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The Crystal Structure of *trans*-Dichlorobis(ethylenediamine)cobalt(III) Hexathionate Monohydrate

OLAV FOSS and KJARTAN MARØY

Chemical Institute, University of Bergen, Bergen, Norway

The salt, $[Co(en)_2Cl_2]_2S_6O_6{\cdot}H_2O$, crystallizes in the space group $C_{2v}{}^8-Pba2$ with two formula units per unit cell, and $a = 12.12$ Å, $b = 19.13$ Å, $c = 6.43$ Å. The structure has been determined by X-ray methods, and refined by least squares for the hkl ($l = 0-3$) and $0kl$ reflections.

The hexathionate ion lies on a twofold axis of symmetry. Its sulphur chain has the extended *trans-trans* form of the helixes of fibrous sulphur. The derived dimensions of the chain are: $S_1-S_2 = 2.120 \pm 0.012$ Å, $S_2-S_3 = 2.025 \pm 0.015$ Å, $S_3-S_3' = 2.054 \pm 0.010$ Å; $\angle\ S_1-S_2-S_3 = 105.8°$, $\angle\ S_2-S_3-S_3' = 107.4°$, each $\pm$ 0.7°; $\angle\ S_1S_2S_3/S_2S_3S_3' = 85.2°$, $\angle\ S_2S_3S_3'/S_3S_3'S_2' = 73.2°$, each $\pm$ 1.5°; where primed atoms are related to the unprimed ones through the twofold axis.

In the cation, $Co-Cl = 2.224$ and 2.235 Å, each ± 0.012 Å, and $\angle\ Cl-Co-Cl = 178.9 \pm 0.2°$. The cobalt-ethylenediamine rings occur in *gauche* forms, and the cation is approximately centrosymmetric.

In the crystals of potassium barium hexathionate,[1] the sulphur chain of the hexathionate ion has the *cis-cis* form of the S_8 and S_6 rings of orthorhombic and rhombohedral sulphur. The extended *trans-trans* rational isomer, corresponding to the helixes of fibrous sulphur, occurs in the crystals of *trans*-dichlorobis(ethylenediamine)cobalt(III) hexathionate monohydrate, the structure of which is described here. Preliminary results have been reported earlier.[2,3]

CRYSTAL DATA

The salt, $[Co(en)_2Cl_2]_2S_6O_6{\cdot}H_2O$, forms long orthorhombic prisms {110}, with[2,4] $a = 12.12$ Å, $b = 19.13$ Å, $c = 6.43$ Å, and two formula units per unit cell. The space group is $C_{2v}{}^8-Pba2$, which requires that the hexathionate ions and the water molecules lie on twofold axes of symmetry.

The salt was first prepared by Weitz and Achterberg[5] in 1928 and later by Weitz and Spohn.[6] Neither of these authors mentioned any water of crystallization.

DETERMINATION OF THE STRUCTURE

The intensities of 149 observed *hk*0 reflections, out of 186 attainable with Fe$K\alpha$ radiation, were estimated visually from zero-layer Weissenberg photographs, taken of a prism {110} with edges 0.08 × 0.09 mm (μr approx. 0.7). The cobalt x and y coordinates were found from the *hk*0 Patterson map, and by use of cobalt as a searcher atom, the two chlorine atoms were located by means of the vector convergence method.[7] The cobalt and chlorine contributions gave the probable signs of one fourth (37) of the strongest *hk*0 reflections, and a Fourier synthesis bases on these indicated the positions of the three sulphur atoms of the asymmetric unit and thereby the signs of 40 additional reflections; none of the signs changed later. The second Fourier map allowed the location of the lighter atoms, and Fourier refinement and next difference refinement were then carried out, until the reliability index, R, was 0.10 for this zone. The Fourier map has been published.[2,3]

The approximate structure in the non-centrosymmetric a-axis projection was worked out on the basis of 54 observed 0*kl* reflections, visually estimated from zero-layer Weissenberg photographs, out of 57 attainable with Fe$K\alpha$ radiation. By use of the known y coordinates, and by placing cobalt at $z = 0$ and using it as a searcher atom, the approximate z coordinates of the chlorine and sulphur atoms were found from the 0*kl* vector convergence map. Fourier syntheses and difference syntheses brought the R index for this zone down to 0.18.

