

CCXXII.—*The Rotatory Dispersion of Optically Active
Co-ordination Compounds.*

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THE principle relating to anomalous rotatory dispersion in co-ordination compounds is not only of interest as showing its general applicability but is also of importance in a field where opposing views are held regarding configuration relationships. Further, it is the application of "propriétés physiques," to which Pasteur

refers ("Leçons sur la dissymétrie moléculaire," 1860) when he states "dont l'une est l'image de l'autre en ce qui concerne toutes les propriétés physiques qui dépendent de ce groupement stéréométrique."

Pasteur, regarding especially the physical property of crystalline shape, postulated that an asymmetric molecule will produce enantiomorphous crystals, and Jaeger, in the course of his crystallographic investigations of this principle, determined the dispersion of certain complex salts for different concentrations (*Proc. K. Akad. Wetensch. Amsterdam*, 1915, **17**, 1217; 1915, **18**, 49; 1917, **20**, 244, 263; 1918, **21**, 203, etc.; *Bull. Soc. chim.*, 1923, **33**, 853; *Rec. trav. chim.*, 1920, **38**, 171).

General Occurrence of Anomaly.—In the case of dextrorotatory * potassium cobaltioxalate, the rotation increases rapidly at first as the wave-length attains a maximum at λ 6000, rapidly decreases, becomes zero at λ 6260, reaches a maximum negative value at λ 6400, and then the curve ascends to the zero axis. The two maxima denote passage through an active absorption band, and although crossing of the axis to the right of the band does not occur, yet the data suggest that crossing, if it had been realised, would have made $d\alpha/d\lambda$ positive.

In the case of dextrorotatory potassium chromioxalate, actual crossing takes place. The curve is similar in type to that described above. A broad absorption band (λ 5200—6000) is present, giving rise to true anomaly; further, crossing of the zero axis from the negative to the positive region of rotation was realised at about λ 6360. This anomaly, as distinct from true anomaly, indicates definitely that the compound is configuratively related to "d"-tartaric acid.

An interesting example of a compound of relative configuration denoted by the symbol "l" is dextrorotatory potassium rhodioxalate. The dispersion curve investigated lies between wave-lengths λ 4860 and 7000, crossing the axis at 5970 from the positive to the negative region of rotation ($d\alpha/d\lambda$ negative), and for this wave-length there is no absorption band.

The dispersion curve for dextrorotatory potassium irido-oxalate has been investigated between λ 4790 and 6800 and lies wholly in the positive region. The dispersion is normal for longer wave-lengths, but as the wave-length decreases, inflexion develops, the curve tending to reach a maximum and to become parallel to the zero axis below λ 5100. The parallelism is due to an absorption band. Bruhat (*Bull. Soc. chim.*, 1915, **17**, 226) has investigated this region and finds that the maximum is due to true anomalous

* Actually the dispersion of the lævorotatory salt was investigated.

rotatory dispersion, for it is accompanied by circular dichroism; he has measured both the ellipticities and the rotation. There is complete analogy, therefore, in this respect between asymmetric iridium and asymmetric carbon (compare Cotton, *Ann. Chim. Phys.*, 1896, 8, 347). The portion of the curve between λ 5420 and 6800 is approximately parallel to that for the rhodium compound, the main difference being the proximity of the absorption band in the visible region. The dispersion curve has been displaced as a whole to a greater wave-length. This disposition of the curve suggests that the crossing of the axis is removed into the red end of the spectrum. If this is the case, the relative configuration is "l."

This independent decision that the dextrorotatory cobalt and chromium salts are of "d" configuration is in accordance with both Werner's and Jaeger's conclusions. The view that the dextrorotatory rhodium and iridium compounds are of "l" configuration, though in harmony with Werner's view, is opposed to Jaeger's.

Opposing Views on Relative Configuration.—Werner (*Ber.*, 1912, 45, 1228; *Bull. Soc. chim.*, 1912, 11, 1) considered that those asymmetric isomerides having corresponding relative configurations gave the more sparingly soluble salts with the same active acid or base but did not necessarily give rotatory power of the same sign; for instance, the isomerides set free from the sparingly soluble triethylenediamine-chromium and -cobalt salts were dextrorotatory and configuratively related to the corresponding sparingly soluble rhodium salt, which was laevorotatory. Werner founded this law, which involves sign of rotation, relative configuration, and analogous solubility relationships, on the following facts:

(1) Compounds of opposite rotatory power were obtained by chemical reactions which are not accompanied by a change of configuration of the complex grouping (Co en_2).

