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The Crystal and Molecular Structure of μ-Oxalato-bis(tetrapyridineruthenium(II)) Fluoroborate

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The crystal structure of μ-oxalato-bis(tetrapyridineruthenium(II)) fluoroborate, $[Ru(C_5H_5N)_4C_2O_4Ru(C_5H_5N)_4](BF_4)_2$, has been determined. The compound crystallizes in the monoclinic space group $P2_1/c$ with $a = 10.926$ (5), $b = 16.740$ (7), $c = 13.732$ (5) Å, and $\beta = 116.51$ (1)° with two molecules per unit cell. $\rho_{obsd} = 1.60$ and $\rho_{calcd} = 1.62$ g cm^{-3} for $Z = 2$. Least-squares refinement of 3165 observed reflections collected by counter methods has yielded a final conventional R factor of 0.071 and a weighted R factor of 0.069. The two ruthenium atoms of the molecule are linked to a centrosymmetric planar tetradentate oxalate ligand. The coordinating ligands form slightly distorted octahedra about each ruthenium atom consisting of two oxygen atoms from the oxalate ligand and four nitrogen atoms from the pyridine ligands. Several features of the structure indicate that the arrangement of four pyridine molecules in the cis configuration causes significantly greater steric interaction than in comparable *trans*-tetrapyridine complexes. This result is in accord with the observed experimental difficulties in preparing *cis*-tetrapyridine complexes. The fluoroborate anion is disordered about one of the threefold B–F axes of the tetrahedron. The unique undisordered fluorine atom is probably held in position by means of a hydrogen bond to an α hydrogen of one of the pyridine rings. The bond lengths and angles of the oxalate ligand are not significantly different from those of the oxalate ion in potassium oxalate monohydrate. The average Ru–N bond distance of 2.08 Å is significantly shorter than that of 2.13 Å in chlorotetraammine(sulfur dioxide)ruthenium(II) chloride.

Introduction

Dinuclear transition metal complexes containing a bridging tetradentate oxalate ligand have been known for several years. The first conclusive evidence was presented in 1938[1] when a partial X-ray analysis of the tri-*n*-butylphosphine complex $(C_4H_9)_3P(Cl)PdC_2O_4Pd(C_4H_9)_3P(Cl)^0$ revealed the Pd···Pd distance to be 5.48 Å. This is quite close to the required distance of 5.3 Å for a bridging oxalate. This result refuted the earlier postulate of bridging chloro ligands which would require a Pd···Pd separation of only 3.4 Å.

The first complete structure analysis of a molecule containing a bridging oxalate was that of the mineral humboltine, $FeC_2O_4 \cdot 2H_2O$, in 1957.[2] The iron atoms are linked by planar bridging tetradentate oxalate ligands, forming planar polymeric chains with the two water molecules completing the octahedron about each iron. Preliminary reports on the structures of the complexes $Cu(NH_3)_2C_2O_4$,[3] $(NH_4)_2[(UO_2)_2(C_2O_4)_3]$,[4] and $Ti_2(C_2O_4)_3 \cdot 10H_2O$[5] have also shown the oxalate ligand to be four-coordinate. In the uranyl complex, two-thirds of the oxalates are tridentate. Tetradentate oxalate complexes have also been claimed for several other transition metals.[6–8]

Almost every two- or three-valent transition metal which supports an octahedral configuration of ligands forms complexes with the stoichiometry $M(C_5H_5N)_4X_2^{n+}$ where X is an anionic ligand and n is 0 or 1.[9,10] Despite several attempts and claims of preparation,[11–13] only one of these complexes displays cis–trans isomerism: dichlorotetrapyridineiridium(III) chloride.[11]

In an attempt to prepare *cis*-tetrapyridine complexes of ruthenium(II), hexapyridineruthenium(II) salts were treated with oxalic acid. Since ruthenium(II) has a low affinity for oxygen donor ligands it was hoped that oxalate could be readily displaced under mild conditions forming cis complexes. Physical data indicated that the complex formed was probably $[(C_5H_5N)_4RuC_2O_4Ru(C_5H_5N)_4](BF_4)_2$.[14] The oxalate could not be displaced from this complex without employing reaction conditions which brought about accompanying loss of pyridine.

It was decided that an X-ray structural analysis of this complex would present an excellent opportunity to study both the bridging tetradentate oxalate ligand and the configuration and interactions of four pyridine ligands in the cis octahedral configuration.

Experimental Section

The complex crystallizes overnight from methanol solution as large, deep-red parallepipeds. The crystals are invariably twinned but after partial re-solution in methanol single-crystal fragments can be obtained.

Anal. Calcd for $C_{42}H_{40}N_8O_4Ru_2B_2F_8$: C, 45.99; H, 3.65; N, 10.22; Ru, 18.45. Found: C, 46.34; H, 3.76; N, 10.13; Ru, 18.68.

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TABLE I
OBSERVED AND CALCULATED STRUCTURE, FACTORS (10 × ABSOLUTE)

