CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND
DEPARTMENT OF CHEMISTRY, UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA

The Crystal and Molecular Structure of Di(tetra-*n*-butylammonium)cobalt(II) Bis(maleonitrile dithiolate) and the Geometry of the Divalent Cobalt(II) Bis(maleonitrile dithiolate) Ion[1]

BY J. D. FORRESTER, ALLAN ZALKIN, AND DAVID H. TEMPLETON

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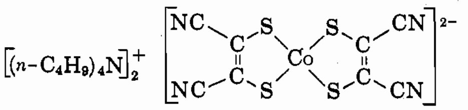
The crystal and molecular structure of di(tetra-*n*-butylammonium)cobalt(II) bis(maleonitrile dithiolate) has been determined from an X-ray diffraction study of a single crystal specimen. A convenient triclinic cell, space group $P\bar{1}$, with a = 10.77 ± 0.01, b = 12.35 ± 0.01, c = 9.81 ± 0.01 Å., α = 88.5 ± 0.1, β = 114.8 ± 0.1, and γ = 93.5 ± 0.1°, contains one formula unit. Atomic parameters were refined by least-squares methods using full three-dimensional data out to $\sin \theta/\lambda = 0.482$. The anion is closely planar with the sulfur atoms in a square arrangement around the cobalt atom. The symmetry of the anion is mmm to a very close degree. Three of the *n*-butyl chains in the cation adopt the *trans* conformation while the other has the *gauche* conformation with a dihedral angle of 71.6°. The cobalt atoms are very well separated with the closest distance of approach as the shortest axial length of 9.81 Å. Positions of all 36 independent hydrogen atoms were determined from an electron density difference function.

Introduction

There has been considerable interest recently in the electronic energy levels of a series of compounds represented by the general formula $R'_z(M'S_4C_4R_4)^{-z}$, where z = 0, 1, 2; M′ = Co, Ni, Cu, Pd, Pt, Zn, Rh; R = CN, CF_3, C_6H_5, etc.; and R′ = $(n\text{-}C_4H_9)_4N^+$, $(CH_3)_4N^+$, $(C_2H_5)_4N^+$, etc. Interpretation of these results, however, has been somewhat hampered by the lack of any detailed knowledge of the molecular structure of these compounds and certain discrepancies have arisen in the electron spin resonance measurements.

$((n\text{-}C_4H_9)_4N)^+{}_2(Co(S_2C_4N_2)_2{}^{2-})$, abbreviated $((n\text{-}C_4H_9)_4N)^+{}_2(Co(MNT)_2{}^{2-})$, and shown schematically below, is a typical member of this series. The anion $(Co(MNT)_2)^{2-}$ was first prepared by Gray and co-workers,[2]



who found that the X-ray powder diffraction patterns for $((n\text{-}C_4H_9)_4N)^+{}_2(M'(MNT)_2{}^{2-})$ with M′ = Co, Pt, Ni, and, later, Rh[3] were identical and differ considerably from that found with M′ = Zn.

Electron spin resonance and magnetic susceptibility measurements on these compounds have been made by Davison, *et al.*,[4-6] as well as by Gray, *et al.*,[2] and Billig, *et al.*[7] Previous evidence[8,9] indicated that four-coordinated, low-spin complexes of Co were likely to be square-planar. This is based on magnetic measurements and apparently the only evidence obtained by X-ray diffraction techniques is the molecular structure of cobalt(II) phthalocyanine where Linstead and Robertson[10,11] showed that the Co atom had a square-planar environment. Little further evidence seems to be available; *e.g.*, Wells.[12]

An X-ray structure determination was completed very recently by Eisenberg, *et al.*,[13] on another member of this family, *viz.*, $((CH_3)_4N)^+{}_2(Ni(MNT)_2{}^{2-})$, and our work is in very close agreement with this.

Maki, *et al.*,[14] have recently used the results of the present crystallographic investigation in a detailed report on the electronic structures of several members of this series of compounds; the principal axes of the g-value tensor and of the hyperfine tensor were associated with the symmetry directions of the anion.

Experimental

X-Ray Diffraction.—We are indebted to Dr. A. H. Maki of Harvard University for sending us some well-formed crystals of the complex $(n\text{-}C_4H_9)_4N)^+{}_2(CoS_4C_4(CN)_4{}^{-2})$. The melting point, analysis, and conductivity of these crystals are described in a paper by Davison, *et al.*[5]

X-Ray photographs obtained by the Weissenberg technique and copper radiation established the diffraction symmetry of the crystal. A single crystal, in the form of a thin plate of approximate dimensions 0.24 × 0.20 × 0.10 mm. and mounted about an a^* axis, was used for collecting the intensity data. Intensities were measured with a General Electric XRD-5 goniostat equipped with a scintillation counter and a pulse height discriminator. Mo Kα radiation was used and the unit cell dimensions are based on λ = 0.70926 Å. for Mo Kα_1.

The 2213 independent reflections permitted by the space group in the sphere of reflection with $\sin \theta/\lambda$ less than 0.482 ($2\theta < 40°$) were measured with counting times of 10 sec. each. Of these,

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[1] Work done under the auspices of the U. S. Atomic Energy Commission.

[2] H. B. Gray, R. Williams, I. Bernal, and E. Billig, *J. Am. Chem. Soc.*, **84**, 3596 (1962).

[3] E. Billig, S. I. Shupack, J. H. Waters, R. Williams, and H. B. Gray, *ibid.*, **86**, 926 (1964).

[4] A. Davison, N. Edelstein, R. H. Holm, and A. H. Maki, *ibid.*, **85**, 2029 (1963).

[5] A. Davison, N. Edelstein, R. H. Holm, and A. H. Maki, *ibid.*, **85**, 3049 (1963).

[6] A. Davison, N. Edelstein, R. H. Holm, and A. H. Maki, *Inorg. Chem.*, **2**, 1227 (1963).

[7] E. Billig, R. Williams, I. Bernal, J. H. Waters, and H. B. Gray, *ibid.*, **3**, 663 (1964).

[8] B. N. Figgis and R. S. Nyholm, *J. Chem. Soc.*, 12 (1954).

[9] B. N. Figgis and R. S. Nyholm, *ibid.*, 338 (1959).

[10] J. M. Robertson, *ibid.*, 615 (1935).

[11] R. P. Linstead and J. M. Robertson, *ibid.*, 1736 (1936).

[12] A. F. Wells, "Structural Inorganic Chemistry," Oxford University Press, 1962, p. 926.

[13] R. Eisenberg, J. A. Ibers, R. J. H. Clark, and H. B. Gray, *J. Am. Chem. Soc.*, **86**, 113 (1964).

[14] A. H. Maki, N. Edelstein, A. Davison, and R. H. Holm, *ibid.*, **86**, 4580 (1964).

TABLE I

FINAL COORDINATES AND ISOTROPIC THERMAL PARAMETERS, TOGETHER WITH THEIR STANDARD DEVIATIONS, FOR ALL ATOMS EXCEPT HYDROGEN

Atom	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$	B^b	$\sigma(B)$
Co	0.0	. . .	0.0	. . .	0.0	. . .	*a*	
S(1)	0.0401	0.0002	0.1381	0.0002	0.1462	0.0002	*a*	
S(2)	−0.2156	0.0002	0.0303	0.0002	−0.1093	0.0002	*a*	
C(1)	−0.1165	0.0007	0.1930	0.0005	0.0876	0.0007	4.7	0.1
C(2)	−0.2283	0.0007	0.1463	0.0006	−0.0214	0.0008	5.0	0.2
C(3)	−0.1243	0.0008	0.2883	0.0007	0.1575	0.0009	6.0	0.2
C(4)	−0.3583	0.0010	0.1883	0.0007	−0.0717	0.0010	7.4	0.2
N(1)	−0.1273	0.0007	0.3662	0.0006	0.2172	0.0008	7.3	0.2
N(2)	−0.4643	0.0010	0.2247	0.0007	−0.1113	0.0010	10.5	0.2
N(3)	0.0867	0.0005	0.2783	0.0004	−0.2929	0.0006	4.4	0.1
C(5)	0.1928	0.0007	0.2589	0.0005	−0.1328	0.0007	4.6	0.1
C(6)	0.2939	0.0007	0.3510	0.0006	−0.0626	0.0008	5.3	0.2
C(7)	0.3797	0.0008	0.3242	0.0007	0.1021	0.0009	6.7	0.2
C(8)	0.4976	0.0010	0.4089	0.0008	0.1789	0.0011	9.2	0.3
C(9)	−0.0023	0.0007	0.1740	0.0006	−0.3418	0.0008	5.0	0.2
C(10)	−0.1167	0.0008	0.1779	0.0007	−0.5001	0.0009	6.9	0.2
C(11)	−0.2078	0.0009	0.0756	0.0008	0.4697	0.0010	8.0	0.2
C(12)	−0.3248	0.0010	0.0689	0.0008	0.3116	0.0011	8.6	0.2
C(13)	0.1538	0.0007	0.3116	0.0005	−0.3963	0.0007	5.0	0.2
C(14)	0.2348	0.0007	0.2286	0.0006	−0.4230	0.0008	5.4	0.2
C(15)	0.3224	0.0008	0.2751	0.0007	−0.5004	0.0009	6.6	0.2
C(16)	0.4075	0.0009	0.1952	0.0007	0.4737	0.0010	7.4	0.2
C(17)	0.0004	0.0007	0.3718	0.0006	−0.2930	0.0008	5.4	0.2
C(18)	−0.0671	0.0008	0.3599	0.0006	−0.1848	0.0009	6.5	0.2
C(19)	−0.1712	0.0009	0.4468	0.0007	−0.2126	0.0010	7.2	0.2
C(20)	−0.2991	0.0011	0.4290	0.0009	−0.3551	0.0012	10.4	0.3

[a] Treated anisotropically—see Table III. [b] In Å.^{2}.

390 were assigned zero intensity and the maximum count was 12,350 counts/sec. for the 100 reflection. No corrections were made for either absorption or extinction. With μ = 5.9 cm.$^{-1}$ for Mo radiation, μR is 0.07 or less, making the absorption effects rather small.

