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

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The crystal structure of *trans*-dichlorobis(ethylenediamine)-cobalt(III) hexathionate monohydrate, $[Co(en)_2Cl_2]_2S_6O_6.H_2O$, has been refined by a full-matrix least squares program using 1994 independent non-zero reflections. The data were collected by means of a single-crystal diffractometer using Mo$K\alpha$ radiation (Nb-filtered). The final conventional R value was 0.037.

The space group is $Pba2$ (No. 32) with two formula units per unit cell, and $a = 12.084(5)$ Å, $b = 19.160(8)$ Å, $c = 6.421(3)$ Å.

The six-membered sulphur chain of the hexathionate ion has the *trans-trans* rotational isomeric form. A twofold symmetry axis passes between the two middle sulphur atoms normal to the bond between these atoms. The dimensions of the chain are: S(1)–S(2) = 2.132(2) Å, S(2)–S(3) = 2.018(3) Å, S(3)–S(3′) = 2.069(3) Å, ∠S(1)–S(2)–S(3) = 105.08(11)°, S(2)–S(3)–S(3′) = 107.35(11)°. The three middle bonds, between divalent sulphur atoms, thus have alternating lengths, the central one being 0.05 Å longer than the two others. The SSS/SSS dihedral angle associated with the shorter bond is 85.7°, compared to 71.4° for the longer bond.

The cation is octahedral with Co–Cl = 2.299(2) and 2.261(2) Å, Co–N = 1.958(4) – 1.974(4) Å, ∠Cl–Co–Cl = 179.4(1)°, ∠Cl–Co–N = 88.8(2)° – 91.2(2)°, ∠N–Co–N = 85.9(2)° and 87.2(2)° for nitrogens from the same ethylenediamine group, and ∠N–Co–N = 92.4(2)° and 94.5(2)° for nitrogens from different ethylenediamine groups.

The hexathionate ion has a six-membered sulphur chain, where the two terminal sulphur atoms are each bonded to three oxygen atoms and form sulphonate groups. The remaining four sulphur atoms are divalent. In the crystal structure of $[Co(en)_2Cl_2]_2S_6O_6.H_2O$, determined on the basis of X-ray film data,[1] there was a small difference in the lengths of two independent divalent sulphur-divalent sulphur bonds. Mainly to decide whether this difference is significant, a refinement based on diffractometer data has been undertaken.

EXPERIMENTAL

The intensity data, and angles for unit cell dimensions, were measured on a Siemens automatic single-crystal diffractometer using Mo$K\alpha$ radiation (Nb-filtered) and a scintillation counter.

The crystal of $[Co(en)_2Cl_2]_2S_6O_6.H_2O$ used for the measurements was of a sample [2] that had been kept for thirteen years in a refrigerator, without showing any signs of decomposition. It was a well developed prism extended along the c axis. The distances between the (110) and between the ($\bar{1}\bar{1}0$) boundary faces were 0.173 and 0.193 mm, respectively, and the length of the crystal was reduced to 0.431 mm by cutting.

The crystal was mounted with the c axis approximately parallel to the ϕ axis of the diffractometer, and setting angles for all reflections were calculated from the θ, χ and ϕ angles of seven non-coplanar reciprocal vectors.

The five-value procedure and $\theta - 2\theta$ scan technique were used. The scan width was 0.70° for all reflections and maximum scan time per degree was 24 sec.

Two reflections of medium strength, 800 and 0 10 0, were measured two times each at intervals of 50 reflections. The net intensities were brought to a common scale by means of these reflections; the scale factors varied from 1.00 to 1.05.

Out of 2149 independent reflections attainable within $\theta = 29°$, only 155 were found to have net intensities below three times its standard deviation. These reflections were assigned an intensity equal to this limit and labelled as unobserved.

The linear absorption coefficient for Mo$K\alpha$ radiation is 19.6 cm^{-1}, and absorption corrections were carried out in addition to Lorentz and polarization corrections. The number of Gaussian grid points used for the absorption corrections were 8, 8, and 12 along the a, b, and c axes, respectively. The absorption factors by which the intensities were multiplied varied from 1.32 to 1.54.

The scattering factor curves used were those listed in *International Tables for X-Ray Crystallography*.[3] The curves for cobalt, chlorine, and sulphur were corrected for anomalous dispersion using the values given by Cromer,[4] and taking the amplitude as the corrected value.

The refinement was carried out with a full-matrix least squares program minimizing the function

$$r = \sum W(|F_o| - K|F_c|)^2$$

The intensity data were eventually corrected for secondary extinction with a program written by K. Åse of this Institute.

For further details concerning the data collection and the programs used, see Ref. 5.

The unit cell dimensions, calculated by means of a least squares program using the θ angles ($\theta = 22 - 28°$) of 11 reflections measured on the diffractometer, are $a = 12.084(5)$ Å, $b = 19.160(8)$ Å, $c = 6.421(3)$ Å.

The space group is $Pba2$ (No. 32) with two $[Co(en)_2Cl_2]_2S_6O_6.H_2O$ formula units per unit cell.[2]

REFINEMENT

Structure factor calculations were carried out using the positional and thermal parameters arrived at by the refinement of the film data,[1] except that isotropic thermal parameters were used for all atoms. On refinement of these parameters, the R value ($\sum||F_o| - |F_c||/\sum|F_o|$) converged at 0.093. Since only relative z coordinates are required for this space group, the z coordinate for cobalt was put equal to zero and was not included in the refinement.

Anisotropic thermal parameters were then introduced for the cobalt, chlorine, and sulphur atoms, whereby the reliability index was reduced to 0.053. The thermal parameters for two of the sulphonate oxygen atoms, the water oxygen atom, and the two carbon atoms of one ethylenediamine group were high compared to those of the other atoms. This is in accordance with the result of the refinement based on the film data.[1] When anisotropic thermal

parameters were used also for the lighter atoms, the R value was reduced to 0.040.

Secondary extinction corrections were then carried out according to Zachariasen.[6] The absorption term in the expression for F_{corr} was put equal to

Table 1. Atomic coordinates for *trans*-dichlorobis(ethylenediamine)cobalt(III) hexathionate monohydrate. Origin on a twofold axis. Standard deviations are given in parentheses.

	x	y	z
S(1)	0.25429(10)	0.09166(6)	0.50648(38)
S(2)	0.16421(13)	0.00393(8)	0.39242(34)
S(3)	0.07232(14)	−0.02888(9)	0.63589(38)
O(1)	0.33544(46)	0.06433(28)	0.64130(139)
O(2)	0.30101(51)	0.11618(28)	0.31087(105)
O(3)	0.17218(34)	0.13776(22)	0.59417(81)
Co	0.04158(5)	0.27620(3)	0
Cl(1)	0.16913(11)	0.29738(9)	0.24271(30)
Cl(2)	−0.08830(11)	0.25590(9)	−0.24602(29)
N(1)	0.08213(34)	0.17649(21)	−0.00163(97)
N(2)	−0.06717(35)	0.25312(27)	0.21536(72)
N(3)	0.00305(36)	0.37630(21)	−0.00721(99)
N(4)	0.14760(38)	0.29820(29)	−0.22361(87)
C(1)	−0.01291(54)	0.13735(28)	0.09967(109)
C(2)	−0.04811(47)	0.17955(30)	0.28668(96)
C(3)	0.08721(67)	0.41443(41)	−0.12442(192)
C(4)	0.14053(90)	0.37272(42)	−0.27471(175)
H_2O	½	0	0.31219(133)

