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The Crystal and Molecular Structure of *trans*-cyano triethylenetetramine Cobalt(III) Perchlorate

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Received October 11, 1972

The crystal and molecular structure of trans-cyanotriethylenetetramine cobalt(III) perchlorate has been determined from three-dimensional X-ray diffraction data collected on a four-circle diffractometer with scintillation counter. The compound crystallizes in space group $P2_1/n$ with $a = 9.85$, $b = 22.35$, $c = 6.68$ Å, $\beta = 100.9°$, and $Z = 4$. The structure was solved by conventional Patterson and Fourier techniques and full matrix least squares refinement of 3314 observed reflections ($F_{obs} > 3\sigma_F$) gave a final discrepancy factor of 0.123. The complex has a distorted octahedral configuration with the trien ligand equatorially coordinated. The bonding mode of the CN group is through the carbon.

Introduction

The bonding mode of cyanide to cobalt has been the subject of considerable controversy in recent years. The energy difference between the cyano and isocyano coordinations is small enough to permit either to occur.[1] However, neutron diffraction studies of cyano-cobalt complexes[2,3,4] have regularly shown that the cyanide group bonds through carbon. Since it is ofttimes difficult to distinguish between atoms which differ by but one electron by X-ray diffraction, bonding via the carbon has sometimes been assumed.[5]

Kuroda and Gentile[6] recently have prepared what they characterize as an isocyano cobalt complex, *cis*-α-isocyano-triethylenetetrammine cobalt(III) perchlorate. Their assignment of the isocyano coordination is based on visible and ultraviolet spectroscopy. Because there is some doubt as to the correctness of this assignment,[7] it was decided to carry out an X-ray structure determination of this compound.

Experimental Section

A sample of the compound, prepared by the method of Kuroda and Gentile,[6] was kindly supplied by Dr. James H. Espenson. Amber-colored crystals were obtained by recrystallization from an aqueous solution. High resolution infrared spectra of the initial sample and of the recrystallized material, both in KBr pellets, were virtually identical, indicating that isomerization had not occurred during solution or recrystallization. Microscopic examination revealed that the crystals have sharply defined faces and are needle-like in appearance with a distorted hexagonal gross section in which alternate sides are of unequal lengths.

Crystals were selected and mounted on a glass fiber with Duco cement thinned with amyl acetate. Preliminary Weissenberg and precession photographs exhibited 2/m Laue symmetry, indicating a monoclinic space group. The following systematic absences were observed: $h0\ell$ when $h+\ell = 2n+1$, and $0k0$ when $k = 2n+1$. These absences are only consistent with the space group $P2_1/n$. The unit cell parameters at 25°C are $a = 9.8544 \pm 0.0015$, $b = 22.3529 \pm 0.0045$, $c = 6.6766 \pm 0.0020$ Å and $\beta = 100.85 \pm 0.03°$. These parameters and their standard deviations were obtained by a least squares fit to the 2θ values of twelve independent reflections whose centers were determined by top-bottom, left-right beam splitting on a previously aligned Hilger-Watts four-circle diffractometer (MoKα radiation, $\lambda = 0.71069$ Å). Any error in the instrumental zero was eliminated by centering the reflection at both $+2\theta$ and -2θ. A calculated density of 1.62 g/cc for four molecules per unit cell agrees quite well with an observed density of 1.65±0.01 g/cc, which was determined by flotation techniques.

For data collection, a crystal was selected having approximate dimensions $0.02 \times 0.08 \times 0.18$ mm along the a, b, and c crystal axes, respectively and was mounted such that the c axis coincided with the φ axis of the diffractometer. Data were collected at room temperature using a Hilger-Watts four-circle diffractometer interfaced to an S.D.S. 910 computer in a real time mode, equipped with a scintillation counter, and using Zr-filtered MoKα radiation. Whithin a two-theta sphere of 60° ($\sin\theta/\lambda = 0.704$ Å^{-1}), all data in the $hk\ell$ and $h\bar{k}\ell$ octants were recorded using the θ-2θ scan technique with a take-off angle of 5.5°. Symmetric scan ranges of 1.2° in 2θ at low two-theta values to 2.0° at large two-theta values were used. Stationary-crystal, stationary-counter background counts of half the scan time were

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(1) J.P. Birk and J.H. Espenson, *J. Amer. Chem. Soc.*, *90*, 2266-2277 (1968).

(2) N.A. Curry and W.A. Runciman, *Acta Cryst.*, *12*, 674-678 (1959).

(3) H.U. Guedel, A. Ludi, P. Fischer, and W. Haelg, *J. Chem. Phys.*, *53*(5), 1917-1923 (1970).

(4) W. Ozbirn and R.A. Jacobson, *Inorg. Chem. Acta*, *4*, 377-382 (1970).

(5) D. Britton and C. Mahon, *Inorg. Chem.*, *10*(3), 586-589 (1971).

(6) K. Kuroda and P. Gentile, *Inorg. Nucl. Chem. Letters*, *3*, 151-156 (1967).

(7) K. Konya, H. Nishikawa, and M. Shibita, *Inorg. Chem.*, *7*, 1165-1168 (1968).

taken at the beginning and end of each scan. A counting rate of 0.4096 seconds per step of 0.01° in θ was employed. A total of 5081 reflections were measured in this way.

As a general check on electronic and crystal stability, the intensities of three standard reflections were remeasured periodically during the data collection period. These reflections did not vary to any significant degree during the entire period of data collection.

Based on a linear absorption coefficient of $\mu = 14.982\ \mathrm{cm}^{-1}$, the maximum and minimum transmission factors[8] were 87.86% and 85.96% respectively, and no absorption correction was deemed necessary.

The intensity data were corrected for Lorentz-polarization effects. The estimated error in each intensity was calculated by

$$\sigma_I^2 = C_T + C_B + (0.03C_I)^2 + (0.03C_B)^2$$

where C_T, C_I, and C_B are the total count, the net count and the background count respectively. The factor 0.03 represents an estimate of non-statistical errors. The estimated deviations in the structure factors were calculated by the finite difference method.[9] Of the 4678 independent reflections, 3314 were considered observed ($> 3\sigma_F$).

