 mmol) of LiOH. The reaction was carried out at 60 °C, and the reaction solution was poured into a column directly. The products separated on the column consisted of two red bands, E_2 and a new E_3 in this order, when eluted with 0.35 M LiCl solution (pH 10 by 1 M LiOH). The E_3 complex was purified by the same method as that of E_2 complex. Found: C, 13.58; H,6.07; N, 14.55%; Co/Te (molar ratio)=2.1. Calcd for $[\{Co(en)_2\}_2(Te_2O_{10})\{Co(en)_2\}_2]Cl_4\cdot CH_3OH\cdot 12H_2O$: C, 13.42; H, 6.10; N, 14.73%; Co/Te=2.0. The metal analyses were carried out using the eluate from the column.

(5) Optical Resolution of E_1, E_2, and E_3 Complexes: Optically active E_1 and E_2 complexes were obtained by the preparation procedure starting from $(+)_{589}$-$[CoCl_2(en)_2]^+$.[7] The $(+)^{\mathrm{CD}}_{589}$-E_1 and $(+)^{\mathrm{CD}}_{589}$-E_2 complexes have $\Delta\varepsilon=+0.18$ and $+0.44$, respectively, at the Na D line. The optical resolution of E_3 complex was made by the solubility difference between diastereomers: A solution of 0.015 g of $K_2[Sb_2(d\text{-}C_4H_2O_6)_2]\cdot 3H_2O$ in 5 cm^3 of water was gradually added with stirring to a solution of 0.07 g of E_3 complex in 10 cm^3 of LiOH solution of pH 10 on an ice bath. To the mixture was added an appropriate amount of acetone, and the solution was kept in a refrigerator overnight. The light red diastereomer deposited was isolated by a centrifuge and washed with methanol a few times. This less soluble diastereomer was stirred at 0 °C in LiOH solution of pH 10 containing anion-exchange Sephadex (QAE A-25, Cl^- form). The solution was filtered to remove Sephadex, and CD of the filtrate was measured. The $(+)^{\mathrm{CD}}_{589}$-E_3 complex has $\Delta\varepsilon=+0.18$ at Na D line.

(6) $[Co(TeO_6H_4)(\mathrm{l}\text{-}pn)_2]Cl\cdot 1/2CH_3OH\cdot 3H_2O$: A solution of 0.42 g (2 mmol) of $Te(OH)_6$ in 10 cm^3 of water was added to a solution of 0.63 g (2 mmol) of *cis*-$[CoCl_2(l\text{-}pn)_2]Cl$ in 10 cm^3 of water, and to the mixture was added 0.16 g (4 mmol) of NaOH. After stirring mechanically at about 35 °C for half an hour, the solution was poured into a column (3 cm × 60 cm) of strong acid cation exchanger (SP Sephadex C-25, Li^+ form). The adsorbed band was eluted with 0.25 M LiCl solution, which was adjusted in advance to pH 10 by 1 M LiCl solution. During the elution the column was cooled by flushing ice-water. Two red bands were eluted, and the absorption spectrum of each eluate was similar with that of ethylenediamine E_1 and E_2 complexes, respectively. It was difficult to isolate the crystals from the later eluate. The earlier eluate was poured once more into another similar column and eluted with 0.5 M LiCl solution (pH 10). To the earlier eluate was added a large amount of acetone. After the solution had been kept in a refrigerator overnight, the light red precipitate deposited was separated by a centrifuge. The product was recrystallized from water (pH 10 by LiOH) at 0 °C by adding methanol-acetone mixture and acetone. Found: C, 14.25; H, 5.38; N, 10.28%. Calcd for $[Co(TeO_6H_4)(l\text{-}pn)_2]Cl\cdot 1/2CH_3OH\cdot 3H_2O$: C, 14.45; H, 5.98; N, 10.37%.

