The Conformation of a Nine-membered Ring

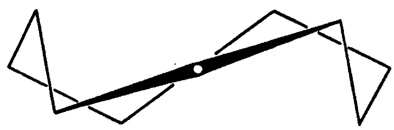
Crystal Structure of the 1 : 1 Addition Compound Mercuric Chloride-Cyclononanone

S. DAHL and P. GROTH

Department of Chemistry, University of Oslo, Oslo 3, Norway

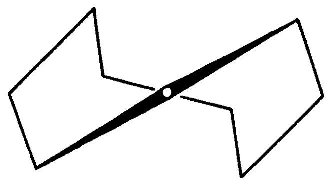
The crystals are monoclinic with space group $P2_1/c$ and cell dimensions $a=7.58_8$ Å, $b=12.21_4$ Å, $c=25.14_8$ Å, and $\beta=96.2_6$°. The unit cell contains eight formula units. The structure was solved by the heavy atom method and refined by full-matrix least squares technique ($R=11.6$ %, and $R_w=11.1$ % for 1550 reflections recorded on an automatic four circle diffractometer). Although the standard deviations of light atom parameters are large, it may be stated that the nine-membered ring conformation corresponds to that of a "twisted chair boat" with approximately C_2 symmetry. The charge transfer bonding system consists of infinite chains of alternating $HgCl_2$ and cyclononanone molecules in the [100]-direction.

Strain-energy minimization calculations [1,2] for the saturated nine-membered ring point out the "twisted boat chair" (TBC), with symmetry D_3:



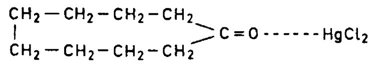
as the energetically favourable conformation. This form has indeed been observed in the crystals of trimeric acetone peroxide,[3] where the ring skeleton consists of six oxygens and three carbon atoms.

The only crystal structure analysis reported of a saturated nine-membered ring is that of cyclononylammonium bromide,[4] where the ring conformation roughly may be described in terms of a "twisted chair boat" (TCB), with a two-fold axis of symmetry:



This conformation has no similarity at all to the TBC-form, and the difference in strain energies amounts to about 3 kcal/mol.

The crystal structure of the 1 : 1 addition compound mercuric chloride-cyclononanone has been carried out in order to settle the conformational problem of the cyclic ketone:



The crystals are monoclinic with space group $P2_1/c$. The cell dimensions, determined on a manual four circle diffractometer, with estimated standard deviations [5]* are:

$$a = 7.588(2)\ \text{Å},\ b = 13.214(3)\ \text{Å},\ c = 25.148(6)\ \text{Å},\ \beta = 96.26(2)^\circ$$

The unit cell contains eight molecules ($\varrho_{calc} = 2.2$ g/cm^3, $\varrho_{obs} \sim 2.0$ g/cm^3).

The crystals decompose quickly in air. When exposed to X-rays, the decomposition is rapid even when the crystals are kept in a sealed capillary tube. Out of 14 specimens mounted, three lasted long enough to allow intensity measurements to be carried out. The intensities were recorded on an automatic four circle diffractometer. Using an observed-unobserved cut-off at 2.0 $\sigma(I)$, a total number of 1550 reflections were observed. The intensities were corrected for absorption effects.

The structure was solved by the heavy atom method and refined by full-matrix least squares technique. Hydrogen positions were calculated assuming C–H bond lengths of 1.08 Å and included in structure factor calculations, but not refined. Anisotropic temperature factors were introduced for mercury and chloride atoms. The weights in least squares were calculated from the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I) = (C_T + (0.02\ C_N)^2)^{\frac{1}{2}}$$

where C_T is the total number of counts, and C_N the net count (peak minus background). Decomposition of the crystals during intensity measurements is probably the main reason for the fact that the weighted R-value arrived at was as large as $R_W = 11.1$ % (conventional value, $R = 11.6$ %) for 1550 observed reflections. The form factors used were those of Hanson *et al.*[6]

Final fractional coordinates and thermal parameters with estimated standard deviations are given in Tables 1 and 2. A comparison between observed and calculated structure factors is presented in Table 3. The principal axes

* All programs used are included in this reference.

Table 1. Fractional atomic coordinates and anisotropic thermal parameters ($\exp[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)]$) for Hg and Cl atoms, with estimated standard deviations.

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Hg_1	.0357	.1413	.1778	.0130	.0067	.0019	.0014	−.0002	−.0002
	3	1	1	9	2	1	5	4	2
Hg_2	.5347	.1289	.1747	.0169	.0064	.0024	−.0001	.0000	.0013
	3	1	1	9	2	1	5	5	2
Cl_1	.224	.196	.2486	.018	.007	.0023	.000	.002	−.0009
	2	1	6	5	1	4	3	2	9
Cl_2	−.152	.094	.1052	.015	.011	.0021	.003	−.001	−.002
	2	1	6	4	1	4	3	2	1
Cl_3	.717	.064	.2431	.021	.010	.0027	.011	.003	.005
	2	1	6	5	1	5	4	2	1
Cl_4	.345	.188	.1050	.017	.009	.0023	.009	−.002	.001
	2	1	6	4	1	4	4	2	1

of the thermal vibration ellipsoids for Hg and Cl atoms were calculated from the thermal paramaters of Table 1. Root mean square amplitudes, the corresponding B-values for the atomic anisotropic thermal vibration along the principal axes, as well as their components along the crystal axes are given in Table 4.

Table 2. Fractional atomic coordinates and isotropic temperature factors for oxygen and carbon atoms, with estimated standard deviations.

Atom	x	y	z	B
O_1	.765 (5)	.292 (2)	.188 (1)	4.5 (0.8)
O_2	.735 (5)	.471 (2)	.318 (2)	5.8 (0.8)
C_1	.757 (6)	.383 (3)	.186 (2)	1.9 (0.9)
C_2	.596 (7)	.433 (3)	.180 (2)	4.0 (1.2)
C_3	.572 (7)	.518 (3)	.133 (2)	5.0 (1.3)
C_4	.523 (7)	.470 (3)	.079 (2)	4.0 (1.2)
C_5	.667 (9)	.398 (4)	.056 (2)	6.4 (1.5)
C_6	.846 (9)	.447 (4)	.047 (3)	7.5 (1.7)
C_7	.972 (9)	.487 (4)	.088 (3)	7.5 (1.7)
C_8	1.026 (8)	.424 (4)	.137 (3)	6.9 (1.5)
C_9	.940 (7)	.436 (3)	.189 (2)	3.8 (1.1)
C_{10}	.743 (9)	.380 (4)	.332 (2)	6.1 (1.5)
C_{11}	.585 (8)	.317 (4)	.326 (2)	5.8 (1.4)
C_{12}	.458 (8)	.342 (3)	.370 (2)	4.8 (1.3)
C_{13}	.525 (9)	.296 (4)	.428 (3)	8.1 (1.8)
C_{14}	.623 (9)	.363 (4)	.469 (3)	7.4 (1.6)
C_{15}	.812 (9)	.405 (4)	.451 (2)	6.9 (1.5)
C_{16}	.938 (9)	.334 (4)	.451 (2)	7.4 (1.6)
C_{17}	.949 (9)	.273 (4)	.393 (3)	8.5 (1.8)
C_{18}	.931 (8)	.333 (4)	.338 (2)	5.5 (1.3)

Table 3. Observed and calculated structure factors on ten times absolute scale.

