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The Crystal Structure of Calcium cis(N)-trans(O_5)-Bis-(L-aspartato)cobaltate(III) cis(N)-trans(O_6)-Bis-(L-aspartato)cobaltate(III) Decahydrate

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Crystals of Ca cis(N)-trans (O_5) -[Co(L-asp)₂]cis(N)-trans (O_6) -[Co(L-asp)₂]. 10H₂O are orthorhombic with space group $P2_12_12_1$: a = 18.688 (3), b = 10.517 (2), c = 17.309 (3) Å and Z = 4. The structure was refined by least-squares methods with anisotropic temperature factors to give an R value of 0.041 on the basis of 3412 observed reflexions collected by the diffractometer method. The structure consists of two isomeric complex ions: cis(N)-trans (O_5) and cis(N)-trans (O_6) isomers, calcium ions and water molecules. Both complex cations exhibit distorted octahedral coordinations. The complex anions are held together by N-H···O hydrogen bonds to form a left-handed helix parallel to the b axis. A calcium ion is surrounded by three carboxylic oxygen atoms and four oxygen atoms of water molecules.

Introduction

There are three possible geometric isomers of the bis-(L-aspartato)cobaltate(III) ion when the ligand acts as a tridentate (Fig. 1). Two of the three isomers were isolated and characterized by Hosaka, Nishikawa & Shibata (1969). Later all three isomers were isolated and geometric configurations were assigned from absorption and circular dichroism spectra and nuclear magnetic resonance measurements (Yamada, Hidaka & Douglas, 1971; Froebe, Yamada, Hidaka & Douglas, 1971; Hidaka, Yamada & Douglas, 1972). Crystals of the calcium and lithium salts, having the composition $CaCo_2C_{16}H_{40}N_4O_{26}$ and $LiCoC_8H_{16}N_2O_{11}$, have been subjected to crystal-structure analysis in order to establish the absolute configurations and conformational details of the complex anions. The former crystals were obtained upon recrystallization of the blue-violet modification, which was considered to be the cis(N)-trans (O₅) isomer. The absorption and circular dichroism spectra of the compound in an aqueous solution are, however, not identical with those of the cis(N)-trans(O₅) isomer, but correspond to those of an equi-molar mixture of the two isomers, cis(N) $trans(O_5)$ and cis(N)-trans(O₆), indicating that the two isomers coexist in the crystal. On the other hand the latter crystals are expected to contain only one isomer. In the present paper the crystal structure of the calcium salt will be described.

Experimental

Crystal data Crystal data CaCo₂C₁₆H₄₀N₄O₂₆, M.W. 431.2 Orthorhombic a = 18.688 (3), b = 10.517 (2) and c = 17.309 (3) Å, U = 3417.9 Å³

 $D_m = 1.73, D_x = 1.68 \text{ g cm}^{-3}, Z = 4$

Mo K α ($\lambda = 0.7107$ Å), $\mu = 13.7$ cm⁻¹

Space group: $P2_{1}2_{1}2_{1}$ (No. 20)

Elementary analysis gave: C 21.97, H 4.79, N 6.74%; CaCo₂C₁₆H₄₀N₄O₂₆ requires C 22.28, H 4.68, N 6.50%. The intensities of the reflexions were measured on a Rigaku automated four-circle diffractometer. A crystal with approximate dimensions $0.3 \times 0.25 \times 0.3$ mm was mounted on the goniostat with the *b* axis roughly parallel to the φ axis. Mo $K\alpha$ radiation was used. 4600 independent reflexions up to $2\theta = 55^{\circ}$ were measured by employing the ω -2 θ scan technique, of which 3412 with $|F| > 3\sigma$ were regarded as 'observed'. Three reflexions were measured as references every 50 reflexions: the net counts of these reflexions did not alter noticeably over the period of data collection. The



Fig.1. Schematic drawings of (a) the trans(N), (b) the cis(N)trans(O_5) and (c) the cis(N)-trans(O_6) isomers of the $[Co(L-asp)_2]^-$ ion.

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Table 1. Observed and calculated structure amplitudes

