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The Crystal Structure of Carbonatotetrakis(pyridine)cobalt(III) Perchlorate Monohydrate

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Carbonatotetrakis(pyridine)cobalt(III) perchlorate monohydrate ($CoC_{21}H_{20}N_4O_7Cl.H_2O$) crystallizes in space group $P2_1/c$ (No. 14) with $a=9{\cdot}457$ (3), $b=16{\cdot}333$ (6) and $c=15{\cdot}582$ (11) Å, $\beta=97{\cdot}93$ (4)°, $Z=4$. There is one formula unit in the asymmetric unit. The structure was solved by Patterson and Fourier techniques and refined by full-matrix least-squares methods to an R value of 0·049, using 3302 diffractometer-collected three-dimensional single-crystal X-ray intensity data. The cobalt atom is octahedrally surrounded by four nitrogen atoms of the pyridine molecules and two oxygen atoms of the bidentate carbonate group. The coordination octahedron is distorted due to strain in the four-membered chelate ring and steric hindrance between the pyridine molecules. The O–Co–O angle is 69·3°. There are no very short intermolecular distances, so the conformation is probably not very much affected by crystal packing.

Introduction

Nearly all known octahedral tetrakis(pyridine) complexes of transition metals have *trans* configuration. Only a few complexes with *cis* configuration have been reported, the dichlorotetrakis(pyridine)iridium(III) ion (Delépine & Lareze, 1963), the *cis*-dihalogenotetrakis(pyridine)ruthenium(II) complexes (Raichart & Taube, 1972), and the μ-oxalatobis[tetrakis(pyridine)-ruthenium(II)] ion. The crystal structure of the latter as fluoroborate has been determined (Cheng, Loescher & Nyburg, 1971).

The scarcity of *cis* isomers is without doubt due to steric hindrance. Springborg & Schäffer (to be published) prepared the carbonatotetrakis(pyridine)cobalt-(III) ion that must be a *cis* configuration as the carbonato group is bidentate. The present X-ray crystallographic investigation was undertaken in order to determine the conformation of the complex ion.

Experimental

The crystal used for data collection was prepared by diffusion. A 1 ml test tube was placed inside a 5 ml tube. The inner tube was filled with saturated aqueous sodium perchlorate solution, the outer one with saturated carbonatotetrakis(pyridine)cobalt(III) perchlorate solution, until the surfaces were about 2 mm from the edge of the inner tube. Then a 5 mm layer of water was placed above to connect the two solutions. After 12 to 24 hr beautiful red monoclinic crystals were formed at the boundary between the carbonatotetrakis(pyridine)cobalt(III) perchlorate solution and the water. Most of the crystals were elongated in the c-axis direction. From Weissenberg and precession photographs the space group was determined as $P2_1/c$. The unit-cell dimensions were obtained from least-squares refinement of a series of diffractometer-measured θ angles ($\lambda=0{\cdot}71069$ Å). $a=9{\cdot}457$ (3), $b=16{\cdot}333$ (6), $c=15{\cdot}582$ (11) Å, $\beta=97{\cdot}93$ (4)°. $V=2384$ Å^3. The formula weight is 552·6 ($CoC_{21}H_{22}N_4O_8Cl$); density, measured at 20°C = 1·547 g cm^{-3} (flotation), calculated = 1·540 gcm^{-3} for $Z=4$. The linear absorption coefficient, μ(Mo $K\alpha$) is 9·23 cm^{-1}.

All X-ray data were obtained from a crystal 0·18 × 0·30 × 0·30 mm in a Nonius 3-circle automatic diffractometer using graphite monochromated Mo $K\alpha$ radiation. The crystal was sealed in a glass capillary and oriented with its c axis along the φ axis of the goniometer. The ω-scanning mode was used. Scan speed 1·2° per min. Each reflexion was scanned over a range of 1·0°. The background was measured on each side of the reflexion for half the scanning time. The background count was set equal to twice the lowest count obtained, as the scanning range apparently had been too small. Intensities for 4188 independent reflexions in a quarter of a sphere in reciprocal space in the range $2{\cdot}5° < \theta < 25°$ were measured. 3302 of these had intensities greater than 2·5 times their corresponding standard deviations (obtained from counting statistics) and were considered observed.

A standard reflexion was remeasured after each 25 reflexions. A minor decline in the intensity of this reflexion appeared during the last half part of the intensity measurements. It amounted to 5%, and was corrected for. No absorption corrections were applied.

Determination and refinement of the structure

The positions of the cobalt and the chlorine atoms were detected from a three-dimensional sharpened Patterson synthesis. An E map phased by these two atoms

Table 1. *Observed and calculated structure factors* (×10) *for carbonatotetrakis(pyridine)cobalt*(III) *perchlorate monohydrate*

Unobserved reflexions are designated with an asterisk. The columns are h, F_o and F_c.