(a)

h	k	l	F_o	F_c
0	0	2	4886	-4480
0	0	4	2243	-2667
0	0	6	3822	4027
0	0	8	3794	-4089
0	0	12	3304	3342
0	0	14	4160	-4335
0	0	16	1562	1585
0	0	18	1188	840
0	0	20	1507	-1334
0	0	22	1056	947
0	1	18	543	-1280
0	1	17	923	1637
0	1	16	1484	1879
0	1	15	1561	-1781
0	1	13	346	-601
0	1	12	2277	-2398
0	1	11	2170	2438
0	1	10	2758	3046
0	1	9	1831	-2125
0	1	8	821	-1011
0	1	7	475	569
0	1	6	1084	-1163
0	1	5	2345	2524
0	1	4	4166	4303
0	1	3	3684	-3891
0	1	2	3852	-3816
0	2	1	4814	-4701
0	2	2	463	546
0	2	3	1840	1800
0	2	5	2901	3060
0	2	7	2068	-2250
0	2	9	1624	1755
0	2	11	1153	1265
0	2	13	3479	-3645
0	2	15	2163	2119
0	2	19	1298	-1169
0	2	21	1030	873
0	3	22	427	342
0	3	21	345	342
0	3	19	692	659
0	3	18	959	-944
0	3	17	1639	-1645
0	3	16	1258	1308
0	3	15	1398	1509
0	3	14	397	-459
0	3	13	632	716
0	3	12	1096	-1285
0	3	11	2590	-2879
0	3	10	2120	2296
0	3	9	2483	2786
0	3	8	777	-829
0	3	7	496	-414
0	3	6	748	-847
0	3	5	1931	-2063
0	3	4	2425	2519
0	3	3	4651	4683
0	3	2	3431	-3405
0	3	1	3814	-3709
0	4	0	5057	-5352
0	4	1	1254	1252
0	4	2	2508	2666
0	4	4	1208	1408
0	4	5	484	-485
0	4	6	3959	-4321
0	4	7	1075	1097
0	4	8	3002	3323
0	4	9	859	-760
0	4	12	1998	-2155
0	4	13	727	830
0	4	14	2619	2646
0	4	15	609	-667
0	4	16	752	-787
0	4	18	1015	-930
0	4	20	1396	1220
0	5	18	1316	1132
0	5	17	608	-563
0	5	16	1435	-1451
0	5	15	716	656
0	5	14	323	315
0	5	12	1925	1941
0	5	11	1147	-1174
0	5	10	2866	-2907
0	5	9	1185	1282
0	5	8	1431	1502
0	5	7	298	-260
0	5	6	897	966
0	5	5	936	-1196
0	5	4	3233	-3532
0	5	3	1649	1713
0	5	2	2679	2899
0	5	1	771	-793
0	6	0	1510	1438
0	6	1	2855	3000
0	6	2	767	-841
0	6	3	390	-274
0	6	4	424	-413
0	6	5	2642	-2729
0	6	6	1227	1291

h	k	l	F_o	F_c
0	6	7	3513	3723
0	6	8	1029	-1126
0	6	9	2174	-2241
0	6	11	722	-803
0	6	12	746	701
0	6	13	1900	1962
0	6	14	712	-690
0	6	15	1234	-1158
0	6	16	335	293
0	6	19	1135	967
0	6	20	419	-526
0	7	19	828	-808
0	7	17	1385	1283
0	7	16	475	-429
0	7	15	782	-781
0	7	13	676	-653
0	7	12	454	380
0	7	11	2099	2119
0	7	10	924	-925
0	7	9	2443	-2540
0	7	8	776	871
0	7	7	265	333
0	7	5	2640	2751
0	7	4	1247	-1296
0	7	3	3103	-3290
0	7	2	1050	1067
0	7	1	1304	1397
0	8	0	1827	1840
0	8	1	1012	-1072
0	8	2	1065	-1056
0	8	3	432	377
0	8	4	876	-875
0	8	5	831	904
0	8	6	2460	2556
0	8	7	1486	-1508
0	8	8	2042	-2081
0	8	9	826	760
0	8	11	275	210
0	8	12	1029	1059
0	8	13	603	-579
0	8	14	1133	-1009
0	8	15	594	611
0	8	16	331	257
0	9	14	356	-323
0	9	12	930	-789
0	9	11	436	318
0	9	10	1881	1924
0	9	9	529	-417
0	9	8	1023	-1078
0	9	7	315	180
0	9	6	1025	-1012
0	9	5	321	240
0	9	4	2107	2130
0	9	3	521	-450
0	9	2	1463	-1503
0	9	1	351	217
0	10	0	891	-877
0	10	1	1215	-1298
0	10	2	719	657
0	10	3	272	264
0	10	5	1159	1268
0	10	6	1229	-1185
0	10	7	1588	-1644
0	10	8	1023	999
0	10	9	597	565
0	10	11	429	471
0	10	12	439	-451
0	10	13	864	-724
0	11	9	1109	1118
0	11	5	1274	-1313
0	11	4	359	71
0	11	3	1492	1513
0	11	1	636	-579
0	12	0	1045	-1077
0	12	1	750	722
0	12	2	667	685
0	12	3	366	-289
1	12	1	375	393
1	12	0	405	542
1	12	-1	286	-342
1	11	-8	808	-797
1	11	-6	307	262
1	11	-4	467	417
1	11	-2	575	-581
1	11	0	278	86
1	11	2	607	605
1	11	4	852	-837
1	11	6	459	496
1	10	10	315	-263
1	10	8	543	628
1	10	7	453	447
1	10	6	633	-515
1	10	5	399	-465
1	10	3	274	-209
1	10	2	424	456
1	10	1	404	521
1	10	0	607	-586
1	10	-1	321	-327

h	k	l	F_o	F_c
1	10	-2	290	213
1	10	-4	376	375
1	10	-5	434	477
1	10	-6	622	-665
1	10	-7	487	-307
1	10	-8	331	428
1	10	-10	355	205
1	10	-11	347	325
1	10	-12	420	-495
1	9	-15	334	294
1	9	-14	304	-175
1	9	-11	573	-490
1	9	-9	805	922
1	9	-8	292	212
1	9	-7	295	-320
1	9	-5	307	-347
1	9	-3	496	525
1	9	0	284	108
1	9	1	326	-304
1	9	3	935	973
1	9	4	260	245
1	9	5	761	-786
1	9	9	549	568
1	9	11	314	-448
1	9	15	504	400
1	8	15	449	396
1	8	13	462	-379
1	8	9	294	268
1	8	7	790	-747
1	8	6	713	-763
1	8	5	755	785
1	8	2	436	450
1	8	1	729	-748
1	8	0	369	-339
1	8	-1	545	544
1	8	-5	252	-300
1	8	-6	364	-384
1	8	-7	635	615
1	8	-8	358	262
1	8	-9	289	-232
1	8	-11	382	-331
1	8	-12	434	-359
1	8	-13	550	442
1	7	-17	397	248
1	7	-16	357	-298
1	7	-14	532	463
1	7	-11	299	208
1	7	-10	635	-652
1	7	-8	616	529
1	7	-6	428	436
1	7	-5	290	258
1	7	-4	582	-643
1	7	-3	331	-307
1	7	-2	578	669
1	7	0	676	-693
1	7	2	464	-567
1	7	3	330	-288
1	7	4	1136	1181
1	7	5	354	333
1	7	8	863	-903
1	7	9	321	-277
1	7	10	586	574
1	7	12	311	-245
1	7	13	351	-328
1	7	16	385	327
1	7	18	406	-352
1	6	16	507	501
1	6	14	488	-450
1	6	11	390	427
1	6	8	666	-709
1	6	7	520	-412
1	6	6	728	784
1	6	5	381	455
1	6	4	396	320
1	6	3	326	255
1	6	2	1136	-1173
1	6	1	374	-418
1	6	0	398	446
1	6	-6	692	752
1	6	-7	258	333
1	6	-8	870	-945
1	6	-10	365	-461
1	6	-11	604	-746
1	6	-12	990	1018
1	6	-15	480	391
1	5	-18	399	-432
1	5	-16	470	295
1	5	-15	521	-518
1	5	-13	979	921
1	5	-12	327	-248
1	5	-9	618	-633
1	5	-7	411	-336
1	5	-6	600	-622
1	5	-5	727	813
1	5	-4	557	602
1	5	-3	729	-834
1	5	-2	572	-590
1	5	-1	1168	1160