REFINEMENT OF THE STRUCTURE

The refinement was based on the *hk*0 and 0*kl* reflections, and in addition to these, on the *hk*1, *hk*2, and *hk*3 reflections. Altogether, 564 independent reflections were observed with measurable intensities, out of 700 accessible with Fe$K\alpha$ radiation. The intensities of the non-equatorial reflections were estimated, visually as the *hk*0 and 0*kl* data, from equi-inclination Weissenberg photographs, and were corrected for the effects of spot extension and contraction by the method of Phillips.[8] The *hk*0, *hk*1, *hk*2, and *hk*3 reflections were initially brought to the same scale by means of the 0*kl* data, and scale factors were later adjusted after each cycle of refinement.

Refinement was carried out by least squares on the IBM 1620 computer, using Mair's[9] program, and his weighting scheme No. 3 with $a = 80$ and $b = 50$. Eight strong *hk*0 reflections and two strong *hk*1 reflections, marked with an asterisk in Table 3, had markedly higher calculated than observed values and were given zero weight. The calculated structure factors were based on the Thomas-Fermi scattering curve for cobalt, those of Dawson[10] for chlorine and sulphur, and of Hoerni and Ibers[11] for oxygen, nitrogen and carbon. The hydrogen contributions were ignored.

Table 1. Atomic coordinates for *trans*-dichlorobis(ethylenediamine)cobalt(III) hexathionate monohydrate, in fractions of cell edges.
Origin on a twofold axis.

	x	y	z	B (Å^2)
S_1	0.2541	0.0919	0.4901	
S_2	0.1647	0.0046	0.3767	
S_3	0.0713	0.0290	0.6181	
O_1	0.1730	0.1373	0.5819	3.60
O_2	0.2999	0.1151	0.2964	5.95
O_3	0.3387	0.0645	0.6212	5.43
Co	0.0415	0.2762	−0.0193	2.28
Cl_1	0.1690	0.2963	0.2219	2.56
Cl_2	−0.0872	0.2567	−0.2614	2.48
N_1	0.0831	0.1779	−0.0144	1.73
C_1	−0.0156	0.1348	0.0780	2.80
C_2	−0.0492	0.1797	0.2731	3.60
N_2	−0.0681	0.2506	0.1963	2.46
N_3	0.0016	0.3764	−0.0193	1.93
C_3	0.0873	0.4161	−0.1355	5.42
C_4	0.1433	0.3715	−0.3054	8.45
N_4	0.1492	0.2981	−0.2307	2.95
H_2O	0.5000	0	0.2716	5.08

Table 2. Temperature parameters for the sulphur atoms, β_{ij}. The expression used is $\exp -(h^2\beta_{11} + k^2\beta_{22} + kl^2\beta_{33} + kl\beta_{23} + hl\beta_{13} + hk\beta_{12})$.

	β_{11}	β_{22}	β_{33}	β_{23}	β_{13}	β_{12}
S_1	0.0035	0.0017	0.0268	0.0014	0.0014	0.0002
S_2	0.0049	0.0021	0.0248	−0.0002	−0.0044	0.0002
S_3	0.0064	0.0026	0.0356	0.0075	−0.0058	−0.0019

Four cycles of refinement, starting with atomic coordinates from the projections and using individual, isotropic temperature parameters, reduced R from 0.16 to 0.09. In two subsequent cycles, anisotropic temperature parameters for the sulphur atoms were introduced. A final structure-factor calculation gave an overall R index of 0.085, with non-observed reflections included when $|F_c|$ exceeds the observable limit, but not the eight strong $hk0$ and two strong $hk1$ reflections referred to above. The individual R values for the $hk0$, $hk1$, $hk2$, $hk3$, and $0kl$ data lie within 0.084 to 0.086.