Solution and Refinement

The positions of the cobalt and chlorine atoms were obtained from analysis of a sharpened Patterson function.[10] The remaining non-hydrogen atoms were found by successive structure factor and electron density map calculations.[11] These atomic positions were then refined by a full-matrix least squares procedure, minimizing the function $\Sigma w(|F_o| - |F_c|)^2$ where $w = 1/\sigma_F^2$ to a conventional discrepancy factor of $R = \Sigma \| F_o| - |F_c\| / \Sigma |F_o| = 0.170$. The scattering factors used were those of Hanson, *et al.*[12] with the cobalt and chlorine scattering factors modified for the real and imaginary parts of anomalous dispersion.[13]

An electron density difference map verified that all the non-hydrogen atoms had been accounted for, but indicated that some anisotropic motion, particularly of the cobalt and chlorine atoms, was present. Accordingly, anisotropic refinement was begun and after six cycles of refinement, values of R and ωR ($= [\Sigma w(|F_o| - |F_c|)^2 / \Sigma w |F_o|^2]^{1/2}$) of 0.154 and 0.169, respectively, were obtained. The positions of the hydrogens bonded to carbons in the triethylenetetramine ligand were calculated with an H-C-C angle of 109.5° and a C-H distance of 1.07 Å. The hydrogen atoms were assigned isotropic thermal parameters of 3.0 Å². Two cycles of refinement with anisotropic thermal parameters for all non-hydrogen atoms, varying only the positional and thermal parameters of the non-hydrogen atoms and the overall scale factor, followed by a recalculation of the hydrogen atom positions and two further cycles of least squares refinement, yielded values for R and ωR of 0.123 and 0.156, respectively.

A final electron density difference map showed no peaks greater than 1.2 e/Å³. A final statistical analysis of the F_o and F_c values as a function of the scattering angle and magnitude of F_o revealed no unusual trends and suggests that the relative weighting scheme used is a reasonable one.

Temperature factors of the atoms in the isocyanide group (assuming bonding via the nitrogen) indicated the assignment of carbon and nitrogen should be reversed. To further verify this, the atom multipliers of these atoms were allowed to vary under two sets of conditions. In the first of these, carbon scattering factors were assigned to C(7) and C(8) and nitrogen scattering factors were assigned to N(5) and N(6). In the second instance, the scattering factor assignments were reversed. All four atom multipliers were initially set at a value of 1.00. The results of these refinements are shown in Table I, and clearly indicate the correct assignments.

The final positional and thermal parameters for the non-hydrogen atoms are listed in Table II. The standard deviations were calculated from the inverse matrix of the final least squares refinement cycle. The calculated hydrogen atom positions are listed in Table III. Bond lengths and bond angles, and significant non-bonded distances are listed in Tables IV and V, respectively, along with their standard deviations.[14] The final values of the observed and calculated structure factors are listed in Table VI.

Shortly after the data had been collected and the crystal removed from the diffractometer, the instrument was found to be slightly misaligned. Thus the data are somewhat poorer than might be expected. However, since all the stereochemical features of interest were well determined and the electron density difference map was rather featureless, the data were not retaken.

Description of the Structure

Our results indicate that the bonding of cyanide to the cobalt is through the carbon and not the nitrogen, and that these groups are *trans* and not *cis* to one another contrary to the predictions of Kuroda and Gentile.[6] Hence, the compound is *trans*-dicyanotriethylenetetramine cobalt(III) perchlorate, and is illustrated in Figure 1.[15] This configuration has been

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(8) D.J. Wehe, W.R. Busing, and A. Levy, « ORABS, A Fortran Program for Calculating Single Crystal Absorption Corrections », Oak Ridge National Laboratory USAEC Report ORNL-TM-229, April, 1962.

(9) S.L. Lawton and R.A. Jacobson, *Inorg. Chem.*, *7*, 2124-2134 (1968).

(10) R.A. Jacobson, J.A. Wunderlich, and W.N. Lipscomb, *Acta Cryst.*, *14*, 598-607 (1961).

(11) The programs used to compute the electron density and Patterson maps were ALF (J. Rodgers and R.A. Jacobson, « ALF: A General Fourier Program in PL 1 for Triclinic, Monoclinic, and Orthorhombic Space Groups », Ames Laboratory USAEC Report, IS-2155, July, 1969) and ALFF (C.R. Hubbard, C.O. Quicksall and R.A. Jacobson, « The Fast Fourier Algorithm and the Programs ALFF, ALFFDP, ALFFPROJ, ALFFT, and FRIEDEL », Ames Laboratory USAEC Report, IS-2625, June, 1971). A local modification of OR FLS (W.R. Busing, K.O. Martin, and H.A. Levy, « OR FLS, A Fortran Crystallographic Least Squares Program », Oak Ridge National Laboratory USAEC Report, ORNL-TM-305, August, 1962) was used to compute structure factors and to refine the structure by the method of least squares.

(12) H.P. Hanson, F. Herman, J.D. Lea, and S. Skillman, *Acta Cryst.*, *17*, 1040-1044 (1960).

(13) D.H. Templeton, « International Tables for X-ray Crystallography », Vol .III, pp. 215-216, Table 3.3.2c, The Knoch Press, Birmingham, England (1962).

(14) W.R. Busing, K.O. Martin, and H.A. Levy, « OR FFE, A Fortran Crystallographic Function and Error Program » Oak Ridge National Laboratory, USAEC Report, ORNL-TM-306, March, 1964.

(15) C.A. Johnson, « OR TEP: A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations », Oak Ridge National Laboratory USAEC Report, ORNL-3794 (2nd Revision), June, 1970.

Table I. Determination of the bonding mode of cyanide in *trans*-$(CN)_2$-trien $Co^{III}ClO_4$.

Atom	Assigned Scattering Factor Table	Atom Multiplier Actual	Theoretical Co C-N	Theoretical Co-N-C	R	wR
C(7)	Carbon	1.033	1.0	1.17		
C(8)	Carbon	1.034	1.0	1.17		
N(5)	Nitrogen	1.026	1.0	0.86		
N(6)	Nitrogen	1.046	1.0	0.86	12.4	12.4
C(7)	Nitrogen	0.823	0.86	1.0		
C(8)	Nitrogen	0.825	0.86	1.0		
N(5)	Carbon	1.293	1.17	1.0		
N(6)	Carbon	1.314	1.17	1.0	15.8	15.8

Table II. Final positional and anisotropic thermal parameters of non-hydrogen atoms in *trans*-$(NC)_2$-trien $Co^{III}ClO_4$ [a,b].