Calculations were made with an IBM 7094 computer using a full matrix least-squares refinement program written by P. K. Gantzel, R. A. Sparks, and K. N. Trueblood, with minor modifications, and Fourier and distance programs written by Zalkin (all unpublished). We minimized the function $\Sigma w(|F_o| - |F_c|)^2/\Sigma w F_o^2$, where F_o and F_c are the observed and calculated structure factors, respectively, and w is the weighting factor.

Atomic scattering factors were taken as the values given by Ibers[15] for neutral Co, S, N, C, and H. Dispersion corrections[16] of 0.4 and 0.1 electron were added to the Co and S scattering factors, respectively. The imaginary part of the dispersion correction is small and was ignored.

Results

Unit Cell and Space Group.—A convenient primitive unit cell contains one formula unit $((n\text{-}C_4H_9)_4N)^+{}_2$-$(Co(S_2C_4N_2)_2{}^{2-})$ and is triclinic with dimensions: a = 10.77 ± 0.01, b = 12.35 ± 0.01, c = 9.81 ± 0.01 Å., α = 88.5 ± 0.1, β = 114.8 ± 0.1, γ = 93.5 ± 0.1°, V = 1182.0 Å.^{3}. With one formula unit per unit cell, the density calculated from the X-ray data is 1.16 g./cc., in comparison with the value of 1.14 g./cc. found by flotation methods using a mixture of benzene and carbon tetrachloride.

There are no systematic extinctions and the space group could be either P1 (C_1) or P$\bar{1}$ (C_i). Previous evidence indicated that the Co complex was likely to be planar and we favored the space group P$\bar{1}$ (C_i); the success of our structure determination confirms this choice.

Determination of the Structure.—After about half of the data had been collected, a three-dimensional Patterson function and a three-dimensional Fourier with phases based on Co placed at the origin were calculated from the observed intensities after correction for Lorentz and polarization effects. From these two functions and a knowledge of the molecular geometry, it was possible to determine unambiguously the positions of all the independent atoms (2S, 4C, and 2N) in the anion. With the exception of the Co atom which is in a special position, 1 (a) 0, 0, 0, all the atoms are in general twofold positions, 2 (i) ±(x, y, z).

At this stage, all the data had been measured, and a second three-dimensional electron density function with phases based on the whole of the anion was calculated from 1627 observed reflections (whose intensity was greater than 3 counts/sec.) after correction for Lorentz and polarization effects. Using this function, together with information regarding bond distances and angles and rejecting any positions which did not lead to peaks in the Patterson function, all 17 independent atoms (1N and 16C) in the tetra-n-butylammonium ion were found in general positions.

A least-squares refinement with all 26 of these atoms, each having an isotropic temperature factor of the form $\exp(-B\lambda^{-2}\sin^2\theta)$, and using the 1627 terms each with unit weight resulted in a conventional unreliability factor $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ of 0.18 after four cycles of refinement. Interatomic distances and angles appeared at this stage to be very reasonable, but there

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(15) J. A. Ibers in "International Tables for X-Ray Crystallography," Kynoch Press, Birmingham, England, 1962, Vol. III, p. 202.

(16) D. H. Templeton, *ibid.*, p. 215.

Table II

Final Coordinates and Isotropic Thermal Parameters for the Hydrogen Atoms

Atom	x	y	z	B, Å.^{2}	Atom	x	y	z	B, Å.^{2}
H(1) C(5)	0.13	0.23	−0.08	1.1	H(1) C(13)	0.22	0.39	−0.35	4.6
H(2)	0.24	0.20	−0.15	1.9	H(2)	0.08	0.33	−0.47	5.4
H(1) C(6)	0.35	0.37	−0.12	1.9	H(1) C(14)	0.29	0.20	−0.34	0.5
H(2)	0.25	0.42	−0.06	4.0	H(2)	0.18	0.17	−0.49	4.9
H(1) C(7)	0.32	0.32	0.16	4.9	H(1) C(15)	0.38	0.33	−0.44	3.4
H(2)	0.41	0.26	0.11	0.3	H(2)	0.26	0.31	−0.58	3.3
H(1) C(8)	−0.43	0.42	0.12	4.6	H(1) C(16)	0.47	0.16	0.56	5.2
H(2)	−0.47	0.35	0.21	1.6	H(2)	0.37	0.13	0.43	4.2
H(3)	−0.53	0.48	0.16	3.6	H(3)	0.46	0.24	0.43	9.9
H(1) C(9)	−0.04	0.15	−0.25	3.4	H(1) C(17)	−0.06	0.37	−0.40	3.1
H(2)	0.06	0.12	−0.34	0.9	H(2)	0.06	0.44	−0.28	2.9
H(1) C(10)	−0.18	0.21	−0.48	1.0	H(1) C(18)	0.01	0.36	−0.07	7.7
H(2)	−0.07	0.18	−0.57	7.9	H(2)	−0.12	0.29	−0.20	2.7
H(1) C(11)	−0.25	0.08	0.55	7.0	H(1) C(19)	−0.21	0.44	−0.12	7.0
H(2)	−0.14	0.05	0.45	2.9	H(2)	−0.14	0.52	−0.23	3.7
H(1) C(12)	−0.38	0.11	0.33	3.5	H(1) C(20)	−0.28	0.44	−0.44	4.4
H(2)	−0.27	0.04	0.26	5.3	H(2)	−0.33	0.37	−0.32	5.5
H(3)	−0.33	0.14	0.25	8.8	H(3)	−0.36	0.49	−0.33	22.8

were several large discrepancies in the observed and calculated structure factors, some of the temperature factors were somewhat abnormal, and we felt that the agreement should be much better. However, it was then realized that we had neglected to apply a special position factor to the Co form factor which was thus twice as large as it ought to be. Correction of this blunder and three cycles of refinement speedily reduced R to 0.11. Several errors in the data taking and in the card punching were corrected and the Co atom given an anisotropic temperature factor of the form exp $(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$, with $4\beta_{ij} = a_i^*a_j^*B_{ij}$, a_i^* being the length of the ith reciprocal axis. With this notation the anisotropic thermal parameters B_{ij} are in the units (Å.^{2}) which are used for isotropic thermal parameters B in the temperature factors of the form $\exp(-B\lambda^{-2}\sin^2\theta)$. Four cycles of refinement with all 2213 terms included resulted in $R = 0.142$. The terms were weighted so that the 1823 nonzero terms were given unit weight and the 390 zero terms were given $^1/_4$ weight.

An electron density difference function, with all the atoms except hydrogen subtracted out, was calculated using the results of this refinement for the terms with $\sin\theta/\lambda < 0.4$. With the exception of several peaks due to an incorrect description of the thermal motion of the sulfur atoms, the highest peaks in this function all corresponded to reasonable positions for the 36 independent hydrogen atoms, all in twofold general positions. Two cycles of least-squares refinement were run with the two sulfur atoms, as well as the cobalt, having anisotropic thermal parameters, and R fell to 0.126.

The number of parameters now exceeded the capacity of our program, so we refined parts of the structure separately with the remainder held fixed. First, the H atoms were included but not refined, while all the other parameters were refined, and R fell to 0.103. Then all atoms except H were held fixed and the 36 H atoms, each with an isotropic temperature factor, were refined and resulted in $R = 0.097$. This procedure was repeated twice to reduce R to 0.091.

Table III

Final Anisotropic Thermal Parameters of the Cobalt and the Two Sulfur Atoms[a]

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}[b]
Co	3.8	5.4	4.1	0.2	1.2	0.0
S(1)	4.3	6.0	5.0	0.1	0.9	−0.6
S(2)	4.2	7.6	5.6	0.3	0.9	−1.8

[a] Estimated standard deviations are all approximately 0.1 Å.^{2}. [b] In Å.^{2}.

We are indebted to a referee who noticed that we had by error used the wrong sign for the dispersion corrections to the atomic scattering factors of cobalt and sulfur. Two additional cycles of least-squares refinement of the heavy atoms after correction of this mistake improved the agreement slightly, with a final R of 0.090. No atom moved more than 0.002 Å. The thermal parameters (other than cross terms) of cobalt and sulfur increased 0.3 and 0.1 Å.,^{2} respectively, while those of carbon and nitrogen decreased about 0.1 Å.^{2}. In the final cycle, no coordinate shifted more than 0.00007 and no thermal parameter by more than 0.01 Å.^{2}.

The parameters resulting from the last cycle are listed in Tables I, II, and III. The observed and calculated structure factors before correction of the dispersion error are compared in Table IV.

Standard deviations of the parameters of the heavy atoms were calculated assuming that the discrepancies in the structure factors represent random errors and with neglect of the effect of the H parameters. Limitations of our computer program did not permit us to estimate the standard deviations of the hydrogen atom parameters by the method of least squares. From the resulting bond distances we estimate the standard deviations of the H atom coordinates to be 0.1 to 0.2 Å.

Description of the Structure.—A projection down the b axis of the over-all structure is shown in Fig. 1. The planar $Co(MNT)_2^{2-}$ ion has a center of symmetry at the cobalt atom and the two tetra-n-butylammonium ions are related to each other by a center of symmetry. The ions are interlaced to form layers in the bc plane.

