with the difference in melting points (220° for the present compound *vs.* 153° for $ReNCl_2(P(C_2H_5)_2C_6H_5)_3$[4]).

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Crystal Structure of Bis(*p*-toluidinium) Hexachlororhenate(IV)

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The structure of bis(*p*-toluidinium) hexachlororhenate(IV) has been determined. The space group is monoclinic $P2_1/c$ with four molecules per unit cell; the cell dimensions are $a = 7.01 \pm 0.02$, $b = 25.04 \pm 0.05$, $c = 11.54 \pm 0.02$ A; $\beta = 90.0 \pm 0.1°$. Octahedral $ReCl_6$ anions are packed with planar *p*-toluidinium cations such that the octahedra form layers alternated with organic groups, and the usual bond lengths and angles are maintained. The compound is apparently not magnetically dilute.

Introduction

The magnetic susceptibility of some simple Re(IV) salts, including K_2ReCl_6, Cs_2ReCl_6, and K_2ReF_6, has been reported by Figgis, Lewis, and Mabbs.[1] The interpretation of these measurements is complicated by the possibility of magnetic exchange. In an attempt to dilute the $ReCl_6$ ions with relatively large organic species and thereby reduce the exchange interaction, Figgis, *et al.*, also measured the susceptibility of bis(*p*-toluidinium) hexachlororhenate(IV) $[(TH)_2ReCl_6]$, bis(pyridinium) hexachlororhenate(IV), and bis(quinolinium) hexachlororhenate(IV). We have determined the crystal structure of $(TH)_2ReCl_6$ in order to observe the nature of this dilution and to provide structural data to facilitate the interpretation of the magnetic measurements.

Experimental Section

Bright yellow-green crystals of bis(*p*-toluidinium) hexachlororhenate $[(CH_3C_6H_4NH_3)_2ReCl_6]$ were prepared by combining 0.25 g of *p*-toluidinium hydrochloride with 0.25 g of potassium hexachlororhenate in 30 ml of concentrated HCl, heating to dissolve, and evaporating on a steam bath until crystals appeared. After standing overnight at room temperature the crystals were filtered on a sintered-glass funnel, washed twice with ethanol and twice with ether, and suction dried. K_2ReCl_6 was made by reduction of $KReO_4$ with H_2PO_3 in concentrated HCl.[2]

A single crystal approximately 0.5 × 0.05 × 0.005 mm was mounted on a glass fiber with the long dimension as the axis of rotation. Cell dimensions were obtained from rotation and Weissenberg photographs using Cu Kα radiation (λ 1.5418 A). Multiple-film equiinclination Weissenberg photographs were taken for layers $h = 0$–4, yielding 1812 independent nonzero reflections. The intensities were estimated by visual comparison with a calibrated film strip, corrected for spot size elongation and contraction,[3] and converted to structure factors in the usual way. Absorption corrections were not made. Interlayer scaling was done according to exposure time.

Unit Cell and Space Group

The crystals are monoclinic with $a = 7.01 \pm 0.02$, $b = 25.04 \pm 0.05$, $c = 11.54 \pm 0.02$ A; $\beta = 90.0 \pm 0.1°$. There are four formula weights per unit cell giving a calculated density of 2.02 g/cc. The density measured by flotation is 1.99 g/cc. The systematic absence of $h0l$ reflections when l is odd and of $0k0$ reflections when k is odd suggests $P2_1/c$ as the space group. The successful structure determination confirms this choice.

Determination of the Structure

A three-dimensional Patterson map yielded the rhenium and chlorine atom coordinates; four cycles of least-squares refinement[4] with these coordinates gave a value of 0.24 for $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. An electron density map with signs based on rhenium and chlorine coordinates revealed the light-atom positions except for hydrogen which was ignored throughout the structure determination. Nitrogen atoms were distinguished from carbon atoms on chemical grounds only, as being closest to the hexachlororhenate octahedra. Seven cycles of full-matrix, least-squares refinement, using isotropic temperature factors for each atom, reduced R to 0.16. In these and subsequent least-squares calculations the function minimized was $\Sigma w(F_o - F_c)^2$ where the weights, w, were assigned according to Hughes.[5] Atomic scattering factors for neutral atoms were obtained from the listing of Ibers.[6] The layers were rescaled by making $\Sigma F_o = \Sigma F_c$. Introduction of anisotropic temperature factors in the form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$ for rhenium and chlorine and removal of 14 intensities which either suffered from extinction or were

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[1] B. N. Figgis, J. Lewis, and F. E. Mabbs, *J. Chem. Soc.*, 3138 (1961).

[2] G. W. Watt and R. J. Thompson, *Inorg. Syn.*, **7**, 190 (1963).

[3] K. Lonsdale, *Acta Cryst.*, **17**, 308 (1964).

[4] Fourier calculations were carried out with the FORDAP program of Dr. Allan Zalkin; least-squares calculations were done with the Gantzel–Sparks–Trueblood program.

[5] E. W. Hughes, *J. Am. Chem. Soc.*, **63**, 1737 (1941).

[6] J. A. Ibers, "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962, p 212.

TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS[a]

K	L	FOBS	FCAL
***	H = 0		****
4	0	1757	2601*
6	0	457	543
8	0	498	319
10	0	147	201
12	0	704	659
14	0	906	921
16	0	2082	2177
18	0	2170	2318
20	0	1721	1799
24	0	246	272
22	0	1001	979
2	1	1328	1161
4	1	2037	2391*
5	1	147	249
6	1	1289	1538
7	1	560	564
8	1	371	502
9	1	620	546
10	1	149	314
11	1	224	259
12	1	872	892
13	1	251	206
14	1	1105	1196
15	1	392	307
16	1	649	597
20	1	714	618
22	1	906	894
24	1	695	672
0	2	2669	2893*
1	2	503	411
2	2	2773	3006
4	2	2299	2259
5	2	365	377
6	2	1255	1215
7	2	481	422
8	2	417	328
11	2	201	111
12	2	1026	1063
13	2	182	278
14	2	1133	1165
15	2	448	455
16	2	1240	1330
17	2	723	702
18	2	1224	1253
19	2	234	325
20	2	1361	1307
22	2	782	749
24	2	489	469
2	3	556	523
3	3	652	673
4	3	1565	1541
5	3	1884	1775
6	3	2902	2817
7	3	1399	1273
8	3	3002	3229
9	3	446	382
10	3	2669	2738
12	3	1522	1684
13	3	736	704
14	3	843	817
15	3	622	574
16	3	216	291
17	3	224	269
22	3	701	615
23	3	246	307
24	3	1174	948
1	4	803	728
2	4	475	520
3	4	1817	1577
4	4	1399	1403
5	4	1070	926
6	4	1420	1264
8	4	931	832
9	4	382	348
10	4	590	540
12	4	1042	1072
13	4	448	467
14	4	1052	1103
15	4	749	718
16	4	707	765
17	4	462	462
18	4	750	748
20	4	348	480
21	4	248	295
22	4	349	410
24	4	336	351
1	5	191	190
2	5	693	590
3	5	754	637
4	5	1590	1460
5	5	895	745
6	5	2122	2176
7	5	855	695
8	5	2342	2280
9	5	186	251
10	5	2595	2616
11	5	629	575
12	5	1916	1978
13	5	705	694
14	5	850	921
16	5	403	431
22	5	423	473
24	5	540	516
0	6	1793	1823
2	6	1173	1138
4	6	1074	1080
5	6	184	281
6	6	1017	847
9	6	496	399
10	6	208	332
11	6	151	197
12	6	969	977
14	6	938	987
15	6	289	367
16	6	1216	1377
18	6	781	821
20	6	349	474
1	7	340	301
2	7	198	301
3	7	345	315
4	7	779	747
5	7	828	777
6	7	1226	1143
7	7	699	648
8	7	1349	1370
10	7	1404	1452
12	7	1259	1298
14	7	979	994
23	7	152	217
24	7	349	305
0	8	1433	1517
1	8	531	483
2	8	1288	1325
3	8	1002	882
4	8	1250	1313
5	8	1219	1110
6	8	780	709
7	8	645	538
8	8	231	266
10	8	168	349
11	8	170	326
12	8	769	834
13	8	650	643
14	8	856	969
15	8	701	693
16	8	699	837
18	8	724	705
20	8	723	606
22	8	488	414
2	9	288	377
4	9	473	470
5	9	238	318
6	9	1059	1066
7	9	171	250
8	9	1032	1111
10	9	817	925
12	9	554	615
14	9	246	325
22	9	289	360
24	9	527	479
0	10	1240	1221
2	10	1347	1224
4	10	780	761
5	10	247	242
6	10	700	629
7	10	175	261
8	10	175	240
9	10	175	200
10	10	174	169
12	10	297	356
[illegible]	[illegible]	313	[illegible]
16	10	590	532
18	10	711	662
20	10	724	672
22	10	552	525
1	11	246	319
2	11	246	401
3	11	246	363
4	11	850	763
5	11	300	352
6	11	975	830
7	11	242	306
8	11	340	459
9	11	168	78
10	11	288	395
12	11	277	348
0	12	1261	1109
2	12	1127	921
4	12	630	578
16	12	627	590
18	12	615	615
****	H = 1		****
3	0	1273	1834
4	0	276	313
5	0	1231	1645
7	0	1025	1368
9	0	1071	1272
10	0	252	251
11	0	1748	1649
12	0	547	430
13	0	1144	1207
14	0	156	201
15	0	870	911
16	0	485	378
17	0	126	196
18	0	186	223
19	0	192	235
20	0	197	206
21	0	603	561
23	0	456	492
25	0	623	483
27	0	569	435
29	0	573	486
31	0	452	422
2	1	693	517
4	1	1103	1227
5	1	1569	1755
6	1	485	506
7	1	618	709
8	1	321	355
11	1	662	667
12	1	1016	814
13	1	976	1106
15	1	1633	1673
17	1	1622	1599
19	1	1023	1085
20	1	198	275
21	1	698	826
23	1	407	429
25	1	196	166
29	1	215	187
31	1	192	265
0	2	1290	1158
1	2	1651	1479
2	2	244	142
3	2	787	786
5	2	1773	1930
6	2	621	539
7	2	2040	2279
8	2	119	97
9	2	2159	2515
10	2	266	243
11	2	1546	1830
13	2	818	933
14	2	161	164
15	2	533	518
16	2	176	183
17	2	183	204
19	2	195	190
21	2	701	650
23	2	813	742
25	2	1034	845
27	2	935	764
29	2	553	467
31	2	233	241
1	3	1814	1888
2	3	787	673
3	3	1681	1582
4	3	343	323
5	3	1932	1799
6	3	684	613
7	3	602	601
8	3	682	676
10	3	445	412
11	3	674	717
12	3	154	235
13	3	735	803
14	3	205	311
15	3	603	699
16	3	181	205
17	3	648	640
18	3	632	130*
19	3	684	745
20	3	201	245
21	3	1186	978
22	3	204	219
23	3	857	668
25	3	191	303
29	3	244	211
31	3	204	206
0	4	368	356
1	4	589	495
2	4	908	795
3	4	924	886
4	4	714	612
5	4	1709	1715
7	4	2371	2548
8	4	312	282
9	4	2027	2105
10	4	150	189
11	4	1547	1689
12	4	199	253
13	4	896	935
15	4	573	558
20	4	575	498
21	4	577	615
23	4	956	779
25	4	1034	844
27	4	906	665
29	4	486	377
1	5	2128	2090
2	5	378	311
3	5	1895	1891
4	5	239	199
5	5	1003	982
6	5	499	454
7	5	468	403
8	5	305	257
11	5	410	379
13	5	724	763
15	5	1003	1051
17	5	874	1009
19	5	932	967
21	5	929	818
22	5	200	165
23	5	434	466
24	5	187	201
29	5	442	124*
0	6	682	684
1	6	517	455
2	6	424	382
3	6	1206	1114
4	6	815	688
5	6	1322	1292
6	6	502	433
7	6	944	956
9	6	903	926
11	6	820	850
13	6	862	898
14	6	236	396
15	6	681	712