		H FU FC	H FO FC	н на но	H FO FC	H FO FC	H F0 FC	H FO FC	н ға ғе	4 FO FC	H T.I FC	H FU FC	- FO FC	H FO FC	H FO FC
- 141 S'A 4 MIT V-0 - 215 217	4 7 4 74 • • • • • •	4 4 51 0 204 A7 1 3 1 1	A 784 64A 7 244 211 8 242 214	14. 40 40 17 114 114 16. 41 34 1/ 109 106	5 163 173 6 146 140 7 158 153 8 494 395	7 305 202 H 317 313 9 390 303 10 133 127	12 170 170 1.1. 45 43 14 44 14	11 127 120 11 241 241 12 20 24 13 128 172	5 105 182 5 105 182 6 55 69	4 13X 144 6 HO HA 7 14; 144 H 75 73	14 107 162 15 171 164 17 142 143	4 71 AN 5 726 724 6 A3 A3	15 83 88 14 90 90 15• 33 30 10 93 68	2 A 234	2 135 127 1 1 1 13
10 100 100 10 100 100 10 100 100	17 19 12	*2 105 105 *1 14 112 *4 4* 63	9 234 244 10 144 140 11 304 305	R.L. Q 2 1/ 74 72 R.L. R 2	9 246 249 10 189 182 11 124 122	11 308 312 12. 44 30 15 93 99	16 77 70 1/0 48 37 1P 60 75	14 79 11 15 68 90 14•1.0 45	U+ 33 48 1 469 451 2 185 202	9 105 104 10 124 1/3 11 157 149	14 111 113 19 151 142 20 104 111 21 113 117	7 226 210 8 71 79 9 221 221	17 46 48 18 64 64 8,1: 8 8	4 3'U 300 4 250 250 5 50 7 90 50	· A1 71 •7 75 91
10 403 400 10 +0 20 2-0 2-1	14+ 30 30 17 41 54 17 51 0	** 7* 73 *7 130 171 ** 130 125	13+ 44 41 14 275 270 15 204 210	A.L. 9 2 U 188 194 1* 30 24	13 00 02 10 90 92 17 55 57	14 111 104 15 85 85 18 209 211 19 125 119	1 04 C4	18 /5 68 14 126 120 20 75 75	4 223 324 5 205 237 6 1 ⁷⁹ 167	14 140 144 15 00 04 10 00 04	6 131 119 1• 47 54	11 124 122 12+ 35 35 14 137 144	1 85 7A 2 91 104 4 64 64	" 1/4 10Å	20 104 101 20 104 101
77 1+4 1+9 Hele 1 A 1 3/7 447 3 72 54	0 1/0 1/21 10 40 50 7 101 147	20 33 72 20 83 81 21 81 21	10 144 144 17 74 Hp 18 19h 198	2 144 133 3 67 73 4 65 54	10 76 71 22 AV 93 K-1 5 3	20 108 105 22 173 143 23 88 69	4 175 148 5 VA 18 6 07 04	21 A2 H2 K-11 4 5 P 51 41	/ 412 407 8 2/4 274 9 314 300	18 05 04 #+1 = 8 4 7 70 47	2 14 14 3 242 248 4 240 230 5 128 143	15 86 91 10 103 100 18+ 58 36 20 74 75	A 182 187 7. 51 49 A 123 124	170 4A 51 17 74 75	1 124 127 2 24" -44 1 11"
4 454 457 5 1 4 1 4 6 506 514	11 1 4 143	n 247 241 1- 40 55 2 385 590	20 123 127 21 50 67 22 121 127	0 159 154 / 53 52 0 67 66	1 180 145 2 204 189 3 393 342	0 57 52 1 204 160 2 109 124	P 171 JP1 6 244 202 10- 40 .16	2 321 322 1 142 369 4 237 229	11 137 137 12 182 190 11 55 59	3 6: 62 4 61 66 5 117 111	A 140 144 7 205 207 A 82 89	21 11A 121 K.L. 1 A 0 127 110	10 182 17A 15 83 87 F.Lz 9 8	14 1 2 100 14 1 1 10 17 11 00	4 145 134 6 202 510 6 117 120 7 185 102
# 220 21A	17 - 37 - 36 14 - 67 - 76 8-1 = 11 - 0	3 48 44 4 17n 180 5 82 85 6 272 273	1 194 205 1 355 345 2 276 274	10 117 117 12 68 63 13 68 85	4 207 270 5 230 225 6 123 115 7 195 185	3 284 250 4 219 239 5 4 2 444 5 287 279	11 129 118 12 218 212 13 184 185 14 51 20	A 513 125 7 145 08 8 3' 94	1/ 126 123 1/ 126 123 1/ 61 65 (0 133 139	A 177 185 7 139 143 8 103 47 9 100 48	10 296 294 11 122 118 12 83 81	2 318 316 3 584 489 4 144 114	2 104 90 3 62 66 4 87 82	K.I.E. n. 4 n. 44 73 1 344 100	# 175 141 • 175 176 •1 276 272
11 117 13A 12 2 ⁴ A 3rn 14 111 1,0	3 1 4 114	7 140 139 R 105 128 Q 109 107	3 146 150 4 174 1'4 5 67, MT	140 46 43 160 16 37 H-L: 10 2	A 260 270 0 262 240 10 219 213	7 122 171 A 104 93 9. 45 44	154 41 12 119 114 18 107 100	9 /r5 210 10 501 502 11 251 /50 12 172 76	<pre>/1 115 117 // 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1</pre>	10 170 177 11 141 141 13 63 64	13 113 108 14 70 46 15 103 102 16 146 140	5 207 200 6 153 140 7 157 149 8 60 62	5 97 91 7 59 52 8 86 87 9 112 123	2 1 7 114 3 1 4 47 4• 65 53 5 1 5 143	·2 14: 130 ·3 134 ·30 ·4 135 :38
25 1 17 1 4 22 46 44 8 11 2 2 0	A 1. A 140 7 1: 9 105 8 97 97	11 88 90 11 172 183 14 150 157	7 453 431 8 304 300 9 207 199	1 214 217 2 207 209 4 100 93	12 241 243 13 116 118 14 146 151	11 225 221 12 221 215 13 106 103	0 AR 70 1 259 241 2 84 25	1 104 184 14 214 214 15. 41 41	2. 30 47 3 386 390 5 236 219	17 59 54	17 97 108 18 83 93 19+ 50 57	9 83 92 10 298 303 11 158 140	12 104 107 13 120 121 14 55 57	A 21, 200 7 1.A 79 # 1.A 74 9 KA /7	17 116 172 44 44 75 *** 47 71
n 242 3rA 3 428 6-7 2 312 211 3 448 44 ⁰	10 100 102 10 100 103 11 00 57 8.1 12 0	16 55 65 17 67 65 16 164 167	10 544 544 11 50 74 12 194 145 13 142 143	0 167 187 0 16 84 8 94 49 4 88 83	14 146 151 16 134 130 17 105 105 19 115 116	14 116 115 15 70 75 15 75 241 17 99 90	3 A5 A6 4 150 140 5 11A 113 0 P1 P2	17 124 121 17 124 121 18. 41 34 19 54 60	<pre>> 121 100 / 270 250 h= 41 41 y 225 212</pre>	3 18/ 177 4- 30 - 20 5 73 - 67	21. 48 48 K-1 5 7 0 300 296	13 267 277 14 103 106 15 79 79	0 144 174 1 154 154 2 41 44	12 117 136 12 17 136 12 18 75	*.1 4 10 1 211 200
4 246 274 6 01 P2 6 316 200 7 101 00	10 27 47 20 11 28 5 25 41 7 54 50	<pre><0 55 47 K.L= 7 1 0 313 307 1 205 202</pre>	14 97 102 15 225 219 16 171 160	10 142 139 11 102 100 12 86 85	20 64 71 21 69 67 K-L2 6 3	10 157 158 20 63 58 21 67 63	/ 116 111 M1 48 43 10 58 52	20 17: 175 21 141 141 7. 1 5 5	10 115 122 11 111 120 12 74 66	A 143 140 7 A: 60 8 114 108	1 137 131 2 101 07 3 118 104 4 151 158	16+ 37 18 17 99 91 18 133 135	1 124 115 1 119 112 /* 50 54	14 H /A	3 175 179 4 143 - 44 5- 24 31
* 107 111 • 256 250 10 116 116	A 112 105 K-11 13 0 3+ 51 04	2 243 241 3 58 51 4 353 351	20 7, 7A 22 56 53	15 A2 74 8.L 11 2 1 AV 71	1 215 213 2 221 215 3 199 193	0 105 130 1 213 221 2 262 245	12 54 52 13 41 44 15 119 114	1 35n 357 2 594 524 3 44 191	14 199 194 15 204 193 16 57 85	11 01 04 12 0, 54 13 96 87	5 A0 A7 A 238 243 7 85 93 8 340 344	20 76 45 21 49 50 K-L= 2 8	• • • • • • • • • • • • • • • • • • •	18 1.1 54	A 00 100 7 141 146 8 154 154 9 AC 44
13 341 310 14 84 90 14 119 125	7 70 3 252 245 4 140 70	5 1/0 144 6 313 310 / 80 /2 0 159 160	23 24 61 x+1 = 3 2 0 11'- 103 1 239 201	3 41 60 4 70 69 7 130 130	4 133 137 5 123 125 6 206 208 8 111 10 ⁸	4 283 281 5. 32 44 6 137 154	H, 1 T 10 4 0 145 104 1 40 71	5 104 185 A 104 .07 7 83 75	18 104 110 19 00 50 70 // 79	15 107 114 5 10 10 10 8 30 25	9 194 188 10 245 247 11 157 157	1 326 3nA 2 145 154 3 114 125	H.1 - 11 A L. 41 -0 1 - 64 -64	1 17 117 1 27 A 250 4 1 5 100	12/172
1, 1, 7 1, 0 1 N 17 07 20 77 77 K 1 8 3 0	A 164 209 7 312 292 8 114 104	9 100 97 10 158 150 12+ 38 33 13 71 74	2 256 263 3 184 194 4 33; 324 5 147 146	A 94 105 / A7 71 8 A3 BA 9 91 AA	9 140 138 10 214 218 11 137 136 12 191 193	7 252 264 8 494 496 9 244 242 10 06 61	2 A1 A0 3 151 153 4 171 176 5 114 176	9 :44 · 14 •1 182 181 •1 04 01	21+ 25 31 22 76 77 NyL1 2 6 1 244 273	1 114 104 2. 40 44 3 144 168 4 83 84	13 97 102 14 93 89 15 70 63	4 45 27 5 142 177 6 234 235 7 210 216	· · · · · · · · · · · · · · · · · · ·	A 1 9 11	······································
1 45 4 2 141 215 3 217 218	7 404 4/7 11 344 310 11 345 353	15 86 85 16 162 159 17 70 74	A 20) 204 7 377 344 Pa 44 41	10 70 70 11 72 73 12 67 61	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	11 314 200 12 144 142 13 82 86	7 124 117 7 124 117 81 47 14	12 157 150 11 100 110 14 207 205	2 108 152 3 /13 193 4 506 571	5 60 52 6* 41 41 / 195 200 8 59 58	1A 140 154 17 71 71 18+ 40 58 19+ 50 38	6 144 150 9 273 266 10 239 237 11 250 251	· · · · · · · · · · · · · · · · · · ·	10 .0 .0 .0	1 153 144