Two diastereomeric isomers of this complex were separated also by the column chromatography. When eluted with 0.2 M LiCl solution (pH 10 by 1 M LiOH), the red band further separated gradually into two bands, P1 and P2 (denoted by elution order). P1 and P2 complexes have $\Delta\varepsilon=+0.28$ and -0.20, respectively, at Na D line. These CD values were calculated referring to molar absorption coefficient of the unresolved complex.

(7) $[Co(\mathrm{l}\text{-}chxn)_2(Te_2O_{10}H_4)Co(\mathrm{l}\text{-}chxn)_2]Cl_2\cdot 8H_2O$: A solution of 0.57 g (2.5 mmol) of $Te(OH)_6$ in 10 cm^3 of water was added to a solution of 0.98 g (2.5 mmol) of *trans*-$[CoCl_2(l\text{-}chxn)_2]Cl$ in 10 cm^3 of water, and to the mixture was added 0.2 g (5 mmol) of NaOH. After stirring mechanically at about 40 °C for an hour, the solution was cooled in a refrigerator. The red precipitate deposited was separated by filtration, recrystallized from a cold water (pH 10 by LiOH) by adding acetone, and washed with methanol-acetone mixture and ether. Found: C, 24.02; H, 6.52; N, 9.28; Te, 19.9%. Calcd for $[Co(l\text{-}chxn)_2(Te_2O_{10}H_4)Co(l\text{-}chxn)_2]Cl_2\cdot 8H_2O$: C, 23.85; H, 6.35; N, 9.27; Te, 21.1%.

The filtrate from the red precipitate was poured into a column of SP Sephadex C-25 (Li^+ form) and eluted with 0.225 M LiCl solution (pH 10 by 1 M LiOH). Three red bands, C1, C2, and C3, were eluted in this order. The absorption spectra of three eluates were similar to that of the corresponding ethylenediamine E_2 complex. Furthermore, the absorption and CD spectra of the first red precipitate, $[Co(l\text{-}chxn)_2(Te_2O_{10}H_4)Co(l\text{-}chen)_2]Cl_2\cdot 8H_2O$, were the same as those of C1 eluate. C1, C2, and C3 complexes have $\Delta\varepsilon=-0.90$, -0.41, and -1.42, respectively, at Na D line. These CD values were calculated referring to the molar absorption coefficient of the first red precipitate. In this case, the complex corresponding to E_1 complex of ethylenediamine was not obtained.

Measurements. The visible and ultraviolet absorption measurements were made by a Shimadzu UV-200 spectrophotometer in aqueous solutions. The CD spectra were recoreded on a JASCO MOE-1 spectropolarimeter.

Results and Discussion

The tellurato Co(III) complexes newly obtained are summerized in Tables 1 and 2 with their d-d absorption and CD data. Other highly condensed complexes were observed in trace amounts on the column, but they were too unstable to be isolated. All the complexes obtained are unstable in solution at room temperature, and their solutions were carefully handled below 5 °C. E_1 complex is particularly unstable to reequilibration, and converted rapidly into E_2 complex in aqueous solution at room temperature. This rapid conversion corresponds to the dehydration condensation of the "mononuclear" complex $[Co(TeO_6H_4)(en)_2]^+$ into the "dinuclear" one $[Co(en)_2(Te_2O_{10}H_4)Co(en)_2]^{2+}$. E_1 and E_2 complexes

TABLE 1. ABSORPTION DATA OF THE TELLURATO COMPLEXES IN THE d-d TRANSITION REGION

Complex	$\sigma_{\max}$[a] (log ε)	
	1st band	2nd band
$[Co(TeO_6H_4)(en)_2]^+$	19.2(2.07)	26.7(1.93)
$[Co(en)_2(Te_2O_{10}H_4)Co(en)_2]^{2+}$	19.2(2.41)	26.7(2.38)
$[\{Co(en)_2\}_2(Te_2O_{10})\{Co(en)_2\}_2]^{4+}$	19.2(2.67)	*ca.* 26.5(*ca.* 2.7)
$[Co(TeO_6H_4)(l\text{-}pn)_2]^+$	19.2(2.10)	26.5(1.95)
$[Co(l\text{-}chxn)_2(Te_2O_{10}H_4)Co(l\text{-}chxn)_2]^{2+}$	19.2(2.42)	26.5(2.36)

a) In the unit of 10^3 cm^{-1}.