Table 2. Thermal parameters expressed in the form $\exp[-2\pi^2(h^2a^{-2}U_{11}+\cdots 2hka^{-1}b^{-1}U_{12}+\cdots)]$. All values have been multiplied by 10^4. Standard deviations are given in parentheses.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
S(1)	288(5)	298(5)	653(9)	6(4)	11(9)	27(9)
S(2)	418(8)	378(7)	634(11)	−12(6)	−73(8)	−26(8)
S(3)	507(8)	413(8)	753(13)	−121(7)	174(9)	−70(10)
O(1)	705(35)	510(28)	1719(72)	11(26)	71(40)	−781(45)
O(2)	1047(47)	562(31)	1006(53)	−181(32)	−139(32)	627(44)
O(3)	392(22)	490(23)	555(28)	40(18)	−140(22)	72(22)
Co	198(2)	292(3)	236(3)	26(2)	−9(3)	−4(3)
Cl(1)	257(5)	550(8)	336(7)	−5(6)	−28(7)	−67(6)
Cl(2)	305(6)	481(8)	284(6)	−22(6)	13(7)	−68(6)
N(1)	395(21)	315(19)	384(23)	92(17)	−51(27)	9(30)
N(2)	232(18)	343(21)	266(26)	23(17)	24(20)	27(18)
N(3)	366(20)	298(19)	434(24)	19(16)	7(27)	25(32)
N(4)	249(20)	503(28)	326(29)	3(21)	9(24)	64(21)
C(1)	488(33)	306(26)	437(33)	25(24)	18(26)	−19(30)
C(2)	412(28)	359(27)	367(33)	9(23)	70(24)	6(26)
C(3)	682(49)	490(40)	1415(91)	68(37)	355(53)	493(62)
C(4)	1261(83)	498(42)	1069(80)	−147(47)	70(50)	763(72)
H_2O	752(47)	819(54)	607(52)	294(43)	0	0

Table 3. Observed and calculated structure amplitudes (× 10) for *trans*-dichlorobis-(ethylenediamine)cobalt(III) hexathionate monohydrate. A minus sign on $F(O)$ indicates an unobserved reflection.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
0	4	0	1137	1244	4	9	0	608	587	8	8	0	75	78	13	4	0	140	136	2	16	1	261	263
0	6	0	565	567	4	10	0	158	176	8	9	0	271	267	13	5	0	286	279	2	17	1	262	255
0	8	0	1055	1036	4	11	0	164	147	8	10	0	143	133	13	6	0	-41	22	2	18	1	237	238
0	10	0	546	537	4	12	0	56	44	8	11	0	262	290	13	7	0	52	44	2	19	1	130	143
0	12	0	-37	27	4	13	0	336	356	8	12	0	153	137	13	8	0	82	75	2	20	1	121	128
0	14	0	212	225	4	14	0	89	67	8	13	0	95	88	13	9	0	199	193	2	21	1	232	234
0	16	0	92	89	4	15	0	236	237	8	14	0	-36	19	13	10	0	-52	29	2	22	1	363	357
0	18	0	54	34	4	16	0	417	421	8	15	0	155	155	13	11	0	230	213	2	23	1	58	64
0	20	0	93	93	4	17	0	554	556	8	16	0	-55	7	13	12	0	-39	4	2	24	1	47	40
0	22	0	256	247	4	18	0	114	121	8	17	0	79	70	13	13	0	168	169	2	25	1	87	91
0	24	0	93	77	4	19	0	447	453	8	18	0	83	82	13	14	0	233	221	3	1	1	344	337
0	26	0	52	44	4	20	0	231	154	8	19	0	-40	15	13	15	0	123	134	3	2	1	477	448
1	2	0	474	482	4	21	0	267	265	8	20	0	64	103	13	16	0	-45	46	3	3	1	1101	1110
1	3	0	1014	1059	4	22	0	256	241	8	21	0	-40	15	14	0	0	292	295	3	4	1	671	678
1	4	0	1127	1113	4	23	0	216	212	8	22	0	88	86	14	1	0	118	106	3	5	1	164	147
1	5	0	-40	59	4	24	0	92	91	9	1	0	256	306	14	2	0	225	214	3	6	1	417	392
1	6	0	1022	1048	4	25	0	54	54	9	2	0	-34	41	14	3	0	84	59	3	7	1	939	954
1	7	0	1550	1569	5	1	0	411	380	9	3	0	536	540	14	4	0	70	69	3	8	1	495	493
1	8	0	35	8	5	2	0	-27	6	9	4	0	965	963	14	5	0	196	213	3	9	1	291	268
1	9	0	1205	1123	5	3	0	405	162	9	5	0	275	281	14	6	0	173	177	3	10	1	466	462
1	10	0	305	355	5	4	0	79	53	9	6	0	203	206	14	7	0	238	235	3	11	1	518	543
1	11	0	748	736	5	5	0	243	264	9	7	0	867	917	14	8	0	49	39	3	12	1	674	687
1	12	0	-30	7	5	6	0	471	455	9	8	0	213	212	14	9	0	137	134	3	13	1	326	314
1	13	0	426	413	5	7	0	305	317	9	9	0	115	96	14	10	0	193	194	3	14	1	45	49
1	14	0	320	315	5	8	0	288	266	9	10	0	341	344	14	11	0	127	139	3	15	1	381	374
1	15	0	127	109	5	9	0	74	79	9	11	0	373	376	14	12	0	-40	9	3	16	1	204	195
1	16	0	108	109	5	10	0	744	758	9	12	0	430	424	14	13	0	-40	14	3	17	1	206	196
1	17	0	210	203	5	11	0	79	66	9	13	0	-36	0	15	1	0	48	51	3	18	1	186	185
1	18	0	113	109	5	12	0	405	407	9	14	0	-36	21	15	2	0	-41	43	3	19	1	135	103
1	19	0	50	57	5	13	0	349	351	9	15	0	-42	16	15	3	0	167	165	3	20	1	185	190
1	20	0	58	56	5	14	0	313	323	9	16	0	217	211	15	4	0	236	227	3	21	1	191	191
1	21	0	-40	41	5	15	0	54	114	9	17	0	115	125	15	5	0	150	159	3	22	1	78	85
1	22	0	147	147	5	16	0	111	155	9	18	0	253	243	15	6	0	187	184	3	23	1	-38	23
1	23	0	-40	9	5	17	0	131	52	9	19	0	68	69	15	7	0	319	321	3	24	1	-35	24
1	24	0	136	127	5	18	0	265	300	9	20	0	74	63	15	8	0	102	83	3	25	1	105	102
1	25	0	-41	15	5	19	0	-35	7	9	21	0	71	70	15	9	0	118	113	4	0	1	801	781
1	26	0	135	97	5	20	0	147	152	10	0	0	670	670	15	10	0	140	129	4	1	1	865	877
2	0	0	1081	895	-5	21	0	207	195	10	1	0	120	123	16	0	0	101	122	4	2	1	319	316
2	1	0	795	675	5	22	0	110	111	10	2	0	171	163	16	1	0	280	279	4	3	1	514	515
2	2	0	1214	1205	5	23	0	-41	20	10	3	0	265	263	16	2	0	196	197	4	4	1	416	447
2	3	0	327	337	5	24	0	90	55	10	4	0	310	288	16	3	0	253	257	4	5	1	399	389
2	4	0	1152	1228	6	0	0	123	111	10	5	0	543	537	16	4	0	238	226	4	6	1	308	339
2	5	0	679	689	6	1	0	432	406	10	6	0	132	95	16	5	0	191	182	4	7	1	340	309
2	6	0	78	91	6	2	0	167	172	10	7	0	415	407	16	6	0	-43	6	4	8	1	517	506
2	7	0	347	316	6	3	0	721	721	10	8	0	135	116	0	0	1	146	139	4	9	1	742	732
2	8	0	935	914	6	4	0	159	159	10	9	0	426	419	0	4	1	266	322	4	10	1	376	396
2	9	0	802	772	6	5	0	427	470	10	10	0	431	415	0	6	1	548	518	4	11	1	238	232
2	10	0	691	701	6	6	0	883	904	10	11	0	140	141	0	8	1	123	117	4	12	1	354	401
2	11	0	605	596	6	7	0	281	217	10	12	0	-38	19	0	10	1	838	819	4	13	1	189	203
2	12	0	201	212	6	8	0	-35	22	10	13	0	125	131	0	12	1	487	486	4	14	1	337	334
2	13	0	555	553	6	9	0	-35	16	10	14	0	251	269	0	14	1	359	358	4	15	1	346	346
2	14	0	127	107	6	10	0	492	471	10	15	0	44	56	0	16	1	201	212	4	16	1	134	149
2	15	0	443	425	6	11	0	45	54	10	16	0	258	293	0	18	1	437	435	4	17	1	220	213
2	16	0	148	137	6	12	0	486	516	10	17	0	84	78	0	20	1	421	431	4	18	1	268	274
2	17	0	156	142	6	13	0	316	318	10	18	0	155	140	0	22	1	108	104	4	19	1	243	249
2	18	0	155	163	6	14	0	761	757	10	19	0	87	81	0	24	1	139	141	4	20	1	177	181
2	19	0	232	207	6	15	0	283	277	10	20	0	-46	58	1	1	1	665	686	4	21	1	259	254
2	20	0	444	455	6	16	0	393	355	11	1	0	137	128	1	2	1	918	923	4	22	1	-39	20
2	21	0	75	75	6	17	0	338	334	11	2	0	145	132	1	3	1	453	416	4	23	1	125	130
2	22	0	-35	24	6	18	0	304	302	11	3	0	66	66	1	4	1	562	574	4	24	1	44	47
2	23	0	137	133	6	19	0	231	234	11	4	0	155	170	1	5	1	763	777	4	25	1	91	95
2	24	0	141	138	6	20	0	96	96	11	5	0	164	176	1	6	1	422	446	5	1	1	34	11
2	25	0	90	93	6	21	0	156	154	11	6	0	226	229	1	7	1	590	588	5	2	1	306	297
3	1	0	50	55	6	22	0	-39	4	11	7	0	149	142	1	8	1	682	709	5	3	1	597	591
3	2	0	1161	1159	6	23	0	132	127	11	8	0	180	174	1	9	1	922	949	5	4	1	301	264
3	3	0	821	788	6	24	0	-44	10	11	9	0	248	232	1	10	1	264	236	5	5	1	271	265
3	4	0	545	466	7	1	0	165	154	11	10	0	76	65	1	11	1	596	614	5	6	1	351	360
3	5	0	372	376	7	2	0	523	513	11	11	0	232	221	1	12	1	249	243	5	7	1	222	232
3	6	0	351	383	7	3	0	-39	37	11	12	0	135	143	1	13	1	412	439	5	8	1	1022	1060
3	7	0	614	613	7	4	0	650	652	11	13	0	211	210	1	14	1	280	286	5	9	1	341	331
3	8	0	492	533	7	5	0	231	235	11	14	0	222	233	1	15	1	259	260	5	10	1	320	311
3	9	0	718	738	7	6	0	722	733	11	15	0	146	138	1	16	1	130	124	5	11	1	72	64
3	10	0	526	538	7	7	0	-64	1	11	16	0	52	83	1	17	1	257	258	5	12	1	376	378
3	11	0	234	224	7	8	0	933	924	11	17	0	-35	5	1	18	1	105	101	5	13	1	122	126
3	12	0	128	132	7	9	0	335	336	11	18	0	56	64	1	19	1	49	44	5	14	1	415	413
3	13	0	721	726	7	10	0	328	333	11	19	0	-41	27	1	20	1	76	69	5	15	1	88	82
3	14	0	351	403	7	11	0	39	46	12	0	0	485	506	1	21	1	165	161	5	16	1	198	201
3	15	0	154	159	7	12	0	144	147	12	1	0	45	25	1	22	1	80	77	5	17	1	222	218
3	16	0	114	111	7	13	0	451	462	12	2	0	256	278	1	23	1	121	125	5	18	1	170	175
3	17	0	324	345	7	14	0	352	349	12	3	0	110	98	1	24	1	161	159	5	19	1	55	33
3	18	0	216	209	7	15	0	-41	22	12	4	0	56	47	1	25	1	44	46	5	20	1	90	92
3	19	0	86	74	7	16	0	54	58	12	5	0	70	68	2	0	1	2010	2110	5	21	1	47	43
3	20	0	92	91	7	17	0	165	160	12	6	0	66	65	2	1	1	736	765	5	22	1	87	88
3	21	0	72	65	7	18	0	77	71	12	7	0	-35	30	2	2	1	623	635	5	23	1	76	77
3	22	0	155	167	7	19	0	78	80	12	8	0	70	76	2	3	1	513	530	5	24	1	195	193
3	23	0	219	210	7	20	0	112	116	12	9	0	51	22	2	4	1	483	458	6	0	1	935	868
3	24	0	156	157	7	21	0	171	174	12	10	0	144	136	2	5	1	1062	1094	6	1	1	715	735
3	25	0	46	23	7	22	0	-44	44	12	11	0	33	34	2	6	1	720	719	6	2	1	51	46
4	0	0	255	118	7	23	0	62	61	12	12	0	218	214	2	7	1	376	336	6	3	1	548	536
4	1	0	458	373	8	0	0	723	654	12	13	0	90	100	2	8	1	503	471	6	4	1	761	774
4	2	0	109	69	8	1	0	1045	1067	12	14	0	132	135	2	9	1	272	264	6	5	1	490	460
4	3	0	357	324	8	2	0	434	436	12	15	0	-41	33	2	10	1	347	375	6	6	1	442	435
4	4	0	731	665	8	3	0	746	782	12	16	0	185	193	2	11	1	115	121	6	7	1	288	297
4	5	0	61	77	8	4	0	776	778	12	17	0	-42	11	2	12	1	125	120	6	8	1	262	273
4	6	0	452	434	8	5	0	625	641	13	1	0	85	96	2	13	1	153	162	6	9	1	103	95
4	7	0	436	456	8	6	0	154	156	13	2	0	121	125	2	14	1	393	381	6	10	1	226	236
4	8	0	159	164	8	7	0	318	320	13	3	0	68	73	2	15	1	128	115	6	11	1	259	268

