CONTRIBUTION FROM THE INSTITUTE FOR MATERIALS RESEARCH,
NATIONAL BUREAU OF STANDARDS, WASHINGTON, D. C. 20234

The Crystal Structure of Sodium Hexamolybdochromate(III) Octahydrate, $Na_3(CrMo_6O_{24}H_6)\cdot 8H_2O$[1,2]

BY ALVIN PERLOFF

Received April 28, 1969

The detailed structure of the heteropoly salt $Na_3(CrMo_6O_{24}H_6)\cdot 8H_2O$ has been determined using three-dimensional X-ray diffraction data. The crystals are triclinic, space group $P\bar{1}$, with cell dimensions $a = 10.9080$ (4) Å, $b = 10.9807$ (4) Å, $c = 6.4679$ (2) Å, $\alpha = 107.594$ (2)°, $\beta = 84.438$ (2)°, and $\gamma = 112.465$ (3)° at 25°. There is one formula unit per unit cell. Final refinement by least-squares analysis with anisotropic temperature factors resulted in an R value of 3.3%. The anion has the same structure as the $TeMo_6O_{24}{}^{6-}$ anion with excellent agreement of comparable bond distances. Charge balance and hydrogen-bonding arguments suggest that the hydrogen atoms of the anion are bonded to the oxygen atoms which are coordinated to the Cr atom. The anions are linked together through sodium octahedra and hydrogen bonding. No hydrogen atoms could be located directly, but a reasonable hydrogen-bonding scheme was inferred from short oxygen-oxygen distances.

Introduction

Prior work[3-7] has established the existence and probable isomorphism for the heteropoly 6-molybdo anions of the following central trivalent ions: Cr, Al, Fe, Co, Rh, and Ga. Recent work has shown that these anions have a −3 charge in solution[4,8] and that the central ion is octahedrally coordinated.[6,9,10] The observation that some of these salts could be completely dehydrated at moderate temperatures (200° or less) without, apparently, destroying the anion was originally interpreted[4] as evidence that all of the water in these salts was merely water of crystallization. This conclusion led to the postulation of an erroneous dimeric structure.[4] However, later work by some of the same workers[8] and others[11] definitely established the monomeric nature of the anion in solution. In 1960, Agarwala[12] investigated the 6-tungstonickelate(II) anion and concluded that this complex definitely contained constitutional water and had the formula $NiW_6O_{24}H_6{}^{4-}$. The heavy-atom arrangement was found to be consistent with the structure originally proposed by Anderson[13] and later found by Evans[14] in the $TeMo_6O_{24}{}^{6-}$ anion. The similarity of the 6-tungsto and 6-molybdo anions, plus the evidence already cited concerning the anion charge and monomeric nature, led Tsigdinos[6] to conclude that the 6-molybdochromate anion and its isomorphs most probably had the Anderson–Evans structure or one very closely related to it, and his explanation of exchange mechanisms for these anions was based on the assumption of such a structure.

In view of the considerations of the confusing dehydration results and the importance of the work on the 6-molybdoheteropoly anions which depend on structural assumptions, it was felt that a complete X-ray structure determination of a member of this class of heteropoly compounds would be of value. The sodium salt of the hexamolybdochromate(II) anion, which has been described by Tsigdinos[6] as $Na_3(CrO_6Mo_6O_{15+n}H_{2n})\cdot(11-n)H_2O$, was chosen as a representative compound.

Experimental Section

Crystal Preparation.—Large, well-formed single crystals of $Na_3(CrO_6Mo_6O_{15+n}H_{2n})\cdot(11-n)H_2O$ were obtained using a slight modification of the procedure described by Tsigdinos.[6] The pH of a solution containing 145 g of $Na_2MoO_4\cdot 2H_2O$ in 300 ml of water was adjusted to 4.5 with concentrated HNO_3. A second solution was made by dissolving 40.0 g of $Cr(NO_3)_3\cdot 9H_2O$ in 40 ml of water. Half of each solution was mixed together, and the mixture was boiled for 1 min and filtered while hot as recommended by Tsigdinos. The filtrate was set aside in a 1500-ml beaker covered with a ribbed watch glass. Cystallization started in 1 hr. The solution was allowed to stand for 2 weeks before the precipitate was filtered off and washed several times with cold water. Apparently too much evaporation had taken place because, along with the desired reddish purple crystals, some colorless crystals and some green crystals had, also, precipitated. However, there was no intergrowth of the three precipitates and the desired crystals were easily picked out and stored in sealed bottles.

The remaining halves of the original solutions were mixed together without heating and stored in a sealed bottle. Within 1 day reddish purple crystals were formed, and after 3 weeks a sizable crop had precipitated that was free of the unwanted products of the previous procedure. This crop was filtered off, washed with cold water, and stored in sealed bottles.

X-Ray powder patterns and single-crystal procession films showed the reddish purple products of the two procedures to be identical, and all further work was done using the uncontaminated crop.

An attempt was made to recrystallize some of the material

---

[1] Abstracted from the Ph.D. thesis of A. Perloff, Georgetown University, June 1966.

[2] Paper presented in part at the American Crystallographic Association Meeting, Atlanta, Ga., Jan 1967.