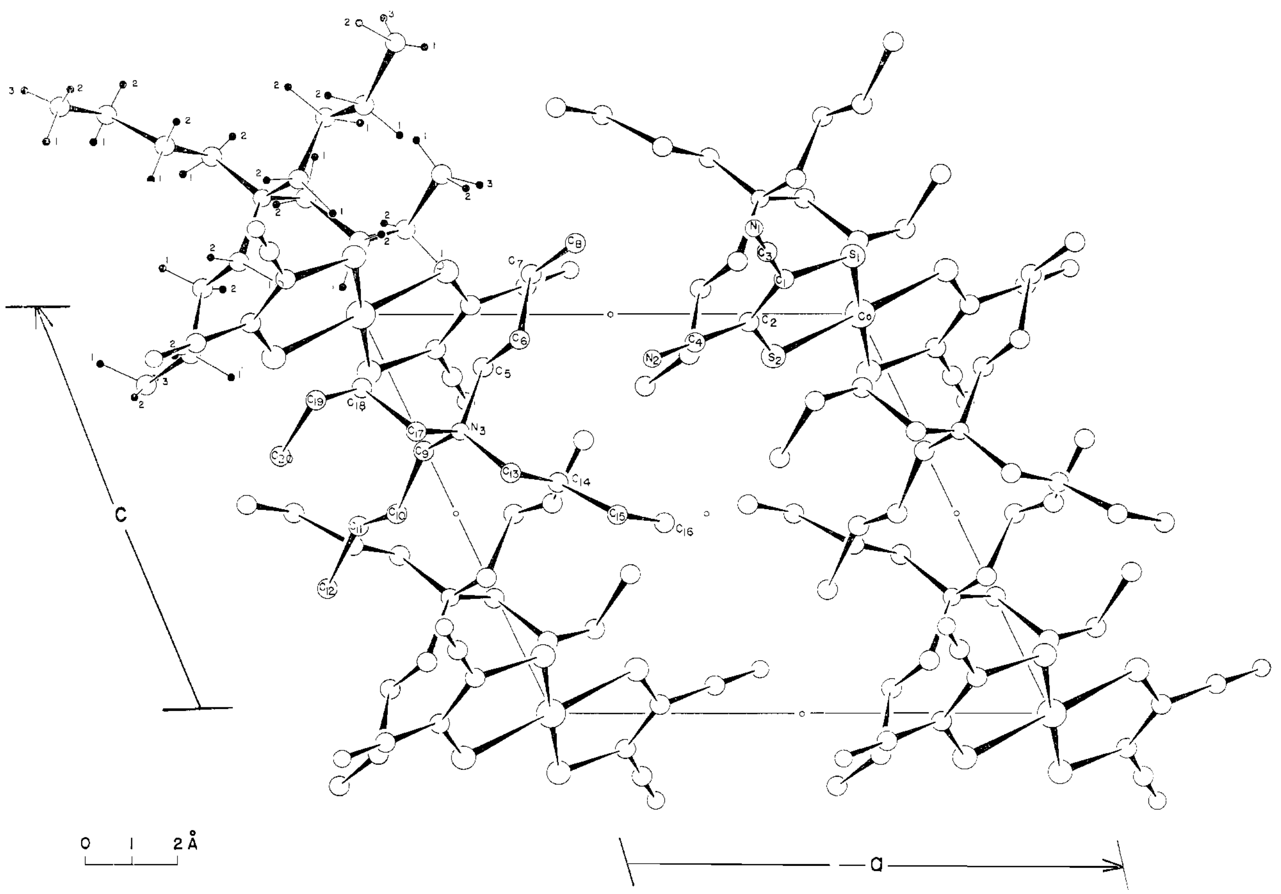

Fig. 1.—Projection of the structure of $((n\text{-}C_4H_9)_4N)^+{}_2(Co(S_2C_4N_2)_2{}^{2-})$ down the b axis. The small black circles represent examples of each type of hydrogen atom.

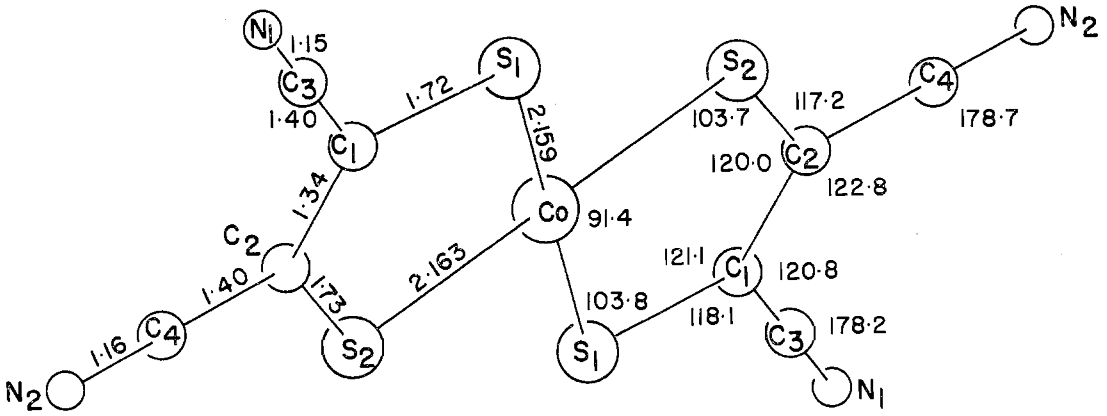

Fig. 2.—Dimensions of the $Co(MNT)_2{}^{2-}$ ion. See Table V for the estimated standard deviations.

The $Co(MNT)_2{}^{2-}$ Ion.—The dimensions of the anion are illustrated in Fig. 2 and compared in Table V with those found in the complex $((CH_3)_4N)^+{}_2(Ni(MNT)_2{}^{2-})$ by Eisenberg, *et al.*[13] The agreement is very close, with the Co–S and Ni–S distances in the two complexes equal to well within the experimental accuracy.

The anion is very closely planar and deviations from planarity are small enough to be chemically insignificant. The three atoms Co, C(1), and C(2) define a plane of equation (triclinic coordinate system)

$$-4.996x - 6.634y + 7.975z = 0$$

The distances of the various atoms from this plane are listed in Table VI. S(1) is somewhat out of the plane of Co, S(2), C(1), and C(2) and makes the five-membered ring slightly puckered, and the cyanide groups are bent a small amount out of the plane. None of these deviations, however, is as much as 0.1 Å.

The chemically equivalent, but crystallographically nonequivalent bonds in the anion, *viz.*, S(1)–C(1) and S(2)–C(2), C(1)–C(3) and C(2)–C(4), C(3)–N(1) and C(4)–N(2), differ by no more than two standard deviations. Although the ion is required to have only a center of symmetry (C_i), the geometry is such that it does not differ significantly from mmm (D_{2h}).

