The Phenomenon of Conglomerate Crystallization. 24. Spontaneous Resolution in Coordination Compounds. Classical and Spontaneous Resolution of the $[Co(en)_2(NH_3)Br]^{2+}$ Cation. Crystal Structures and Absolute Configurations of Resolved $(+)_{589}$-[*cis*-Λ(δλ)-$Co(en)_2(NH_3)Br]S_4O_6$ (I) and of Conglomerate Crystals of [*cis*-Λ(δλ)-$Co(en)_2(NH_3)Br]S_4O_6$ (II)

Ivan Bernal* and James Cetrullo

Department of Chemistry, University of Houston, Houston, Texas 77204-5641

W. Gregory Jackson

Chemistry Department, University College, The University of New South Wales, Australian Defence Force Academy, Campbell, ACT, Australia 2600

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Compound I, $BrCoS_4O_6N_5C_4H_{19}$, crystallizes in the monoclinic space group $P2_1$ with cell constants $a = 7.971(3)$ Å, $b = 9.610(4)$ Å, $c = 10.953(4)$ Å, and $\beta = 103.90(5)°$; $V = 814.43$ Å^3 and $d(\mathrm{MW} = 500.32, z = 2) = 2.040$ g cm^{-3}. The structure factors were corrected for absorption: $\mu = 40.036$ cm^{-1} using ψ-scan curves. The absolute configuration of the $(+)_{589}$ enantiomorph was determined from refinement of the original and inverted coordinates, and the correct absolute configuration is $(+)_{589}$-[*cis*-Λ(δλ)-$Co(en)_2(NH_3)Br]^{2+}$. The most interesting aspect of the crystallographic study of I is the nature of the interaction between the cation and anion, which are hydrogen-bonded such that one oxygen of $-SO_3$ binds one of the hydrogens of the $-NH_3$ ligand, while a second oxygen is attached to a hydrogen on an $-NH_2$ of an adjacent en ligand. This is precisely the interaction mode in $(+)_{589}$-[*cis*-Λ(δλ)-$Co(en)_2(NH_3)Cl]$(*d*-3-bromocamphorsulfonate)$_2\cdot H_2O$, the cation of which is known to be very efficiently resolved by bromocamphorsulfonate. Finally, the $S_4O_6^{2-}$ anion is itself intrinsically chiral (torsional angle S–S–S–S = +90.5°); its absolute configuration is coupled with that of the chiral [*cis*-$Co(en)_2(NH_3)Cl]^{2+}$ cation, with which it crystallizes. Crystallization of a racemic mixture of [*cis*-$Co(en)_2(NH_3)Br]S_4O_6$ **II** produces a conglomerate crystallizing in the same space group and with the same cell constants as those found for **I**.

Introduction

Twelve years after he predicted[1] that octahedral compounds containing ions such as $[Co(en)_2ox]^+$ should be resolvable into antipodal pairs, Werner was able to announce[2] that he and his student Victor King had succeeded in separating the enantiomers of [*cis*-$Co(en)_2(NH_3)X]^{2+}$ (X = Cl, Br). The magic agent that performed the desired resolution was the *d*-3-bromocamphorsulfonate anion. It is now known[3] that that the *cis*-aminobromo derivative is particularly easy to separate due to the insolubility of the ion pair formed by (+)-bromocamphorsulfonate and the $(+)_{589}$ cation.

In 1978, Yoneda et al.[4] determined the structure and absolute configuration of the diastereoisomeric pair $(+)_{589}$-[*cis*-Λ(δλ)-$Co(en)_2(NH_3)Cl]$(*d*-3-bromocamphorsulfonate)$_2\cdot H_2O$ **III** and described in detail the cation–anion interaction present therein. For the purpose of what follows, we will summarize their report by saying that the sulfonate groups of the two anions present bind the cation by short hydrogen-bonded contacts. The reader is referred to the original work for details. If, as suggested,[4] the specificity of the interaction between the [*cis*-$Co(en)_2(NH_3)$-$Cl]^{2+}$ cation and the *d*-3-bromocamphorsulfonate (BCS^-) resolving agent resides in the strong hydrogen-bonded interaction between the $-SO_3$ oxygens and the hydrogens of a pair of adjacent $-NH_3$ and $-NH_2$ moieties, then some other charge-compensating anion bearing an $-SO_3^-$ group may form hydrogen-bonded interactions resembling those described by Yoneda et al.,[4] giving rise to conglomerate crystallization. After all, such interactions resemble those which we observed for other systems in which conglomerate crystallization took place and which we attributed to hydrogen-bonded interactions.[5,6] Thus, we decided to determine the crystallization characteristics of Werner's famous cation using related counteranions. In this report, we describe our experiences with the tetrathionate anion, $S_4O_6^{2-}$.