5 270 3nm 6 55 52 7 162 1/5	13 61 68 14 167 115 15 439 438	19- 30 33 1-L= 8 1 0 4 ⁰ 2 4 ⁰ 5	10 251 240 11 276 266 17 75 76	1* 51 52 3 108 104 4 55 56	17 77 74 18 69 70 19 82 85	14 145 148 15, 51 48 16 82 61 18 70 66	10 25 81 10 25 37 17 12 114	A A4 74 7 01 84 8 140 146	6 7/7 260 / 1/6 163 8 407 447	v 7/ 75 10 A0 M2 11 111 111	2n 77 80 K-1 = 0 7 C 725 274	12 59 53 13 60 70 14 99 101	105 117 - 76 74 20 94 60	10 /1 /0	2 /0. /00 3 157 101 4 156 109
0 174 31A	19+ 45 54 10 144 142 23+ 39 32	2 99 100 3 184 193 4 321 333	13 143 144 15+ 38 20 16 118 118 17- 32 30	9+ 38 36 *-Lr 13 2 U 123 120	K.Lz 7 3 0 100 140 1 223 216	20 66 64 21 122 124 22 64 71	14 2,5 AS K.FE 11 4	K.L. A 5 0 210 218 1 120 117	10 91 90 11 76 82 12 329 331	15 A3 61 14. 44 5n A.L.: 11 A	2 143 139 3 263 270 4 139 139	10 45 14 18 47 44 19 A9 71	4 174 1'A	1 1 12 1 1 12 2 1 22	4 /44 /39 7- 30 41 4 134 134
12 111 112 13 109 105 14 127 141	K-1 = 1 1 A 6L ⁹ A40 1 2:3 243 7 98 65	> 130 125 6 113 105 7 06 71 8 147 147	18 pd 62 19 116 119 21 63 76	2 R7 R3 3+ 44 35 4 12 ⁹ 132 K-1= 0 3	2 232 230 3 111 105 4 214 211	K·L: 3 4 0 133 119 1 5 ⁰ 0 36 ⁷ 2 443 444	1 02 03 74 40 52 8 04 307 4 344 309	4 130 134 5 120 13 6 120 13 6 120 13	15 77 78 14 135 137 10 00 01 18 130 141	0 110 114 1 136 136 2 92 94 3 79 75	5 108 101 5 113 112 7 149 144 5 256 247	K.Lz 3 8 0 306 313 1 203 209 2 160 150	5 141 176 44 15 41 5 111 116	4 04 101 5 1/3 1/0 7 115 145	43 ¥3 89 14 114 116 15 77 72
17 189 184 18 141 172 195 20 16	1 505 A11 4 317 275 5 214 269	9 85 82 10 177 178 11 137 140	R,1 = 4 2 0 68 53 1 151 162 2 177 168	1 117 149 2 412 456 3 612 561 4 130 128	7 187 A9 A 145 191 9 136 176	3 423 422 4 207 192 5 217 200	5 1.17 110 6 /6 /4 9 1.5 184 9 64 59	7 114 151 8 104 9 01 86	20 20 23 22 102 20	5 123 124 5 48 54 7* 48 54 5* 45 40	9 123 125 10 107 112 11. 46 48	3 170 170 4 262 264 5 164 163 6 158 158	2 211 200 2 1.4 344 4 22 11	A 17 43 9 17 17 10 19 54 11 15 58	30 1/0 1/0 *.L1 6 10 0 109 105
8.5 4 0 0 190 145 1 91 44	7 3 4 379 8 67 36 9 614 492	13 59 57 14 141 138 15 108 106	3 204 204 4 139 150 5. 19 20	5. 37 29 6 75 15 7 151 175	11 A6 A5 12 55 49 13 75 70	7 403 390 A 274 25A 9 152 152	11 × 70 1. 1. 1 42 1. 1. 7 110	1) 12, 07 13 8, 73	0 431 437 1 213 209 2 259 255	V+ 52 49 10 53 50 11 1^C 162	11 0A 08 14 98 105 15 96 98	7 255 251 8 255 255 9 99 86 10 247 248	12 2-1 160 11 1-2 142 15 148 1-7	17 61 /3	1° 44 40 2 1'3 187 3 94 95 4 71 76
2 388 304 3 45 40 4 247 245 7 188 120	14 3.7 375 17 129 135 13 179 178	16 212 207 17* 28 33 18 60 60	6 164 164 7 205 20n 8 308 3 ^H 4 90 34 44	9 606 601 11 127 114 1/ 127 115	14+ 40 37 14+ 40 37 15 89 97 16 70 74	10 321 331 11 72 76 12 152 155 13 147 130	110	· A · 4 · 34 · 7 · 6 · A 2 · 7 · 6 · A 2	4 3'0 354 5 259 235 9 175 14/	U 70 64 1• 14 33	17 59 57 18 61 62 19 126 126	11 96 91 12 744 237 11 44 44	20 - 12 - 14 20 - 12 - 14	7 /0	5 177 170 6 140 154 7 146 160 8 217 225
9 AN 74 10 244 241 11+ 42 41 12 243 2-8	*5 113 114 *6 206 205 *7* 53 50 *0 147 144	1. 40 40 2. 41 42 3 135 133 4 134 128	10 177 178	13 288 289 15 74 77 16 124 131 17 173 180	17 93 103 18 40 70 19 67 47 K.L. 8 3	14 197 201 15 87 90 16 131 138 18 140 145	4 3,7 86 5 3,5 68 6 1,6 177	20 6 61	8 110 100 9 209 245 16 262 257	4 A5 83 7* 50 42 6 192 188	1 217 222 2 73 72 3 155 152	15+ 37 37 16 114 118 17+ 50 51	2 4 9 4 5 3 7 7 7 4	- 14 /A	9 217 210 10 1 ⁴ 1 182 11 97 65
13 160 141 14 78 73 15 111 123	20 134 13A 21+ 45 45 22+ 42 56 23 0 86	5 jun 97 5 01 54 7 185 184	14 8- 83 15 61 61 10 63 65	18 187 185 19. 48 85 77. 42 37	0. 34 32	19 132 130 20 70 75 22 55 62	7 (A 78 9 78 76 0 76 77	1 250 257 2 139 134 3 180 184 4 113 110	11 237 233 17 215 217 13 242 241 14 95 55	/ A0 63 A-LE 0 7 3 A5 81 7 350 355	4 117 108 5 108 106 6 122 122 7 35 17	18 64 65 19 97 98 20 96 101 #.Lz 4 8	2 yes yes 50 x0 5 y 7 87 57 7 1 4 1 8	11 1/4 1/2 17 0A 1/2	13 111 112 14 132 137 16 134 129
20 11 ⁹ 174	K.I.= 2 1 0 143 167 1 576 581	9 12A 128 10 94 92 11 158 148	18 58 56 •9 133 132 20• 28 79	1 214 210 2 265 298 3 65 45	4 108 106 5 73 66 6 174 163	0 115 113 1 320 315 2 409 407	n 24 4,2 kg Kuto (14	6 04 01 A An 70 7. 20 12	15 7: 80 15 135 131 17 165 163	3 105 110 4 148 101 5 87 96	9. 40 45 10 70 70 11 114 122 12 71 72	n 94 107 1 214 21A 2 309 3ng 3 304 29A	H 244 240 U 464 344 4. 144 141	14 0 50 14 0 53 K.I.E 52 0 6 45 52	17 77 69 18 46 94 P-LF 7 10 0+ 44 45
4 14 1.3 4 4 4 4 1 4 7 7 1 40	3 259 267 4 131 125 5 24° 244	12 137 130 14 86 87 16* 46 49 1/ 57 47	K+L= 5 2 n 14n 124 1 245 203	4 103 179 5 102 118 6 297 246 7 402 348	8 50 A2 0 06 87 10 88 89	4 279 269 5 323 315 6 184 198	3 4/2 448 4 5/2 510 5 214 216	0 00 00 10 81 85 11 11/ 101	10 01 MR 25 01 60	/ 132 111 8 317 318 9 259 262	13 130 127 14 155 108 15 100 108	4 94 104 5 107 105 6 199 144	1, 1, 3, 3, 7	1,7 00 7 /A M7 3, 0 40	1 117 114 2 107 100 3 113 118 4 140 138
A 544 545 74 17 44 F 113 110	A 144 77 7 121 123 A 144 136 9 315 312	1 176 163 2 128 131 3 175 177	2 2/2 241 4 183 179 5 230 210 6 199 202	8 111 140 9 340 349 10• 45 60 11 170 158	11 140 148 12 167 114 13 154 151 14 145 147	A 190 197 9 132 137 10 226 270	7. 19 44 A 117 309 D 15 44	· 1 12/ 125 · 4 45 41 · 4 123 122	1 640 651 2 140 150 3 214 146	11 175 171 12 78 75 13 104 197	18 58 47 K.L= 8 7 0 147 145	A 159 159 9 77 83 10 98 94	17 - 46 - 44	5 117 174 A 147 143 7 19 74	211 206 53 56 7 100 171
0 170 151 11 244 240 12 114 111	1 05 7J	4* 45 50 5 66 64 6 134 128 7 90 86	7 126 115 A 311 207 9 93 94	12 137 128 13 226 220 14 165 169 15 93 95	14+ 48 41 18 140 180 K.L: 9 3	10 07 164 10 100 111 11 63 60	11 314 298 11+ 38 34 12 1-2 100 13 157 150	10 121 24 17 55 50 18 71 70 10 03 95	4 107 2117 5 474 402 6 21: 210 7- 34 23	15+ 47 43 1+ 75 69 2u 48 70 22 138 140	2 158 154 3 96 99 4 104 109	17 130 125 17 164 169 14 65 64	21 01 (7 # 11 2 2 9 F 141 317	0 52 56 11 145 148 11 141 144	9 162 163 10 77 54 11 93 97
10 128 127 17 07 44 18 00 44	4 203 244 17 14: 186 18 11: 135	8 57 53 9 87 84 10 127 124	11 168 167 12 161 161 15 98 96	1A 156 1A7 17 233 231 18 80 80	1 109 111 2 181 179 3. 51 50	1/1 14 14 1/1 14 16 180 47 87	15 4.4 40 17 67 92 18 68 72	7 145 -5V 7 145 -5V 7 44 34	A H2 NA 0 21: 225 11 230 245	6 01 100 1 116 99	5 148 148 6 64 71 7 116 115	15 66 67 16• 29 31 17 75 75	1 241 248 24 41 42 3 2 4 2 2 4 2 2 2 5	17 57 68 K.I.T. 12 9 1. 46 49 2.1.14 144	15 103 101 10 7 04
20 42 43 21 122 117 K.1 t 6 0	21 14 111 22 4 7 23 47 6	14 /1 /4 15 00 02	18 153 155 19 56 54 20 85 85	21 152 140 23• 49 41 K·L= 2 3	5 110 113 6 151 159 7 70 77	21 141 143 22 28 25 K-I = 5 4	24 /5 84 24 67 94 27 81 83	2 246 274 7 00 00 4 112 113 5 145 151	12 174 144 11 40 83 14 41 44	3 118 111 4 228 214 7 216 212	9 169 167 10 56 55 11 120 112	19 130 126 20 104 105 K.L. 5 A	6 107 214 A 14A 144 7 114 113 2 144 117	7 84 86 8 85 81 8 85 81	1 NA NO 1 NA NO 2 JU N5 3 JO NJ