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
6	12	1	210	206	11	6	1	150	148	1	17	2	276	264	5	22	2	81	75	10	13	2	308	310
6	13	1	140	131	11	7	1	334	321	1	18	2	257	245	5	23	2	-40	31	10	14	2	105	105
6	14	1	226	236	11	8	1	211	205	1	19	2	168	166	5	24	2	77	75	10	15	2	154	156
6	15	1	313	307	11	9	1	220	215	1	20	2	96	97	6	0	2	313	359	10	16	2	240	247
6	16	1	234	224	11	10	1	201	202	1	21	2	185	195	6	1	2	608	791	10	17	2	136	140
6	17	1	236	275	11	11	1	149	143	1	22	2	-40	17	6	2	2	492	445	10	18	2	132	131
6	18	1	243	259	11	12	1	105	103	1	23	2	146	141	6	3	2	528	520	10	19	2	99	94
6	19	1	236	242	11	13	1	251	251	1	24	2	89	76	6	4	2	675	672	11	1	2	173	175
6	20	1	135	129	11	14	1	135	131	1	25	2	165	173	6	5	2	336	337	11	2	2	352	350
6	21	1	154	152	11	15	1	159	158	2	0	2	580	564	6	6	2	366	346	11	3	2	272	271
6	22	1	-40	43	11	16	1	93	92	2	1	2	642	646	6	7	2	159	161	11	4	2	113	121
6	23	1	115	117	11	17	1	146	144	2	2	2	905	907	6	8	2	766	783	11	5	2	402	395
6	24	1	72	61	11	18	1	90	89	2	3	2	615	598	6	9	2	152	166	11	6	2	187	192
7	1	1	634	641	11	19	1	-42	44	2	4	2	301	336	6	10	2	699	703	11	7	2	402	420
7	2	1	320	333	12	0	1	567	561	2	5	2	657	645	6	11	2	136	149	11	8	2	294	284
7	3	1	212	229	12	1	1	167	171	2	6	2	208	235	6	12	2	211	207	11	9	2	313	301
7	4	1	634	631	12	2	1	99	81	2	7	2	605	618	6	13	2	227	228	11	10	2	90	90
7	5	1	111	122	12	3	1	218	210	2	8	2	323	315	6	14	2	183	186	11	11	2	207	197
7	6	1	542	511	12	4	1	323	310	2	9	2	591	599	6	15	2	166	171	11	12	2	151	146
7	7	1	346	336	12	5	1	85	74	2	10	2	66	68	6	16	2	288	287	11	13	2	228	238
7	8	1	153	155	12	6	1	329	309	2	11	2	507	491	6	17	2	207	217	11	14	2	114	107
7	9	1	-45	52	12	7	1	86	83	2	12	2	786	775	6	18	2	55	65	11	15	2	163	166
7	10	1	634	641	12	8	1	169	174	2	13	2	370	394	6	19	2	281	277	11	16	2	51	70
7	11	1	255	269	12	9	1	49	43	2	14	2	421	408	6	20	2	54	55	11	17	2	68	68
7	12	1	451	447	12	10	1	319	323	2	15	2	144	159	6	21	2	167	164	11	18	2	66	69
7	13	1	31	51	12	11	1	64	70	2	16	2	55	108	6	22	2	71	82	12	0	2	344	386
7	14	1	220	225	12	12	1	96	103	2	17	2	65	69	6	23	2	91	97	12	1	2	85	97
7	15	1	140	132	12	13	1	45	26	2	18	2	351	373	7	1	2	362	356	12	2	2	614	618
7	16	1	156	167	12	14	1	256	263	2	19	2	170	172	7	2	2	212	239	12	3	2	42	44
7	17	1	137	147	12	15	1	94	94	2	20	2	334	322	7	3	2	362	380	12	4	2	604	596
7	18	1	205	209	12	16	1	255	246	2	21	2	116	124	7	4	2	171	169	12	5	2	98	93
7	19	1	71	67	12	17	1	-41	31	2	22	2	-36	35	7	5	2	383	363	12	6	2	237	225
7	20	1	111	113	13	1	1	-43	13	2	23	2	81	79	7	6	2	62	79	12	7	2	97	84
7	21	1	175	91	13	2	1	217	211	2	24	2	90	96	7	7	2	177	183	12	8	2	142	126
7	22	1	95	83	13	3	1	245	235	2	25	2	-35	28	7	8	2	505	502	12	9	2	65	68
7	23	1	46	42	13	4	1	207	205	3	1	2	768	782	7	9	2	176	177	12	10	2	147	157
8	0	1	232	243	13	5	1	134	126	3	2	2	209	213	7	10	2	232	227	12	11	2	49	57
8	1	1	367	361	13	6	1	180	195	3	3	2	1162	1146	7	11	2	50	63	12	12	2	195	204
8	2	1	474	482	13	7	1	323	324	3	4	2	163	193	7	12	2	273	269	12	13	2	56	53
8	3	1	530	583	13	8	1	96	109	3	5	2	865	863	7	13	2	-36	23	12	14	2	161	159
8	4	1	74	75	13	9	1	160	173	3	6	2	367	391	7	14	2	419	421	12	15	2	-40	10
8	5	1	241	256	13	10	1	118	116	3	7	2	660	633	7	15	2	117	129	12	16	2	92	89
8	6	1	451	458	13	11	1	152	158	3	8	2	926	947	7	16	2	203	192	13	1	2	43	48
8	7	1	309	307	13	12	1	101	96	3	9	2	701	711	7	17	2	166	172	13	2	2	189	174
8	8	1	215	209	13	13	1	213	205	3	10	2	522	505	7	18	2	-39	21	13	3	2	196	202
8	9	1	340	339	13	14	1	74	77	3	11	2	435	441	7	19	2	96	93	13	4	2	78	92
8	10	1	316	305	13	15	1	139	144	3	12	2	443	442	7	20	2	53	53	13	5	2	378	363
8	11	1	236	216	14	0	1	174	169	3	13	2	440	434	7	21	2	43	45	13	6	2	102	101
8	12	1	165	167	14	1	1	245	248	3	14	2	502	495	7	22	2	105	106	13	7	2	254	257
8	13	1	306	296	14	2	1	226	215	3	15	2	418	415	8	0	2	347	357	13	8	2	206	204
8	14	1	80	70	14	3	1	226	234	3	16	2	115	127	8	1	2	84	94	13	9	2	213	224
8	15	1	260	261	14	4	1	334	319	3	17	2	121	112	8	2	2	103	123	13	10	2	-39	4
8	16	1	277	262	14	5	1	142	157	3	18	2	92	94	8	3	2	49	50	13	11	2	187	197
8	17	1	220	217	14	6	1	79	91	3	19	2	65	66	8	4	2	269	283	13	12	2	96	83
8	18	1	111	107	14	7	1	69	48	3	20	2	164	166	8	5	2	58	65	13	13	2	177	176
8	19	1	236	240	14	8	1	94	88	3	21	2	89	89	8	6	2	241	235	13	14	2	-40	25
8	20	1	81	90	14	9	1	96	83	3	22	2	55	46	8	7	2	102	103	14	0	2	267	272
8	21	1	104	107	14	10	1	135	141	3	23	2	76	71	8	8	2	41	31	14	1	2	134	135
8	22	1	132	137	14	11	1	55	52	3	24	2	91	88	8	9	2	-33	23	14	2	2	142	146
9	1	1	306	313	14	12	1	153	162	3	25	2	-39	26	8	10	2	172	169	14	3	2	59	60
9	2	1	395	390	14	13	1	111	105	4	0	2	1174	1214	8	11	2	49	47	14	4	2	176	171
9	3	1	255	255	15	1	1	57	53	4	1	2	1433	1441	8	12	2	182	185	14	5	2	-40	17
9	4	1	175	201	15	2	1	151	153	4	2	2	757	759	8	13	2	151	151	14	6	2	250	244
9	5	1	442	455	15	3	1	176	175	4	3	2	1357	1365	8	14	2	208	211	14	7	2	80	77
9	6	1	310	317	15	4	1	68	50	4	4	2	197	198	8	15	2	288	291	14	8	2	123	135
9	7	1	238	218	15	5	1	190	188	4	5	2	733	750	8	16	2	115	113	14	9	2	169	175
9	8	1	308	270	15	6	1	142	135	4	6	2	709	743	8	17	2	317	327	14	10	2	-40	31
9	9	1	421	421	15	7	1	129	123	4	7	2	185	190	8	18	2	269	274	14	11	2	161	172
9	10	1	211	213	15	8	1	179	187	4	8	2	250	255	8	19	2	258	259	14	12	2	76	74
9	11	1	211	202	15	9	1	144	146	4	9	2	322	325	8	20	2	165	157	15	1	2	117	108
9	12	1	125	137	15	10	1	85	84	4	10	2	257	246	8	21	2	240	239	15	2	2	63	55
9	13	1	234	242	16	0	1	159	155	4	11	2	194	206	8	22	2	86	96	15	3	2	92	98
9	14	1	308	306	16	1	1	173	177	4	12	2	187	171	9	1	2	145	165	15	4	2	143	135
9	15	1	61	64	16	2	1	145	150	4	13	2	68	59	9	2	2	113	120	15	5	2	96	88
9	16	1	100	93	16	3	1	136	137	4	14	2	127	123	9	3	2	143	133	15	6	2	155	142
9	17	1	177	176	16	4	1	81	60	4	15	2	108	110	9	4	2	347	349	15	7	2	94	94
9	18	1	-40	49	16	5	1	115	113	4	16	2	213	219	9	5	2	173	182	15	8	2	66	62
9	19	1	67	69	0	0	2	412	415	4	17	2	76	64	9	6	2	294	293	15	9	2	81	84
9	20	1	90	90	0	2	2	463	482	4	18	2	82	86	9	7	2	119	108	16	0	2	103	99
9	21	1	47	48	0	4	2	677	667	4	19	2	151	147	9	8	2	70	61	16	1	2	64	54
10	0	1	241	213	0	6	2	338	307	4	20	2	94	78	9	9	2	152	150	16	2	2	-40	27
10	1	1	380	367	0	8	2	151	162	4	21	2	114	116	9	10	2	259	256	C	0	3	1177	1197
10	2	1	562	560	0	10	2	661	673	4	22	2	112	103	9	11	2	283	296	0	2	3	240	230
10	3	1	240	247	0	12	2	402	414	4	23	2	95	93	9	12	2	250	231	0	4	3	762	800
10	4	1	470	475	0	14	2	455	454	4	24	2	41	32	9	13	2	273	271	0	6	3	374	345
10	5	1	214	224	0	16	2	669	664	5	1	2	191	196	9	14	2	-38	19	0	8	3	681	704
10	6	1	140	137	0	18	2	516	538	5	2	2	256	241	9	15	2	272	283	0	10	3	127	126
10	7	1	285	290	0	20	2	380	381	5	3	2	477	455	9	16	2	69	76	0	12	3	595	572
10	8	1	131	91	0	22	2	488	487	5	4	2	903	914	9	17	2	207	203	C	14	3	228	217
10	9	1	43	13	0	24	2	158	152	5	5	2	266	262	9	18	2	65	72	0	16	3	263	264
10	10	1	166	173	1	1	2	427	408	5	6	2	756	765	9	19	2	105	102	0	18	3	418	416
10	11	1	157	161	1	3	2	734	728	5	7	2	208	196	9	20	2	105	107	C	20	3	446	449
10	12	1	269	307	1	3	2	73	83	5	8	2	576	577	9	21	2	87	95	0	22	3	106	100
10	13	1	82	79	1	4	2	258	216	5	9	2	113	122	10	0	2	466	451	C	24	3	123	134
10	14	1	148	149	1	5	2	134	136	5	10	2	605	806	10	1	2	139	119	1	1	3	115	106
10	15	1	124	121	1	6	2	170	173	5	11	2	222	220	10	2	2	200	204	1	2	3	116	120
10	16	1	167	171	1	7	2	357	432	5	12	2	552	559	10	3	2	171	167	1	3	3	406	421
10	17	1	118	109	1	8	2	240	243	5	13	2	151	151	10	4	2	275	303	1	4	3	247	234
10	18	1	211	205	1	9	2	420	426	5	14	2	108	113	10	5	2	207	220	1	5	3	71	65
10	19	1	93	86	1	10	2	409	413	5	15	2	300	291	10	6	2	498	503	1	6	3	500	499
10	20	1	165	163	1	11	2	529	545	5	16	2	47	52	10	7	2	165	164	1	7	3	472	464
11	1	1	96	94	1	12	2	457	455	5	17	2	195	195	10	8	2	241	242	1	8	3	280	280
11	2	1	123	120	1	13	2	340	335	5	18	2	73	72	10	9	2	229	230	1	9	3	639	641
11	3	1	252	264	1	14	2	97	106	5	19	2	132	131	10	10	2	74	67	1	10	3	81	90
11	4	1	237	229	1	15	2	320	313	5	20	2	124	128	10	11	2	276	277	1	11	3	637	628
11	5	1	352	356	1	16	2	253	257	5	21	2	42	41	10	12	2	155	164	1	12	3	123	122