L	FO	FC
H= 0, K= 0		
2	1926	-1971
4	373	366
6	151	172
8	1292	-1264
10	814	811
14	125	-122
H= 0, K= 1		
13	346	-370
12	112	-46
11	589	567
9	288	-275
8	105	-103
7	272	-304
5	114	97
4	555	499
3	284	272
2	291	-315
1	1075	1146
H= 0, K= 2		
0	1982	2107
1	933	-978
2	1148	-1042
3	549	511
4	756	717
5	288	293
6	368	355
7	155	-169
8	767	-730
9	417	362
10	585	559
11	270	-230
14	136	-112
H= 0, K= 3		
13	389	-365
12	436	-434
11	540	557
10	169	160
9	266	-282
8	227	225
7	198	-197
6	476	-463
5	1291	1225
4	409	399
3	1452	-1422
2	1830	-1842
1	556	563
H= 0, K= 4		
0	373	352
1	453	479
2	214	-289
3	124	-140
4	270	324
5	467	477
6	276	271
7	498	-509
8	254	-241
9	430	409
10	412	440
11	241	-268
13	126	-100
14	177	-163
H= 0, K= 5		
14	218	208
13	267	-285
12	526	-546
11	218	175
10	155	200
9	165	-173
8	152	127
6	993	-990
5	1029	956
4	1720	1667
3	248	-221
2	853	-919
1	190	160
H= 0, K= 6		
0	295	228
1	832	-804
2	1130	-1119
3	274	248
4	409	432
5	549	562
6	567	535
7	683	-684
8	273	-265
9	521	560
10	191	165
11	291	-309
12	115	-99
H= 0, K= 7		
13	169	-187
12	451	-473
9	135	53
8	241	229
7	204	156
6	785	-781
5	159	-120
4	863	849
3	362	333
2	574	-547
1	654	-641
H= 0, K= 8		
0	171	-162
1	674	-698
2	476	-468
3	702	682
4	208	-192
7	1025	-1025
9	478	478
10	105	75
11	295	-346
12	133	-124
H= 0, K= 9		
12	338	-334
11	95	-57
10	129	129
7	370	411
6	677	-684
4	1057	1019
2	544	-534
1	447	-400
H= 0, K= 10		
0	717	-678
1	421	-452
2	226	191
3	236	260
5	225	231
6	258	-233
7	551	-536
8	326	300
9	357	357
10	183	-167
11	289	-287
12	108	-54
H= 0, K= 11		
12	130	-233
11	158	-130
10	241	261
8	212	205
6	455	-496
5	562	-589
4	746	711
3	196	200
2	785	-762
1	102	-78
H= 0, K= 12		
0	446	-435
1	284	-286
2	408	393
3	130	80
4	155	-149
6	461	-452
7	313	-357
8	232	238
9	262	222
10	354	-333
H= 0, K= 13		
11	137	-259
8	197	199
6	321	-373
5	579	-563
4	274	262
3	352	348
2	391	-413
1	121	-109
H= 0, K= 14		
0	649	-663
1	339	-350
2	469	494
4	133	-111
6	399	-346
7	158	-109
8	393	390
9	199	181
10	302	-309
H= 0, K= 15		
9	180	93
8	113	100
7	176	169
6	103	-63
5	513	-520
3	385	349
2	136	-107
1	185	-176
H= 0, K= 16		
0	661	-670
1	119	18
2	431	402
3	101	-13
4	127	-75
6	271	-304
8	330	350
H= 0, K= 17		
7	122	139
6	196	169
5	399	-397
4	146	-169
3	349	360
1	186	-163
H= 0, K= 18		
0	332	-316
1	178	144
2	235	243
6	220	-159
H= 0, K= 19		
4	179	-185
3	303	269
2	209	186
1	137	-95
H= 1, K= 19		
1	128	-82
2	139	145
3	273	239
-4	197	233
-3	254	260
-2	244	-213
-1	130	-133
H= 1, K= 18		
2	280	276
0	400	-402
-1	210	-173
-2	306	309
-4	125	-122
-6	121	-125
H= 1, K= 17		
3	479	426
5	325	-320
6	160	121
7	164	133
-5	408	-410
-3	347	341
-2	111	-109
-1	184	-173
H= 1, K= 16		
8	335	315
6	262	-272
5	93	-65
2	271	250
0	497	-466
-1	104	20
-2	422	431
-4	119	-81
-6	315	-308
-8	281	280
H= 1, K= 15		
1	189	-197
2	248	-155
3	351	357
5	572	-573
6	97	-64
7	136	99
8	106	55
9	253	62
-10	98	-56
-8	109	-91
-7	263	259
-5	408	-419
-3	337	347
-2	227	208
-1	111	-90
H= 1, K= 14		
9	98	112
9	370	377
7	102	-111
6	358	-366
5	110	-23
4	165	-162
3	156	-139
2	355	341
1	112	-68
0	600	-633
-1	130	137
-2	480	479
-3	128	142
-4	192	-115
-5	144	-105
-6	271	-305
-8	345	341
-9	205	-208
-10	320	-324
H= 1, K= 13		
0	166	211
1	272	-292
2	335	-321
3	545	535
4	109	186
5	465	-473
6	374	-395
7	117	114
-11	262	-293
-9	148	138
-8	277	-284
-7	186	189
-6	280	315
-5	618	-608
-4	409	-396
-3	480	493
-2	289	302
-1	221	-192
H= 1, K= 12		
11	151	-114
10	301	-286
9	316	286
8	420	425
7	397	-414
6	317	-295
4	213	-208
3	210	235
2	484	470
1	449	-392
0	581	-565
-1	390	399
-2	537	519
-4	382	-374
-5	152	-131
-6	293	-294
-7	374	370
-8	219	227
-9	355	-376
-10	226	-239
-12	151	154
H= 1, K= 11		
2	412	-379
3	259	263
4	529	549
5	412	-409
6	400	-436
9	129	54
10	206	194
11	180	-147
-12	300	322
-11	278	-264
-10	109	-13
-9	193	213
-8	241	-252
-7	260	252
-6	634	617
-5	506	-538
-4	819	-805
-3	313	318
-2	461	467
-1	236	-261
H= 1, K= 10		
12	409	-64
11	267	-266
10	177	-174
9	502	510
8	239	214
7	406	-350
6	338	-341
5	284	269
4	99	16
3	431	443
2	149	80
1	712	-690
0	643	-615
-1	592	625
-2	220	203
-3	564	-561
-4	118	85
-5	110	-125
-6	114	-121
-7	444	639
-8	271	245
-11	271	321
-12	92	-51
-13	87	-20
H= 1, K= 9		
0	305	-249
1	400	-377
2	470	-485
3	114	105
4	896	905
5	194	-192
6	590	-621
10	333	332
11	92	-90
12	372	-388
-13	125	129
-12	278	277
-10	103	-114
-9	114	-150
-7	200	194
-6	605	569
-5	211	-236
-4	1146	-1122
-3	345	351
-2	396	396
-1	141	-156
H= 1, K= 8		
12	110	-80
11	237	-222
7	679	-680
5	643	635
4	234	235
3	386	369
2	207	-195
1	892	-830
-1	984	959
-2	261	-255
-3	608	-646
-4	322	311
-5	303	-278
-7	856	898
-8	140	122
-9	820	-852
-11	274	294
-12	217	-221
H= 1, K= 7		
0	511	504
2	1034	-987
3	202	-215
4	1056	1026
5	344	296
6	719	-738
7	133	59
8	193	200
9	122	175
10	270	282
11	89	32
-14	309	-277
-13	141	-172
-12	360	342
-10	152	-209
-9	276	-260
-8	166	-166
-7	161	145
-6	627	634
-5	234	192
-4	1016	-996
-3	88	-12
-2	1048	1070
-1	513	-523
H= 1, K= 6		
13	107	-124
11	293	-285
9	490	493
8	221	-236
7	565	-590
6	582	588
3	581	588
2	676	-668
1	760	-737
0	400	388
-1	782	752
-2	431	-418
-3	240	-166
-4	338	324
-5	426	-424
-6	742	787
-7	804	777
-8	384	-360
-9	544	-528
-11	423	423
-12	112	-77
H= 1, K= 5		
0	149	110
1	138	103
2	724	-448
3	850	-806
4	946	1146
5	942	915
6	326	-311
8	235	201
10	163	197
12	394	-390
13	276	-270
-14	344	-311
-13	261	-295
-12	387	393
-11	238	221
-10	204	-200
-9	135	-116
-8	115	-135
-7	205	-222
-6	1019	999
-5	555	526
-4	1128	-1110
-3	153	-135
-1	365	-335
H= 1, K= 4		
11	325	-310
10	333	357
9	458	453
8	442	-413
7	511	-534
6	111	-81
5	391	328
4	300	270
3	308	291
2	228	-243
1	1463	-1443
0	1587	1540
-1	469	487
-2	828	-818
-3	531	-549
-4	86	-23
-6	357	333
-7	560	520
-8	468	-449
-9	561	-556
-10	430	415
-11	296	281
H= 1, K= 3		
0	728	669
1	1084	1068
2	508	-485
3	1395	-1228
4	598	616
5	935	894
6	684	-659
7	160	-119
8	231	191
9	460	-229
10	167	191
11	506	530
12	273	-263
13	248	-272
-14	225	-199
-13	460	-440
-12	286	295
-11	487	470
-10	191	-149
-9	365	-354
-7	350	-349
-5	960	923
-4	552	-582
-3	1242	-1192
-2	1175	1225
-1	771	861
H= 1, K= 2		
13	84	-71
10	595	605
9	249	270
8	558	-527
7	371	-378
6	516	462
5	186	198
4	517	472
3	835	-767
2	1339	-1276
1	198	210
0	1909	2147
-1	682	672
-2	1375	-1422
-3	304	352
-4	598	440
-5	144	144
-6	286	294
-7	521	502
-8	833	-848
-9	625	-613
-10	636	629
-12	218	-226
-13	155	144
-14	113	-65
-15	128	-139
H= 1, K= 1		
1	932	947
2	735	734
3	793	-758
4	560	539
5	689	647
6	411	-428
7	318	-326