16	6	489	539
17	6	202	288
18	6	204	248
19	6	204	286
21	6	487	405
23	6	492	433
25	6	165	315
27	6	430	329
1	7	2166	2129
2	7	166	203
[illegible]	7	[illegible]	1444
4	7	508	384
5	7	757	784
6	7	174	177
8	7	312	307
10	7	264	363
11	7	330	400
12	7	548	553
13	7	763	807
15	7	1107	1109
17	7	1186	1310
19	7	1166	1018
21	7	603	584
0	8	256	292
1	8	257	301
2	8	257	288
3	8	808	795
5	8	988	925
7	8	931	920
9	8	893	897
11	8	881	929
13	8	574	633
15	8	576	603
16	8	287	371
17	8	201	280
18	8	241	215
21	8	177	286
23	8	312	336
25	8	400	341
1	9	827	815
3	9	898	838
5	9	874	802
6	9	199	189
7	9	200	203
8	9	570	565
10	9	538	522
11	9	288	406
12	9	204	314
13	9	642	544
15	9	745	689
17	9	603	575
19	9	561	473
21	9	418	396
23	9	229	234
2	10	203	251
3	10	288	376
4	10	538	515
5	10	864	758
7	10	1186	1066
9	10	1447	1276
11	10	1065	986
12	10	198	333
13	10	618	632
14	10	191	319
21	10	186	266
22	10	163	232
23	10	396	365
1	11	606	483
3	11	570	486
5	11	694	570
6	11	199	285
7	11	198	304
8	11	277	300
10	11	191	337
13	11	333	395
15	11	437	440
17	11	331	368
19	11	395	281
0	12	268	317
2	12	189	278
3	12	266	326
4	12	187	245
5	12	414	406
6	12	183	235
7	12	651	622
9	12	767	681
11	12	697	630
12	12	158	262
13	12	454	395
14	12	144	233
16	12	124	188
1	13	659	607
3	13	609	511
4	13	161	259
5	13	463	406
6	13	156	224
7	13	152	216
8	13	148	189
13	13	295	356
1	-1	1638	2693*
2	-1	583	502
3	-1	2232	3289*
4	-1	266	327
5	-1	1110	1262
7	-1	450	544
10	-1	128	160
11	-1	331	407
12	-1	653	491
13	-1	1355	1331
15	-1	1633	1738
17	-1	1777	1868
18	-1	254	339
19	-1	1252	1252
20	-1	469	391
21	-1	897	754
23	-1	281	376
29	-1	150	169
31	-1	311	284
1	-2	1799	1618
2	-2	333	330
3	-2	974	978
4	-2	870	836
5	-2	1801	1869
6	-2	213	186
9	-2	1829	2250*
10	-2	325	437
11	-2	1693	2046
12	-2	277	354
13	-2	1002	1124
14	-2	456	433
15	-2	826	803
17	-2	248	346
18	-2	182	198
19	-2	266	333
20	-2	273	342
21	-2	556	537
23	-2	742	631
25	-2	714	591
27	-2	649	528
29	-2	462	376
31	-2	328	256
1	-3	1758	1676
2	-3	747	649
3	-3	1855	1747
5	-3	1451	1383
6	-3	699	652
7	-3	652	722
9	-3	125	146
10	-3	323	263
11	-3	855	831
13	-3	933	1037
15	-3	1012	1103
17	-3	956	974
19	-3	907	896
21	-3	1019	880
23	-3	624	569
29	-3	197	221
31	-3	240	270
0	-4	368	350
1	-4	337	323
2	-4	477	319
3	-4	505	523
4	-4	438	395
5	-4	1528	1580
7	-4	2381	2422
8	-4	395	348
9	-4	2266	2476
10	-4	369	358
11	-4	1729	2038
12	-4	514	498
13	-4	981	1130
14	-4	118	223
15	-4	603	619
21	-4	628	558
23	-4	552	513
25	-4	836	656
27	-4	681	583
29	-4	446	374
31	-4	156	200
1	-5	1675	1643
3	-5	1468	1519
4	-5	992	850
5	-5	1599	1472
7	-5	662	429
10	-5	308	340
11	-5	618	665
12	-5	286	403
13	-5	995	1124
14	-5	395	386
15	-5	1059	1267
17	-5	847	983
19	-5	740	701
20	-5	526	540
21	-5	840	708
22	-5	239	337
23	-5	269	401
0	-6	199	189
1	-6	488	475
3	-6	701	661
4	-6	145	130
5	-6	1334	1206
7	-6	1394	1426
9	-6	1468	1554
10	-6	236	281
11	-6	1118	1272
13	-6	1010	1056
15	-6	538	563
21	-6	275	325
23	-6	342	360
25	-6	515	416
27	-6	645	503
29	-6	478	462
1	-7	1561	1724
2	-7	449	353
3	-7	1444	1421
4	-7	560	456
5	-7	953	984
6	-7	166	198
7	-7	536	464
11	-7	450	453
12	-7	187	238
13	-7	905	950
15	-7	867	979
17	-7	1070	970
19	-7	784	747
21	-7	561	467
23	-7	171	270
0	-8	174	181
1	-8	247	271
3	-8	934	853
4	-8	532	475
5	-8	1042	1047
6	-8	573	555
7	-8	1193	1225
8	-8	186	226
9	-8	1096	1195
11	-8	1027	1034
12	-8	553	566
13	-8	558	541
14	-8	281	410
15	-8	487	467
21	-8	247	318
23	-8	555	488
25	-8	663	556
1	-9	1001	1071
2	-9	378	424
3	-9	1103	1137
4	-9	504	503
5	-9	778	795
7	-9	275	319
11	-9	628	326
13	-9	280	419
15	-9	534	522
17	-9	530	516
19	-9	604	551
21	-9	644	562
23	-9	434	420
3	-10	685	561
5	-10	1131	946
7	-10	1107	924
9	-10	1104	944
11	-10	734	691
13	-10	271	401
21	-10	345	365
1	-11	1100	1084
3	-11	985	796
5	-11	679	578
15	-11	540	466
17	-11	687	625
19	-11	673	622
21	-11	455	495
1	-12	228	243
3	-12	640	563
5	-12	531	474
6	-12	180	291
7	-12	397	423
9	-12	363	363
11	-12	303	361
13	-12	366	396
15	-12	339	346
1	-13	945	841
3	-13	622	575
5	-13	248	300
13	-13	350	325
****	H = 2		****
3	0	584	654
4	0	648	817
5	0	336	365
6	0	684	773
7	0	604	631
8	0	186	212
9	0	759	747
10	0	512	554
11	0	715	732
12	0	1197	1185
13	0	244	306
14	0	1260	1179
16	0	1239	1238
18	0	1063	942
20	0	624	459
30	0	233	282
2	1	123	45
3	1	745	594
4	1	746	833
5	1	506	375
6	1	1579	1791
7	1	437	386
8	1	1986	2380*
10	1	2000	2315
12	1	1872	2005
13	1	174	199
14	1	1082	1091
16	1	484	450
17	1	410	372
18	1	211	199
19	1	307	247
22	1	224	271
24	1	534	451
26	1	634	519
28	1	646	586
30	1	495	484
0	2	142	150
1	2	799	725
2	2	1581	1532