H,0,0		
2	1071	-1175
3	727	682
5	609	-627
6	425	456
7	598	596
8	278	-272
9	370	-351
10	291	304
11	117	112
H,1,0		
1	1527	-1675
2	946	-951
3	606	579
4	385	-373
5	539	-536
6	512	523
7	204	190
8	425	-420
9	174	191
10	250	253
H,2,0		
1	53	21
2	251	189
3	330	-324
4	779	-771
5	416	437
6	89	100
7	271	-279
9	320	305
11	199	-199
H,3,0		
1	1460	1477
2	1187	-1175
3	511	-530
4	323	326
5	347	300
6	577	-573
7	205	194
8	377	354
9	225	-212
10	214	-209
11	101	106
H,4,0		
0	1736	-1808
1	86	51
2	42	-23
3	93	-64
4	580	596
5	378	-377
6	490	-490
7	299	256
8	94	83
9	286	-281
10	156	-162
H,5,0		
1	42	-17
2	359	-329
3	301	291
4	141	-160
5	195	-206
6	223	212
7	67	62
8	160	-160
10	246	239
H,6,0		
0	1026	-1013
1	105	76
2	332	327
3	261	-252
4	265	-268
5	332	326
6	207	194
7	205	-203
8	171	161
9	129	140
10	151	-149
H,7,0		
1	96	85
2	132	-137
3	377	-375
4	343	358
5	393	400
6	132	-127
7	136	-131
8	92	74
10	141	-138
H,8,0		
0	625	591
1	267	252
2	335	-338
3	134	124
4	477	443
5	345	-337
6	269	262
7	176	177
9	143	-130
10	67	53
H,9,0		
1	350	-348
2	263	257
3	217	181
4	356	-390
5	323	331
6	225	209
7	95	-76
8	285	-284
H,10,0		
1	145	134
2	162	-175
3	83	56
4	107	-100
5	386	355
7	139	-139
8	136	-128
9	121	125
H,11,0		
2	119	-95
3	79	51
4	404	401
5	130	-136
6	93	32
7	195	-166
8	85	85
H,12,0		
1	142	-146
3	197	-185
4	269	-266
5	161	-172
6	289	-285
7	114	-116
H,13,0		
1	331	331
2	292	-285
3	245	-242
4	63	54
5	215	-219
6	183	-182
7	156	157
8	105	77
H,14,0		
0	119	142
1	143	-139
2	55	35
4	127	123
5	122	-118
6	115	111
7	198	204
H,15,0		
1	127	-135
5	62	53
6	109	101
H,16,0		
0	459	-463
1	55	77
2	257	258
3	67	-88
4	67	67
5	166	160
H,17,0		
1	131	124
3	111	-113
5	115	115
H,18,0		
1	66	-34
2	124	-128
3	103	89
4	113	112
H,19,0		
1	179	-175
2	220	223
H,1,1		
1	355	-410
-1	145	45
2	580	539
-2	121	92
3	589	-613
-3	380	-426
4	263	-259
-4	355	-336
5	297	312
-5	174	178
6	60	-71
-7	257	265
8	128	140
-8	75	40
9	80	71
-9	209	-218
10	70	-44
11	91	71
H,2,1		
0	666	634
1	676	-677
-1	753	756
2	270	-273
-2	2059	-2267
3	236	230
-3	146	-137
4	527	-545
-4	438	446
5	265	240
-5	939	-913
6	263	278
-6	76	-70
7	234	211
-7	211	216
8	352	-345
-8	62	-57
9	106	106
-9	98	-98
-10	108	112
-11	77	-57
H,3,1		
0	72	31
1	77	-109
-1	475	415
2	895	863
-2	345	-345
3	70	67
-3	472	-484
4	551	-554
-4	366	413
5	132	-105
-5	310	324
6	192	193
-6	61	-98
7	156	-155
-7	459	-452
8	180	-176
-8	69	26
9	98	87
-9	84	72
-11	67	105
H,4,1		
0	722	708
-1	311	317
2	265	-286
-2	239	237
3	268	-249
-3	293	-286
4	159	127
-4	292	-266
5	231	213
-5	276	268
6	202	191
-6	504	517
7	292	-280
-7	251	231
-8	282	-305
9	183	177
10	93	-106
H,5,1		
0	192	165
1	669	634
-1	439	-437
2	411	-427
-2	122	-130
3	342	349
-3	536	552
4	601	590
-4	137	-152
5	414	-412
-5	344	-335
6	219	-215
-6	183	200
7	358	369
-7	211	211
8	230	235
-8	99	-113
9	240	-226
-10	135	124
H,6,1		
0	93	-112
1	157	-165
-1	259	234
2	85	-35
-2	284	-257
3	640	645
-3	102	-77
4	135	-155
-4	343	369
5	436	-424
6	73	72
-6	381	-384
7	302	309
8	198	-191
-8	336	336
9	199	-200
-9	122	-123
10	250	217
-10	107	-111
H,7,1		
1	223	190
2	234	225
-2	213	218
3	178	-161
-3	597	-613
4	499	-497
-4	346	-348
-5	383	377
6	107	115
-6	282	283
-7	341	-336
8	192	-175
9	180	175
-9	329	326
10	83	59
-10	252	-261
H,8,1		
0	109	83
1	238	250
-1	70	-99
2	276	256
-2	761	782
3	771	-770
-3	246	247
-4	861	-850
5	600	588
-5	62	51
6	280	-272
-6	332	350
-7	105	-107
8	317	315
9	94	-73
-9	204	211
10	303	-294
H,9,1		
0	161	167
1	315	300
-1	165	-176
2	876	-881
-2	92	-97
3	195	178
-3	658	654
4	628	632
5	117	-127
-5	543	-552
6	110	-35
-6	154	-138
-7	329	343
8	66	46
-8	156	-165
9	206	-192
-9	169	-197
H,10,1		
0	64	85
1	461	-467
-1	312	341
2	342	-341
-2	439	-421
3	480	474
-3	350	-355
4	274	-282
-4	529	529
5	345	-353
6	65	49
-6	614	-623
7	169	159
-7	111	111
8	165	-142
-8	178	178
-9	245	-241
H,11,1		
0	725	-737
1	580	-585
-1	506	517
2	755	741
-2	276	290
3	120	-125
-3	570	-567
4	708	-711
-4	321	326
5	360	350
-5	561	573
6	338	348
-6	378	-379
7	328	-329
-7	124	-127
8	68	-37
-8	216	231
9	144	159
H,12,1		
0	53	-13
1	433	427
-1	334	-319
2	109	114
-2	188	191
3	511	-520
4	230	244
-4	269	-262
5	308	305
6	96	79
-6	409	409
7	267	-262
8	132	116
-8	216	-224
H,13,1		
0	516	527
1	77	79
-1	225	-219
2	309	-318
-2	256	-265
-3	353	352
4	313	325
-4	64	45
5	110	100
-5	253	-254
6	308	-307
7	166	172
-7	168	172
8	156	148
-8	101	-106
H,14,1		
0	461	473
1	494	-499
-1	373	383
2	157	-155
-2	466	-473
3	402	402
-4	192	178
5	261	-267
6	242	239
7	157	145
-7	170	175
H,15,1		
0	137	-139
1	131	-116
-1	129	141
-2	240	250
3	155	157
-4	240	-240
5	89	-106
-5	61	61
6	138	143
-7	162	-165
H,16,1		
0	263	-281
1	149	147
-1	181	-187
-2	208	196
3	103	-106
4	78	-100
5	97	86
-5	101	-93
-6	105	110
H,17,1		
0	149	141
1	83	52
-1	275	-273
2	262	-261
-2	64	-51
-3	169	171
4	221	220
-5	234	-238
H,18,1		
1	81	-73
2	81	78
-2	87	-75
3	137	132
-4	103	86
H,19,1		
0	85	-91
-1	93	112
2	109	122
-2	83	70
H,0,2		
0	67	-145
1	1488	1467
-1	2801	-2776
2	327	-355
-2	397	326
3	239	-286
-3	929	942
4	663	705
-4	49	-71
5	61	33
-5	163	-164
6	1172	-1162
-6	440	464
7	449	434
-7	350	-344
8	657	650
-8	293	-289
9	216	-203
-9	115	122
-10	163	163
-11	106	-121
H,1,2		
0	1560	1538
1	1122	-1144
-1	369	345
2	95	-77
-2	1363	-1425
3	457	-468
-3	1049	-1058
4	105	134
-4	613	626
5	654	-660
-5	416	-416
6	113	-94
7	466	455
-7	323	330
8	114	-112
-8	75	82
9	353	-342
-9	410	-420
-10	61	71
-11	280	276
H,2,2		
0	253	-292
1	538	-550
-1	730	674
2	556	605
-2	567	524
3	328	302
-3	226	-247
4	443	-424
-4	413	403
5	326	-316
-5	385	401
6	439	451
-6	149	-159
-7	298	-284
8	274	-271
-8	380	394
10	129	123
-10	262	-276
-11	85	83
H,3,2		
0	1972	-2108
1	1102	1073
-1	607	587
2	463	438
-2	650	643
3	813	-864
-3	638	-634
4	499	-504
-4	567	-569
5	747	752
-5	539	561
6	139	138
-6	345	346
7	574	-555
-7	329	-334
8	209	195
-8	137	138
9	177	180
-9	121	101
10	234	-227
-11	206	-198
H,4,2		
0	487	-505
1	907	861
-1	660	-648
2	1161	-1157
-2	748	-747
3	177	-194
-3	640	629
4	422	416
-4	57	-48
5	339	334
-5	557	-575
6	313	-309
-6	297	307
7	61	-44
-7	346	345
8	84	87
-8	461	-471
-9	62	-42
10	146	-133
-10	298	299
H,5,2		
0	123	-111
1	330	313
-1	203	-206
2	178	-179
-2	862	-836
3	712	664
-3	107	115
4	524	499
-4	261	281
5	185	-201
6	62	51
-6	329	-324
7	135	137
-7	328	332
8	84	66
-8	78	68
9	204	-214
-9	141	-129
10	78	61
H,6,2		
0	946	954
1	476	-509
-1	446	449
2	628	610
-2	216	-233
3	564	578
-3	881	-888
4	385	-390
-4	248	250
5	317	-300
-5	191	195
6	384	398
-6	395	-395
7	410	-400
8	316	-311
-8	122	103
9	215	208
-9	173	-157
10	103	114
H,7,2		
0	184	-174
1	543	501
-1	519	479
2	317	303
-2	444	446
3	551	-560
-3	153	152
4	263	-265
-4	367	-346
5	79	100
-5	217	231
6	201	-199
-6	325	330
7	116	105
-7	271	-282
-8	104	-98
9	268	278
-9	142	127
-10	115	-98
H,8,2		
0	247	-245
-1	333	-339
2	190	179
3	179	-186
-3	381	398
4	219	207
-4	206	-208
6	97	-90
-6	165	160
-7	200	198
8	362	358
-8	90	-81
9	73	-44
-9	99	86
H,9,2		
0	778	771
1	140	118
-1	414	-400
2	226	-225
-2	378	-365
3	60	39
4	117	-106
5	446	-447
-5	449	-443
6	167	162
-6	91	62
7	334	342
-7	114	112
8	126	-125
9	126	-138
-9	73	-67
H,10,2		
0	213	-196
1	58	61
-1	237	216
2	65	-44
-2	151	149
3	131	-127
-3	96	100
-4	101	93
5	96	88
6	65	-66
-6	130	-116
7	175	185
-7	306	-304
8	112	-105
-8	99	97
9	104	-119
-9	98	106
H,11,2		
0	141	125
1	137	-136
-1	258	-259
-2	68	36
-3	116	-104
4	235	-246
-4	112	-115
5	61	7
-5	187	190
6	73	-71
-6	113	118
7	112	-85
-8	202	-208
H,12,2		
0	169	157
2	228	-235
-2	104	115
3	140	-108
-3	115	112
-4	365	-377
-5	236	234
6	130	103
7	119	112
-7	103	102
-8	77	61
H,13,2		
0	440	-460
1	134	131
-1	251	245
2	296	299
-2	480	482
3	101	-95
-3	165	-152
4	144	127
-4	89	-70
5	343	336
-5	292	289
-6	149	-153
7	191	-187
8	98	98
-8	174	171
H,14,2		
1	79	81
-1	134	-144
-2	88	84
3	131	-143
-3	96	-155
-4	64	21
5	71	51
-5	210	-202
6	134	-124
-6	121	119
H,15,2		
0	132	128
-2	110	-117
-3	153	-158
-4	244	243
5	76	-59
-5	91	-82
-7	77	87
H,16,2		
1	254	-251
-1	435	449
2	102	79
-2	104	-96
3	115	104
-3	222	-219
4	129	-136
-4	137	147
6	209	217
-6	108	-90
H,17,2		
0	120	-119
-1	96	-76
2	70	68
-2	128	117
4	74	50
-4	104	-117
H,18,2		
0	141	140
1	93	82
-1	76	-65
2	88	-88
3	95	85
-3	133	128
H,19,2		
0	120	117
1	78	-93
-2	173	-173
H,1,3		
0	442	381
1	837	-859
-1	1251	-1331
2	144	118
-2	828	-877
3	566	587
4	456	-443
-5	245	-276
6	62	21
-6	58	34
7	217	-205
-7	139	131
-8	180	-190
-10	99	92
-11	104	-115
H,2,3		
0	913	872
1	2211	2333
-1	520	-546
2	1392	-1423
-2	257	-288
-3	166	199
4	366	371
-4	550	-550
5	465	-493
-5	248	-241
6	151	-143
-6	299	323
7	529	517
9	237	-222
-9	159	172
-11	74	-61
H,3,3		
0	230	-267
1	536	506
-1	1386	1363
2	422	-466
-2	400	354
3	1015	974
4	450	434
-4	615	608
5	90	-99
6	178	169
-6	416	-424
7	179	173
-7	93	-46
8	319	-356
-8	305	299
9	96	56
10	202	193
-11	73	79
H,4,3		
0	836	-826
1	1089	1056
-1	347	336
2	231	213
-2	573	570
3	194	169
-3	694	-656
4	120	-127
-4	520	492
5	391	-358
-5	155	161
-6	91	-72
7	199	-202
-7	442	-447
8	62	32
-8	236	-252
9	187	183
-9	296	293
-10	102	-120
H,5,3		
0	703	-716
1	97	-81
-1	164	119
2	515	492
-2	979	953
3	967	-955
-3	349	350
4	222	256
-4	460	-460
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8	405	404
-8	163	-165
9	66	-22
-9	111	100
10	141	-123
H,6,3		
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2	260	-301
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-5	377	-379
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7	111	-92
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8	113	111
9	212	-209
-9	251	-248
-10	166	174
H,7,3		
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2	219	213
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-4	551	546
5	120	-132
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-6	424	-431
-7	165	-153
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-8	258	261
-9	200	-197
-10	139	-147
H,8,3		
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-3	387	-390
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7	117	-95
-7	162	-162
-8	173	110
9	316	323
-10	112	-104
H,9,3		
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1	563	571
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2	152	-160
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3	669	-676
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-6	236	227
7	205	-192
8	136	127
-8	337	-335
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H,10,3		
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4	382	385
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6	269	-267
7	247	243
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8	107	93
-8	84	-87
9	197	-199
-9	259	-265
H,11,3		
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1	417	-439
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2	119	-117
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3	886	883
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-4	213	209