h	k	l	F_o	F_c
1	5	0	773	715
1	5	1	1085	-1110
1	5	2	288	-316
1	5	3	381	-441
1	5	5	578	673
1	5	7	1270	1330
1	5	9	764	-804
1	5	12	483	572
1	5	13	402	-393
1	5	14	442	-416
1	5	18	360	333
1	4	19	366	188
1	4	17	348	436
1	4	15	564	-564
1	4	9	621	584
1	4	8	277	296
1	4	7	685	697
1	4	6	398	511
1	4	5	654	-756
1	4	3	1272	-1212
1	4	2	283	-259
1	4	1	1258	1323
1	4	-2	261	243
1	4	-7	911	-860
1	4	-8	255	-104
1	4	-9	413	400
1	4	-10	414	-405
1	4	-11	711	669
1	4	-12	447	458
1	4	-13	438	-401
1	4	-16	321	-430
1	4	-20	336	116
1	4	-21	374	457
1	3	-21	419	-289
1	3	-19	392	274
1	3	-17	399	-358
1	3	-16	402	260
1	3	-15	382	460
1	3	-14	1066	-1112
1	3	-12	685	819
1	3	-11	367	-254
1	3	-10	365	448
1	3	-9	376	336
1	3	-7	682	710
1	3	-6	1205	-1221
1	3	-5	1082	-1144
1	3	-4	873	750
1	3	-3	419	459
1	3	-2	322	-304
1	3	-1	961	-961
1	3	0	2158	2078
1	3	1	1242	1187
1	3	2	653	-569
1	3	3	336	-293
1	3	6	221	-132
1	3	8	747	803
1	3	9	532	529
1	3	10	922	-1040
1	3	11	489	-557
1	3	13	632	763
1	3	14	456	-292
1	3	15	366	-334
1	3	18	359	163
1	2	21	371	155
1	2	17	325	-450
1	2	16	470	-496
1	2	14	355	343
1	2	11	627	-577
1	2	10	568	548
1	2	9	396	554
1	2	7	346	322
1	2	6	668	-758
1	2	5	346	-393
1	2	2	1290	1210
1	2	-4	707	-577
1	2	-5	243	127
1	2	-7	293	247
1	2	-8	544	558
1	2	-9	437	-498
1	2	-12	393	-323
1	2	-13	312	435
1	2	-15	703	-622
1	2	-17	366	383
1	2	-21	409	-411
1	1	-20	415	-435
1	1	-18	465	460
1	1	-14	316	-312
1	1	-12	313	195
1	1	-8	970	-952
1	1	-7	1195	1024
1	1	-6	1153	1016
1	1	-5	393	-334
1	1	-4	581	-528
1	1	-3	748	698
1	1	-2	371	366
1	1	-1	525	560
1	1	0	770	825
1	1	1	612	-649

Table 3. Continued.

h	k	l	F_o	F_c
1	1	4	307	366
1	1	6	563	530
1	1	7	1548	-1645
1	1	8	860	- 917
1	1	9	527	534
1	1	12	402	- 306
1	1	15	409	388
1	1	21	479	308
1	0	22	404	440
1	0	16	404	441
1	0	14	450	- 576
1	0	12	503	- 390
1	0	10	486	680
1	0	8	566	- 589
1	0	-6	290	- 298
1	0	-10	279	343
1	0	-12	287	- 145
2	0	-22	490	388
2	0	-20	2016	-1886
2	0	-18	1895	1850
2	0	-14	1495	-1609
2	0	-12	1907	1881
2	0	-10	1183	-1121
2	0	-8	2070	-2125
2	0	-6	5590	5633
2	0	-4	5734	-5053
2	0	-2	376	- 393
2	0	0	4912	4304
2	0	2	3178	-3000
2	0	6	3521	3450
2	0	8	5176	-5178
2	0	10	2192	2119
2	0	12	1673	1732
2	0	14	1798	-1758
2	0	16	1247	1193
2	0	20	1615	-1393
2	1	18	872	-1134
2	1	17	707	988
2	1	16	771	774
2	1	13	799	-1160
2	1	12	2299	-2240
2	1	11	2491	2517
2	1	10	3088	2934
2	1	9	1885	-1927
2	1	8	410	- 474
2	1	7	1067	-1082
2	1	6	2731	-2855
2	1	5	2740	2863
2	1	4	3034	2944
2	1	3	1554	-1632
2	1	2	655	- 660
2	1	1	264	- 233
2	1	0	978	-1258
2	1	-1	1811	2031
2	1	-2	3511	3581
2	1	-3	3828	-3527
2	1	-4	4416	-3977
2	1	-5	1998	1912
2	1	-6	669	- 712
2	1	-7	2084	2038
2	1	-8	3309	3281
2	1	-9	2671	-2634
2	1	-10	2184	-2093
2	1	-11	1121	1017
2	1	-14	1153	1131
2	1	-15	1426	-1325
2	1	-16	1899	-1789
2	1	-17	1153	1321
2	1	-18	763	1055
2	1	-21	634	- 692
2	1	-22	741	- 938
2	2	-21	1221	- 980
2	2	-19	2179	1970
2	2	-17	1005	- 916
2	2	-15	1252	-1149
2	2	-14	350	381
2	2	-13	2552	2393
2	2	-11	2177	-2150
2	2	-10	282	313
2	2	-9	437	- 495
2	2	-7	4702	4648
2	2	-6	850	- 860
2	2	-5	5776	-5834
2	2	-4	575	269
2	2	-3	902	1060
2	2	-1	2596	3063

h	k	l	F_o	F_c
2	2	0	468	- 463
2	2	1	3066	-3657
2	2	2	329	268
2	2	3	2397	2554
2	2	5	905	953
2	2	7	4877	-5164
2	2	8	717	709
2	2	9	4109	4039
2	2	11	312	- 184
2	2	13	2132	-2089
2	2	15	2136	2024
2	2	17	666	- 669
2	2	19	814	- 553
2	2	21	1701	1401
2	3	19	1484	1419
2	3	18	911	- 908
2	3	17	1394	-1264
2	3	16	707	642
2	3	13	1264	1239
2	3	12	1608	-1685
2	3	11	3007	-3088
2	3	10	2158	2107
2	3	9	2079	2101
2	3	8	388	- 346
2	3	7	1629	1689
2	3	6	1787	-1933
2	3	5	3605	-3941
2	3	4	2500	2534
2	3	3	3047	3281
2	3	2	1508	-1610
2	3	1	746	- 729
2	3	0	976	-1110
2	3	-1	3197	-3761
2	3	-2	2954	3365
2	3	-3	4397	4874
2	3	-4	2148	-2390
2	3	-5	1376	-1493
2	3	-7	1638	-1610
2	3	-8	2044	1964
2	3	-9	3130	3074
2	3	-10	1990	-1970
2	3	-11	1742	-1720
2	3	-12	542	490
2	3	-13	321	- 90
2	3	-14	760	786
2	3	-15	1623	1583
2	3	-16	1270	-1157
2	3	-17	1872	-1798
2	3	-18	651	623
2	3	-19	616	524
2	3	-21	716	679
2	3	-22	724	- 588
2	4	-20	1327	1189
2	4	-18	1416	-1327
2	4	-15	420	304
2	4	-14	2058	1984
2	4	-13	648	- 672
2	4	-12	2350	-2264
2	4	-11	442	453
2	4	-10	1367	1480
2	4	-8	1627	1533
2	4	-7	898	- 865
2	4	-6	4165	-4399
2	4	-5	1188	1277
2	4	-4	2184	2648
2	4	-2	1150	1321
2	4	-1	629	- 736
2	4	0	3241	-3764
2	4	2	3416	3932
2	4	3	920	- 906
2	4	4	933	- 948
2	4	6	2739	-2766
2	4	7	922	874
2	4	8	3591	3538
2	4	9	1101	-1055
2	4	10	1573	-1544
2	4	11	324	337
2	4	12	1162	-1151
2	4	14	2162	2021
2	4	16	1526	-1457
2	4	20	1194	1054
2	5	19	453	496
2	5	18	1488	1314
2	5	17	701	- 668
2	5	16	1134	-1026