There was an independent interest in the $S_4O_6^{2-}$ anion. It has long been known to be dissymmetric, having a two-bladed propeller structure in the solid state.[7] It is of course a labile species, and from the prospect of conglomerate crystallization (which does not hinge on the $S_4O_6^{2-}$ anion being chiral), it was anticipated that a preresolved cation such as (+)-[*cis*-$Co(en)_2(NH_3)Br]^{2+}$ might lead to its resolution in the solid state and, moreover, effect a second-order dissymmetric synthesis in converting 1 equiv of (±)-$S_4O_6^{2-}$ totally into (+)- or (−)-$S_4O_6^{2-}$.

Experimental Section

Syntheses. $Na_2S_4O_6$(IV) and $K_2S_4O_6$ (V). Sodium thiosulate (49.6 g; 0.2 mol) in water (50 mL) was treated with I_2 (25.4 g; 0.2 mol). White crystals separated from the mixture during the process. Ethanol was added slowly to give a total volume of 800 mL. This mixture was stirred for 1 h at 0 °C, and the crystals were collected, washed with ethanol and ether, and air-dried. Yield: 23.4 g (0.087 mol; 43.3%). To the filtrate was added saturated KI (ca. 100 mL), resulting in the crystallization of appreciable $K_2S_4O_6$ after ca. 12 h at 0 °C. The material was collected, washed with ethanol and ether, and air-dried. Yield: 4.0 g. Both salts were recrystallized from the minimum volume of water with addition of ethanol. The sodium salt is the more soluble.

Unresolved **$[Co(en)_2(NH_3)Br]Br_2$ (VI)** was prepared by the procedure of Werner[8] and converted to the purple **$[Co(en)_2(NH_3)Br]S_4O_6$ (II)** salt by mixing aqueous solutions of the cobalt complex and $Na_2S_4O_6$ or $K_2S_4O_6$.

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Table I. Crystallographic Data for $(+)_{589}$-[*cis*-$\Lambda(\delta\lambda)$-$Co(en)_2(NH_3)Br]S_4O_6$ (I)

chem formula $BrCOS_4O_6N_5C_4H_{16}$	fw 500.32
$a = 7.971(3)$ Å	space group $P2_1$ (No. 4)
$b = 9.9610(4)$ Å	$T = 18$ °C
$c = 10.953(5)$ Å	$\lambda = 0.710\,73$ Å
$\beta = 103.90(5)°$	$\mu = 40.036$ cm^{-1}
$V = 814.43$ Å^3	$R(F_o) = 0.0283^a$
$Z = 2$	$R_w(F_o) = 0.0276^b$

[a] $R(F_o) = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $R_w(F_o) = [\sum w^2(|F_o| - |F_c|)^2/\sum|F_o|^2]^{1/2}$.

Table II. Positional and Isotropic Thermal Parameters for I[a]

atom	*x/a*	*y/b*	*z/c*	*B* (Å^2)
Br	0.0661(1)	0.1990	0.3323(1)	3.44(2)
Co	−0.2086(1)	0.3023(1)	0.2413(1)	1.63(1)
S1	−0.3388(2)	−0.2056(3)	0.1666(2)	2.53(3)
S2	−0.3619(2)	−0.2031(3)	0.3546(2)	3.08(4)
S3	−0.6198(2)	−0.1940(3)	0.3362(2)	2.89(4)
S4	−0.6926(2)	−0.4067(2)	0.3442(2)	2.07(3)
O1	−0.1567(7)	−0.209(1)	0.1849(6)	7.7(2)
O2	−0.4257(7)	−0.3265(6)	0.1064(4)	4.0(1)
O3	−0.4170(8)	−0.0802(6)	0.1088(5)	4.1(1)
O4	−0.8306(6)	−0.3923(6)	0.4088(4)	3.1(1)
O5	−0.7500(6)	−0.4533(5)	0.2159(4)	2.6(1)
O6	−0.5414(6)	−0.4769(5)	0.4160(4)	2.9(1)
N1	−0.2190(6)	0.1953(7)	0.0880(5)	2.3(1)
N2	−0.1119(7)	0.4501(6)	0.1565(5)	2.6(1)
N3	−0.3150(6)	0.1548(6)	0.3174(5)	2.2(1)
N4	−0.4437(6)	0.3681(6)	0.1715(5)	2.1(1)
N5	−0.1803(7)	0.4135(8)	0.3953(5)	3.5(1)
C1	−0.1023(8)	0.2621(7)	0.0153(6)	2.4(2)
C2	−0.1263(9)	0.4147(9)	0.0231(6)	3.0(2)
C3	−0.4987(7)	0.1896(8)	0.3114(6)	2.1(1)
C4	−0.5675(8)	0.2562(7)	0.1851(6)	2.4(2)