Atom	x	y	z	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
Co	0.6629(1)	0.3488(1)	0.6552(2)	55.9(1.4)	9.2(0.2)	169.9(3.6)	1.1(0.5)	13.9(1.6)	0.7(0.9)
Cl	0.6616(3)	0.1289(1)	0.6466(6)	79.7(3.0)	15.6(0.6)	306.3(9.7)	3.6(1.1)	12.7(4.3)	6.1(2.2)
O(1)	0.6601(9)	0.0863(4)	0.4868(15)	139(12)	21(2)	360(32)	10(4)	-26(16)	11(7)
O(2)	0.5498(9)	0.1700(4)	0.5928(16)	121(12)	30(2)	486(39)	32(4)	-79(17)	43(8)
O(3)	0.7893(8)	0.1635(4)	0.6748(15)	92(10)	26(2)	386(32)	-5(4)	-7(14)	13(7)
O(4)	0.6586(11)	0.0988(5)	0.8283(16)	204(17)	34(3)	333(33)	-.2(5.6)	90(19)	-3(9)
N(1)	0.4727(8)	0.3159(4)	0.5898(14)	69(9)	14(2)	198(24)	-9(3)	2(12)	10(6)
N(2)	0.5927(8)	0.4122(3)	0.8075(12)	77(9)	11(2)	148(21)	3(3)	11(11)	4(5)
N(3)	0.8378(8)	0.3891(4)	0.7368(15)	66(9)	14(2)	224(26)	-4(3)	23(13)	-9(6)
N(4)	0.7579(9)	0.2898(4)	0.5051(13)	97(11)	14(2)	197(25)	8(4)	39(13)	7(6)
C(7)	0.6226(10)	0.3942(5)	0.4078(18)	68(11)	12(2)	205(30)	3(4)	17(15)	-.8(6.7)
C(8)	0.6997(10)	0.3013(4)	0.8952(18)	63(11)	9(2)	250(33)	-.7(3.5)	31(15)	-7(7)
C(1)	0.3914(11)	0.3494(6)	0.7122(20)	77(12)	22(3)	274(36)	-7(5)	15(17)	-.5(9)
C(2)	0.4392(11)	0.4144(5)	0.7393(20)	62(11)	20(3)	313(39)	10(4)	39(17)	15(9)
C(3)	0.6694(12)	0.4679(5)	0.7878(19)	93(13)	12(2)	280(37)	-4(4)	3(17)	-11(8)
C(4)	0.8173(13)	0.4546(5)	0.7531(24)	118(17)	16(3)	388(47)	-7(5)	11(22)	-19(9)
C(5)	0.9274(12)	0.3720(6)	0.6003(21)	81(13)	26(3)	289(40)	-5(5)	42(19)	-15(10)
C(6)	0.9102(13)	0.3046(6)	0.5589(23)	105(16)	26(3)	407(50)	18(6)	96(23)	14(11)
N(5)	0.6027(11)	0.4174(5)	0.2488(19)	116(13)	19(2)	349(38)	3(4)	27(18)	9(8)
N(6)	0.7204(10)	0.2762(4)	1.0540(15)	115(12)	16(2)	230(28)	3(4)	28(15)	5(7)

[a] In this and subsequent tables, numbers in parentheses represent standard deviations in the least significant digits. [b] Anisotropic thermal parameters are defined by: $T = \exp[-h^2\beta_{11}+k^2\beta_{22}+l^2{}_{33}+2hk\beta_{12}+2hl\beta_{23}+2hl\beta_{13})]$.

Table III. Calculated Hydrogen Atomic Positions in *trans*-$(CN)_2$-trien $Co^{III}ClO_4$.

Atom	x	y	z
H(AC1)	0.4041	0.3289	0.8624
H(BC1)	0.2834	0.3479	0.6404
H(AC2)	0.3934	0.4374	0.8519
H(BC2)	0.4113	0.4386	0.5960
H(AC3)	0.6762	0.4947	0.9921
H(BC3)	0.6135	0.4932	0.6569
H(AC4)	0.8303	0.4765	0.6143
H(BC4)	0.8906	0.4724	0.8798
H(AC5)	1.0339	0.3827	0.6673
H(BC5)	0.8990	0.3961	0.4566
H(AC6)	0.9606	0.2912	0.4366
H(BC6)	0.9593	0.2799	0.6960

anticipated by Konya, Nishikawa, and Shibita[7] on the basis of spectroscopic examination of similar compounds. The *trans* configuration is also consistent

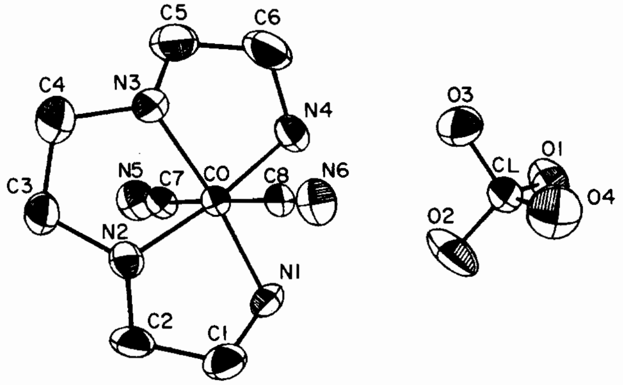

Figure 1. A formula unit of *trans*-dicyano triethylne tetramine cobalt(III) perchlorate.

Table IV. Selected Bond Distances and Angles in Angstroms and Degrees for *cis*-$(CN)_2$-trien $Co^{III}ClO_4$.

Cl-O(1)	1.427(9)	C(8)-N(6)	1.180(13)
Cl-O(2)	1.424(8)	N(1)-C(1)	1.462(15)
Cl-O(3)	1.456(9)	N(2)-C(2)	1.488(12)
Cl-O(4)	1.392(10)	N(2)-C(3)	1.476(13)
Co-N(1)	1.978(8)	N(3)-C(4	1.484(14)
Co-N(2)	1.951(8)	N(3)-C(5)	1.444(15)
Co-N(3)	1.922(8)	N(4)-C(6)	1.506(15)
Co-N(4)	2.001(8)	C(1)-C(2)	1.527(16)
Co-C(7)	1.909(12)	C(3)-C(4)	1.551(17)
Co-C(8)	1.894(12)	C(5)-C(6)	1.536(18)
C(7)-N(5)	1.161(14)		
O(1)-Cl-O(2)	110.7(6)	N(4)-Co-C(8)	91.5(4)
O(1)-Cl-O(3)	109.1(6)	C(7)-Co-C(8)	177.9(4)
O(1)-Cl-O(4)	109.3(6)	Co-N(1)-C(1)	106.7(6)
O(2)-Cl-O(3)	107.1(6)	Co-N(2)-C(2)	108.1(7)
O(2)-Cl-O(4)	112.3(7)	Co-N(2)-C(3)	108.9(7)
O(3)-Cl-O(4)	108.4(6)	Co-N(3)-C(4)	110.8(7)
N(1)-Co-N(2)	88.0(4)	Co-N(3)-C(5)	109.0(7)
N(1)-Co-N(3)	172.5(4)	Co-N(4)-C(6)	106.3(7)
N(1)-Co-N(4)	99.4(4)	Co-C(7)-N(5)	173.7(10)
N(1)-Co-C(7)	89.2(4)	Co-C(8)-N(6)	174.1(9)
N(1)-Co-C(8)	89.2(4)	N(1)-C(1)-C(2)	111.3(9)
N(2)-Co-N(3)	84.5(4)	N(2)-C(2)-C(1)	105.9(8)
N(2)-Co-N(4)	172.6(4)	N(2)-C(3)-C(4)	111.4(8)
N(2)-Co-C(7)	91.8(4)	N(3)-C(4)-C(3)	110.2(9)
N(2)-Co-C(8)	89.6(4)	N(3)-C(5)-C(6)	108.3(10)
N(3)-Co-N(4)	88.1(4)	N(4)-C(6)-C(5)	109.1(9)
N(3)-Co-C(7)	90.7(4)	C(2)-N(2)-C(3)	116.8(8)
N(3)-Co-C(8)	91.0(4)	C(4)-N(3)-C(5)	114.5(10)
N(4)-Co-C(7)	87.3(4)		

Table V. Significant Non-bonded Distances in *trans*-$(CN)_2$-trien $Co^{III}ClO_4$ [a].