h	k	l	F_o	F_c
2	5	15	384	301
2	5	14	496	- 428
2	5	13	563	463
2	5	12	1920	1788
2	5	11	1192	-1159
2	5	10	2007	-1931
2	5	9	724	722
2	5	8	284	183
2	5	7	474	475
2	5	6	2451	2413
2	5	5	1586	-1610
2	5	4	3231	-3249
2	5	3	1341	1385
2	5	2	1424	1570
2	5	0	1287	1561
2	5	-1	1159	-1201
2	5	-2	2794	-2980
2	5	-3	1584	1601
2	5	-4	2464	2585
2	5	-5	400	- 363
2	5	-7	904	- 854
2	5	-8	2389	-2437
2	5	-9	1310	1337
2	5	-10	2333	2157
2	5	-11	825	- 816
2	5	-12	719	- 706
2	5	-13	418	- 263
2	5	-14	1249	-1252
2	5	-15	898	843
2	5	-16	1878	1763
2	5	-17	752	- 599
2	5	-18	986	- 810
2	6	-19	1161	- 985
2	6	-18	470	386
2	6	-17	546	487
2	6	-15	905	778
2	6	-14	786	- 701
2	6	-13	2313	-2307
2	6	-12	875	806
2	6	-11	2155	2107
2	6	-10	420	- 362
2	6	-8	643	- 674
2	6	-7	1802	-1901
2	6	-6	953	893
2	6	-5	2249	2367
2	6	-4	831	- 823
2	6	-3	561	- 661
2	6	-1	2389	-2580
2	6	0	1430	1430
2	6	1	3632	3854
2	6	2	1171	-1157
2	6	3	1970	-2023
2	6	5	597	- 609
2	6	6	723	717
2	6	7	2294	2399
2	6	8	1107	-1122
2	6	9	1776	-1862
2	6	10	488	366
2	6	13	1705	1684
2	6	14	699	- 649
2	6	15	1819	-1753
2	6	16	680	598
2	6	17	767	583
2	7	17	1415	1318
2	7	16	436	- 478
2	7	15	407	- 274
2	7	13	1242	-1220
2	7	11	1830	1739
2	7	10	620	- 538
2	7	9	1029	- 990
2	7	8	319	270
2	7	7	789	- 735
2	7	6	650	615
2	7	5	2779	2881
2	7	4	1178	-1194
2	7	3	2506	-2585
2	7	2	707	794
2	7	0	491	479
2	7	-1	2132	2355
2	7	-2	932	- 945
2	7	-3	2458	-2648
2	7	-4	630	564
2	7	-5	853	739
2	7	-6	306	257
2	7	-7	1220	1262

h	k	l	F_o	F_c
2	7	-8	861	- 891
2	7	-9	2344	-2341
2	7	-10	603	453
2	7	-11	1480	1445
2	7	-14	391	- 364
2	7	-15	1548	-1411
2	7	-16	594	537
2	7	-17	1250	1139
2	8	-15	492	- 413
2	8	-14	1388	-1265
2	8	-13	1105	1048
2	8	-12	1806	1697
2	8	-11	612	- 605
2	8	-10	768	- 781
2	8	-8	718	- 704
2	8	-7	863	793
2	8	-6	1497	1508
2	8	-5	893	- 941
2	8	-4	1039	-1119
2	8	-3	325	260
2	8	-2	475	- 522
2	8	-1	741	624
2	8	0	2296	2420
2	8	1	1533	-1543
2	8	2	2369	-2438
2	8	3	921	963
2	8	4	483	480
2	8	5	384	364
2	8	6	1120	1139
2	8	7	842	- 773
2	8	8	1375	-1366
2	8	9	751	657
2	8	10	606	636
2	8	12	648	633
2	8	13	595	- 482
2	8	14	1508	-1329
2	8	15	985	847
2	9	12	1486	-1398
2	9	11	461	390
2	9	10	1323	1287
2	9	8	[illegible]	- 76
2	9	6	1388	-1387
2	9	5	634	574
2	9	4	2265	2230
2	9	3	528	- 504
2	9	2	1262	-1306
2	9	0	1035	-1087
2	9	-1	541	300
2	9	-2	2063	2106
2	9	-3	612	- 590
2	9	-4	1358	-1382
2	9	-8	1411	1386
2	9	-9	580	- 410
2	9	-10	2049	-1931
2	9	-12	745	687
2	9	-14	916	874
2	10	-12	950	- 852
2	10	-11	927	- 838
2	10	-8	757	713
2	10	-7	1216	1107
2	10	-6	972	- 803
2	10	-5	1354	-1314
2	10	-4	731	654
2	10	-3	406	358
2	10	-2	381	174
2	10	-1	1206	1153
2	10	0	1293	-1273
2	10	1	1953	-1976
2	10	2	1334	1295
2	10	3	1089	1062
2	10	4	340	- 260
2	10	5	699	623
2	10	6	813	- 755
2	10	7	1355	-1315
2	10	8	844	788
2	10	9	958	835
2	10	10	494	- 506
2	11	5	1427	-1310
2	11	4	390	- 7
2	11	3	1437	1320
2	11	-1	1412	-1345
2	11	-2	389	125
2	11	-3	1407	1294
2	11	-5	497	- 388
2	11	-7	647	- 587

(b)

h	k	l	F_o	F_c
3	0	-6	807	652
3	0	12	549	- 309
3	1	8	474	564
3	1	6	338	- 313
3	1	3	322	329
3	1	1	329	- 370
3	1	-2	381	363
3	1	-4	284	292
3	1	-5	726	783
3	1	-6	384	- 330
3	1	-7	327	- 324
3	1	-10	387	- 395
3	2	-21	359	- 320
3	2	-18	466	- 420
3	2	-15	384	- 301
3	2	-14	283	362
3	2	-12	273	- 407
3	2	-6	383	- 404
3	2	-4	1066	1138
3	2	-2	1377	-1366
3	2	0	383	- 445
3	2	1	204	270
3	2	2	502	471
3	2	5	364	- 347
3	2	10	839	- 816
3	2	11	324	- 251
3	2	12	404	475
3	2	17	449	- 448
3	3	19	349	264
3	3	17	343	- 303
3	3	16	599	- 505
3	3	9	370	- 404
3	3	8	411	- 424
3	3	7	580	629
3	3	5	329	- 414
3	3	4	456	- 436
3	3	3	541	579
3	3	2	800	922
3	3	1	424	- 451
3	3	-4	438	- 435
3	3	-5	291	139
3	3	-6	918	874
3	3	-7	579	- 657
3	3	-8	511	- 459

Table 3. Continued.