1 44 1-1 1 41 43 2 184 1-1 2 474 4-2	n /14 691 1 344 (5) 2 354 727	1 11º 121 3 114 111 4 08 05 7 110 114	U 297 305 1 288 278 2 184 184	0 333 354 1 282 316 2 364 345 3 159 168	0 70 86 10 171 118 11 168 166	1 540 541 2 244 247 3 400 544	175 128 2 5 6 498 3 130 128	A 64 40 H 55 4A 9 178 127	17 195 204 20 87 84 21 18: 188	/ 158 152 n= 48 62 = 48 41	14 94 94 17 /7 74 K-LE 9 7	1 326 326 2 110 10A 3 176 183	6 107 141 10 156 1.7 11 144 1:0	1 A4 70 2 134 181 4.12 9 16	4 171 172 5 114 113 7 10 73
4 113 110 54 44 - 13 6 104 110 7 764 250	3 37: 296 4 267 243 5 263 245 6 374 137	/ 102 10A 8 142 145 9 105 107	3 115 120 40 44 30 5 217 232 6 9/ 103	4 450 417 5 390 391 6 2 ⁷ 2 263 7 374 337	17 126 131 13 82 78 14 97 102 15 64 63	4 300 200 5 100 107 5 154 154 7 1-6 10	4 230 226 5 167 144 6 120 133 7 164 135	**************************************	3 348 343 1 213 203 2 330 324	10° 34 30 11 158 150 17 198 189 15 78 79	2 100 101	5 175 172 6 154 152 7 212 214	12 124 2.1	1 418 402 2 111 101 3 334 554	0 A7 64 10 141 121 21 120 114
4 4A7 4A4 11 158 15A 12 1/0 1/4	7 15/ 57 A 82 00 9 184 173	10 20 29 11 60 65 13 49 40	/ 55 53 9 214 214 11 183 185 12 113 112	N 57 92 9 240 244 10 84 74	16 84 86 K-L= 10 3 0 63 67 1 139 132	# 104 100 v 164 149 10 169 160 11 160 141	A 201 241 9 14.0 165 10 2 4 230 11 255 258	140 4A 44 14 A A4 170 51 46	3 220 227 4 231 220 5 1M8 191 5 123 115	14 124 123 15 89 84 16 118 124 17 107 119	6 144 148 7 68 74 8 72 73	0 76 78 10 111 117 11 184 169	17 04 104 17 04 104 164 30 14 21 80 87	5 156 152 6 15, 145 7 231 232	14+ 36 37 14+ 36 37 15 112 113
13 27A 272 14 145 121 15 10 20	1 160 141 2 41 63 11 41 75	0 0 74 1 19 59 3 05 00	13 136 135 14 140 144 15 74 60	12 89 A8 14 135 133 15 96 102	2 00 00 3 07 08 5 118 115	12 PA 77	12 210 220	n 140 (52 1 70 76 24 42 53	7 65 68 6 117 113 9 226 228	10 110 215 194 41 44 21 85 78	0 A0 A0 10 111 111 11 58 A7 12 80 80	14. 49 5n 15 189 190 15 78 80 17 91 98	21 1-7 1-2 E-1 2 4 f 31n 3-1 1 248 2-4	n 126 - 22 - 2 13/ 143	**L* 9 10 0 212 212 1 72 52 2 1#3 182
17 3/0 100 18 /5 71 27 /1 04	16 165 145 16 165 145 17 16 16	0 55 55 173 126 0 57 80	10 110 111 1/ 06 08 10 08 101 10 50 60	17 05 01 10 53 56 20 85 47	7. 44 52 10 172 149 11 98 91	14 78 1 17 141 1-1 19 120 1-1	140 55 17 /0 74 18 1,4 177	4 03 87 5 110 197 7- 44 47	11 73 63 12• 3'' 40 13 23# 241	N.L. 2 7 U 5n1 484 1 215 213	14 111 117 15• 34 26 13 124 125	14. 34 45 19 154 153 20 77 74	. 1.3 1-0 1 112 3 10 4 115 102	10 45 67 10 45 87 17 107 104 18 49 55	5 115 114 4 134 143 6 84 83 7 74 81
1 1 4 100	16 AV A0 10 140 136 20 145 145 27 115 275	*.L= 13 1 1* 51 44 2* 36 37	20 77 82 NIE 7 2 0 215 235 1 107 107	21 104 111 22 99 98 8.L= 5 3 0 480 451	14 46 46 15 79 78 K.L. 11 3	21 A4 /n x.1 = 6 4 n 216 2-7	27 - 16 - 52 K.1 5 - 5 N 323 - 346	9 88 97 10 45 68 12 97 /1	15 110 114 16, 57 48 17 164 164	3 427 430 4 245 240 7 779 789	0 62 70 1 44 62 3 61 65	0 348 344 1 134 132 2 107 105	A 121 115 / 114 111 A 74 / 1	10 215 210 20 70 43	8 13A 136 9. 30 32 11 54 85
4 14" 172 5 11 1 102 6 11 5 107 7 217 20*	n : :-1 1 : :-1 2 :55 :	4 82 84 N.L: 0 2 U 252 201 1 222 243	2 140 170 3 246 237 4 68 64	1 048 686 2 215 196 3 520 548 4 3 ⁷ 0 3 ⁶⁰	n= 51 40 1 162 159 2 153 150 3 148 146	1 214 2.7 2 167 143 1 215 211 4 116 117	7 2:5 225 7 4:4 453 4 1:7 1/2 5 1 P 1/1	14 99 101 16 137 156	19 55 51 21 4, 78 8415 6 4	0 163 1/1 / 386 390 n 184 137 v 225 228	6 76 77 7 62 61 8 148 142	4 85 93 5 94 98 6 33 31	17 1 0 1 7	2 /R4 /A1 3 147 00	A.L. 10 10 0 84 1 105 107
A 20 00 0 16 90 10 2 5 245	3 23, 207 4 11, 105 5 110, 96	2 470 496 3 317 303 4 161 411	A2 69 7 323 321 8 110 114	5 370 344 6 131 123 7 177 145	5* 48 46 6 160 157 7 125 124	5- 40 5A - 1-7 355 7 205 164	A 171 143 7 5.5 464 8 217 146 9 212 247	U 58 67 1 70 73 2 53 54 3 207 209	1 140 144 2 42 44 3 21 213	10 143 134 11 175 169 17 140 136 13 212 209	9. 41 43 10 96 88 11 70 68 12 86 83	7 198 205 8 125 128 9 140 130	12 10 14 15 14 3 0 17 14 1 10	+ 11+ 113 + 144 157 7 10: 105	2 118 127 4 h2 88 5 106 107 6 67 61
17 78 /5 16 (3 A1 17 (5 41	7 /01 /11 1 144 :50 16 110 113	0 383 284 / 164 162 8 218 192	10+ 45 54 11 2+6 268 12 43 71	9 243 245 10 146 150 11 141 140	10 100 103 11 55 67 K.L. 12	0 247 2/1 10 44 46 11 1-1 3-1	10 120 100 11 13 80 17 00 08	4 104 190 5 140 135 6. 40 43	4 100 287 5 131 148 2.12 7 8	14 A4 B2 17 104 101 17* 52 55	K.LI 11 7 0 277 777 1 165 171 3 115 111	11 138 131 12 138 130 14- 40 40	2(P3 H +.1 = 4 0 	A 247 240 V 145 - 37 - 1 245 244	9 58 62 10 82 84 11 78 74
K, I y H N 1 4/ 0 474 5 6 30 ⁹	120 5, 64 130 5, 64 11 80 84 14 225 215	10+ 44 35 11 73 78 12 Vn 08	14 142 143 15 266 200 16 88 90	13 (14 219 K.L. 4 3 14 81 49	2 123 126 3 89 47 5 52 54	14 84 51 15 111 112	15 1.7 137	8 254 260 10 75 64 11 109 105	7 134 138 8 208 214	1v 140 153 20 54 51 21 72 69	4 127 130 5 112 115 6 60 60	16 110 110 17 64 63 18 55 50	241 217	12 185 158 13 91 91 14 4 53	0 70 88 2 79 76 3 111 114
4 200 2/8 5 2/4 2/7	** 35 45 ** 35 45 *7 1*4 15 *8 65 73	13- 46 37 14 21# 220 15 13# 134 10 134 174	1/- 16 25 19 01 94 K,L: A 7 U 07 90	K.L= 3 3 15 168 164 16 136 130 17 146 140	7 140 130 A. 30 3A 9 104 103	17. 42 10 17. 42 10 10 110 117 20 77 74	19 211 296 21 -3 75	13 /7 81 14 V/ 88 A-L= 11 5	174 41 38 11 104 104 12 175 121	41 4.L. 3 7 0 294 305 1 430 449	A 70 7A 9 74 7A 10 77 76	0 115 118 1. 31 12	1 1 1 1 1 1 1.0 200 8 241 241	18 114 123 10 9, 9] 20 64 67	7 77 7. 7 77 7.
A. 44 49 7 167 142 9 3 1 195 11 2 4 2/1	-U AV 01 21 AV 83 K.IE 5 1	17 82. 30 18 189 262 19 130 114 21 91 97	1 37/ 32A 2 12H 127 3 /4/ 744 4 125 12A	1A 102 108 19 104 106 20 99 111 21 150 145	K.L 2 13 K 1 03 70 2. 44 45	# +1 = 7 4 0 100 1/4 1 114 101 2 1-7 1-	27 .A /2 	0 134 135 1 98 99 2 55 55 3 70 77	13 120 12A 14 113 114 15 67 67	2 331 328 3 355 350 4 207 201 5 308 387	1 121 127 2 128 125	2 127 144 3 115 110 4 220 222 5 139 145	2 2.7 774 11 115 114 11 115 114	1 30 33 1 30 34 2 1 ⁶ 1 ⁷	5 147 139 4 65 78
17 UN 47 15 2/0 2/4 14 A 74	1 214 -21	2 54 54 2 54 639 1 232 24	7 120 127 7 202 194 143 147	22 00 73 K-1 4 3 0• 44 01	0 269 279	3 01 03 4 134 142 5 130 114	1 2-2 240 4 216 216 5 215 237	4 1/1 1/0 / 111 11/ 11 00 03	18 124 170 19 117 108 Kalt 7 A 0 295 200	0 117 116 / 196 194 0 240 247 9 147 150	3. 49 50 5 120 123 6 78 87 K,Lz n 8	6 167 144 7 216 215 8 110 109 9 157 110	14 104 141 14 104 141 14 41 44	3 217 204 4 40 75 60 5, 41 6 161 173	> 200 204 > 212 207 2 100 191 8 121 150
7 3 1 311	4 (1, 1)/ 4 4 31 7 14 36	2 453 778 34 44 77 4 203 274	11° 16 36 12 11 125 13 149 130	2 165 160 3 348 31/ 4 125 127	4 81 94 5 714 658 6 401 384	A A PO 10 215 200 11 75 Pt	7 7.4 200 R 1/4 1/2 Q 103 194	1 141 134 2 67 67 3• 37 33	1 01 01 2 247 241 3 115 1/0	14 212 224 13 290 285 12 80 75	n 56 54 1 144 151 2 91 188	14 134 135 11 54 55 12 199 197	14 1/1 jn1 19 /1 09 Kila 5 9	7- 34 31 0 174 172 11 247 /32	9 178 183 10 174 167 31 478 420