5	259	-265
6	356	367
-6	217	-213
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-7	298	295
8	240	-238
-8	282	293
H,12,3		
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-3	196	-198
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6	293	293
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7	184	-180
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8	96	-80
H,13,3		
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-2	214	204
3	352	-353
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4	152	161
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6	212	-211
-6	410	417
7	141	-134
-8	194	-202
H,14,3		
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1	176	180
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2	392	-396
-2	186	-193
-3	224	228
4	306	302
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5	350	-349
-5	379	-407
6	298	-311
-6	145	135
7	249	253
-7	104	104
H,15,3		
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-1	372	362
2	77	71
-2	222	-216
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4	153	-139
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5	166	-190
6	81	85
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H,16,3		
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2	125	109
-2	270	270
3	119	-122
-3	158	-148
4	227	-234
-4	232	-223
5	118	106
-5	154	166
H,17,3		
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-1	115	-115
2	170	-180
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3	313	-324
-3	109	125
4	66	22
-4	141	-147
H,18,3		
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1	149	-157
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2	99	-88
-2	67	-69
3	67	42
-3	75	56
-4	113	108
H,19,3		
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H,0,4		
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1	202	138
-1	1396	1406
2	635	646
-2	1576	1626
3	584	-607
-3	371	-422
4	81	62
-4	868	-914
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6	290	-271
7	1031	-1030
-7	169	-176
8	71	26
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9	151	140
-9	73	63
10	214	-215
-10	108	93
-11	135	-112
H,1,4		
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1	826	770
2	137	-147
3	458	-451
-3	676	711
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5	220	207
-5	408	-431
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-6	412	427
7	103	101
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8	290	298
-8	203	-204
-9	166	-187
10	82	-87
-10	236	238
H,2,4		
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3	411	394
-3	175	139
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-4	567	547
5	423	-398
-5	309	-301
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-6	331	-351
7	461	467
-7	404	400
-8	148	163
9	106	-94
-9	201	-213
10	144	129
-11	215	224
H,3,4		
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3	69	-37
-3	996	-993
4	870	-896
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5	64	-30
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6	801	811
-6	354	-372
7	311	-310
-7	88	-23
8	246	-241
-8	210	216
9	168	165
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H,4,4		
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1	1113	1091
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2	283	-281
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3	748	-722
-3	441	413
4	81	-86
-4	818	-836
5	351	344
-5	505	509
7	133	-128
-7	565	-570
8	76	59
-8	89	-73
-9	292	293
10	125	-121
H,5,4		
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1	304	270
-1	725	-730
2	1203	-1166
-2	330	-303
3	688	667
-3	1190	1172
4	340	335
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-7	314	336
8	96	89
-8	478	-492
9	110	-100
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H,6,4		
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2	221	-256
-2	758	-753
3	526	538
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4	159	144
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5	369	-383
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6	83	74
-6	305	-306
7	291	295
-7	156	169
8	230	-220
9	143	-142
-9	81	-81
H,7,4		
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1	478	-479
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3	170	164
-3	640	-613
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-6	139	-141
7	118	-107
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8	81	-60
-8	170	169
9	110	104
-10	130	-119
H,8,4		
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2	355	367
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3	142	-142
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7	169	-156
-8	144	-151
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H,9,4		
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3	446	-430
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4	222	220
-4	211	-194
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6	433	-429
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8	220	210
-8	146	151
9	111	-105
-9	69	-41
H,10,4		
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3	73	-64
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-6	83	-77
-7	109	95
8	112	90
-8	154	148
-9	72	-63
H,11,4		
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3	244	246
4	104	94
-4	178	171
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-5	102	-95
-6	65	-35
8	67	-42
H,12,4		
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2	383	379
3	87	83
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4	67	-52
-4	240	239
5	101	-89
6	102	-98
-6	218	-207
H,13,4		
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1	263	-265
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2	100	91
-2	403	-404
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4	415	-411
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5	125	-120
-5	69	-64
6	186	179
-7	68	6
-8	141	-144
H,14,4		
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2	64	-47
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3	134	-123
-3	62	54
4	103	-80
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-5	117	122
-6	115	123
7	146	-145
H,15,4		
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3	120	117
4	105	102
-4	93	104
-5	205	-205
-6	103	117
H,16,4		
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2	130	-112
-2	252	-243
3	154	151
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5	127	-139
-5	74	-67
H,17,4		
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1	78	-75
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-2	187	183
-3	284	-277
-5	194	212
H,18,4		
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1	109	99
-1	111	-102
-2	177	179
3	69	-72
H,1,5		
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2	431	-424
-2	703	673
3	270	298
-3	385	384
4	211	229
-4	80	-102
5	195	-190
-5	220	-231
6	141	-145
7	152	-125
-7	160	-169
8	193	182
-9	98	89
10	87	82
H,2,5		
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1	148	122
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2	687	665
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3	223	-245
-3	51	-28
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-4	143	131
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6	151	-138
-6	93	-95
7	145	137
-7	133	133
8	324	319
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9	116	115
-9	145	-141
10	148	-143
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H,3,5		
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1	869	844
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2	703	-711
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3	482	-457
-3	113	-138
-4	232	-222
5	71	-67
-5	172	-191
6	162	161
7	281	280
-7	143	156
8	149	-151
9	173	-175
-9	99	-75
-11	88	60
H,4,5		
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1	778	-769
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2	580	-557
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3	111	91
-3	221	-206
4	85	-90
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6	153	151
-6	402	-398
7	295	-285
-7	124	-107
-8	216	210
-9	72	-77
H,5,5		
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1	137	-156
2	544	535
-2	456	410
3	70	-37
-3	227	-247
4	470	-507
5	420	432
-5	395	373
6	244	241
-6	161	164
7	71	-74
-7	67	62
8	172	-172
9	200	209
-9	78	101
H,6,5		
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1	435	430
-1	1175	-1147
2	127	-118
-2	69	-42
3	680	-704
-3	918	921
4	498	501
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5	62	48
-5	363	-360
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-6	218	236
8	108	93
-8	352	-355
9	130	-132
-9	220	230
-10	91	80
H,7,5		
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1	1008	-1003
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-2	837	-839
3	157	172
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4	253	250
-4	562	569
5	171	-167
-5	362	-352
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-6	228	-225
-7	344	334
9	169	-182
-9	305	-313
-10	150	151
H,8,5		
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1	474	-495
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-2	704	-703
3	669	655
-3	203	-202
4	163	-175
-4	269	265
5	263	-266
-5	73	-54
6	203	193
-6	89	-93
7	118	93
8	469	-479
-8	293	323
9	80	84
-9	101	-106
H,9,5		
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1	88	79
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2	557	552
-2	630	615
-3	362	-373
4	136	-127
-4	226	-214
5	268	254
-5	221	214
6	107	-101
7	210	-209
-7	329	-337
-8	119	109
-9	256	259
H,10,5		
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1	547	559
-1	334	-344
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-2	303	298
3	134	-133
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4	255	254
-4	136	-134
5	211	212
-5	156	-154
6	243	-244
-6	297	289
7	71	-65
8	257	252
-8	330	-334
-9	104	105
H,11,5		
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1	306	317
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2	727	-746
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-3	205	266
4	265	289
-4	204	-185
5	364	-370
-5	142	-131
6	268	-279
-6	93	78
7	362	370
-7	127	127
8	83	97
-8	85	-78
-9	159	-170
H,12,5		
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1	491	-500
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2	76	-86
-2	95	84
3	259	257
-3	104	-185
4	143	-135
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5	117	-110
6	215	225
-6	285	-280
-8	202	209
H,13,5		
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1	212	203
-1	99	-87
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3	155	-157
-3	97	-90
4	162	-173
-4	164	-158
5	108	201
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6	103	91
-6	69	61
7	252	-256
-7	303	-303
H,14,5		
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1	297	308