(2) Both the dextrorotatory chloronitrocobaltic and the laevorotatory chloro*is*thiocyanatocobaltic series gave salts with *d*-bromocamphorsulphonic acid which were much less soluble than the corresponding salts with the *l*-acid. By the action of potassium thiocyanate on the chloride of the former, and of sodium nitrite on the chloride of the latter, the same dextrorotatory nitro*is*thiocyanatocobaltic chloride is obtained.

(3) The laevorotatory tetraethylenediamine- μ -aminoperoxodibaltic series is transformed by the action of nitrous acid into the dextrorotatory tetraethylenediamine- μ -aminonitrodicobaltic series, and both series give difficultly soluble *d*-bromocamphorsulphonates. Such transformations as the above led to no inconsistencies, thus making unlikely a Walden inversion. Further, *d*-cobalt-, *d*-chrom-

ium-, and *l*-rhodium-triethylenediamine salts are configuratively related. Werner considered that the sign of the rotatory power depends on (a) the nature of the central atom, and (b) the radicals which occupy the other two co-ordination positions.

Jaeger opposes Werner's conclusions on the following grounds. (1) In the analogous optically active complex salts of cobalt and rhodium the two metals will replace each other isomorphously. (2) The less soluble chloride-*d*-tartrates of triethylenediamine-cobalt and -rhodium possess different crystalline forms, the former having a triclinicpedial symmetry and crystallising with 5 mols. of water, whereas the latter has a monoclinic and perhaps sphenoidal symmetry and crystallises with 4 mols. of water. This absence of isomorphism of cobalt and rhodium in analogous combination led to the deduction that they are not of the same but of opposite relative configuration. The triethylenediaminerhodium iodide set free from this chloride-*d*-tartrate, together with all salts derived from it, is lævorotatory, the corresponding cobalt salts being dextrorotatory, so that rotatory power of opposite sign accompanies opposite relative configuration.

Jaeger regards the replacing of the central cobalt by a rhodium atom as manifesting itself only by a difference in magnitude, but not in sign, of the rotation; and he further regards the radicals, basic or acidic, arranged unsymmetrically around the central metallic atom and the specific asymmetry of the arrangement as determining in the first instance the sign of the rotation.

The dispersion curves of the dextrorotatory triethylenediamine salts of cobalt and chromium are analogous and do not resemble those for the corresponding salts of rhodium. The chief point of interest is that the former are normal; the curve for triethylenediaminechromium iodide runs almost parallel to that for the corresponding cobalt salt, but, unlike the latter, it exhibits a point of inflexion. The dispersion curves for triethylenediaminerhodium iodide, bromide, and nitrate are remarkable: for a particular wave-length, increase of rotatory power is in the order given; for longer wave-lengths, the curves are practically parallel; for shorter wave-lengths, the curve for the bromide diverges from that of the nitrate and converges on that of the iodide. They all show inflexion and maxima or approach to maxima; for instance, the dispersion curve for the iodide ascends to a maximum, becomes almost parallel to the zero axis between λ 4420 and 4010, and thereafter from λ 4010 to 3700 (measurements uncertain) the rotatory power decreases.

In the literature there is no mention of the maxima being due to true anomaly. The order of the absorption bands is to a shorter

wave-length in passing from cobalt through chromium * to rhodium.

The published data, which are not conclusive, appear to indicate that (1) both the cobalt and the chromium salts are passing to an active absorption band in the *positive* region, where they will exhibit true anomaly, and (2) the rhodium salts exhibit anomaly in the positive region, the active absorption band being therefore in the *negative* region. Assuming these points and Werner's law, we may draw up the following scheme :

Series.	Sign of rotation.	Relative configuration deduced from dispersion.	Less soluble compound. (General type : " l " -complex <i>d</i> -acid.)
Co en ₃	Dextro	" l "	Chloride- <i>d</i> -tartrate.
Cr en ₃	Dextro	" l "	<i>d</i> -Camphornitronate.*
Rh en ₃	Lævo	" l "	Chloride- <i>d</i> -tartrate.