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(C)	F(C)	H	K	L	F(C)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
1	12	3	367	379	5	22	3	72	71	11	1	3	57	49	2	8	4	405	388	7	3	4	157	187
1	14	3	322	310	5	23	3	-40	20	11	2	3	77	81	2	9	4	331	329	7	4	4	323	318
1	15	3	256	260	6	0	3	254	315	11	3	3	142	151	2	10	4	229	229	7	5	4	142	139
1	16	3	54	52	6	1	3	489	496	11	4	3	205	198	2	11	4	441	447	7	6	4	472	461
1	17	3	173	171	6	2	3	674	665	11	5	3	336	344	2	12	4	48	47	7	7	4	170	168
1	18	3	133	125	6	3	3	513	526	11	6	3	145	158	2	13	4	324	327	7	8	4	563	558
1	19	3	158	165	6	4	3	183	151	11	7	3	248	242	2	14	4	158	151	7	9	4	50	23
1	20	3	137	138	6	5	3	341	349	11	8	3	-38	20	2	15	4	196	203	7	10	4	313	318
1	21	3	67	59	6	6	3	465	469	11	9	3	226	230	2	16	4	112	113	7	11	4	210	209
1	22	3	54	55	6	7	3	261	276	11	10	3	103	87	2	17	4	222	219	7	12	4	145	144
1	23	3	123	119	6	8	3	251	265	11	11	3	261	255	2	18	4	339	342	7	13	4	72	87
1	24	3	85	76	6	9	3	72	73	11	12	3	153	155	2	19	4	90	97	7	14	4	239	228
2	0	3	1126	1115	6	10	3	218	214	11	13	3	176	175	2	20	4	175	180	7	15	4	250	254
2	1	3	541	548	6	11	3	63	62	11	14	3	59	61	2	21	4	132	133	7	16	4	53	49
2	2	3	567	557	6	12	3	268	255	11	15	3	155	170	2	22	4	102	103	7	17	4	53	58
2	3	3	666	661	6	13	3	241	251	11	16	3	73	70	2	23	4	-41	8	7	18	4	-40	40
2	4	3	411	396	6	14	3	112	105	11	17	3	55	63	3	1	4	145	148	7	19	4	85	86
2	5	3	115	94	6	15	3	243	245	12	0	3	310	305	3	2	4	223	193	7	20	4	57	53
2	6	3	340	356	6	16	3	76	67	12	1	3	192	195	3	3	4	58	63	8	0	4	148	149
2	7	3	421	387	6	17	3	255	255	12	2	3	332	339	3	4	4	129	146	8	1	4	704	706
2	8	3	-30	29	6	18	3	103	107	12	3	3	54	45	3	5	4	239	228	8	2	4	580	559
2	9	3	73	77	6	19	3	163	155	12	4	3	155	173	3	6	4	55	44	8	3	4	508	516
2	10	3	276	264	6	20	3	126	125	12	5	3	92	85	3	7	4	231	221	8	4	4	325	336
2	11	3	171	174	6	21	3	144	149	12	6	3	310	310	3	8	4	578	579	8	5	4	391	389
2	12	3	150	154	6	22	3	157	156	12	7	3	64	65	3	9	4	110	116	8	6	4	81	72
2	13	3	63	55	7	1	3	333	320	12	8	3	242	238	3	10	4	90	84	8	7	4	367	363
2	14	3	86	80	7	2	3	131	126	12	9	3	99	93	3	11	4	452	450	8	8	4	74	62
2	15	3	79	85	7	3	3	167	166	12	10	3	136	136	3	12	4	157	160	8	9	4	39	28
2	16	3	412	411	7	4	3	491	515	12	11	3	-40	29	3	13	4	180	172	8	10	4	103	109
2	17	3	134	132	7	5	3	275	265	12	12	3	221	221	3	14	4	126	123	8	11	4	-37	11
2	18	3	144	149	7	6	3	459	472	12	13	3	-41	24	3	15	4	323	324	8	12	4	89	89
2	19	3	205	206	7	7	3	146	156	12	14	3	124	114	3	16	4	-35	41	8	13	4	153	156
2	20	3	236	240	7	8	3	251	210	12	15	3	-42	11	3	17	4	112	114	8	14	4	82	84
2	21	3	103	103	7	9	3	141	137	13	1	3	133	137	3	18	4	102	104	8	15	4	95	87
2	22	3	136	147	7	10	3	361	363	13	2	3	57	54	3	19	4	124	123	8	16	4	-43	28
2	23	3	111	117	7	11	3	76	76	13	3	3	76	86	3	20	4	78	78	8	17	4	73	69
2	24	3	76	74	7	12	3	356	353	13	4	3	166	159	3	21	4	105	105	8	18	4	106	107
3	1	3	303	298	7	13	3	124	122	13	5	3	195	191	3	22	4	-42	42	8	19	4	-41	28
3	2	3	158	161	7	14	3	148	155	13	6	3	194	206	4	0	4	364	350	9	1	4	161	168
3	3	3	346	330	7	15	3	64	72	13	7	3	210	205	4	1	4	-32	34	9	2	4	73	78
3	4	3	339	315	7	16	3	193	206	13	8	3	136	140	4	2	4	322	329	9	3	4	108	121
3	5	3	717	720	7	17	3	48	59	13	9	3	203	209	4	3	4	119	111	9	4	4	220	235
3	6	3	554	548	7	18	3	134	131	13	10	3	110	96	4	4	4	59	69	9	5	4	511	511
3	7	3	228	237	7	19	3	123	124	13	11	3	148	146	4	5	4	127	134	9	6	4	379	363
3	8	3	44	40	7	20	3	121	123	13	12	3	46	48	4	6	4	179	179	9	7	4	227	210
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3	16	3	132	130	8	6	3	167	166	14	6	3	62	69	4	14	4	124	125	9	15	4	58	54
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3	19	3	154	154	8	9	3	237	233	14	9	3	47	34	4	17	4	275	269	9	18	4	134	131
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3	21	3	-41	23	8	11	3	252	266	15	1	3	65	86	4	19	4	276	283	10	1	4	126	133
3	22	3	37	81	8	12	3	211	203	15	2	3	123	110	4	20	4	122	117	10	2	4	265	260
3	23	3	111	112	8	13	3	235	244	15	3	3	-42	27	4	21	4	252	263	10	3	4	243	239
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4	12	3	349	345	9	6	3	170	156	0	20	4	73	71	5	13	4	56	43	10	17	4	48	37
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4	16	3	133	135	9	10	3	112	104	1	3	4	356	333	5	17	4	47	57	11	4	4	143	152
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4	18	3	166	154	9	12	3	118	124	1	5	4	641	641	5	19	4	66	72	11	6	4	-39	29
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4	22	3	-39	29	9	16	3	54	52	1	9	4	751	767	6	1	4	398	406	11	10	4	-40	26
4	23	3	87	89	9	17	3	-41	43	1	10	4	219	227	6	2	4	50	61	11	11	4	96	104
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5	2	3	531	525	9	19	3	113	116	1	12	4	115	111	6	4	4	73	78	11	13	4	138	141
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5	7	3	57	67	10	4	3	425	426	1	17	4	-37	25	6	9	4	180	173	12	2	4	81	77
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5	9	3	245	244	10	6	3	178	176	1	19	4	84	81	6	11	4	125	124	12	4	4	168	179
5	10	3	450	465	10	7	3	108	96	1	20	4	170	179	6	12	4	539	547	12	5	4	73	60
5	11	3	183	173	10	8	3	52	67	1	21	4	-39	18	6	13	4	138	137	12	6	4	81	80
5	12	3	148	134	10	9	3	126	118	1	22	4	-38	13	6	14	4	62	64	12	7	4	75	55
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5	14	3	356	403	10	11	3	117	115	2	0	4	392	405	6	16	4	143	143	12	9	4	-40	49
5	15	3	134	134	10	12	3	116	112	2	1	4	326	328	6	17	4	194	192	12	10	4	54	38
5	16	3	98	100	10	13	3	85	68	2	2	4	650	639	6	18	4	73	75	12	11	4	-40	29
5	17	3	67	69	10	14	3	200	197	2	3	4	79	88	6	19	4	172	180	12	12	4	98	100
5	18	3	60	52	10	15	3	128	134	2	4	4	567	565	6	20	4	124	113	12	13	4	-40	18
5	19	3	85	92	10	16	3	102	95	2	5	4	53	54	6	21	4	145	138	13	1	4	94	96
5	20	3	75	73	10	17	3	91	100	2	6	4	203	206	7	1	4	-37	29	13	2	4	44	42
5	21	3	91	89	10	18	3	156	151	2	7	4	417	412	7	2	4	515	511	13	3	4	93	93