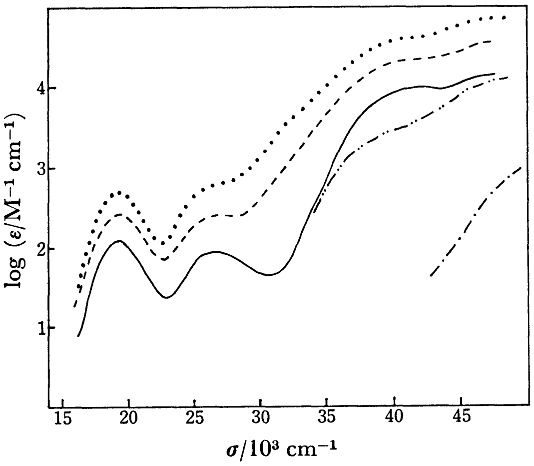

Fig. 1. Absorption spectra of $[Co(TeO_6H_4)(en)_2]^+$ (——), $[Co(en)_2(Te_2O_{10}H_4)Co(en)_2]^{2+}$ (– – –), and $[\{Co(en)_2\}_2(Te_2O_{10})\{Co(en)_2\}_2]^{4+}$ (……). Absorption spectra of $[TeO_6H_{6-n}]^{n-}$ (—·—) and $[Te_2O_{10}H_{8-n}]^{n-}$ (—··—) are shown for comparison (pH 10—11).

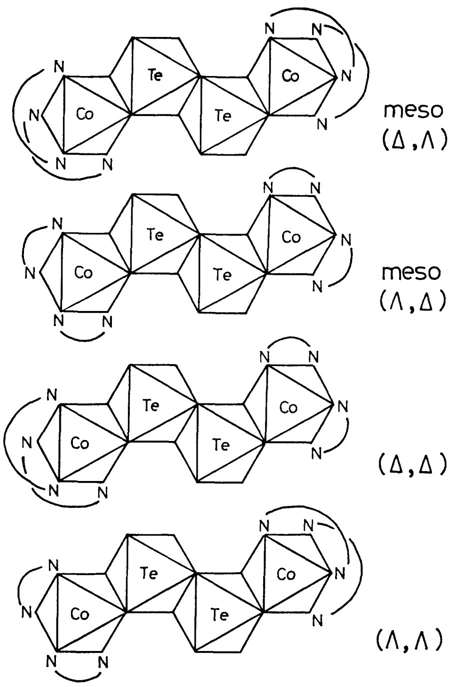

Fig. 2. Proposed structures for the four diastereomeric isomers of $[Co(diamine)_2(Te_2O_{10}H_4)Co(diamine)_2]^{2+}$.

were prepared as their chlorides from the reaction of *cis*-$[CoCl_2(en)_2]^+$ and $Te(OH)_6$ at 35 °C. The formation ratio changed with the reaction temperature. At 60 °C, the former was not obtained at all, and the latter predominantly. The E_1 sulfate $[Co(TeO_6H_4)(en)_2]_2SO_4$ is sparingly soluble in water, but can be converted into the chloride solution by the anion exchange resin Dowex 1-X8 (Cl^- form) at 0 °C. The absorption and CD data of E_1 complex previously reported[5] have been corrected in this paper.