[3] H. Struve, *J. Prakt. Chem.*, **61**, 449 (1854); J. G. Gentele, *ibid.*, **81**, 413 (1860); E. Marckwald, Dissertation, University of Basel, Basel, Switzerland, 1895; R. D. Hall, *J. Amer. Chem. Soc.*, **29**, 692 (1907); G. A. Barbieri, *Atti Accad. Naz. Lincei, Cl. Sci. Fis., Mat. Natur., Rend.*, [1] **23**, 338 (1914); A. Rosenheim and H. Schwer, *Z. Anorg. Allg. Chem.*, **89**, 224 (1914).

[4] L. C. W. Baker, G. Foster, W. Tan, F. Scholnick, and T. P. McCutcheon, *J. Amer. Chem. Soc.*, **77**, 2136 (1955).

[5] C. W. Wolfe, M. Block, and L. C. W. Baker, *ibid.*, **77**, 2200 (1955).

[6] G. A. Tsigdinos, Doctoral Dissertation, Boston University, 1961.

[7] O. W. Rollins and J. E. Earley, *J. Amer. Chem. Soc.*, **81**, 5571 (1959).

[8] G. A. Tsigdinos, M. T. Pope, and L. C. W. Baker, Abstracts, 135th National Meeting of the American Chemical Society, Boston, Mass., April 1959, p 48M.

[9] P. Rây, A. Bhaduri, and B. Sarma, *J. Indian Chem. Soc.*, **25**, 51 (1948).

[10] Y. Shimura, H. Ito, and R. Tsuchida, *Nippon Kagaku Zasshi*, **75**, 560 (1954).

[11] E. Matijevic and M. Kerker, *J. Amer. Chem. Soc.*, **81**, 5560 (1959).

[12] U. C. Agarwala, Doctoral Dissertation, Boston University, 1960.

[13] J. S. Anderson, *Nature (London)*, **140**, 850 (1937).

[14] H. T. Evans, Jr., *J. Amer. Chem. Soc.*, **70**, 1291 (1948).

from water in an effort to get smaller, well-shaped crystals. The recrystallized product looked very much the same in color and general morphology, but X-ray examination showed it to be a new phase different from both the original compound and its dehydration products. This new phase is being investigated.

The original crystals readily lose water when exposed to the atmosphere and decompose to a pink powder. For the X-ray powder patterns the crystals were ground in Vaseline. For the single-crystal film work the crystals were coated with Canada balsam or shellac. This was found to be adequate protection for a few days but not for a long enough time to permit a complete set of intensity data to be obtained from one crystal. A small single crystal sealed in a thin-walled Pyrex capillary with some saturated mother liquor was protected indefinitely. This technique was used with the crystal from which the final X-ray intensity data were obtained.

Dehydration Results.—Weight loss was measured on ground samples exposed to the atmosphere at room temperature and on large crystals heated on a recording balance. The results confirm the presence of a total of 11 water molecules (weight loss: calcd, 16.1%; obsd, 16.0%) per empirical formula unit and indicate the existence of a stable, lower hydrate containing five molecules of water per empirical formula unit. The sample dried at room temperature attained constant weight after a weight loss approximating six molecules of water (weight loss: calcd, 8.8%; obsd, 8.4%). A recording balance trace (Figure 1) reveals that the water is lost in two stages, with six water molecules being readily lost at temperatures of about 200° and five additional water molecules being lost at more elevated temperatures (about 300°). There appears to be no distinction between the loss of water of hydration and constitutional water in the second stage. X-Ray powder patterns show the lower hydrate to be a distinct crystalline phase.

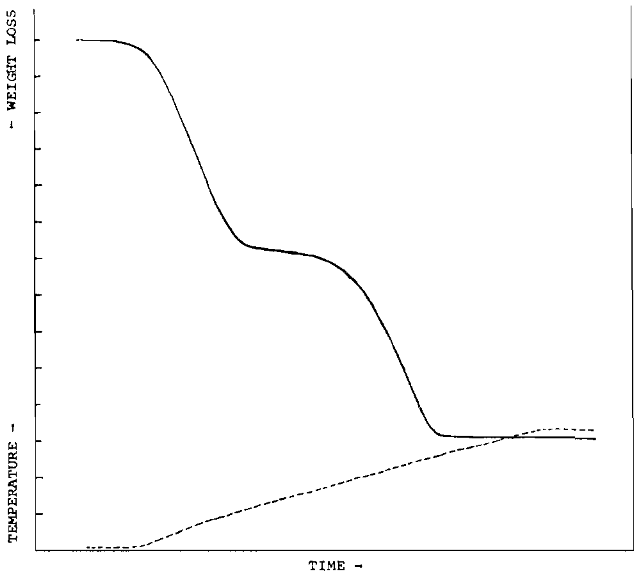

Figure 1.—Weight loss (solid line) and temperature (dashed line) *vs.* time curves. The first break in the weight loss curve corresponds to the loss of six H_2O molecules and the second to the loss of five more H_2O molecules. One unit on the ordinate axis corresponds to a weight loss of 1 mg (weight loss curve) or 1-mV potential change across the Pt—Pt–10% Rh thermocouple (temperature curve). The abscissa is time increasing to the right. The heating rate was 6°/min. The center of the intermediate plateau is observed at 200° and the cessation of weight loss occurs at approximately 330°.

Crystal Data

Morphology.—The rapidly grown crystals varied widely in morphology. Crystals which grew slowly or had the opportunity to perfect themselves generally showed a tabular habit with a tendency to elongate in one direction. Referred to the unit cell used in this paper, the dominant form is {010} and the secondary forms are {100} and {001}. The direction of elongation is the *c* axis. The crystals have perfect (010) cleavage.