The arrangement of the sulfur atoms around the co-

Table IV

Observed Structure Factor Magnitudes (FOBS) and Calculated Structure Factors (FCAL), each Multiplied by 10

```
H,K=  0,  0
 L FOBS FCAL
 1  590  551
 2  315  302
 3  377 -379
 4  184 -171
 5  185 -160
 6  157  158
 7  136  131
 8   90   99

H,K=  0,  1
 L FOBS FCAL
-8  114  109
-7  160  152
-6   63   79
-5  180 -176
-4  242 -244
-3   44  -46
-2 1001 1005
-1   52   21
 0  473  415
 1  631  587
 2  337  306
 3  255  236
 4  285  261
 5   22  -16
 6  175  171
 7  120  131
 8  131  137

H,K=  0,  2
 L FOBS FCAL
-8  122  129
-7   92   91
-6    0  -23
-5  138  136
-4  136  142
-3  354  344
-2  233 -240
-1  673  655
 0  472 -486
 1   45   19
 2   99   89
 3  286  271
 4  217  198
 5  232  216
 6  155  146
 7    0    9
 8    0    3

H,K=  0,  3
 L FOBS FCAL
-8   97   83
-7   89   85
-6   78  -81
-5   50   58
-4    0    8
-3  145  161
-2   43  -38
-1  507  503
 0  245  231
 1  143  157
 2    0    2
 3  205  207
 4  197  192
 5  128  115
 6   35   23
 7   27   25
 8    0  -13

H,K=  0,  4
 L FOBS FCAL
-8   42   48
-7  120  110
-6   44   46
-5    0   -1
-4   96  106
-3   51   60
-2  148  157
-1  180  184
 0    0  -14
 1   40   29
 2  520  509
 3  180  196
 4    0   38
 5   57  -49
 6   98  -99
 7    0  -12
 8  111   91

H,K=  0,  5
 L FOBS FCAL
-7   63   69
-6  143  133
-5  146  143
-4  205  195
-3   51   31
-2   91  -80
-1    0   21
 0  368  369
 1  506  501
 2  581  572
 3  224  239
 4    0  -11
 5  103  -93
 6  108 -118
 7  116 -111

H,K=  0,  6
 L FOBS FCAL
-7    0   37
-6  138  137
-5  170  190
-4   53   54
-3   70  -80
-2   21  -56
-1  156  157
 0  313  308
 1  419  433
 2  517  519
 3  312  301
 4    0   45
 5   84  -76
 6   61  -53
 7    0   -7

H,K=  0,  7
 L FOBS FCAL
-6  105   96
-5   92  108
-4    0   24
-3    0   19
-2    0   17
-1  112  126
 0  191  192
 1  282  264
 2  108  107
 3   85   90
 4    0    1
 5    0   35
 6  162  151

H,K=  0,  8
 L FOBS FCAL
-6   97   93
-5   39   56
-4    0  -43
-3    0   39
-2   34   50
-1  189  202
 0   93   85
 1  237  226
 2   34  -37
 3   25   39
 4  166  151
 5  103   96
 6   51   42

H,K=  0,  9
 L FOBS FCAL
-5   50   53
-4    0  -27
-3    0    8
-2   45   43
-1   71   75
 0    0    6
 1  133  137
 2  152  160
 3  145  123
 4   92   88
 5  142  144

H,K=  0, 10
 L FOBS FCAL
-4   82   81
-3   28   33
-2   82   88
-1    0   14
 0    0  -16
 1    0  -36
 2   82   78
 3  143  140
 4   29   52

H,K=  0, 11
 L FOBS FCAL
-3   42   63
-2   29   31
-1    0    1
 0   69   59
 1    0   10
 2    0   20
 3   89   76

H,K=  1,-11
 L FOBS FCAL
-3  113   90
-2   70  -81
-1   70  -73
 0   40   41
 1    0    8
 2   71   80

H,K=  1,-10
 L FOBS FCAL
-4   64   73
-3  121  123
-2   86   85
-1   96  -87
 0    0   10
 1   76   73
 2   78   80
 3   57   69
 4   59   62

H,K=  1, -9
 L FOBS FCAL
-5  103  102
-4  130  131
-3    0  -13
-2  102  103
-1   56   63
 0    0   -4
 1   51   45
 2   97  101
 3   60   67
 4    0   15
 5   42   36

H,K=  1, -8
 L FOBS FCAL
-6  118  125
-5   90   91
-4  183  194
-3   43   18
-2  107 -112
-1   33   58
 0  119  111
 1   71   77
 2   24   34
 3    0   20
 4    0   27
 5   28   25

H,K=  1, -7
 L FOBS FCAL
-7   93   93
-6  132  140
-5  127  130
-4   42   47
-3  208  228
-2   67   70
-1  196  203
 0  104   92
 1    0   -5
 2   61   39
 3   24   33
 4   68   57
 5  101   95
 6   82   74

H,K=  1, -6
 L FOBS FCAL
-7   80   80
-6    0   19
-5   86  105
-4   52   58
-3  266  257
-2  256  271
-1  238  238
 0  117  112
 1   79  -58
 2   37  -50
 3   51   75
 4  196  174
 5  285  279
 6  238  228

H,K=  1, -5
 L FOBS FCAL
-8   72   59
-7    0   -6
-6   25  -18
-5   98   76
-4  319  319
-3  368  366
-2  396  386
-1  262  261
 0   68  -72
 1  153 -158
 2    0   18
 3  159  170
 4  300  282
 5  172  177
 6  145  152
 7    0   36

H,K=  1, -4
 L FOBS FCAL
-8   50  -40
-7  142 -129
-6   60  -58
-5  186  186
-4  442  415
-3  593  572
-2  576  553
-1  329  328
 0    0   19
 1   80   78
 2  106   93
 3  246  241
 4   50   57
 5   87   95
 6   26   20
 7   69   63

H,K=  1, -3
 L FOBS FCAL
-8   85  -89
-7   45  -41
-6   54  -27
-5  155  163
-4  349  356
-3  391  383
-2  388  376
-1  325  337
 0  509 -524
 1  372  369
 2   84 -101
 3  226  229
 4  184  165
 5  235  231
 6   81   70
 7  111  106

H,K=  1, -2
 L FOBS FCAL
-8    0  -17
-7   36  -39
-6  118  128
-5  296  296
-4  238  266
-3   41   52
-2  218 -225
-1  189  185
 0  277 -317
 1  156  140
 2  258  260
 3  101  -97
 4  112   97
 5  191  196
 6   50   63
 7  123  122
 8  107  118

H,K=  1, -1
 L FOBS FCAL
-8  100   93
-7  128  125
-6   99   91
-5  109   97
-4    0    8
-3   42   44
-2  496  496
-1  318  304
 0  504  483
 1    0  -10
 2  520  504
 3   56  -67
 4  256 -246
 5    0    6
 6  168  154
 7  189  182
 8  102  108

H,K=  1,  0
 L FOBS FCAL
-8   39   36
-7  142  128
-6  195  190
-5   72   65
-4  164  150
-3  212  212
-2  245  235
-1  274  239
 0  993  953
 1  298  290
 2  507 -500
 3   31  -19
 4   49   31
 5  185  173
 6  196  197
 7  221  197
 8  132  115

H,K=  1,  1
 L FOBS FCAL
-8  118  117
-7   44  -48
-6  161  154
-5   69   56
-4   91  101
-3  495  502
-2 1146 1176
-1  514  494
 0  382  350
 1  793  795
 2  593 -572
 3  332 -308
 4   79   69
 5  149  167
 6  218  219
 7  139  131
 8   59   64

H,K=  1,  2
 L FOBS FCAL
-8  166  156
-7   73  -64
-6  167 -163
-5  240 -240
-4   99   93
-3  544  549
-2 1415 1481
-1 1241 1261
 0  458  463
 1  304 -315
 2  228  201
 3   96  -86
 4  267  273
 5   89  106
 6  116  121
 7   91  103
 8   42   21

H,K=  1,  3
 L FOBS FCAL
-8    0   14
-7    0   23
-6   87  -86
-5   66   55
-4  258  248
-3  278  269
-2  140  116
-1  321 -330
 0  847  859
 1   75   61
 2  122  121
 3   73   62
 4  227  199
 5  151  160
 6  126  127
 7  158  145

H,K=  1,  4
 L FOBS FCAL
-8   41  -51
-7  129  113
-6  108  110
-5  141  146
-4   84  101
-3   66   59
-2  106   89
-1   44  -16
 0  188 -181
 1  215  218
 2  195  178
 3  270  264
 4  140  133
 5    0   -5
 6    0  -26
 7   70   69

H,K=  1,  5
 L FOBS FCAL
-8    0   17
-7    0    9
-6   26   35
-5    0    7
-4   66   97
-3  210  216
-2   55  -43
-1  183  174
 0  239  233
 1  187  180
 2  356  338
 3  119  122
 4   74  -68
 5   56  -52
 6   27   -8
 7    0    4

H,K=  1,  6
 L FOBS FCAL
-7    0   18
-6   80   77
-5   86   72
-4   62   71
-3   38   35
-2   73  100
-1  188  209
 0  506  518
 1  148  160
 2   84   72
 3   92  -77
 4  145 -138
 5  128 -123
 6  101   87

H,K=  1,  7
 L FOBS FCAL
-7  125  118
-6  103   99
-5   45   32
-4    0   -4
-3    0   33
-2  146  136
-1  346  350
 0  433  421
 1  228  239
 2   84   87
 3   60  -65
 4  118 -103
 5  112 -110
 6   50   58

H,K=  1,  8
 L FOBS FCAL
-6   41   32
-5    0    7
-4   45  -55
-3   70   71
-2  123  106
-1  231  223
 0  270  260
 1  280  273
 2   70   70
 3    0   11
 4   27   31
 5   64   63

H,K=  1,  9
 L FOBS FCAL
-5   50   57
-4    0   28
-3   26   36
-2  121  117
-1  183  168
 0    0   -0
 1   44   22
 2   37   46
 3   86   75
 4   90  100
 5  136  137

H,K=  1, 10
 L FOBS FCAL
-4   76   78
-3   74   80
-2   47   48
-1  108   92
 0    0   -6
 1   54   42
 2  108   99
 3  137  130
 4    0   30

H,K=  1, 11
 L FOBS FCAL
-3   42   35
-2    0  -20
-1   57   64
 0   76   88
 1  108  115
 2   51   61

H,K=  2,-11
 L FOBS FCAL
-3   88  114
-2    0    5
-1   57  -40
 0    0   29
 1   29   26
 2   84   73

H,K=  2,-10
 L FOBS FCAL
-5   98   99
-4   70   69
-3   39  -59
-2   47   54
-1  114 -114
 0  101  109
 1  182  188
 2  132  143
 3   82   81

H,K=  2, -9
 L FOBS FCAL
-6  141  141
-5   69   51
-4   38   39
-3  163 -172
-2   44   16
-1   80   77
 0   51   46
 1  141  133
 2  199  196
 3   68   70
 4    0   45

H,K=  2, -8
 L FOBS FCAL
-6  120  120
-5  142  133
-4   77   65
-3   95   91
-2  152 -161
-1   67   68
 0  213  212
 1  142  141
 2   61   77
 3    0    2
 4   28   24
 5    0    4

H,K=  2, -7
 L FOBS FCAL
-7  111  114
-6  112  113
-5  147  152
-4    0  -22
-3  109  117
-2   74   77
-1    0    1
 0    0   13
 1   64   60
 2   41   23
 3   86   92
 4  115  121
 5  125  125
 6   42   43

H,K=  2, -6
 L FOBS FCAL
-8  126  113
-7  104  129
-6  122  129
-5    0   30
-4   46   54
-3   31   28
-2  194  184
-1   98   89
 0   46   40
 1  120 -106
 2   55   46
 3  140  143
 4  147  148
 5  179  178
 6  129  124

H,K=  2, -5
 L FOBS FCAL
-8  104   99
-7   54   61
-6   90   92
-5   74   82
-4  241  259
-3  214  221
-2  129  164
-1    0  -20
 0  290  284
 1   88   87
 2   76   64
 3  213  211
 4  239  223
 5  119  118
 6   84   81

H,K=  2, -4
 L FOBS FCAL
-8    0  -15
-7   45   29
-6  148  144
-5  311  301
-4  337  344
-3  378  394
-2   93   92
-1  161 -175
 0  241 -232
 1   61  -66
 2  418  409
 3  545  531
 4  423  425
 5   72   71
