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The Crystal and Molecular Structure of Dichloro(ethylenedimorpholine)cobalt(II)

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The crystal structure of dichloro(ethylenedimorpholine)cobalt(II) ($Co(C_{10}H_{20}N_2O_2)Cl_2$) has been determined from three-dimensional single-crystal X-ray diffraction data collected by counter methods. The structure has been refined anisotropically by full-matrix least-squares methods to a conventional R factor of 6.2% for 750 nonzero reflections. The compound crystallizes in the monoclinic space group C2/c with $a = 10.59 \pm 0.02$ Å, $b = 11.19 \pm 0.02$ Å, $c = 12.35 \pm 0.02$ Å, and $\beta = 109° 15' \pm 15'$. An experimental density of 1.56 g/cm³ agrees with the calculated density of 1.54 g/cm³ based on four molecules per unit cell. The discrete monomeric pseudotetrahedral molecules possess crystallographically required C_2 symmetry. The Co–Cl bond length is 2.229 (3) Å, while the Co–N bond distance is 2.116 (8) Å. The six-membered rings have the chair conformation. Distortions are observed in the idealized tetrahedral coordination of the cobalt atom: N–Co–Cl, 109.0 (3) and 115.0 (3)°; N–Co–N, 88.9(4)°; Cl–Co–Cl, 116.8 (2)°.

Introduction

Various N-alkylethylenediamines and systems derived from ethylenediamine have been observed to force unusual coordination geometries. 1,1,7,7-Tetraethyldiethylenetriamine (Et_4dien) forms a pentacoordinate complex $Co(Et_4dien)Cl_2$.[2] The MLX_2 complexes when L is N,N,N′,N′-tetramethylated ethylenediamine (Me_4en), 1,2-propylenediamine (Me_4pn), or trimethylenediamine (Me_4tn) have been investigated. All cobalt(II) halide complexes[3] and the iron(II) bromide complexes[4] are pseudotetrahedral. The nickel(II) bromides and iodides have been assigned a pseudotetrahedral structure.[3] In the solid, Me_4en and Me_4pn derivatives of nickel chloride are octahedral chloride-bridged polymers while the Me_4tn derivative has been assigned a pseudotetrahedral structure.[3] The copper(II) bromide complex of Me_4tn was found to be tetrahedral while the Me_4en- and Me_4pn–Cu(II) complexes are square planar.[4] Thus a bulky ligand and/or large anion are required to force tetrahedral coordination.

The ligand N,N′-ethylenedimorpholine (EDM), $C_{10}H_{20}N_2O_2$, has been observed to form a series of complexes $M(EDM)X_2$ (M = Co, Ni, Cu; X = Cl, Br, I)[5] which have been assigned tetrahedral coordinations from their physical properties.

The complexes (except for $Ni(EDM)Br_2$, $Ni(EDM)I_2$, $Co(EDM)I_2$) are isomorphous based on the similarity of their powder patterns. A three-dimensional X-ray crystal structure of the cobalt complex was undertaken to verify the tetrahedral structure of these complexes and to investigate the factors which cause N,N′-ethylenedimorpholine to form pseudotetrahedral complexes so readily.

Experimental Section

Crystals of $Co(EDM)Cl_2$ were kindly supplied by Mr. A. L. Lott, II.

Unit Cell and Space Group.—Crystals of $Co(EDM)Cl_2$ are monoclinic with lattice parameters $a = 10.59 \pm 0.02$ Å, $b = 11.19 \pm 0.02$ Å, $c = 12.35 \pm 0.02$ Å, and $\beta = 109° 15' \pm 15'$. Unit cell parameters were determined from the $hk0$ and $0kl$ precession photographs. The angle β was determined from the $h0l$ Weissenberg photograph. The precision in the measurement of the spot positions on the photographs was used to estimate lattice constant errors. Cu Kα radiation (λ 1.5418 Å) was used for all photographs. The systematic absences $h + k = 2n + 1$ for all hkl and $l = 2n + 1$ for $h0l$ limited the choice of probable space groups to Cc (C_s^4) or C2/c (C_{2h}^6).[6] The space group C2/c was confirmed by the solution of the structure. The volume of the unit cell is 1440 (7) Å³. The density, determined by flotation in chloroform–carbon tetrachloride, was 1.56 ± 0.03 g/cm³, which agrees with the calculated density of 1.54 g/cm³ based on four molecules per unit cell.

The cobalt atoms are located on twofold axes corresponding to sets of fourfold positions (4e), and the remaining atoms occupy general eightfold positions (8f). The total number of electrons per unit cell, $F(000)$, is 684.