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Table 1 (cont.)

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4 FU FC	H FO FC	H FO FC 2 91 87	H FD FC	H FO FG 9• 45 45	H FO FC K.Lz 5 17	H FO FC
3 166 167 3 111 112	11 117 116	3 01 03 4 79 A5 5 03 05	A0 44 4A / 78 78	11 170 1AB 14 03 08	1 120 124	2 55 54 5 76 74 7 114 109
1/ 104 111	0 84 78	6 AŬ A5 7 90 91	4 54 AG	K.L. 1 16 0 44 93	3 100 103 • 39 27	9 60 65 K.L.# 1 20
0 72 M1 1 114 106	2. 47 48 3 113 123	A 47 46	K- # 9 14 0 9.5 -4	1 220 221 2 145 147	2. 45 44	1 92 94
3 46 100 4 01 63	7 134 130 8 76 78	11 97 98 12 69 AR	4 1.4 1.4	4 11/ 11/ 6 114 113	10 57 57 11 A0 66	7 113 110
0 A0 64 7 06 99	9. 52 57 16 110 108	8.L. 9 13 8 96 100	1 1 1 1	7 107 107 8 72 81	1 101 VO	N- 3/ 37 K.Lz 2 24
K+L= 11 11 1 54 60	K.L= 10 12 2 190 190	3 06 0A 4* 51 5A	K-1 = 10 14	10 141 144 12 05 76	3 200 213 0. 42 30	2+ 42 37 5 80 M1
2 08 99 4 n3 86	3 66 65	5 144 146	1	13 /3 70 14 64 58	/ 13/ 134 8 134 137	7 55 54
u 423 420	A 70 72 K.L. 11 12	A 67 75 9 121 123	3 + 20 - 40 4 - 7 - 5	1 109 101	K.L.S 7 17 U. 31 10	2 131 133
34 19 3• 30 30	1. 32 41 2. 42 48	10 64 70 K.L= 10 13	4 12 FR	2 47 94 3 42 94	1 25 95	5 64 65
5 40 9A 6• 40 30	1 235 230 3 130 126	1 71 75	* 127 122	5 V0 K0	7. 44 44 A.L. A 17	7 109 107 K.L = 4 20
5 102 10A	4 222 220 5. 45 55 6 143 151	3 83 89 4 76 48	12 129 121	0 125 177	1 94 98 R.LT 0 18 U 72 66	3 74 /2 6 126 126
12 73 A1 15* 40 47	7. 48 47 8 234 242	0 83 89 1 305 295	14+ 57 63	10+ 37 44 11 153 148	1 124 114	K-L# 5 20 0 94 88
19+ 50 56 10 169 178	10 255 248	2 243 234 3 258 247 5 245 235	1 2:5 217	13 47 44 15 108 108	5 137 130 6 62 66	2. 45 55
10 55 46	12 91 98	7 178 176	3 141 175	0 110 110	7 95 94 9 147 147	K.L= 0 21 3 87 93 4 84 78
2 214 211 3 173 174	14 49 52	14 104 110 15 83 91	A 277 274 9 174 123	2 100 96	11 75 77 K-1 1 16	K.L. 1 31
4 214 235	17 130 141 18+ 52 52 K.L = 1 13	14 99 99	14 104 185	4 64 7ŋ 5 01 61	0 112 100 1 68 101 20 52 55	K-L+ 2 21
9 196 202 9 111 10A	0 156 147 1 84 85	*.L. 1 14 51 57	14 /1 07	9 h2 h6 10 73 73	3 107 102	4 86 82 4 75 76
11 107 103	3 185 176	1 118 119	14 67 59	12 11/ 120	9 81 79 13 75 47	A 77 77
13 105 10A 14 72 70	5 162 163 6 228 224	4 143 148 5 163 105	2 2 203	0 103 105	1 74 98	1 70 74 5 07 04
1/ 104 105	7 250 254 8 107 104 9 66 61	6 195 184 7 135 136 9 164 177	5 156 111	2 200 147	3 78 40	n 149 147 1 72 49
19 86 90 8.Ls 2 12	10 174 175	10 77 63 11 187 188	A A7 A7	92 93 5 106 171	5 59 62 7 110 1º2	2• 49 50 3 78 74
1 175 122	13 A3 01 14 A3 A8	13 67 63	10 130 138	7 8 ₂ 87 8 85 91	11 73 74	
3 279 283 4 71 69 7 198 194	14 75 74	14 56 55 K-LE 2 14	14 57 49	9 167 166 10 69 62	K.L. 3 18 0 171 170	
42 40 9 213 223	1 73 73	1 147 150 3 97 101	14 1.2 106 8.4 5 3 15	13 A5 A3 14 67 A2	4 133 126 6 93 91	
11 131 134 14 103 104	2 300 307 3 72 A9 4 64 55	5 132 132	1 1/2 173	0 67 43	7 95 98 9 115 116 10 75 66	
13 2A7 2AA 13 A1 71	117 112	7 84 87 8 65 68	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	2 71 73 3 83 81	11 74 76 12 82 84	
10. 40 47 Mile 3 12	0 A0 02 10 133 178	10 124 131 11 118 117	\$ 39 98	6 133 131 7 A/ A7	0 119 120	
1 102 100 2 A7 7A 3 1A4 180	11 75 A1 13• 47 4A 14 144 147	12 62 62 13 73 77 15 131 130	8 75 76 0 47 93	8+ 34 29	2 50 6 4 60 64 5 136 135	
100 00	16 R0 00	19 80 76	12 00 00	12+ 39 35 13 84 82	9 98 AS	
/ 114 113 6 A7 6A	18 125 132 K-L 3 13	R.LE 3 14 0+ 33 34 1 146 145	15 45 48 15 79 81	X.L= 6 14 0 158 150 2 74 75	0 124 129 10 55 46	
0 00 00 10 04 06 11° 47 49	0 224 231 1• 39 41 2 84 81	2 200 252 3 200 192	10 34	4 105 1A4	11 78 85	
15 AV 69 14 78 75	3 11° 119 200 210	5 184 180	3 114 112	7 NS 87 8 131 128	2 114 111	
18+ 28 25 K,La 4 17	9 22 A3	7 65 77	5 nA /3 4 r.4 pB 7 114 104	90 50 51 10. 34 34	3 05 09 4 75 77 6 109 110	
U 468 461 1 104 103	8 76 A9	10 82 8A 11 180 179	0 1/1 1/0	0 116 113	8 A9 71 K-L3 A 48	
3 130 121 4 179 175	11 103 100 17 170 170	13 03 62	13 120 107	3 72 74	4 124 125 7 91 84	
5 98 100 5 59 64 7 150 154	13 10 103 16 111 103 13 76 303	15 106 105 K.L. 4 14	K.14 5 15 N A5 RA 1 OV 30	5 70 47	K.L.2 / 18 0 A8 00	
8 61 A1 9 85 79	1 223 215	2 103 111	2 105 102 3* 40 4A	10 134 134	2 59 58	
11 133 128 12 168 148	2. 52 48	4 75 81 5 84 87	6 54 58 7- 40 43	1 21 24	K.L. 0 19 2 49 47	
14 07 07 15 110 111	4 A3 A4 5 137 134	6 72 78 7• 44 36 8• 36 40	A 1º1 117 9 110 117	4 8/ 84	3+ 43 14	
10 177 179	A+ 4A 40 0+ 52 A3	10 91 89 12 109 111	1 11 112	0 125 124	8 04 05 10 109 108	
1 154 101 3 67 70	11 178 181 12 71 70	15 62 64	0. 28 28	2 71 75	0 109 104 1• 33 #5	
5 125 120 5 00 52	13 01 04 14 A2 50 15 1A1 1A8	n 155 127	1 154 155	6 257 2A1 70 31 21	2 AB A4 3 A4 77	
7 112 112	14 124 123 K.L. 5 13	3 145 146	4 14A -46	17. 40 42	5 52 54 6 50 43	
11 H2 78 17 136 141	2 104 113	54 34 33 5 50 57	7 88 62 8 79 82	2 165 174	10 100 150 10 108 109	
KiL 30 12	4 71 73 5• 40 41 6 62 69	A 113 11A 10 110 113	100 52 53	4 05 04 7. 45 40	N.L. 2 10 N 145 134	
1 77 73	7 171 173	11. 45 48 12 83 81	7 17	11 73 74	2 124 120 3. 48 40	
4. 41 38 5 96 47	11 184 180 17+ 35 26	14 81 85 15- 43 38	3. 121 193	14 07 01 K,1 2 17	4 A2 A3 50 51 43	
7 75 86 8 89 44 9. 35 42	13 114 120	f 1 10 1 14	1 112 · 13 4 7n 78 5 97 109	1 114 123	7 126 129	
11 02 02 11- 39 43	0 95 9A 1 171 179	1 101 101	\$ 12.5 105 7. 5a 47	3 110 107 4 54 55 5 A9 67	10+26 30 K-L: 3 19	
13 117 117	1 131 115	A 1/0 1/4	1. 52 . 54	/ 140 140 0 107 112 0 27 74	0+ 18 44 1 175 1+7 2 72 75	
1 117 110	5 10 ⁰ 10 ⁰ 6 81 84 7-38 39	A 133 1 3	A.L. 8 13	11 A0 A0 12 A1 70	3 07 01	
2 95 VB 3 84 A7	9 102 108 10 91 100	11 01 10	3 1 10	8 227 217 1 146 154	6 55 56 7. 32 29	
5 106 102	12+ 47 51		A 27 22 7 14: 144	4 183 175 4 81 89	A A4 A7	
7 130 131 A 98 101	14 88 98 15 82 80	1 1 1 1 1 1	. 1.4 113	6 73 79 / 84 79	2 304 109	
10 239 247	0 94 A4 1 172 145	4 44 47	1 2 7	8 91 90 10 78 74	3 R2 R2 5 74 75 6 91 R	
12 126 117 11 75 75 14 140 112	2 184 185 3 177 177	7 117 148 # 15 #m	20 10 41 10 41 47	13+ 31 28 Kale 4 17	7 130 133 9 121 124	
15. 42 27 K.L. A 12	6 124 133	1 43 50	5 AN 45	1* 11 54 2 184 187 3 AV A	1 A1 AA 7 1m7 97	
1 121 120	9 118 121 9 118 121 10 127 132	130 - A 41 512 - A 14	1 141 142 3 AL 54	4 A2 57 5 43 34	3 A5 A2 4 A8 7	
3 68 73 4 170 180 5 51 54	12+ 4A 45 13+ 17 27	8 · 44 43	t 12 15	/ 04 07 0* 3* 33	K.L. A 10 3. 47 A0	
7 71 74	n 134 135 1 141 135		/ [n4 185 8 81 85	V A1 78 10 170 176 11 67 65	4 /8 /9 K.L* 0 /h 9 56 41	