The final atomic coordinates are listed in Table 1, together with the isotropic temperature parameters, B in $\exp [- B(\sin^2 \theta/\lambda^2)]$. The anisotropic temperature parameters used for the sulphur atoms are given in Table 2, and observed and calculated structure amplitudes in Table 3.

Table 3. Observed and calculated structure amplitudes for *trans*-dichlorobis(ethylenediamine)cobalt(III) hexathionate monohydrate.

k	$\|F_o\|$	$\|F_c\|$
	$0k0$	
2	107*	112
4	103*	125
6	58	58
8	104	98
10	59	56
12	< 15	2
14	19	21
16	< 14	2
18	< 11	10
	$1k0$	
1	8	13
2	42	49
3	85*	103
4	102*	111
5	< 8	7
6	96	104
7	139	150
8	< 11	7
9	109	105
10	41	32
11	68	69
12	18	2
13	36	46
14	34	37
15	< 15	10
16	< 15	16
17	18	17
18	11	19
19	< 8	7
	$2k0$	
0	72*	83
1	57	65
2	90*	116
3	26	36
4	97*	120
5	85	86
6	< 10	8
7	38	37
8	53	51
9	77	81
10	70	72
11	59	63
12	20	17
13	58	62
14	19	15
15	41	49
16	< 14	4
17	12	15
18	13	17
19	14	20
	$3k0$	
1	< 8	5
2	95*	116
3	74	78
4	56	52
5	34	33
6	31	31
7	68	69
8	45	48
9	64	65
10	48	48
11	19	18
12	21	14
13	60	70
14	37	36
15	18	16
16	13	13
17	32	43
18	18	25
	$4k0$	
0	13	12
1	49	44
2	17	11
3	39	34
4	71	70
5	15	15
6	43	42
7	40	40
8	17	19
9	60	61
10	20	21
11	21	19
12	< 15	6
13	31	31
14	< 15	6
15	19	19
16	37	40
17	43	56
18	< 8	15
	$5k0$	
1	38	37
2	< 11	3
3	22	22
4	17	10
5	25	25
6	43	42
7	30	30
8	39	37
9	< 14	8
10	69	68
11	< 15	1
12	44	47
13	39	38
14	28	30
15	< 13	6
16	13	17
17	< 9	14
	$6k0$	
0	14	11
1	36	29
2	18	20
3	68	71
4	23	20
5	40	42
6	91	90
7	27	23
8	< 15	5
9	< 15	2
10	52	52
11	< 15	7
12	51	59
13	28	31
14	71	81
15	21	24
16	35	32
	$7k0$	
1	20	22
2	52	50
3	< 14	5
4	65	64
5	27	26
6	70	67
7	< 15	2
8	90	89
9	36	32
10	28	28
11	< 15	5
12	< 14	14
13	43	47
14	29	30
15	< 10	7
	$8k0$	
0	68	65
1	97	95
2	42	40
3	73	71
4	79	78
5	63	60
6	20	17
7	32	33
8	< 15	8
9	30	26
10	16	15
11	28	28