Single-Crystal Data.—A crystal 0.3 × 0.03 × 0.03 mm was mounted with the long axis coincident with the crystal rotation axis. Intensity data were collected with a Weissenberg geometry automated diffractometer (Charles Supper Co.), used in conjunction with a fully stabilized constant-potential Philips X-ray generator (operated at 40 kV/12 mA), a Philips transistorized scintillation counter, and a Philips electronic panel. The pulse height analyzer was adjusted to receive 90% of the Mo Kα peak. The balanced filter technique using Zr and Y foil was used to filter the Mo radiation. A fixed-counter, moving-crystal method was used where the crystal scan range in degrees ($\Delta\phi$) was defined by $\Delta\phi = (r/\cos^2 \nu \sin \gamma) + k$, where γ is the counter setting, ν is the equiinclination angle, and r and k are empirical constants which were $r = 0.35$ and $k = 3$. The crystal scan range was broken into 10 scan and count steps (I_i). A scan speed of 1°/min was used whenever $\Delta\phi \leq 4°$, and 2°/min was used whenever $\Delta\phi > 4°$. The step scan data I_i were used to determine which steps belonged to background regions. The background was based on a least-squares line through all steps in the background region.[7] Ten levels of data corresponding to $hk0$–$hk9$ were collected. A total of 1332 unique reflections were recorded; 750 had $F_o^2 > 3\sigma(F^2)$ and were included in the refine-

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[1] Taken in part from the Ph.D. thesis of W. R. Scheidt, The University of Michigan, 1968.

[2] Z. Dori and H. B. Gray, *J. Am. Chem. Soc.*, **88**, 1394 (1966); A. Dori, R. Eisenberg, and H. B. Gray, *Inorg. Chem.*, **6**, 483 (1967).

[3] L. Sacconi, I. Bertini, and F. Mani, *ibid.*, **6**, 262 (1967).

[4] I. Bertini and F. Mani, *ibid.*, **6**, 2032 (1967).

[5] A. L. Lott, II, and P. G. Rasmussen, *J. Inorg. Nucl. Chem.*, in press.

[6] "International Tables for X-Ray Crystallography," Vol. 1, The Kynoch Press, Birmingham, England, 1953, p 101.

[7] J. C. Hanson, Ph.D. Thesis, The University of Michigan, 1969.

TABLE I[a]

ATOMIC PARAMETERS WITH STANDARD DEVIATIONS

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{23}	b_{13}
Co	5000 (–)[b]	−659 (1)	2500 (–)[b]	59 (2)	37 (1)	47 (2)	0 (–)[b]	0 (–)[b]	12 (2)
Cl	5036 (3)	−1703 (2)	974 (3)	110 (3)	85 (2)	68 (4)	−32 (4)	53 (4)	22 (5)
N	3525 (7)	−691 (7)	1962 (7)	71 (7)	32 (9)	32 (9)	−2 (11)	−5 (10)	34 (11)
C_5	4385 (10)	+1754 (8)	1980 (10)	85 (10)	51 (7)	96 (13)	−8 (12)	−34 (14)	35 (16)
O	942 (7)	−376 (8)	1536 (8)	69 (7)	130 (9)	78 (9)	13 (11)	24 (14)	25 (12)
C_1	2738 (9)	+898 (8)	2710 (10)	80 (10)	76 (8)	41 (13)	−19 (12)	−1 (12)	29 (15)
C_2	1814 (8)	−169 (10)	2673 (10)	58 (9)	111 (10)	49 (11)	52 (14)	11 (15)	20 (14)
C_3	1684 (10)	−582 (9)	801 (11)	70 (9)	89 (9)	73 (13)	−3 (14)	33 (16)	−4 (16)
C_4	2568 (9)	−484 (10)	871 (10)	67 (9)	122 (11)	42 (10)	−72 (16)	−2 (17)	−7 (14)
$H_1(C_5)$[c]	38 (1)	+25 (1)	19 (1)						
$H_2(C_5)$[c]	48 (1)	+18 (1)	13 (1)						
$H_1(C_1)$[c]	23 (1)	+17 (1)	26 (1)						
$H_2(C_1)$[c]	32 (2)	+10 (1)	35 (1)						
$H_1(C_2)$[c]	24 (1)	−11 (1)	30 (1)						
$H_2(C_2)$[c]	12 (1)	0 (1)	33 (1)						
$H_1(C_3)$[c]	22 (1)	−13 (1)	9 (1)						
$H_2(C_3)$[c]	9 (1)	−8 (1)	1 (1)						
$H_1(C_4)$[c]	10 (1)	+10 (1)	5 (1)						
$H_2(C_4)$[c]	29 (1)	+1 (1)	3 (1)						