-1	313	-312
2	229	-259
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3	301	-293
-3	125	125
4	197	190
-4	259	-263
5	173	169
-5	70	-58
6	309	-306
-6	190	186
H,15,5		
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1	153	-165
-2	234	-234
3	164	145
4	138	146
-4	448	445
5	239	-242
-5	317	-326
-6	101	-92
H,16,5		
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1	120	-123
-1	189	187
2	211	217
-2	61	-74
3	79	74
-3	232	-228
4	224	-232
-4	316	318
-5	124	137
H,17,5		
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2	143	141
-2	64	-67
4	166	-185
-4	146	-157
H,18,5		
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1	142	143
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-3	154	142
H,0,6		
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1	231	-258
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2	1035	1058
-2	312	-322
3	369	395
-3	375	-399
4	778	-805
-4	967	996
5	186	191
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6	1067	1085
-6	279	-296
7	217	-217
-7	116	114
8	162	-162
-8	224	-220
9	277	266
-9	318	328
10	89	89
H,1,6		
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1	410	395
-1	460	-464
2	217	212
-2	182	202
3	544	-537
-3	91	-52
4	331	320
-4	976	-1018
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-5	139	-129
6	297	-317
-6	732	753
7	281	-290
-7	413	-424
8	161	158
-8	311	-245
9	102	106
-9	142	127
10	135	-154
-10	67	37
-11	208	-198
H,2,6		
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1	516	500
-1	526	-542
2	1144	-1136
-2	58	22
3	205	198
-3	334	311
4	235	245
-4	74	-62
5	238	-249
-5	625	-648
6	292	-295
-6	275	274
7	122	154
-7	420	422
8	114	113
-8	262	-259
9	172	-178
-9	236	-242
-10	166	173
H,3,6		
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1	494	-505
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2	371	-405
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3	688	712
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-5	293	-296
-6	274	-272
7	383	395
-7	276	289
8	380	-354
-8	182	173
9	90	-68
-10	184	-186
H,4,6		
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1	176	-181
-1	646	619
2	665	676
-2	362	351
3	603	-673
-3	1013	-1023
4	321	-330
-4	575	-573
-5	670	673
6	130	126
7	104	-118
-7	455	-450
-8	368	371
9	113	116
-9	89	91
-10	366	-363
H,5,6		
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1	435	418
-1	241	-250
-2	569	555
3	423	-459
-3	193	-186
4	317	311
-4	674	-674
5	293	298
-5	169	-161
6	182	176
-6	336	350
7	161	-164
-7	75	-52
8	195	205
-8	72	-77
9	75	-45
-9	282	294
-10	107	92
H,6,6		
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1	180	191
-1	58	24
2	230	198
-2	156	-150
3	221	-226
-3	395	375
4	405	411
-4	272	-268
5	180	190
-5	258	-259
6	574	-588
-6	173	-172
7	201	190
-7	86	-72
9	267	-262
-9	70	-70
-10	125	129
H,7,6		
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1	601	-617
-1	877	865
2	107	96
-2	296	-267
3	150	160
-3	77	43
4	370	-391
-4	312	325
-5	204	203
6	136	125
-6	288	-287
-8	85	81
-9	225	-223
H,8,6		
0	159	-163
1	164	-157
-1	100	91
2	209	208
-2	242	243
3	107	118
-3	126	104
4	187	-187
-4	120	110
-5	184	186
6	170	188
-6	114	115
8	72	-63
-8	71	79
-9	81	73
H,9,6		
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1	123	116
-1	67	-66
2	264	257
-2	139	142
3	209	-216
-3	243	-224
4	196	-196
-4	171	167
5	270	279
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6	68	49
-6	115	-106
7	84	-89
-9	88	-81
H,10,6		
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1	184	196
-1	158	-141
2	172	-175
-2	246	-243
-4	196	200
5	65	-57
-5	120	-113
6	132	129
-6	112	104
-7	95	99
-8	130	-122
H,11,6		
-1	333	333
2	403	-411
-2	82	-97
4	72	32
-4	67	-49
5	65	-42
-6	84	-58
H,12,6		
0	102	-112
1	406	-404
-1	161	-147
2	271	-280
3	196	184
-3	120	-130
-4	76	53
-5	137	-135
7	131	-116
-7	64	37
H,13,6		
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-1	126	-115
2	80	66
3	168	150
-3	121	114
4	197	205
-4	63	-25
5	116	-121
-5	123	-118
6	116	-103
H,14,6		
-1	102	107
2	132	136
3	96	97
-3	105	123
-4	68	86
-5	95	95
-7	74	-66
H,15,6		
0	107	-98
1	199	190
-1	123	-120
-4	191	-194
5	81	78
-6	184	186
H,16,6		
0	85	52
1	146	152
-1	117	-122
2	109	-89
-2	75	-36
-3	190	200
4	175	176
-4	69	-45
-5	155	-131
H,17,6		
0	146	154
1	133	-137
-1	73	61
-2	182	-162
3	177	189
-3	134	-125
-4	179	179
H,18,6		
0	127	-125
-1	148	163
-2	148	151
H,1,7		
0	185	160
1	78	-64
-1	169	-163
-2	272	233
3	304	-317
-3	267	-290
4	83	70
5	125	133
-5	392	393
6	91	103
-6	123	-124
8	124	122
9	123	-119
H,2,7		
0	244	-229
1	721	-717
-1	367	-348
2	524	514
-2	278	250
3	57	29
-3	94	-86
4	97	-105
-4	103	115
5	173	173
-5	233	-252
6	64	65
-6	73	54
7	79	-75
8	219	-215
-8	102	94
9	153	159
-9	143	-126
H,3,7		
0	52	-58
1	446	427
-1	337	-339
2	307	283
-2	555	-557
-3	270	268
-4	89	59
5	90	-83
-5	145	-135
6	206	-207
7	70	-61
-7	145	132
-8	73	-81
9	114	124
-10	84	74
H,4,7		
0	890	876
-1	440	407
2	62	-84
-2	355	-349
3	144	129
-3	76	-65
4	97	-78
-4	155	171
5	126	-133
-5	70	-75
6	163	166
-6	69	-58
-7	202	290
-8	201	-189
9	80	-75
-9	101	-111
-10	73	75
H,5,7		
0	160	147
1	318	-329
-1	174	151
-2	59	-86
3	643	668
-3	286	251
4	347	-356
-4	70	-47
5	335	-338
-5	69	-61
6	181	171
-6	93	86
7	121	125
-7	70	-30
8	151	-146
-6	203	207
-9	112	-119
-10	98	-104
H,6,7		
0	434	-446
1	334	318
-1	249	-243
2	93	108
-2	587	571
3	382	-405
-3	363	-337
4	349	-352
-4	436	-439
5	338	338
-5	212	220
-6	76	59
7	81	-109
-7	157	-155
8	74	55
9	109	116
-9	246	247
-10	103	-106
H,7,7		
0	316	308
1	427	436
-1	863	-871
2	369	-378
-2	213	198
3	361	-367
-3	562	567
4	643	573
-4	196	-186
5	82	-67
-5	305	-312
6	264	-265
-6	274	274
7	119	125
8	114	105
-8	340	-331
-9	135	142
-10	194	188
H,8,7		
0	413	400
1	93	108
-1	166	-175
2	322	-332
-2	299	-307
-3	64	84
4	487	504
-4	277	281
5	374	-393
-5	75	-71
6	91	-93
7	353	373
-7	111	114
-8	93	79
-9	179	-176
H,9,7		
0	184	-191
1	317	-332
-1	880	863
2	261	270
-2	238	-220
3	143	155
-3	704	-749
4	332	-342
-4	249	237
5	132	126
-5	173	170
6	292	302
-6	136	-124
-7	202	-208
8	201	-221
-8	256	259
-9	75	-63
H,10,7		
0	539	-536
1	306	303
-1	102	-79
-2	748	749
3	313	-317
-3	246	-241
4	137	-138
-4	477	-473
5	253	263
-5	136	144
-6	181	183
7	273	-270
-7	312	-315
-9	192	184
H,11,7		
0	174	-172
1	297	301
-1	224	-230
2	147	-151
-2	136	129
3	206	-194
-3	214	223
4	68	63
-4	81	-62
5	222	231
6	317	-326
-6	77	73
-7	130	154
-8	140	-147
H,12,7		
0	363	355
-1	175	191
2	113	-142
-2	452	-444
3	184	178
-3	186	183
-4	153	152
5	180	-174
-5	155	-157
6	114	104
-6	122	-111
7	199	200
-7	321	314
-8	80	22
H,13,7		
0	69	-66
1	305	-311
-1	222	223
2	134	134
-2	152	148
3	77	85
-3	277	-280
4	147	-145
-4	222	227
-5	151	136
6	263	272
-6	306	-316
-7	71	-47
H,14,7		
0	226	-234
-1	163	158
2	246	256
-2	68	-59
3	99	-118
4	137	-143
-4	126	-113
5	241	248
-5	177	180
-6	122	138
-7	125	-115
H,15,7		
0	57	53
1	173	184
-1	167	-168
2	154	-137
-2	73	67
3	84	-75
-3	246	252
4	242	243
-4	248	-264
-5	212	-211
-6	202	203
H,16,7		
0	224	220
1	133	-112
-1	119	-123
2	171	-171
3	174	184
4	98	92
-4	193	205
-5	196	-197
H,17,7		
0	51	47
1	195	-202
-1	108	112
-3	90	-76
H,0,8		
0	312	281
1	420	-420
-1	168	-163
2	201	186
3	539	556
-3	473	-611
-4	172	150
5	188	-184
-5	109	120
-6	816	-882
-7	81	-99
8	303	-293
-8	439	458
9	158	-153
-10	145	-156
H,1,8		
0	703	-688
1	102	-118
-1	271	272
2	379	402
-2	386	395
3	403	-390
-3	314	-323
4	274	-288
-4	321	-311
-5	729	765
6	158	-149
-6	306	-316
7	327	-336
-7	577	-591
8	189	-191
9	238	242
-9	220	232
-10	111	-115
H,2,8		
0	286	-316
1	642	645
-1	501	-511
2	122	114
-2	295	284
3	438	-445
-3	387	378
4	317	316
-4	329	-351
-5	69	81
6	205	-226
-6	637	662
7	106	-108
-7	80	-66
8	311	308
-8	237	-250
-9	131	142
-10	115	128
H,3,8		
0	533	518
1	220	205
-1	302	-287
2	530	-531
-2	186	-209
3	179	-167
-3	69	78
4	470	476
-4	61	-25
-5	424	-416
6	441	-445
-6	83	-79
7	286	287
-8	69	61
9	190	-172
-9	218	-222
-10	140	130
H,4,8		
0	190	175
1	654	-652
-1	686	676
-2	696	-688
3	677	690
-3	126	-152
4	308	-317
-4	611	625
5	399	-407
-5	97	-81
6	212	223
-6	321	-320
7	164	169
-7	70	73
8	292	-294
9	113	113
-9	192	-205
-10	97	-89
H,5,8		
0	152	-165
-1	582	579
2	688	672
-2	207	198
3	306	-310
-3	481	-481
4	469	-471
5	115	102
-5	432	441
6	257	250
7	171	-178
8	86	86
-8	137	130
-9	89	104
-10	153	-160
H,6,8		
0	258	-245
1	169	-148
2	224	226
-2	354	339
3	450	-476
4	126	-115
5	469	474
-5	65	16
7	161	-164
8	260	255
-8	93	-95
-10	116	103
H,7,8		
0	569	571
-1	409	-414
2	137	-130
-2	330	-329
3	284	284
-3	436	406
4	180	191
-4	101	107
5	142	-119
-5	311	-304
-6	193	208
7	191	194
-7	167	161
-8	110	-95
-9	125	-127
H,8,8		
0	136	-133
1	106	-92
-1	445	450
3	65	83
-3	60	-76
-4	147	139
5	82	-80
-6	186	-190
8	88	-60
-8	149	133
-9	70	-92
H,9,8		
0	133	-141
1	133	-121
-1	289	-290
2	102	-169
-2	102	-95
3	307	320
-3	234	227
4	256	-281
-4	77	65
5	90	-60
6	94	89
-6	66	58
-8	76	-31
H,10,8		
0	136	146
1	268	288
-1	626	-624
2	220	-225
-3	84	93
4	99	93
-4	71	-53
5	68	-57
-8	122	112
H,11,8		
1	69	-18
-1	63	26
2	125	-110
-2	258	-264
-3	279	-274
4	88	99
-4	97	99
5	144	-144
6	71	37
-6	86	-79
-7	101	88
-8	71	-80
H,12,8		
0	170	172
1	243	245
-1	65	-44
3	157	171
-4	209	-208
-6	95	88
-7	80	-32
H,13,8		
0	174	168
1	167	188
-1	259	256
-2	96	106
3	177	-184
4	68	65
-4	68	-68
5	121	98
H,14,8		
0	58	-67
1	242	-239
-1	74	61
2	67	-43
-2	137	135
3	94	-90
-3	121	-118
-6	100	-72
H,15,8		
0	212	-208
2	194	198
-2	109	110
4	73	-58
-5	127	128
H,16,8		
1	153	147
-1	81	67
3	174	-176
-4	79	-42
H,17,8		
0	108	109
-1	74	-72
-2	81	-91
-3	160	159
H,1,9		
0	138	-129
1	235	-225
2	90	82
-2	62	38
3	66	35
4	196	-200
-4	309	300
5	272	-279
-5	237	-258
6	97	109
-6	238	-248
-7	154	154
-8	184	-191
-9	101	-82
H,2,9		
0	52	-63
1	79	-71
-1	145	-146
2	207	192
-2	105	-116
3	124	-127
-3	479	482
4	162	158
-4	188	192
6	90	97
-6	365	406
7	262	267
-7	326	-340
8	207	-205
-8	175	176
-9	89	74
H,3,9		
1	243	228
-1	199	-203
-2	355	342
-3	85	89
4	170	177
-4	163	-168
5	373	388
6	67	-5
-6	334	342
7	169	-165
-7	243	-255
-8	101	93
-9	172	173
H,4,9		
0	64	73
1	349	355
-1	376	-378
2	386	-384
-2	93	-95
-3	339	331
4	385	394
-4	127	-140
5	151	-168
-5	182	-183
6	223	-228
-6	199	215
7	75	87
-7	83	-33
8	70	25
-8	252	-251
H,5,9		
0	167	168
1	94	-93
2	344	-353
-2	187	-157
3	93	106
-3	99	63
4	359	376
-4	71	-63
5	196	-187
-5	128	139
6	196	-202
-6	82	-86
7	137	126
-7	130	129
-8	119	117
-9	124	-118
H,6,9		
0	387	-396
1	252	-266
-1	662	644
2	437	424
-2	188	-192
3	240	260
-3	470	-466
4	567	-592
-4	162	164
-5	205	196
6	181	184
-6	178	-164
7	113	-98
8	78	-67
-8	188	201
-9	88	-71
H,7,9		
0	384	-373
1	309	314
-1	80	-72
2	185	192
-2	502	487
3	449	-455
-3	178	-187
4	90	-70
-4	416	-416
5	409	426
-5	199	199
6	99	-91
-6	91	77
7	167	-138
-7	189	-189
8	128	111
-8	84	57
-9	144	142
H,8,9		
-1	371	-385
2	71	-48
-2	189	195
3	144	-136
-3	82	69
4	217	219
5	128	132
-5	135	-120
6	293	-306
-6	92	-87
-7	94	104
-8	103	-99
-9	83	-66
H,9,9		
0	81	25
1	169	-163