Unit Cell and Space Group.—The cell data are given in Table I. The crystals are triclinic. The unit cell directions indicated by the morphology were found to provide a convenient unit cell and this was adopted. The Laue symmetry and approximate unit cell dimensions were obtained from precession films. Final cell dimensions were determined from a least-squares refinement at 19 high-angle reflections ($2\theta > 150°$) measured at 25° on the goniostat using Ni-filtered Cu $K\alpha_1$ radiation (λ 1.54051 Å). A 1° takeoff angle was used for these measurements.

TABLE I
CRYSTAL DATA[a]

$a = 10.9080\ (4)$ Å	Space group $P\bar{1}$
$b = 10.9807\ (4)$ Å	
$c = 6.4679\ (2)$ Å	$Z = 1$
$\alpha = 107.594\ (2)°$	Density (obsd) = 2.950 g/cm³
$\beta = 84.438\ (2)°$	Density (calcd) = 2.995 g/cm³
$\gamma = 112.465\ (3)°$	

[a] Values in parentheses are the standard deviation as computed by the least-squares program and refer to the last decimal place.

The crystals were tested for a piezoelectric effect with negative results, suggesting that the probable space group is $P\bar{1}$ rather than $P1$. The space group was assumed to be $P\bar{1}$ and the successful structure determination proved this assumption to be correct.

Density measurements (Table I), made on a Berman microbalance using toluene as the immersion liquid, established that there is one formula unit per unit cell.

Collection and Reduction of Intensity Data

Single-crystal intensity data were obtained using the stationary-crystal, stationary-counter method. The crystal used was a fragment roughly approximating a rectangular prism with dimensions of 0.203 × 0.224 × 0.294 mm. It was sealed in a thin-walled Pyrex capillary (0.03-mm wall thickness) with the long direction approximately parallel to the capillary axis. Complete three-dimensional data to $2\theta = 60°$ were measured with a General Electric counter diffractometer using molybdenum radiation (λ 0.71069 Å) and a β filter of 0.025-mm thick niobium. A 4° takeoff angle was used. The detector was a scintillation counter equipped with a pulse-height analyzer set to pass the central 90% of the Mo $K\alpha$ peak. Attenuation filters of niobium were used on all reflections for which the unattenuated peak intensity exceeded 20,000 counts/sec so that all measurements were within the linear response range of the equipment.

Angular settings were computed[15] from the refined

---

(15) The angular settings and data reduction to *F* values were done using programs written by F. A. Mauer of the National Bureau of Standards. All other computations were done with the appropriate programs of the X-Ray 63 system, Technical Report TR-64-6, Computer Science Center, University of Maryland, 1964.

TABLE II
OBSERVED AND CALCULATED STRUCTURE FACTORS[a]