[a] All values $\times 10^4$ except hydrogen $\times 10^2$. Coordinates are fractional coordinates of the monoclinic cell. Thermal parameters are of the form $\exp[-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{23}kl + b_{13}hl)]$. Numbers in parentheses here and in succeeding tables are standard deviations in the least significant digits. [b] Fixed parameters. [c] B(isotropic) = 3.5 Å^2.

ment. Of the unobserved data, 193 reflections were found to have values of F_o in the range of 1–2σ, 10 between 2σ and 3σ, and 20 greater than 3σ. Intensities were corrected for Lorentz and polarization factors. No corrections were made for absorption; the transmission coefficients varied from 2.29 to 2.12. Standard reflections were monitored every 25th reflection. No decomposition of the crystal was noted.

Determination of the Structure.—The data were placed on an approximate absolute scale by the Wilson method.[8] A three-dimensional Patterson function, with origin peak removed, gave approximate positions for the cobalt, nitrogen, and chlorine atoms. A three-dimensional Fourier synthesis phased by these atoms clearly showed the carbon and oxygen atoms. A structure factor calculation of these atoms gave an agreement factor, $R = \Sigma||F_o - F_c||/\Sigma|F_o|$, of 25%. Scattering factors were from Cox and Bonham.[9] No corrections for anomalous dispersion were applied.

Five cycles of full-matrix isotropic least-squares refinement[10] of the nine nonhydrogen atoms in the asymmetric unit minimizing $\Sigma w(F_o - F_c)^2$ yielded an R of 16%. Weights were assigned as follows: $w = 1$ if $F_o < 4F_{\min}$; $\sqrt{w} = 4F_{\min}/F_o$ if $F_o > 4F_{\min}$. A difference Fourier suggested anisotropic thermal motion for the cobalt and chlorine atoms. Five cycles of refinement where the chlorine and cobalt atoms had anisotropic parameters reduced R to 9.2%. Two additional cycles of refinement using anisotropic thermal parameters for all atoms reduced R to 8.2%. A difference Fourier map function clearly showed the positions of the ten unique hydrogen atoms. One cycle of refinement with anisotropic thermal parameters for the nonhydrogen atoms and isotropic thermal parameters for the hydrogens gave an R of 7.3%. The thermal parameters of the hydrogens showed considerable variation and were therefore held constant ($B = 3.5$) for the remainder of the refinement. Two additional cycles of refinement of positional parameters of all atoms and anisotropic thermal parameters for all nonhydrogen atoms gave a final R of 6.2%. A final difference Fourier showed no peaks higher than 10% of a carbon atom peak. Final positional and thermal parameters with estimated standard deviations are listed in Table I. Table II lists the observed and calculated structure factors on the scale of electron units. The atom position and thermal ellipsoids are shown in Figure 1.

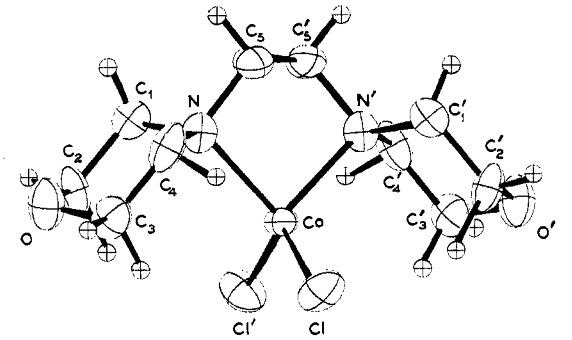

Figure 1.—The monomeric Co(EDM)X_2 unit showing 50% probability thermal ellipsoids for nonhydrogen atoms. One hydrogen has been omitted from each of C_1, C_1', C_4, C_4', C_5, and C_5' for clarity. The required twofold axis is in the plane of the paper.

Results and Discussion

Dichloro(N,N′-ethylenedimorpholine)cobalt(II) exists as discrete monomers as can be seen in Figure 2.[11] The molecule has crystallographically required point group symmetry C_2. The cobalt atom exhibits pseudotetrahedral coordination, where the tetrahedron is somewhat distorted, especially the N′–Co–N angle which is only 88.9 (4)°. Bond distances and angles with estimated standard deviations are listed in Table

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(8) D. Rogers in "Computing Methods in Crystallography," J. S. Rollett, Ed., Pergamon Press, London, 1965, pp 133–148; J. W. Schilling, "ANW, Anisotropic Wilson Plot Program for the IBM 7090," Ph.D. Thesis, the University of Michigan, 1968.