Table 1 (*cont.*)

	H,9,9	
-1	67	92
-2	626	-632
3	346	345
-3	90	72
4	73	-62
-4	319	311
5	423	-433
-5	178	-185
6	95	130
-6	223	-238
7	147	159
-7	149	147
-9	139	-144
	H,10,9	
0	59	-35
1	72	68
-1	286	273
2	306	299
-2	277	277
3	105	97
-3	468	-467
4	245	-257
-4	138	127
-5	295	316
6	239	233
-6	142	-128
7	150	-138
-7	163	-152
-8	143	136
	H,11,9	
0	85	-77
1	131	136

-5	461	-465
6	99	-88
-6	97	104
7	231	239
-7	239	246
8	77	-91
-8	78	-86
-9	139	-143
	H,5,10	
1	428	-418
-1	311	299
2	93	96
-2	427	-432
3	504	511
-3	219	-214
4	217	-216
-4	464	469
5	126	-143
6	248	255
-6	197	-169
-7	145	138
8	120	-130
-8	138	136
-9	91	-82
	H,6,10	
1	92	62
2	144	145
-2	119	111
3	117	110
-3	153	-144
4	219	-212
-4	249	-263
5	126	118

only ($R=\sum w(|F_o|-|F_c|)/\sum|F_o|=0·45$) showed the six ligating atoms (four nitrogens and two oxygens) in octahedral positions around the cobalt atom. On this basis a new E map ($R=0·40$) revealed the rest of the non-hydrogen atoms except for the water oxygen atom.

All atoms were assumed to be non-charged and the scattering factors were taken from *International Tables for X-ray Crystallography* (1962).

During two cycles of least-squares refinement the R value decreased from 0·30 to 0·18 and the missing oxygen atom was found on a Fourier map. Two cycles of isotropic full-matrix refinement led to an R value of 0·11 and further isotropic refinement did not improve the result. A difference Fourier synthesis showed that the thermal vibrations of several atoms were distinctly anisotropic. After three cycles of anisotropic refinement the R value was 0·096, and a difference Fourier synthesis revealed all the hydrogen atoms except those of the water molecule. The hydrogen positions were in accordance with those calculated from the positions of the bonded atoms, assuming that the hydrogen atoms were placed at the extensions of the diagonals of the pyridine rings. The carbon–hydrogen bond length was set to 1·08 Å.

In the following cycles of refinement, new positions for the hydrogen atoms were calculated after each cycle, but refinement of the hydrogen atom parameters was not attempted. The isotropic temperature factor for the hydrogen atoms was fixed at $B=5·0$ Å. The refinement ceased at $R=0·049$. At this stage the shifts were less than half the corresponding standard deviation (average shift/error = 0·22). Because of capacity restrictions of the least-squares program, each of the pyridine rings was treated as an isolated block and so was the perchlorate ion and the cobalt atom plus the carbonato group. Finally the blocking was changed for a cycle of refinement to assure that the blocking had not biased the result.