C(7)-H(BC3)	2.780(10)
C(7)-H(AC4)	2.884(10)
C(8)-O(2)	2.694(13)
C(8)-O(3)	3.601(13)
N(4)-O(3)	3.034(12)
O(1)-H(AC5)′	2.34(1)
O(4)-H(BC1)′	2.49(1)

[a] Primed atoms indicate atoms in another molecule.

with an exceedingly sharp band for the C-N stretching frequency at 2145 cm^{-1}, with no evidence of splitting.

The cyanide groups are within a degree of being perpendicular to the nitrogens of the trien ligand (Table IV), and the variations from an ideal octahedral configuration displayed by the N-Co-N angles appear to simply reflect the restrictions imposed by the quadradentate ligand (Figure 2). This trien ligand coordinated to cobalt in a *cis* configuration has been investigated by Freeman and Maxwell,[16] and Dwyler and Maxwell;[17] their results have been used as the basis of many of the comparisons which follow.

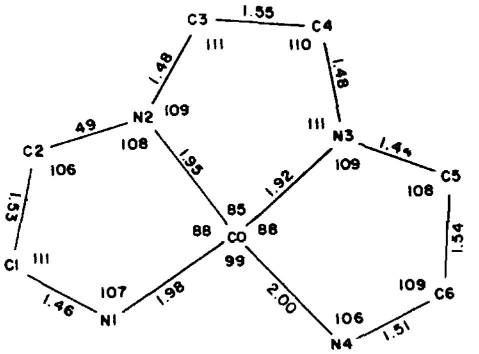

Figure 2. Bond distances and angles within the trien ligand.

The N(1)-Co-N(2) and N(3)-Co-N(4) angles of 88.0 and 88.1° are reasonable and consistent with similar results obtained in other investigations of the trien ligand. A slight compression of the Co-N(2)-C(3)-C(4)-N(3) ring due to the equatorial coordination of the ligand adequately explains the somewhat small value of 84.5° for the N(2)-Co-N(3) angle. Hydrogen-hydrogen repulsions would be expected to produce an increase in the N(1)-Co-N(4) angle compared to the nominal 90°, and the 99° angle found is quite reasonable. A similar result was obtained during the investigation of a cobalt compound containing a homologous ligand.[18] The bond angles within the trien ligand, with the exception of the C-N-C angles, do not differ from the expected tetrahedral angles by more than three standard deviations. In addition, they fall within two standard deviations of the range of values of 108.4° to 112.3° for Co-N-C bonds, and of 105.2° to 111.4° for N-C-C bonds reported by other investigators of this ligand. The C-N-C bond angles of 116.8° and 114.6° are also reasonable in view of the fact that the Co-N-C angles are, on the average, somewhat less than tetrahedral. These C-N-C angles also agree within three standard deviations with other results obtained in investigations of this ligand.

The bond distances within the trien ligand range from 1.92 to 2.00 Å for Co-N bonds, from 1.44 to 1.51 Å for N-C bonds and from 1.53 to 1.55 Å for C-C bonds, with average values of 1.96, 1.48 and 1.54 Å, respectively. These distances in no instance differ significantly from previously reported values for other compounds involving this ligand.

The four trien nitrogens and the cobalt atom all fall within 0.01 Å of the least-squares plane. The three five-membered rings formed by the quadradentate ligand and the cobalt atom are not planar how-

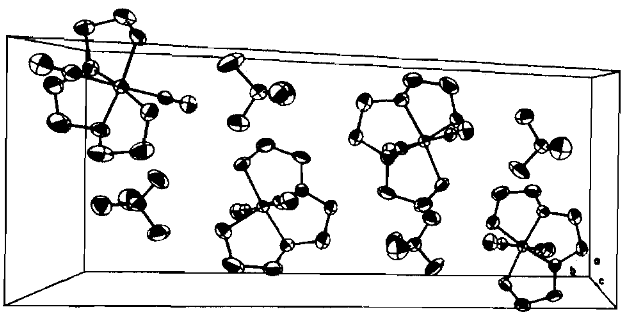

Figure 3. Unit cell showing packing of *trans*-dicyano triethylene tetramine cobalt(III) perchlorate.

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[16] H.C. Freeman and I.E. Maxwell, *Inorg. Chem.*, *8(6)*, 1293-1298 (1969).

[17] M. Dwyler and I.E. Maxwell, *Inorg. Chem.*, *9(6)*, 1459-1464 (1970).

[18] J.W. Turley and R.G. Asperger, *Inorg. Chem.*, *10(3)*, 558-565 (1971).

Table VI. Observed and calculated structure factors.

19	0	14	15
19	1	7	9
19	2	36	35
19	3	5	-4
19	4	25	-36
19	5	7	-8
20	0	13	14
20	1	24	25
20	2	7	6
20	3	7	-6
20	7	6	8
21	0	17	17
21	1	12	10
21	2	11	11
21	3	13	-14
21	4	12	-16
21	5	11	13
22	0	34	33
22	1	9	-9
22	2	26	-28
22	4	11	-10
22	6	9	12
23	0	21	-22
23	1	6	-8
23	2	27	-27
23	2	28	-27
23	3	5	-4
23	6	27	35
24	0	29	-29
24	1	6	-9
24	2	17	18
24	3	6	6
24	6	7	-8
25	1	5	4
25	2	8	-9
25	5	17	16
26	0	17	-17
26	1	17	17
26	2	14	14
26	3	7	-5
26	5	6	-7
27	0	18	16
27	2	20	21
27	3	8	-10
27	4	16	-20
28	0	21	22
28	2	24	-24
28	3	8	-7
29	0	12	-10
29	2	8	-6
30	1	11	-11
31	0	6	-6
31	1	8	3

