

without the necessary intervention of other ordering processes. Nevertheless, the possibility persisted that subcritical antiferromagnetic interactions such as described by Moriya¹¹ and others¹² might be found in Cs₃VCl₆·3H₂O. These interactions only appear in a measurement involving a magnetic field, such as the susceptibility but not the specific heat, and have been observed in a number of nickel compounds with large zero-field splittings, such as NiSnCl₆·6H₂O¹³ and [Ni(C₅H₅N-O)₆](ClO₄)₂.¹⁴ In fact, the fit did not improve significantly when eq 2 and 3 were corrected to allow for such a molecular field interaction, and the fit was judged inferior to that when the TIP term was added instead.

Calculations on trivalent vanadium have appeared¹⁵ which lend credence to the presence of a TIP term. It arises from the mixing of the ³E(³T₁) level with the ground state; the excited level is expected to be of the order of 1000 cm⁻¹ above the ³A₂(³T₁) state which is the main contributor to the magnetic susceptibility.

Another interesting aspect of this compound which remains to be explored experimentally, as mentioned above, concerns its behavior in an external magnetic field. As the present data show, the *z* principal axes of the two molecules in the unit cell have the same orientation with respect to the parallel or green axis, which then allows these molecular axes to be aligned parallel to an external field. As the field is turned on, the *m* = ±1 levels will diverge linearly in this orientation, Figure 1a, while the *m* = 0 level will remain essentially unchanged in energy. One can then calculate that the |0⟩ and |-1⟩ levels should cross at some *H*₀ = 90 kOe, which then provides a doubly degenerate ground state of effective spin - 1/2 at *H*_{eff} = 0. If the orientation were not so favorable, divergence of the levels, as in Figure 1b, would occur instead. Determination of the magnetization in high fields, such as recently carried out on [C(NH₂)₃]V(SO₄)₂·6H₂O¹⁶ and FeSiF₆·6H₂O,¹⁷ then allows a direct evaluation of the zero-field splitting. Furthermore, magnetic ordering may then take place at *H*₀ at some temperature of the order of the exchange interaction *J*/*k* between the metal ions, whereas magnetic ordering otherwise does not take place at *H*₀ = 0. Experiments of this type have been reported for Cu(NO₃)₂·2.5H₂O¹⁸ and are currently being carried out on Cs₃VCl₆·3H₂O.

Acknowledgment. We thank Professor P. J. McCarthy of Canisius College for calling this system to our attention and for supplying us with several single crystals. Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for the partial support of this research.

Registry No. Cs₃VCl₆, 16448-91-2.

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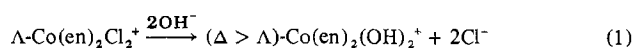
Concerted Base Hydrolysis Bailar Inversion of Λ-Dichlorobis(ethylenediamine)cobalt(III). A Circular Dichroism Study¹

Won S. Kwak and Ronald D. Archer*

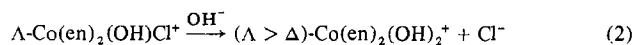
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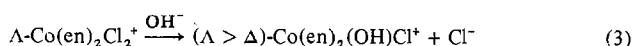
Several years ago Dittmar and Archer² reported that the inversion reaction



is concerted because the reactions



and



both go with a predominance of retention (for the *cis* ion formed—actually (3) gives *trans* > *cis*). Whereas analysis of reactions 1 and 2 was clear-cut with or without multicomponent spectral analysis, the stereochemical analysis of reaction 3 relied upon concurrent polarimetric and multicomponent spectral results. The results were internally consistent, but in the analogous base hydrolysis of the corresponding *trans*-Co(en)₂(OH)Cl⁺ ion, the multicomponent analysis proved to be fallible.³ Slight errors in extinction coefficients or a minor impurity caused an error of up to 7% in the product analysis of the *trans* reaction. Therefore, with the availability of high-precision circular dichroism instrumentation in our laboratory⁴ we decided to investigate the chirality of this system, the silver-free base hydrolysis analogue of the classic Bailar inversion reaction.⁵ Actually all base hydrolysis products are studied as the aqua species obtained through acid quenching of the reactions.