8	108	119
9	159	-144
11	515	504
12	89	-97
13	379	-372
14	225	70
-15	309	267
-14	100	-64
-13	398	-415
-11	775	784
-10	119	-59
-9	262	-209
-8	172	-72
-7	698	-689
-6	96	123
-5	811	783
-4	670	647
-3	421	-376
-2	101	59
-1	653	767
H= 1, K= 0		
12	111	89
10	591	598
8	987	-981
6	561	562
4	715	674
2	546	-447
0	1755	1848
-2	1466	-1596
-4	987	-931
-6	283	315
-8	782	-791
-10	953	947
-12	152	-167
-14	119	-90
H= 2, K= 0		
0	1561	1625
2	585	-590
4	117	101
6	661	635
8	856	-851
10	389	383
12	107	-116
-12	146	-174
-8	873	-887
-6	423	431
-4	471	-404
-2	1875	-2030
H= 2, K= 1		
13	341	-357
12	124	-122
11	354	352
10	100	-41
9	121	-164
8	169	130
7	346	-304
5	1113	1116
4	83	110
3	1244	-1139
2	180	-171
1	1659	1650
0	1107	-1129
-1	610	651
-2	119	-102
-3	1880	-1979
-4	534	-485
-5	473	684
-6	211	221
-7	661	-667
-8	126	88
-11	759	766
-12	115	95
-13	407	-409
-14	114	-95
-15	299	279
H= 2, K= 2		
0	865	865
1	1150	-1142
2	352	-374
3	146	116
4	126	-123
5	717	721
6	449	398
7	386	-406
8	651	-644
9	181	128
10	289	268
11	197	-153
-15	147	-93
-14	87	-11
-12	165	-141
-10	753	724
-9	246	-198
-8	823	-857
-7	245	279
-6	700	688
-5	76	-66
-4	451	406
-3	1513	-1602
-2	343	89
-1	1460	1558
H= 2, K= 3		
13	263	-296
12	247	-221
11	365	367
10	173	129
8	174	157
7	619	-636
6	148	-183
5	1041	1051
4	633	597
3	1385	-1353
2	196	-132
1	632	689
0	741	-779
-1	882	851
-2	158	-131
-3	241	-268
-4	1112	-1080
-5	1218	1200
-6	337	252
-7	615	-583
-9	133	-97
-10	142	-141
-11	500	496
-12	347	340
-13	398	-405
-14	241	-261
-15	228	300
H= 2, K= 4		
0	269	285
1	1248	-1230
3	587	572
4	236	-205
5	369	338
6	587	585
7	305	-317
8	412	-422
9	389	426
10	131	107
11	216	-246
12	125	69
13	167	-104
-15	272	-261
-14	127	-114
-12	99	-93
-11	268	243
-10	393	371
-9	544	-504
-8	486	-471
-7	496	480
-6	582	611
-5	175	-171
-3	398	-403
-2	781	-784
-1	702	657
H= 2, K= 5		
12	296	-283
11	223	197
10	385	340
9	132	146
8	134	-115
7	394	-430
6	287	-302
5	463	473
4	962	953
3	895	-785
2	1506	-1448
1	80	1
0	148	134
-1	245	214
-2	256	-261
-3	730	-716
-4	1326	-1309
-5	740	767
-6	991	1014
-7	589	-534
-8	220	-210
-9	146	117
-10	204	-187
-11	284	280
-12	354	331
-13	317	-292
-14	344	-316
-15	166	199
H= 2, K= 6		
0	104	63
1	1543	-1495
2	276	-254
3	503	462
4	111	93
5	125	136
6	331	304
7	481	-484
8	217	-233
9	518	562
10	185	183
11	296	-307
12	100	50
-14	94	-16
-13	111	91
-12	155	-154
-11	296	315
-10	141	139
-9	730	-739
-8	169	-160
-7	893	904
-6	179	142
-5	330	-306
-4	238	236
-3	527	-510
-2	405	-371
-1	584	537
H= 2, K= 7		
12	420	-416
10	257	310
8	135	-132
7	152	-147
6	509	-507
5	129	77
4	748	735
3	171	-134
2	459	-439
1	172	-162
0	142	125
-1	87	59
-2	219	256
-3	127	-147
-4	1168	-1183
-5	405	421
-6	644	649
-7	176	156
-8	454	-450
-9	126	72
-12	364	325
-13	114	-97
-14	319	-319
H= 2, K= 8		
1	1092	-1057
3	445	400
4	133	83
5	257	283
7	547	-499
8	115	116
9	537	561
11	283	-304
12	101	-12
-14	125	-67
-13	90	3
-12	160	-158
-11	388	392
-9	900	-912
-7	620	618
-5	300	-290
-4	220	183
-3	521	-548
-2	86	-37
-1	921	949
H= 2, K= 9		
8	105	-23
7	216	215
6	470	-472
5	225	-174
4	649	619
3	121	-128
2	323	-330
1	100	24
-1	363	-416
-2	495	470
-3	270	248
-4	757	-773
-5	111	-184
-6	709	692
-7	291	272
-8	134	-148
-10	280	-254
-11	265	-261
-12	238	256
-13	102	86
-14	364	-346
H= 2, K= 10		
0	438	-441
1	758	-769
5	174	157
6	215	-178
7	570	-591
8	296	300
9	506	485
10	117	-130
11	109	-160
-12	114	27
-11	311	297
-10	371	-349
-9	580	-557
-7	454	447
-5	226	-227
-4	110	-108
-3	376	-387
-2	341	307
-1	942	942
H= 2, K= 11		
10	182	184
7	191	185
6	363	-365
5	302	-303
4	240	229
3	450	448
2	448	-447
1	321	-291
0	286	278
-1	327	-323
-2	525	511
-3	461	474
-4	524	-551
-5	396	-389
-6	435	431
-7	120	99
-8	158	-160
-10	187	-191
-11	363	-355
-12	326	347
-13	211	180
H= 2, K= 12		
0	467	-439
1	385	-331
2	329	311
5	161	93
6	200	-216
7	401	-423
8	310	304
9	278	293
10	191	-218
-12	132	147
-11	194	170
-10	277	-252
-9	358	-393
-8	372	398
-7	227	188
-6	184	-236
-5	245	-251
-4	376	-389
-2	381	409
-1	538	536
H= 2, K= 13		
9	139	117
7	153	149
6	235	-255
5	301	-326
2	151	-112
1	243	-221
0	157	128
-1	137	-126
-2	301	271
-3	375	354
-4	409	-426
-5	449	-476
-6	350	317
-11	238	-262
-12	154	116
H= 2, K= 14		
0	638	-629
1	164	-150
2	392	389
4	114	-113
6	302	-284
7	110	-52
8	268	266
-10	330	-340
-9	230	-235
-8	340	321
-7	141	144
-6	402	-398
-4	99	-123
-3	124	-91
-2	496	522
-1	264	264
H= 2, K= 15		
7	145	104
6	105	-95
5	497	-493
4	200	171
3	492	500
2	154	-167
1	244	-261
-1	110	-49
-2	165	126
-3	444	470
-5	582	-585
-6	94	21
-7	175	204
-8	104	-48
-9	84	-65
H= 2, K= 16		
0	433	-395
2	354	351
4	127	-82
5	94	-49
6	281	-304
-9	90	-19
-8	282	284
-6	286	-275
-5	123	109
-4	215	-197
-3	103	58
-2	469	470
H= 2, K= 17		
6	224	110
5	294	-309
4	128	-108
3	458	462
2	110	88
1	180	-188
-1	156	-199
-2	168	-169
-3	335	343
-4	185	176
-5	407	-429
-7	216	206
H= 2, K= 18		
0	346	-318
1	116	82
2	232	203
3	105	-11
-7	120	-101
-5	113	58
-4	162	-145
-3	170	148
-2	290	267
-1	104	-116
H= 2, K= 19		
2	143	146
-2	138	-116
-3	178	191
-4	196	215
-5	296	-251
H= 3, K= 19		
-4	101	182
-3	185	178
H= 3, K= 18		
2	203	180
0	296	-306
-1	148	-93
-2	249	224
-3	127	99
-4	192	-169
-5	129	94
-6	122	-167
-7	156	-96
H= 3, K= 17		
1	317	-307
2	122	95
3	427	415
4	102	-65
5	313	-319
-7	232	237
-5	475	-471
-4	207	188
-3	330	338
-1	165	-144
H= 3, K= 16		
6	163	-214
5	91	37
3	110	64
2	406	378
0	251	-268
-1	110	40
-4	136	-151
-6	289	-289
-8	391	382
-9	102	-42
H= 3, K= 15		
1	276	-271
5	394	-387
-10	118	-9
-7	218	196
-5	579	-586
-3	271	319
H= 3, K= 14		
8	258	311
7	107	-88
6	282	-300
2	327	315
1	144	-123
0	585	-547
-1	300	273
-2	549	548
-4	110	-49
-6	426	-450
-7	122	95
-8	431	414
-9	173	-166
H= 3, K= 13		
1	479	-424
2	110	-106
3	253	261
4	310	306
5	444	-447
7	203	227
-12	187	186
-10	159	-102
-9	163	-129
-7	254	236
-6	253	243
-5	363	-376
-4	193	-230
-3	264	326
-1	247	-225
H= 3, K= 12		
9	234	298
8	334	333
6	348	-338
5	103	151
3	152	146
2	153	109
1	350	-341
0	772	-753
-1	647	612
-2	542	555
-3	468	-465
-4	172	-171
-6	207	-214
-7	153	166
-9	291	-265
-10	524	-504
-11	232	249
-12	142	79
H= 3, K= 11		
1	338	-343
2	367	-350
3	191	174
4	378	393
5	428	-416
6	310	-355
7	179	160
10	253	184
-13	190	191
-12	379	374
-11	293	-318
-10	311	-256
-8	231	-212
-6	375	369
-5	337	-313
-3	489	487
-2	502	514
-1	171	-214
H= 3, K= 10		
10	102	-117
9	452	444
8	166	169
7	559	-563
6	159	-95
5	124	59
4	114	-13
3	105	15
1	712	-704
0	358	-336
-1	883	836
-2	264	241
-3	355	-356
-4	174	-59
-5	140	187