H = 0

K	L	FO	FC
0	2	83	-81
0	4	47	-42
0	6	28	24
1	1	10	11
1	2	153	-146
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21	2	6	5
21	3	8	12
21	4	17	-15
21	5	7	-8
22	1	26	29
22	5	20	-20
23	0	6	5
23	0	6	5
23	3	24	-24
23	4	8	-6
23	5	23	18
24	1	16	-18
24	2	9	-9
24	3	9	9
25	0	18	-17
25	2	7	1
25	4	11	13
26	0	13	-12
26	0	14	-12
26	1	13	-13
26	2	15	11
26	3	7	7
27	0	7	8
27	1	7	5
27	2	13	10
27	3	9	12
28	1	16	20
28	1	19	20
28	2	8	-1
30	0	8	7

H = 5

K	L	FO	FC
0	1	105	105
0	3	16	-21
0	5	20	-29
0	7	7	13
1	0	20	-19
1	1	15	17
1	2	32	36
1	3	48	-44
1	4	17	-24
1	5	9	12
1	7	10	12
2	0	48	40
2	1	7	5
2	2	59	-64
2	3	5	-9
2	4	5	-9
2	5	10	12
3	0	47	-43
3	1	34	31
3	2	43	-45
3	3	22	-21
3	4	30	33
3	5	8	10
3	7	8	7
4	0	35	-34
4	0	34	-34
4	1	90	-85
4	2	15	12
4	3	13	16
4	4	23	25
4	5	26	28
4	6	12	-12
4	7	10	-10
5	0	62	76
5	1	11	-11
5	2	7	10
5	3	72	71
5	4	16	-17
5	6	16	-13
5	7	13	-11
6	0	4	-4
6	1	36	32
6	3	10	11
6	4	11	-10
6	5	31	-31
6	6	9	-9
6	7	11	10
7	0	24	22
7	1	16	-16
7	1	15	-16
7	3	9	-9
7	4	10	-12
8	0	33	33
8	1	39	36
8	2	9	-6
8	3	6	-7
8	4	10	-10
8	5	18	-14
8	6	31	27
8	7	12	9
9	0	25	-26
9	1	12	13
9	2	43	-39
9	3	39	-36
9	4	17	15
9	5	19	15
9	7	12	7
10	0	44	-42
10	1	46	-44
10	2	13	11
10	4	7	7
10	5	35	28
10	7	20	-14
11	0	14	-14
11	1	22	-22
11	2	19	19
11	3	9	8
11	5	19	-13
11	7	6	-6
12	0	18	-16
12	2	20	19
12	3	5	-1
12	4	8	8
12	6	22	-18
13	0	6	4
13	2	43	40
13	3	27	24
13	4	32	-27
13	5	6	-6
13	6	6	-1
14	0	47	45
14	1	28	26
14	2	34	-34
14	2	35	-34
14	4	15	-13
14	5	18	-14
14	6	16	12
15	1	14	13
15	2	19	-19
15	3	15	-13
15	3	15	-13
15	4	6	7
15	5	14	10
16	0	10	-9
16	1	13	-13
16	3	8	7
16	6	6	4
17	1	11	-11
17	2	27	-26
17	3	7	1
17	4	30	24
17	5	6	-3
18	0	38	-38
18	1	19	-21
18	2	29	28
18	4	14	13
18	6	17	-14
19	0	8	6
19	2	17	19
19	3	21	19
19	4	23	-21
20	0	12	11
20	1	16	14
20	2	7	-6
21	0	6	2
21	2	17	17
21	3	20	-20
21	4	12	-11
21	5	6	4
22	0	29	31
22	2	10	-10
22	4	10	-11
23	0	16	-18
23	0	18	-18
23	1	7	7
23	2	16	-16
23	4	18	17
24	0	24	-25
24	1	8	-7
24	2	12	10
25	1	7	-1
25	3	7	9
26	0	17	-17
26	0	16	-17
26	1	12	14
26	2	9	8
27	0	9	9
27	1	6	7
27	2	13	14
28	0	18	19
28	1	6	-2

H = 6

K	L	FO	FC
0	0	77	77
0	2	17	-19
0	4	31	-40
0	6	9	13
1	0	19	18
1	1	4	-6
1	2	46	-52
1	3	16	-19
1	4	18	21
1	6	11	13
2	0	8	5
2	2	7	7
2	4	14	19
3	0	39	39
3	2	30	-30
3	3	16	16
3	4	9	10
3	7	6	-5
4	0	72	-69
4	0	72	-69
4	1	9	2
4	2	4	2
4	3	9	11
4	4	30	27
4	5	13	-11
4	6	19	-17
5	0	9	-9
5	1	17	17
5	2	55	50
5	4	15	-14
5	5	6	-6
5	6	14	-13
6	0	48	48
6	1	4	2
6	2	9	-7
6	3	7	-5
6	4	22	-22
6	5	6	-3
6	6	10	11
6	7	7	2
7	0	7	-9
7	1	15	14
7	3	4	-2
7	4	7	6
7	7	9	6
8	0	45	44
8	1	47	-46
8	2	20	-20
8	3	6	-5
8	4	16	-12
8	5	23	20
8	6	9	8
9	0	7	-7
9	1	58	-57
9	2	38	-36
9	3	15	11
9	4	5	3
9	5	7	4
9	6	15	15
10	0	56	-56
10	1	11	9
10	2	12	12
10	3	22	20
10	4	22	21
10	5	6	-6
10	6	18	-16
11	0	7	-7
11	2	34	33
11	3	8	-7
11	4	11	-12
12	0	12	-10
12	1	24	25
12	2	21	22
12	4	6	-4
12	5	25	-21
13	0	8	11
13	1	35	33
13	2	15	14
13	3	25	-27
13	4	13	-8
13	6	10	-6
14	0	15	15
14	1	24	-23
14	3	18	-16
14	4	23	-21
14	5	19	15
14	6	14	9
15	1	23	-22
15	2	30	-31
15	3	11	9
15	3	11	9
15	4	20	18
15	6	10	8
16	0	14	-14
16	2	6	10
16	3	7	-7
16	4	15	14
16	5	12	10
16	6	10	-7
17	1	11	-11
17	3	34	33
17	4	7	2
18	0	34	-8
18	1	26	27
18	2	15	-14
18	3	13	11
18	5	22	-18
19	1	12	12
19	2	13	13
19	3	27	-27
19	4	12	-11
20	0	20	21
20	1	11	-9
20	3	7	2
20	4	10	-8
21	1	12	13
21	2	6	-5
21	3	14	-12
22	0	6	-9
22	1	16	-16
22	3	11	-11
23	1	12	-12
23	3	21	20
24	0	12	-13
24	1	18	19
24	1	18	19
25	2	9	6
25	3	7	-5
26	0	16	16
26	0	15	16
26	1	11	11
27	1	15	15