(9) H. L. Cox and R. A. Bonham, *J. Chem. Phys.*, **47**, 2599 (1967).

(10) W. R. Busing, K. O. Martin, and H. A. Levy, "ORFLS, a Fortran Crystallographic Least-Squares Program," Oak Ridge Technical Manual 305, modified by R. Shiono, mimeographed paper, Oak Ridge least-squares program modified for the Crystallography Laboratory of the University of Pittsburgh, and by J. W. Schilling, Ph.D. Thesis, The University of Michigan, 1968.

(11) C. K. Johnson, "ORTEP, a Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustration," Oak Ridge Technical Manual 3794, Oak Ridge National Laboratory, Oak Ridge, Tenn., June 1965.

Table II

Calculated and Observed Structure Factors

H	K	L	FOBS	FCAL
0	0	2	2345	-2021
0	0	4	246	259
0	0	6	1274	-1333
0	0	8	311	301
0	2	0	2159	1973
0	2	1	537	510
0	2	2	1245	-1152
0	2	3	716	688
0	2	4	853	858
0	2	5	338	340
0	2	6	311	-339
0	2	7	842	-835
0	2	8	185	161
0	2	9	202	232
0	4	0	246	-230
0	4	1	454	448
0	4	3	1215	-1213
0	4	5	364	360
0	4	6	251	225
0	4	9	565	607
0	6	1	523	588
0	6	2	664	674
0	6	3	292	-308
0	6	4	808	-803
0	6	5	211	162
0	6	7	367	-358
0	6	8	164	-160
0	6	9	336	333
0	8	0	1083	-1090
0	8	2	468	482
0	8	3	210	231
0	8	4	146	-178
0	8	6	449	464
0	8	7	127	-140
0	8	8	193	-197
0	8	9	103	-102
0	10	0	323	-354
0	10	1	194	-212
0	10	5	256	-250
0	10	6	191	202
0	10	7	416	415
0	10	9	136	-111
0	12	0	264	291
0	12	1	215	-214
0	12	3	253	245
0	12	5	322	-267
0	12	6	154	-152
0	14	2	141	-171
0	14	4	216	213
0	16	0	125	133
1	1	-7	669	-678
1	1	-6	803	755
1	1	-5	283	308
1	1	-4	274	-299
1	1	-3	764	789
1	1	-2	1666	1764
1	1	-1	306	259
1	1	0	1252	-1168
1	1	3	504	-554
1	1	4	977	-929
1	1	5	482	-451
1	1	6	828	843
1	1	7	786	788
1	3	-7	411	-385
1	3	-6	279	-270
1	3	-5	702	733
1	3	-4	722	-652
1	3	-3	920	-504
1	3	-2	534	525
1	3	-1	323	318
1	3	0	1401	1496
1	3	1	253	-229
1	3	2	94	-116
1	3	3	741	737
1	3	4	1032	-1044
1	3	5	799	-767
1	5	-5	406	383
1	5	-4	548	569
1	5	-3	1154	-1179
1	5	-2	549	-573
1	5	2	536	-547
1	5	3	1053	1074
1	5	4	584	559
1	5	5	490	-537
1	5	7	252	266
1	7	-7	262	-272
1	7	-6	300	-330
1	7	-5	118	94
1	7	-4	634	613
1	7	-3	301	310
1	7	-2	915	-937
1	7	0	447	447
1	7	1	183	-160
1	7	2	549	-573
1	7	3	232	-237
1	7	4	579	623
1	7	5	223	-248
1	7	6	163	-171
1	7	7	549	535
1	9	-7	310	294
1	9	-6	540	-547
1	9	-4	202	212
1	9	-2	493	-523
1	9	-1	312	-273
1	9	0	734	753
1	9	1	280	239
1	9	3	156	-170
1	9	6	311	-307
1	9	7	176	-159
1	11	-7	374	320
1	11	-5	242	-255
1	11	-1	272	-324
1	11	0	220	-180
1	11	1	300	338
1	11	3	164	-194
1	11	5	364	334
1	11	7	369	-303
1	13	-3	273	297
1	13	0	189	-189
1	13	2	125	134
1	13	3	305	-303
1	13	5	178	159
1	15	2	208	161
1	17	0	138	-128
2	0	-8	772	835
2	0	-4	365	379
2	0	2	667	-660
2	0	4	647	-583
2	0	6	93	-60
2	0	8	342	305
2	2	-9	151	-112
2	2	-8	499	513
2	2	-7	718	735
2	2	-5	556	-548
2	2	-4	721	695
2	2	-3	511	-532
2	2	-2	177	-114
2	2	-1	896	-875
2	2	0	849	-873
2	2	2	861	-857
2	2	3	368	-357