[a] Estimated standard deviations are listed in parentheses. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as $(4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table III. Bond Distances and Angles for I[a]

	(A) Bond Distances (Å)		
Br–Co	2.393(1)	Co–N1	1.954(5)
Co–N3	1.940(4)	Co–N2	1.954(5)
Co–N4	1.951(4)	Co–N5	1.963(6)
N3–C3	1.488(6)	N4–C4	1.491(6)
C1–C2	1.483(9)	N1–C1	1.506(7)
N2–C2	1.478(7)	C3–C4	1.503(7)
S1–S2	2.111(2)	S1–O1	1.417(4)
S1–O2	1.430(5)	S1–O3	1.433(5)
S3–S4	2.133(2)	S4–O4	1.450(3)
S4–O5	1.440(3)	S4–O6	1.439(4)
S2–S3	2.019(2)		
	(B) Bond Angles (deg)		
Br–Co–N1	89.1(1)	Br–Co–N2	94.2(2)
Br–Co–N3	88.2(1)	Br–Co–N4	173.9(1)
Br–Co–N5	87.9(2)	N1–Co–N2	85.1(2)
N1–Co–N3	93.0(2)	N1–Co–N4	89.7(2)
N1–Co–N5	175.7(2)	N2–Co–N3	176.9(2)
N2–Co–N4	91.7(2)	N2–Co–N5	92.0(3)
N3–Co–N4	85.8(2)	N3–Co–N5	90.1(2)
N4–Co–N5	93.5(2)	Co–N1–C1	109.0(4)
Co–N2–C2	110.9(4)	Co–N3–C3	110.1(3)
Co–N4–C4	108.9(3)	N1–C1–C2	106.6(5)
N2–C2–C1	107.7(5)	N3–C3–C4	105.9(4)
N4–C4–C3	107.2(4)	S2–S1–O1	100.9(2)
S2–S1–O2	108.2(2)	S2–S1–O3	106.9(2)
O1–S1–O2	114.4(4)	O1–S1–O3	113.9(4)
O2–S1–O3	111.6(2)	S1–S2–S3	103.2(7)
S2–S3–S4	103.6(1)	S3–S4–O4	100.0(2)
S3–S4–O5	106.5(2)	S3–S4–O6	105.7(2)
O4–S4–O5	113.9(2)	O4–S4–O6	114.3(2)
O5–S4–O6	114.6(2)		
	(C) Hydrogen Bond Distances (Å) and Angles (deg)		
O2–H1	2.18	N1–H1···O2	167.9
O3–H10	2.31	N3–H10···O3	151.5
O3–H16	2.14	N4–H16···O3	159.3
O4–H9	2.04	N3–H9···O4	168.8
O5–H8	2.07	N2–H8···O5	153.9
O6–H18	2.17	N5–H18···O6	175.1

[a] Numbers in parentheses are estimated standard deviations in the least significant digits.

(+)-$[Co(en)_2(NH_3)Br]S_4O_6$ (I). VI was resolved by the procedure of Kauffman and Linley[9] and converted to the bromide. The latter was converted to (+)-$[Co(en)_2(NH_3)Br]S_4O_6$ (I) by the addition of $Na_2S_4O_6$. Slow evaporation of an aqueous solution produced the desired crystals (purple).