-7	240	225
-9	514	-497
-10	326	-296
-11	394	398
-12	120	97
H= 3, K= 9		
0	128	118
1	458	-488
2	642	-615
3	311	316
4	473	461
6	443	-423
8	131	-87
9	97	68
10	292	324
-14	330	-336
-11	322	-329
-10	362	-372
-9	98	91
-8	183	-163
-6	611	653
-5	134	-144
-4	785	-823
-3	104	101
-2	497	540
-1	130	104
H= 3, K= 8		
11	182	-162
10	92	15
9	423	435
8	103	-37
7	599	-624
5	100	80
4	117	-78
1	868	-855
0	312	-317
-1	1397	1427
-2	236	-238
-3	801	-818
-4	141	-136
-5	374	-359
-7	503	502
-8	108	38
-9	520	-495
-11	475	458
H= 3, K= 7		
0	106	84
1	183	179
2	851	-851
3	282	-284
4	665	677
5	137	63
6	273	-275
7	100	10
8	138	-143
9	102	-101
10	299	308
-14	382	-390
-13	143	-129
-12	501	519
-11	130	103
-10	162	-154
-8	276	-264
-7	158	-189
-6	594	639
-5	321	347
-4	971	-1004
-3	348	-398
-2	222	168
-1	160	157
H= 3, K= 6		
11	202	-232
10	121	126
9	414	408
8	222	-232
7	462	-474
6	152	118
5	135	48
4	125	-70
3	254	247
2	214	-198
1	1319	-1299
0	158	189
-1	1085	1033
-2	361	-331
-3	718	-743
-4	120	99
-5	297	-290
-6	112	108
-7	599	644
-8	105	-142
-9	759	-771
-11	357	361
-15	262	-277
H= 3, K= 5		
0	699	-653
1	888	844
2	718	-754
3	225	-226
4	632	650
5	565	572
6	373	-383
7	249	-261
8	202	-201
9	117	-51
10	218	228
11	176	156
12	375	-394
-15	118	162
-14	346	-367
-13	344	-352
-12	232	221
-11	255	277
-10	220	-199
-9	200	198
-8	131	-109
-7	344	-333
-6	1231	1266
-5	859	860
-4	855	-821
-3	1005	-1035
-2	494	496
-1	143	151
H= 3, K= 4		
11	287	-305
10	158	194
9	320	347
8	373	-367
7	382	-394
6	509	506
5	326	298
3	149	161
2	223	-237
1	873	-827
0	836	790
-1	401	400
-2	302	-293
-3	667	-686
-4	181	-219
-5	246	271
-6	479	494
-7	388	423
-8	476	-496
-9	402	-430
-10	534	546
-11	282	317
-13	158	-154
-14	151	-140
-15	191	-190
H= 3, K= 3		
0	444	-448
1	1170	1089
2	390	-372
3	723	-714
4	543	522
5	828	828
6	112	-84
7	549	-556
8	245	-233
11	216	201
12	301	-300
-15	283	273
-14	253	-271
-13	356	-305
-12	379	421
-11	389	364
-10	248	-250
-9	319	318
-7	675	-670
-6	1194	1162
-5	860	888
-4	951	-969
-3	1197	-1228
-2	549	481
-1	405	371
H= 3, K= 2		
11	161	-160
10	206	219
9	200	216
8	621	-622
7	269	-284
6	695	702
5	397	399
4	289	-274
3	453	422
2	179	-107
1	717	-749
0	1199	1201
-1	363	-350
-2	605	-610
-3	478	-577
-4	571	521
-5	215	229
-6	851	783
-7	98	-13
-8	1029	-1037
-9	112	-89
-10	725	740
-12	108	-130
-13	97	-61
-14	136	-77
-15	99	-53
H= 3, K= 1		
0	327	-392
1	453	444
2	261	274
3	1227	-1247
4	91	-27
5	939	935
6	117	-126
7	372	-357
11	344	356
12	104	-55
-15	348	341
-14	119	-174
-13	482	-475
-12	200	192
-11	523	506
-10	209	-230
-9	107	67
-7	439	-417
-6	310	327
-5	1349	1305
-4	514	-451
-3	323	-339
-2	76	-82
-1	443	481
H= 3, K= 0		
10	370	409
8	458	-463
6	868	859
2	751	-736
0	1051	1048
-2	85	59
-4	1044	1066
-6	827	837
-8	913	-883
-10	820	839
-12	191	-163
-14	178	-179
H= 4, K= 19		
-2	150	-132
H= 4, K= 18		
2	215	175
1	124	145
0	257	-267
-1	177	-157
-2	202	213
-4	164	-122
-5	109	61
H= 4, K= 17		
1	267	-253
2	120	120
3	398	401
-7	230	245
-6	95	-87
-5	429	-409
-4	165	136
-3	234	251
H= 4, K= 16		
3	98	61
2	228	239
0	402	-410
-1	122	-109
-2	163	143
-6	249	-265
-8	435	438
H= 4, K= 15		
1	364	-364
3	547	560
4	126	68
5	376	-383
-8	106	-77
-7	375	349
-6	195	203
-5	627	-654
-3	276	284
H= 4, K= 14		
6	228	-221
2	258	274
1	363	-334
0	451	-436
-1	203	238
-2	245	242
-3	148	-116
-4	506	-480
-5	102	-26
-7	120	119
-8	477	470
-10	387	-365
H= 4, K= 13		
1	420	-386
2	297	-280
3	297	282
4	180	150
5	343	-341
6	123	-102
7	194	177
-12	181	229
-11	218	-213
-10	90	-94
-9	150	-131
-8	213	-178
-7	259	289
-6	287	318
-5	331	-369
-4	206	-239
-3	427	430
-2	109	141
H= 4, K= 12		
8	260	270
7	297	-309
6	241	-263
5	135	129
3	181	166
1	219	-252
0	534	-521
-1	628	622
-2	392	363
-3	485	-529
-6	295	-257
-7	214	173
-8	327	321
-9	350	-308
-10	417	-428
-11	196	213
-12	88	71
-13	137	-24
H= 4, K= 11		
1	257	-251
2	201	-175
3	123	107
4	325	326
5	379	-340
6	228	-244
9	134	107
-13	231	217
-12	402	407
-11	189	-195
-10	227	-254
-7	157	192
-6	120	115
-5	396	-407
-4	600	-611
-3	415	419
-2	570	591
H= 4, K= 10		
9	287	308
8	198	243
7	464	-476
6	176	-197
5	103	110
4	133	-150
2	128	79
1	298	-244
0	335	-353
-1	1272	1305
-2	357	384
-3	682	-717
-4	119	132
-6	149	-113
-7	116	-140
-8	140	157
-9	527	-548
-10	303	-298
-11	406	399
-12	154	96
-14	154	84
H= 4, K= 9		
0	130	119
1	274	-243
2	490	-487
3	136	104
4	378	355
5	188	-180
6	393	-408
8	127	-114
9	90	16
10	218	191
-14	344	-360
-13	98	103
-12	573	560
-10	303	-311
-9	231	-206
-8	345	-335
-7	170	181
-6	472	490
-5	113	-103
-4	911	-938
-3	202	216
-2	651	663
H= 4, K= 8		
9	372	403
8	110	23
7	571	-606
5	110	157
4	211	-203
3	371	385
2	129	77
1	753	-757
-1	805	831
-3	669	-700
-4	200	176
-5	149	-163
-7	447	470
-8	177	153
-9	454	-455
-11	345	376
-12	173	-133
-13	93	-93
H= 4, K= 7		
0	169	114
1	94	86
2	716	-729
4	614	611
6	367	-379
7	130	-136
10	230	250
-14	473	-488
-13	241	-242
-12	545	572
-10	177	-170
-9	144	108
-8	443	-448
-7	95	-128
-6	839	847
-4	737	-791
-3	475	-463
-2	461	506
-1	164	136
H= 4, K= 6		
10	100	108
9	283	267
8	405	-394
7	446	-416
6	114	127
5	383	384
4	147	92
1	624	-631
0	279	266
-1	910	870
-3	627	-622
-4	130	-99
-5	111	103
-6	400	427
-7	640	629
-8	179	-184
-9	569	-611
-10	307	335
-11	413	410
-12	154	-176
-15	282	-286
H= 4, K= 5		
0	214	-231
1	358	314
2	489	-513
3	349	-363
4	787	803
5	356	337
6	350	-341
7	111	-90
9	163	-140
10	220	282
11	116	144
-15	173	177
-14	371	-367
-13	306	-317
-12	338	341
-11	122	103
-10	543	-561
-9	165	172
-7	176	-156
-6	790	827
-5	750	765
-4	705	-730
-3	331	-292
-2	669	656
-1	224	-245
H= 4, K= 4		
11	175	-141
10	222	243
9	187	210
8	324	-390
7	423	-422
6	264	283
5	345	342
2	173	-185
1	852	-822
0	941	963
-1	398	387
-2	532	-559
-3	1262	-1247
-5	669	623
-6	290	269
-7	613	622
-8	567	-585
-9	484	-491
-10	696	724
-11	357	327
-12	149	-152
-13	231	-202
-14	202	-185
-15	193	-202
H= 4, K= 3		
0	905	-876
1	541	495
2	631	-584
3	559	-558
4	501	502
5	680	666
6	147	131
7	101	-80
8	137	107
9	175	-154
11	298	302
-16	162	87
-15	285	283
-14	415	-426
-13	372	-357
-12	236	281
-11	336	315
-10	334	-313
-8	279	242
-7	621	-612
-6	721	742
-5	658	644
-4	478	-483
-3	786	-797
-2	332	329
-1	280	266
H= 4, K= 2		
11	147	-148
10	273	276
9	338	351
8	363	-383
7	233	-288
6	630	615
5	343	338
4	210	200
3	122	-77
2	674	-664
1	971	-983
0	1048	1040
-1	276	247
-2	881	-861
-3	344	-323
-4	119	121
-5	372	332
-6	189	168
-7	710	744
-8	1069	-1102
-9	156	-206
-10	605	610
-11	152	118
-12	114	-108
-13	194	-176
-16	313	335
H= 4, K= 1		
0	73	71
1	1104	1058