During the later stages of the refinement an empirical weighting scheme was used, which gave highest weight to reflexions of moderate intensity and lower weight to reflexions with high intensity or low value of $\sin\theta$. $1/w=-0·5+2\sigma(F)^2+0·85/\sin\theta-0·09F+0·002F^2$. A final analysis of $\sum w(F_o-F_c)^2$ as a function of $|F_o|$ and $\sin\theta$ showed a smooth distribution indicating the validity of the weighting scheme. Observed and calculated structure factors are listed in Table 1. The refined coordinates and thermal parameters of the

Table 2. *Final parameters (and e.s.d.'s) of non-hydrogen atoms*

$$\text{T.F.}=\exp\{-2\pi^2(U_{11}a^{*2}h^2+2U_{12}a^*b^*hk+2U_{13}a^*c^*hl+U_{22}b^{*2}k^2+2U_{23}b^*c^*kl+U_{33}c^{*2}l^2)\}.$$

Thermal parameters are in Å$^2\times10^2$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co	0·28213 (5)	0·27169 (3)	0·39756 (3)	2·69 (3)	2·79 (3)	3·91 (3)	0·08 (2)	0·01 (2)	−0·11 (2)
Cl	0·68656 (11)	0·40849 (7)	0·15140 (8)	4·39 (5)	4·97 (6)	6·17 (6)	0·14 (4)	−0·07 (5)	−0·07 (5)
O(1)	0·1367 (3)	0·3508 (2)	0·4022 (2)	3·3 (1)	4·1 (1)	6·2 (2)	0·7 (1)	0·1 (1)	−0·8 (1)
O(2)	0·3081 (3)	0·3620 (1)	0·3253 (2)	4·0 (1)	3·0 (1)	5·4 (2)	0·0 (1)	−0·3 (1)	0·4 (1)
O(3)	0·1519 (3)	0·4661 (2)	0·3241 (3)	5·8 (2)	3·6 (2)	10·3 (3)	1·0 (1)	−1·3 (2)	0·0 (2)
O(4)	0·8336 (7)	0·3738 (6)	0·4033 (5)	9·7 (4)	30·9 (1·1)	15·2 (6)	3·0 (5)	4·6 (4)	3·7 (6)
O(5)	0·6481 (5)	0·3334 (2)	0·1083 (3)	11·7 (3)	5·6 (2)	11·0 (3)	−2·0 (2)	−4·1 (3)	−0·1 (2)
O(6)	0·5850 (6)	0·4319 (4)	0·2034 (4)	11·0 (4)	17·2 (6)	13·9 (5)	3·7 (4)	7·1 (4)	1·3 (4)
O(7)	0·8192 (4)	0·3958 (3)	0·2043 (3)	6·5 (2)	9·1 (3)	12·6 (4)	0·6 (2)	−3·6 (2)	−1·7 (3)
O(8)	0·7060 (6)	0·4699 (2)	0·0902 (3)	17·0 (5)	5·7 (2)	8·2 (3)	0·3 (3)	2·9 (3)	0·5 (2)
N(1)	0·4516 (3)	0·2094 (2)	0·3682 (2)	3·5 (1)	3·0 (2)	4·4 (2)	0·1 (1)	0·1 (1)	0·0 (1)
N(2)	0·3963 (3)	0·3331 (2)	0·4919 (2)	3·7 (2)	3·7 (2)	4·6 (2)	−0·3 (1)	0·1 (1)	−0·4 (1)
N(3)	0·2106 (3)	0·1957 (2)	0·4822 (2)	3·7 (2)	3·8 (2)	3·9 (2)	0·0 (1)	0·7 (1)	0·0 (1)
N(4)	0·1649 (3)	0·2145 (2)	0·3002 (2)	3·6 (2)	3·2 (2)	4·2 (2)	−0·1 (1)	0·1 (1)	0·1 (1)
C(1)	0·1959 (4)	0·3994 (2)	0·3487 (3)	4·0 (2)	3·4 (2)	6·2 (3)	0·1 (2)	−0·8 (2)	−1·0 (2)
C(11)	0·5158 (5)	0·2396 (3)	0·3030 (3)	5·2 (2)	4·4 (2)	5·4 (2)	0·8 (2)	1·7 (2)	0·8 (2)
C(12)	0·6342 (5)	0·2016 (3)	0·2770 (3)	6·3 (3)	6·1 (3)	6·8 (3)	1·0 (2)	2·7 (2)	0·3 (2)
C(13)	0·6870 (5)	0·1312 (3)	0·3174 (3)	4·8 (2)	6·1 (3)	7·2 (3)	1·6 (2)	1·2 (2)	−0·9 (2)
C(14)	0·6212 (5)	0·1008 (3)	0·3835 (3)	4·8 (2)	4·9 (2)	6·3 (3)	1·6 (2)	0·5 (2)	0·1 (2)
C(15)	0·5035 (4)	0·1404 (2)	0·4068 (3)	4·4 (2)	4·1 (2)	5·0 (2)	0·7 (2)	0·2 (2)	0·0 (2)
C(21)	0·5318 (4)	0·3550 (3)	0·4872 (3)	3·6 (2)	5·0 (2)	5·4 (2)	−0·5 (2)	−0·4 (2)	−0·2 (2)
C(22)	0·6055 (5)	0·4089 (3)	0·5466 (3)	4·5 (2)	5·7 (3)	5·9 (3)	−1·4 (2)	−1·0 (2)	0·0 (2)
C(23)	0·5392 (6)	0·4411 (3)	0·6119 (3)	7·2 (3)	5·8 (3)	5·0 (3)	−1·8 (2)	−0·7 (2)	−0·3 (2)
C(24)	0·4012 (6)	0·4175 (3)	0·6170 (3)	7·3 (3)	6·3 (3)	4·9 (3)	1·9 (2)	1·0 (2)	−1·6 (2)
C(25)	0·3322 (5)	0·3641 (3)	0·5568 (3)	5·4 (2)	5·4 (2)	4·9 (2)	−1·0 (2)	0·7 (2)	−1·1 (2)
C(31)	0·2923 (4)	0·1657 (2)	0·5525 (3)	5·0 (2)	4·2 (2)	3·8 (2)	0·3 (2)	0·6 (2)	−0·2 (2)
C(32)	0·2402 (5)	0·1125 (3)	0·6102 (3)	6·8 (3)	4·5 (2)	4·4 (2)	0·7 (2)	1·0 (2)	−0·2 (2)
C(33)	0·0976 (6)	0·0889 (3)	0·5944 (3)	7·6 (3)	4·8 (2)	5·3 (3)	−0·8 (2)	2·0 (2)	−0·1 (2)
C(34)	0·0132 (5)	0·1219 (3)	0·5242 (3)	5·3 (2)	6·1 (3)	6·4 (3)	−1·4 (2)	1·6 (2)	−0·6 (2)
C(35)	0·0715 (4)	0·1751 (3)	0·4698 (3)	3·7 (2)	5·4 (2)	5·1 (2)	−0·7 (2)	0·8 (2)	−0·5 (2)
C(41)	0·0699 (4)	0·2544 (2)	0·2424 (3)	4·5 (2)	3·5 (2)	4·7 (2)	−0·2 (2)	−0·4 (2)	0·5 (2)
C(42)	−0·0164 (4)	0·2150 (3)	0·1758 (3)	4·4 (2)	5·0 (2)	5·3 (2)	−0·6 (2)	−0·5 (2)	0·9 (2)
C(43)	−0·0063 (5)	0·1312 (3)	0·1683 (3)	5·0 (2)	5·1 (2)	5·4 (3)	−1·7 (2)	−0·1 (2)	−0·3 (2)
C(44)	0·0885 (4)	0·0891 (2)	0·2284 (3)	4·6 (2)	3·6 (2)	6·4 (3)	−0·5 (2)	0·1 (2)	−0·4 (2)
C(45)	0·1720 (4)	0·1320 (2)	0·2925 (3)	3·7 (2)	3·7 (2)	5·0 (2)	−0·2 (2)	0·1 (2)	−0·1 (2)

non-hydrogen atoms are listed in Table 2, and the approximate coordinates of the hydrogen atoms in Table 3.

Table 3. *Approximate hydrogen positions*

An isotropic temperature factor $U=0{\cdot}063$ Å^2 was used.

	x	y	z
H(11)	0·4737	0·2948	0·2710
H(12)	0·6869	0·2258	0·2253
H(13)	0·7789	0·1010	0·2976
H(14)	0·6633	0·0457	0·4157
H(15)	0·4512	0·1161	0·4586
H(21)	0·5840	0·3301	0·4355
H(22)	0·7143	0·4265	0·5424
H(23)	0·5944	0·4828	0·6585
H(24)	0·3494	0·4425	0·6688
H(25)	0·2234	0·3463	0·5608
H(31)	0·4034	0·1833	0·5637
H(32)	0·3074	0·0875	0·6660
H(33)	0·0534	0·0473	0·6377
H(34)	−0·0979	0·1044	0·5131
H(35)	0·0045	0·2001	0·4141
H(41)	0·0629	0·3201	0·2481
H(42)	−0·0914	0·2479	0·1394
H(43)	−0·0726	0·0988	0·1173
H(44)	0·0956	0·0233	0·2228
H(45)	0·2466	0·0989	0·3389

The input tapes for the diffractometer were produced on a GIER computer by *INDIFF* (Sørensen, unpublished). The rest of the calculations were performed on the IBM 360/75 computer at NEUCC, Lundtofte, Denmark, using the N.R.C. 2A Picker data reduction program (Ahmed, revised by Alcock and Sørensen) and *The X-ray System* (Stewart, Kundell & Baldwin 1970). The drawings were prepared by *ORTEP* (Johnson, 1965).