H = 7

K	L	FO	FC
0	1	11	-15
0	3	23	-31
0	5	12	16
1	0	9	-7
1	1	41	-45
1	3	17	23
1	4	5	0
1	7	13	-12
2	0	5	6
2	1	13	-12
2	3	10	14
2	5	6	-9
2	6	6	-5
3	0	12	-13
3	1	25	-28
3	4	10	-10
3	5	7	7
3	6	8	-4
4	0	17	16
4	1	18	18
4	2	4	2
4	3	35	38
4	4	5	-7
4	5	16	-17
5	0	28	27
5	1	66	65
5	3	27	-30
5	4	14	11
5	5	12	-14
5	6	7	7
6	1	16	-16
6	3	27	-27
6	4	5	-1
6	5	17	17
7	0	30	30
7	1	11	-9
7	2	20	-20
7	3	8	12
7	6	7	6
8	0	37	-37
8	1	13	-13
8	3	21	-20
8	4	6	4
8	5	6	4
8	6	7	-6
9	0	31	-30
9	1	64	-64
9	2	22	19
9	3	20	18
9	5	18	16
9	6	10	-6
10	1	28	29
10	2	23	23
10	3	27	26
10	4	12	-10
10	5	21	-18
10	6	9	5
11	0	10	-10
11	1	39	41
11	2	16	17
11	4	5	-1
11	6	7	-7
12	0	30	30
12	1	6	1
12	2	7	5
12	3	7	-5
12	4	27	-23
12	5	9	6
13	0	12	10
13	1	19	19
13	2	40	-40
13	3	9	-9
13	5	9	-7
13	6	13	10
14	0	15	-14
14	2	8	-9
14	3	18	-19
14	4	29	24
14	5	18	15
15	1	29	-30
15	2	17	14
15	3	16	17
15	3	16	17
15	4	6	6
15	4	8	6
16	1	10	11
16	2	17	-16
16	3	9	7
16	4	13	15
16	5	11	-8
17	0	12	-13
17	2	24	27
18	0	31	30
18	0	30	30
18	1	5	-4
18	4	24	-23
19	0	15	15
19	1	8	9
19	2	23	-23
19	3	12	-11
20	0	9	-8
20	0	8	-3
20	1	10	-6
20	2	9	7
20	3	11	-9
20	4	6	3
21	0	10	9
21	2	12	-10
21	3	12	11
22	0	25	-25
22	1	7	5
22	3	7	5
23	0	17	-19
23	2	23	22
24	0	19	20
24	1	7	6
25	0	6	-3
25	1	7	8
26	0	11	11
26	0	9	11

H = 8

K	L	FO	FC
0	0	34	-34
0	2	36	-36
0	4	27	33
1	0	49	-47
1	1	5	3
1	2	23	26
1	4	10	15
1	6	13	-18
2	1	6	9
2	2	15	15
2	3	13	16
2	6	12	-17
3	0	25	-25
3	1	5	3
3	2	5	4
3	4	16	15
3	5	6	-7
4	0	20	19
4	1	22	23
4	2	41	43
4	3	11	-11
4	4	21	-23
4	5	8	11
5	0	67	64
5	0	66	64
5	1	10	-8
5	2	38	-38
5	4	15	-18
5	6	18	17
6	0	16	-17
6	2	35	-32
6	3	15	-15
6	4	22	22
6	5	7	4
7	0	15	-15
7	1	27	-27
7	2	10	11
7	4	6	-6
7	5	14	12
8	1	8	-7
8	2	18	-18
8	3	17	13
8	3	16	13
8	4	8	7
8	5	10	-7
9	0	51	-52
9	1	31	30
9	2	38	35
9	3	5	-5
9	4	18	17
9	5	13	-13
10	0	16	16
10	2	30	30
10	3	11	-11
10	4	24	-20
11	0	25	26
11	1	18	19
11	2	14	-16
11	5	7	-7
12	1	13	15
12	2	6	-6
12	3	17	-17
12	5	10	6
13	0	16	17
13	1	37	-37
13	2	8	-6
13	5	16	12
14	1	8	-7
14	2	21	-22
14	3	21	21
14	4	21	21
15	0	25	-26
15	1	5	7
15	2	11	10
16	1	7	-7
16	3	10	12
16	4	7	-9
17	1	24	25
17	2	7	-7
18	0	9	8
18	0	8	6
18	1	10	9
18	2	6	7
18	3	26	-24
18	4	6	-8
19	0	14	14
19	1	14	-13
20	0	7	-3
20	1	6	-6
20	2	7	-9
20	3	6	6
21	0	18	-18
21	1	8	-9
21	2	8	6
22	1	[illegible]	-9
23	1	[illegible]	20
24	[illegible]	7	-1

H = 9

K	L	FO	FC
0	1	33	-32
0	3	30	33
1	0	9	9
1	1	34	26
1	3	14	17
1	4	8	10
1	5	13	-18
2	0	9	8
2	1	13	13
2	2	13	11
2	3	10	-13
2	4	9	-8
3	1	9	12
3	3	8	9
3	4	14	-13
3	5	10	-10
4	0	6	4
4	1	26	26
4	2	14	-14
4	3	28	-29
4	4	12	12
4	5	6	1
5	0	20	-20
5	0	20	-20
5	1	30	-29
5	5	18	-18
5	5	23	20
6	1	19	-17
6	1	20	-17
6	2	7	-7
6	3	23	22
6	4	5	1
7	0	15	-16
7	1	10	12
7	2	7	4
7	4	10	8
8	0	7	-7
8	1	13	-15
8	2	16	16
8	3	9	6
8	3	10	8
9	0	27	28
9	1	19	20
9	3	10	10
9	4	12	-13
9	5	17	-14
10	1	21	21
10	2	17	-15
10	3	14	-14
11	1	17	-16
11	4	6	1
11	5	11	6
12	0	12	10
12	1	10	-5
12	2	13	-12
12	4	8	6
13	0	26	-26
13	2	6	0
13	4	19	16
14	0	10	-12
14	1	18	-20
14	2	19	19
14	3	12	11
15	0	7	5
15	1	10	10
15	4	8	-9
15	4	12	-9
16	0	6	3
16	1	12	11
16	2	6	7
16	4	7	-5
17	0	27	27
17	3	8	-6
18	0	11	10
18	0	9	10
18	2	21	-21
18	3	6	-6
19	0	14	-15
19	1	7	-6
19	2	8	-5
20	0	10	-11
20	2	8	5
21	0	11	-8
23	0	17	14