3	2	2330	2264
4	2	1911	1830
5	2	2102	2070
6	2	1631	1577
7	2	1230	1125
8	2	751	708
9	2	879	850
10	2	310	401
11	2	838	865
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16	1	460	412
17	1	874	905
19	1	666	650
21	1	374	363
22	1	363	345
24	1	494	398
26	1	139	211
0	2	1262	1271
2	2	1301	1316
3	2	796	736
4	2	943	935
5	2	725	668
6	2	330	302
7	2	398	274
9	2	217	247
11	2	331	308
12	2	296	320
13	2	177	201
14	2	513	500
16	2	535	487
18	2	746	736
19	2	192	264
20	2	657	636
21	2	454	413
22	2	508	433
23	2	171	265
25	2	149	185
1	3	723	663
2	3	598	489
3	3	1015	940
4	3	958	959
5	3	987	916
6	3	1190	1178
7	3	712	635
8	3	1039	1048
10	3	993	1000
11	3	171	248
12	3	585	653
13	3	600	563
14	3	585	526
15	3	533	572
16	3	191	278
17	3	544	548
19	3	381	369
21	3	258	321
24	3	441	448
26	3	443	394
0	4	611	504
2	4	437	310
3	4	511	462
5	4	265	235
6	4	272	298
7	4	738	596
9	4	1014	955
11	4	690	640
16	4	272	184
17	4	192	318
18	4	270	324
20	4	183	243
1	5	552	439
2	5	707	559
3	5	714	580
4	5	979	914
5	5	907	823
6	5	1010	935
7	5	759	580
8	5	1048	1026
10	5	994	993
11	5	585	619
12	5	861	916
13	5	699	766
14	5	606	663
15	5	471	465
17	5	190	228
19	5	259	230
20	5	177	202
22	5	226	309
23	5	149	190
24	5	271	255
26	5	139	269
0	6	598	476
2	6	575	514
4	6	466	348
5	6	503	402
6	6	254	221
7	6	773	709
9	6	792	726
11	6	738	744
13	6	430	420
14	6	385	489
15	6	191	295
16	6	354	431
18	6	362	386
25	6	138	197
1	7	611	519
3	7	185	128
6	7	533	424
8	7	633	644
10	7	638	648
12	7	543	569
13	7	135	201
14	7	189	321
15	7	186	280
17	7	177	265
19	7	162	180
0	8	382	358
2	8	703	678
3	8	636	586
4	8	665	615
5	8	815	814
6	8	609	590
7	8	667	652
8	8	192	288
9	8	543	553
10	8	135	244
11	8	536	491
12	8	458	524
13	8	553	617
14	8	477	564
15	8	248	382
16	8	208	355
18	8	154	272
20	8	188	231
21	8	169	205
22	8	145	238
23	8	181	258
1	9	470	359
3	9	234	176
6	9	268	289
8	9	495	419
10	9	483	452
19	9	169	223
21	9	114	159
0	10	521	526
2	10	519	468
3	10	317	343
4	10	446	418
5	10	361	395
6	10	253	342
7	10	467	433
8	10	174	185
9	10	341	336
11	10	229	269
14	10	203	212
16	10	177	247
18	10	239	263
1	11	553	520
3	11	481	443
4	11	231	317
5	11	228	289
6	11	225	319
0	12	421	435
2	12	405	382
1	-1	582	917
2	-1	50	114
3	-1	436	754
4	-1	625	824
5	-1	221	224
6	-1	493	515
8	-1	489	556
9	-1	209	292
10	-1	489	467
11	-1	219	310
12	-1	530	540
13	-1	348	309
14	-1	472	423
15	-1	441	365
17	-1	235	291
19	-1	542	453
20	-1	485	370
21	-1	417	355
22	-1	333	342
24	-1	217	273
26	-1	188	232
27	-1	118	179
28	-1	277	242
0	-2	903	1235
1	-2	265	257
2	-2	782	814
3	-2	304	192
4	-2	1004	997
5	-2	787	776
6	-2	966	1017
7	-2	955	1008
8	-2	465	451
9	-2	593	690
12	-2	593	560
11	-2	720	700
13	-2	506	443
14	-2	748	730
15	-2	614	538
16	-2	694	658
18	-2	539	507
20	-2	542	454
22	-2	467	417
23	-2	225	307
24	-2	302	323
25	-2	399	371
27	-2	160	260
1	-3	443	377
3	-3	124	107
4	-3	439	354
6	-3	1124	1104
8	-3	1403	1532
9	-3	307	252
10	-3	1216	1286
11	-3	341	322
12	-3	748	882
13	-3	152	148
14	-3	223	300
21	-3	167	168
22	-3	162	207
24	-3	464	369
26	-3	424	426
28	-3	295	339
0	-4	326	269
1	-4	311	231
2	-4	447	359
3	-4	723	549
4	-4	973	823
5	-4	1068	948
6	-4	954	802
7	-4	882	763
8	-4	508	369
9	-4	819	714
10	-4	143	196
11	-4	703	691
12	-4	652	665
13	-4	583	446
14	-4	632	670
15	-4	289	353
16	-4	415	438
17	-4	171	227
18	-4	243	318
20	-4	237	317
22	-4	157	217
23	-4	149	246
24	-4	140	223
25	-4	128	227
27	-4	181	205
1	-5	391	341
2	-5	353	339
4	-5	706	693
6	-5	814	818
8	-5	1295	1193
10	-5	1123	1091
12	-5	885	867
14	-5	505	551
15	-5	170	269
17	-5	454	424
22	-5	150	204
24	-5	258	297
26	-5	271	266
0	-6	942	859
2	-6	850	791
4	-6	504	398
5	-6	148	174
6	-6	213	174
7	-6	376	381
9	-6	452	387
11	-6	438	367
12	-6	238	292
13	-6	170	228
14	-6	453	473
16	-6	541	602
18	-6	551	562
20	-6	220	279
1	-7	442	357
2	-7	415	301
3	-7	723	651
4	-7	779	751
5	-7	681	642
6	-7	744	668
7	-7	465	427
8	-7	675	619
10	-7	689	649
11	-7	121	321
12	-7	786	830
13	-7	486	485
14	-7	484	532
15	-7	479	518
16	-7	289	349
17	-7	200	314
19	-7	152	208
21	-7	135	189
22	-7	175	206
23	-7	155	148
24	-7	218	228
0	-8	911	832
2	-8	767	719
3	-8	168	287
4	-8	559	493
12	-8	170	268
14	-8	234	358
16	-8	417	431
18	-8	436	403
20	-8	312	304
22	-8	141	224
1	-9	454	454
2	-9	172	215
3	-9	384	358
4	-9	486	401
5	-9	384	374
6	-9	594	575
7	-9	121	201
8	-9	589	630
10	-9	473	480
12	-9	230	345
13	-9	112	170
14	-9	154	228
15	-9	149	240
0	-10	714	700
2	-10	650	625
4	-10	528	521
6	-10	233	348
16	-10	170	279
18	-10	269	351
4	-11	216	317
6	-11	365	397
8	-11	321	355
0	-12	680	609
2	-12	529	528
4	-12	388	348
5	-12	179	241
6	-12	175	226