Description and discussion of the structure

The structure of carbonatotetrakis(pyridine)cobalt(III) perchlorate monohydrate is built up from discrete carbonatotetrakis(pyridine)cobalt(III) cations and perchlorate anions held together by electrostatic interaction. Fig. 1 shows the atomic labelling of the carbonatotetrakis(pyridine)cobalt(III) ion. Fig. 2 shows the content of a unit cell and the atomic labelling of the perchlorate ion and the water molecule.

Conformation of the complex ion

The interatomic distances and angles, uncorrected for thermal motion, are given in Table 4. As seen in Fig. 1 and 3, the angles in the octahedron are distorted. This is probably due to strain in the four-membered chelate ring and to space requirements of the pyridine molecules.

The four atoms of the carbonate group, the cobalt atom and the two nitrogen atoms N(1) and N(3) form one plane, the equatorial plane. The equation of this plane and the deviations of the atoms from it are given in Table 5. In the carbonatotetraminecobalt(III) ion (Barclay & Hoskins, 1962) and in the carbonatobis(trimethylenediamine)cobalt(III) ion (Geue & Snow, 1971), an angle of 7° was found between the carbonate group and the O–O–Co–N–N plane. Otherwise the dimensions of the coordinated carbonate group as well as the bond distances and angles of the coordination octahedron in carbonatotetrakis(pyridine)cobalt(III) ion are close to previously reported values.

The two pyridine rings in *trans* positions (rings 2 and 4) are almost perpendicular (83°) to each other and form angles of 80 and 3°, respectively, with a plane containing N(2), N(4), Co, C(1) and O(3). This plane is perpendicular (89°) to the equatorial plane. Ring 1 is almost coplanar with the equatorial plane (the angle is 8°). Ring 3 is rotated in a direction opposite to ring 1 so that it forms an angle of 52° with the equatorial plane. The cobalt atom in no case lies on the extension of the N–γC diagonal of the pyridine rings. The most striking deviation is that of ring 2, which forms an

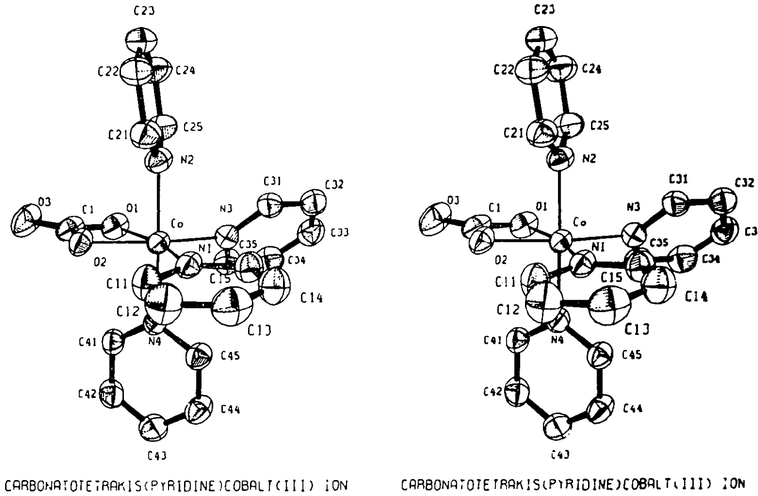

Fig. 1. Stereoscopic drawing of the complex ion. The thermal ellipsoids enclose 50% probability. The hydrogen atoms have been omitted for clarity.

angle of 13° with the normal to the equatorial plane. The planes of two of the pyridine rings (Table 5) contain the cobalt atom, and thus only exhibit in-plane bending. The most pronounced is the 5° bending of ring 1 towards the carbonate group. This clearly increases the ring 1–ring 3 distance. The shortest distance between α-carbon atoms in adjacent rings is the C(35)–C(45) distance of 3·123 Å, which is significantly shorter than the 3·21 Å in *trans*-tetrakis-(3,4-dimethylpyridine)nickel(II) perchlorate (Madaule-Aubry, Busing & Brown, 1968) and shorter than the 3·17 Å in μ-oxalatobis[tetrakis(pyridine)ruthenium(II)] ion (Cheng, Loescher & Nyburg, 1971), the only *cis*-tetrakis(pyridine) complex structure reported so far.

The arrangement of the pyridine rings is completely asymmetric. The crystals thus contain complex ions of opposite chirality.

The pyridine ligand

The pyridine rings are planar and identical within experimental error. Table 5 gives the equations for the least-squares planes through the pyridine molecules and the deviations of the atoms from it. Table 4 gives the bond distances and angles. The means and the estimated standard deviations of the means have been calculated assuming that the pyridine molecules have symmetry 2. The four crystallographically independent pyridine molecules are oriented differently in the crystal and the determinations of their atomic positions are thus affected differently by such systematic errors as absorption. So, the good internal agreement and the agreement with values obtained by microwave spectroscopy (Bak, Hansen-Nygaard & Rastrup-Andersen 1958) and from other pyridine derivatives (Downie, Harrison & Raper, 1972) show that the pyridine mol-

Table 4. *Interatomic distances* (Å) *and angles* (°)

Co–N(1)	2·004 (4)	O(1)····C(25)	2·835 (12)	C(1)–O(1)	1·330 (6)
Co–N(2)	1·974 (6)	O(1)····C(35)	3·146 (6)	C(1)–O(2)	1·319 (5)
Co–N(3)	1·996 (5)	O(1)····C(41)	2·940 (6)	C(1)–O(3)	1·210 (3)
Co–N(4)	1·985 (7)	O(2)····C(11)	2·857 (5)	Cl—O(5)	1·421 (4)
Co–O(1)	1·895 (3)	O(2)····C(21)	3·063 (13)	Cl—O(6)	1·393 (8)
Co–O(2)	1·892 (3)	O(2)····C(41)	3·004 (8)	Cl—O(7)	1·417 (7)
		C(11)···C(21)	3·420 (6)	Cl—O(8)	1·414 (5)
O(1)–Co–O(2)	69·28 (12)	C(15)···C(31)	3·250 (12)		
O(1)–Co–N(2)	87·16 (12)	C(15)···C(45)	3·387 (13)	C(1)–O(1)···O(4)	115·4 (3)
O(1)–Co–N(3)	94·85 (12)	C(25)···C(31)	3·263 (6)	O(1)–C(1)–O(2)	108·7 (3)
O(1)–Co–N(4)	91·07 (12)	C(35)···C(45)	3·123 (9)	O(1)–C(1)–O(3)	125·5 (4)
O(2)–Co–N(1)	94·97 (12)			O(2)–C(1)–O(3)	125·8 (4)
O(2)–Co–N(2)	86·84 (13)	Co–O(1)–C(1)	90·7 (2)		
O(2)–Co–N(4)	90·96 (12)	Co–O(2)–C(1)	91·2 (2)	O(5)–Cl—O(6)	111·1 (4)
N(1)–Co–N(2)	93·19 (12)	Co–N(1)–C(11)	116·3 (3)	O(5)–Cl—O(7)	107·3 (3)
N(1)–Co–N(3)	100·91 (12)	Co–N(1)–C(15)	125·5 (3)	O(5)–Cl—O(8)	110·0 (3)
N(1)–Co–N(4)	88·06 (13)	Co–N(2)–C(21)	121·5 (3)	O(6)–Cl—O(7)	109·1 (3)
N(2)–Co–N(3)	91·38 (14)	Co–N(2)–C(25)	119·6 (3)	O(6)–Cl—O(8)	111·2 (4)
N(3)–Co–N(4)	90·45 (13)	Co–N(3)–C(31)	123·9 (3)	O(7)–Cl—O(8)	108·0 (3)
		Co–N(3)–C(31)	123·9 (3)		
		Co–N(3)–C(35)	118·3 (3)		
		Co–N(4)–C(41)	122·2 (2)		
		Co–N(4)–C(45)	120·4 (2)		

Pyridine rings

N–αC		αC–βC		βC–γC	
N(1)–C(11)	1·348 (6)	C(11)–C(12)	1·389 (7)	C(12)–C(13)	1·371 (7)
N(1)–C(15)	1·337 (5)	C(14)–C(15)	1·379 (7)	C(13)–C(14)	1·368 (8)
N(2)–C(21)	1·342 (5)	C(21)–C(22)	1·392 (7)	C(22)–C(23)	1·372 (8)
N(2)–C(25)	1·347 (7)	C(24)–C(25)	1·377 (7)	C(23)–C(24)	1·373 (8)
N(3)–C(31)	1·344 (6)	C(31)–C(32)	1·388 (7)	C(32)–C(33)	1·391 (7)
N(3)–C(35)	1·346 (5)	C(34)–C(35)	1·381 (7)	C(33)–C(34)	1·372 (8)
N(4)–C(41)	1·349 (6)	C(41)–C(42)	1·388 (7)	C(42)–C(43)	1·377 (6)
N(4)–C(45)	1·354 (5)	C(44)–C(45)	1·377 (7)	C(43)–C(44)	1·385 (7)
Mean	1·346 (5)	Mean	1·384 (6)	Mean	1·376 (8)
Microwave	1·3402	Microwave	1·3945	Microwave	1·3944

N-vertex		αC-vertex		βC-vertex		γC-vertex	
N(1)	118·2 (3)	C(11)	121·4 (4)	C(12)	119·9 (5)	C(13)	118·4 (5)
		C(15)	122·4 (4)	C(14)	119·7 (4)		
N(2)	118·2 (3)	C(21)	121·7 (4)	C(22)	119·8 (4)	C(23)	118·1 (4)
		C(25)	121·9 (4)	C(24)	120·2 (5)		
N(3)	117·8 (4)	C(31)	122·8 (4)	C(32)	118·7 (4)	C(33)	118·4 (4)
		C(35)	122·3 (4)	C(34)	119·9 (4)		
N(4)	117·3 (3)	C(41)	122·9 (3)	C(42)	119·0 (4)	C(43)	118·8 (4)
		C(45)	122·6 (3)	C(44)	119·4 (4)		
Mean	117·9 (4)	Mean	122·3 (5)	Mean	119·6 (5)	Mean	118·4 (3)
Microwave	116·83	Microwave	123·88	Microwave	118·53	Microwave	118·33

ecule is not distorted even in a complex with pronounced steric hindrance. This also agrees, within the respective experimental error, with the values obtained in other tetrakis(pyridine) complexes.