* The same optically active acid was not employed in this case as resolving agent. Werner (*loc. cit.*) considers the above series to be configuratively related.

The dispersion diagram would now be a comparative series of curves (those for cobalt and chromium situated in the positive region, that for rhodium in the negative region), passing and, in the case of rhodium, about to pass to an active absorption band in the positive region.

A further important point noted by Jaeger is that the crystals of the optically active triethylenediamine-cobalt and -rhodium nitrates, which in solution have the *opposite* sign of rotation, exhibit sphenoids of the *same* algebraic sign, *viz.*, the dextrorotatory cobalt salt (relative configuration " l " by the argument above) exhibits a right-handed sphenoid (+) and the lævorotatory rhodium nitrate (" l ") also shows the right-handed form.

Confirmatory Evidence of the Complex Oxalates and Malonates.

If it be conceded that the dispersion curve for dextrorotatory potassium cobaltioxalate is altogether analogous to the similar dispersion curve for dextrorotatory potassium chromioxalate, and that the dispersion curve for dextrorotatory potassium irido-oxalate is altogether analogous to that for dextrorotatory potassium rhodi-oxalate, then the following interesting correlation is possible. (It should be noted that the order of the absorption bands is to a shorter wave-length on passing from cobalt through chromium and iridium to rhodium.)

* Inflection mentioned above indicates approach to a maximum (anomaly or true anomaly).

Optically active potassium salt.	Dispersion.	"Sign" of rotation of complex salt.	Relative configuration deduced from dispersion.
Cobalti oxalate.*	True anomaly.	Lævo "l"	Give less soluble strychnine compounds of the general type l-strychnine + "l" complex.
Chromi oxalate.*	True anomaly and anomaly (in negative region).	Lævo "l"	
Iridio oxalate.	True anomaly.	Dextro "l"	
Rhodi oxalate.	Anomaly (in negative region).	Dextro "l"	

* The lævorotatory compounds are considered for simplicity of treatment.

A point of further importance with regard to the enantiomorphism of the crystalline forms, which has been noted by Jaeger, is that lævorotatory potassium cobaltioxalate exhibits a right-handed trigonal bipyramid, whereas dextrorotatory potassium irido-oxalate and dextrorotatory potassium rhodioxalate similarly exhibit a right-handed trigonal bipyramid. All three complex salts exhibit hemihedral forms of the same algebraic sign (+).

The dispersion data indicate that the relative configuration of the complex salts of cobalt, chromium, iridium, and rhodium may be denoted by the symbol "l," which is in accordance with Werner's law but opposed to Jaeger's conclusions.

The dispersion diagram* for these compounds of the same configuration is of considerable interest: with the movement of the absorption band into the violet, that part of the dispersion curve to the right of the band increases over a greater range of wave-length into the visible spectrum. The curves (direction, red to blue) all ascend to absorption bands in the positive region. From the diagram, which consists of a series of dispersion curves of a truly comparative series of active co-ordination compounds, one can realise the specific influence of the central atom on the dispersion, and further, the relationship of corresponding absorption bands to, and influence on, the dispersion. Comparison of this diagram with the dispersion diagram for the comparative series of complex triethylenediamine salts, as tabulated in the previous section, brings out the influence of the substituents and the comparative asymmetry of the molecule, just as in the case of the carbon compounds two series of the same relative configuration can be compared.

Cinchonine l-rhodimalonate is the less soluble salt and the isolated lævorotatory potassium rhodimalonate exhibits a maximum dispersion at λ 5800 in the negative region, at which wave-length there is no absorption band; accordingly the compound is assumed to be of relative configuration "l." The substitution of three oxalyl for the malonyl radicals round the central atom results in a diminution of rotation in the region of anomaly, and actual crossing

* This can be constructed approximately from data given.

of the zero axis is realised for the complex oxalate. Although the alkaloids used in the resolutions were different in the two cases of the isolation of the optically active complex malonate and of the oxalate, yet the isolated salts from the less soluble compound have the same relative configuration "l." Data given for potassium malonochromate are in full agreement: salt obtained, *l*-strychnine malonochromate-acid; dispersion, analogous to that of the corresponding complex oxalate; configuration, deduced from the dispersion, "l." The strychnine *l*-salt is anomalous in the negative region, but no conclusion can be drawn from this fact as to the configuration of the chromium complex. In the complex malonates, as in the other cases considered, the absorption band has moved to a shorter wave-length in passing from chromium to rhodium.