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
13	4	4	78	78	4	17	5	209	208	10	11	5	-40	19	4	8	6	137	131	0	0	7	100	130
13	5	4	89	91	4	18	5	124	128	10	12	5	103	106	4	9	6	-39	28	0	2	7	251	247
13	6	4	52	45	4	19	5	209	212	10	13	5	42	41	4	10	6	98	97	0	4	7	84	100
13	7	4	171	175	4	20	5	157	151	10	14	5	174	180	4	11	6	-39	33	0	6	7	197	192
13	8	4	106	103	5	1	5	91	66	11	1	5	73	75	4	12	6	-39	22	0	8	7	107	127
13	9	4	76	76	5	2	5	227	211	11	2	5	83	79	4	13	6	87	96	0	10	7	245	239
13	10	4	-41	25	5	3	5	183	178	11	3	5	73	86	4	14	6	68	70	0	12	7	137	120
14	0	4	228	232	5	4	5	165	159	11	4	5	86	85	4	15	6	44	47	0	14	7	193	199
14	1	4	82	85	5	5	5	234	211	11	5	5	235	225	4	16	6	67	66	1	1	7	80	75
14	2	4	124	124	5	6	5	174	183	11	6	5	101	107	4	17	6	72	76	1	3	7	82	61
14	3	4	87	97	5	7	5	162	155	11	7	5	164	171	5	1	6	143	136	1	3	7	67	56
14	4	4	128	127	5	8	5	367	361	11	8	5	-41	26	5	2	6	102	112	1	4	7	130	133
14	5	4	141	150	5	9	5	70	57	11	9	5	132	135	5	3	6	111	110	1	5	7	107	107
14	6	4	123	123	5	10	5	344	333	11	10	5	78	76	5	4	6	325	321	1	6	7	71	69
14	7	4	163	173	5	11	5	134	141	11	11	5	133	142	5	5	6	-39	20	1	7	7	148	153
0	0	5	732	719	5	12	5	217	215	11	12	5	58	105	5	6	6	400	400	1	8	7	127	124
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0	4	5	691	681	5	14	5	234	235	12	1	5	85	100	5	8	6	280	290	1	10	7	48	49
0	6	5	244	237	5	15	5	53	52	12	2	5	175	176	5	9	6	120	111	1	11	7	246	239
0	8	5	350	360	5	16	5	52	86	12	3	5	54	59	5	10	6	319	330	1	12	7	70	56
0	10	5	192	191	5	17	5	-40	23	12	4	5	104	98	5	11	6	83	93	1	13	7	160	160
0	12	5	265	263	5	18	5	33	55	12	5	5	56	50	5	12	6	180	173	1	14	7	112	112
0	14	5	203	198	5	19	5	70	66	12	6	5	248	244	5	13	6	117	120	1	15	7	98	93
0	16	5	120	122	6	0	5	268	279	12	7	5	-41	11	5	14	6	42	47	2	0	7	339	339
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0	20	5	133	135	6	2	5	404	401	12	9	5	74	71	5	16	6	-41	33	2	2	7	236	235
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1	3	5	404	424	6	5	5	154	178	13	3	5	56	58	6	1	6	272	276	2	5	7	205	202
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1	5	5	228	223	6	7	5	161	161	13	5	5	178	178	6	3	6	256	250	2	7	7	92	104
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1	7	5	443	446	6	9	5	54	67	0	0	6	82	74	6	5	6	161	153	2	9	7	128	124
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1	12	5	73	86	6	14	5	127	122	0	10	6	117	132	6	10	6	283	289	2	14	7	98	95
1	13	5	153	161	6	15	5	139	143	0	12	6	147	152	6	11	6	84	84	2	15	7	63	64
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1	16	5	54	55	6	18	5	56	57	0	18	6	220	220	6	14	6	141	140	3	3	7	199	200
1	17	5	69	75	6	19	5	102	101	1	1	6	70	92	6	15	6	118	125	3	4	7	178	170
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2	17	5	78	66	8	3	5	287	289	2	3	6	267	266	8	5	6	50	54	4	10	7	118	118
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3	15	5	90	97	9	5	5	147	169	3	4	6	90	42	9	10	6	94	96	6	1	7	192	199
3	16	5	81	75	9	6	5	156	147	3	5	6	270	280	9	11	6	107	112	6	2	7	157	159
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3	18	5	81	76	9	8	5	271	266	3	7	6	355	356	10	0	6	204	198	6	4	7	191	204
3	19	5	-41	35	9	9	5	134	122	3	8	6	311	326	10	1	6	110	116	6	5	7	178	170
3	20	5	88	84	9	10	5	111	116	3	9	6	227	230	10	2	6	166	184	6	6	7	116	102
4	0	5	154	166	9	11	5	100	99	3	10	6	264	262	10	3	6	50	54	6	7	7	74	87
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4	3	5	320	322	9	14	5	185	181	3	13	6	45	54	10	6	6	231	236	6	10	7	56	57
4	4	5	153	139	9	15	5	129	133	3	14	6	221	233	10	7	6	104	105	6	11	7	-44	55
4	5	5	124	114	9	16	5	69	66	3	15	6	43	21	10	8	6	145	139	6	12	7	59	59
4	6	5	128	143	10	0	5	239	233	3	16	6	157	158	10	9	6	62	69	7	1	7	-43	39
4	7	5	254	263	10	1	5	201	211	3	17	6	55	43	10	10	6	62	72	7	2	7	74	80
4	8	5	153	148	10	2	5	204	205	3	18	6	-40	21	11	1	6	-41	17	7	3	7	72	69
4	9	5	152	178	10	3	5	207	202	4	0	6	486	488	11	2	6	100	99	7	4	7	137	136
4	10	5	182	178	10	4	5	276	267	4	1	6	632	646	11	3	6	135	131	7	5	7	74	70
4	11	5	266	268	10	5	5	173	156	4	2	6	235	242	11	4	6	77	77	7	6	7	158	153
4	12	5	206	214	10	6	5	78	73	4	3	6	462	444	11	5	6	184	203	7	7	7	90	87
4	13	5	128	129	10	7	5	131	129	4	4	6	206	218	11	6	6	107	97	7	8	7	101	106
4	14	5	52	65	10	8	5	109	103	4	5	6	244	247	11	7	6	244	244	7	9	7	54	55
4	15	5	146	150	10	9	5	66	66	4	6	6	235	235	12	0	6	289	296	7	10	7	146	150
4	16	5	160	176	10	10	5	65	62	4	7	6	186	191	12	1	6	81	89	7	11	7	64	49