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[a] Values listed for F_o and F_c are 10 times the actual F_o and F_c. Structure factors marked with an asterisk were not used for final refinement.

TABLE II

FINAL ATOMIC PARAMETERS[a,b]

A. Atomic Coordinates and Isotropic Temperature Factors

Atom	x	y	z	B, A^2
Re	0.2736 (2)	0.02819 (4)	0.2532 (1)	
Cl (1)	0.2737 (16)	−0.0339 (3)	0.0999 (7)	
Cl (2)	0.2625 (14)	0.0880 (3)	0.4096 (6)	
Cl (3)	0.0204 (15)	−0.0198 (3)	0.3411 (7)	
Cl (4)	0.5306 (16)	0.0748 (3)	0.1729 (7)	
Cl (5)	0.4961 (13)	−0.0252 (3)	0.3526 (6)	
Cl (6)	0.0537 (14)	0.0796 (3)	0.1491 (6)	
N (1)	−0.252 (4)	0.078 (1)	0.431 (2)	3.5 (5)
N (2)	0.250 (4)	0.437 (1)	0.385 (2)	4.8 (6)
C (1)	−0.229 (5)	0.135 (1)	0.460 (3)	4.5 (7)
C (2)	−0.157 (6)	0.171 (1)	0.378 (3)	5.0 (8)
C (3)	−0.133 (6)	0.223 (1)	0.400 (3)	4.7 (7)
C (4)	−0.179 (6)	0.246 (1)	0.503 (3)	5.0 (8)
C (5)	−0.268 (7)	0.208 (2)	0.592 (4)	6.2 (9)
C (6)	−0.276 (6)	0.153 (2)	0.567 (3)	5.4 (8)
C (7)	−0.160 (7)	0.303 (2)	0.538 (4)	6.7 (10)
C (8)	0.266 (5)	0.379 (1)	0.349 (3)	4.2 (6)
C (9)	0.334 (5)	0.344 (1)	0.430 (3)	3.7 (6)
C (10)	0.362 (6)	0.293 (1)	0.395 (3)	5.0 (8)
C (11)	0.307 (6)	0.275 (2)	0.287 (3)	5.8 (9)
C (12)	0.242 (7)	0.312 (2)	0.206 (4)	6.4 (10)
C (13)	0.219 (5)	0.364 (1)	0.239 (3)	4.6 (7)
C (14)	0.338 (6)	0.217 (2)	0.249 (3)	5.4 (8)