As is seen in Table 1 and Fig. 1, the absorption spectrum of $[Co(TeO_6H_4)(en)_2]^+$ complex shows a characteristic feature of the *cis*-CoN_4O_2 type and is similar to that of $[Co(CO_3)(en)_2]^+$ (σ_{max}=19500 and 27700 cm^{-1})[6] in the d-d absorption band region. Thus it is concluded that the ligand $TeO_6H_4^{2-}$ stands near after CO_3^{2-} in the spectrochemical series. The second absorption bands of E_2 and E_3 complexes appear as a shoulder because of overlapping with an absorption band at 30000—35000 cm^{-1}. The latter band is assigned to the ditellurate band modified by coordination, since the free ditellurate ion has its longest wavelength absorption band at about 38000 cm^{-1} as a shoulder in contrast to the free orthotellurate ion which scarcely absorbs in the region of 35000—42000 cm^{-1} (Fig. 1). The occurrence of the ditellurate band for E_2 complex and the E_1 to E_2 conversion in solution support that E_2 complex [Co(en)$_2$(Te$_2$-

TABLE 2. CD DATA OF THE TELLURATO COMPLEXES IN THE d-d TRANSITION REGION

Complex	σ_{ext}[a] ($\Delta\varepsilon$)	
	1st band	2nd band
$(+)^{CD}_{589}$-$[Co(TeO_6H_4)(en)_2]^+$	19.0(+1.12)	25.0(+0.092) 27.0(−0.012) 29.8(+0.023)
$(+)^{CD}_{589}$-$[Co(en)_2(Te_2O_{10}H_4)Co(en)_2]^+$	19.0(+2.63)	25.0(+0.071) 27.5(−0.071)
$(+)^{CD}_{589}$-$[\{Co(en)_2\}_2(Te_2O_{10})\{Co(en)_2\}_2]^{4+}$ [b]	18.5(+0.83)	24.5(+0.038) 27.5(−0.023)
$[Co(TeO_6H_4)(l\text{-}pn)_2]^+$: P1	19.1(+1.56)	25.1(+0.18) 29.3(+0.06)
: P2	18.4(−0.79) 20.7(+0.38)	25.5(+0.13)
$[Co(l\text{-}chxn)_2(Te_2O_{10}H_4)Co(l\text{-}chxn)_2]^{2+}$: C1	18.5(−3.03) 20.7(+0.84)	26.5(+0.43)
: C2	18.3(−1.36) 20.5(+1.44)	26.0(+0.37)
: C3	18.5(−5.24) 21.5(+0.26)	26.5(+0.57)

a) In the unit of 10^3 cm^{-1}. b) Partly resolved complex.

$O_{10}H_4)Co(en)_2]^{2+}$ corresponds to the condensation product of E_1 complex $[Co(TeO_6H_4)(en)_2]^+$ (see Fig. 2). Similarly, it is supposed that E_3 complex $[\{Co(en)_2\}_2(Te_2O_{10})\{Co(en)_2\}_2]^{4+}$ is a condensation product of $[Co(en)_2(TeO_6H_2)Co(en)_2]^{2+}$, which has not been isolated in this work.

The optically active forms were obtained for E_1, E_2, and E_3 complexes, and two diastereomeric isomers of $[Co(TeO_6H_4)(l\text{-pn})_2]^+$, P1 and P2, were separated by column chromatography. Furthermore, three diastereomeric isomers of $[Co(l\text{-chxn})_2(Te_2O_{10}H_4)Co(l\text{-chxn})_2]^{2+}$, C1, C2, and C3, were obtained. Among several possible structures for the "dinuclear" complex, $[Co(diamine)_2(Te_2O_{10}H_4)Co(diamine)_2]^{2+}$, the most probable ones are those shown in Fig. 2. In this type of condensation, four diastereomeric isomers are possible, which have *meso*-(Δ,Λ), *meso*-(Λ,Δ), (Δ,Δ), and (Λ,Λ) configurations, respectively, in regard to skew pairs of two diamine chelate rings. Many optical isomers are possible for $[\{Co(en)_2\}_2(Te_2O_{10})\{Co(en)_2\}_2]^{4+}$, but further investigation on this complex has not been intended in the present study, because it seems the optical resolution was successful only partly.