2	2	4	517	519
2	2	5	738	763
2	2	6	346	351
2	2	7	798	-807
2	2	8	142	135
2	4	-9	542	-523
2	4	-8	153	181
2	4	-6	228	235
2	4	-5	558	-577
2	4	-3	1141	1179
2	4	-2	126	-85
2	4	-1	466	-483
2	4	0	381	-379
2	4	1	1138	1154
2	4	2	226	-259
2	4	3	1404	-1486
2	4	4	161	143
2	4	5	592	556
2	4	8	94	-95
2	4	9	311	299
2	6	-9	251	-239
2	6	-8	263	-249
2	6	-7	269	252
2	6	-4	802	-802
2	6	-3	119	-94
2	6	-2	463	458
2	6	-1	201	-209
2	6	0	388	405
2	6	1	660	658
2	6	2	462	486
2	6	3	319	-338
2	6	4	656	-678
2	6	5	226	195
2	6	7	240	-249
2	6	8	272	-307
2	6	9	89	105
2	8	-9	175	153
2	8	-8	403	-413
2	8	-7	162	110
2	8	-6	500	492
2	8	-4	401	-406
2	8	-3	590	-618
2	8	-2	482	508
2	8	-1	159	159
2	8	0	666	-677
2	8	2	455	462
2	8	3	383	388
2	8	5	208	-82
2	8	6	283	282
2	8	8	317	-340
2	8	9	206	-199
2	10	-7	299	-300
2	10	-6	217	222
2	10	-5	237	207
2	10	-4	230	-243
2	10	-2	162	178
2	10	-1	348	390
2	10	0	234	-272
2	10	1	456	-482
2	10	2	243	264
2	10	3	213	198
2	10	5	334	-344
2	10	6	172	152
2	10	7	450	452
2	10	9	110	-144
2	12	-6	173	-190
2	12	-5	357	252
2	12	-3	241	-273
2	12	-1	188	207
2	12	0	200	213
2	12	1	284	-283
2	12	3	305	303
2	12	5	279	-241
2	12	7	244	157
2	14	-4	212	211
2	14	4	220	171
2	16	-4	187	145
2	16	-2	106	-162
3	1	-7	434	-462
3	1	-6	1189	1245
3	1	-4	1210	-1213
3	1	-3	723	699
3	1	-2	855	893
3	1	-1	1027	1019
3	1	0	1486	-1555
3	1	1	1021	-1025
3	1	2	166	197
3	1	3	145	-116
3	1	4	349	322
3	1	5	428	-486
3	1	6	764	747
3	1	7	599	610
3	3	-7	274	-270
3	3	-6	320	-291
3	3	-5	579	587
3	3	-4	876	-921
3	3	-3	837	-827
3	3	-2	667	656
3	3	-1	1557	1561
3	3	0	435	407
3	3	1	1010	-1005
3	3	2	482	455
3	3	3	639	661
3	3	4	625	-606
3	3	5	734	-753
3	3	7	426	395
3	5	-5	380	393
3	5	-4	333	325
3	5	-3	1191	-1227
3	5	-2	93	61
3	5	-1	1093	1108
3	5	0	435	-377
3	5	1	405	-422
3	5	2	101	-108
3	5	3	707	704
3	5	4	349	339
3	5	7	101	70
3	7	-7	360	-341
3	7	-6	331	-376
3	7	-4	459	473
3	7	-3	276	246
3	7	-2	375	-359
3	7	-1	444	492
3	7	0	161	166
3	7	1	253	-271
3	7	2	264	-258
3	7	3	292	-316
3	7	4	256	270
3	7	5	234	-167
3	7	6	197	-195
3	7	7	314	301
3	9	-7	135	147
3	9	-6	524	-511
3	9	-4	193	209
3	9	0	592	593
3	9	1	346	335
3	9	3	260	-256
3	9	5	342	186
3	9	6	375	-365
3	9	7	225	-218
3	11	-7	215	232
3	11	-5	277	-251
3	11	-1	354	-340
3	11	1	400	420
3	11	3	155	-172
3	11	4	179	138
3	11	5	255	278
3	11	7	263	-244
3	13	-6	210	170
3	13	-3	254	258
3	13	-1	136	-143
3	13	3	159	-177
3	15	-2	105	144
3	15	4	173	-185
4	0	-8	618	700
4	0	-6	1598	-1488
4	0	-4	113	68
4	0	0	1285	1303
4	0	2	339	-373
4	0	4	471	429
4	0	6	1045	-1034
4	0	8	360	388
4	2	-8	296	261
4	2	-7	576	581
4	2	-6	311	-262
4	2	-5	167	-220
4	2	-4	360	351
4	2	-3	193	-167
4	2	-2	85	-131
4	2	-1	684	-675
4	2	0	189	-164
4	2	1	763	746
4	2	2	607	-591
4	2	3	273	-290
4	2	4	992	1019
4	2	5	407	432
4	2	6	419	-434
4	2	7	390	-410
4	2	8	119	163
4	4	-9	476	-449
4	4	-7	199	193
4	4	-6	291	262
4	4	-5	454	-443
4	4	-3	1387	1403
4	4	-2	337	322