 6    0    0
 7   29  -30

H,K=  2, -3
 L FOBS FCAL
-8   62  -42
-7   85   84
-6  201  188
-5  453  443
-4  525  527
-3  481  472
-2  106 -105
-1  398 -384
 0  204 -205
 1  226  228
 2  389  392
 3  232  229
 4  258  254
 5    0   47
 6  123  117
 7    0   35

H,K=  2, -2
 L FOBS FCAL
-9   51  -50
-8   82  -83
-7   44   56
-6  165  181
-5  444  438
-4  354  357
-3  236  213
-2   21   20
-1  927  931
 0  107  106
 1  102  110
 2   40   38
 3   84  -59
 4   58   57
 5  218  205
 6   99   89
 7  107  110

H,K=  2, -1
 L FOBS FCAL
-9   29  -24
-8   27   34
-7  152  162
-6   72   72
-5  171  166
-4  162  169
-3  322  326
-2  370 -389
-1  292  273
 0  238 -247
 1   59   54
 2  211  197
 3  638  628
 4  155 -156
 5  254  240
 6  155  155
 7  145  145

H,K=  2,  0
 L FOBS FCAL
-9    0   23
-8   66   54
-7  199  187
-6  301  304
-5   65   70
-4  104   88
-3  178 -197
-2  219 -213
-1   86   68
 0   44   36
 1  458  443
 2  281 -299
 3  256  233
 4  133  148
 5  195  206
 6  206  189
 7   90  100

H,K=  2,  1
 L FOBS FCAL
-9   88   88
-8  152  139
-7   97 -102
-6  121  126
-5   74   71
-4  203  210
-3  204  196
-2  738  746
-1  431  418
 0  281  266
 1  244 -234
 2   69   64
 3  292 -288
 4  301  290
 5  289  296
 6  196  207
 7    0   26

H,K=  2,  2
 L FOBS FCAL
-9   42   10
-8  143  139
-7    0   35
-6  106 -103
-5  171  187
-4  345  375
-3  584  599
-2 1128 1173
-1  220  200
 0  305 -321
 1  755 -739
 2   31   39
 3   90   96
 4  263  264
 5  264  253
 6  238  222
 7   99  104

H,K=  2,  3
 L FOBS FCAL
-8   56  -59
-7   86   72
-6    0   21
-5  290  300
-4  571  595
-3  544  585
-2  375  411
-1  234  236
 0   77  -76
 1  140  140
 3  128  134
 4  205  223
 5  257  245
 6    0   30
 7   65   75

H,K=  2,  4
 L FOBS FCAL
-8   64  -61
-7   99  -88
-6  127  110
-5  212  188
-4  316  296
-3  287  279
-2  279  290
-1  138  137
 0  155 -149
 1  555  546
 2  293  294
 3  243  244
 4   85   92
 5   95  100
 6    0   14
 7   98   86

H,K=  2,  5
 L FOBS FCAL
-8   41   50
-7    0   -7
-6  108  106
-5   71   60
-4  101   94
-3   72   45
-2   92   94
-1  156  148
 0   99   85
 1   75  -52
 2  253  243
 3   32   34
 4  120  121
 5  142  137
 6  123  138

H,K=  2,  6
 L FOBS FCAL
-7   69   64
-6   59   80
-5   60   77
-4   47   60
-3   86   74
-2   80   74
-1   42  -82
 0  428  414
 1   31   23
 2   39   42
 3   42  -49
 4    0  -25
 5    0    8
 6  127  131

H,K=  2,  7
 L FOBS FCAL
-7   41   46
-6   48   65
-5    0   40
-4    0    5
-3   41   44
-2  240  257
-1  185  169
 0  298  287
 1  133  127
 2   64  -65
 3  107 -106
 4    0  -30
 5   49  -41

H,K=  2,  8
 L FOBS FCAL
-6   40   32
-5    0    2
-4    0   34
-3  168  184
-2  255  265
-1  169  181
 0  128  141
 1    0  -19
 2  122 -124
 3  141 -133
 4    0   35
 5  102   97

H,K=  2,  9
 L FOBS FCAL
-5   49   49
-4   78   72
-3  143  132
-2  210  202
-1  191  197
 0    0   35
 1   26    9
 2   47  -48
 3    0   11
 4   58   56

H,K=  2, 10
 L FOBS FCAL
-4   58   68
-3  126  116
-2   39   29
-1  160  148
 0    0   39
 1   56   59
 2   81   78
 3  110  102

H,K=  2, 11
 L FOBS FCAL
-3   94   87
-2    0    4
-1    0   -5
 0   58   65
 1   66   51

H,K=  3,-11
 L FOBS FCAL
-3   88   73
-2   65   75
-1    0   -8
 0   41   59
 1    0   32

H,K=  3,-10
 L FOBS FCAL
-5   78   65
-4    0   17
-3   48  -69
-2    0  -20
-1    0    4
 0  107   94
 1  233  221
 2  119  126
 3   42   54

H,K=  3, -9
 L FOBS FCAL
-6   83   75
-5   69   73
-4    0  -16
-3   64  -59
-2    0    2
-1  186  208
 0  130  122
 1  199  200
 2  171  167
 3   57   51
 4   30  -24

H,K=  3, -8
 L FOBS FCAL
-7  132  116
-6   63   55
-5    0  -22
-4    0  -41
-3   25   10
-2   24   34
-1  178  186
 0  286  281
 1  140  132
 2   86   85
 3   27   25
 4   49   51

H,K=  3, -7
 L FOBS FCAL
-7   86   83
-6   60   63
-5   25  -28
-4    0   -7
-3   62  -71
-2  194  208
-1    0    1
 0  178  184
 1  182  164
 2   55   64
 3    0  -11
 4   39   21
 5  105   96

H,K=  3, -6
 L FOBS FCAL
-8  125  114
-7  107  118
-6  121  112
-5    0   -4
-4    0    3
-3   76  -79
-2  184  195
-1  375  380
 0   21   51
 1   22   17
 2   74   70
 3   89   74
 4   37   39
 5   49   57

H,K=  3, -5
 L FOBS FCAL
-8  111  118
-7   80   82
-6   25   45
-5   87   80
-4    0   19
-3  297  288
-2  217 -202
-1  138 -142
 0  164  150
 1  154  154
 2  189  201
 3  271  262
 4  228  221
 5   72   74
 6    0   12

H,K=  3, -4
 L FOBS FCAL
-8   69   71
-7   45   39
-6   84   93
-5  233  248
-4   99  101
-3   61   39
-2  202  200
-1  309 -326
 0   59  -41
 1  229  229
 2  349  358
 3  297  296
 4  323  321
 5    0  -33
 6   57  -53

H,K=  3, -3
 L FOBS FCAL
-9   30   24
-8   61   57
-7  132  132
-6  214  232
-5  145  160
-4  230  248
-3    0   24
-2  280  278
-1   86   71
 0  343  327
 1  373  340
 2  487  485
 3  108  115
 4  108  109
 5   58  -46
 6    0  -37

H,K=  3, -2
 L FOBS FCAL
-9    0   -8
-8   54   59
-7  224  224
-6  349  348
-5  419  413
-4  143  158
-3  181 -173
-2  345 -329
-1  148 -138
 0  449  421
 1  719  674
 2  459  453
 3  233  226
 4    0   14
 5   81   85
 6   28   34

H,K=  3, -1
 L FOBS FCAL
-9    0   29
-8   71   73
-7  325  319
-6  344  345
-5  304  312
-4  203  197
-3   69  -67
-2  492 -498
-1  202  209
 0  190  193
 1  144  150
 2  171  167
 3  370  354
 4  136  130
 5  102  101
 6  110  104
 7   30   49

H,K=  3,  0
 L FOBS FCAL
-9   41   41
-8   54   56
-7  154  165
-6  206  208
-5   46  -54
-4   32  -42
-3  709  732
-2  240  230
-1   51   55
 0  127  137
 1   96   93
 2   19   42
 3  281  276
 4  257  251
 5   67   75
 6  155  146
 7   42   31

H,K=  3,  1
 L FOBS FCAL
-9   82   98
-8  111  108
-7    0   -8
-6    0    9
-5  280  286
-4  159 -152
-3    0   -8
-2  207 -209
-1  330  332
 0  265  253
 1  228  204
 2    0   -1
 3   60   51
 4  270  286
 5  316  313
 6  146  141
 7    0    7

H,K=  3,  2
 L FOBS FCAL
-9   51   37
-8  152  161
-7  214  213
-6    0   -1
-5   73   59
-4  116   98
-3  156  141
-2  208  187
-1  120  133
 0  175 -176
 1  229 -227
 2  126  126
 3  530  528
 4  152  160
 5  155  136
 6    0   30

H,K=  3,  3
 L FOBS FCAL
-9    0   -4
-8   48  -28
-7   26   29
-6  166  182
-5  114  130
-4  670  685
-3  251  273
-2  190  165
-1  104 -190
 0  206 -221
 1  248  237
 2  160  152
 3  355  345
 4  285  294
 5   53   71
 6    0  -29

H,K=  3,  4
 L FOBS FCAL
-8    0   -3
-7   53  -48
-6  264  264
-5  368  392
-4  509  553
-3  246  262
-2   88  -93
-1   60  -80
 0  320 -323
 1    0  -29
 2  322  346
 3  135  139
 4  238  233
 5  225  203
 6   64   58

H,K=  3,  5
 L FOBS FCAL
-8   65   49
-7  173  179
-6  139  141
-5  294  288
-4  118  136
-3  158  180
-2   46   72
-1  208  224
 0    0    6
 1    0  -38
 2  235  236
 3  232  238
 4    0   14
 5  110  109
 6   51   83

H,K=  3,  6
 L FOBS FCAL
-8    0   -1
-7  108  101
-6   64   61
-5   90   65
-4   62   61
-3   32   22
-2    0    7
-1  129  157
 0  358  379
 1  318  316
 2   41   45
 3   76   94
 4    0   12
 5  118  111

H,K=  3,  7
 L FOBS FCAL
-7    0  -18
-6  103  105
-5   26   32
-4   56   37
-3    0  -18
-2  205  185
-1    0   11
 0   47   43
 1   42   45
 2    0   19
 3   26   51
 4  155  131
 5   98  100

H,K=  3,  8
 L FOBS FCAL
-6   50   37
-5   39   38
-4   79   90
-3   85   78
-2  248  221
-1   66   59
 0    0   -8
 1   81  -98
 2    0  -36
 3   73  -63
 4  120  114

H,K=  3,  9
 L FOBS FCAL
-5    0   45
-4  100   94
-3  124  122
-2  180  185
-1   88   96
 0    0  -11
 1   61  -74
 2   39  -55
 3    0  -18

H,K=  3, 10
 L FOBS FCAL
-4  120  120
-3  127  132
-2   28   49
-1   48   29
 0   40  -48
 1   50  -56
 2    0   32

H,K=  3, 11
 L FOBS FCAL
-2   42   36
-1   42  -28
 0   30   37

H,K=  4,-11
 L FOBS FCAL
-3   42   91
-2   59   51
-1    0    4
 0    0   -4

H,K=  4,-10
 L FOBS FCAL
-5   98  106
-4   58   61
-3   40   48
-2    0    3
-1   48   48
 0   28   22
 1  132  122
 2    0   22

H,K=  4, -9
 L FOBS FCAL
-6    0   30
-5   40   47
-4   48   38
-3    0    4
-2   46   51
-1  143  137
 0  186  177
 1   91   78
 2  120  121
 3   29   15

H,K=  4, -8
 L FOBS FCAL
-7  103  108
-6    0   14
-5  108 -103
-4   26  -33
-3    0    3
-2  205  214
-1  210  203
 0  278  288
 1  101   95
 2    0   18
 3    0   -9
 4   30   23

H,K=  4, -7
 L FOBS FCAL
-7    0   20
-6   38  -31
-5   58  -63
-4   43   44
-3  165  171
-2  247  256
-1  344  358
 0  170  159
 1  175  165
 2    0  -45
 3   77  -66
 4    0  -16

H,K=  4, -6
 L FOBS FCAL
-8   88   96
-7    0   19
-6   45  -38
-5    0  -13
-4    0  -13
-3  204  205
-2  153  131
-1  440  430
 0    0  -14
 1    0    9
 2   89   73
 3  169  173
 4   28   36
 5   59   65

H,K=  4, -5
 L FOBS FCAL
-8   86   86
-7   46   40
-6    0   -4
-5    0  -21
-4   39   55
-3  139  149
-2    0   -8
-1    0    8
 0  268  255
 1   55   64
```