B. Anisotropic Temperature Factors

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Re	0.0292 (6)	0.0013 (2)	0.0055 (1)	0.0007 (2)	0.0007 (3)	−0.0001 (1)
Cl (1)	0.0439 (33)	0.0016 (1)	0.0056 (6)	0.0019 (10)	−0.0037 (21)	−0.0029 (5)
Cl (2)	0.0318 (33)	0.0012 (1)	0.0061 (6)	0.0013 (9)	0.0021 (18)	−0.0010 (4)
Cl (3)	0.0303 (36)	0.0015 (1)	0.0072 (6)	−0.0027 (9)	0.0029 (20)	−0.0001 (5)
Cl (4)	0.0379 (38)	0.0017 (2)	0.0073 (7)	−0.0023 (10)	0.0038 (22)	0.0023 (5)
Cl (5)	0.0215 (31)	0.0013 (1)	0.0059 (5)	0.0004 (9)	−0.0022 (16)	0.0001 (4)
Cl (6)	0.0295 (34)	0.0018 (1)	0.0060 (6)	0.0026 (10)	−0.0040 (19)	0.0013 (5)

[a] Anisotropic temperature factors are of the form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$. [b] Standard deviations are given in parentheses for the last significant figure of atomic coordinates and thermal parameters.

obvious blunders reduced R to a final value of 0.109 after four more cycles of refinement. The final values of $|F_o|$ and $|F_c|$ are listed in Table I. Table II gives the final atomic parameters. The anisotropic temperature factors probably have little physical significance since they tend to compensate for inaccurate scaling of layers, neglect of absorption corrections, and neglect of anomalous dispersion corrections.

Results

The crystal structure consists of octahedral hexachlororhenate anions packed with planar p-toluidinium cations. Figures 1 and 2 show projections onto the yz and xz planes, respectively. Bond lengths and bond angles are given in Table III. The standard deviations in bond lengths estimated from least-squares calculations are 0.06 A for bonds between light atoms and 0.02 A for Re–Cl bonds. Although these standard deviations are somewhat high, the Re–Cl bonds average 2.35 A, in reasonable agreement with the value 2.37 A found in K_2ReCl_6,[7] and the average C–C bond within a ring is 1.39 A, in good agreement with the value 1.394 A given by Sutton[8] for an aromatic C–C bond.

TABLE III

INTERATOMIC DISTANCES AND ANGLES

A. Re–Cl Octahedron

Distances, A		Angles, deg			
Re–Cl(1)	2.35	Cl(1)–Re–Cl(3)	89.3	Cl(2)–Re–Cl(5)	90.6
Re–Cl(2)	2.35	Cl(1)–Re–Cl(4)	91.8	Cl(2)–Re–Cl(6)	91.3
Re–Cl(3)	2.37	Cl(1)–Re–Cl(5)	89.5	Cl(3)–Re–Cl(5)	90.0
Re–Cl(4)	2.34	Cl(1)–Re–Cl(6)	88.7	Cl(3)–Re–Cl(6)	90.3
Re–Cl(5)	2.35	Cl(2)–Re–Cl(3)	88.2	Cl(4)–Re–Cl(5)	88.1
Re–Cl(6)	2.34	Cl(2)–Re–Cl(4)	90.7	Cl(4)–Re–Cl(6)	91.7

B. p-Toluidinium Rings

Distances, A

C(1)–C(2)	1.40	C(8)–C(9)	1.37
C(2)–C(3)	1.35	C(9)–C(10)	1.37
C(3)–C(4)	1.36	C(10)–C(11)	1.39
C(4)–C(5)	1.52	C(11)–C(12)	1.38
C(5)–C(6)	1.42	C(12)–C(13)	1.38
C(6)–C(1)	1.34	C(13)–C(8)	1.36
N(1)–C(1)	1.48	N(2)–C(8)	1.52
C(4)–C(7)	1.47	C(11)–C(14)	1.54

Angles, deg

C(1)–C(2)–C(3)	123	C(8)–C(9)–C(10)	117
C(2)–C(3)–C(4)	123	C(9)–C(10)–C(11)	122
C(3)–C(4)–C(5)	115	C(10)–C(11)–C(12)	119
C(5)–C(6)–C(1)	120	C(11)–C(12)–C(13)	120
C(6)–C(1)–C(2)	120	C(12)–C(13)–C(8)	119
N(1)–C(1)–C(2)	120	C(13)–C(8)–C(9)	123
N(1)–C(1)–C(6)	121	N(2)–C(8)–C(9)	117
C(7)–C(4)–C(5)	117	N(2)–C(8)–C(13)	120
C(7)–C(4)–C(3)	128	C(14)–C(11)–C(10)	121
C(4)–C(5)–C(6)	119	C(14)–C(11)–C(12)	119

The $ReCl_6$ octahedra are arranged in parallel sheets stacked perpendicularly to [010] with a threefold axis

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(7) R. W. G. Wyckoff, "Crystal Structures," Vol. III, Interscience Publishers, Inc., New York, N. Y., 1960, Chapter IX, tables, p 11.

(8) "Table of Interatomic Distances and Configurations in Molecules and Ions," The Chemical Society, London, 1965, p S 16s.