4	4	-1	336	-365
4	4	0	501	-526
4	4	3	1075	-1134
4	4	4	246	221
4	4	5	403	388
4	4	7	205	165
4	4	9	355	345
4	6	-9	231	-208
4	6	-8	358	-366
4	6	-7	373	365
4	6	-5	139	-126
4	6	-4	612	-606
4	6	-3	357	365
4	6	-2	571	645
4	6	-1	328	-344
4	6	1	208	225
4	6	2	530	535
4	6	4	722	-756
4	6	5	246	287
4	6	6	160	156
4	6	7	128	-127
4	6	8	198	-175
4	6	9	221	168
4	8	-9	144	133
4	8	-8	463	-448
4	8	-7	162	129
4	8	-6	550	556
4	8	-4	206	-214
4	8	-3	336	-321
4	8	-2	359	370
4	8	0	544	-553
4	8	2	349	248
4	8	4	189	-216
4	8	5	263	64
4	8	7	119	-104
4	8	8	230	-247
4	8	9	89	-87
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4	10	5	220	-189
4	10	6	203	200
4	12	-9	156	127
4	12	-5	273	272
4	12	-3	335	-337
4	12	-1	241	261
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4	12	1	167	-194
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4	12	5	219	-147
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4	14	2	142	-163
4	14	4	229	196
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5	1	4	232	-222
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5	3	-1	329	351
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5	5	-3	986	-1012
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5	5	4	672	663
5	5	6	245	-277
5	7	-7	120	-99
5	7	-6	83	-102
5	7	-4	320	323
5	7	-3	200	212
5	7	-2	468	-478
5	7	-1	265	260
5	7	0	407	381
5	7	2	589	-587
5	7	4	673	579
5	7	6	275	-315
5	7	7	319	273
5	9	-7	279	261
5	9	-6	367	-382
5	9	-3	135	177
5	9	-2	138	-155
5	9	-1	206	-216
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5	9	1	121	132
5	9	2	194	-204
5	9	5	232	124
5	9	6	316	-288
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5	11	-5	356	-337
5	11	-3	161	192
5	11	-1	236	-268
5	11	1	316	345
5	11	3	157	-169
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5	13	3	230	-197
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6	4	-1	265	-281
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6	4	3	314	-324
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6	4	6	97	71
6	4	7	190	-172
6	4	9	214	190
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6	6	-2	388	368
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6	6	1	275	223
6	6	2	418	410
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6	6	7	290	-261
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6	8	-4	143	-84
6	8	-3	267	-284
6	8	-2	267	264
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6	8	2	154	192
6	8	3	160	184
6	8	6	261	271
6	8	8	128	-178
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6	10	-1	284	314
6	10	0	191	-168
6	10	2	110	119
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6	10	5	157	-128
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7	1	-4	144	-164
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7	1	4	191	-205
7	1	5	195	-208
7	1	6	188	188
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7	3	-6	293	-309