H,0,0		
1	534	718
2	406	-380
3	1331	1452
4	706	796
5	677	-645
6	251	249
7	1276	1324
8	846	815
9	81	-105
10	456	464
11	887	867
12	94	-96
13	129	-117
14	605	569
H,1,0		
7	501	482
8	86	-21
9	493	-481
10	96	87
11	72	76
12	311	-303
13	198	-199
H,-1,0		
-6	41	38
-5	705	-687
-4	56	73
-3	1024	1029
-2	620	-629
-1	347	-369
0	1252	1441
1	786	708
2	869	-931
3	587	567
4	721	794
5	461	-437
6	85	45
7	670	651
8	867	863
9	377	-379
10	193	222
11	833	810
12	136	117
13	219	-219
14	517	522
H,-2,0		
-13	149	-150
-12	111	120
-11	152	-148
-10	351	-345
-9	31*	17
-8	205	-210
-7	549	-561
-6	147	-148
-5	126	-141
-4	325	-328
-3	707	-702
-2	237	-260
-1	745	-774
0	373	-370
1	639	-657
2	497	-493
3	575	-578
4	401	-415
5	426	-437
6	744	-758
7	45	22
8	78	-78
9	351	-353
10	217	-221
11	32*	27
12	160	-152
13	233	-238
14	64	-63
H,-3,0		
-12	628	618
-11	209	209
-10	231	-226
-9	445	432
-8	903	905
-7	191	-165
-6	215	-219
-5	821	623
-4	603	611
-3	636	-651
-2	146	141
-1	1105	1160
0	162	-217
1	768	-765
2	928	957
3	415	431
4	156	-196
5	121	-132
6	898	909
7	418	-424
8	469	-480
9	29*	-6
10	321	327
11	371	-370
12	280	-277
13	219	215
14	37*	-50
15	284	-310
H,-4,0		
-12	545	542
-11	450	452
-10	161	-156
-9	380	367
-8	900	914
-7	340	333
-6	499	-493
-5	754	787
-4	1271	1328
-3	501	-501
-2	851	634
-1	2167	2622
0	490	516
1	229	-244
2	1493	1553
3	964	1063
4	227	-217
5	113	97
6	1373	1437
7	1181	1218
8	302	-320
9	433	446
10	1113	1161
11	85	-97
12	147	-151
13	565	587
14	469	475
15	199	-198
H,-5,0		
-11	39*	25
-10	129	-132
-9	81	-60
-8	346	344
-7	74	-82
-6	487	-464
-5	211	-214
-4	380	330
-3	402	-391
-2	273	-273
-1	1442	1513
0	750	741
1	549	-592
2	139	128
3	127	156
4	175	-156
5	542	-545
6	695	726
7	935	923
8	117	-135
9	31*	0
10	921	941
11	121	119
12	237	-252
13	525	569
14	461	461
15	107	-107
H,-6,0		
-10	60	-36
-9	37*	-30
-8	314	-311
-7	111	-126
-6	68	-67
-5	300	-304
-4	243	-236
-3	253	-265
-2	151	-150
-1	569	-572
0	333	-342
1	590	-596
2	319	-333
3	510	-532
4	452	-470
5	254	-268
6	113	-130
7	241	-245
8	276	-277
9	294	-291
10	30*	-27
11	235	-246
12	179	-191
13	123	-130
14	80	75
15	174	-162
H,-7,0		
-9	549	546
-8	138	-89
-7	143	-138
-6	632	647
-5	469	475
-4	454	-456
-3	175	161
-2	707	710
-1	92	98
0	571	-562
1	646	629
2	664	695
3	487	-501
4	150	-154
5	475	471
6	88	76
7	536	-544
8	199	208
9	306	321
10	286	-289
11	279	-207
12	230	239
13	35*	-52
14	331	-354
15	74	-58
H,-8,0		
-8	190	193
-7	285	-268
-6	626	628
-5	724	741
-4	64	-71
-3	156	163
-2	1213	1243
-1	361	369
0	90	-78
1	594	591
2	1099	1150
3	279	257
4	29*	30
5	1287	1338
6	825	837
7	394	-397
8	393	372
9	976	1029
10	79	96
11	223	-232
12	669	659
13	485	516
14	135	-136
H,-9,0		
-7	323	-317
-6	207	-207
-5	201	206
-4	226	-226
-3	76	-54
-2	564	564
-1	355	348
0	298	-290
1	31*	-8
2	385	366
3	203	-199
4	374	-386
5	324	307
6	880	884
7	273	-272
8	77	85
9	733	738
10	156	152
11	250	-253
12	356	370
13	450	446
14	38*	-20
H,-10,0		
-6	159	-162
-5	327	-322
-4	122	-127
-3	137	-125
-2	82	-79
-1	186	-106
0	214	-230
1	227	-230
2	522	-529
3	327	-336
4	201	-205
5	169	-169
6	208	-209
7	108	-106
8	269	-260
9	175	-169
10	128	-135
11	231	-235
12	36*	-6
13	37*	-22
14	140	-137
H,-11,0		
-5	140	-142
-4	56	73
-3	544	550
-2	72	54
-1	342	-346
0	333	325
1	366	399
2	247	-249
3	243	-235
4	574	578
5	93	76
6	346	-346
7	76	81
8	334	341
9	169	-194
10	245	-240
11	160	169
12	37*	22
13	259	-260
H,-12,0		
-3	532	537
-2	401	400
-1	139	-128
0	310	307
1	894	909
2	154	154
3	61	-48
4	726	741
5	577	581
6	194	-184
7	266	263
8	791	768
9	141	131
10	105	-89
11	442	445
12	499	537
H,-13,0		
-1	151	-143
0	109	-100
1	198	201
2	56	36
3	237	-236
4	328	324
5	409	416
6	175	-184
7	36*	-9
8	430	419
9	139	133
10	181	-184
11	204	202
H,-14,0		