h	k	l	F_o	F_c
3	3	-9	289	358
3	3	-10	518	534
3	3	-11	636	-631
3	3	-12	418	-390
3	3	-13	350	331
3	3	-18	382	405
3	4	-20	385	348
3	4	-13	332	-347
3	4	-10	282	136
3	4	-9	555	651
3	4	-5	493	600
3	4	-4	332	-304
3	4	-3	605	-618
3	4	-1	552	-572
3	4	1	428	449
3	4	3	605	549
3	4	4	499	-492
3	4	5	1000	-1057
3	4	7	390	423
3	4	9	513	-534
3	4	18	429	434
3	5	17	597	559
3	5	15	535	-528
3	5	12	373	355
3	5	11	350	345
3	5	8	504	-543
3	5	7	361	-278

h	k	l	F_o	F_c
3	5	6	375	367
3	5	5	436	478
3	5	4	333	-354
3	5	3	1307	-1265
3	5	2	358	304
3	5	1	616	605
3	5	-1	932	917
3	5	-2	308	-375
3	5	-5	883	-879
3	5	-6	269	328
3	5	-7	523	533
3	5	-8	321	-275
3	5	-9	382	-344
3	5	-10	615	626
3	5	-11	313	327
3	5	-12	363	-371
3	5	-19	542	530
3	6	-18	660	454
3	6	-17	388	-380
3	6	-16	567	-462
3	6	-15	359	375
3	6	-14	412	-395
3	6	-12	486	505
3	6	-11	321	-244
3	6	-6	561	667
3	6	-4	926	-946
3	6	-3	566	611

h	k	l	F_o	F_c
3	6	-2	475	472
3	6	-1	256	188
3	6	0	703	749
3	6	1	421	-456
3	6	2	412	-485
3	6	8	616	-695
3	6	10	765	827
3	6	14	592	-580
3	6	16	380	243
3	7	12	498	-473
3	7	8	279	93
3	7	4	722	784
3	7	3	313	-169
3	7	2	634	-620
3	7	0	345	-331
3	7	-1	359	266
3	7	-2	853	834
3	7	-5	332	221
3	7	-6	491	-575
3	7	-8	341	187
3	7	-10	342	-376
3	7	-13	364	-137
3	7	-14	388	470
3	7	-16	378	-325
3	8	-13	472	466
3	8	-11	526	-482

h	k	l	F_o	F_c
3	8	-7	312	279
3	8	-5	606	-670
3	8	-4	469	457
3	8	-3	604	588
3	8	-1	300	358
3	8	0	378	-355
3	8	1	806	-821
3	8	2	463	448
3	8	3	445	494
3	8	9	406	416
3	8	12	358	-332
3	9	11	563	-590
3	9	7	497	513
3	9	5	570	-518
3	9	0	347	-237
3	9	-1	1037	-1115
3	9	-3	647	672
3	9	-7	559	-513
3	9	-9	569	607
3	9	-13	540	-544
3	10	-6	403	-387
3	10	0	550	-508
3	10	1	417	450
3	10	2	636	580
3	10	8	485	420
3	11	-2	605	-619

(c)

4	0	-18	890	953
4	0	-16	536	-510
4	0	-14	999	-1027
4	0	-12	2989	3184
4	0	-10	2391	-2470
4	0	-8	624	-530
4	0	-6	2998	2546
4	0	-4	3595	-2875
4	0	-2	1469	806
4	0	0	3052	2899
4	0	2	5525	-5348
4	0	4	2605	2606
4	0	6	1558	1476
4	0	8	2882	-2944
4	0	10	1637	1845
4	0	14	1444	-1502
4	0	16	2033	2167
4	0	18	893	-816
4	1	18	1433	-1229
4	1	14	1001	1153
4	1	13	1251	-1287
4	1	12	1303	-1629
4	1	8	658	641
4	1	7	2048	-1509
4	1	6	3254	-2803
4	1	5	3490	3009
4	1	4	3276	3037
4	1	3	1687	-1574
4	1	2	588	-142
4	1	1	1178	-1249
4	1	0	2111	-2226
4	1	-1	2123	2243
4	1	-2	2234	2303
4	1	-3	1578	-1442
4	1	-4	1092	-1100
4	1	-6	1155	-1062
4	1	-7	2130	1887
4	1	-8	2908	2811
4	1	-9	2529	-2523
4	1	-10	1804	-2001
4	1	-13	1165	1115
4	1	-14	1301	1397
4	1	-15	639	-1185
4	2	-19	934	1023
4	2	-17	1024	-1042
4	2	-13	1593	1918
4	2	-12	733	-291
4	2	-11	2461	-3032
4	2	-10	1259	287
4	2	-9	1101	1298
4	2	-8	606	161
4	2	-7	1859	1959
4	2	-6	875	-393
4	2	-5	2629	-2667
4	2	-4	730	227
4	2	-3	1632	1738
4	2	-2	454	-124
4	2	-1	566	460
4	2	0	494	-214
4	2	1	3122	-3418
4	2	2	988	619
4	2	3	3590	3711
4	2	4	1085	-397
4	2	5	670	-624
4	2	7	2194	-2231
4	2	8	853	249
4	2	9	2153	2207
4	2	10	729	-232
4	2	11	956	-1019
4	2	13	740	-703

4	2	15	1688	1891
4	2	17	1115	-1283
4	3	17	647	-805
4	3	15	411	-514
4	3	14	573	639
4	3	13	1247	1377
4	3	12	1057	-1057
4	3	11	1294	-1463
4	3	10	676	693
4	3	9	363	395
4	3	8	689	449
4	3	7	1564	1677
4	3	6	1738	-1575
4	3	5	2528	-2668
4	3	4	1626	1687
4	3	3	1432	1510
4	3	2	363	-157
4	3	1	1371	1547
4	3	0	1555	-1628
4	3	-1	2562	-2850
4	3	-2	1521	1557
4	3	-3	1461	1468
4	3	-4	592	-577
4	3	-5	429	452
4	3	-6	1069	-858
4	3	-7	1896	-2033
4	3	-8	1627	1613
4	3	-9	1992	2281
4	3	-10	849	-985
4	3	-11	570	-560
4	3	-12	517	-321
4	3	-13	939	-1196
4	3	-14	901	1082
4	3	-15	972	1311
4	4	-18	812	-1175
4	4	-17	408	260
4	4	-16	374	615
4	4	-14	464	614
4	4	-13	364	-447
4	4	-12	1720	-2028
4	4	-11	876	683
4	4	-10	1573	1886
4	4	-9	547	-240
4	4	-8	381	382
4	4	-7	509	-455
4	4	-6	2382	-2669
4	4	-5	1034	855
4	4	-4	2470	2889
4	4	-3	819	-509
4	4	-2	673	-719
4	4	-1	471	-207
4	4	0	1583	-1811
4	4	1	809	671
4	4	2	2779	3406
4	4	3	1045	-815
4	4	4	1471	-1813
4	4	5	386	144
4	4	6	1012	-1222
4	4	7	562	376
4	4	8	2004	2382
4	4	9	782	-542
4	4	10	1400	-1770
4	4	11	562	368
4	4	14	1086	1389
4	4	16	1275	-1562
4	5	14	959	-1167
4	5	13	738	674
4	5	12	1551	1930
4	5	11	804	-963
4	5	10	898	-1064