balt atom is very close to square, although the S-metal-S angle within the five-membered ring is found to be somewhat greater than 91° in both the present structure determination and in that of Eisenberg, *et al.*[13]

The closest intermolecular contacts of the cobalt atom are two butyl carbon atoms from each of the cations at distances of C(9) = 3.927 ± 0.008 and C(5) = 4.179 ± 0.008 Å., together with the hydrogen atoms associated with these two carbon atoms, *viz.*, H(1)C(5) at 3.34, H(2)C(5) at 4.13, H(1)C(9) at 2.96, and H(2)-C(9) at 3.90 Å.

The cobalt atoms are very well separated, as the nearest distance of approach is the shortest lattice distance, 9.81 Å.

The Tetra-*n*-butylammonium Ion.—The configura-

TABLE IV (*Continued*)

L	FOBS	FCAL
2	41	41
3	101	107
4	81	93
5	81	68
H,K= 4, -4		
-8	62	74
-7	0	-14
-6	0	16
-5	45	40
-4	169	169
-3	0	2
-2	480	492
-1	171	-152
0	124	150
1	204	194
2	172	183
3	81	83
4	58	53
5	28	-26
H,K= 4, -3		
-9	42	42
-8	55	65
-7	51	60
-6	85	95
-5	93	95
-4	160	161
-3	253	-269
-2	75	79
-1	265	250
0	205	216
1	383	380
2	490	496
3	109	100
4	26	12
5	87	-89
6	72	-70
H,K= 4, -2		
-9	0	29
-8	121	119
-7	205	208
-6	210	213
-5	121	117
-4	69	-82
-3	130	-133
-2	130	-137
-1	239	222
0	595	592
1	483	475
2	411	419
3	40	-36
4	104	-100
5	0	-52
6	0	36
H,K= 4, -1		
-9	100	98
-8	137	130
-7	189	193
-6	134	136
-5	108	112
-4	48	54
-3	31	37
-2	280	275
-1	602	576
0	512	492
1	325	323
2	67	74
3	0	-7
4	35	38
5	38	-10
6	82	84
H,K= 4, 0		
-9	144	135
-8	178	166
-7	233	242
-6	210	208
-5	0	-27
-4	323	-353
-3	316	307
-2	111	117
-1	320	299
0	259	265
1	288	281
2	168	157
3	126	132
4	154	151
5	170	155
6	77	80
H,K= 4, 1		
-9	142	136
-8	194	199
-7	130	135
-6	65	69
-5	261	272
-4	190	-192
-3	99	-101
-2	172	160
-1	56	-58
0	244	245
1	346	330
2	137	133
3	222	207
4	125	121
5	127	130
6	0	13
H,K= 4, 2		
-9	29	37
-8	61	55
-7	25	33
-6	0	-33
-5	0	-17
-4	375	385
-3	38	4
-2	255	248
-1	78	84
0	261	263
1	234	229
2	240	242
3	229	240
4	172	169
5	67	-47
6	0	-16
H,K= 4, 3		
-9	0	20
-8	0	-1
-7	45	19
-6	155	134
-5	59	-73
-4	66	56
-3	272	286
-2	117	133
-1	91	-89
0	80	-51
1	96	106
2	237	247
3	270	271
4	343	328
5	28	-23
6	0	-57
H,K= 4, 4		
-8	49	58
-7	79	82
-6	116	110
-5	378	365
-4	110	107
-3	128	108
-2	338	-354
-1	41	-41
0	42	-28
1	346	339
2	349	353
3	134	123
4	0	21
5	0	32
H,K= 4, 5		
-8	0	38
-7	226	238
-6	198	222
-5	226	229
-4	73	63
-3	0	-19
-2	164	-179
-1	197	170
0	215	225
1	209	213
2	186	200
3	305	289
4	116	-93
5	29	5
H,K= 4, 6		
-8	67	60
-7	138	142
-6	235	245
-5	25	60
-4	87	111
-3	0	17
-2	51	-51
-1	0	8
0	152	163
1	226	220
2	36	44
3	181	174
4	98	109
H,K= 4, 7		
-7	0	10
-6	100	94
-5	70	58
-4	25	63
-3	78	97
-2	85	97
-1	91	89
0	96	99
1	105	95
2	116	113
3	56	57
4	77	111
H,K= 4, 8		
-6	0	-35
-5	0	16
-4	0	4
-3	45	41
-2	190	188
-1	146	163
0	59	90
1	0	-1
2	62	52
3	87	86
H,K= 4, 9		
-5	0	16
-4	132	107
-3	0	25
-2	55	37
-1	0	-6
0	28	-30
1	0	-8
2	50	49
H,K= 4, 10		
-4	133	125
-3	65	62
-2	0	-6
-1	0	-48
0	50	-69
1	51	-47
H,K= 5,-10		
-4	51	69
-3	41	45
-2	0	15
-1	0	-47
0	0	-7
H,K= 5, -9		
-6	0	9
-5	91	86
-4	84	77
-3	28	3
-2	27	11
-1	0	12
0	62	73
1	0	12
2	0	25
H,K= 5, -8		
-6	64	63
-5	0	-24
-4	0	-5
-3	117	122
-2	222	224
-1	197	207
0	103	97
1	0	30
2	130	-129
3	42	-31
H,K= 5, -7		
-7	50	-43
-6	0	3
-5	0	8
-4	95	94
-3	196	202
-2	209	205
-1	291	284
0	72	65
1	117	108
2	0	4
3	40	-39
H,K= 5, -6		
-8	42	43
-7	48	-52
-6	46	-45
-5	36	41
-4	177	181
-3	308	321
-2	204	201
-1	169	178
0	94	87
1	139	-148
2	83	81
3	151	130
4	65	84
H,K= 5, -5		
-8	50	-67
-7	0	11
-6	0	-9
-5	106	99
-4	222	217
-3	384	381
-2	320	325
-1	32	26
0	73	89
1	34	-35
2	110	-102
3	131	126
4	120	116
H,K= 5, -4		
-8	0	-11
-7	37	-42
-6	35	-14
-5	102	104
-4	173	172
-3	235	230
-2	93	87
-1	61	80
0	50	-19
1	243	265
2	78	80
3	37	55
4	0	37
5	78	71
H,K= 5, -3		
-9	0	-10
-8	68	79
-7	0	14
-6	54	70
-5	45	51
-4	0	10
-3	21	-35
-2	54	59
-1	334	335
0	121	120
1	113	113
2	133	153
3	85	88
4	38	-24
5	58	-47
H,K= 5, -2		
-9	0	-34
-8	27	32
-7	62	64
-6	86	85
-5	0	-10
-4	232	255
-3	70	83
-2	130	144
-1	210	223
0	423	429
1	235	233
2	139	134
3	138	-137
4	159	-139
5	64	-41
H,K= 5, -1		
-9	65	83
-8	66	57
-7	84	77
-6	78	97
-5	31	-38
-4	65	56
-3	0	6
-2	348	341
-1	499	493
0	536	535
1	69	90
2	46	-44
3	90	-94
4	0	-36
5	81	70
H,K= 5, 0		
-9	194	176
-8	202	191
-7	145	146
-6	0	-1
-5	126	-151
-4	129	-122
-3	244	241
-2	544	548
-1	482	481
0	235	248
1	0	18
2	0	-8
3	35	-38
4	38	24
5	114	109
H,K= 5, 1		
-9	96	104
-8	124	122
-7	51	38
-6	0	-16
-5	121	129
-4	163	149
-3	150	161
-2	428	431
-1	285	280
0	277	267
1	137	137
2	110	103
3	0	50
4	0	19
5	76	71
H,K= 5, 2		
-9	66	68
-8	73	75
-7	36	36
-6	68	-64
-5	50	66
-4	184	183
-3	80	79
-2	120	110
-1	178	210
0	127	116
1	201	204
2	156	152
3	179	179
4	207	194
5	0	5
H,K= 5, 3		
-9	66	64
-8	48	51
-7	69	69
-6	35	39
-5	0	9
-4	108	-95
-3	262	261
-2	139	140
-1	102	109
0	69	61
1	196	205
2	260	265
3	26	52
4	78	73
5	29	-37
H,K= 5, 4		
-8	0	6
-7	0	-15
-6	44	19
-5	186	201
-4	103	109
-3	50	-47
-2	159	169
-1	117	118
0	297	292
1	269	280
2	215	223
3	26	-29
4	105	-106
H,K= 5, 5		
-8	0	30
-7	117	116
-6	146	130
-5	0	17
-4	127	125
-3	121	-118
-2	73	-59
-1	74	83
0	210	222
1	225	211
2	183	189
3	98	95
4	50	-65
H,K= 5, 6		
-7	140	143
-6	205	205
-5	37	32
-4	146	-157
-3	69	53
-2	59	-47
-1	149	144
0	206	204
1	225	220
2	38	7
3	0	19
4	0	27
H,K= 5, 7		
-7	66	64
-6	0	-4
-5	72	83
-4	102	-95
-3	129	118
-2	105	93
-1	198	185
0	123	131
1	175	176
2	0	40
3	29	25
H,K= 5, 8		
-6	0	11
-5	0	22
-4	0	28
-3	0	31
-2	123	130
-1	101	96
0	67	84
1	56	51
2	58	61
H,K= 5, 9		
-5	0	3
-4	116	117
-3	0	37
-2	49	64
-1	0	23
0	50	53
1	72	70
H,K= 5, 10		
-2	0	12
H,K= 6,-10		
-3	0	23
-2	0	5
H,K= 6, -9		
-5	102	92
-4	64	71
-3	0	-20
-2	0	6
-1	0	16
0	41	28
H,K= 6, -8		
-6	116	126
-5	0	6
-4	0	9
-3	0	-25
-2	0	37
-1	55	50
0	39	32
1	0	50
2	42	34
H,K= 6, -7		
-7	0	-1
-6	40	52
-5	77	76
-4	53	58
-3	73	64
-2	127	125
-1	45	40
0	0	-3
1	73	-75
2	50	75
H,K= 6, -6		
-8	0	1
-7	0	17
-6	72	-77
-5	133	156
-4	309	306
-3	349	346
-2	170	165
-1	56	49
0	36	-40
1	158	-151
2	0	5
3	92	107
H,K= 6, -5		
-8	77	-105
-7	0	38
-6	134	131
-5	192	202
-4	156	150
-3	320	331
-2	160	161
-1	59	45
0	0	-25
1	96	90
2	38	-38
3	182	184
H,K= 6, -4		
-8	0	38
-7	0	-36