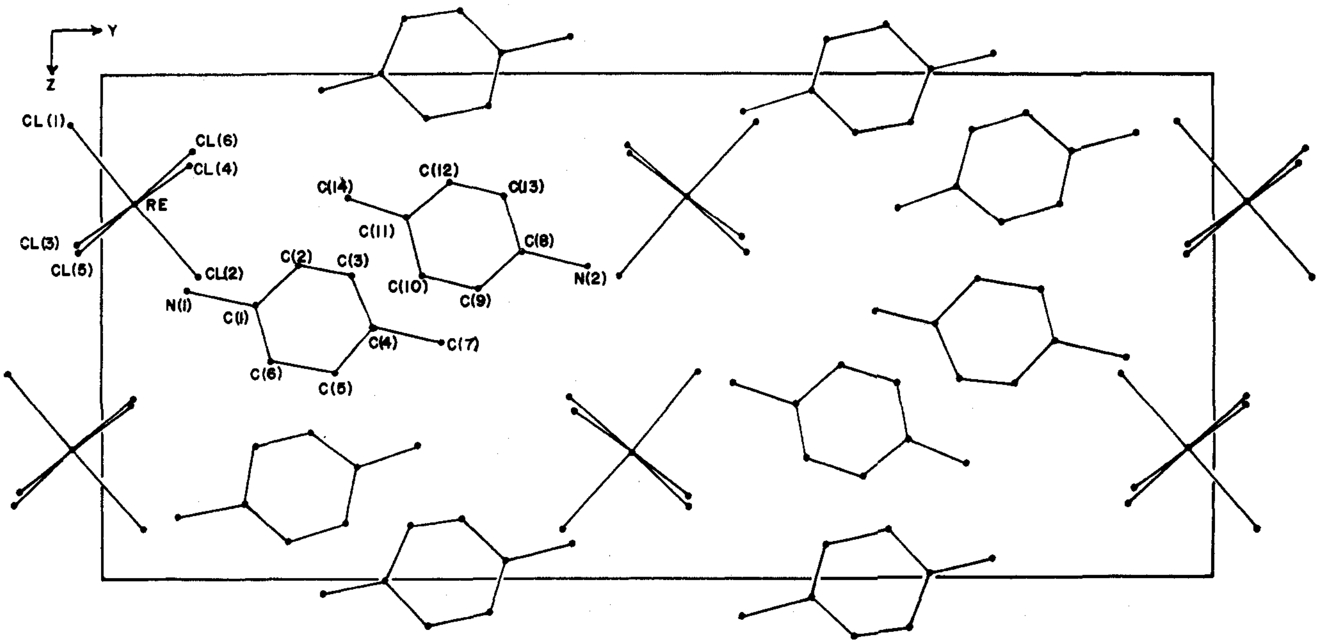

Figure 1.—View of structure of $(TH)_2ReCl_6$ along [100].

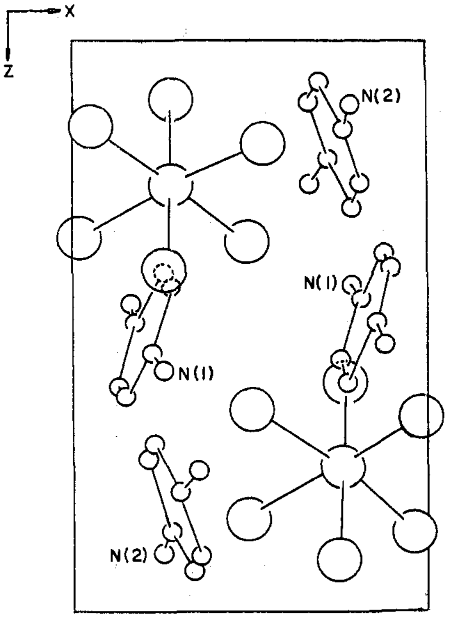

Figure 2.—View of structure of $(TH)_2ReCl_6$ along [010] showing one-half of unit cell from $y = -1/4$ to $y = 1/4$.

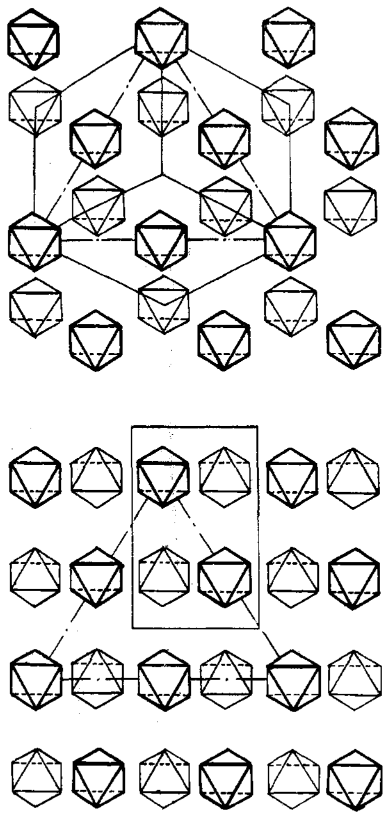

Figure 3.—Top: projection of $ReCl_6$ octahedra onto [111] planes of K_2ReCl_6. Bottom: projection of $ReCl_6$ octahedra onto [010] planes of $(TH)_2ReCl_6$. (Heavily outlined octahedra are in one plane, while light octahedra are in another. Only two planes are shown in each figure.)

of the octahedron parallel to [010]. These sheets are separated by the organic cations such that the distance between sheets is $^1/_2b$ (12.52 A). Similar sheets are found in the K_2ReCl_6 structure, stacked perpendicularly to [111] of the cubic unit cell. The principal differences between the sheets in the two structures are: (1) in K_2ReCl_6 the Re atoms are coplanar whereas in $(TH)_2ReCl_6$ they are staggered 0.70 A above and below a mean plane and (2) in K_2ReCl_6 the planes are separated only by potassium atoms so that the separation is 5.70 A whereas the organic cation packing in $(TH)_2ReCl_6$ results in the 12.52-A separation. The stacking of the sheets in the two structures is shown in Figure 3. A layer structure similar to $(TH)_2ReCl_6$ consisting of sheets of heavy-atom octahedra alternated with substituted ammonium groups has been proposed by Wyckoff[9] for $(CH_3NH_3)_2SnCl_6$, $(C_2H_5NH_3)_2SnCl_6$, $(CH_3NH_3)_2PtCl_6$, and $(C_2H_5NH_3)_2PtCl_6$, which is further supported by the structure we have determined.