Experimental Section

Syntheses. The complexes were prepared and resolved according to reported procedures: Λ-*cis*-[Co(en)₂Cl₂]Cl·H₂O,⁶ Λ-*cis*-[Co(en)₂(H₂O)Cl]Br₂·H₂O,^{7,8} and Λ-[Co(en)₂CO₃]ClO₄.⁷ The last was converted to Λ-*cis*-[Co(en)₂(H₂O)₂]³⁺ in cold 0.5 M HClO₄, a reaction known to be completely stereoretentive.⁹

Anal. Calcd for Co(C₂H₈N₂)₂Cl₃·H₂O: C, 15.8; H, 6.0; N, 18.5. Found:¹⁰ C, 16.1; H, 6.2; N, 18.2. Calcd for Co(C₂H₈N₂)₂Cl(H₂O)Br₂·H₂O: C, 11.7; H, 4.9; N, 13.6. Found: C, 11.8; H, 4.8; N, 13.3. Calcd for Co(C₂H₈N₂)₂CO₃ClO₄: C, 17.7; H, 4.6; N, 16.5. Found: C, 17.2; H, 4.6; N, 16.4.

All reaction vessels, deionized deaerated water, chemicals, solutions, and transfer syringes and pipets were maintained at 0 °C for 2 h prior to use. The dissolution of samples, the quenching of reactions, and chromatographic separations were all conducted in a 0 °C bath surrounded by air at <5 °C. Replicate experiments were performed under all relevant conditions. A typical base hydrolysis experiment follows. Λ-[Co(en)₂Cl₂]Cl·H₂O (30 mg, 10⁻⁴ mol) dissolved in 4 ml of water was rapidly mixed with 1 ml of 1 M NaOH. After 15 s of vigorous shaking, the reaction was quenched with 10 ml of 0.5 M HClO₄ solution. The final volume was adjusted to 25 ml with water. A circular dichroism spectrum of the total solution was measured and ion-exchange separation of Co(en)₂Cl₂⁺ and Co(en)₂(H₂O)Cl₂²⁺ from Co(en)₂(H₂O)₂³⁺ was accomplished on Dowex 50W-X8, 20–50 mesh, pretreated with 2 M NaClO₄ at pH 1.0 (adjusted with HClO₄). Ten milliliters of the solution was passed through the column and eluted with 10 ml of 2 M NaClO₄ acidified (pH 1.0) solution. A circular dichroism spectrum of the eluent was obtained. (Further elution with more acidified NaClO₄ produced no further spectral changes.)

Analogous reactions were studied with Λ-Co(en)₂Cl(OH)⁺, i.e., Λ-Co(en)₂(H₂O)Cl²⁺ in strongly basic solutions.

Circular Dichroism Spectra. The circular dichroism (CD) spectra of the optically active complexes were measured on a Cary 60

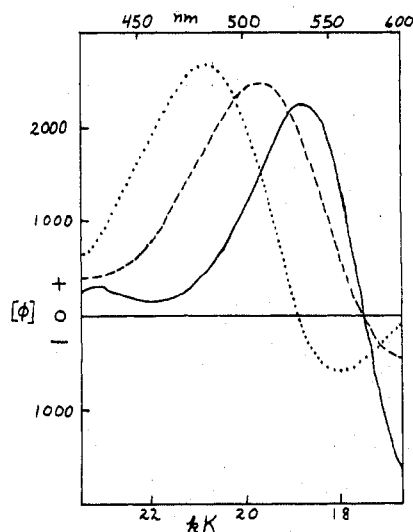


Figure 1. Circular dichroism spectra for the Δ -cis isomers of $\text{Co}(\text{en})_2\text{Cl}_2^+$ (—), $\text{Co}(\text{en})_2\text{Cl}(\text{H}_2\text{O})_2^{2+}$ (---), and $\text{Co}(\text{en})_2(\text{H}_2\text{O})_2^{3+}$ (····).