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7	3	5	439	-468
7	3	7	195	197
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7	11	-1	175	-193
7	11	1	197	202
7	11	2	141	-148
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8	2	-5	124	-158
8	2	-4	571	578
8	2	-2	456	-445
8	2	0	156	141
8	2	1	483	427
8	2	2	184	-149
8	2	5	189	166
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8	2	9	105	142
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8	4	-5	100	-98
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8	4	0	185	-174
8	4	2	155	140
8	4	3	471	-478
8	4	7	207	-163
8	6	-9	146	-154
8	6	-8	213	-182
8	6	-6	112	114
8	6	-4	577	-578
8	6	-3	221	256
8	6	-2	508	502
8	6	-1	161	-171
8	6	0	153	-185
8	6	1	111	86
8	6	2	370	390
8	6	3	131	-137
8	6	4	327	-310
8	8	-9	127	110
8	8	-8	230	-222
8	8	-6	346	352
8	8	-4	319	-264
8	8	-2	224	241
8	8	0	385	-374
8	8	2	193	219
8	8	6	244	158
8	10	-7	259	-156
8	10	-6	154	136
8	10	-5	272	124
8	10	-4	120	-155
8	10	-1	241	230
8	10	0	152	-159
8	10	1	276	-279
8	10	5	125	-150
8	12	-6	167	-139
8	12	3	258	165
8	14	-4	162	163
9	1	-7	255	-201
9	1	-6	716	735
9	1	-5	219	162
9	1	-4	310	-287
9	1	-3	145	115
9	1	0	470	-481
9	1	1	320	-329
9	1	2	243	232
9	1	4	310	-235
9	1	6	294	320
9	3	-7	126	-144
9	3	-5	258	265
9	3	-4	296	-289
9	3	-2	285	288
9	3	1	297	-319
9	3	2	123	144
9	3	3	249	272
9	3	4	352	-286
9	3	7	183	166
9	5	-5	189	170
9	5	-4	257	279
9	5	-3	235	-263
9	5	-2	217	-226
9	5	1	177	-172
9	5	2	108	-133
9	5	3	319	353
9	5	7	204	99
9	7	-7	168	-181
9	7	-6	239	-215
9	7	-4	213	209
9	7	-2	308	-287
9	7	-1	176	145
9	7	0	230	213
9	7	1	279	-274
9	7	2	84	-116
9	7	4	214	169
9	9	-8	105	176
9	9	-1	125	-164
9	9	0	201	214
9	11	-1	182	-164
9	11	1	208	203
10	0	0	409	368
10	0	2	166	-174
10	0	4	119	167
10	2	-8	166	153
10	2	-7	265	224
10	2	-5	267	-270
10	2	-4	204	199
10	2	-3	105	131
10	2	1	405	391
10	2	2	161	-161
10	4	-9	227	-210
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10	4	-2	104	-128
10	4	-1	279	-304
10	4	1	209	213
10	4	3	273	-316
10	6	-8	205	-184
10	6	-4	260	-230
10	6	-3	114	130
10	6	-2	286	275
10	6	-1	308	-314
10	6	1	219	213
10	6	4	265	-225
10	8	-9	120	133
10	8	-8	224	-247
10	8	-6	191	189
10	8	-2	151	156
10	8	0	149	-147
10	10	-3	104	-107
10	10	-1	206	180
10	10	1	167	-177
11	1	1	180	-195
11	3	-7	184	-176
11	3	-5	322	310
11	3	-4	219	-226
11	3	-3	292	-297
11	3	0	174	175
11	3	1	209	-201
11	5	-4	125	138
11	5	-3	271	-281
11	5	-2	154	-122
11	5	-1	129	136
11	7	-2	219	-161
11	7	2	259	-154
11	7	4	203	145
11	9	0	96	129
11	9	1	203	156
11	9	2	149	-153
11	11	-1	140	-141
11	11	1	174	169
12	0	0	423	413
12	0	2	177	-221
12	2	-8	144	118
12	2	-7	239	216
12	2	0	190	181
12	2	1	225	189
12	4	-7	186	106
12	4	-3	200	219
12	4	3	111	-197
12	6	-7	229	150
12	8	0	201	-180
13	1	0	272	-247
13	5	-3	183	-211
13	7	0	129	129
13	9	0	178	127
14	0	2	157	-173
14	8	0	140	-116
15	1	0	136	-126
15	3	1	93	-103