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
6	0	7	66	71	5	5	7	62	63	1	8	8	-43	56	3	2	8	57	62	4	7	8	48	43
6	1	7	64	102	5	6	7	56	124	1	9	8	208	212	3	3	8	-42	29	4	8	8	-44	33
6	2	7	83	65	0	0	8	505	505	1	10	8	64	60	3	4	8	-41	16	5	1	8	-43	27
6	3	7	98	103	0	2	8	312	330	2	0	8	144	163	3	5	8	-42	29	5	2	8	-42	17
6	4	7	52	52	0	4	8	278	275	2	1	8	101	112	3	6	8	93	84	5	3	8	59	64
6	5	7	136	131	0	6	8	181	171	2	2	8	155	170	3	7	8	-41	31	5	4	8	-42	31
6	6	7	70	65	0	8	8	86	68	2	3	8	70	55	3	8	8	112	107	5	5	8	53	63
6	7	7	152	152	0	10	8	64	57	2	4	8	145	147	3	9	8	117	101	5	6	8	81	86
6	8	7	72	71	1	1	8	-44	39	2	5	8	-42	33	4	0	8	45	35	5	7	8	75	62
6	9	7	158	152	1	2	8	-41	26	2	6	8	106	114	4	1	8	-42	39	6	0	8	-44	47
6	1	7	-41	5	1	3	8	157	163	2	7	8	71	78	4	2	8	-40	13	6	1	8	185	186
6	2	7	114	107	1	4	8	105	103	2	8	8	117	111	4	3	8	46	44	6	2	8	-43	25
6	3	7	135	126	1	5	8	154	155	2	9	8	137	132	4	4	8	64	62	6	3	8	157	163
6	4	7	57	67	1	6	8	106	104	2	10	8	131	15	4	5	8	-41	5	6	4	8	58	67
					1	7	8	225	225	3	1	8	-41	24	4	6	8	60	53	6	5	8	126	116