No analyses were performed on IV–VI, since these are compounds of well-established compositions given by the authors of their syntheses. Elemental analyses (C, H, N) for I and II were performed at the Australian National University. Both compounds share the same composition, $BrCoS_4O_6N_4C_4H_{19}$ (MW = 500.32). Anal. Calcd: C, 9.60; H, 3.83; N, 14.00. Found for I: C, 9.56; H, 3.91; N, 13.92. Found for II: C, 9.62; H, 3.80; N, 13.87.

X-ray Diffraction. Data for I were collected first with a crystal of preresolved (+)-$[Co(en)_2(NH_3)Br]S_4O_6$; later, a crystal of II, "the racemic material", prepared as described above, was mounted on the diffractometer and, as hoped, was found to give the exact same cell as crystals of I. Moreover, the same 25 reflections used during the centering of the optically resolved crystal were now recollected with the "racemic" one and the relative intensity ratios of the two sets compared. They were essentially identical. Thus, no further data were collected with the crystal (II) derived from the conglomerate crystallization experiment.

All intensity data were collected with an Enraf-Nonius CAD-4 diffractometer operating with a Molecular Structure Corp. TEXRAY-230 modification[10] of the SDP-Plus software package.[11] The crystals of I were centered with data in the 18° ≤ 2θ ≤ 25° range, and examination of the cell constants and Niggli matrix[12] clearly showed it to crystallize in a primitive, monoclinic lattice whose systematic absences are those of space group $P2_1$ or $P2_1/m$. Since the crystal was grown from preresolved material, the former was selected. The intensity data set was corrected for absorption using empirical curves derived from ψ scans[10,11] of five reflections. The scattering curves were taken from Cromer and Waber's compilation.[13]

The structure of I was determined from the Patterson function, using the Br^- ligand as the heavy atom. Refinement, followed by difference Fourier maps, located the missing atoms. Refinement proceeded smoothly, and upon isotropic convergence, the hydrogen atoms of the en rings were computed at idealized positions (N–H, C–H = 0.95 Å). These were added as fixed contributions to the structure factors (B = 5.0 Å^2), and after each set of refinement cycles, the hydrogen positions were recomputed.

Refinement of I with heavy atoms anisotropic and hydrogens at fixed positions with B = 5.00 Å^2 converged [coordinates + + + = $\Lambda(\delta\lambda)$] to $R(F) = 0.0316$ and $R_w(F) = 0.0312$. When the configuration was inverted (− − −), refinement converged to $R(F) = 0.0393$ and $R_w(F) = 0.0377$; thus, the former was chosen. At this point, a difference map produced the hydrogen positions of the $-NH_3$ ligand, which were added as fixed contributions. New hydrogen atom positions were computed for the en rings, and refinement was completed with the correct absolute configuration. After elimination of four low-angle reflections, which suffered severely from extinction, the final $R(F)$ and $R_w(F)$ factors were 0.0283 and 0.0276, respectively.

Details of data collection and processing are summarized in Table I. Figure 1 gives a labeled stereoview of the asymmetric unit in I, and

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(9) Kauffman, G. B.; Linley, E. V., Jr. *Inorg. Synth.* **166**, *8*, 198.

(10) TEXRAY-230 is a modification of the SDP-Plus[11] set of X-ray crystallographic programs distributed by Molecular Structure Corp., 3200 Research Forest Dr., The Woodlands, TX 77386, for use with their automation of the CAD-4 diffractometer.

(11) SDP-Plus is the Enraf-Nonius Corp. X-ray diffraction data processing set of programs distributed by B. A. Frenz & Associates, 1140 E. Harvey Rd., College Station, TX 77840.

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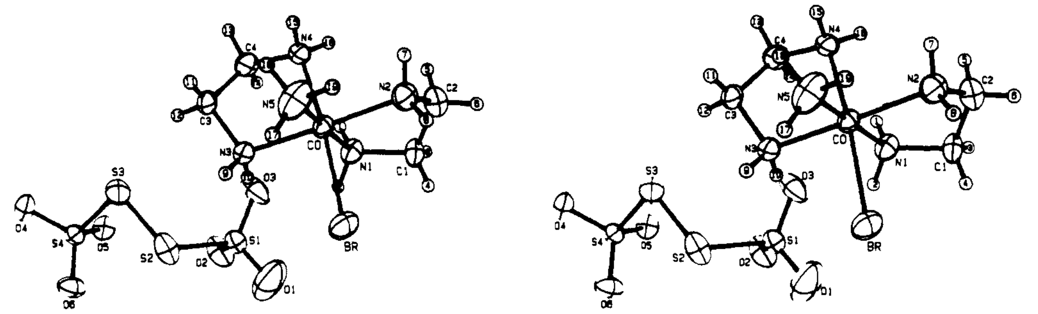