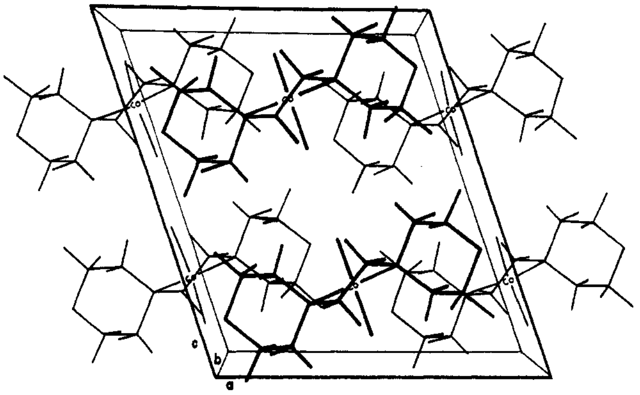

Figure 2.—The *b*-axis perspective view of unit cell contents.

TABLE III
BOND DISTANCES AND BOND ANGLES IN Co(EDM)Cl_2 WITH ESTIMATED STANDARD DEVIATIONS[a]

Atoms	Dist, Å	Atoms	Dist, Å
Co–Cl	2.229 (3)	C_1–H_1	1.00
Co–N	2.116 (8)	C_1–H_2	0.96
C_3–O	1.40 (2)	C_2–H_1	1.20
C_2–O	1.42 (1)	C_2–H_2	1.17
C_3–C_4	1.52 (2)	C_3–H_1	0.93
C_2–C_1	1.54 (1)	C_3–H_2	1.14
C_4–N	1.50 (1)	C_4–H_1	0.98
C_1–N	1.45 (2)	C_4–H_2	0.92
C_5–C_5'	1.50 (2)	C_5–H_1	0.97
C_5–N	1.50 (1)	C_5–H_2	1.03

Atoms	Angle, deg	Atoms	Angle, deg
N–Co–N′	88.9 (4)	C_1–N–Co	115.7 (6)
Cl–Co–Cl′	116.8 (2)	C_4–N–Co	112.8 (6)
Cl–Co–N	109.0 (3)	N–C_1–C_2	110.6 (8)
Cl′–Co–N	115.0 (3)	C_1–C_2–O	110.9 (9)
C_1–N–C_4	107.4 (8)	C_2–O–C_3	110.2 (7)
C_1–N–C_5	109.8 (8)	O–C_3–C_4	111.2 (7)
C_4–N–C_5	110.6 (9)	C_3–C_4– N	109.2 (9)
C_5–N–Co	100.4 (5)	C_5'–C_5–N	111.4 (8)

[a] Primed atoms indicate twofold related atoms.

III.[12] The Cl–Co–Cl′ angle has opened up to 116.8 (2)°. The Cl′–Co–N (115.0 (3)°) angle is also larger than the tetrahedral value.

It should also be noted that the N–Co–N′ angle of 88.9 (4)° is larger than N–M–N angles observed with unsubstituted ethylenediamine. Cu(en)$_2$(NO$_3$)$_2$ has a N–Cu–N angle of 86.2°; all others are smaller.[13] Some strain in the chelate ring system is suggested by the deviations from tetrahedral values of the angles about the nitrogen (especially C_5–N–Co (100.4 (5)°). The Co–Cl distance of 2.229 Å agrees well with the 2.252-Å length in Cs_3CoCl_5,[14] the 2.23-Å length in Cs_2CoCl_4,[15] and the 2.26-Å bond in dichlorodi-*p*-toluidinecobalt(II).[16] The Co–N bond length of 2.116 (8) Å is normal.[17,18]

TABLE IV
NONBONDED INTRAMOLECULAR DISTANCES[a]

Atoms	Dist, Å	Atoms	Dist, Å
C_4–Cl	3.53 (1)	N–N′	2.96 (2)
C_1–Cl′	3.76 (1)	Cl–Cl′	3.796 (7)
C_3–Cl	3.70 (1)	Co–O	4.141 (7)
C_2–Cl′	3.63 (1)		

[a] Primed atoms indicate twofold related atoms.

Table IV summarizes the important nonbonded intramolecular distances. The intramolecular distances of all four ring carbons and the closest chloride are less than the sum of the van der Waals radii of 3.8 Å.[19] The C_4–Cl and C_2–Cl′ distances, in particular, are quite short—3.53 and 3.63 Å. The C_2–Cl′ repulsion prevents the ring from twisting further to relieve the C_4–Cl interaction. The interaction of the carbons of the ring and the chlorines forces the opening of the N–Co–Cl′ angle and decreases the Co–N–C_5 angle. The N–N′ intraligand distance of 2.96 (2) Å is larger than that of 2.7 Å observed in unsubstituted ethylenediamines. These interactions, plus the preference of the ligand for the conformation in which the Co–N bonds are axial to the ring protons, cause this ligand to be very forcing toward tetrahedral geometry at the metal atom.

Both morpholine groups have the normal chair conformation and are joined to the ethylene bridge by equatorial C–N bonds. The ethylene bridge has a near-staggered conformation with the N′–C_5–′C_5–N dihedral angle of 62.4°. Deviations of bond angles of the chelating ligand from the tetrahedral values are not statistically significant.

Figure 2 shows the unit cell viewed approximately down the *b* axis. The minimum intermolecular packing distances (excluding hydrogen atoms) are 3.64 Å between C_2 and C_4 of the screw-axis-related molecules and 3.87 Å between Cl and C_5' of the glide-related molecules.

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