The CD data of the present complexes are summerized in Table 2 and Figs. 3—6. The CD patterns of the $[Co(TeO_6H_4)(en)_2]^+$ and $[Co(en)_2(Te_2O_{10}H_4)Co(en)_2]^{2+}$ complexes are similar to that of Λ-$[Co(CO_3)(en)_2]^+$ ($\Delta\varepsilon = +3.7$ at 18900 cm^{-1})⁶⁾ in the d-d absorption band region, and show only one CD band in the first band region (Figs. 4 and 6). Thus, the absolute configurations of $(+)^{\mathrm{CD}}_{589}$-E_1 and $(+)^{\mathrm{CD}}_{589}$-E_2 complexes are assigned to be Λ and (Λ,Λ), respectively. This corresponds well to the fact that the optically active E_1 and E_2 complexes have been obtained from $(+)_{589}$-$[CoCl_2(en)_2]^+$ (Λ configuration)⁶,⁸⁾. The CD curves of two diastereomeric isomers, P1 and P2, of $[Co(TeO_6H_4)(l\text{-pn})_2]^+$ are shown in Fig. 3. Their CD patterns somewhat differ from that of $[Co(TcO_6H_4)(en)_2]^+$, because of the presence of vicinal contribution due to the (*R*)-1,2-propanediamine ligand. It has been well known that the configurational and vicinal CD contributions are separable for many kinds of cobalt(III) complexes by utilizing an additivity rule for a pair of CD curves of diastereomeric isomers.⁹⁾ When the additivity rule is applied for the CD curves of P1 and P2 isomers, the calculated configurational CD curve agrees well with the observed CD curve of E_1 complex (Fig. 4). Thus the sign pattern of $(+)^{\mathrm{CD}}_{589}$-E_1 complex (Λ configuration) suggests that P1 and P2 isomers are Λ and Δ configurations, respectively.

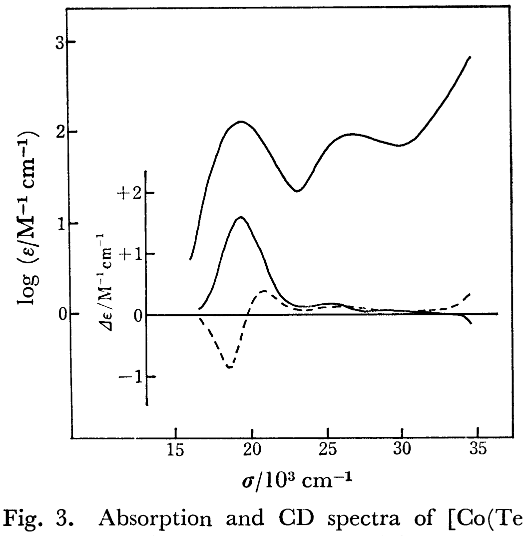

Fig. 3. Absorption and CD spectra of $[Co(TeO_6H_4)(l\text{-pn})_2]^+$: P1 (——) and P2 (----) isomers.

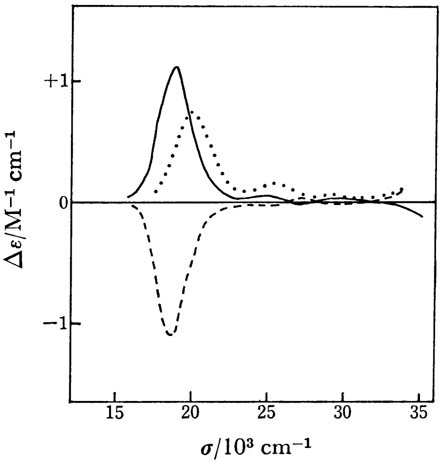

Fig. 4. Calculated configurational (----) and vicinal (······) CD curves of $(-)^{\mathrm{CD}}_{589}$-$[Co(TeO_6H_4)(l\text{-pn})_2]^+$, and observed CD curve (——) of $(+)^{\mathrm{CD}}_{589}$-$[Co(TeO_6H_4)(en)_2]^+$.