4	5	8	508	-534
4	5	7	860	713
4	5	6	1885	2310
4	5	5	1187	-1264
4	5	4	1736	-2102
4	5	3	583	576
4	5	2	221	-145
4	5	1	713	585
4	5	0	1847	2148
4	5	-1	1389	-1431
4	5	-2	2348	-2767
4	5	-3	1118	1162
4	5	-4	1141	1231
4	5	-5	350	-51
4	5	-6	863	1062
4	5	-7	1091	-876
4	5	-8	2031	-2322
4	5	-9	1065	1041
4	5	-10	1351	1564
4	5	-13	573	-571
4	5	-14	950	-1303
4	5	-15	628	670
4	5	-16	720	1223
4	6	-16	350	-135
4	6	-13	803	-1006
4	6	-12	535	556
4	6	-11	1179	1435
4	6	-10	519	-437
4	6	-9	407	-484
4	6	-8	289	-122
4	6	-7	1252	-1487
4	6	-6	861	791
4	6	-5	2239	2555
4	6	-4	1043	-945
4	6	-3	1674	-1835
4	6	-2	582	422
4	6	-1	471	-417
4	6	0	694	630
4	6	1	1843	2144
4	6	2	998	-1002
4	6	3	1494	-1743
4	6	4	566	534
4	6	7	1262	1520
4	6	8	814	-891
4	6	9	1733	-2148
4	6	10	821	767
4	6	11	801	913
4	6	13	450	617
4	7	12	693	577
4	7	11	1251	1574
4	7	10	451	-547
4	7	8	353	65
4	7	7	1130	-1367
4	7	6	803	615
4	7	5	1472	1813
4	7	4	578	-631
4	7	3	790	-948
4	7	2	251	-87
4	7	1	857	-1066
4	7	0	861	761
4	7	-1	2014	2375
4	7	-2	1026	-914
4	7	-3	1793	-2013
4	7	-4	489	476
4	7	-5	253	11
4	7	-6	573	325
4	7	-7	1419	1748
4	7	-8	736	-594
4	7	-9	1266	-1471
4	7	-11	328	274

4	7	-12	286	104
4	7	-13	418	705
4	7	-14	383	-351
4	8	-12	781	1024
4	8	-11	528	-531
4	8	-10	580	-789
4	8	-9	271	139
4	8	-8	284	-304
4	8	-7	502	490
4	8	-6	1475	1696
4	8	-5	1070	-1123
4	8	-4	1757	-1989
4	8	-3	841	768
4	8	-2	468	448
4	8	-1	292	321
4	8	0	984	1100
4	8	1	785	-785
4	8	2	1359	-1570
4	8	3	802	796
4	8	4	684	801
4	8	6	528	659
4	8	7	628	-605
4	8	8	1346	-1619
4	8	9	876	883
4	8	10	1058	1279
4	9	7	307	-30
4	9	6	1045	-1345
4	9	5	379	206
4	9	4	740	901
4	9	1	424	-148
4	9	0	1024	-1196
4	9	-1	699	336
4	9	-2	1616	1886
4	9	-3	497	-276
4	9	-4	707	-752
4	9	-5	332	30
4	9	-6	599	-768
4	9	-7	484	250
4	9	-8	1030	1272
4	9	-9	377	-268
4	10	-2	331	-308
4	10	0	533	-491
5	1	-3	279	300
5	1	-4	284	-308
5	2	-2	304	326
5	2	-1	237	-143
5	2	0	245	-271
5	2	2	228	377
5	2	4	457	-513
5	2	7	327	168
5	3	11	342	-171
5	3	10	438	-401
5	3	7	369	427
5	3	6	324	358
5	3	4	263	305
5	3	3	335	-343
5	3	2	358	-404
5	3	1	249	333
5	3	-1	293	-329
5	3	-2	528	-505
5	3	-3	385	492
5	3	-4	395	282
5	3	-5	241	-246
5	3	-6	318	245
5	3	-7	265	-348
5	3	-10	324	-381
5	3	-12	419	552
5	3	-13	396	-167
5	4	-11	335	334
5	4	-9	608	-664

Table 3. Continued.