one, and C was found to be 1.2×10^{-6}. Additional refinement cycles were then carried out, and the final R value was 0.037, with unobserved reflections included when $|F_c|$ exceeds the observable limit. In the last refinement cycle, all parameter shifts were less than 10 % of its standard deviation. The atomic coordinates of Table 1 and the thermal parameters of Table 2 are from the last of these refinement cycles.

The atoms O(1), O(2), C(3), and C(4) have the highest and most anisotropic thermal parameters. An electron density map showed considerably lower and more extended positive regions for these atoms than for the other atoms of the same kind.

A structure factor calculation, not including C(3) and C(4), was carried out ($R = 0.066$), followed by a difference electron density map. From this map the carbon atoms were located very close to the positions arrived at by the least squares refinement. The regions of positive electron density were extended approximately normal to the plane through cobalt and the four nitrogen atoms, but there was no tendency of splitting of the peaks.

A difference electron density map based on the structure factors calculated from the parameters of Tables 1 and 2 allowed the location of the hydrogen atoms, except those of C(3) and C(4). With these atoms included in the structure factor calculations, the R value was 0.033. An attempt to include the hydrogen atoms in the refinement was, however, not successful, as some of them were shifted to pisitions giving unreasonable bond lengths and angles. The hydrogen atoms are therefore not included in the final structure factor calculations listed in Table 3.

RESULTS

The hexathionate ion has the extended *trans-trans* form in this salt, and is thus different from the *cis-cis* form found in the potassium barium salt.[7,8] The difference between the two rotational isomeric forms is a rotation of approximately 180° about the middle S – S bond, or a rotation of approximately 180° about the S(2) – S(3) and S(4) – S(5) bonds.

The dimensions of the hexathionate ion in the *trans-trans* form are listed in Table 4. The standard deviations given in parentheses include estimated uncertainties in unit cell dimensions. A twofold symmetry axis passes between the two middle sulphur atoms normal to the bond between these atoms, and a prime denotes the equivalent of an atom across this axis. Fig. 1 is a stereo-

Table 4. Dimensions of the *trans-trans* form of the hexathionate ion. Standard deviations are given in parentheses.

Bond lengths and angles	
S(1) – S(2) = 2.1323(21) Å	∠S(1) – S(2) – S(3) = 105.08(11)°
S(2) – S(3) = 2.0184(29)	∠S(2) – S(3) – S(3′) = 107.35(11)°
S(3) – S(3′) = 2.0687(34)	
S(1) – O(1) = 1.410(7)	∠S(2) – S(1) – O(1) = 105.9(2)°
S(1) – O(2) = 1.455(7)	∠S(2) – S(1) – O(2) = 99.0(3)°
S(1) – O(3) = 1.443(5)	∠S(2) – S(1) – O(3) = 105.4(2)°
	∠O(1) – S(1) – O(2) = 112.4(4)°
	∠O(1) – S(1) – O(3) = 117.8(4)°
	∠O(2) – S(1) – O(3) = 114.9(3)°
Dihedral angles	
S(1)S(2)S(3)/S(2)S(3)S(3′) = 85.7°	S(2)S(3)S(3′)/S(3)S(3′)S(2′) = 71.4°
S(3)S(2)S(1)/S(2)S(1)O(1) = 72.3°	S(2)S(1)O(1)/S(2)S(1)O(2) = 116.5°
S(3)S(2)S(1)/S(2)S(1)O(2) = 171.1°	S(2)S(1)O(1)/S(2)S(1)O(3) = 125.5°
S(3)S(2)S(1)/S(2)S(1)O(3) = 53.1°	S(2)S(1)O(2)/S(2)S(1)O(3) = 118.0°
Non-bonded distances	
S(1) – S(3′) = 4.2089(22) Å	S(2) – S(2′) = 3.9715(31) Å
S(1) – S(2′) = 5.4282(20)	S(1) – S(1′) = 7.0786(28)

scopic view of the ion as seen along the twofold axis, and Fig. 2, with principal. bond lengths and angles, gives a view normal to this axis.

The differences between the present, refined dimensions of the hexathionate ion, and those arrived at on basis of the film data,[1] are within the errors estimated for the latter. The three independent bonds of the sulphur chain are thus of significantly different lengths.

The terminal S – S bonds are 2.132(2) Å, compared to 2.119(1) and 2.110(1) Å in the potassium barium salt, 2.124(6) and 2.110(6) Å in potassium pen-

Fig. 1. The *trans-trans* form of the hexathionate ion in $[Co(en)_2Cl_2]_2S_6O_6.H_2O$ as seen along the twofold axis. The ellipsoids represent 50 % probability.

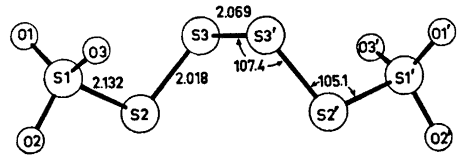

Fig. 2. The hexathionate ion as seen normal to the twofold axis.

tathionate hemitrihydrate,[9] and an average value of 2.12(2) Å in the barium pentathionates.[10] In all cases, these are the longer bonds of the sulphur chain.