k	$\lvert F_o\rvert$	$\lvert F_c\rvert$
12	16	13
13	< 11	8
14	< 9	1
	9*k*0	
1	32	35
2	< 15	4
3	53	52
4	67	65
5	26	21
6	18	15
7	77	76
8	17	17
9	16	5
10	28	33
11	32	35
12	36	39
13	< 7	1
	10,*k*,0	
0	62	58
1	14	8
2	14	8
3	26	25
4	28	27
5	51	50
6	< 13	9
7	22	20
8	15	13
9	39	40
10	38	40
	11,*k*,0	
1	15	12
2	15	14
3	< 12	4
4	18	15
5	13	15
6	23	22
7	9	9
8	17	17
	12,*k*,0	
0	48	59
1	< 9	3
2	22	24
3	< 8	9
4	< 7	1
	0*k*1	
0	19	14
2	145*	228
4	20	34
6	88	87
8	20	12
10	82	86
12	50	48
14	36	38
16	21	16
18	35	41
	1*k*1	
1	64	71
2	82	93
3	47	42
4	55	59
5	68	74
6	45	43
7	46	44
8	71	69
9	85	89
10	29	16
11	56	57
12	25	25
13	38	44
14	28	29
15	21	22
16	18	15
17	24	31
18	< 15	11
	2*k*1	
0	141*	214
1	66	75
2	51	56
3	52	52
4	54	51
5	98	107
6	69	71
7	41	32
8	53	48
9	29	28
10	38	37
11	< 21	12
12	< 21	12
13	< 22	14
14	38	33
15	< 21	9
16	25	22
17	23	26
18	20	23
	3*k*1	
1	33	35
2	47	47
3	98	110
4	63	65
5	19	18
6	40	34
7	86	88
8	47	46
9	21	20
10	44	43
11	50	52
12	67	67
13	35	31
14	< 22	20
15	38	37
16	19	22
17	20	22
18	18	20
	4*k*1	
0	70	74
1	77	80
2	28	23
3	49	44
4	43	44
5	41	34
6	34	36
7	36	34
8	50	50
9	71	71
10	40	39
11	21	24
12	38	39
13	22	24
14	33	33
15	30	32
16	19	16
17	17	18
	5*k*1	
1	< 16	4
2	29	30
3	60	57
4	25	22
5	30	29
6	36	32
7	25	27
8	95	98
9	33	31
10	26	26
11	< 22	7
12	34	35
13	< 21	11
14	30	40
15	< 18	8
16	19	24
17	18	20
	6*k*1	
0	86	85
1	63	63
2	< 18	4
3	51	43
4	74	79
5	45	43
6	45	43
7	30	27
8	30	30
9	< 22	7
10	27	24

k	$\|F_o\|$	$\|F_c\|$
11	30	24
12	26	23
13	$<$ 20	10
14	25	20
15	28	29
16	28	33
	7k1	
1	28	31
2	32	28
3	26	27
4	61	59
5	$<$ 21	8
6	50	47
7	34	31
8	$<$ 22	9
9	$<$ 22	6
10	61	58
11	32	28
12	42	44
13	$<$ 18	12
14	22	25
15	13	15
	8k1	
0	21	18
1	37	30
2	44	44
3	48	55
4	$<$ 22	7
5	35	34
6	42	46
7	30	27
8	27	22
9	35	35
10	33	30
11	25	22
12	19	21
13	29	31
14	$<$ 12	8
	9k1	
1	32	31
2	38	39
3	30	23
4	23	22
5	43	42
6	28	29
7	24	20
8	36	34
9	38	38
10	18	17
11	19	19
12	$<$ 13	13
	10,k,1	
0	27	19
1	36	33

k	$\|F_o\|$	$\|F_c\|$
2	54	52
3	24	23
4	45	44
5	21	23
6	$<$ 18	12
7	28	27
8	$<$ 16	8
9	$<$ 14	5
10	16	18
	11,k,1	
1	$<$ 17	10
2	17	11
3	22	24
4	21	22
5	27	32
6	14	13
7	24	29
8	24	24
	12,k,1	
0	37	48
1	14	13
2	$<$ 11	7
	0k2	
0	30	27
2	50	50
4	74	72
6	33	28
8	17	17
10	68	63
12	41	43
14	40	37
16	60	61
18	38	55
	1k2	
1	40	38
2	73	72
3	$<$ 10	11
4	32	21
5	12	10
6	21	20
7	36	34
8	31	27
9	41	37
10	42	40
11	51	51
12	57	51
13	36	29
14	$<$ 20	13
15	31	30
16	28	35
17	26	22
18	21	28

k	$\|F_o\|$	$\|F_c\|$
	2k2	
0	51	49
1	58	63
2	81	90
3	60	55
4	30	31
5	88	86
6	28	28
7	62	61
8	36	33
9	63	61
10	$<$ 19	10
11	55	50
12	77	78
13	40	46
14	39	39
15	$<$ 18	19
16	$<$ 16	13
17	$<$ 14	5
18	23	35
	3k2	
1	73	80
2	19	25
3	106	110
4	21	22
5	83	79
6	34	34
7	62	61
8	89	90
9	68	70
10	52	49
11	47	40
12	42	43
13	26	21
14	51	48
15	23	21
16	$<$ 15	15
17	$<$ 13	11
	4k2	
0	106	119
1	140	156
2	71	70
3	122	130
4	22	12
5	77	73
6	73	77
7	22	17
8	27	24
9	34	33
10	27	23
11	22	19
12	20	17
13	$<$ 19	1
14	$<$ 18	12
15	$<$ 16	9
16	18	20
17	$<$ 11	4