-6	155	147
-5	257	263
-4	232	211
-3	134	147
-2	0	4
-1	47	-22
0	80	-80
1	255	242
2	158	140
3	83	76
4	102	89
H,K= 6, -3		
-9	73	-71
-8	0	25
-7	0	18
-6	125	127
-5	244	245
-4	260	257
-3	147	144
-2	59	70
-1	0	24
0	0	41
1	42	-20
2	134	131
3	142	124
4	82	68
H,K= 6, -2		
-9	0	-16
-8	48	-52
-7	101	110
-6	65	78
-5	70	65
-4	92	87
-3	73	80
-2	167	168
-1	96	98
0	175	182
1	80	92
2	0	-11
3	0	4
4	64	61
H,K= 6, -1		
-9	101	100
-8	39	46
-7	52	51
-6	54	53
-5	57	-84
-4	59	61
-3	191	188
-2	191	188
-1	283	282
0	238	201
1	41	30
2	67	-74
3	129	-130
4	49	-28
H,K= 6, 0		
-9	41	32
-8	67	70
-7	0	-15
-6	84	108
-5	69	75
-4	135	137
-3	325	318
-2	402	407
-1	354	364
0	162	157
1	133	-142
2	212	-206
3	38	-30
4	0	39
H,K= 6, 1		
-9	42	37
-8	48	56
-7	0	4
-6	0	-32
-5	52	60
-4	254	256
-3	416	422
-2	321	333
-1	143	144
0	133	-108
1	72	-68
2	0	-1
3	170	153
4	122	124
H,K= 6, 2		
-9	89	99
-8	39	38
-7	0	-30
-6	50	-46
-5	121	130
-4	270	284
-3	282	285
-2	89	93
-1	81	96
0	96	91
1	0	37
2	36	20
3	109	102
4	96	116
H,K= 6, 3		
-8	0	16
-7	54	53
-6	88	67
-5	49	40
-4	133	113
-3	134	145
-2	352	359
-1	165	176
0	164	168
1	50	70
2	111	118
3	0	-5
4	41	42
H,K= 6, 4		
-8	0	3
-7	39	22
-6	58	53
-5	75	84
-4	200	193
-3	48	-54
-2	161	146
-1	86	86
0	173	169
1	112	128
2	226	200
3	0	2
H,K= 6, 5		
-8	0	9
-7	49	56
-6	47	53
-5	45	55
-4	140	153
-3	61	53
-2	35	-52
-1	201	198
0	190	173
1	106	109
2	28	25
3	65	-37
H,K= 6, 6		
-7	65	55
-6	74	73
-5	0	-4
-4	146	-139
-3	191	190
-2	143	159
-1	241	245
0	162	163
1	48	49
2	40	-59
H,K= 6, 7		
-6	0	-45
-5	48	45
-4	61	-58
-3	0	-3
-2	178	169
-1	115	112
0	136	130
1	64	68
2	0	-21
H,K= 6, 8		
-5	0	-8
-4	64	60
-3	0	18
-2	141	123
-1	110	101
0	0	43
1	51	32
H,K= 6, 9		
-3	122	113
-2	72	75
-1	78	83
H,K= 7, -9		
-4	0	21
-3	0	-12
-2	0	24
H,K= 7, -8		
-5	0	-10
-4	29	-23
-3	28	-50
-2	0	10
-1	58	72
0	72	74
H,K= 7, -7		
-6	41	40
-5	0	28
-4	0	7
-3	0	14
-2	39	51
-1	55	41
0	0	10
1	0	-31
H,K= 7, -6		
-7	29	49
-6	28	-21
-5	38	24
-4	172	178
-3	69	64
-2	0	7
-1	38	-23
0	0	18
1	0	14
2	51	53
H,K= 7, -5		
-8	42	4
-7	40	40
-6	217	205
-5	87	83
-4	126	122
-3	116	112
-2	0	18
-1	82	-82
0	53	-35
1	0	55
2	100	106
H,K= 7, -4		
-8	29	46
-7	39	42
-6	205	197
-5	420	427
-4	198	183
-3	65	72
-2	0	-26
-1	71	-81
0	0	4
1	123	127
2	152	173
3	42	45
H,K= 7, -3		
-8	0	39
-7	234	235
-6	69	44
-5	266	273
-4	122	129
-3	48	40
-2	0	-44
-1	78	67
0	88	68
1	137	139
2	177	170
3	143	147
H,K= 7, -2		
-8	0	17
-7	170	164
-6	162	168
-5	82	67
-4	0	-5
-3	0	-9
-2	0	20
-1	94	96
0	117	110
1	167	152
2	78	68
3	112	95
H,K= 7, -1		
-8	93	91
-7	0	34
-6	210	206
-5	77	81
-4	145	137
-3	41	60
-2	85	87
-1	72	74
0	55	42
1	86	86
2	54	57
3	0	-7
H,K= 7, 0		
-8	63	63
-7	0	-10
-6	67	66
-5	73	84
-4	116	106
-3	220	204
-2	174	178
-1	110	102
0	0	-9
1	142	-125
2	38	-24
3	118	120
H,K= 7, 1		
-8	0	19
-7	0	24
-6	0	-18
-5	135	131
-4	169	160
-3	290	296
-2	181	192
-1	42	-19
0	139	-135
1	69	-88
2	72	-58
3	96	117
H,K= 7, 2		
-8	0	-4
-7	38	25
-6	82	88
-5	218	214
-4	294	296
-3	281	283
-2	90	99
-1	35	-53
0	80	-82
1	0	-11
2	48	34
3	101	103
H,K= 7, 3		
-8	0	2
-7	55	34
-6	138	139
-5	203	211
-4	185	180
-3	78	74
-2	43	39
-1	56	-52
0	0	24
1	123	131
2	109	124
3	142	122
H,K= 7, 4		
-8	0	20
-7	63	73
-6	118	117
-5	105	93
-4	154	142
-3	36	37
-2	44	55
-1	113	115
0	46	32
1	68	76
2	91	97
H,K= 7, 5		
-7	0	-4
-6	74	58
-5	0	23
-4	182	184
-3	188	195
-2	64	65
-1	110	128
0	173	160
1	0	-6
2	0	-3
H,K= 7, 6		
-6	100	94
-5	0	11
-4	0	-34
-3	82	74
-2	187	173
-1	62	61
0	144	138
1	41	-29
H,K= 7, 7		
-5	50	58
-4	40	31
-3	0	11
-2	160	138
-1	86	81
0	0	20
H,K= 7, 8		
-4	111	111
-3	150	143
-2	72	65
H,K= 8, -8		
-3	30	52
H,K= 8, -7		
-5	41	-40
-4	0	9
-3	50	23
-2	122	121
-1	121	111
H,K= 8, -6		
-6	0	-12
-5	85	-113
-4	0	55
-3	28	27
-2	28	39
-1	49	41
0	137	123
H,K= 8, -5		
-7	29	12
-6	85	109
-5	39	50
-4	0	6
-3	38	64
-2	0	-23
-1	48	-34
0	28	51
1	125	110
H,K= 8, -4		
-7	86	86
-6	92	85
-5	247	234
-4	0	-11
-3	59	-45
-2	65	-55
-1	0	5
0	68	75
1	132	142
H,K= 8, -3		
-8	110	100
-7	194	203
-6	67	53
-5	65	60
-4	116	116
-3	58	-76
-2	45	-49
-1	46	39
0	122	137
1	153	166
2	59	75
H,K= 8, -2		
-8	117	109
-7	188	188
-6	302	284
-5	0	-8
-4	0	3
-3	36	-51
-2	73	63
-1	176	158
0	158	155
1	84	108
2	41	70
H,K= 8, -1		
-8	116	129
-7	83	82
-6	80	74
-5	82	75
-4	0	-13
-3	0	28
-2	57	54
-1	110	107
0	170	155
1	156	149
2	185	174
H,K= 8, 0		
-8	41	47
-7	108	93
-6	38	-41
-5	58	61
-4	0	16
-3	62	55
-2	51	51
-1	116	111
0	85	83
1	0	28
2	117	92
H,K= 8, 1		
-8	51	36
-7	56	54
-6	60	64
-5	91	88
-4	99	99
-3	142	149
-2	85	99
-1	0	-5
0	54	-48
1	0	-15
2	29	40
H,K= 8, 2		
-8	51	34
-7	0	30
-6	102	90
-5	176	176
-4	161	164
-3	68	76
-2	52	-53
-1	110	-128
0	103	-88
1	127	114
2	145	145
H,K= 8, 3		
-7	70	75
-6	152	140
-5	209	205
-4	96	88
-3	37	-29
-2	96	-96
-1	54	-51
0	28	-28
1	126	123
H,K= 8, 4		
-7	93	95
-6	139	123
-5	111	121
-4	105	108
-3	0	27
-2	0	12
-1	48	53
0	0	27
1	98	83
H,K= 8, 5		
-6	101	93
-5	0	24
-4	56	60
-3	39	47
-2	0	13
-1	40	49
0	152	153
H,K= 8, 6		
-5	51	48
-4	0	-7
-3	58	52
-2	112	112
-1	0	15
H,K= 9, -6		
-5	30	13
-4	98	81
-3	118	111
-2	0	40
H,K= 9, -5		
-6	0	7
-5	41	41
-4	41	32
-3	135	127
-2	104	93
-1	0	25
H,K= 9, -4		
-6	0	1
-5	0	34
-4	0	11
-3	63	-60
-2	120	117
-1	108	85
0	123	121
H,K= 9, -3		
-7	93	104
-6	57	69
-5	40	-45
-4	28	39
-3	55	-42
-2	0	5
-1	90	101
0	181	165
H,K= 9, -2		
-7	130	124
-6	90	100
-5	0	-10
-4	123	-117
-3	27	33
-2	48	56
-1	149	157
0	105	124
H,K= 9, -1		
-7	65	74
-6	0	-12
-5	0	36
-4	54	-41
-3	54	45
-2	95	112
-1	146	152
0	112	117
H,K= 9, 0		
-7	65	61
-6	0	-8
-5	48	-61
-4	122	124
-3	119	104
-2	168	153
-1	101	107
0	116	122
H,K= 9, 1		
-7	0	1
-6	80	86
-5	0	-21
-4	48	34
-3	39	51
-2	88	96
-1	106	102
0	157	137
H,K= 9, 2		
-7	42	35
-6	41	15
-5	79	91
-4	0	21
-3	28	46
-2	0	-0
-1	0	-16
0	42	33
H,K= 9, 3		
-6	58	52
-5	154	158
-4	80	85
-3	0	-8
-2	75	-78
-1	0	-9
H,K= 9, 4		
-6	84	86
-5	51	36
-4	0	-33
-3	71	-79
-2	58	-51
-1	78	80
H,K= 9, 5		
-4	42	-36
-3	0	-14
H,K= 10, -3		
-5	42	41
-4	72	51
-3	84	79
H,K= 10, -2		
-5	0	-5
-4	29	-45
-3	101	91
-2	145	132
H,K= 10, -1		
-6	0	-14
-5	0	-15
-4	58	56
-3	0	35
-2	177	170
H,K= 10, 0		
-6	0	3
-5	138	-67
-4	87	101
-3	109	111
-2	89	98
H,K= 10, 1		
-5	51	34
-4	0	17
-3	138	127
-2	99	108
H,K= 10, 2		
-4	89	95
-3	115	97