1	296	-299
2	254	-258
3	138	-139
4	217	-222
5	119	-117
6	93	-60
7	166	-162
8	38*	-70
9	136	-133
H,0,1		
1	1231	1261
2	607	-631
3	920	888
4	961	1027
5	390	-336
6	302	-311
7	1017	1031
8	1077	1065
9	132	122
10	115	120
11	984	975
12	156	151
13	334	-332
14	334	324
H,1,1		
7	1229	1272
8	730	727
9	187	-197
10	398	402
11	612	615
12	96	-94
13	271	-279
H,-1,1		
-6	233	-253
-5	547	-547
-4	226	-228
-3	724	-660
-2	502	-502
-1	827	-854
0	331	-367
1	33	-53
2	1085	-1101
3	671	-632
4	282	216
5	138	-151
6	653	-656
7	117	93
8	111	103
9	248	-244
10	301	-300
11	66	45
12	58	42
13	362	-345
14	66	-31
H,-2,1		
-13	119	-117
-12	622	608
-11	316	300
-10	456	-440
-9	299	296
-8	585	553
-7	266	-250
-6	433	-432
-5	570	563
-4	69	59
-3	229	141
-2	426	-405
-1	1051	1050
0	418	-384
1	970	-952
2	438	420
3	323	290
4	518	-531
5	340	-339
6	246	234
7	91	-101
8	761	-752
9	179	-180
10	117	111
11	294	-281
12	320	-312
13	83	69
14	122	-118
H,-3,1		
-12	579	559
-11	610	593
-10	319	-309
-9	109	84
-8	1132	1142
-7	360	357
-6	48	-28
-5	1137	1145
-4	1586	1658
-3	368	-358
-2	56	40
-1	1986	2279
0	1071	1028
1	496	-484
2	1082	1067
3	1919	2225
4	509	-458
5	86	62
6	1088	1119
7	1099	1091
8	348	-342
9	235	223
10	933	897
11	75	74
12	279	-270
13	552	540
14	461	444
15	134	-151
H,-4,1		
-12	162	151
-11	249	229
-10	280	-281
-9	250	-249
-8	404	402
-7	359	345
-6	623	-605
-5	333	323
-4	977	966
-3	73	-72
-2	336	-361
-1	1470	1486
0	1023	1001
1	646	-624
2	208	205
3	241	349
4	232	218
5	395	-402
6	914	919
7	1496	1563
8	124	91
9	194	181
10	1060	1047
11	278	278
12	535	-520
13	478	467
14	666	655
15	39*	27
H,-5,1		
-11	168	-150
-10	80	-70
-9	169	-164
-8	226	-224
-7	153	-149
-6	199	-199
-5	460	-447
-4	320	-327
-3	405	-392
-2	655	-666
-1	144	-170
0	416	-422
1	217	-240
2	481	-474
3	82	67
4	854	-837
5	469	-464
6	293	-291
7	372	327
8	109	-125
9	389	-387
10	181	171
11	70	-77
12	502	-500
13	78	-76
14	209	203
15	64	-42
H,-6,1		
-10	244	246
-9	457	430
-8	145	-128
-7	209	-211
-6	246	242
-5	517	511
-4	320	-320
-3	268	-271
-2	768	750
-1	41	28
0	682	-690
1	110	92
2	357	346
3	605	-594
4	273	-271
5	431	411
6	67	-23
7	480	-493
8	230	-218
9	263	257
10	565	-561
11	301	-296
12	74	-65
13	58	-45
14	263	-253
15	104	-102
H,-7,1		
-9	742	732
-8	318	311
-7	165	-166
-6	599	587
-5	943	949
-4	104	100
-3	28*	-18
-2	1262	1307
-1	837	819
0	555	-538
1	976	977
2	1337	1406
3	236	235
4	37	-45
5	1056	1072
6	893	894
7	424	-423
8	70	35
9	1043	1041
10	293	295
11	307	-299
12	596	584
13	578	561
14	177	-166
15	38*	1
H,-8,1		
-9	198	189
-8	156	154
-7	203	-201
-6	92	104
-5	641	633
-4	128	-120
-3	223	-223
-2	614	584
-1	575	559
0	257	-261
1	58	-66
2	761	762
3	299	304
4	482	-491
5	633	622
6	1203	1200
7	145	116
8	153	-149
9	1045	1044
10	217	212
11	288	-265
12	422	414
13	711	709
14	71	58
15	39*	35
H,-9,1		
-8	92	-73
-7	178	-182
-6	211	-207
-5	239	-233
-4	205	-199
-3	284	-286
-2	103	-97
-1	103	-115
0	268	-274
1	286	-289
2	573	-563
3	351	-341
4	411	-416
5	195	-198
6	270	246
7	79	-83
8	236	-240
9	147	135
10	209	-193
11	469	-464
12	119	-114
13	123	125
14	62	-63
H,-10,1		
-6	309	306
-5	207	-217
-4	205	-194
-3	497	482
-2	33*	38
-1	504	-499
0	206	204
1	348	351
2	263	-256
3	370	-371
4	256	259
5	69	55
6	388	-392
7	70	20
8	103	99
9	251	-244
10	421	-416
11	83	-95
12	53	-57
13	223	-218
14	137	-121
H,-11,1		
-5	95	94
-4	71	-75
-3	776	779
-2	556	561
-1	148	-146
0	326	329
1	919	925
2	139	127
3	31*	-12
4	654	660
5	796	811
6	161	-164
7	65	45
8	673	867
9	212	209
10	291	-280
11	475	456
12	516	510
13	67	-49
14	82	-77
H,-12,1		
-4	167	-153
-3	190	177
-2	348	354
-1	210	-219
0	34*	-10
1	532	525
2	305	304
3	252	-249
4	375	375
5	683	684
6	157	-162
7	111	-130
8	624	612
9	388	385
10	171	-165
11	359	359
12	583	572