H = 10

K	L	FO	FC
0	0	26	-26
0	2	23	26
0	4	7	1
1	0	21	23
1	2	14	14
1	3	7	11
1	4	17	-15
1	5	8	-6
2	1	6	9
2	2	8	-9
2	5	11	-8
3	0	7	5
3	1	5	-1
3	2	12	9
3	3	12	-14
3	4	12	-10
4	0	24	25
4	1	14	-16
4	2	25	-25
5	0	27	-28
5	0	28	-28
5	1	7	1
5	2	10	-11
5	4	16	15
6	0	13	-13
6	1	5	2
6	2	20	20
7	0	15	15
7	1	8	7
8	0	16	-17
8	1	18	19
8	2	12	13
8	3	7	-6
8	3	7	-6
9	0	18	18
9	3	13	-11
9	4	15	-11
10	0	17	17
10	1	10	-11
10	2	15	-16
10	4	6	0
11	0	13	-15
11	3	9	7
12	1	19	-19
13	0	6	-6
13	1	7	5
13	3	14	14
14	1	22	22
14	2	6	8
15	0	16	15
15	3	12	-10
15	3	12	-10
16	0	6	5
16	3	7	-6
17	0	6	4
18	1	17	-18
19	0	10	-11
20	1	9	7
21	0	8	2

H = 11

K	L	FO	FC
0	1	29	31
1	1	15	16
1	2	8	7
1	3	14	-16
1	4	8	-5
2	0	16	14
2	1	9	-6
2	2	9	-9
3	2	15	-17
3	3	8	-10
3	4	8	8
4	0	19	-21
4	1	23	-29
5	1	10	-10
5	2	6	7
5	3	19	19
6	0	6	-5
6	1	15	16
7	0	7	4
7	1	7	4
7	2	10	9
8	0	29	30
8	1	18	18
8	2	12	-14
8	3	6	-1
9	2	14	-15
9	3	17	-16
10	0	7	-5
10	1	22	-21
11	3	7	7
12	0	14	-17
12	1	7	-5
12	2	9	7
13	0	10	7
13	2	10	11
14	0	16	15
14	1	12	13
14	2	7	-9
15	2	8	-3
17	0	7	-8
18	0	17	-11
18	0	17	-17

H = 12

K	L	FO	FC
0	0	28	27
0	2	10	1
1	0	6	8
1	1	6	2
1	2	15	-15
2	1	12	-13
3	1	12	-12
3	2	6	-6
4	0	21	-21
5	0	7	-9
5	1	14	13
5	2	21	21
6	0	11	10
6	1	6	6
7	1	8	9
8	0	13	14
8	2	7	-5
9	0	10	9
9	1	17	-15
9	2	19	-19
10	0	13	-14
12	1	8	6
13	1	13	13
14	0	9	12

H = 13

K	L	FO	FC
0	1	6	-8
1	1	8	-12
2	0	6	-10
3	1	9	-7
4	0	8	1
5	1	12	12
8	0	6	-7
9	0	12	-12

Table VI. (Continued)

H =-13

K	L	FO	FC
0	1	19	20
0	3	15	-15
1	1	6	-1
1	3	7	-6
2	0	11	10
2	2	10	-13
3	1	7	-7
3	3	6	-8
3	4	10	9
4	1	13	-16
4	2	7	2
4	3	8	8
5	3	15	16
6	3	7	-7
7	2	6	5
7	4	9	-10
8	1	7	9
8	3	6	-1
9	0	12	-12
9	3	16	-13
10	1	7	-12
10	2	11	7

H =-12

K	L	FO	FC
0	0	26	27
0	2	21	-24
0	6	19	15
1	0	6	-6
1	1	7	3
1	2	20	-20
1	4	10	11
1	6	7	4
2	1	15	-16
2	6	9	-5
3	2	12	-12
3	3	10	12
3	4	7	6
4	0	24	-21
4	1	11	9
4	2	12	16
4	4	7	6
4	5	12	-8
4	6	14	-10
5	0	9	9
5	2	16	16
5	4	15	-14
5	5	6	-1
6	0	11	10
6	3	9	4
7	3	10	-11
8	0	14	14
8	1	13	-15
8	4	7	-4
8	5	15	11
9	0	11	-9
9	2	8	-7
9	3	13	16
9	4	9	10
10	0	12	-14
10	1	6	8
10	4	12	9
10	5	10	-7
11	4	8	-11
12	2	6	0
13	2	8	6
13	3	13	-15
14	0	11	12
14	1	13	-16
16	3	6	1
19	2	10	-8

H =-11

K	L	FO	FC
0	1	34	-35
0	3	12	-12
0	5	22	20
1	1	27	-29
1	1	20	-29
1	2	15	-16
1	3	20	19
1	5	7	6
1	7	14	-10
2	0	13	-14
2	2	7	7
2	4	6	7
2	5	11	-7
3	1	6	-5
3	2	26	27
3	3	8	8
3	4	9	-8
3	6	8	-8
4	0	19	21
4	1	24	28
4	2	6	-5
4	3	15	14
4	4	13	-12
4	5	21	-17
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18	4	32	-36
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19	6	13	14
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22	0	29	-31
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22	6	8	-9
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23	4	6	-9
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24	0	24	25
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25	4	6	-3
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26	0	17	17
26	1	13	-12
27	0	10	9
27	2	14	-10
27	3	6	2
27	4	7	5
28	0	18	-19

H = -4

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0	4	27	45
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0	8	17	-12
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1	1	38	-35
1	2	5	12
1	3	6	5
1	4	12	18
1	5	10	9
1	6	29	-25
1	8	8	7
2	0	66	-55
2	1	11	-9
2	2	6	7
2	3	41	42
2	4	16	-17
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2	8	9	4
2	9	11	9
3	0	29	-31
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3	2	4	3
3	3	5	-7
3	4	19	18
3	5	16	-17
3	6	24	-20
3	6	22	-20
3	7	8	6
3	8	5	1
4	0	124	112
4	1	13	9
4	1	12	9
4	2	48	48
4	3	22	-23
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4	9	9	-6
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6	0	12	14
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6	4	29	32
6	8	10	-11
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9	7	16	19
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22	3	17	18
22	4	5	-6
22	5	12	-15
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23	5	13	-14
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24	1	6	-6
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24	3	9	-9
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26	3	7	-4
27	0	8	-8
27	1	22	-23
27	3	9	8
28	3	17	17
29	1	10	9
30	0	10	7

H = -3

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0	7	22	-21
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1	1	92	96
1	3	16	15
1	4	21	27
1	5	87	-82
1	6	14	-15
1	7	12	10
1	8	7	5
2	0	11	10
2	1	34	-30
2	2	29	25
2	3	42	-43
2	4	40	-45
2	6	5	-3
2	7	5	7
2	8	16	13
2	9	7	-4
3	0	71	66
3	1	34	33
3	2	24	-22
3	3	42	42
3	4	23	-25
3	5	43	-40
3	6	26	25
3	6	23	25
3	7	19	18
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4	1	91	92
4	1	91	92
4	2	20	-22
4	3	71	-65
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4	5	8	11
4	7	17	18
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5	3	15	-13
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6	9	10	11
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7	3	23	-18
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7	6	14	-19
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7	9	6	1
8	1	29	-29
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8	3	43	44
8	4	16	-26
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8	7	9	-12
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18	6	7	11
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19	3	7	-7
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24	2	19	-21
24	3	7	-7
24	4	12	11
25	1	19	-18
25	2	7	8
25	4	7	11
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26	4	8	8
27	0	25	-24
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27	2	6	5
27	3	10	11
27	4	6	8
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29	0	9	9
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30	2	7	-1