h	k	l	F_o	F_c
5	4	-8	410	362
5	4	-5	379	400
5	4	-2	305	- 210
5	4	-1	421	- 463
5	4	1	307	342
5	4	2	266	112
5	4	3	522	- 574
5	4	5	718	743
5	5	11	548	581
5	5	10	304	35
5	5	9	303	- 339
5	5	8	289	- 286
5	5	7	310	- 315
5	5	5	329	183
5	5	2	290	- 288
5	5	1	340	- 346
5	5	0	346	344
5	5	-1	335	385
5	5	-2	298	- 333
5	5	-3	375	- 562
5	5	-6	257	110
5	5	-7	485	528
5	6	-12	293	303
5	6	-11	320	- 81
5	6	-10	468	- 634
5	6	-9	299	365
5	6	-8	402	434
5	6	-5	325	- 167
5	6	-4	441	- 532
5	6	-3	243	103
5	6	-1	250	174
5	6	2	330	- 393
5	6	3	293	- 11
5	6	4	673	759
5	6	5	269	- 127
5	6	6	272	- 295
5	6	7	277	- 105
5	6	8	367	- 441
5	6	10	408	404
5	7	7	333	- 263
5	7	6	482	- 581
5	7	-2	413	509
5	7	-5	308	40
5	7	-6	475	- 477
5	7	-8	373	386
5	8	-5	353	- 479
5	8	-4	328	219
5	8	-3	317	349
5	8	-1	277	64
5	8	3	300	332
6	0	-12	1168	1161
6	0	-10	1448	-1374
6	0	-8	759	720
6	0	-6	967	756
6	0	-4	3023	-2558
6	0	-2	2690	2297
6	0	0	680	550
6	0	2	1807	-2021
6	0	4	1129	1547
6	0	8	999	-1185
6	0	10	1537	1870
6	1	9	772	462
6	1	8	1148	985
6	1	7	1307	-1156
6	1	6	1169	-1188
6	1	5	977	1001
6	1	4	745	612
6	1	3	493	- 173
6	1	2	792	679
6	1	1	1413	-1394
6	1	0	1839	-1978
6	1	-1	1874	1978
6	1	-2	1743	1798
6	1	-3	994	- 853
6	1	-4	670	161
6	1	-5	976	- 669
6	1	-6	1437	-1336
6	1	-7	1353	1237
6	1	-8	1189	1138
6	1	-9	815	- 646
6	1	-12	845	- 817
6	1	-13	1000	859
6	2	-12	589	- 247
6	2	-11	1389	-1591
6	2	-10	671	222
6	2	-9	962	987
6	2	-7	457	393
6	2	-6	578	- 199
6	2	-5	2097	-2192
6	2	-4	1050	391
6	2	-3	2215	2450
6	2	-2	794	- 251
6	2	-1	479	- 390
6	2	0	431	- 6
6	2	1	1576	-1778
6	2	2	721	339
6	2	3	2017	2246
6	2	4	854	- 252
6	2	5	859	- 951
6	2	7	682	- 632
6	2	9	1668	1923
6	3	9	621	- 576
6	3	8	876	679
6	3	7	1465	1608
6	3	6	1125	- 947
6	3	5	1287	-1321
6	3	4	604	546
6	3	2	629	390
6	3	1	1382	1554
6	3	0	1422	-1349
6	3	-1	2011	-2179
6	3	-2	1152	1179
6	3	-3	861	710
6	3	-4	554	236
6	3	-5	1013	940
6	3	-6	1041	- 984
6	3	-7	1444	-1447
6	3	-8	975	834
6	3	-9	886	890
6	3	-10	358	- 281
6	4	-10	1375	1430
6	4	-9	538	- 330
6	4	-8	576	- 640
6	4	-6	914	- 930
6	4	-5	657	383
6	4	-4	1832	1990
6	4	-3	874	- 514
6	4	-2	1264	-1429
6	4	-1	389	91
6	4	0	626	- 577
6	4	1	525	303
6	4	2	1766	1930
6	4	3	777	- 373
6	4	4	1459	-1582
6	4	5	553	294
6	4	6	315	275
6	5	5	857	- 768
6	5	4	922	- 936
6	5	3	321	250
6	5	2	638	- 678
6	5	1	740	553
6	5	0	1452	1621
6	5	-1	911	- 824
6	5	-2	1218	-1306
6	5	-3	450	295
6	5	-4	419	- 96
6	5	-5	563	390
6	5	-6	1253	1322
6	5	-7	1000	- 792
6	5	-8	1408	-1481
6	6	-5	1145	1171
6	6	-4	794	- 615
6	6	-3	1302	-1403
6	6	-2	556	422
6	6	-1	375	310
6	6	1	1019	1108
6	6	2	754	- 645
0	1	24	572	- 679
0	3	23	755	- 858
0	4	22	633	- 763
0	5	22	515	- 544
0	6	21	989	-1039
0	6	22	444	361
0	7	19	658	- 808
0	8	18	502	507
0	8	20	880	- 890
0	9	18	884	- 880
0	9	16	818	851
0	10	14	490	534
0	10	15	540	555
0	10	16	417	- 332
0	11	11	867	- 944
0	12	5	498	- 510
0	12	6	788	- 843
0	12	7	879	965
0	12	8	702	736
0	12	9	405	- 508
0	13	5	359	85
0	13	4	1074	-1162
0	13	2	805	880
1	13	-3	403	- 522
1	13	-2	289	97
1	13	1	303	366
1	13	2	299	- 15
1	13	3	618	- 609
1	12	7	351	410
1	12	6	329	251
1	12	-4	274	- 374
1	12	-5	450	469
1	12	-6	305	499
1	11	-11	338	- 219
1	11	-10	520	648
1	10	13	305	289
1	9	17	392	- 500
1	8	19	445	- 367
1	8	18	389	- 296
1	6	-21	411	77
1	5	21	454	- 464
1	3	-23	407	- 119
1	1	-25	379	- 159
2	0	-24	434	591
2	0	22	1120	1366
2	2	23	653	- 863
2	3	23	445	- 638
2	3	-23	591	- 751
2	4	22	815	- 941
2	5	20	622	- 656
2	6	-21	474	485
2	6	19	520	568
2	6	21	873	- 888
2	7	19	963	-1038
2	7	-19	382	- 147
2	8	16	840	931
2	8	17	416	- 446
2	9	16	667	737
2	9	-16	536	- 951
2	10	-14	443	668
2	10	-13	733	1080
2	10	13	711	- 794
2	10	14	622	789
2	11	12	321	68
2	11	11	894	-1012
2	11	10	315	194
2	11	9	524	501
2	11	-9	840	1259
2	11	-11	591	- 898
2	12	-7	515	- 593
2	12	-6	553	- 704
2	12	-5	371	527
2	12	-4	456	579
2	12	-1	406	- 374
2	12	0	606	- 673
2	12	1	751	872
2	12	2	494	622
2	12	3	371	- 450
2	12	5	290	- 175
2	12	6	490	- 537
2	12	7	516	472
2	12	8	628	642
3	12	3	332	- 482
3	12	2	369	- 361
3	12	1	363	451
3	11	-10	309	216
3	11	-4	378	523
3	11	4	313	- 505
3	10	-11	358	301
3	10	-14	381	162
3	9	-15	484	648
3	9	12	373	- 184
3	9	13	428	357
3	8	15	323	358
3	8	14	379	398
3	7	16	512	485
3	6	19	452	- 153
3	3	20	442	199
4	0	-20	713	- 837
4	1	20	633	858
4	1	18	711	-1229
4	3	19	594	1051
4	3	-21	562	839
4	4	-20	474	797
4	5	18	669	877
4	6	-19	469	- 915
4	6	-17	602	881
4	6	15	1025	-1185
4	6	17	712	774
4	7	13	1173	-1424
4	7	-15	735	-1183
4	7	-16	417	356
4	7	-17	506	762
4	8	-14	327	- 445
4	8	-13	445	565
4	8	11	663	- 662
4	8	14	640	- 850
4	9	12	1088	-1301
4	9	11	348	294
4	9	10	609	797
4	9	8	481	609
4	9	-10	562	- 717
4	9	-13	410	303
4	10	-9	400	447
4	10	-7	359	429
4	10	-6	599	- 684
4	10	-5	978	-1144
4	10	-4	820	876
4	10	-3	822	822
4	10	1	767	- 905
4	10	2	639	705
4	10	3	734	844
4	10	4	445	- 351
4	10	7	507	- 753
4	10	8	639	693
4	10	9	824	928
4	11	3	391	517
4	11	1	333	427
4	11	0	411	- 85
4	11	-1	1025	-1224
4	11	-2	486	129
4	11	-3	881	1085
4	11	-4	287	- 93
5	9	-9	368	277
5	9	-7	526	- 591
5	9	5	372	- 437
5	9	7	406	426
5	8	10	327	- 55
5	8	9	335	455
5	7	10	505	435
5	1	-19	404	- 14
6	0	-16	802	-1296
6	0	12	806	- 873
6	0	14	509	- 624
6	1	14	1109	987
6	1	13	1269	-1278
6	1	12	965	-1261
6	1	11	551	811
6	1	-14	1040	1275
6	1	-15	771	- 980
6	2	-16	706	138
6	2	-13	715	948
6	2	10	1215	- 336
6	2	11	1342	-1683
6	2	12	1228	237
6	3	13	1210	1285
6	3	12	963	- 800
6	3	11	771	- 843
6	3	-12	575	- 558
6	3	-13	860	-1052
6	3	-14	861	776
6	3	-15	517	1074
6	4	-12	1066	-1151
6	4	-11	682	411
6	4	8	1103	1147
6	4	9	685	- 382
6	4	10	1472	-1545
6	4	11	839	384
6	4	12	504	732
6	5	9	562	- 166
6	5	8	1105	-1114
6	5	7	914	745
6	5	6	1603	1652
6	5	-9	603	592
6	5	-10	569	586
6	5	-11	437	- 74
6	5	-12	564	644
6	5	-13	705	- 411
6	6	-11	1184	1338
6	6	-10	707	- 491
6	6	-9	992	-1038
6	6	-8	426	207
6	6	-6	430	359
6	6	-5	1100	1171
6	6	3	1525	-1618
6	6	4	814	719
6	6	5	870	843
6	6	6	318	- 208
6	6	7	465	450
6	6	8	356	- 421
6	6	9	1105	-1214
6	7	6	761	564
6	7	5	1195	1256
6	7	4	540	- 430
6	7	2	291	- 137
6	7	1	947	-1093
6	7	0	605	438
6	7	-1	1109	1195
6	7	-2	460	- 271
6	7	-3	443	- 354
6	7	-4	457	- 183
6	7	-5	712	- 735
6	7	-6	686	494
6	7	-7	1337	1430
6	7	-8	579	- 414
6	7	-9	891	- 933
6	8	-4	982	-1004
6	8	-3	617	489
6	8	-2	553	606
6	8	0	346	306
6	8	1	449	- 295
7	5	1	392	- 336
7	4	-1	338	446
7	3	-7	370	- 202
7	3	-4	323	- 72
7	3	0	343	107
7	3	1	278	204
7	2	5	334	- 186
7	2	0	370	325
7	2	-2	410	- 398

Table 4. The principal axes of the thermal vibration ellipsoids given by the components of a unit vector in fractional coordinates e_x, e_y, e_z, the corresponding r.m.s. amplitudes, and the B-values.

Atom	e_x	e_y	e_z	$(\bar{u}^2)^{\frac{1}{2}}$ (Å)	B (Å)
	.030	.046	.029	.252	5.00
Hg_1	−.003	.059	.025	.239	4.52
	.129	−.010	.012	.190	2.85
	−.020	.030	.036	.288	6.53
Hg_2	.056	.065	−.010	.229	4.13
	.119	−.026	.015	.217	3.72
	.011	−.040	.034	.276	6.00
Cl_1	.007	.064	.021	.244	4.71
	.132	.000	.000	.228	4.10
	.019	.067	−.018	.323	8.25
Cl_2	−.019	.036	.034	.245	4.75
	.130	−.004	.012	.200	3.16
	.043	.055	.026	.363	10.38
Cl_3	.085	.023	−.026	.255	5.13
	.092	−.047	.018	.198	3.08
	.069	.060	−.011	.299	7.04
Cl_4	−.008	.032	.036	.281	6.24
	.113	−.034	.014	.181	2.58

Bond distances and angles may be found in Figs. 1 and 2. Estimated standard deviations in bond distances and angles involving the light atoms only are about 0.07 Å and 5°, respectively. The asymmetric unit contains two independent cyclononanone molecules with enantiomeric conformations of the TCB-form. The approximate two-fold axes of symmetry are indicated in Fig. 1. In view of the large standard deviations, the considerable variations in bond distances and angles of the nine-membered rings are in fact insignificant and will not be discussed further. The C−C bond distances and the C−C−C bond angles average to 1.533 Å and 115.7°, the corresponding mean values for cyclononylammonium bromide [4] being 1.532 Å and 116.7°, respectively.