Table I. Circular Dichroism Parameters of Δ -cis- $\text{Co}(\text{en})_2\text{Cl}_2^+$, Δ -cis- $\text{Co}(\text{en})_2\text{Cl}(\text{H}_2\text{O})_2^{2+}$, and Δ -cis- $\text{Co}(\text{en})_2(\text{H}_2\text{O})_2^{3+}$ ^a

Complex	CD max, 0, and min, nm (kK)	$\Delta\epsilon$ ([ϕ])
Δ -cis- $\text{Co}(\text{en})_2\text{Cl}_2^+$	535 (18.69)	+0.685 (+2260)
	572 (17.48)	0
	595 (18.61) ^b	-0.457 (-1510)
Δ -cis- $\text{Co}(\text{en})_2\text{Cl}(\text{H}_2\text{O})_2^{2+}$	510 (19.61)	+0.748 (+2470)
	570 (17.54)	0
	595 (18.61) ^b	-0.139 (-460)
Δ -cis- $\text{Co}(\text{en})_2(\text{H}_2\text{O})_2^{3+}$ ^c	480 (20.83)	+0.806 (+2660)
	530 (18.87)	0
	555 (18.02)	-0.182 (-600)

^a CD spectra were measured in aqueous solution at 0 °C. ^b The CD minimum is actually located above 600 nm. ^c Δ -[$\text{Co}(\text{en})_2\text{-CO}_3\text{]ClO}_4$ was dissolved in 0.5 M HClO_4 solution at 0 °C.

spectropolarimeter fitted with a CD accessory and a sensitive end-on photomultiplier system. The spectra were obtained at 0 °C in 1- or 5-cm jacketed Suprasil cells with appropriate masks to avoid stray radiation. Replicate spectra were obtained and compared to baselines obtained without disturbing the cell using Teflon syringe tubing to fill and empty the cell. Only spectra with dynode voltage values of <400 V were used.¹¹

Results

Circular dichroism spectra for the three optically active Δ -cis complexes of importance to this study are shown in Figure 1, and their circular dichroism maxima, intensities, and ellipticities for the visible region are given in Table I. To the lower energy side of the maxima are minima at 555 nm (18.0 kK) for the Δ -cis- $\text{Co}(\text{en})_2(\text{H}_2\text{O})_2^{3+}$ ion and above 600 nm for the other two ions. In the classic circular dichroism study by Mathieu,¹² the diaqua and aquachloro spectra were not measured into this negative region. We have been limited to ≤ 600 nm by the instrument at our possession. Our calibrated molar ellipticities are slightly higher than reported by Mathieu,¹² suggesting a slightly improved optical purity in either the light source or the complex ions.

Typical base hydrolysis results for the Δ -cis- $\text{Co}(\text{en})_2\text{Cl}(\text{OH})^+$ ion at 0 °C after acidification are shown in Figure 2 and Table II. Complete retention within experimental error is apparent for reaction 2 inasmuch as the protonation during acidification does not change the chirality.¹³

Typical base hydrolysis results for the Δ -cis- $\text{Co}(\text{en})_2\text{Cl}_2^+$ ion at 0 °C after acid quenching are shown in Figures 2 and 3 and in Table III. The retention for the $\text{Co}(\text{en})_2\text{Cl}(\text{H}_2\text{O})_2^{2+}$

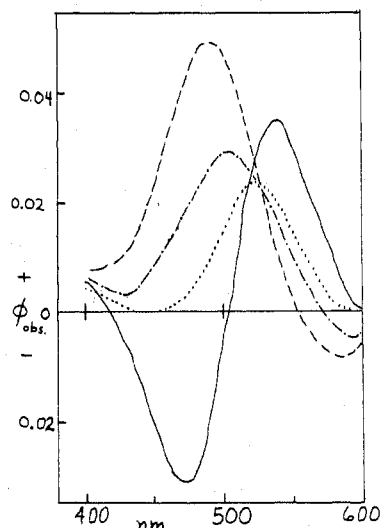


Figure 2. Typical circular dichroism spectra of the 0 °C base hydrolysis reaction products of (1) Δ -cis- $\text{Co}(\text{en})_2\text{Cl}_2^+$ (—) and (2) Δ -cis- $\text{Co}(\text{en})_2\text{Cl}(\text{H}_2\text{O})_2^{2+}$ (---) after acid quenching. Ion-exchange removal of most of the $\text{Co}(\text{en})_2(\text{H}_2\text{O})_2^{3+}$ species results in the ···· spectrum for (1) and in the --- spectrum for (2).