k	$\lvert F_o \rvert$	$\lvert F_c \rvert$
	5k2	
1	20	22
2	26	21
3	50	46
4	89	84
5	29	27
6	70	72
7	23	18
8	55	52
9	< 20	5
10	76	75
11	22	23
12	53	51
13	< 18	16
14	< 17	10
15	28	29
16	< 12	3
	6k2	
0	34	32
1	72	68
2	47	44
3	48	47
4	69	70
5	33	29
6	41	37
7	< 20	15
8	78	77
9	< 20	16
10	71	72
11	< 19	15
12	21	23
13	20	18
14	18	19
15	12	18
	7k2	
1	39	41
2	20	24
3	38	36
4	20	17
5	36	33
6	< 20	2
7	< 20	16
8	47	42
9	< 19	17
10	< 19	16
11	< 18	8
12	21	23
13	< 14	4
14	33	40
	8k2	
0	42	37
1	20	15
2	< 20	7
3	< 20	12
4	27	25
5	< 20	6
6	26	23
7	< 19	7
8	< 19	7
9	< 18	1
10	17	19
11	< 15	6
12	16	16
13	11	14
	9k2	
1	< 19	17
2	< 19	11
3	< 19	14
4	33	33
5	18	15
6	28	29
7	< 17	9
8	< 16	8
9	< 15	13
10	22	19
11	20	26
	10,k,2	
0	45	39
1	< 17	6
2	17	15
3	< 17	16
4	23	28
5	21	22
6	43	46
7	16	20
8	19	24
9	18	24
	11,k,2	
1	15	15
2	31	33
3	21	25
4	< 12	10
5	30	37
6	13	21
	0k3	
0	106	116
2	18	12
4	68	64
6	42	39
8	70	70
10	19	18
12	56	58
14	18	18
16	24	30
	1k3	
1	9	18
2	10	8
3	38	36
4	30	28
5	< 14	9
6	51	51
7	43	41
8	28	25
9	61	59
10	< 19	13
11	60	61
12	< 20	11
13	38	34
14	33	32
15	29	32
16	< 14	9
17	10	18
	2k3	
0	86	101
1	49	51
2	58	57
3	67	66
4	42	38
5	< 15	5
6	34	35
7	44	42
8	< 18	2
9	< 19	12
10	32	30
11	22	19
12	20	18
13	< 19	10
14	< 18	7
15	< 16	5
16	31	40
	3k3	
1	30	29
2	21	21
3	36	35
4	33	29
5	71	69
6	47	48
7	29	28
8	< 19	5
9	59	58
10	66	68
11	41	38
12	29	28
13	24	24
14	18	19
15	15	16
16	< 12	17
	4k3	
0	22	20
1	65	71
2	48	50
3	62	61
4	43	46

k	$\lvert F\rvert$	$\lvert F_c\rvert$
5	27	31
6	$<$ 18	2
7	49	51
8	36	33
9	23	22
10	$<$ 20	9
11	32	29
12	37	42
13	34	34
14	16	16
15	$<$ 13	6
	$5k3$	
1	37	40
2	50	53
3	$<$ 18	16
4	19	13
5	37	34
6	56	56
7	$<$ 20	8
8	53	55
9	26	25
10	40	39
11	19	16
12	$<$ 18	17
13	$<$ 16	5
14	30	38
15	$<$ 11	11
	$6k3$	
0	28	35
1	43	45
2	69	67
3	44	47
4	23	23
5	30	34
6	46	48
7	24	24
8	27	29
9	$<$ 19	6