The researches of Jaeger and his collaborators have been of fundamental importance in deciding the relative configuration of co-ordination compounds.

Anomalous Rotatory Dispersion with Circular Dichroism.

Longchambon (*Compt. rend.*, 1924, **178**, 1828) has compared the rotatory dispersion of dextrorotatory potassium rhodioxalate in solution and in the crystalline state. The curves are similar, visual anomaly being shown in both cases by crossing of the zero axis and by maxima (for α and ρ) in the negative region. Crossing of the axis, however, has been displaced to a shorter wave-length (λ 5190) in the crystalline state. Longchambon considers that the rotatory dispersion in both cases may be represented by a two-term Bruhat equation, one term, the dispersion constant of which is negative, representing the anomalous rotatory dispersion at an absorption band near or in the visible region, and the second term, a low-frequency term, the dispersion constant of which is positive, representing the normal rotatory dispersion proceeding to a band in the near ultra-violet.

The rotatory dispersion of the co-ordination compound is analogous to that exhibited by 1:2-diphenyl-3-*l*-bornyl-imidoxanthide [*l*-bornyl *N*-thiobenzoyl-*N*-phenylthioncarbamate] (Tschugaev, *Ber.*, 1909, **42**, 2244; *Z. physikal. Chem.*, 1910, **74**, 503; *Ann. Chim. Phys.*, 1911, **22**, 137) and has been investigated quantitatively by Bruhat (*Ann. Physique*, 1915, **22**, 237). The measurements obtained are in agreement with a three-term equation

$$\alpha = \frac{D(\lambda^2 - \lambda_0^2)}{(\lambda^2 - \lambda_0^2)^2 + T^2\lambda^2} + \frac{A}{(\lambda^2 - \lambda_1^2)} + \frac{B}{\lambda^2}$$

True Anomaly Term.

Low-Frequency Term.
High-Frequency Term.

Anomaly.

The first term represents anomaly due to the absorption band in the green (λ 5240); D , T , and λ_0 were determined from the curve of circular dichroism, D being negative. A is positive; the second term represents normal positive rotatory dispersion exhibited in the visible region on proceeding to the band λ_1 3520 on the longer wave-length side of the band.

Bruhat found that, in order to obtain agreement between observed and calculated values, it was necessary to introduce the third (high-frequency) term, the constant B being negative. This term represents visible normal negative rotatory dispersion on proceeding to a band in the remote inaccessible ultra-violet region. λ_1 was deduced from the dispersion of the refractive index, A and B from rotatory dispersion.

This quantitative expression, involving data given by circular dichroism, variation of refractive index, and rotatory dispersion, is significant. The results indicate that (a) crossing of the axis is accompanied by circular dichroism; (b) the high-frequency term is negative, indicating a compound of relative configuration " l ," which is in agreement with the mode of crossing the axis ($d\alpha/d\lambda$ negative); (c) crossing of the axis is on the longer wave-length side of the band and not at the band; (d) the rotatory dispersion does not proceed to $\pm \infty$ at the band; (e) the case is not analogous to the rotatory dispersion of tartaric acid, of which the anomaly is not accompanied by circular dichroism (Bruhat, *loc. cit.*) and is represented as the sum of two simple partial rotations, the characteristic frequencies being both in the ultra-violet; (f) the rotatory dispersion of the l -bornyl derivative can be referred to the superposition of three partial rotations: one represents true anomaly at the absorption band in the visible region, the other two are simple partial rotations, the characteristic frequencies of both being in the ultra-violet, and are characteristic of anomalous and potentially anomalous optically active, transparent compounds.

The phenomena exhibited by this compound appear to be similar in general character to those exhibited by methyl and ethyl d -bornyl xanthogenide and l -bornyl dixanthogenide (Tschugaev, *loc. cit.*; compare also derivatives of fenchyl alcohol). The compounds follow the rule with regard to the occurrence of anomalous rotatory dispersion and the relative configuration; for instance, the rotatory dispersion curve for the transparent l -menthyl ester of triphenylacetic acid exhibits a maximum in the negative region, that for l -menthol dithiourethane exhibits inflexion, maximum, and crossing of the axis from the negative to the positive region.*

* Maximum light absorption occurs at λ 5160, and crossing of the axis at λ 5280 (approx.).