Crystal Data.—The system is monoclinic of space group $P2_1/c$, with $a = 10.926$ (5), $b = 16.740$ (7), $c = 13.732$ (5) Å, $\beta = 116.51$ (1)°, formula weight 1095.9, $V = 2247.4$ (7) Å^3, $\rho_{\text{obsd}} = 1.60$ (3) g cm^{-3} (flotation in $CHBr_3$-$CHCl_3$), and $\rho_{\text{calcd}} = 1.619$ (3) g cm^{-3} for $Z = 2$. Unit cell dimensions and their esd's were obtained from a least-squares refinement of 15 measurements of high-order 2θ.

Intensity Data Collection.—The crystal used was an irregularly shaped block mounted about the y axis with an approximate cross section of 0.08 × 0.12 mm and a thickness (along y) of 0.12 mm

TABLE I (*Continued*)

L	FO	FC
H=	4, K=	1
2	178	-229
3	842	-907
4	204	220
5	865	900
6	156	168
7	179	-165
8	106	28
9	186	-170
11	247	281
-15	403	399
-14	241	-243
-13	420	-480
-11	416	433
-10	162	-142
-9	155	200
-8	145	169
-7	838	-848
-6	804	775
-5	1379	1419
-3	770	-752
-2	383	-393
-1	234	-225
H=	4, K=	0
10	399	447
8	292	-314
6	629	612
4	214	-217
2	784	-785
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-2	218	193
-3	346	-344
-4	123	-89
-7	258	254
-8	171	103
-9	513	-501
-10	171	-165
-11	371	366
-12	128	131
-13	127	-115
H=	8, K=	9
1	108	-73
2	498	-469
3	122	153
4	337	355
5	128	-55
-13	126	137
-12	257	238
-10	206	-202
-8	344	-339
-6	432	449
-4	337	-355
-3	131	131
-2	179	169
H=	8, K=	8
5	168	187
4	88	-28
2	196	206
1	365	-357
-1	537	537
-2	154	152
-3	503	-506
-7	203	178
-9	609	-690
-10	109	-77
-11	424	409
-12	96	85
-13	189	-185
H=	8, K=	7
0	192	192
1	137	158
2	459	-482
3	141	-108
4	309	321
-14	350	-339
-13	86	-77
-12	300	307
-11	144	119
-10	129	-168
-8	480	-460
-7	296	-336
-6	523	496
-5	193	183
-4	434	-434
-3	113	-93
-2	155	165
-1	193	208
H=	8, K=	6
5	186	203
2	105	-99
1	380	-386
0	291	291
-1	630	634
-3	343	-329
-4	150	136
-6	223	-190
-7	412	421
-8	255	-235
-9	702	-715
-10	147	88
-11	422	440
-13	241	-234
-15	111	-158
H=	8, K=	5
0	137	127
1	149	148
2	353	-356
3	177	-193
4	273	260
6	91	-106
-15	239	252
-14	367	-393
-13	142	-168
-12	146	135
-11	246	232
-10	192	-180
-8	295	-286
-7	451	-437
-6	469	504
-5	306	321
-4	269	-295
-3	379	-400
H=	8, K=	4
6	148	185
5	139	197
4	130	-171
2	111	-88
1	169	-182
0	407	415
-1	410	402
-2	428	-440
-3	361	-372
-4	221	248
-5	97	-88
-6	202	-173
-7	464	450
-8	416	-414
-9	628	-636
-10	446	439
-11	341	320
-12	171	-119
-13	326	-339
-15	199	-215
H=	8, K=	3
0	181	163
1	316	261
2	267	-295
3	302	-305
4	252	264
5	224	208
6	154	-157
-15	322	323
-14	263	-286
-13	415	-413
-12	103	102
-11	212	209
-8	213	-222
-7	407	-428
-6	172	175
-5	449	473
-4	468	-480
-3	532	-558
-2	161	139
H=	8, K=	2
6	319	336
5	92	53
4	168	-172
2	220	-258
1	148	-17
0	351	351
-1	237	227
-2	688	-684
-3	278	-253
-4	477	489
-6	126	95
-7	167	155
-8	338	-317
-9	316	-312
-10	343	322
-11	272	273
-12	270	-221
-13	193	-194
-14	146	-149
-15	136	-94
H=	8, K=	1
1	129	188
3	462	-497
4	149	141
5	303	333
7	127	-14
-16	116	60
-15	486	475
-14	117	-79
-13	767	-746
-11	221	216
-9	280	266
-7	616	-612
-6	195	185
-5	1119	1125
-4	295	-267
-3	706	-729
-1	279	-264
H=	8, K=	0
6	344	342
4	141	-143
2	408	-417
0	291	269
-2	890	-922
-4	370	342
-6	240	226
-8	117	34
-10	270	272
-12	200	-221
-14	179	-167
-16	296	269
H=	9, K=	0
0	312	331
2	196	-184
4	134	-109
-12	453	-435
-10	398	398
-8	102	-87
-6	96	54
-4	325	325
-2	600	-567
H=	9, K=	1
5	211	215
3	391	-419
2	195	-192
1	224	250
0	106	-24
-1	163	-115
-2	177	-193
-3	522	-520
-5	791	788
-6	186	197
-7	453	-438
-8	98	-90
-9	108	108
-11	346	313
-13	562	-538
-14	159	-140
-15	401	392
H=	9, K=	2
0	361	433
1	184	-153
2	167	-187
4	163	-160
5	103	48
-15	110	-97
-12	306	-283
-10	496	491
-9	206	-168
-8	353	-388
-7	215	214
-6	106	14
-5	137	-166
-4	491	495
-3	184	-170
-2	530	-540
H=	9, K=	3
5	155	188
4	119	159
3	307	-314
2	293	-279
1	151	145
0	157	134
-3	393	-281
-4	301	-304
-5	542	482
-6	249	268
-7	338	-313
-8	224	-194
-9	134	131
-10	100	70
-11	142	128
-12	188	197
-13	313	-311
-14	315	-316
-15	369	374
H=	9, K=	4
0	391	408
1	258	-266
2	131	-124
4	101	-96
5	121	152
-13	162	-153
-12	216	-213
-11	299	304
-10	442	436
-9	518	-515
-8	335	-339
-7	255	234
-6	233	233
-3	421	-416
-2.	542	-573
-1	352	356
H=	9, K=	5
4	191	174
3	257	-231
2	371	-360
1	190	196
0	120	132
-2	131	104
-4	329	-340
-5	244	219
-6	371	357
-7	301	-258
-8	364	-337
-10	124	-150
-11	140	174
-12	237	227
-13	236	-267
-14	291	-277
-15	255	207
H=	9, K=	6
0	205	151
1	360	-361
2	115	-90
-13	256	-222
-11	441	416
-10	178	171
-9	572	-554
-8	161	-128
-7	193	179
-3	486	-517
-1	494	443
H=	9, K=	7
4	166	195
2	399	-382
1	179	198
0	202	201
-3	113	-51
-4	395	-452
-5	111	77
-6	327	352
-7	174	-121
-8	339	-343
-10	159	-146
-11	165	186
-12	336	344
-13	125	-117
-14	321	-354
H=	9, K=	8
1	286	-275
2	86	11
-13	131	-160
-11	310	320
-9	527	-507
-8	95	50
-7	292	258
-3	395	-391
-1	310	317
H=	9, K=	9
2	337	-327
1	116	-99
-1	139	98
-2	182	159
-4	416	-398
-5	201	-176
-6	441	459
-7	126	30
-8	415	-405
-9	153	-111
-10	95	-83
-12	242	235
H=	9, K=	10
1	167	-197
-12	122	89
-11	283	291
-10	189	-199
-9	428	-431
-7	166	189
-6	125	-115
-3	298	-313
-2	106	93
-1	409	417
H=	9, K=	11
1	133	-138
0	108	90
-2	103	98
-3	186	164
-4	303	-299
-5	294	-306
-6	309	288
-7	246	264
-8	296	-301
-9	109	-72
-11	169	-125
H=	9, K=	12
0	181	-173
-10	279	-273
-9	262	-261
-8	230	255
-7	184	179
-4	145	-128
-3	233	-244
-2	255	239
-1	220	220
H=	9, K=	13
-3	183	197
-4	125	-140
-5	280	-278
-6	210	200
-7	249	256
H=	9, K=	14
-7	146	109
-4	168	-175
-3	156	-106
H=	10, K=	12
-3	182	-198
-4	134	-145
-6	107	-39
-7	112	157
-8	198	196
H=	10, K=	11
-8	168	-162
-7	164	173
-6	289	299
-5	219	-192
-4	286	-269
-3	103	121
-1	112	37
H=	10, K=	10
-1	209	213
-2	130	144
-3	313	-293
-4	122	-78
-5	97	67
-7	158	209
-8	120	123
-9	298	-314
-10	106	-96
-11	259	264
H=	10, K=	9
0	103	110
1	151	-52
-12	241	247
-9	93	-22
-8	326	-299
-7	109	99
-6	472	471
-5	186	-157
-4	350	-351
H=	10, K=	8
1	219	-227
-1	300	332
-3	291	-301
-5	126	118
-6	111	-82
-7	201	194
-9	317	-330
-11	267	305
H=	10, K=	7
0	174	146
1	117	83
2	250	-267
-12	288	288
-9	98	-19
-8	323	-309
-7	130	-126
-6	519	503
-4	514	-502
-3	110	-53
-2	257	130
-1	120	-100
H=	10, K=	6
2	117	-107
1	153	-153
0	181	191
-1	344	358
-2	98	-116
-3	369	-346
-4	115	111
-5	196	173
-8	168	-170
-9	460	-407
-10	164	155
-11	330	312
-13	159	-160
H=	10, K=	5
0	160	141
1	212	187
2	256	-276
3	186	-156
-14	236	-260
-13	170	-165
-12	283	264
-11	109	131
-10	169	-148
-9	104	45
-8	255	-277
-7	274	-237
-6	300	322
-5	149	156
-4	343	-333
-2	211	219
H=	10, K=	4
2	130	-121
1	177	-165
0	275	278
-1	307	323
-2	476	-442
-3	260	-239
-4	216	212
-3	212	182
-6	152	166
-8	325	-297
-9	403	-366
-10	413	423
-11	293	264
-12	340	-324
H=	10, K=	3
0	235	212
1	234	221
2	191	-193
3	229	-233
-14	232	-211
-13	284	-303
-12	189	171
-11	162	165
-8	325	-284
-7	187	-190
-6	286	269
-5	192	189
-4	251	-227
-3	131	-105
-2	135	124
-1	148	-155
H=	10, K=	2
2	119	-126
1	114	-130
0	373	374
-1	201	223
-2	489	-486
-4	260	226
-5	131	-38
-6	114	89
-7	121	76
-8	472	-460
-9	138	-154
-10	577	560
-12	408	-400
-13	111	-27
H=	10, K=	1
1	347	341
2	96	-86
3	395	-385
-14	118	-92
-13	447	-450
-9	119	110
-5	318	-273
-7	435	-404
-6	213	162
-5	618	564
-3	156	-137
-1	100	-74
H=	10, K=	0
2	115	-128
0	369	365
-2	360	-378
-4	287	295
-6	244	216
-8	439	-449
-10	436	453
-12	481	-511
H=	11, K=	0
0	261	257
-10	487	467
-8	451	-425
-4	160	128
-2	510	-474
H=	11, K=	1
1	285	300
0	127	86
-1	188	-180
-3	185	-184
-4	104	-47
-5	159	174
-7	418	-395
-8	175	-130
-9	181	143
-10	90	-21
-11	124	123
-13	406	-369
-14	97	-48
H=	11, K=	2
0	239	249
-13	110	-31
-12	354	-347
-10	445	438
-9	192	-178
-8	385	-392
-4	125	121
-2	430	-399
-1	192	178
H=	11, K=	3
1	188	225
0	127	98
-1	91	-138
-2	126	109
-3	163	-148
-4	275	-282
-5	327	288
-6	266	257
-7	360	-358
-8	289	-261
-12	157	116
-13	290	-273
H=	11, K=	4
0	157	169
1	141	-65
-12	317	-302
-11	283	279
-10	241	246
-9	356	-331
-8	298	-281
-7	90	-18
-4	216	193
-3	193	-207
-2	235	-221
-1	275	284
H=	11, K=	5
0	150	123
-1	98	-99
-2	203	171
-4	396	-363
-5	253	240
-6	338	300
-7	244	-194
-8	310	-341
-12	238	193
-13	161	-140
H=	11, K=	6
-12	131	-131
-11	323	288
-10	116	123
-9	364	-363
-8	206	-163
-3	135	123
-3	317	-316
-1	365	342
H=	11, K=	7
-2	89	79
-4	279	-276
-6	390	396
-8	281	-264
-10	95	-24
-11	88	-47
-12	176	166
H=	11, K=	8
-11	214	232
-9	354	-348
-7	168	160
-3	289	-297
-1	291	258
H=	11, K=	9
-4	310	-306
-6	388	382
-6	234	-243
-9	124	67
H=	11, K=	10
-9	263	-254
H=	12, K=	7
-9	178	61
-8	239	-259
-6	310	318
H=	12, K=	6
-8	176	-141
-9	306	-308
-10	149	128
H=	12, K=	5
-11	136	11
-9	93	61
-8	292	-283
-7	208	-230
-6	211	216
-5	192	180
-4	219	-226
H=	12, K=	4
-2	228	-204
-3	217	-201
-4	184	148
-8	177	-188
-9	342	-313
-10	226	210
-11	226	295
H=	12, K=	3
-12	127	114
-10	89	54
-9	128	120
-8	310	-285
-7	267	-243
-6	138	130
-5	288	288
-4	184	-183
-3	177	-130
H=	12, K=	2
-2	286	-295
-3	96	-94
-4	231	218
-5	140	-102
-8	241	-201
-9	203	-201
-10	331	307
-11	144	175
-12	295	-266
H=	12, K=	1
-11	152	120
-9	175	153
-8	165	-133
-7	364	-347
-5	289	295
-4	111	-108
-3	198	-214
H=	12, K=	0
-2	313	-322
-4	199	196
-8	312	-275
-10	359	333
-12	292	-290
H=	13, K=	0
-8	208	-147
H=	13, K=	1
-7	309	-277
-8	133	-66