k	$\lvert F_o\rvert$	$\lvert F_c\rvert$
10	24	17
11	$<$ 17	6
12	25	26
13	22	24
14	$<$ 11	10
	$7k3$	
1	35	35
2	20	11
3	20	17
4	45	46
5	30	29
6	44	43
7	$<$ 19	16
8	19	16
9	$<$ 18	18
10	31	31
11	$<$ 15	8
12	30	36
	$8k3$	
0	54	56
1	27	18
2	22	16
3	28	22
4	$<$ 19	13
5	33	28
6	18	9
7	24	24
8	$<$ 17	13
9	26	24
10	14	18
11	23	26
	$9k3$	
1	18	17
2	33	32
3	17	15
4	$<$ 17	10

k	$\lvert F_o\rvert$	$\lvert F_c\rvert$
5	30	30
6	16	13
7	34	36
8	32	39
9	15	20
	$10,k,3$	
0	20	14
1	33	27
2	33	35
3	18	20
4	31	38
5	17	17
6	15	19
	$0k4$	
0	110	103
2	139	152
4	55	53
6	82	76
8	$<$ 15	5
10	15	18
12	13	9
14	9	10
	$0k5$	
0	74	73
2	56	51
4	68	69
6	29	26
8	40	39
10	24	21
12	28	33
	$0k6$	
0	10	10
2	$<$ 11	2
4	13	14
6	10	15

THE HEXATHIONATE ION

Bond lengths and angles in the hexathionate ion, from the atomic coordinates of Table 1, are listed in Table 4 together with the standard deviations produced by the least-squares treatment. The ion lies across a twofold axis of symmetry, and a prime is used to denote the equivalent of an atom on the other side of the twofold axis. S_1 and S_1' are the terminal, sulphonate sulphur atoms, and S_3 and S_3' the middle ones, closest to the twofold axis. Two views of the ion are shown in Fig. 1.

The sulphur-sulphur bonds have within the errors the same lengths in the present *trans-trans* form of the hexathionate ion as in the *cis-cis* form occurring in the potassium barium salt:[1] 2.120 ± 0.012 Å against 2.10 ± 0.02 Å for the sulphonate sulphur-divalent sulphur bonds, and 2.025, 2.054,

Table 4. Dimensions of the hexathionate ion.

Bond lengths and angles.

$S_1-S_2 = 2.120 \pm 0.012$ Å
$S_2-S_3 = 2.025 \pm 0.015$
$S_3-S_3' = 2.054 \pm 0.010$

$S_1-O_1 = 1.44 \pm 0.02$ Å
$S_1-O_2 = 1.43 \pm 0.04$
$S_1-O_3 = 1.43 \pm 0.03$

$\angle S_1-S_2-S_3 = 105.8 \pm 0.7°$

$\angle S_2-S_3-S_3' = 107.4 \pm 0.7°$

$\angle S_2-S_1-O_1 = 105.6 \pm 1.0°$
$\angle S_2-S_1-O_2 = 98.2 \pm 1.3$
$\angle S_2-S_1-O_3 = 106.3 \pm 1.1$

$\angle O_1-S_1-O_2 = 115.8 \pm 1.6°$
$\angle O_1-S_1-O_3 = 118.1 \pm 1.8$
$\angle O_2-S_1-O_3 = 110.4 \pm 1.6$

Dihedral angles.

$S_1S_2S_3/S_2S_3S_3' = 85.2 \pm 1.5°$

$S_2S_3S_3'/S_3S_3'S_2' = 73.2 \pm 1.5°$

2.025 ± 0.010—0.015 Å against 2.05, 2.04, 2.04 ± 0.02 Å for the middle bonds between divalent sulphur atoms.