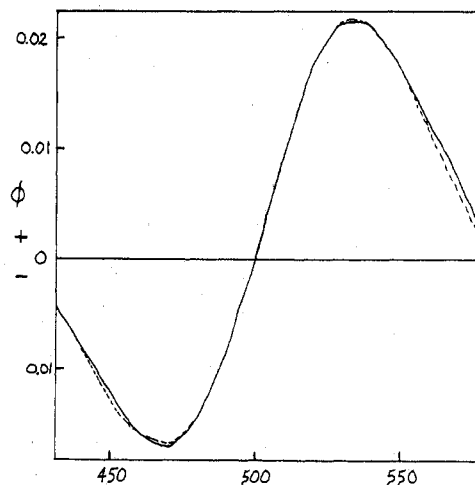


Figure 3. A computer fit (---) to a 0 °C base hydrolysis reaction product spectrum (—) for Δ -cis- $\text{Co}(\text{en})_2\text{Cl}_2^+$.

Table II. Circular Dichroism Data of the Base Hydrolysis of Δ -cis- $\text{Co}(\text{en})_2\text{Cl}(\text{OH})^+$ at 0 °C

[Complex], M	[NaOH], M	Reactn time, ^a s	% Δ -cis- $\text{Co}(\text{en})_2\text{-}(\text{H}_2\text{O})_2^{3+}$	% Δ -cis- $\text{Co}(\text{en})_2\text{Cl}(\text{H}_2\text{O})_2^{2+}$ ^b
1.0×10^{-2}	2.0×10^{-2}	30	16.0	84.4
9.2×10^{-3}	6.0×10^{-2}	30	45.7	54.8
1.2×10^{-2}	6.0×10^{-2}	45	53.5	45.4

^a The reaction times are approximate. ^b The amount of the starting material which was not base hydrolyzed.

ion and the inversion for the $\text{Co}(\text{en})_2(\text{H}_2\text{O})_2^{3+}$ ion, both of which are formed as reaction products in the base hydrolysis reaction of the dichloro ion, are apparent both from the simple ion-exchange experiment and from the calculated spectra. No reasonable fit can be obtained for both ions either as retained or as inverted species.

Discussion

The complete retention (>98%) observed for base hydrolysis reaction of Δ -cis- $\text{Co}(\text{en})_2\text{Cl}(\text{OH})^+$ is appreciably different from the results of Chan and Tobe,¹⁴ who suggested 61% retention, 36% inversion, and 3% trans complexes, but is very similar to the results of Dittmar and Archer,² who noted

Table III. Relative Amounts of Optically Active Cis Products in the Base Hydrolysis Solutions of Λ -*cis*-Co(en)₂Cl₂⁺ at 0 °C

[OH ⁻], M	Time, ^a s	Initial Λ - Co(en) ₂ - Cl ₂ ⁺ M	Product optical activity (net), ^b %		
			Λ - Co(en) ₂ - Cl ₂ ⁺	Λ - Co(en) ₂ - Cl(OH) ⁺	Λ - Co(en) ₂ - (OH) ₂ ⁺
0.30	15	0.0235	0 ±	4.0 ±	6.5 ±
			0.2	0.2	0.2
0.20	15	0.0200	0.2 ±	5.0 ±	6.3 ±
			0.3	0.2	0.2
0.20	25	0.0217	0.1 ±	3.5 ±	4.0 ±
			0.3	0.2	0.2
0.20	35	0.0203	0.1 ±	2.3 ±	3.8 ±
			0.2	0.2	0.2
0.10	20	0.0232	1.2 ±	10.0 ±	2.7 ±
			0.6	0.6	0.2

^a Times only approximate. ^b Reactions quenched with HClO₄; product percentages based on protonation causing no stereochemical changes; ranges based on only concentrations which gave reasonable fits to the optical activity data; σ for best fit between calculated and observed values is less than 0.0007, 0.0004, 0.0005, 0.0004, and 0.0006^o, respectively.