Reflection intensities were measured on a Picker four-circle diffractometer equipped with a pulse height analyzer using Cu Kα radiation. A θ–2θ scan was employed at a scanning speed of 2°/min. Background was counted for 40 sec at each end of the

TABLE II

ATOMIC COORDINATES AND THERMAL PARAMETERS × 10^4 [a]

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ru	82.9 (7)	317.1 (5)	1911.0 (6)	68 (0.7)	21 (0.2)	34 (0.4)	1 (0.6)	24 (0.4)	−1 (0.4)
C_1	−261 (11)	1714 (6)	3306 (7)	148 (16)	42 (5)	41 (7)	20 (8)	36 (9)	−2 (5)
C_2	267 (12)	2384 (6)	3962 (8)	194 (19)	34 (5)	60 (8)	2 (8)	55 (11)	−4 (5)
C_3	1618 (12)	2634 (7)	4322 (8)	161 (18)	32 (5)	72 (9)	17 (8)	33 (11)	13 (6)
C_4	2412 (11)	2209 (6)	3967 (8)	131 (15)	27 (4)	71 (9)	−6 (7)	22 (10)	1 (5)
C_5	1878 (10)	1543 (6)	3246 (7)	91 (12)	29 (4)	49 (7)	−10 (6)	7 (8)	3 (4)
C_6	−743 (12)	−1074 (5)	2922 (7)	136 (14)	24 (4)	34 (6)	−12 (6)	22 (8)	1 (4)
C_7	−1456 (11)	−1548 (6)	3338 (8)	145 (16)	37 (5)	49 (8)	−15 (7)	21 (9)	6 (5)
C_8	−2550 (11)	−1232 (7)	3485 (8)	143 (16)	54 (6)	59 (8)	−11 (8)	52 (10)	−1 (6)
C_9	−2903 (10)	−417 (7)	3178 (7)	128 (14)	50 (6)	50 (7)	−4 (8)	39 (8)	0 (6)
C_{10}	−2147 (9)	34 (6)	2778 (7)	93 (12)	42 (5)	62 (8)	−1 (6)	55 (8)	−0 (5)
C_{11}	2209 (10)	−294 (7)	4001 (8)	147 (15)	31 (4)	58 (7)	−4 (8)	26 (8)	3 (6)
C_{12}	3380 (11)	−729 (7)	4697 (8)	140 (17)	50 (6)	70 (9)	32 (8)	14 (10)	7 (6)
C_{13}	4080 (11)	−1147 (7)	4228 (9)	132 (17)	56 (7)	75 (10)	32 (9)	12 (11)	15 (6)
C_{14}	3600 (11)	−1117 (8)	3101 (9)	121 (16)	64 (7)	101 (11)	36 (9)	49 (11)	29 (7)
C_{15}	2438 (10)	−669 (7)	2458 (9)	117 (14)	51 (6)	89 (10)	34 (8)	67 (10)	14 (6)
C_{16}	−1865 (11)	1689 (6)	588 (9)	176 (18)	26 (4)	88 (10)	19 (8)	77 (12)	9 (5)
C_{17}	−3017 (12)	2041 (7)	−213 (9)	150 (17)	44 (6)	78 (10)	19 (8)	50 (11)	5 (6)
C_{18}	−4202 (12)	1593 (8)	−778 (9)	131 (17)	62 (7)	87 (10)	31 (9)	59 (11)	9 (7)
C_{19}	−4247 (11)	766 (8)	−575 (8)	116 (15)	58 (7)	71 (9)	24 (8)	41 (10)	11 (6)
C_{20}	−3038 (19)	427 (7)	252 (7)	71 (11)	62 (9)	57 (7)	−11 (7)	25 (7)	−2 (6)
C_{21}	390 (8)	398 (5)	59 (6)	78 (10)	24 (4)	42 (6)	−2 (6)	27 (6)	0 (4)
N_1	556 (7)	1291 (4)	2942 (6)	98 (10)	27 (4)	54 (6)	−2 (5)	29 (7)	3 (4)
N_2	−1079 (7)	−282 (5)	2668 (5)	95 (9)	36 (3)	43 (5)	11 (6)	36 (6)	8 (4)
N_3	1750 (7)	−263 (5)	2895 (6)	107 (10)	29 (3)	52 (5)	4 (6)	35 (6)	−1 (4)
N_4	−1888 (7)	880 (5)	817 (5)	74 (9)	35 (4)	48 (6)	5 (5)	20 (6)	−5 (4)
O_1	747 (6)	783 (4)	917 (4)	101 (8)	28 (2)	56 (5)	1 (4)	43 (5)	1 (3)
O_2	−608 (6)	−600 (3)	736 (5)	113 (9)	25 (3)	57 (5)	−3 (4)	50 (5)	−2 (3)
F_1	6338 (8)	1691 (5)	2697 (7)	174 (12)	84 (5)	193 (10)	26 (7)	40 (10)	−17 (6)
F_2	4646 (17)	1000 (9)	1553 (13)	367 (36)	80 (14)	186 (35)	58 (20)	88 (36)	−86 (16)
F_{2B}	4640 (39)	1355 (28)	1123 (23)	506 (81)	223 (54)	148 (37)	−59 (58)	231 (48)	−22 (31)
F_3	4508 (16)	2266 (9)	1564 (11)	295 (30)	74 (9)	141 (20)	6 (13)	26 (19)	15 (11)
F_{3B}	4209 (25)	2216 (14)	2094 (20)	275 (47)	54 (11)	144 (32)	96 (19)	69 (30)	22 (15)
F_4	4621 (14)	1650 (12)	2925 (11)	267 (29)	148 (13)	138 (18)	−50 (17)	106 (19)	−54 (14)
F_{4B}	4605 (23)	1007 (15)	2614 (21)	221 (45)	72 (13)	124 (35)	41 (22)	10 (32)	28 (18)
B	4985 (15)	1606 (9)	2145 (10)	119 (18)	39 (6)	57 (12)	10 (9)	20 (13)	0 (6)

[a] The temperature factor is of the form: $T(hkl) = \exp(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)$.

scan range of about 3° which was adjusted to account for α_1–α_2 splitting. The takeoff angle used was 0.16°. Throughout the course of the data collection there was no significant deviation in the two standard reflections 032, 087 which were remeasured after every 50 reflections. The esd in F_o^2 was taken as $\sigma(F_o^2) = [(Lp)^{-2} + CF_o^4]^{1/2}$ with $C = 0.02$, chosen arbitrarily to make allowance for systematic errors. Of the 4000 independent reflections measured, 3165 were considered significant on the criterion $|F_o| < \sigma(F_o)$.

The linear absorption coefficient μ for Cu Kα is 64.4 cm^{-1}. Considering the small crystal size and the fact that the maximum variation in count rate from the mean for several ϕ sweeps was ±8%, no absorption corrections were applied. The structure refinement was carried out using the XFLS[15] program which allows for anomalous dispersion. Hartree–Fock atomic scattering factors were used.[16]

Determination and Refinement of the Structure

Standard Patterson and Fourier techniques led to the location of all except hydrogen atoms in the cell. Anisotropic least-squares refinement yielded a conventional R factor of 0.076. A difference Fourier map revealed the positions of all 20 hydrogen atoms associated with the pyridine rings. Due to the large amount of computer time required for each least-squares cycle, the hydrogen atoms were not included in the refinement nor in F_c. The map indicated disordering about one of the threefold axes of the BF_4^- tetrahedron. The three disordered fluorine atoms were found to be in two sets; site occupancies 0.65 and 0.35 gave the lowest residual. Refinement, including the $|BF(F_{0.65})_3(F_{0.35})_3|^-$ entity, yielded a final conventional R of 0.071 and a weighted R of 0.068. F_c and F_o are listed in Table I.

A final difference Fourier showed no large areas of electron density greater than 0.4 e$^-$ Å^{-3} in the region of the fluoroborate anion. The peaks assignable to hydrogen atoms varied from 0.8 to 0.4 e$^-$ Å^{-3} with an average value of 0.6 e$^-$ Å^{-3}.

Description of the Structure

The structure consists of discrete centrosymmetric

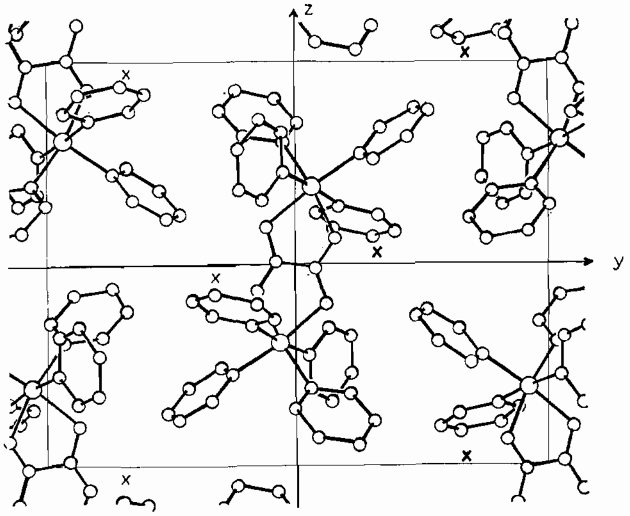

Figure 1.—Structure in x projection. X's mark BF_4^- ions.