H = -2

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1	3	18	13
1	4	69	-79
1	5	29	-29
1	6	36	30
1	7	11	9
1	8	16	11
2	0	32	24
2	1	46	-49
2	2	15	-18
2	3	55	-44
2	4	20	19
2	5	8	-7
2	6	9	9
2	7	19	17
2	9	7	-4
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3	2	7	9
3	4	26	-34
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3	6	12	14
3	6	9	14
4	0	5	12
4	1	7	4
4	2	64	-77
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4	3	7	-3
4	6	33	37
4	7	11	-15
4	8	13	-15
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5	3	36	-36
5	4	36	35
5	5	12	-15
5	6	15	-21
5	7	11	-18
5	8	11	-12
6	0	20	-20
6	1	17	-14
6	2	137	126
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6	6	14	-22
6	7	5	-6
6	9	7	3
7	0	46	-44
7	1	20	19
7	2	30	-25
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7	5	3	-6
7	8	6	8
7	9	8	10
8	0	43	-39
8	1	94	87
8	2	49	48
8	3	28	-25
8	7	10	20
8	8	4	8
9	0	28	29
9	1	44	-41
9	2	42	37
9	3	23	-25
9	4	8	-40
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9	6	7	10
9	9	6	-8
10	0	37	34
10	2	126	-117
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10	4	11	17
11	0	35	-33
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11	6	5	-10
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13	4	4	14
13	5	15	-23
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14	0	32	-33
14	1	37	41
14	2	25	24
14	3	32	-28
14	4	5	-4
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14	6	8	-14
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14	8	6	6
15	0	65	62
15	1	9	-5
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15	3	18	-16
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16	0	31	34
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17	5	16	24
18	1	58	-60
18	2	5	-1
18	3	29	27
18	4	6	-5
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18	6	8	9
18	7	9	-15
19	0	30	-30
19	2	8	10
19	3	23	24
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21	6	7	5
22	0	10	12
22	1	41	42
22	2	7	-8
22	3	24	-24
22	4	6	6
23	1	17	-19
23	3	23	-22
23	3	23	-22
23	5	15	20
24	1	28	-29
24	2	12	-12
24	3	11	12
25	0	11	-11
25	4	9	12
25	5	6	-4
26	0	8	-8
26	1	20	-20
26	2	16	16
26	3	17	17
26	5	7	6
27	0	9	9
27	1	10	10
27	3	15	15
28	1	18	18
28	2	5	0
28	3	13	-19
29	1	9	-10
29	2	7	5
29	3	12	-12
30	2	9	-11
31	0	14	-12
31	1	8	4

H = -1

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0	5	75	-71
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0	9	6	3
1	0	40	33
1	1	6	8
1	2	63	-52
1	3	71	-65
1	4	5	-4
1	5	42	39
1	6	5	-2
1	7	19	17
1	8	10	6
1	9	8	-5
2	0	72	54
2	1	7	-7
2	2	6	-7
2	3	9	10
2	4	7	15
2	5	10	15
3	0	14	-10
3	1	15	23
3	2	7	8
3	3	4	-1
3	4	6	28
3	6	3	-8
4	0	40	-39
4	1	156	-150
4	2	50	-45
4	3	9	7
4	5	17	43
4	6	6	-20
4	7	8	-21
5	0	15	12
5	1	22	-21
5	2	25	19
5	3	119	114
5	4	3	-9
5	5	10	-27
5	9	7	12
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6	1	118	114
6	2	8	7
6	3	22	-22
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6	8	6	4
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8	2	67	-63
8	3	26	-25
8	6	9	25
9	0	55	-52
9	1	8	7
9	2	5	3
9	3	63	-79
10	0	41	36
10	1	84	-78
10	2	49	47
10	3	32	34
10	5	5	18
11	0	21	20
11	1	17	16
11	2	34	-35
11	3	56	35
11	4	4	-5
12	0	30	-31
12	1	7	6
12	2	26	24
12	3	14	13
12	5	7	-16
12	6	6	-20
13	0	67	61
13	1	17	-17
13	2	16	18
13	3	24	24
13	5	7	-16
14	0	23	25
14	1	11	11
14	2	50	-48
14	5	8	-14
14	6	7	20
14	7	7	12
14	8	4	-7
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15	1	11	-9
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15	8	8	-3
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16	1	29	-27
16	2	4	-7
16	3	13	14
16	5	11	19
17	0	44	-43
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18	0	63	-61
18	1	10	-8
18	2	34	37
18	3	15	-17
18	4	5	7
18	6	10	-20

ever, as the carbon atoms are located above and below the N-Co-N planes. Nonetheless all of the atoms in the trien ligand fall within 0.37 Å of a least-squares plane.

As has been described above, the coordination of the CN groups to the cobalt is clearly through the carbon. The Co-C average bond distance of 1.90 Å is in good agreement with previously published values. The average C-N distance of 1.17 Å also agrees quite well with the range of previously reported values, 1.15 to 1.18 Å.[2,19-21] The fact that no part of the chain N(6)-C(8)-Co-C(7)-N(5) is linear, with bond angles of N(6)-C(8)-Co, C(8)-Co-C(7), and Co-C(7)-N(5) of 174.1, 177.9 and 173.7°, respectively, is somewhat unusual but not surprising. Curry and Runciman[2] have already observed such a phenomenon. Much of this can be attributed to steric effects both inside the moiety and between moieties (Figure 3). Significant non-bonded distances are given in Table V.

The Cl-O bond distances in the perchlorate group average 1.42 Å and the angles average 109.5°. The relatively large thermal parameters, possibly due to spatial as well as temporal disorder probably account for the large individual deviations from the average values. However, these average values agree reasonably well with previously published values for this anion.[18] A weak hydrogen bond is possible between N(4) and O(3), which are 3.03 Å apart. Such a hydrogen bond would help stabilize the perchlorate group.

(19) B.M. Chadwick and A.G. Sharpe, *Adv. Inorg. Chem. Radiochem.*, *8*, 84-176 (1966).

(20) W.P. Shaefer, B. Wang, and R.E. Marsh, *Inorg. Chem.*, *10(7)*, 1429-1497 (1971).

(21) A. Wolberg, *Acta Cryst.*, *B25(pt 1)*, 161-162 (1969).