approximately 98 ± 4% retained *cis*-dihydroxo (and 4–8% *trans*) complex.¹⁵ Therefore, any "real" *cis*-chlorohydroxo species formed in multistep reactions should also undergo subsequent base hydrolysis with >98% retention of configuration. As noted by Dittmar and Archer,² the almost exclusive formation of Λ -*cis*-Co(en)₂(OH)₂⁺ during the base hydrolysis of Λ -*cis*-Co(en)₂Cl(OH)⁺ supports a tetragonal pyramid^{16,17} or *cis* displacement.¹⁸

The fact that the spectra are additive for this part of the study implies a high reliability for the molar ellipticities and the applications of these values in the calculations of the multicomponent system to follow. As expected, the amount of Λ -*cis*-Co(en)₂(OH)₂⁺ formed in the base hydrolysis of the Λ -*cis*-chlorohydroxo species increases as either the time or the hydroxide ion concentration increases. The product quantities cannot be used to determine reaction rates as the time of mixing is an appreciable fraction of the total reaction time. The alternate approach of predissolution followed by quenched flow studies was abandoned because of aequation complications.

The Λ -*cis*-Co(en)₂Cl₂⁺ ion undergoes base hydrolysis in hydroxide solutions with a novel stereochemistry which cannot be explained by any *simple* intermediates, either symmetric or dissymmetric. That is, the attainment of more Λ - than Δ -*cis*-Co(en)₂Cl(OH)⁺ (and appreciable *trans*-Co(en)₂Cl(OH)⁺)^{2,14} in the base hydrolysis of Λ -*cis*-Co(en)₂Cl₂⁺ is logical from any approach to octahedral stereochemistry,^{13,16–18} but the fact that there is a predominance of the Δ isomer of *cis*-Co(en)₂(OH)₂⁺ in this reaction whereas Λ -*cis*-Co(en)₂Cl(OH)⁺ reacts to give only Λ -*cis*-Co(en)₂(OH)₂⁺ requires a concerted reaction.¹⁹

A number of concerted reaction paths have been suggested previously for such inversion reactions.^{2,20} The ion-pair suggestion of Bailar²⁰ together with mobilization by a second hydroxide is quite attractive. In order to explain the concurrent inversion and loss of both chloro ligands, the authors favor a temporary spin change analogous to that suggested earlier for the ammonia inversion²¹ and in agreement with simple ligand field calculations.²² That is, a trigonal bipyramid (or an analogous intermediate necessary to go from one tetragonal pyramid to another) requires almost 3 times the ligand field activation energy required for a tetragonal pyramid *unless a spin change occurs*.^{22,23} We contend that the remarkable inversion is a result of both chlorides being lost during the period of intermediate spin. The spin state must exist for only a very short time or else complete decomposition or racemization would occur. However, as noted previously by Bailar,²⁰

if one end of an ethylenediamine in Λ -*cis*-Co(en)₂Cl₂⁺ is moved, a *trans* product results, whereas the movement by the other end produces a Δ -*cis* product. The fact that some racemization often occurs in base hydrolysis reactions of cobalt(III) complexes¹⁹ suggests that this mechanism may be more general but that in general it is not the predominant pathway.

Finally, the octahedral Bailar inversion is not limited to cobalt(III) complexes. Such inversions are known for chromium(III) triethylenetetramine (trien) complexes,²⁴ which the α -*cis* ion undergoes an optical inversion when transforming to the β -*cis* ion, but this is a stereochemically required inversion for the simplest rearrangement. Another inversion, that of Fe(phen)₃²⁺ with CN⁻,^{4,25} can also be explained via a spin change. An alternate suggestion²⁶ for the cyanide attack on the ligand followed by a tautomeric shift in the apparent bimolecular reaction does not easily accommodate the inversion for this ion, especially since the corresponding Fe(bpy)₃²⁺ ion reaction produces a net retention.²⁷

Registry No. Λ -*cis*-Co(en)₂Cl₂⁺, 18660-62-3; Λ -*cis*-Co(en)₂Cl(H₂O)²⁺, 45837-52-3; Λ -*cis*-Co(en)₂(H₂O)₂³⁺, 18485-08-0; Λ -*cis*-Co(en)₂Cl(OH)⁺, 19428-61-6.