The nitrogen atoms of the p-toluidinium groups

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(9) R. W. G. Wyckoff, "Crystal Structures," Vol. IV, Interscience Publishers, Inc., New York, N. Y., 1960, Chapter XIII, p 10; illustration, p 4.

occupy approximately the same position relative to the $ReCl_6$ octahedra in a sheet as the potassium atoms in K_2ReCl_6. The average N–Cl distance is 3.59 A; that of K–Cl is 3.49 A. The average N–Re distance is 4.38 A; that of K–Re is 4.29 A.

Magnetic exchange is still possible in $(TH)_2ReCl_6$ since the nearest neighbor Re–Re distance (six nearest neighbors, average distance 6.94 A) is about the same as that in K_2ReCl_6 (6.98 A, 12 nearest neighbors). The nearest neighbor nonbonded Cl–Cl distances (not in the same octahedra) average 3.73 A in $(TH)_2ReCl_6$ and are 3.63 A in K_2ReCl_6.

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Some Reactions of the Octahalodirhenate(III) Ions.
III. The Stability of the Rhenium–Rhenium Bond toward Oxygen and Sulfur Donors[1]

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The reactions of the $Re_2Cl_8^{2-}$ and $Re_2Br_8^{2-}$ ions with several carboxylic acids and sulfur ligands have been studied. The following carboxylates were prepared and their spectral properties investigated: $Re_2(O_2CCH_3)_2X_4 \cdot 2L$, $Re_2(O_2CCH_3)_3Br_3 \cdot H_2O$, $Re_2(O_2CR)_2(O_2CCl_3)_2Cl_2$, and $Re_2(O_2CCCl_3)_2Cl_4$, where L = H_2O or pyridine, X = Cl or Br, and R = CH_3 or $(CH_3)_3C$. Evidence for their dinuclear structures is presented. The $Re_2X_8^{2-}$ ions react with thiourea (tu) and tetramethylthiourea (tmtu) in acidified methanol to yield the complexes $[ReX_3(tu)_3]$ and $[ReX_3(tmtu)]_n$, respectively. In acetone the thiourea reaction gave the acetone solvates $ReX_3(tu)_3 \cdot {}^1/_3(CH_3)_2CO$. The thiourea complexes are believed to be true complexes of the type ReX_3L_3, only two of which have previously been reported in the literature. Spectral evidence in the case of $[ReX_3(tmtu)]_n$ suggests that $n = 2$ and that these complexes contain a Re–Re bond. $[(C_6H_5)_4As]_2[Re_2(C_2S_2(CN)_2)_4]$ is only the second compound reported in which a *cis*-1,2-disubstituted ethylene-1,2-dithiolate ligand is bonded to rhenium. The reaction of 2,5-dithiahexane (DTH) with $Re_2X_8^{2-}$ yields the complexes $[ReX_3(DTH)]_n$, $Re_2Cl_5(DTH)_2$, or $[ReBr_2(DTH)]_n$ depending upon the reaction conditions. Possible structures are suggested for these complexes on the basis of diffuse reflectance and infrared spectral studies. From their infrared spectra it is concluded that 2,5-dithiahexane assumes its *gauche* conformation on coordination. The infrared spectrum of the polymeric complex $Re_3Cl_9DTH_{1.5}$ is also reported and the ligand shown to have the *trans* conformation. This is the first example where this molecular form has been stabilized by complex formation. The complexes $ReCl_3(bipy)$ and $ReCl_3(CEP)_2 \cdot C_2H_5OH$, where CEP = tris(2-cyanoethyl)phosphine, have also been prepared and characterized.

Introduction

The reactivity and stability of the trimeric rhenium(III) halides is now well understood.[2] However, although the octahalodirhenate(III) ions $Re_2X_8^{2-}$, where X = Cl or Br, have been isolated,[3] and their electronic[4] and molecular[5,6] structures investigated, little information is yet available on their stability and reactivity. The most significant results of the earlier studies[4,6] are that the $Re_2X_8^{2-}$ species contain a Re–Re quadruple bond and an eclipsed rotational configuration.

We are at present investigating the reactions of these ions with a variety of donor molecules in an attempt to isolate new complexes in which the Re–Re bond may be broken, retained, or modified. A study of those complexes which contain a Re–Re bond should then provide information on such factors as the relationship between bond multiplicity and the molecular configuration (eclipsed or staggered).

Previous papers in this series have described the reactions of $Re_2X_8^{2-}$ with phosphines[7] and carboxylic acids.[8] A Re–Re bond is believed to be present in several of these phosphine complexes,[7] as is almost certainly the case with the tetracarboxylatodirhenate(III) species $Re_2(O_2CR)_4X_2$.[8] In the latter complexes the bridging nature of the carboxylate groups would be expected to favor the retention of a Re–Re bond.[9] In the present work further reactions of the octahalodirhenate(III) ions are reported and several new complexes containing rhenium–sulfur bonds described.

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(1) Research supported by the United States Atomic Energy Commission under Contract AT(30-1)-1965 and the National Science Foundation under grant No. GP-4329.

(2) F. A. Cotton and R. A. Walton, *Inorg. Chem.*, **5**, 1802 (1966), and earlier references therein.

(3) F. A. Cotton, N. F. Curtis, B. F. G. Johnson, and W. R. Robinson, *ibid.*, **4**, 326 (1965).

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(5) F. A. Cotton and C. B. Harris, *ibid.*, **4**, 330 (1965).

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