13	140	127
H,-13,1		
-2	164	-156
-1	97	-91
0	277	-275
1	267	-259
2	222	-212
3	276	-280
4	180	-176
5	105	102
6	49	-29
7	159	-154
8	35*	22
9	201	-207
10	282	-279
11	109	-104
12	39*	14
H,-14,1		
0	266	271
1	246	-246
2	273	-273
3	185	180
4	70	70
5	167	-159
6	65	-40
7	170	184
8	112	-125
9	308	-311
10	123	-138
H,-15,1		
4	557	562
5	60	-46
6	96	90
7	544	547
H,0,-1		
0	1560	1740
1	289	-284
2	639	-626
3	1475	1568
4	157	154
5	503	-506
6	324	299
7	847	836
8	227	-232
9	380	-381
10	502	512
11	206	212
12	291	-292
13	64	-88
14	236	230
H,1,-1		
7	368	-361
8	316	-315
9	30*	-47
10	311	-310
11	157	-159
12	103	-93
13	139	-148
H,-1,-1		
-6	139	-156
-5	117	-128
-4	1013	1046
-3	904	923
-2	877	-860
-1	1162	1174
0	2094	2627
1	478	448
2	270	-253
3	1927	2148
4	1606	1726
5	230	-248
6	390	377
7	1442	1588
8	111	113
9	328	-328
10	536	540
11	933	938
12	34*	3
13	163	183
14	647	665
H,-2,-1		
-13	135	-131
-12	253	-244
-11	53	48
-10	199	-210
-9	398	-389
-8	111	-113
-7	28*	-14
-6	307	-303
-5	625	-618
-4	295	244
-3	262	232
-2	400	-410
-1	241	-230
0	562	562
1	62	74
2	983	-1001
3	18*	27
4	834	805
5	35	-20
6	452	-465
7	671	698
8	176	162
9	653	-624
10	284	291
11	546	558
12	33*	16
13	35*	2
14	394	407
H,-3,-1		
-12	130	131
-11	141	-125
-10	311	-299
-9	84	67
-8	44	42
-7	368	-373
-6	256	-248
-5	523	495
-4	764	-758
-3	366	-374
-2	238	-250
-1	495	-469
0	691	-681
1	372	-379
2	60	9
3	75	-110
4	785	-792
5	533	-528
6	472	-451
7	371	-373
8	375	-383
9	217	-222
10	208	-200
11	32*	-29
12	237	-228
13	107	-117
14	113	-113
H,-4,-1		
-11	199	191
-10	64	-59
-9	563	559
-8	872	889
-7	245	-234
-6	29*	-39
-5	1105	1128
-4	644	634
-3	697	-680
-2	812	800
-1	1339	1456
0	311	-316
1	206	-232
2	631	646
3	888	844
4	813	-809
5	395	372
6	977	980
7	26*	-33
8	322	-331
9	559	580
10	298	287
11	345	-352
12	165	-155
13	296	296
14	65	-64
H,-5,-1		
-11	333	325
-10	134	-135
-9	478	478
-8	797	796
-7	32*	29
-6	416	-407
-5	766	768
-4	638	639
-3	213	-222
-2	531	513
-1	1442	1559
0	512	497
1	356	-343
2	1505	1559
3	1269	1318
4	95	-120
5	344	320
6	1331	1416
7	417	412
8	420	-422
9	747	779
10	970	1003
11	145	136
12	48	-42
13	760	726
14	406	381
15	249	-254
H,-6,-1		
-10	153	-154
-9	114	-115
-8	34*	30
-7	203	-200
-6	294	-283
-5	125	-114
-4	29*	6
-3	255	-263
-2	386	-393
-1	456	441
0	85	-79
1	491	-495
2	50	-63
3	960	957
4	431	-437
5	215	-228
6	542	535
7	57	55
8	537	-528
9	29*	3
10	484	475
11	178	177
12	61	-45
13	378	398
14	335	358
H,-7,-1		
-9	122	121
-8	366	-363
-7	119	-116
-6	146	141
-5	236	-241
-4	301	-288
-3	43	-47
-2	50	-55
-1	381	-384
0	267	-285
1	226	-232
2	187	-191
3	578	-586
4	237	-242
5	83	-80
6	528	-521
7	395	-403
8	351	-361
9	30*	-33
10	289	-285
11	60	-64
12	87	-78
13	100	-99
14	149	-157
H,-8,-1		
-8	65	-60
-7	37*	-24
-6	833	854
-5	337	332
-4	335	-321
-3	450	447
-2	664	684
-1	29*	-3
0	305	-313
1	824	835
2	725	730
3	424	-425
4	28*	-12
5	811	814
6	53	-17
7	370	-370
8	468	479
9	374	376
10	186	-182
11	160	-159
12	297	316
13	70	69
14	323	-346
H,-9,-1		
-7	188	-197
-6	398	424
-5	507	515
-4	186	-183
-3	218	217
-2	946	969
-1	358	349
0	180	-171
1	589	601
2	865	875
3	68	-66
4	66	58
5	1125	1158
6	474	470
7	51	-50
8	504	508
9	913	959
10	79	22
11	95	-83
12	543	579
13	399	445
14	203	-236
H,-10,-1		
-5	75	-40
-4	141	-134
-3	242	-231
-2	122	117
-1	33*	45
0	384	-379
1	137	117
2	347	352
3	177	-178
4	118	-141
5	207	212
6	31*	6
7	358	-365
8	32*	-51
9	418	417
10	188	191
11	141	-133
12	240	249
13	339	372
H,-11,-1		
-4	38*	-22
-3	76	45
-2	196	-190
-1	295	-290
0	60	-47
1	111	-123
2	301	-296
3	168	-173
4	32*	-42
5	248	-254
6	386	-400
7	120	-132
8	215	-206
9	249	-262
10	73	-67
11	36*	0
12	81	-97
H,-12,-1		
-2	39*	62
-1	113	-124
0	410	409
1	516	520
2	262	-274
3	55	60
4	569	590