intensities were corrected for Lorentz and polarization effects. No corrections for absorption and extinction were made.

Solution and refinement of the structure

The positions of the cobalt and calcium atoms were deduced from the Patterson maps. These coordinates were used to calculate the structure factors. The Rvalue was 0.35. Three-dimensional electron density maps were then calculated with all the terms, the phases of which were calculated on the basis of the cobalt and calcium atoms. The maps revealed the positions of all the remaining non-hydrogen atoms of the complex ions. The atomic positions and isotropic temperature factors were refined by the leastsquares method with the block-diagonal program HBLS-4 written by Dr Ashida. A weighting scheme, w = 0.2 for $|F_a| \le 15$ and w = 1 for all other F_a 's was employed. The scattering factor for Co, Ca, O, N, C and H atoms were taken from International Tables for X-ray Crystallography (1962). After several refinement cycles the structure converged with R=0.10. A difference Fourier synthesis was then calculated, which revealed the positions of 10 oxygen atoms of water molecules. After inclusion of the water oxygen atoms, further cycles of least-squares refinement were carried out with anisotropic modes. R then decreased to 0.045. A difference synthesis at this stage revealed all the positions of hydrogen atoms except those of water molecules. In this difference synthesis and electron density synthesis, there was one well defined peak with a height of about one half that of an oxygen atom. The maps were well explained if one of the 10 oxygen atoms is distributed over this new position and its original position with equal probability. This disorder of water molecules was taken into account in subsequent refinement. Further refinement cycles were carried out with anisotropic temperature factors for non-hydrogen atoms and with isotropic temperature factors for hydrogen atoms. The final R value was 0.041 for all the 3412 observed reflexions. At the final stage of the refinement all the parameter shifts of non-hydrogen atoms were



Fig. 2. A perspective drawing of cis(N)-trans (O_5) -[Co(L-asp)₂]⁻ and the numbering scheme of the atoms. The ellipsoids show the thermal motions of the atoms with a probability of 50%.

well within the corresponding standard deviations. Table 1 gives the observed and calculated structure factors. The atomic parameters and their standard deviations are listed in Table 2. observed and calculated differences shows that the complex ions have the absolute configurations illustrated in Figs. 2 and 3.

Determination of the absolute configuration

Equi-inclination Weissenberg photographs of the first layer around the *b* axis were taken using Cu $K\alpha$ radiation. Differences between intensities of some reflexions and those of their counter-reflexions were clearly discernible, as shown in Table 3. Comparison of the Description of the structure and discussion

The crystal is essentially ionic and is built up of cis(N)trans (O_5) - $[Co(L-asp)_2]^-$, cis(N)-trans (O_6) - $[Co(L-asp)_2]^ Ca^{2+}$ and water molecules. Thermal ellipsoids of the complex ions, the cis(N)-trans (O_5) isomer (complex A), and cis(N)-trans (O_6) isomer (complex B) are illustrated in Figs. 2 and 3, which correctly represent the absolute

Table 2. Atomic parameters

(a) Positional and thermal parameters for the non-hydrogen atoms ($\times 10^4$), with their e.s.d.'s in parentheses.

The β_{lj} 's are defined by exp $[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})].$

	x	У	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Co(1)	2270 (1)	6100 (1)	3790 (1)	13 (1)	34 (1)	15(1)	3 (1)	- 6(1)	-2(1)
$C_0(2)$	342 (1)	3511 (1)	2077 (1)	10 (1)	23 (1)	13 ÌÌ	-1(1)	1 (Ì)	-2(1)
Ca	2242 (1)	9622 (1)	892 (1)	18 (1)	39 (1)	20(1)	2(1)	12 (1)	10 (1)
O (1)	1268 (2)	6228 (5)	3730 (3)	11 (1)	67 (5)	16(2)	11(4)	-9(3)	7 (5)
$\tilde{O}(2)$	286 (3)	5832 (6)	4420 (3)	11 (1)	85 (6)	34(2)	8 (5)	-4(3)	6 (6)
0(3)	2144(3)	8423 (5)	5641 (3)	22 (2)	58 (5)	25 (2)	- 10 (6)	-7(3)	- 24 (6)
O(4)	2321(3)	7452 (5)	4542(3)	$\frac{21}{21}$ (2)	40(4)	21(2)	-12(5)	2(3)	-12(5)
Õ(Š)	3278(3)	5866 (5)	3843(3)	15 (1)	80 (6)	$\bar{20}(\bar{2})$	-5(5)	$-\bar{8}(\bar{3})$	-10(6)
0(6)	4161 (3)	5129 (8)	3139 (3)	14(2)	178(11)	44(3)	19 (7)	-1(4)	-58(10)
O(7)	2524 (4)	8206 (6)	1876 (4)	46(3)	95 (7)	30 (3)	40 (8)	14(4)	52(7)
O(8)	2336 (3)	7391 (5)	3015 (3)	28 (2)	50 (5)	21(2)	14(5)	1(3)	15 (5)
0(0)	1027(3)	4786 (5)	1767 (3)	$\frac{20}{11}$ (1)	33 (4)	17(2)	-4(4)	6(2)	-5(4)
O(10)	1755 (3)	5209 (5)	787 (3)	19(2)	52 (5)	24(2)	- 19 (5)	17(3)	-3(5)
O(11)	-667(3)	4074 (6)	61(3)	19(2)	115 (8)	22(2)	13 (6)	-11(3)	10 (7)
O(12)	-280(2)	3942 (4)	1251(3)	12(1)	45 (4)	14(1)	10(4)	1(3)	$\frac{1}{7}(5)$
O(13)	-208(2)	4664 (4)	2695 (3)	16(2)	24 (4)	16(2)	-6(4)	6(2)	-1(4)
O(14)	-1033(3)	4634 (5)	3618 (3)	10(2) 14(1)	43(5)	$\frac{10}{28}(2)$	0(4)	14(3)	-8(5)
ous	1256 (3)	2756 (6)	4109 (3)	26(2)	66 (6)	$\frac{1}{25}(\frac{1}{2})$	-11 (6)	-24(3)	14 (6)
odió	964 (3)	3151 (5)	2915 (3)	15(1)	44(5)	17(2)	-3(4)	-1(3)	6 (5)
N(I)	2094 (4)	4892 (6)	4607 (4)	16 (2)	39 (6)	18 (2)	- 1 (6)	-8(4)	-2(6)
N(2)	2290 (4)	4825 (6)	2995 (4)	18 (2)	51 (6)	17 (2)	3 (6)	3 (4)	-10(7)
N(3)	879 (4)	2408 (6)	1427 (4)	15 (2)	28 (6)	20 (2)	- 2 (6)	2 (4)	-5 (6)
N(4)	- 315 (4)	2248 (6)	2414 (4)	12 (2)	30 (5)	14 (2)	- 9 (6)	-1(4)	-5(5)
Cùí	946 (4)	5828 (7)	4334 (5)	15 (2)	43 (7)	26 (3)	- 3 (7)	- 12 (4)	-16(8)
C(2)	1432 (4)	5347 (8)	4979 (Š)	13 (2)	41 (7)	21 (3)	1 (6)	-6(4)	1 (8)
C(3)	1597 (4)	6440 (8)	5523 (4)	20 (2)	50 (7)	16 (3)	- 7 (7)	-3(4)	-13(8)
C (4)	2051 (4)	7516 (8)	5212 (5)	14 (2)	44 (7)	22 (3)	4 (7)	-15 (4)	-15 (8)
C(5)	3539 (5)	5345 (9)	3242 (5)	14 (2)	80 (9)	26 (3)	8 (8)	-6(5)	-6 (9)
C(6)	2995 (4)	5019 (8)	2605 (5)	20 (2)	63 (8)	20 (3)	7 (8)	-3 (4)	4 (8)
C(7)	2955 (5)	6121 (9)	2034 (5)	26 (3)	74 (9)	19 (3)	19 (8)	5 (5)	6 (9)
C(8)	2575 (5)	7327 (9)	2329 (5)	22 (3)	61 (8)	23 (3)	2 (8)	-3 (5)	8 (9)
C(9)	1331 (4)	4502 (7)	1131 (5)	9 (2)	36 (6)	22 (3)	8 (6)	-4 (4)	7 (7)
C(10)	1130 (4)	3232 (7)	786 (4)	11 (2)	42 (7)	16 (2)	3 (6)	4 (4)	0 (7)
C (11)	537 (4)	3407 (9)	184 (4)	9 (2)	79 (8)	13 (2)	-10 (7)	0 (3)	-9 (8)
C(12)	- 183 (4)	3833 (8)	519 (4)	14 (2)	49 (7)	17 (2)	1 (7)	-5 (4)	7 (7)
C(13)	- 623 (4)	4089 (7)	3183 (4)	13 (2)	31 (6)	16 (3)	2 (6)	-1(4)	-10(7)
C(14)	- 540 (4)	2639 (7)	3206 (4)	18 (2)	25 (6)	15 (2)	-2(6)	. 1 (4)	-3(6)
C(15)	58 (4)	2296 (7)	3780 (4)	21 (2)	40 (7)	16 (3)	-7(7)	-2(4)	2 (8)
C(16)	800 (4)	2776 (7)	3593 (5)	20 (2)	22 (6)	24 (3)	15 (6)	-11(4)	-12(7)
W(1)	3364 (4)	9048 (7)	268 (5)	28 (2)	76 (8)	47 (3)	-2(8)	33 (5)	-24(9)
W(2)	1690 (5)	4/1 (8)	-222(5)	38 (3)	113 (9)	39 (4)	-20(10)	27(3)	-33(10)
W(3)	1845 (5)	//02 (/)	2/4 (5)	48 (3)	58 (7) 76 (6)	39 (3)	-17(8)	4 (6)	-19(0)
W(4)	2164 (4)	1059 (6)	2025 (4)	23(2)	70 (0)	20(2)	4(7)	1(4)	-7(7)
W(5)	1/44 (4)	9860 (7)	3309 (4)	32 (3) 58 (4)	97 (8)	54(5)	14(0) 22(13)	-21(8)	1(0)
W(0) W(7)*	2/80 (0) 4440 (10)	2341 (9)	30/U (0) 1202 (15)	JO (4) 47 (7)	$\frac{120(11)}{216(30)}$	34(4)	-10(26)	-21(0) -54(18)	127 (38)
W (1)* W(9)	4440 (10)	1022 (21)	1292 (13)	47 (7) 45 (4)	210 (30)	37(3)	-17(20)	-11(6)	-17(30)
W(0)	4390 (3) 4860 (10)	1424 (13)	1038 (10)	134 (9)	369 (11)	119(17)	-36(34)	122 (22)	157 (49)
W(10)	4000 (10)	101 (16)	4793 (7)	46 (10)	386 (34)	70 (10)	11 (32)	1(18)	142 (34)
W(11)*	3500 (6)	1275 (28)	2539 (8)	11(4)	571 (57)	34 (6)	-9(26)	15 (8)	-173(34)
** (11) ·	5577 (0)	1213 (20)	2007 (0)	** (*)	511 (51)	54 (0)	× (20)	(0)	1.5 (54)

* Population 0.5.