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Crystal and Molecular Structure of *trans*-Oxochlorotetrakis(methyl isocyanide)molybdenum(IV) Triiodide

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The synthesis and characterization of cationic molybdenum(IV) oxohalo isocyanide complexes have been described.¹ Here we report the x-ray crystal structure of $[\text{MoOCl}(\text{CNCH}_3)_4]\text{I}_3$. The relationship between this complex and other isocyanide and cyanide complexes of molybdenum is discussed elsewhere.²

Experimental Section

Collection and Reduction of X-Ray Data. Maroon-red needles of $[\text{MoOCl}(\text{CNCH}_3)_4]\text{I}_3$ were obtained by slow evaporation of solvent from a nitromethane-methanol solution. Approximate unit cell parameters and the space group were determined on a precession camera using nickel-filtered $\text{Cu K}\alpha$ radiation (λ 1.5418 Å). From the Laue symmetry C_{2h} ($2/m$) and observed extinctions hkl , $h + k \neq 2n$, the space group was determined to be $C2$ [C_2 ; No. 5], Cm [C_2 ; No. 8], or $C2/m$ [C_{2h} ; No. 12].

Using a Picker FACS-I-DOS diffractometer, data were collected on an irregular hexagonal prism with faces (210) and $(\bar{2}10)$ 0.45 mm apart, (210) and $(\bar{2}10)$ 0.45 mm apart, (100) and $(\bar{1}00)$, 0.06 mm apart, and (201) and $(\bar{2}0\bar{1})$ 0.050 mm apart. The crystal mounting was nearly along $[\bar{1}40]$, which approximates the needle axis. The details of data collection are given in Table I. The apparatus, experimental technique, and data reduction were as described previously.³ The orientation matrix used for data collection and the lattice parameters were computed from least-squares refinement of the setting angles of 12 carefully centered reflections.

Determination and Refinement of the Structure. An origin-removed, sharpened Patterson map revealed the positions of the iodine and molybdenum atoms, one carbon atom, and one nitrogen atom.⁴ A structure factor calculation and difference Fourier map in space group Cm showed the oxygen atom of the $\text{Mo}=\text{O}$ moiety and the chlorine atom to be disordered in such a manner as to be related by a twofold axis. Subsequent refinement was carried out in space group $C2/m$, with the Mo, O, and Cl atoms equally disordered in sites x , 0 , z , and \bar{x} , 0 , \bar{z} and assigned half-weights. The higher symmetry of this space group requires the terminal iodine atoms of the triiodide anion to be related by a C_2 operation and is consistent with the Patterson map solution and with refinement in Cm .

Because of the small value of the lattice parameter β in $C2/m$, the hkl indices and atomic coordinates were transformed to the non-standard space group $I2/m$ (Table I), in which all subsequent computations were made. Least-squares refinement, minimizing the function $\sum w(|F_o| - |F_c|)^2$ with weights $w = 4F_o^2/\sigma^2(F_o^2)$, converged at final values of 0.042 and 0.078 for $R_1 = \sum ||F_o| - |F_c||/\sum |F_o|$ and $R_2 = (\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2)^{1/2}$, respectively. No final parameter shift was greater than 0.071 times its estimated standard deviation. All atoms were assigned anisotropic temperature factors of the form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$. A total of 56 parameters were varied to fit 855 data for which $F_o^2 > 2\sigma(F_o^2)$. In all calculations of F_o , the atomic scattering factors and anomalous dispersion corrections for iodine, molybdenum, and chlorine were taken from ref 5.