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[16] D. T. Cromer and J. B. Mann, *Acta Crystallogr., Sect. A*, **24**, 321 (1968).

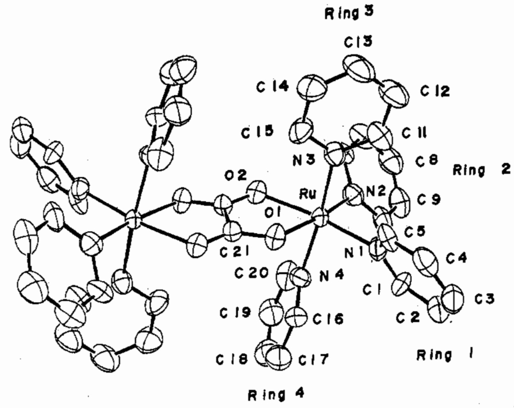

Figure 2.—Atomic numbering scheme and thermal ellipsoid plot.

dimeric cations and fluoroborate anions, Figure 1. The ruthenium ions lie ±0.0404 (8) Å from the plane $x = 0$ and the boron atoms ±0.007 (16) Å from the plane $x = {}^1/_2$.

Figure 2 shows an isolated cation and indicates the lettering for Tables II through V. The pyridine and oxalate ligands form a slightly distorted octahedron about each ruthenium atom. The maximum deviation from the best least-squares equatorial plane (Table V) is 0.026 Å. As the oxalate ligand has a bite of only 80°, the other bond angles in the plane are somewhat greater than 90° (Table IV). The angle between the two axial Ru–N bonds above and below the equatorial plane is 177.8 (8)°. Pyridine rings 3 and 4 are at 81° to each other with ring 3 approximately parallel and ring 4 approximately perpendicular to the Ru–Ru axis of the dimer (see Figure 2). Rings 1 and 2 are inclined such that their normals cut the equatorial plane at 46 and 56°, respectively.

TABLE III

(a) Bond Lengths (Å) and Angles (deg) in Pyridine Rings

Bond Lengths

N–αC		αC–βC		βC–γC	
N_1–C_1	1.395 (11)	C_1–C_2	1.392 (13)	C_2–C_3	1.396 (14)
N_1–C_5	1.378 (11)	C_4–C_5	1.431 (12)	C_3–C_4	1.368 (14)
N_2–C_6	1.350 (10)	C_6–C_7	1.400 (12)	C_7–C_8	1.429 (14)
N_2–C_{10}	1.378 (11)	C_9–C_{10}	1.399 (13)	C_8–C_9	1.402 (14)
N_3–C_{11}	1.372 (10)	C_{11}–C_{12}	1.410 (13)	C_{12}–C_{13}	1.389 (14)
N_3–C_{15}	1.337 (11)	C_{14}–C_{15}	1.398 (13)	C_{13}–C_{14}	1.397 (14)
N_4–C_{16}	1.393 (12)	C_{16}–C_{17}	1.391 (13)	C_{17}–C_{18}	1.394 (15)
N_4–C_{20}	1.373 (11)	C_{19}–C_{20}	1.421 (13)	C_{18}–C_{19}	1.417 (15)

In-Plane Bending Angles

Ru–N_1–C_1	124.8 (6)	Ru–N_3–C_{11}	120.3 (6)
Ru–N_1–C_5	115.8 (6)	Ru–N_3–C_{15}	120.9 (6)
Ru–N_2–C_6	122.8 (6)	Ru–N_4–C_{16}	119.6 (6)
Ru–N_2–C_{10}	117.0 (6)	Ru–N_3–C_{20}	119.7 (6)

Out-of-Plane Bending Angles

Ru–ring 1	1.1	Ru–ring 3	4.6
Ru–ring 2	6.1	Ru–ring 4	5.4

Bond Angles

N vertex		αC vertex	
C_1–N_1–C_5	119.2 (8)	N_1–C_1–C_2	119.6 (10)
		N_1–C_5–C_4	119.8 (10)
C_6–N_2–C_{10}	119.7	N_2–C_6–C_7	119.7
		N_2–C_{10}–C_9	121.1
C_{11}–N_3–C_{15}	118.5	N_2–C_{11}–C_{12}	122.7
		N_3–C_{15}–C_{14}	112.6
C_{16}–N_4–C_{20}	119.6	N_4–C_{16}–C_{17}	119.7
		N_4–C_{20}–C_{19}	120.9
βC vertex		**γC vertex**	
C_1–C_2–C_3	122.5 (10)	C_2–C_3–C_{11}	117.2 (10)
C_3–C_4–C_5	121.9 (10)		
C_6–C_7–C_8	120.1	C_7–C_8–C_9	117.3
C_8–C_9–C_{10}	120.5		
C_{11}–C_{12}–C_{13}	118.0	C_{12}–C_{13}–C_{14}	119.2
C_{13}–C_{14}–C_{15}	120.1		
C_{16}–C_{17}–C_{18}	119.9	C_{17}–C_{18}–C_{19}	121.6
C_{18}–C_{19}–C_{20}	117.0		

(b) Shortest αC···αC Distances (Å)

C_6–C_{11}	3.17	C_1–C_{16}	3.34
C_{10}–C_1	3.37	C_5–C_{11}	3.21
C_{10}–C_{20}	3.22		

TABLE IV

OTHER BOND LENGTHS (Å) AND ANGLES (DEG)

Bond Lengths

Ru–N_1	2.069 (7)	B–F_1	1.335 (15)
Ru–N_2	2.066 (7)	B–F_2	1.247 (17)
Ru–N_3	3.089 (7)	B–F_{2B}	1.348 (31)
Ru–N_4	2.095 (7)	B–F_3	1.327 (18)
Ru–O_1	2.096 (5)	B–F_{3B}	1.309 (23)
Ru–O_2	2.113 (5)	B–F_4	1.300 (17)
C_{21}–C_{21}'	1.551 (6)	B–F_{4B}	1.352 (26)
C_{21}–O_1	1.244 (9)	Av B–F	1.317
C_{21}'–O_2	1.262 (9)		

Bond Angles

O_1–Ru–O_2	79.8 (2)	O_1–C_{21}–C_{21}'	118.4 (10)
O_2–Ru–N_2	90.4 (2)	O_1–C_{21}–O_1'	125.7 (9)
N_2–Ru–N_1	97.6 (3)	O_2–C_{21}'–O_2'	125.7 (9)
N_1–Ru–O_1	92.3 (3)	F_1–B–F_2	113 (1)
N_3–Ru–O_1	84.7 (3)	F_1–B–F_{2B}	110 (2)
N_3–Ru–O_2	86.1 (3)	F_1–B–F_3	107 (1)
N_3–Ru–N_2	92.3 (3)	F_1–B–F_{3B}	118 (2)
N_3–Ru–N_1	93.4 (3)	F_1–B–F_4	101 (1
N_4–Ru–O_1	91.5 (3)	F_1–B–F_{4B}	108 (2)
N_4–Ru–O_2	90.7 (3)	F_2–B–F_4	118 (2)
N_4–Ru–N_2	90.9 (3)	F_3–B–F_4	115 (1)
N_4–Ru–N_1	89.4 (3)	F_3–B–F_2	111 (2)
Ru–O–C_{21}	112.4 (5)	F_{2B}–B–F_{3B}	108 (2)
Ru–O_2'–C_{21}'	112.2 (5)	F_{2B}–B–F_{4B}	105 (2)
		F_{3B}–B–F_{4B}	106 (2)

TABLE V

EQUATIONS OF BEST LEAST-SQUARES PLANES, WHERE X, Y, AND Z ARE REAL ORTHOGONAL COORDINATES WITH AXES X ALONG x AND Z ALONG z*

Ring 1	$0.0616X + 0.5960Y - 0.8006Z - 1.672 = 0$
Ring 2	$-0.2363X - 0.2776Y - 0.9312Z - 2.242 = 0$
Ring 3	$-0.5883X - 0.8021Y - 0.1024Z - 0.0900 = 0$
Ring 4	$0.6958X - 0.2223Y - 0.6830Z - 2.791 = 0$
Oxalate	$-0.7708X + 0.4944Y - 0.4017Z = 0$
Octahedral plane	$-0.6731X + 0.4720Y - 0.5692Z - 0.2196 = 0$

In the pyridine rings, the N–C bonds seem to be significantly longer than those reported in other crystal structures containing pyridine derivatives where the bond length is usually about 1.35 Å. In addition the C–N–C angle is usually about 118° and N–C–C about 122° whereas in the present case, for reasons which are not clear, these angles are both closer to 120° (see Table IIIa). Since the rings are all essentially planar with no deviations as great as 2σ from the best planes and there is no apparent lengthening of the β–γ carbon–carbon bonds, it is concluded that there is no observable contribution from quinoid type structures which would result from d_π–p_π back-bonding.

The Ru–N bonds of the equatorial pyridine rings 1 and 2 are 2.069 (7) and 2.066 (7) Å, which are 3σ shorter than the Ru–N bonds of the pyridine rings 3 and 4 (2.089 (7) and 2.095 (7) Å). Since oxalate is a better π donor and poorer π acceptor than pyridine,[17] the Ru–N bonds of the equatorial pyridine ligands would be expected to be shortened due to increased electron density available for back-donation relative to the case of two competing axial pyridine ligands. The difference, although slight, is in accord with the trans effect.