tion of this ion can be seen in Fig. 1 and the important dimensions are listed in Table VII. Three of the butyl chains adopt the *trans* conformation but one (C(17)–C(20)) adopts a *gauche* conformation, presumably to improve the packing and to avoid too close approach to the Co atom. The dihedral angles for all the chains are listed in Table VIII. The angles for the *trans* chains are very close to zero, showing them to be very nearly planar. The dihedral angle for the *gauche* chain is 71.6°, in comparison with a mean dihedral angle of 61 ± 3° found by Bartell and Kohl[17] in an electron diffraction study of free hydrocarbon chains in the gas phase and the ideal value at 60°.

The four C–N bonds are equal to within the experimental accuracy and have an average value of 1.52 ± 0.01 Å. The six tetrahedral angles at the nitrogen

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(17) L. S. Bartell and D. A. Kohl, International Congress of X-Ray Crystallography, Rome, 1963, Abstract 5.31.

TABLE V

A COMPARISON OF INTERATOMIC DISTANCES (Å.) AND ANGLES IN THE TWO IONS $Co(MNT)_2^{2-}$ AND $Ni(MNT)_2^{2-}$

	$Co(MNT)_2^{2-}$		$Ni(MNT)_2^{2-}$	
Atoms	Distance	E.s.d.	Distance	E.s.d.
Ni/Co–S(1)	2.159	0.003	2.16	0.008
Ni/Co–S(2)	2.163	0.003	2.16	0.008
S(1)–C(1)	1.715	0.007	1.75	*a*
S(2)–C(2)	1.731	0.007	1.75	*a*
C(1)–C(3)	1.40	0.010	1.44	*a*
C(2)–C(4)	1.40	0.010	1.42	*a*
C(1)–C(2)	1.34	0.010	1.30	0.02
C(3)–N(1)	1.15	0.010	1.13	0.02
C(4)–N(2)	1.16	0.010	1.13	0.02
Atoms	Angles	E.s.d.	Angles[b]	
S(1)–Co/Ni–S(2)	91.4°	0.1°	91.7°	
Co/Ni–S(1)–C(1)	103.8°	0.3°	103°	
S(1)–C(1)–C(2)	121.1°	0.5°	123°	
C(1)–C(2)–S(2)	120.0°	0.6°	120°	
Co/Ni–S(2)–C(2)	103.7°	0.3°	103°	
S(2)–C(2)–C(4)	117.2°	0.6°	...	
S(1)–C(1)–C(3)	118.1°	0.5°	...	
C(2)–C(1)–C(3)	120.8°	0.7°	120°	
C(1)–C(3)–N(1)	178.2°	0.8°	179°	
C(1)–C(2)–C(4)	122.8°	0.7°	124°	
C(2)–C(4)–N(2)	178.7°	1.0°	177°	

[a] Intermediate between ±0.008 and ±0.02 Å. [b] E.s.d. all approximately ±1.0°.

TABLE VI

DISTANCES OF THE VARIOUS ATOMS (Å.) IN THE $Co(MNT)_2^{2-}$ ION FROM THE PLANE THROUGH ATOMS Co, C(1), AND C(2)

Atom	Distance from plane	E.s.d. of the atomic position	E.s.d. of the plane at a particular atom
Co	0	0	0
S(1)	+0.049	0.002	0.004
S(2)	+0.004	0.002	0.004
C(1)	0	0.007	0.007
C(2)	0	0.007	0.007
C(3)	−0.035	0.008	0.010
C(4)	−0.031	0.009	0.010
N(1)	−0.062	0.007	0.012
N(2)	−0.058	0.009	0.012

TABLE VII

DISTANCES AND ANGLES INVOLVING THE CARBON AND NITROGEN ATOMS IN THE TETRA-*n*-BUTYLAMMONIUM GROUP

Atoms	Distance,[a] Å.	Atoms	Angles,[b] deg.
N(3)–C(5)	1.53	C(5)–N(3)–C(9)	106.3
N(3)–C(9)	1.52	C(5)–N(3)–C(13)	111.7
N(3)–C(13)	1.51	C(5)–N(3)–C(17)	109.4
N(3)–C(17)	1.53	C(9)–N(3)–C(13)	112.9
		C(9)–N(3)–C(17)	110.3
		C(13)–N(3)–C(17)	106.3
Mean C–N dist.	1.52	Mean C–N–C angle	109.5

Atoms	Distance,[c] Å.	Distance,[d] Å.	Atoms	Angles, deg.	E.s.d., deg.
C(5)–C(6)	1.49	1.49	N(3)–C(5)–C(6)	115.6	0.5
C(6)–C(7)	1.53	1.54	C(5)–C(6)–C(7)	108.9	0.6
C(7)–C(8)	1.53	1.56	C(6)–C(7)–C(8)	112.5	0.7
C(9)–C(10)	1.53	1.54	N(3)–C(9)–C(10)	114.0	0.6
C(10)–C(11)	1.51	1.52	C(9)–C(10)–C(11)	109.5	0.7
C(11)–C(12)	1.54	1.54	C(10)–C(11)–C(12)	114.3	0.8
C(13)–C(14)	1.48	1.49	N(3)–C(13)–C(14)	115.7	0.6
C(14)–C(15)	1.52	1.53	C(13)–C(14)–C(15)	112.4	0.6
C(15)–C(16)	1.48	1.49	C(14)–C(15)–C(16)	114.2	0.7
C(17)–C(18)	1.52	1.53	N(3)–C(17)–C(18)	115.3	0.6
C(18)–C(19)	1.54	1.54	C(17)–C(18)–C(19)	111.4	0.7
C(19)–C(20)	1.51	1.53	C(18)–C(19)–C(20)	113.3	0.8
Mean C–C dist.	1.515	1.525	Mean angle	113.1	

[a] E.s.d. all ±0.01 Å. [b] E.s.d. all ±0.5°. [c] E.s.d. all ±0.01 Å. [d] Corrected for thermal vibration assuming that atom C(n + 1) "rides" on atom C(n).

TABLE VIII

THE DIHEDRAL ANGLES FOR THE FOUR BUTYL CHAINS IN THE TETRA-*n*-BUTYLAMMONIUM ION

Plane (1) defined by atoms			Plane (2) defined by atoms			Dihedral angle, deg.
C(5)	C(6)	C(7)	C(6)	C(7)	C(8)	6.5
C(9)	C(10)	C(11)	C(10)	C(11)	C(12)	1.6
C(13)	C(14)	C(15)	C(14)	C(15)	C(16)	1.3
C(17)	C(18)	C(19)	C(18)	C(19)	C(20)	71.6

atom (C–N–C) average 109.5° with a spread of ±3° and are very close to the tetrahedral angle.

In the butyl chains, the mean C–C distance is 1.515 Å. Applying a thermal correction, assuming that each carbon atom "rides" on its neighbor nearer to the central nitrogen atom, increases this distance by 0.010 Å. This assumption is reasonable as the temperature factors of the carbon atoms increase progressively along each carbon chain (see Table I). The mean value of the twelve N–C–C and C–C–C angles is 113.1°, somewhat greater than the tetrahedral angle. The four N–C–C angles all have higher than average values.

Most of the hydrogen atoms are suitably placed with respect to bond distances and angles. The C–H bond lengths range from 0.8 to 1.1 Å., with the average of the 36 values being 0.98 Å. Several of the bond angles deviate substantially from tetrahedral, but the majority are very close. In particular, except for the hydrogen atoms on the terminal carbon atoms of the four *n*-butyl chains, all the hydrogen bond angles are within 15° of the expected value; consistent with the estimated standard deviations of the hydrogen atom positions. It is noteworthy that all the hydrogen atoms except one have calculated temperature factors less than those of the carbon atoms with which they are associated.

X-Ray powder diffraction photographs of material from the sample studied in this investigation and of material used in the e.s.r. experiments (kindly supplied by Dr. N. Edelstein) are consistent but do not agree with the *d*-spacings reported by Davison, *et al.*[5]

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