Perchlorate ion

The perchlorate ion is a nearly perfect tetrahedron with dimensions close to previously reported values (Vijayan & Viswamitra, 1966). The average Cl–O distance is 1·411 (13) Å and the average O–Cl–O angle 109·5 (1·6)°. The thermal vibrations, which are relatively high, seem to be determined by the packing of the ions.

Water molecule

The water molecule – O(4) – forms no close contacts to the rest of the structure, in accordance with the relatively poor definition of its position and the high values of its thermal parameters. The closest approach of the water molecule is to the ligating oxygen O(1) of the carbonate group. This distance (2·89 Å) might be interpreted as a hydrogen bond although the hydrogen atom has not been located. The angle C(1)–O(1)···O(4) is 115° and O(4) is situated only 0·27 Å from the equatorial plane. The distance of 3·106 Å to O(7) of the perchlorate ion does not differ significantly from the sum of the van der Waals radii.

Packing

The closest intermolecular approaches (Table 6) are all between pyridine rings and oxygen atoms (either the non-ligating oxygen atom of the carbonate group or the oxygen atoms of the perchlorate ion).

Each perchlorate ion is surrounded by five complex ions and a water molecule and each complex ion by five perchlorate ions and a water molecule. The conformation of the complex ion is only slightly affected by the neighbour ions, though as mentioned above ring 2 is bent 13° probably due to close contacts to ring 1 (3.50 Å).

Table 5. *Least-squares planes*

The equations of the planes can be expressed as $Px+Qy+Rz=S$ in direct space.

Plane determined by:

	Pyridine ring 1	Pyridine ring 2	Pyridine ring 3	Pyridine ring 4	Carbonate group Co, N(1), N(2)
P	4·887	−2·388	−2·911	−7·531	4·530
Q	8·916	12·595	12·684	−1·769	7·230
R	9·067	−8·477	9·146	10·897	10·668
S	7·407	−0·925	6·297	1·658	7·489

Distances of the atoms from least-squares planes in Å and the corresponding standard deviation.

N(1)	0·005	N(2)	0·004	N(3)	−0·018	N(4)	−0·008	C(1)	0·005
C(11)	−0·002	C(21)	−0·002	C(31)	0·006	C(41)	0·007	O(1)	−0·043
C(12)	0·001	C(22)	−0·003	C(32)	0·011	C(42)	0·001	O(2)	−0·006
C(13)	−0·002	C(23)	0·007	C(33)	−0·017	C(43)	−0·008	O(3)	0·026
C(14)	0·004	C(24)	−0·005	C(34)	0·005	C(44)	0·007	Co	−0·005
C(15)	−0·006	C(25)	−0·001	C(35)	0·013	C(45)	0·001	N(1)	−0·001
Co*	−0·001	Co*	0·304	Co*	−0·036	Co*	0·069	N(3)	0·024
								O(4)*	0·270
e.s.d.	0·004	e.s.d.	0·004	e.s.d.	0·014	e.s.d.	0·007	e.s.d.	0·023

* Not included in the calculation of the plane.

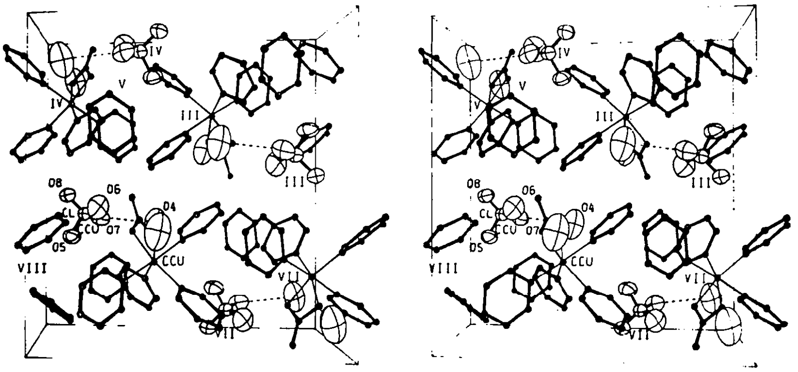

Fig. 2. Stereoscopic illustration of the contents of the unit cell. The view is down **a**, with **b** vertical and **c** horizontal. Thermal ellipsoids are shown for the perchlorate ion and the water molecule and enclose 50% probability.

Table 6. *Interionic distances* (Å) *between non-hydrogen atoms*

The first column contains atoms belonging to the crystal chemical unit (CCU), see Fig. 2.

Key to symmetry operations:

i:	$1+x$	y	z	v:	$\bar{x}$	$\frac{1}{2}+y$	$\frac{1}{2}-z$
ii:	$x-1$	y	z	vi:	$1+x$	$\frac{1}{2}-y$	$z-\frac{1}{2}$
iii:	$1-x$	$1-y$	$1-z$	vii:	x	$\frac{1}{2}-y$	$\frac{1}{2}+z$
iv:	$1-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$	viii:	x	$\frac{1}{2}-y$	$z-\frac{1}{2}$

O–O and O–C distances ≤3·5 Å

O(1)····O(4) (ii)	2·894 (7)
O(2)····C(32) (viii)	3·350 (7)
O(3)····C(22) (iii)	3·496 (11)
O(3)····C(23) (iii)	3·321 (9)
O(3)····C(43) (v)	3·037 (5)
O(3)····C(44) (v)	3·060 (7)
O(4)····O(7)	3·106 (10)
O(4)····C(21)	3·313 (12)
O(4)····C(22)	3·361 (14)
O(5)····C(12)	3·414 (7)
O(5)····C(15) (viii)	3·275 (11)
O(5)····C(31) (viii)	3·357 (9)
O(6)····C(32) (viii)	3·458 (12)
O(7)····C(33) (vi)	3·342 (12)
O(7)····C(41) (i)	3·305 (6)
O(7)····C(42) (i)	3·397 (6)
O(7)····C(44) (iv)	3·403 (6)
O(8)····C(14) (viii)	3·412 (9)
O(8)····C(15) (iv)	3·421 (6)
O(8)····C(45) (iv)	3·331 (7)

C–C distances ≤3·7 Å

C(1)····C(23) (iii)	3·609 (7)
C(12)···C(23) (viii)	3·495 (8)
C(12)···C(24) (viii)	3·651 (13)
C(13)···C(23) (viii)	3·517 (11)
C(21)···C(23) (iii)	3·693 (7)
C(22)···C(23) (iii)	3·611 (9)
C(24)···C(44) (vii)	3·632 (12)
C(32)···C(41) (vii)	3·533 (9)
C(33)···C(41) (vii)	3·479 (6)
C(33)···C(42) (vii)	3·663 (7)
C(34)···C(42) (vii)	3·597 (7)

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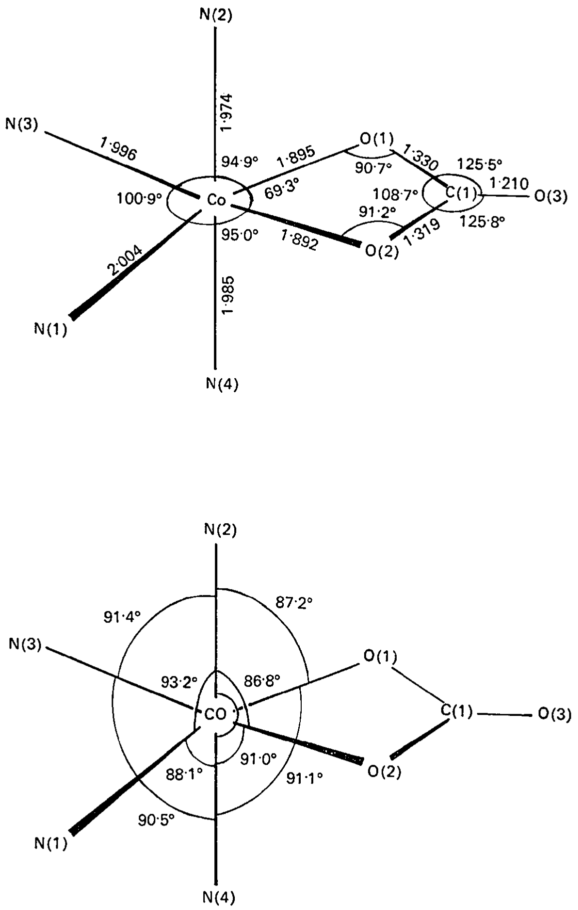

Fig. 3. Bond distances and angles of the coordination octahedron.

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