The carbonyl groups occupy positions corresponding to the shortest transannular H···H contacts in the TBC-form of cyclononane. In the present compound, such contacts range from 1.9 to 2.7 Å (1.8 to 2.4 Å in cyclononylammonium bromide).

Fig. 3 shows dihedral angles (averaged for the two isomers) of the present compound (A) and cyclononylammonium bromide (B), compared with those calculated by Hendrickson [2] (C), and Bixon and Lifson [1] (D) for the TCB-conformation. Apart from a somewhat larger eclipsing at the carbonyl group, the agreement is satisfactory. Standard deviations in dihedral angles are about 6°.

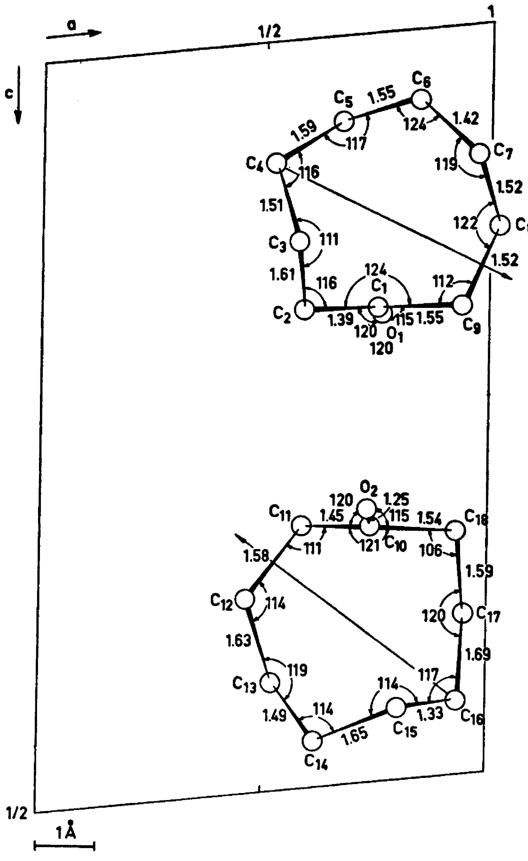


Fig. 1. Schematical drawing (viewed along [010]) showing bond distances and angles of the cyclononanone molecules.

When oxygen acts as a donor in charge-transfer complexes, only one of the lone pairs is usually engaged in the bond. Among the exceptions is the 1 : 1 complex between acetone and bromine,[7] where the Br· · ·O· · ·Br angle is 110°. From Fig. 2, showing the charge-transfer binding system, it is clear that the present structure constitutes another example, the $Hg_1 \cdots O_1 \cdots Hg_2$ and $Hg_1 \cdots O_2 \cdots Hg_2$ angles being 84.1° and 82.0°, respectively. Each mercury atom is complexed with two independent cyclononanone molecules, thus forming infinite chains in the [100]-direction. The mean value of the four charge-transfer bonds is 2.87 Å. The arrangement around the Hg-atoms is widely different from what has been observed for the complex $HgCl_2.2(C_6H_5)_3AsO$ [8] and the 1 : 1 addition compound between mercuric chloride and cyclohexane-1,4-dione,[9] but resembles that of $HgCl_2$.1,4-dioxane.[10]

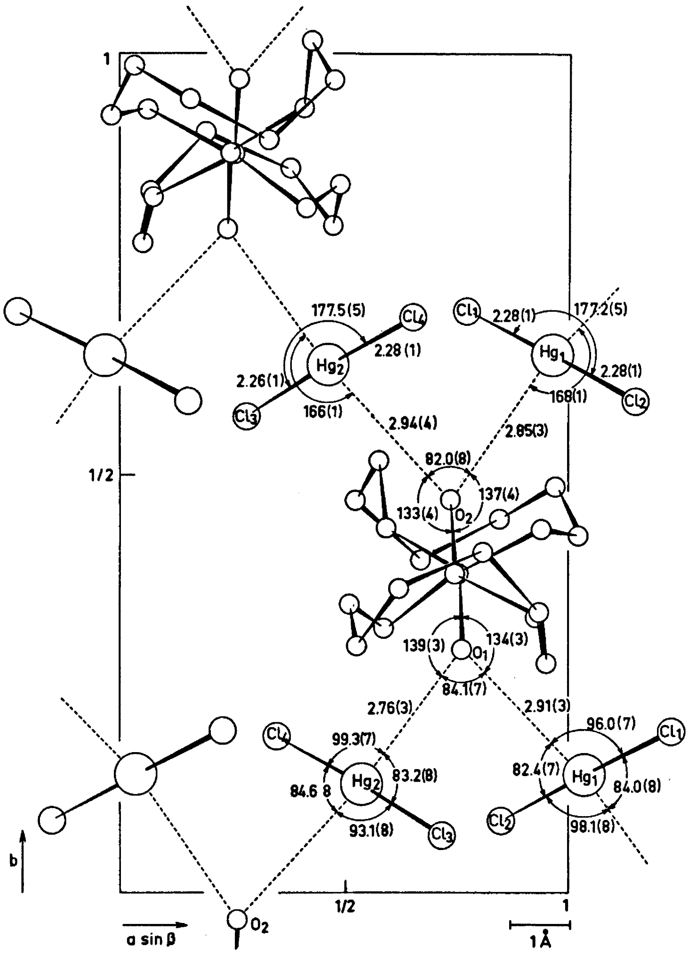


Fig. 2. Schematical drawing (viewed along [001]) showing the charge-transfer bonding system.

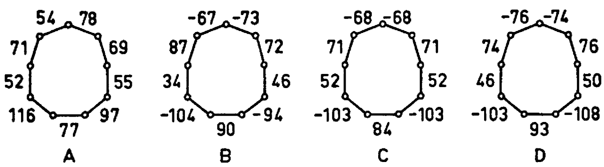


Fig. 3. Dihedral angles; of the present compound (A), cyclononylammonium bromide (B), and calculated [2] (C) and [1] (D).

REFERENCES

1. Bixon, M. and Lifson, S. *Tetrahedron* **23** (1967) 769.
2. Hendrickson, J. B. *J. Am. Chem. Soc.* **89** (1967) 7036.
3. Groth, P. *Acta Chem. Scand.* **23** (1969) 1311.
4. Bryan, R. F. and Dunitz, J. D. *Helv. Chim. Acta* **43** (1960) 3.
5. Dahl, T., Gram, F., Groth, P., Klewe, B. and Rømming, C. *Acta Chem. Scand.* **24** (1970) 2232.
6. Hanson, H. P., Herman, F., Lea, J. D. and Skillman, S. *Acta Cryst.* **17** (1964) 1040.
7. Hassel, O. and Strømme, K. O. *Acta Chem. Scand.* **13** (1969) 275.
8. Brändén, C. I. *Acta Chem. Scand.* **17** (1963) 1363.
9. Groth, P. and Hassel, O. *Acta Chem. Scand.* **18** (1964) 1327.
10. Hassel, O. and Hvoslef, J. *Acta Chem. Scand.* **8** (1954) 1953.

Received August 19, 1970.