The error on an observation of unit weight, $[\sum w(|F_o| - |F_c|)^2/(\text{NO} - \text{NV})]^{1/2}$, was estimated to be 2.459; since $\sum w\Delta^2$ was fairly independent of $|F_o|$ and of $(\sin \theta)/\lambda$, no compensating weighting scheme

Table I. Experimental Data for the X-Ray Diffraction Study of $[\text{MoOCl}(\text{CNCH}_3)_4]\text{I}_3$

(A) Crystal Parameters at $23 \pm 1^\circ\text{C}$		
Space group [C_{2h} ; No. 12]	$C2/m$	$I2/m$
a , Å	17.024 (10)	6.068 (4)
b , Å	10.670 (9)	10.670 (9)
c , Å	6.068 (4)	15.087 (10)
$\cos \beta$	0.4791 (3)	-0.1385 (3)
β , deg	61.37 (3)	97.96 (3)
V , Å ³	967.4	967.4
Z	2	
Mol wt	692.3	
ρ (calcd), g cm ⁻³	2.376	
ρ (obsd), g cm ⁻³ (by neutral buoyancy in $\text{CH}_2\text{I}-\text{CH}_2\text{I}_2$)	2.37 (1)	

(B) Measurement of Intensity Data

Radiation: graphite-monochromatized $\text{Mo K}\alpha$ (λ 0.71069 Å)

Attenuators: used for counts $>10,000/\text{s}$

Takeoff angle: 2.15°

Detector aperture: 6.25 mm \times 6.25 mm

Crystal-detector distance: 33 cm

Scan mode: coupled θ (crystal)- 2θ (counter)

Scan speed: $1^\circ/\text{min}$

Range: $2.7 < 2\theta < 50^\circ$

Scan length: from $2\theta(K\alpha_1) - 1^\circ$ to $2\theta(K\alpha_2) + 1^\circ$

Background measurement: stationary crystal, stationary counter; 20 s at beginning and end of each 2θ scan

Std reflections: three remeasured every 97 reflections; an isotropic linear decay correction was applied to compensate for a 6% intensity loss owing to decomposition over the 140-h data collection period

Reflections collected: 2194 including two or more equivalent forms averaged^b to give 989 unique data with $R_{F_o^2}(\text{av}) = 0.057$

(C) Reduction of Intensity Data

Conversion to F_o^2 and $\sigma(F_o^2)$: as in ref 3 with $\epsilon = 0.05$

Absorption coeff: $\mu = 52.2 \text{ cm}^{-1}$; minimum and maximum transmission coefficients were 0.71 and 0.80, respectively^c

^a Data were collected and reduced with crystal parameters corresponding to $C2/m$. Final refinement and all reported results refer to the nonstandard setting $I2/m$. ^b Using AVERAGE by J. T. Gill. ^c Absorption corrections were carried out using a local version of ORABS; the Wehe-Busing-Levy program, adapted to the Picker FACS geometry.

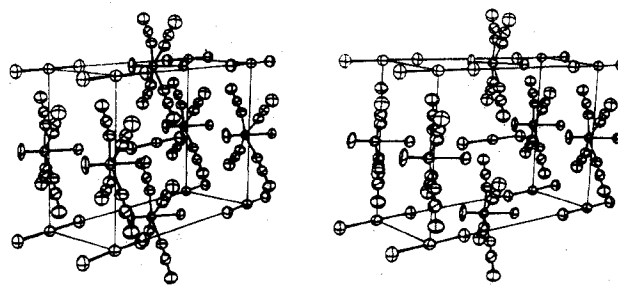


Figure 1. Stereoscopic view of the unit cell packing. The cation is shown as ordered for the purpose of this illustration (see text).

was applied. An attempt to locate and refine the hydrogen atoms was unsuccessful. A final difference Fourier map showed residual electron density of $\sim 1.51 \text{ e}/\text{\AA}^3$ near the triiodide ion and less than $0.77 \text{ e}/\text{\AA}^3$ elsewhere.

Final parameters are given in Table II and the molecular geometry is summarized in Table III. A listing of observed and calculated structure factor amplitudes and a table of root-mean-square amplitudes of vibration are available.⁶ The unit cell packing is shown in Figure 1, and the molecular geometry and atom labeling scheme, in Figure 2.

Discussion

The coordination geometry of the $[\text{MoOCl}(\text{CNCH}_3)_4]^+$ cation has the chloride and oxygen atoms in *trans* positions, as suggested previously.¹ The site symmetry is close to C_{4v} , with the molybdenum atom displaced toward the oxygen by 0.33 \AA from the plane defined by the four coordinated methyl