The three divalent sulphur-divalent sulphur bonds are 2.018(3) Å, 2.069(3) Å, and 2.018(3) Å. They probably possess some double-bond character, arising from overlap of the $p\pi$ electron pair of one divalent sulphur atom with available 3*d* orbitals of a bond partner.[10] If it is assumed that in the sulphonate sulphur-divalent sulphur bond there is little double-bond character, the S(II) atom next to the sulphonate group has its $p\pi$ electron pair available for π-bond formation with one bond partner only, whereas each of the two central S(II) atoms has to share its $p\pi$ electron pair between two neighbours. One might therefore assume that the S(II) – S(II) bonds next to the sulphonate groups should be shorter than the central one.

When the dihedral angle associated with a sulphur-sulphur bond of this kind deviates much from 90°, there seems to be an increase in the bond length, probably caused by increase in the repulsion between the $p\pi$ electron pairs or by less favourable conditions for π-bonding.[11] The bond lengths, with associated dihedral angles given in parentheses, are 2.018 Å (85.7°), 2.069 Å (71.4°), and 2.018 Å (85.7°) in the present salt, and 2.042 Å (109.4°), 2.056 Å (89.0°), and 2.039 Å (106.3°) in the potassium barium salt. Assuming that the S(II) – S(II) bonds next to the sulphonate groups should be shorter than the central one, the differences between the bond lengths in the two salts might be correlated with the differences in dihedral angles. The small dihedral angle, 71.4°, in the present salt causes a lengthening of the central S(II) – S(II) bond and thus increases the difference between this bond and the terminal ones, which have dihedral angles of 85.7°. The large dihedral angles, 109.4° and 106.3°, in the potassium barium salt cause a lengthening of the two terminal S(II) – S(II) bonds and thus decrease the difference between these bonds and the central one, which has a dihedral ongle of 89.0°.

In potassium pentathionate hemitrihydrate [9] the average value of the S(II) – S(II) bond lengths is 2.029(7) Å with an average value of the dihedral angles of 83°, compared to 2.04 Å and 108° in the barium pentathionates.[10] The sulphur chain has the *trans* form in the potassium salt and the *cis* form in the barium salts.

The S – S – S angles vary more in the *cis-cis* chain than in the *trans-trans* chain. The individual values are 101.3(1)°, 110.2(1)°, 109.0(1)°, and 100.0(1)° in the former, and 105.1(1)°, 107.4(1)°, 107.4(1)°, and 105.1(1)° in the latter.

The sulphonate groups have the usual distorted tetrahedral form, the O – S – O angles being larger and the S – S – O angles being smaller than the tetrahedral angle. The S – S – O angle, involving the oxygen atom O(2) situated near the S(1) – S(2) – S(3) plane and with the S – O bond pointing in a direction opposite to that of the S(2) – S(3) bond, is 6.7° smaller than the average value of the two others. The reason for the apparent shortness of the S(1) – O(1) bond, 1.410(7) Å, might be the large thermal motion of O(1) normal to this bond. The largest differences between the sulphonate groups in this salt and in the potassium barium salt are in the degrees of rotation about the S – S bonds which differ by approximately 16°.

The dimensions of the *trans*-dichlorobis(ethylenediamine)cobalt(III) ion are listed in Table 5. The four nitrogen atoms are coordinated to cobalt in a

Table 5. Dimensions of the *trans*-dichlorobis(ethylenediamine)cobalt(III) ion. Standard deviations are given in parentheses. The ethylenediamine groups are N(1) – C(1) – C(2) – N(2) and N(3) – C(3) – C(4) – N(4).

Co – Cl(1) = 2.229(2) Å	∠Cl(1) – Co – N(1) = 90.5(2)°	∠N(1) – Co – N(2) = 87.2(2)°
Co – Cl(2) = 2.261(2)	∠Cl(1) – Co – N(2) = 90.7(2)°	∠N(3) – Co – N(4) = 85.9(2)°
Co – N(1) = 1.972(4)	∠Cl(1) – Co – N(3) = 90.2(2)°	∠N(1) – Co – N(4) = 92.4(2)°
Co – N(2) = 1.958(4)	∠Cl(1) – Co – N(4) = 91.2(2)°	∠N(2) – Co – N(3) = 94.5(2)°
Co – N(3) = 1.974(4)	∠Cl(2) – Co – N(1) = 90.1(2)°	∠Co – N(1) – C(1) = 106.8(3)°
Co – N(4) = 1.970(5)	∠Cl(2) – Co – N(2) = 89.4(2)°	∠Co – N(2) – C(2) = 109.0(3)°
N(1) – C(1) = 1.518(8)	∠Cl(2) – Co – N(3) = 89.3(2)°	∠Co – N(3) – C(3) = 109.5(4)°
N(2) – C(2) = 1.500(8)	∠Cl(2) – Co – N(4) = 88.8(2)°	∠Co – N(4) – C(4) = 109.5(5)°
N(3) – C(3) = 1.461(11)	∠Cl(1) – Co – Cl(2) = 179.4(1)°	∠N(1) – C(1) – C(2) = 106.8(4)°
N(4) – C(4) = 1.467(10)	∠N(1) – Co – N(3) = 178.2(2)°	∠N(2) – C(2) – C(1) = 107.7(4)°
C(1) – C(2) = 1.509(9)	∠N(2) – Co – N(4) = 178.1(2)°	∠N(3) – C(3) – C(4) = 112.8(7)°
C(3) – C(4) = 1.409(14)		∠N(4) – C(4) – C(3) = 115.2(7)°

planar arrangement, the maximum deviation of an atom from a least squares plane being 0.025 Å. The distances of the carbon atoms from this plane are: – 0.48 Å for C(1), 0.20 Å for C(2), 0.31 Å for C(3), and – 0.06 Å for C(4).The carbon atoms of one ethylenediamine group, C(3) and C(4), are thus closer to the cobalt-nitrogen plane than are the two other carbon atoms, and they form shorter C – N and C – C bonds, 1.46 and 1.41 Å, respectively, compared to 1.51 and 1.51 Å. C(3) and C(4) have large thermal motions nearly normal to the cobalt-nitrogen plane, and this is probably the reason for the short lengths and short distances from the plane. Similar effects of high thermal motions are found in the structure of $Cu_3(en)_2(CN)_4.H_2O$,[12] where disorder of the ethylenediamine groups or a dynamic flipping of the carbon atoms across the copper-nitrogen plane is suggested.

Hydrogen bonds are probably formed from the water molecule to O(3) and its image across the twofold axis on which the water oxygen atom is situated. The $H_2O – O$ distance is 3.153(9) Å, and the $O – H_2O – O$ angle is

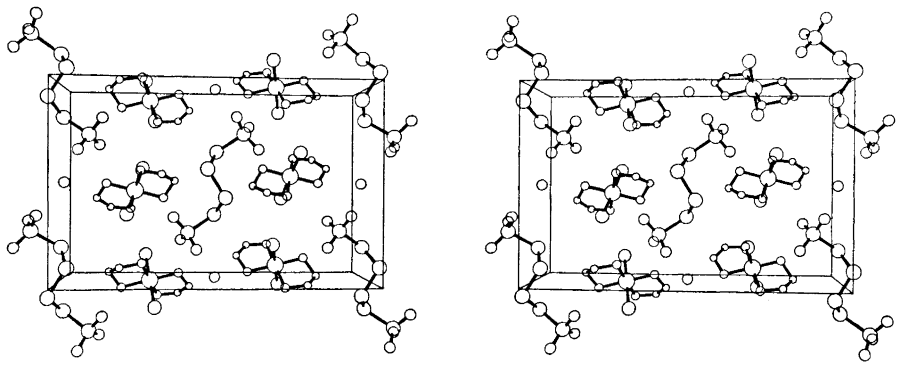

Fig. 3. A stereoscopic view of the cell packing in $[Co(en)_2Cl_2]_2S_6O_6.H_2O$ as seen normal to the *c* crystal plane.

95.8(3)°. N(1) probably forms a hydrogen bond to O(1) at $x,y,z-1$, the N–O distance being 2.910(7) Å. The hydrogen atoms located in the difference electron density map are in accordance with such hydrogen bonds.

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