Figure 1. Stereoview of the ion pair present in $(+)_{589}$-[*cis*-Λ(δλ)-$Co(en)_2(NH_3)Br]S_4O_6$. Note the interaction between the tetrathionate oxygens and the $-NH_2$ hydrogens of the en ligands, the conformations (δ and λ) of the en ligands are not the correct ones for the Λ configuration at the Co cation, and that of the en ligand on the lower part of the figure, hydrogen-bonded to the tetrathionate anion, is wrong. See text for discussion.

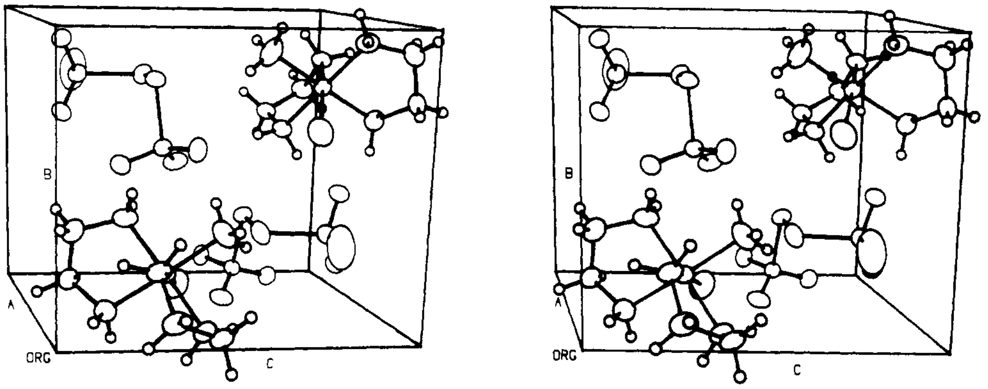

Figure 2. Packing diagram for $(+)_{589}$-[*cis*-Λ(δλ)-$Co(en)_2(NH_3)Br]S_4O_6$.

Figure 2 depicts the stereo packing of the ions in the unit cell. Final positional and equivalent isotropic thermal parameters are given in Table II; bond lengths, bond angles, and torsional angles for I are given in Table III. Anisotropic thermal parameters are presented as supplementary material.

Description of the Structures

Elsewhere,[14] we discussed the structure of Werner's $[Co(en)_2(NH_3)Br]Br_2$ (VI), which is a racemate crystallizing in space group *C*2/*c*. Therefore, our starting material for either I or II was a racemate and no chiral bias was introduced in our experiments due to an unexpected conglomerate crystallization of the parent reagent. We will merely remark here that one of the two enantiomorphic cations present in the lattice of VI is in the $(lel)_2$ conformation and Λ(δδ) configuration, which are the lowest energy conformation and configuration for such a cation.

We now describe the results obtained for compound I and, *inter alia*, for II. Figure 1 depicts in stereo the diastereoisomeric pair present in the asymmetric unit, which shows the preresolved $(+)_{589}$ complex to be [*cis*-Λ(δλ)-$Co(en)_2(NH_3)Br]S_4O_6$, the conformation of which is not the lowest energy conformation for a pair of such en rings.[15] Figure 2 gives the packing diagram.

Discussion

The diastereoisomeric pair depicted on Figure 1 shows that a terminal $-SO_3$ of the tetrathionate anion is hydrogen-bonded to an $-NH_3$ hydrogen as well as to a hydrogen of an axial $-NH_2$ moiety of an en ligand and that it is this spanning across the two points, in a bidentate fashion, which lends stability to the configuration depicted. Note that this hydrogen-bonded interaction is such that the en ligand bonded to the tetrathionate oxygens has the λ conformation. (For a cis-Λ-conformed $Co(en)_2$ fragment, the helical chirality of the N–C–C–N fragments prefers to be δ; however, one is not.) Precisely the same effects were observed by Yoneda et al.[4] for the conformation, configuration, and hydrogen-bonding scheme in the case of $(+)_{589}$-[*cis*-Λ(δλ)-$Co(en)_2(NH_3)Cl]$(*d*-3-bromocamphorsulfonate)$_2$·H_2O (III). Thus, we agree with their suggestion that the specificity of the ion-pair formation[3] mentioned earlier is probably due to this interaction. Interestingly, the implication is that, if the $-SO_3$···cation interaction is present, the anion needs not necessarily be a chiral and optically stable resolving agent such as *d*-3-bromocamphorsulfonate.