Further, Tschugaev has pointed out with respect to certain of the above compounds "that in many cases the anomalous dispersion curves are shifted by the influence of temperature and by the nature of the solvent in the same direction as in the case of tartaric acid and its esters."

The borneol esters employed by Tschugaev were not contaminated with the corresponding but differently rotating isoborneol esters (*J. Russ. Phys. Chem. Soc.*, 1904, **36**, 988; Pickard and Littlebury, *J.*, 1907, **91**, 1973).

Further consideration of the phenomenon of circular dichroism is deferred. On the above grounds it is considered that the dispersion data for dextrorotatory potassium rhodioxalate, both in solution and in the crystalline form, suggest the relative configuration "l."

Configuration and the Algebraic Sign of the Hemihedral Form.

A point of interest arising from the study of co-ordination compounds is whether the right- or left-handed (R.-H. or L.-H.) character of hemihedral crystals is, in general, directly related to the relative configuration of the constituent molecules. In the following scheme, d represents the density of the crystalline form, and ρ_D is the rotation per mm.

Compound.	$\frac{d[\alpha]_D}{100}$	ρ_D .	Character of crystal.	Configuration.	Difference of rotation (cryst. — soln.).
Quartz.....	—	+21.73 ^{o†}	L.-H.	"l"	—
l-Tartaric acid* ...	-0.12°	+11.4§	L.-H.	"l"	+11.52°
l-NaKC ₄ H ₄ O ₆ ...	-0.47	- 1.35§	R.-H.	"l"	- 0.88
l-NH ₄ NaC ₄ H ₄ O ₆ ...	-0.52	+ 1.55§	L.-H.	"l"	+ 2.07
l-(NH ₄) ₂ C ₄ H ₄ O ₆ ...	-0.61	+ 8.9	L.-H.	"l"	+ 9.51
l-Rb ₂ C ₄ H ₄ O ₆	-0.69	+10.24¶	L.-H.	"l"	+10.93
l-Cs ₂ C ₄ H ₄ O ₆	-ve. rotn.†	+19	L.-H.	"l"	+ve. rotn.
Ammonium molybdomalate	+5	+32.3	L.-H.	"l"	+27.3
d-Camphor	+0.55	+ 0.65	L.-H.	"l"	+ 0.10
l-Asparagine	-0.06 × d (λ 5893)	- 6.2 (λ 5780)	R.-H.	"l"	-ve. rotn.
d-K ₃ [Rh(C ₂ O ₄) ₃].... (for λ 6400)	-1.3 (approx.)	- 9.5	R.-H.	"l"	- 8.2

* l-Tartaric acid and derivatives have been considered for simplicity.

† Exact data not available.

‡ Lowry.

§ Dufet, *J. Physique*, 1904, [iv], **3**, 757.

|| Longchambon, *Compt. rend.*, 1922, **175**, 174; 1921, **173**, 89.

¶ Traube.

The following examples are important.

1. In quartz the asymmetry of the crystalline form has been

induced by the non-asymmetric silicon dioxide molecules striving after a tetrahedral arrangement of the oxygen atoms round the silicon atom.* The dispersion data indicate that the tetrahedral arrangement in left-handed quartz is of relative configuration "l." In the table the convention of chemists with regard to the taking of polarimetric measurements opposite to the direction of the propagation of the light is adhered to. In this case the molecules give rise to optical activity only while they are constituents of the crystal, and the optical activity is induced by crystal structure alone.

2. In the crystalline form, potassium rhodioxalate exhibits an increase of rotation in the negative region (to which region the compound belongs since it is of "l" configuration) and a displacement of the crossing of the axis to a shorter wave-length.

This difference of the dispersion curves in the two states could be interpreted by orientation producing distortion of the molecules in the crystalline state and so affecting the ratio of the positive and negative dispersion constants; the molecules may occupy a spiral arrangement in the crystal, and this arrangement may affect the magnitude of the rotation.

The crystalline symmetry of the oxalate ($L^3 \cdot 3L^2$) is identical with the molecular symmetry indicated by Werner's stereochemical representation. Longchambon (*loc. cit.*) considers that all the molecules have a more or less perfect, parallel orientation; Burgers (*Proc. Roy. Soc.*, 1927, *A*, **116**, 553), however, concludes that the similarity of the dispersion curves in the two states is not due to a parallel, but may be partly due to the spiral, arrangement of the molecules in the crystal.