5	34*	15
6	331	-322
7	220	219
8	391	389
9	81	-81
10	119	-113
11	366	386
H,-13,-1		
0	332	337
1	595	593
2	37*	26
3	36*	-43
4	633	643
5	422	419
6	94	-85
7	363	365
8	645	649
9	139	133
10	116	-116
H,-14,-1		
4	59	70
5	66	79
6	107	-109
7	57	-50
H,0,2		
1	486	442
2	529	-520
3	593	-615
4	431	396
5	144	111
6	510	-512
7	186	-197
8	487	487
9	287	-289
10	192	-182
11	256	252
12	231	233
13	305	-299
H,1,2		
8	1046	1072
9	100	-106
10	81	92
11	834	833
12	293	287
13	216	-200
H,-1,2		
-6	452	-450
-5	258	241
-4	251	238
-3	590	-612
-2	169	108
-1	64	78
0	411	-386
1	900	-923
2	107	-132
3	172	-185
4	326	-337
5	627	-629
6	175	-179
7	80	71
8	631	-623
9	245	-245
10	139	-144
11	276	-279
12	174	-166
13	118	-114
14	143	-145
H,-2,2		
-12	532	526
-11	724	722
-10	194	-192
-9	56	-59
-8	1209	1247
-7	376	365
-6	100	-117
-5	1105	1091
-4	1536	1624
-3	103	-94
-2	243	-217
-1	1250	1324
0	1451	1465
1	589	-566
2	395	379
3	1622	1762
4	324	288
5	720	-724
6	1103	1142
7	560	573
8	275	-280
9	29*	14
10	816	821
11	33*	17
12	229	-220
13	312	321
14	298	286
H,-3,2		
-12	150	154
-11	520	523
-10	225	-219
-9	493	-483
-8	597	590
-7	709	765
-6	422	-431
-5	896	858
-4	1443	1519
-3	109	96
-2	304	-294
-1	45	81
0	691	776
1	317	-324
2	287	248
3	1976	2238
4	1129	1149
5	387	-398
6	1099	1131
7	1211	1273
8	233	-215
9	85	72
10	1000	1017
11	516	501
12	217	-211
13	258	241
14	784	789
H,-4,2		
-12	111	-103
-11	91	-80
-10	89	-78
-9	403	-403
-8	186	-190
-7	239	-241
-6	300	-289
-5	311	-321
-4	49	39
-3	427	-426
-2	610	-609
-1	290	-283
0	173	-138
1	144	-177
2	604	-592
3	399	365
4	363	317
5	1031	-1073
6	147	-59
7	497	486
8	116	-121
9	377	-374
10	346	338
11	162	162
12	373	-368
13	119	-108
14	335	319
15	80	83
H,-5,2		
-11	209	-207
-10	36*	-33
-9	413	415
-8	63	-77
-7	335	-330
-6	61	52
-5	175	166
-4	335	-336
-3	401	-407
-2	422	403
-1	235	-237
0	317	-329
1	440	-421
2	267	243
3	610	-606
4	449	-460
5	78	46
6	130	-142
7	718	-728
8	297	-301
9	120	-111
10	311	-289
11	328	-328
12	137	-131
13	171	-165
14	67	-60
15	180	-179
H,-6,2		
-10	65	48
-9	684	680
-8	447	437
-7	184	-182
-6	532	509
-5	1112	1124
-4	164	147
-3	27*	-49
-2	1133	1157
-1	1156	1160
0	765	-744
1	525	488
2	1521	1536
3	123	93
4	421	-418
5	803	802
6	1028	1056
7	394	-399
8	92	94
9	751	744
10	255	248
11	304	-283
12	292	294
13	423	406
14	72	-65
15	174	-163
H,-7,2		
-9	394	393
-8	361	349
-7	156	-151
-6	337	330
-5	804	790
-4	289	281
-3	566	-558
-2	457	460
-1	853	846
0	25*	-6
1	142	-148
2	1534	1577
3	747	725
4	351	-344
5	806	792
6	1070	1100
7	95	90
8	88	-75
9	964	970
10	715	697
11	250	-230
12	392	385
13	822	827
14	143	145
15	140	-141
H,-8,2		
-9	240	-246
-8	74	-53
-7	160	-149
-6	126	-127
-5	33*	55
-4	160	-168
-3	304	-312
-2	541	-543
-1	205	-202
0	346	-329
1	489	-487
2	304	281
3	204	155
4	433	-424
5	157	-163
6	512	486
7	427	-428
8	223	-222
9	143	136
10	194	174
11	383	-378
12	153	-153
13	310	299
14	124	115
15	138	-123
H,-9,2		
-8	265	-265
-7	109	104
-6	227	226
-5	264	-255
-4	269	-264
-3	153	147
-2	110	99
-1	505	-508
0	26*	-2
1	302	299
2	358	-343
3	376	-360
4	171	-166
5	183	-182
6	483	-467
7	160	-167
8	29*	-29
9	217	-215
10	365	-345
11	258	-249
12	87	-64
13	257	-258
14	135	-125
H,-10,2		
-7	182	174
-6	725	728
-5	169	169
-4	147	-149
-3	789	795
-2	666	676
-1	241	-231
0	251	253
1	879	885
2	214	214
3	265	-266
4	769	755
5	744	734
6	97	-84
7	170	-169
8	713	708
9	262	278
10	321	-300
11	195	186
12	521	511
13	36*	2
14	168	-176
H,-11,2		
-5	190	196
-4	237	-241
-3	274	287
-2	676	674
-1	165	-162
0	32*	-21
1	846	839
2	445	436
3	205	-200
4	300	266
5	907	867
6	144	146
7	142	-143
8	784	792
9	632	635
10	90	-90
11	244	238
12	764	770
13	136	122
14	79	-71
H,-12,2		
-4	275	-263
-3	312	-308
-2	130	-137
-1	159	-156
0	310	-309