The Chirality of the Anion

It is noteworthy that the tetrathionate anion is inherently chiral inasmuch as the S1–S2–S3–S4 fragment is a helical entity with a torsional angle of +90.4°. In fact, $Na_2S_4O_6{\cdot}2H_2O$ crystallizes as a conglomerate[7]—a fact that was recently verified by a complete redetermination of its structure and absolute configuration.[16]

In their report of the structure and absolute configuration of $Na_2S_4O_6{\cdot}2H_2O$, Christides et al.[16] gave the following data, which are contrasted with ours (the second entry in each case):

Distances (Å)

S1–S2	S2–S3	S3–S4
2.115(1) 2.111(2)	2.019(1) 2.019(2)	same as S1–S2[a] 2.133(2)

Angles (deg)

S1–S2–S3	S2–S3–S4
103.95(1) 103.7(7)	*a* 103.6(1)

Torsional Angles (deg)

S1–S2–S3–S4
−89.59 90.4

[a] The anion lies on a 2-fold axis of the crystal lattice.

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(14) Bernal, I.; Cetrullo, J.; Jackson, W. G. *J. Coord. Chem.*, submitted for publication.

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Thus, the two structural studies give very similar results, despite the marked difference in the nature of the charge-compensating cation.

It is also revealing that other cobalt amine species containing thionate anions also crystallize via a conglomerate pathway, a particularly interesting one being bis(2-aminoethyl 3-aminopropyl sulfide)(1,8-diaza-4-thiaoct-5-yl) cobalt(III) dithionate, studied by Bjerrum et al.[17] which crystallizes in the enantiomorphic space group $P2_1$. Figure 6 of their paper clearly depicts the robust hydrogen-bonding network present in the lattice, which the authors describe as follows: "...the hydrogen bonding between the oxygen atoms of this anion and the hydrogen atoms of the four nitrogens atoms seems to greatly stabilize the crystal as is also implied by the high calculated density of 1.73 g/cm^3. Only one nitrogen-bound hydrogen atom (on N11) does not participate in the hydrogen bonding."

The compound of Bjerrum et al.[17] is not the only metal amine dithionate available in the literature that has been found to crystallize as a conglomerate. Lehmann et al.[18] reported the synthesis and crystal structure of $[Ru(NH_3)_5(\text{fumaric acid})]S_2O_6$, space group $P2_12_12_1$, and Diamantis et al.[19] determined the crystal structure of $[Ru(NH_3)_3(acac)(OH_2)][S_2O_6]\cdot 2H_2O$, which also crystallizes as a conglomerate in space group $P2_12_12_1$. Moreover, even as simple a salt as $K_2S_2O_6$ has been reported[20] to crystallize in the space group $P321$, a clear case of conglomerate crystallization. Finally, $(NH_4)_2Se_2O_5$ was reported[20] to crystallize in the enantiomorphic space group $P2_12_12_1$.

Conclusions

The observations of Yoneda and associates,[4] suggesting the high efficiency noted in the resolution of $[cis\text{-}Co(en)_2(NH_3)Br]^{2+}$ cations by *d*-3-bromocamphorsulfonate anions is connected with specific cation–anion interactions, led us to explore the possibility that such hydrogen-bonded interactions were generic and not connected with the inherent chirality of the bromocamphorsulfonate anion, thus leading to the observation of similar interactions in the case of an anion such as tetrathionate. This inference seems to have been correct, as we observed conglomerate crystallization in the case of compound **II**. We are exploring other polythionates as charge-compensating anions, specially in view of the comments concerning the intrinsic conglomerate crystallization tendencies of these simple anions even with achiral cations.

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Supplementary Material Available: Tables of crystallographic parameters, anisotropic thermal parameters, hydrogen atom coordinates, and torsional angles for I (4 pages). Ordering information is given on any current masthead page.

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