The algebraic sign of the hemihedrism is directly related to the relative configuration for the complex salt mentioned above. It is therefore considered that the movement in the rotatory dispersion is due to the superimposed effect of the spiral arrangement, accompanied by a slight distortion of the molecules themselves. This latter effect is similar to that produced by temperature, solvents, etc.

The dextrorotatory complex oxalate crystallises in the rhombic system with the symmetry of quartz and exhibits a single right-handed trigonal bipyramid; Jaeger (*loc. cit.*) states that a trapezoidal face is never present.

Asparagine and sodium potassium tartrate are further examples of compounds of relative configuration "l" associated with right-

* Compare the case of lithium potassium sulphate (Burgers, *loc. cit.*) where the oxygen tetrahedra are found to be regular.

handed forms. Using the convention of physicists, the algebraic sign of the rotation in the crystal and in solution as well as the crystalline form is +; this convention leads to simplicity in the tabulation of results.

The cases of quartz and the rhodioxalate are interesting examples showing, respectively, optical rotation induced in inactive molecules by crystalline structure, the active molecules then being arranged spirally, and rotation due to active molecules little influenced by crystalline structure although the active molecules are arranged spirally.

Tartaric acid presents a case where a spiral arrangement in the crystal may influence the rotatory dispersion, this spiral being due, not to the arrangement of the crystal molecules, but to the differences between the crystal and the chemical molecule. Astbury (*Proc. Roy. Soc.*, 1922, A, **102**, 506) has shown that the arrangement of the two tartaric acid molecules in the crystal unit produces through the hydroxyl groups an opposite spiral to that produced by the asymmetric carbon atoms.

The distortion effect of the molecules upon one another favours the negative dispersion coefficient, and the resulting crystal shape is opposite in stereometrical character to that of the molecules. On dissolution of the substance, the negative dispersion coefficient regains its normal value, visual anomaly resulting, and an explanation based on polymerisation is not necessary to account for the anomaly in solution, since rotatory dispersion both in the crystal and in solution is essentially molecular, the effect of crystal structure being analogous to the effect of solvents.

Other cases are afforded by sodium ammonium tartrate, and ammonium, rubidium, and caesium tartrates.

In the majority of the tartrates it is the left-handed crystal which is associated with a compound of relative configuration "*l*," and in the above cases, whenever there is a change of sign in rotation from solution to crystal, the relative configuration is indicated, e.g., left-handed sodium ammonium "*l*"-tartrate has *negative* rotation in solution, and *positive* in the crystal. The data are, however, too restricted to support a generalisation.

Ammonium molybdomalate and camphor are two interesting examples of compounds of relative configuration "*l*" exhibiting positive rotations both in solution and in the crystalline form. The positive dispersion coefficient of the molecules, distorted but exhibiting the shape of a molecule of "*l*" configuration, has persisted in and, in the case of the malate, has been enhanced by crystal structure, the crystals being of opposite stereometrical character to that of the relative configuration of the molecules.

This involves the point that right-handed "l"-asparagine is in crystalline structure more closely related to *right*-handed sodium ammonium tartrate than to the *left*-handed tartrate, even though "l"-asparagine and sodium ammonium *d*-tartrate are of opposite relative configuration (see table).

Ostromisslensky (*Ber.*, 1908, **41**, 3035) observed that an inactive supersaturated solution of sodium ammonium *d*- and *l*-tartrates, when inoculated (at 6°) by a nucleus of *l*-asparagine, deposited exclusively the *right*-handed variety, but a supersaturated solution of sodium ammonium *d*-tartrate does not deposit crystals when inoculated with a nucleus of the *l*-tartrate (Gerez, *Compt. rend.*, 1866, **63**, 843). The enantiomorphous nucleus has an exclusive directional influence.

Summary.

1. Optically active co-ordination compounds exhibit anomaly and true anomaly, the former indicating the relative configuration.
2. The configuration as deduced from dispersion data is in agreement with Werner's law.
3. The algebraic sign of the hemihedral form of the co-ordination compound considered indicates the relative configuration, but no generalisation can be made at present.

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