The S—S—S bond angles in the potassium barium salt are 101°, 113°, 109°, 100°, and the SSS/SSS dihedral angles, 108°, 89°, 105°, from one end of the sulphur chain to the other. The bond angles differ more in the *cis-cis* form than in the *trans-trans* form, although the average is nearly the same in the two forms, 105.8° against 106.6°. The dihedral angles are larger, by 23°, 16°, and 20°, in the potassium barium salt than in the present salt, the involved non-bonded distances, S_1-S_4, S_2-S_5, and S_3-S_6 being 4.57, 4.42, and 4.40 Å in the former and 4.21, 4.00, and 4.21 Å in the latter. The *cis-cis* form leads to a closer approach between the terminal sulphur atoms, 4.85 Å as compared with 7.09 Å in the extended *trans-trans* form. The reason for the larger dihedral angles in the former may be the space requirements of the sulphonate groups.

In cesium hexasulphide,[12] as well as in barium tetrasulphide monohydrate,[13] there appears to be a variation in the lengths of the sulphur bonds along the chain: 1.99, 2.10, 2.03, 2.12, 2.03 ± 0.03 Å in the hexasulphide and 2.02, 2.07, 2.02 ± 0.025 Å in the tetrasulphide. In the hexathionate ion, each of the terminal sulphur atoms has three oxygen atoms attached to it, and the terminal S—S bonds are longer than the middle ones, but one might from analogy with the polysulphide ions perhaps expect a variation in the lengths of the three middle divalent-divalent S—S bonds. More accurate data are required to decide this point.

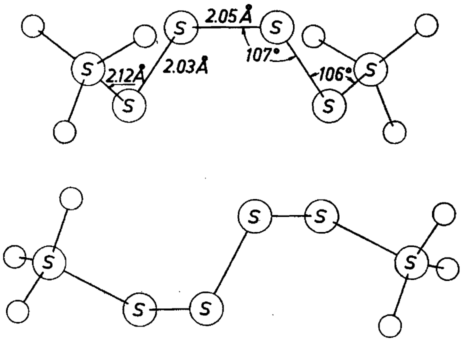

Fig. 1. The *trans-trans* form of the hexathionate ion, as seen along the twofold axis (below), and normal to the twofold axis and to a line through S_2 and S_3' (above).

THE CATION

For the *trans*-diclorobis(ethylenediamine)cobalt(III) cation the resulting Co—Cl bond lengths are 2.224 Å (Cl_1) and 2.235 Å (Cl_2), each ± 0.012 Å, and the Cl—Co—Cl angle, 178.9 ± 0.2°. The cobalt-nitrogen coordination group is planar within the error, the maximum deviation of atoms from the least-squares plane of the group being 0.015 Å for N_1 and N_3. The Co—Cl_1 and Co—Cl_2 bonds make angles of 89° and 88°, respectively, with this plane. Lengths of 2.22 Å and 2.33 Å, respectively, have been reported for the Co—Cl bond in *trans*-dichlorobis(ethylenediamine)cobalt(III) chloride[14] and chloride hydrochloride dihydrate;[15] in both these salts the cation is centrosymmetric.

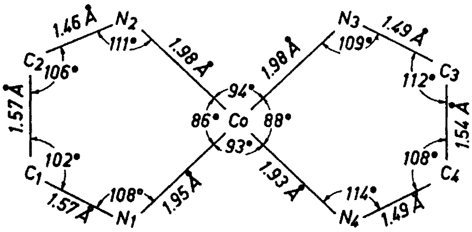

Fig. 2. Dimensions of the cobalt-ethylenediamine parts of the cation.

Bond lengths and angles in the cobalt-ethylenediamine rings are shown in Fig. 2. The standard deviations are 0.02—0.03 Å, 0.04—0.05 Å, and 0.05—0.06 Å, respectively, for Co—N, N—C, and C—C bond lengths, and about 1°, 2°, and 3°, respectively, for N—Co—N, Co—N—C, and N—C—C bond angles. The abnormal C—N length and N—C—C angle associated with C_1 are probably due to maximum errors in the coordinates of this atom.

The distances of the carbon atoms from the cobalt-nitrogen least-squares plane are: C_1, −0.52 Å; C_2, 0.25 Å; C_3, 0.33 Å; C_4, −0.15 Å, ± 0.04—0.05 Å. The metal-ethylenediamine rings thus have the usual *gauche* form, and the cation approaches centrosymmetry.

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