Table 2 (cont.)

(b)	Positional parameters for the hydrogen atoms ($\times 10^3$)
	Mean isotropic temperature factor of the hydrogen atoms is 5.2 Å ² .

	x	У	Z
H(1)	254 (8)	486 (16)	493 (8)
H(2)	212 (6)	401 (11)	438 (11)
H(3)	117 (8)	465 (15)	519 (8)
H(4)	116 (6)	682 (10)	562 (6)
H(5)	189 (7)	623 (14)	598 (8)
H(6)	194 (6)	491 (11)	269 (10)
H(7)	227 (7)	401 (10)	319 (6)
H(8)	307 (7)	411 (13)	245 (8)
H(9)	344 (6)	645 (12)	192 (7)
H(10)	274 (8)	594 (13)	152 (8)
H(11)	60 (9)	177 (16)	119 (10)
H(12)	124 (7)	185 (13)	170 (7)
H(13)	152 (6)	288 (10)	46 (6)
H(14)	68 (6)	415 (10)	- 23 (6)
H(15)	51 (6)	266 (13)	- 13 (7)
H(16)	- 14 (8)	155 (15)	243 (9)
H(17)	- 68 (6)	230 (10)	207 (7)
H(18)	- 96 (7)	230 (13)	343 (8)
H(19)	- 13 (6)	256 (12)	428 (7)
H(20)	4 (5)	134 (10)	376 (6)

Table 3. Detern	nination of t	he absol	lute conf	iguration
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h	k l	$ F_c(hkl) ^2$	Obs.	$ F_c(\bar{h}kl) ^2$
1	1 2	4096	>	3996
1	1 3	4844	>	2652
1	1 4	2016	<	3410
1	1 12	7327	>	6384
3	1 3	640	>	408
3	1 5	756	<	1156
3	17	790	<	1616
3	1 15	1998	<	2362
4	1 2	4396	<	6384
4	1 3	2540	>	986
6	1 Ī	1436	>	829

configurations. Two aspartic acid residues are octahedrally coordinated to a cobalt atom as a tridentate ligand through two amino nitrogen atoms and four carboxylic oxygen atoms. A five-membered, a sixmembered and a seven-membered chelate ring are formed. Two nitrogen atoms are in *cis*-positions in both of the complex ions. In the complex A, two oxygen atoms of the five-membered chelate rings are in *trans*positions and those of the six-membered chelate rings are in *cis*-positions, whereas in the complex B oxygen atoms of the five-membered chelate rings and those of the six-membered chelate rings and those of the six-membered chelate rings and those of the six-membered chelate rings are in *cis*- and *trans*positions, respectively.

The bond distances and angles within the complex ions are listed in Tables 4 and 5, together with their estimated standard deviations. The Co-N distances are 1.929 and 1.922 Å in the complex A and 1.902 and 1.901 Å in the complex B, all shorter than those observed in other cobalt(III) complexes. The Co-O distances range from 1.879 to 1.931 Å in the complex A and from 1.897 to 1.929 Å in the complex B. As can be seen from Figs. 2 and 3 and Table 4 the two Co-O distances are longer for those in *cis*-positions than for those in *trans*-positions. The deviations from 90° of N-Co-O angles in the chelate rings range from +3.9to -4.7° in the complex A and from +5.5 to -5.2° in the complex B. The coordination octahedrons are slightly distorted. For example, the Co-N(1) and Co(1)-O(8) bonds in the complex A are not collinear but make an angle of 173.1°. Likewise the angles N(2)-Co(1)-O(4) and O(1)-Co(1)-O(5) are 174.9 and 176.7° respectively. These distortions seem to be due to the non-bonded hydrogen interaction as well as to formation of the strained chelate rings. The deviations of the carbon atoms from the plane formed by cobalt, nitrogen and oxygen atoms are listed in Table 6. As can be seen from the table, the conformations of the four five-membered chelate rings in the complex ions A and B are the symmetric envelope form, and those of the six-membered chelate rings are all asymmetric skewboat.

Tabl	e 4	Interatomic	c distance.	s within	the	complex	ion
with	their	estimated	standard	deviation	ns in	parenthe	eses

Complex 4	
Complex A	
$Co(1) \cdots N(1)$	1·929 (7) Å
$C_{\tau}(1) = N(2)$	1 0 2 2 (7) 7
$Co(1) \cdots N(2)$	1.922 (7)
$C_0(1)\cdots O(1)$	1.879 (5)
	1077(5)
$Co(1) \cdots O(4)$	1.931 (5)
$C_0(1)\cdots O(5)$	1.003 (5)
	1 203 (3)
$Co(1) \cdots O(8)$	1.912 (5)
$N(1) \cdots C(2)$	1.475 (10)
	1 475 (10)
$C(1) \cdots C(2)$	1.526 (11)
$C(2) \cdots C(3)$	1.517(11)
C(2) $C(4)$	1 512 (11)
$C(3) \cdots C(4)$	1.212 (11)
$C(1) \cdots O(1)$	1.278(10)
C(1) $O(2)$	1 241 (10)
$C(1) \cdots O(2)$	1.241 (10)
$C(4) \cdots O(3)$	1.222(10)
$C(A) \dots O(A)$	1.266 (0)
C(4) = O(4)	1.200 (9)
$N(2) \cdot \cdot \cdot C(6)$	1.495 (11)
$C(5) \dots C(6)$	1.520 (12)
C(3) = C(0)	1.339 (12)
$C(6) \cdots C(7)$	1.525 (12)
$C(7) \cdots C(8)$	1.541 (13)
C(1) $C(0)$	1 541 (15)
$C(5) \cdots O(5)$	1.273 (11)
$C(5) \cdots O(6)$	1.197 (10)
	1 1 / (10)
$C(8) \cdots O(7)$	1 216 (12)
$C(8) \cdots O(8)$	1.271(10)
Complex B	
$Co(2) \cdots N(3)$	1·902 (7) A
$Co(2) \cdots N(4)$	1.901 (6)
$C_{\alpha}(2) \dots O(0)$	1.030 (5)
CO(2) = O(9)	1.929 (3)
$Co(2) \cdots O(12)$	1.897 (5)
$C_0(2) \cdots O(13)$	1.917 (5)
$O_{2}(2) = O(15)$	1 007 (5)
$CO(2) \cdots O(16)$	1.897 (3)
$N(3) \cdot \cdot \cdot C(10)$	1.485 (10)
$C(\dot{0}) \dots C(\dot{1})$	1.511 (11)
$C(10) \cdots C(11)$	1.231 (10)
$C(11) \cdots C(12)$	1.533(10)
C(0) $O(0)$	1.274 (0)
$C(9) \cdots O(9)$	1.274 (9)
$C(9) \cdots O(10)$	1.239 (9)
$C(12) \cdots O(11)$	1.220 (10)
	1 229 (10)
$C(12) \cdots O(12)$	1.284 (9)
$N(4) \cdots C(14)$	1.402 (10)
C(12) $C(14)$	1 + 72 (10)
$C(13) \cdots C(14)$	1.233 (10)
$C(14) \cdots C(15)$	1.537(11)
$C(15) \dots C(16)$	1.511 (11)
$C(13) \cdots C(10)$	1.211 (11)
$C(13) \cdots O(13)$	1.295 (9)
$C(13) \cdots O(14)$	1.217 (0)
	1 217 (3)
$C(16) \cdots O(15)$	1.235 (10)
$C(16) \cdots O(16)$	1.275(10)

Table	e 5.	Bond	angles	within	the	compl	'ex	ions	with	their
	est	imated	d stand	ard dei	viatio	ons in	par	enth	eses	

Complex A	
N(1)—Co(1)–N(2)	93·9 (2)°
N(1) - Co(1) - O(1)	85.3 (2)
N(1) - Co(1) - O(4)	89·9 (2)
N(1) - Co(1) - O(5)	92.7 (2)
N(2) - Co(1) - O(1)	91.7 (2)
N(2) - Co(1) - O(5)	85·7 (2)
N(2) - Co(1) - O(8)	89·5 (2)
O(1) - Co(1) - O(4)	92·0 (2)
O(1) - Co(1) - O(8)	88·6 (2)
O(4) - Co(1) - O(5)	90·8 (2)
O(4) - Co(1) - O(8)	87.0 (2)
O(5) - Co(1) - O(8)	93·5 (2)
Co(1)-N(1)-C(2)	104.4 (3)
Co(1) - O(1) - C(1)	113.6 (5)
O(1) - C(1) - C(2)	115.3 (7)
O(1) - C(1) - O(2)	124.4 (7)
O(2) - C(1) - C(2)	120.3 (7)
C(1) - C(2) - N(1)	106.7 (6)
C(1) - C(2) - C(3)	108.9 (4)
N(1) - C(2) - C(3)	110.2 (5)
C(2) - C(3) - C(4)	117.4 (6)
C(3) - C(4) - O(4)	120.7 (4)
C(3) - C(4) - O(3)	116.6 (6)
O(4)C(4)O(3)	122.7 (5)
C(4) - O(4) - Co(1)	129.5 (4)
Co(1)-N(2)-C(6)	104.2 (4)
Co(1) - O(5) - C(5)	113.3 (5)
O(5) - C(5) - C(6)	115.4 (7)
O(5) - C(5) - O(6)	125.1 (8)
O(6) - C(5) - C(6)	119.5 (7)
C(5) - C(6) - N(2)	106.8 (7)
C(5) - C(6) - C(7)	109.2 (5)
N(2) - C(6) - C(7)	110.7 (6)
C(6) - C(7) - C(8)	115.7 (6)
C(7) - C(8) - O(8)	121.0 (5)
C(7) - C(8) - O(7)	116.6 (7)
O(7) - C(8) - O(8)	122.4 (6)
C(8) - O(8) - Co(1)	129.9 (4)



Fig. 3. A perspective drawing of cis(N)-trans (O_6) -[Co(L-asp)₂]⁻ and numbering scheme of atoms. The ellipsoids show the thermal motions of the atoms with a probability of 50 %.