No Ru–N bond is quite coplanar with its pyridine ring, Ru atoms being as much as 0.2 Å out of the plane. In addition, the equatorial pyridine rings exhibit significant in-plane bending; that is, the two αC–N–Ru angles differ by 9° for ring 1 and by 5° for ring 2. These deviations are most readily accounted for by steric interactions discussed below.

As in previously reported complexes[2,5] the bridging oxalate ligand is planar, in the present case to within 0.000 (8) Å. The bond lengths and angles of the oxalate ligand are not significantly different from those of the free oxalate ion.[18] Any C–O bond lengthening which might result from the formation of the four Ru–O donor bonds is too small to be detected. The Ru–O bond lengths of 2.113 (5) and 2.096 (5) Å differ by 3σ and the C–O bond lengths at 1.244 (9) and 1.262 (9) differ only by 2σ. However, the longer C–O bond is associated with the longer Ru–O bond, suggesting that there is no chemical significance attributable to the variation in Ru–O bond lengths.

The Ru atoms bridged by the oxalate ion lie above and below the oxalate plane such that the plane formed by the two Ru–O bonds cuts the oxalate plane at an angle of 169.6 (6)°. This out-of-plane bending increases the distance between the H atom attached to C_{15} and the carbons of the oxalate ligand. These distances in the complex are 2.69 and 2.64 Å, somewhat less than the sum of their van der Waals radii which is *ca.* 2.9 Å.

The Ru–N bond lengths are significantly shorter than the Ru–N bond length of 2.127 (7) Å in the complex *trans*-$[Ru(NH_3)_4ClSO_2]Cl$.[19] In many metal–ammine and metal–pyridine complexes of cobalt, nickel, and copper the metal–ammine and metal–pyridine bond lengths are effectively equal. The relative shortness in the present case indicates the ability of the Ru(II) ion to form strong bonds with the "weak" base pyridine. Ir spectra of several ruthenium(II)–pyridine complexes have exhibited bands in the region of 300 cm^{-1} which have been tentatively assigned to the Ru–N stretching mode.[14] This quite high value is in accord with the observed shortness of the Ru(II)–N bonds in this complex.

The BF_4^- anion is disordered about a threefold axis of the tetrahedron. Each set of three disordered fluorine atoms subtends bond angles equal to the tetrahedral angle within experimental error. However, their large vibrational amplitudes may imply that disorder is more extensive than the model used here. The unique, ordered, fluorine atom F(1) is perhaps hydrogen bonded to the hydrogen atom attached to C(10) (Figure 3). The F···H distance is 2.28 Å (sum of van der Waals radii about 2.43 Å) with an F(1)–H–C(10) angle of 141° and B–F(1)–H angle of 115°.

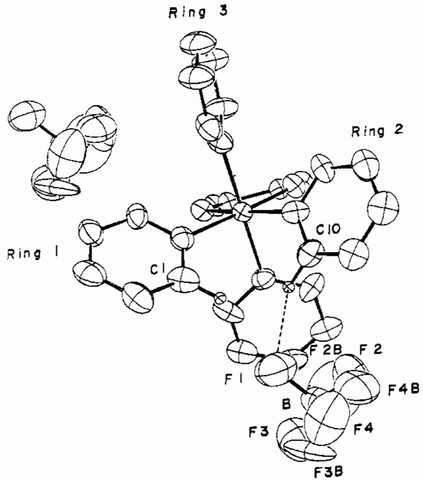

Figure 3.—Perspective view of molecule showing (a) disordering of BF_4^- ion and (b) possible C–H···F hydrogen bond.

In the complex diiodocarbomethoxycarbonyl(2,2′-bipyridyl)iridium(III),[20] the carbomethoxy ligand is considered to be held in the equatorial plane by means of an α-C–H···O hydrogen bond with H···O distance of length 2.0 Å and angle 139°. Although the proposed C–H···F hydrogen bond in our complex is longer, the possibility of sufficient interaction to prevent complete disordering of the fluoroborate anion exists. There are no other short H···F contacts and all F···C distances are normal.

It has been suggested[21] that *cis*-tetrapyridine complexes are thermodynamically unstable relative to trans due to steric interactions, possibly between hydrogen atoms attached to α-carbon atoms. As far as the α-carbon atoms themselves are concerned they are no closer together in the present complex than the 3.21 Å in the typical trans complex $Ni(3,5-(CH_3)_2py)_4(ClO_4)_2$[22] (see Table IIb). However, in the case of one pair of adjacent rings, 1 and 2 (Figure 3), the calculated separation between hydrogen atoms attached to α-carbon atoms C(1) and C(10) is only 1.8 Å. (The latter hydrogen atom appears on the difference map in the predicted position; the peak for the C(1) hydrogen atom is, however, too broad for its position to be assigned in this way.) No other pairs of hydrogen atoms attached to α-carbon atoms are at all close. Thus this one H···H interaction may contribute to the instability of cis complexes relative to trans. In the latter, no such close H···H interactions are to be expected. There is some pmr evidence[14] that the pyridine rings are librating in solution. This may give rise to further H···H interactions.

The close contact between α-carbon atoms appears to be the cause of the Ru–N bonds of rings 1 and 2 being subject to bending in the plane of the ring ("in-plane bending") and the cause of out-of-plane bending (*i.e.*, Ru is displaced from the mean ring plane) in rings 2, 3, and 4. All these distortions are such as to reduce the interaction between α-carbon atoms of adjacent rings.

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Thus, overall, the cis complex appears to be subject to more internal strain than a comparable trans complex. In addition, the oxalate ligand, having a bite of only 80°, allows the N–Ru–N bond angle for the equatorial pyridine rings to expand to 98°. Thus if the oxalate ligand were replaced by two chlorine atoms, the resultant cis complex, requiring a larger Cl–Ru–Cl angle, would certainly be more crowded and thus would be expected to be unstable relative to the trans complex.

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CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
TEXAS CHRISTIAN UNIVERSITY, FORTH WORTH, TEXAS

The Crystal and Molecular Structure of Dichlorobis(4-methylpyridine 1-oxide)copper(II), Yellow Modification

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The crystal structure of the yellow modification of dichlorobis(4-methylpyridine 1-oxide)copper(II), $[(C_6H_7NO)_2CuCl_2]_2$, has been determined by single-crystal X-ray diffraction methods. Counter methods were used to collect 1469 nonzero reflections. The complex crystallizes in the monoclinic space group $P2_1/n$ with two dimeric molecules per unit cell. The cell dimensions are a = 7.368 (10), b = 19.857 (5), c = 10.773 (5) Å, and β = 111° 25 (10)′. The observed and calculated densities are 1.60 and 1.59 g cm^{-3}, respectively. The structure was refined by least-squares methods to a conventional R factor of 0.061%. The molecules consist of oxygen-bridged dimer units. The coordination around the copper ion may be described as a highly distorted square-based pyramid with a chlorine atom at the apical site at a distance of 2.354 (3) Å. The oxygen bridging system contains unequal Cu–O bond lengths of 1.957 (6) and 2.153 (6) Å, and the magnetic moment should be larger than those observed for the 1:1 dimeric species. The unstable dimeric molecule of the yellow modification is postulated to be an intermediate in the formation of the thermodynamically stable trans 2:1 monomer.

Introduction

The various types of 2:1 aromatic *N*-oxide complexes of copper(II) have been discussed in previous papers.[1,2] The infrared spectra have been reported for many of these complexes, and copper(II) coordination geometries were predicted from a consideration of the metal–chlorine stretching modes.[3] The green form of the 2:1 4-methylpyridine 1-oxide complex was shown to have a trans square-planar geometry about the copper(II) ions.[2] The monomers are isolated units which show little intramolecular steric interaction and exhibit normal magnetic moments from 77°K to well above room temperature. This form is thermodynamically the most stable, and all 2:1 green crystalline forms are predicted to be trans square planar.

The yellow form of the dichlorobis(2,6-dimethylpyridine 1-oxide)copper(II) complex was shown to have a geometry intermediate between tetrahedral and cis square planar.[4] This is consistent with the prediction of Hatfield and Morrison[5] based upon the diffuse reflectance spectrum of dibromobis(2,6-dimethylpyridine 1-oxide)copper(II). The magnetic moments of these two complexes were normal. In general, only small quantities of the yellow form can be prepared, and the infrared spectra and magnetic moments of only a few of the complexes have been reported. The dichlorobis(pyridine 1-oxide) copper(II) complex has been shown to be an oxygen-bridged dimer,[6] and the complex has a magnetic moment around 0.5 BM.[7] The corresponding bromide and dibromobis(4-methylpyridine 1-oxide)copper(II) complexes have magnetic moments of 1.4[7] and 1.33[8] BM, respectively. Since the 1:1 dimeric bromide complexes usually have lower magnetic moments than the corresponding chloride complexes, these intermediate moments are not consistent with the known structures in this series. A dimeric structure was predicted,[2] but some significant differences in geometry must exist.

We wish to report the structure of the yellow form of dichlorobis(4-methylpyridine 1-oxide)copper(II) and rationalize the physical properties in terms of this structure.

Experimental Section

Small quantities of the yellow 2:1 complex were prepared by mixing an excess of 4-methylpyridine 1-oxide with the 1:1 complex in boiling ethanol. The solution was allowed to evaporate slowly at room temperature, and a few yellow crystals were found together with a large excess of the green form. Attempts to improve the yield of yellow modification were unsuccessful. The crystals were unstable, and the small quantities of sample hindered the complete chemical and physical characterization of this material.

The yellow complex was irreversibly converted to the green form above 73°, and the converted green form retained its color on returning to room temperature. An infrared spectrum was obtained of the yellow form pressed into a KBr pellet. Two

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