As is seen in Fig. 5, the CD curves of three diasteromeric isomers, C1, C2, and C3, of $[Co(l\text{-chxn})_2(Te_2O_{10}H_4)Co(l\text{-chxn})_2]^{2+}$ differ from that of E_2 complex by the same reason as in the case of P1 and P2 complexes. By the application of the additivity rule for the CD curves of C2 and C3 isomers, the calculated configu-

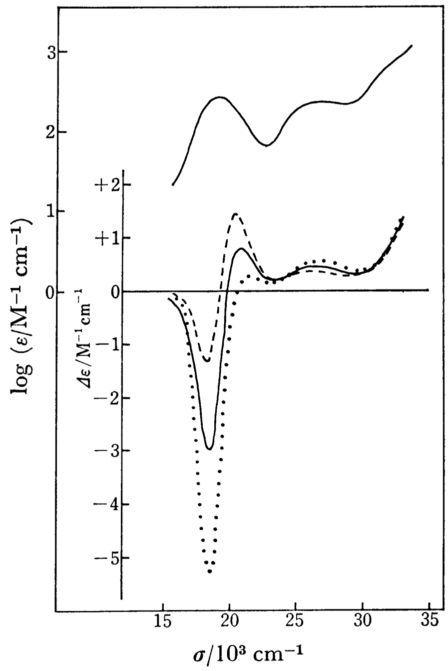

Fig. 5. Absorption and CD spectra of $[Co(l\text{-chxn})_2(Te_2O_{10}H_4)Co(l\text{-chxn})_2]^{2+}$: C1 (——), C2 (----), and C3 (······) isomers.

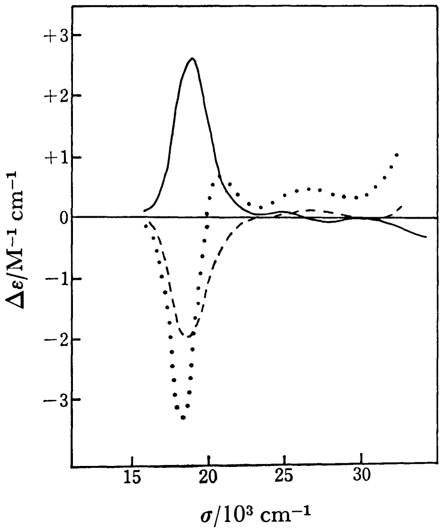

Fig. 6. Calculated configurational (----) and vicinal (······) CD curves of $[Co(l\text{-}chxn)_2(Te_2O_{10}H_4)Co(l\text{-}chxn)_2]^{2+}$(C3 isomer), and observed CD curve (——) of $(+)^{CD}_{589}$-$[Co(en)_2(Te_2O_{10}H_4)Co(en)_2]^{2+}$.

rational and vicinal CD curves agree well with the observed CD curves of E_2 complex and C1 isomer, respectively (Fig. 6). Thus the CD of C1 isomer is contributed only by the vicinal effect due to the four (*R*,*R*)-*trans*-1,2-cyclohexanediamine ligands; namely C1 isomer corresponds to a *meso* one, (Δ,Λ) or (Λ,Δ) in Fig. 2, or to a mixture of the two *meso* ones. Furthermore, by comparison of the sign patterns of C2 and C3 isomers to that of $(+)^{CD}_{589}$-(Λ,Λ)-$[Co(en)_2(Te_2O_{10}H_4)$-$Co(en)_2]^{2+}$ complex, it is confirmed that C2 isomer has (Λ,Λ) configuration, and C3 isomer (Δ,Δ) one.

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