1000 J (0000.	Tal	ble	5 (cont.
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Complex B	
N(3) - Co(2) - N(4)	95.5 (2)
N(3) - Co(2) - O(9)	84.8(2)
N(3) - Co(2) - O(12)	91.3(2)
N(3) - Co(2) - O(16)	90.4(2)
$N(4) - C_0(2) - O(12)$	90.1(2)
$N(4) - C_0(2) - O(13)$	85.6 (2)
N(4) - Co(2) - O(16)	91.2(2)
O(9) - Co(2) - O(12)	91.8(2)
O(9) - Co(2) - O(13)	94.1(2)
O(9) - Co(2) - O(16)	86.8 (2)
O(12) - Co(2) - O(13)	86.6 (2)
O(13) - Co(2) - O(16)	91.6 (2)
Co(2) - N(3) - C(10)	104.6 (3)
Co(2) - O(9) - C(9)	111.9 (4)
O(9) - C(9) - C(10)	115·9 (4)
O(9) - C(9) - O(10)	124.1 (4)
O(10)-C(9)-C(10)	120.0 (6)
C(9) - C(10) - N(3)	107.4 (5)
C(9) - C(10) - C(11)	110.1 (4)
N(3) - C(10) - C(11)	110.5 (5)
C(10)-C(11)-C(12)	114.4 (6)
C(11)-C(12)-O(12)	121.5 (6)
C(11)-C(12)-O(11)	117.5 (6)
O(11)-C(12)-O(12)	121.0 (7)
C(12) - O(12) - Co(2)	129.5 (4)
Co(2)-N(4)-C(14)	105.8 (3)
Co(2) - O(13) - C(13)	112.9 (3)
O(13)-C(13)-C(14)	114.9 (5)
O(13)-C(13)-O(14)	124.1 (3)
O(14) - C(13) - C(14)	121.0 (5)
C(13) - C(14) - N(4)	106.1 (4)
C(13) - C(14) - C(15)	108.9 (5)
N(4) - C(14) - C(15)	108.9 (6)
C(14) - C(15) - C(16)	116.8 (6)
C(15) - C(16) - O(16)	121.4 (7)
C(15) - C(16) - O(15)	118.2 (7)
O(15) - O(16) - O(16)	120.4 (7)
C(16) = O(16) = Co(2)	128.2 (5)

The structure projected along the b axis is shown in Fig. 4. The important molecular contacts are listed in Table 7. A complex B and the one related by a twofold screw axis parallel to the b axis are held together by two N-H···O hydrogen bonds formed between amino nitrogen atoms and carboxylic oxygen atoms of the five-membered chelate rings. A complex A is linked to two complexes B that are related to each other by a twofold screw axis parallel to the b axis by four N-H···O hydrogen bonds; two of them are formed between carboxylic oxygen atoms in the six-membered chelate rings of the complex B and amino nitrogen atoms of the complex A, and the other two are between the amino nitrogen atoms of the complex B and oxygen atoms in the five-membered chelate rings of the complex A. By these hydrogen bonds, the complex ions form a left-handed helix parallel to the b axis, as shown in Fig. 5. These helices are held together by electrostatic forces between calcium ions and the carboxylic oxygen atoms not used to form hydrogen bonds. A calcium ion is coordinated to three carboxylic oxygen atoms and four oxygen atoms of the water molecules. The coordination polyhedron is a distorted pentagonal bipyramid. The rest of the water of crystallization molecules are arranged in a channel formed by four



Fig.4. A projection of the structure along the b axis.



Fig. 5. A projection of the structure along the a axis, showing a helix formed by the complex ions.

Table 6. Deviations of the carbon atoms from the plane formed by N, Co and O atoms Five-membered chelate rings

								
	Plane determined by:				Distances of the atoms from the planes in Å:			
	Co(1) Co(1) Co(2) Co(2)	N(1) N(2) N(3) N(4)	O(1) O(5) O(9) O(13)	C(1) C(5) C(9) C(13)	0·38 0·39 0·45 0·33	C(2) C(6) C(10) C(14)	0·77 0·78 0·80 0·75	
Six-membere	ed chelate ri	ngs		. ,				
Plane de	termined by	:		Distances o	f the atoms	from the p	lanes in Å:	
Co(1) Co(1) Co(2) Co(2)	N(1) N(2) N(3) N(4)	O(4) O(8) O(12) O(16)	C(2) C(6) C(10) C(14)	1·23 1·27 1·22 1·24	C(3) C(7) C(11) C(15)	1·01 1·10 0·95 1·02	C(4) C(8) C(12) C(16)	

helices of complex anions. They are held together by $O-H\cdots O$ hydrogen bonds and are linked to the carboxylic oxygen atoms.

Table 7. Interatomic distances less than 3.4 Å Key to symmetry operations

$ \begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	$\begin{array}{cccc} y & z \\ \frac{1}{2} + y & \frac{1}{2} - z \\ \frac{1}{2} + y & \frac{1}{2} - z \\ 1 - y & \frac{1}{2} + z \\ \frac{1}{2} - y & 1 - z \\ 1 + y & z \end{array}$
$\begin{array}{c} Ca \cdots O(3) \\ Ca \cdots O(7) \\ Ca \cdots O(14) \end{array}$	2·394 (6) Å 4 2·322 (9) 1 2·413 (5) 2
$\begin{array}{c} 0(1) \cdots N(4) \\ 0(2) \cdots O(13) \\ 0(2) \cdots O(14) \\ 0(2) \cdots N(3) \\ 0(2) \cdots C(11) \\ 0(3) \cdots O(11) \\ 0(5) \cdots C(11) \\ 0(6) \cdots W(8) \\ 0(6) \cdots W(8) \\ 0(6) \cdots W(9) \\ 0(8) \cdots W(5) \\ 0(9) \cdots N(2) \\ 0(9) \cdots N(4) \\ 0(9) \cdots C(14) \\ 0(10) \cdots C(7) \\ 0(10) \cdots C(7) \\ 0(10) \cdots W(3) \\ 0(13) \cdots W(4) \\ 0(14) \cdots N(3) \\ 0(15) \cdots W(1) \\ 0(15) \cdots W(6) \\ 0(15) \cdots W(1) \\ 0(15) \cdots W(1) \\ 0(15) \cdots C(1) \\ 0(16) \cdots N(2) \end{array}$	$\begin{array}{cccccccc} 2\cdot871 & (8) & 2 \\ 3\cdot358 & (7) & 1 \\ 3\cdot097 & (8) & 1 \\ 3\cdot104 & (9) & 2 \\ 3\cdot189 & (10) & 2 \\ 3\cdot164 & (10) & 2 \\ 3\cdot093 & (8) & 2 \\ 3\cdot297 & (9) & 4 \\ 2\cdot764 & (13) & 3 \\ 2\cdot799 & (19) & 1 \\ 2\cdot888 & (9) & 1 \\ 3\cdot176 & (8) & 1 \\ 3\cdot239 & (8) & 2 \\ 3\cdot136 & (9) & 2 \\ 3\cdot258 & (10) & 1 \\ 2\cdot773 & (9) & 1 \\ 2\cdot895 & (7) & 2 \\ 2\cdot933 & (2) & 2 \\ 2\cdot933 & (2) & 2 \\ 2\cdot933 & (2) & 2 \\ 2\cdot931 & (12) & 1 \\ 2\cdot871 & (9) & 1 \\ 3\cdot305 & (10) & 1 \\ 3\cdot043 & (8) & 1 \end{array}$
$\begin{array}{c} Ca \cdots W(1) \\ Ca \cdots W(2) \\ Ca \cdots W(3) \\ Ca \cdots W(4) \end{array}$	2·435 (8) Å 1 2·362 (8) 6 2·403 (8) 1 2·480 (7) 6

.

Table 7 (cont.)

0·40 0·38 0·34

0.53

$N(1) \cdots W(6)$	3.388 (12)	1
$N(1) \cdots O(10)$	2.968 (9)	4
$N(2) \cdots W(6)$	3.004(12)	i
$N(3) \cdots W(4)$	2.974 (9)	1
$C(4) \cdots O(1)$	3.098 (10)	2
$C(4) \cdots W(2)$	3.252(12)	ī
$C(6) \cdots W(6)$	3.390 (13)	1
$W(1) \cdots W(3)$	3.171(11)	ī
$W(1) \cdots W(7)$	2.975(23)	ī
$W(2) \cdots W(10)$	2.678 (15)	6
$W(4) \cdots W(7)$	3.353(12)	Ĩ
$W(4) \cdots W(11)$	2.835(23)	1
$W(5) \cdots O(12)$	2.975 (9)	2
$W(5) \cdots W(4)$	2.760 (10)	6
$W(5) \cdots W(6)$	3.291 (13)	6
$W(6) \cdots W(3)$	2.864(13)	4
$W(6) \cdots W(11)$	2.725 (22)	1
$W(8) \cdots W(9)$	2.709 (23)	1
$W(8) \cdots W(11)$	2.595 (21)	1
$W(9) \cdots W(10)$	2.732 (22)	3
$W(10)\cdots O(2)$	2·771 (14)	5

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