1	66	60
2	73	77
3	297	-284
4	32*	-42
5	72	81
6	142	-136
7	257	-256
8	33*	24
9	78	60
10	229	-233
11	90	-82
12	250	247
13	39*	61
H,-13,2		
-2	336	-343
-1	53	-47
0	210	199
1	185	-177
2	233	-222
3	34*	-6
4	34*	-50
5	236	-230
6	244	-246
7	34*	10
8	86	-76
9	348	-353
10	151	-156
11	134	-132
12	203	-211
H,-14,2		
0	597	592
1	236	244
2	108	-99
3	285	274
4	534	514
5	36*	-41
6	56	-50
7	500	504
8	308	322
9	258	-257
10	110	94
11	444	441
H,-15,2		
3	172	190
4	580	569
5	161	164
6	148	-141
7	466	474
8	446	452
H,0,-2		
0	95	-64
1	452	-493
2	530	-504
3	60	50
4	433	-451
5	939	-948
6	173	-177
7	158	-159
8	289	-298
9	216	-227
10	32*	-14
11	213	-218
12	236	-233
13	87	-35
H,1,-2		
7	200	-194
8	290	297
9	58	-45
10	361	-335
11	129	126
12	335	343
13	169	-162
H,-1,-2		
-7	972	973
-6	449	-430
-5	625	593
-4	1814	1960
-3	194	154
-2	192	130
-1	283	343
0	1311	1368
1	522	-514
2	207	-188
3	1686	1776
4	1194	1186
5	615	-601
6	805	794
7	605	821
8	574	-581
9	123	-133
10	667	704
11	279	269
12	164	-172
13	114	118
14	426	399
H,2,-2		
13	83	-77
H,-2,-2		
-12	36*	5
-11	513	517
-10	142	137
-9	470	-459
-8	450	444
-7	747	737
-6	327	-318
-5	266	250
-4	1336	1400
-3	646	636
-2	407	-429
-1	552	542
0	420	409
1	19*	-3
2	184	-187
3	1651	1724
4	1866	1914
5	465	-471
6	796	780
7	1258	1301
8	50	-42
9	353	-374
10	807	857
11	645	697
12	34*	9
13	126	135
14	604	641
H,-3,-2		
-12	190	-189
-11	92	-74
-10	190	-199
-9	261	-260
-8	92	-93
-7	239	-249
-6	303	-308
-5	354	-360
-4	122	-129
-3	466	-450
-2	315	-315
-1	530	-547
0	395	353
1	245	-274
2	674	-665
3	165	153
4	421	396
5	341	-342
6	444	-435
7	343	334
8	146	-158
9	424	-445
10	100	103
11	307	318
12	53	-56
13	126	-124
14	286	307
H,-4,-2		
-11	187	-182
-10	162	-165
-9	219	220
-8	150	151
-7	389	-384
-6	51	-25
-5	415	404
-4	344	-329
-3	349	-368
-2	218	194
-1	24*	-41
0	564	-581
1	331	-342
2	376	356
3	499	-496
4	548	-551
5	327	-312
6	202	187
7	686	-693
8	220	-239
9	71	-67
10	188	-201
11	298	-323
12	139	-157
13	118	-117
14	88	-104
H,-5,-2		
-10	118	-105
-9	691	689
-8	619	609
-7	201	-188
-6	304	301
-5	1043	1077
-4	378	381
-3	329	-341
-2	789	787
-1	1200	1263
0	76	-71
1	132	-136
2	1401	1478
3	668	656
4	868	-850
5	562	558
6	877	866
7	111	-121
8	29*	-32
9	657	660
10	476	485
11	281	-292
12	86	101
13	382	400
14	37*	-3
H,-6,-2		
-9	212	213
-8	510	514
-7	184	-178
-6	53	54
-5	800	813
-4	435	436
-3	179	-193
-2	50	57
-1	680	682
0	105	88
1	464	-476
2	1224	1273
3	1251	1256
4	58	-46
5	493	481
6	1151	1185
7	150	-140
8	184	-195
9	650	663
10	773	795
11	75	86
12	35*	51
13	686	751
14	289	304
H,-7,-2		
-8	193	-199
-7	132	-143
-6	166	-159
-5	146	-141
-4	81	-34
-3	313	-303
-2	304	-298
-1	246	-252
0	229	-232
1	406	-416
2	149	131
3	370	360
4	331	-327
5	261	-272
6	215	209
7	236	-240
8	363	-371
9	32*	-23
10	329	346
11	86	-75
12	115	-104
13	150	151
14	164	175
H,-8,-2		
-7	38*	-21
-6	321	316
-5	125	-127
-4	414	-413
-3	162	170
-2	60	40
-1	319	-307
0	216	-203
1	191	179
2	127	-129
3	464	-475
4	29*	27
5	53	-21
6	437	-436
7	372	-383
8	46	-47
9	187	-192
10	307	-315
11	114	-120
12	35*	0
13	124	-131
H,-9,-2		
-6	680	684
-5	266	276
-4	137	-141
-3	436	453
-2	864	876
-1	85	-114
0	60	0
1	861	896
2	390	400
3	362	-370
4	282	279
5	777	783
6	32*	35
7	181	-172
8	575	585
9	472	485
10	212	-208
11	80	-70
12	464	474
13	38*	35
H,-10,-2		
-4	209	-222
-3	53	67
-2	556	564
-1	123	116
0	208	-203
1	669	673
2	723	723
3	237	-236
4	198	214
5	645	642
6	258	265
7	108	-102
8	460	471
9	690	709
10	57	70
11	36*	-13
12	605	651
H,-11,-2		
-3	258	-262
-2	194	-205
-1	185	-177
0	289	-298
1	35*	5
2	148	148
3	34*	-49
4	105	-106
5	72	87
6	222	-225
7	293	-298
8	35*	-50
9	146	150
10	36*	25
11	158	-155
H,-12,-2		
-1	202	-196
0	213	202
1	37*	-15
2	184	-181
3	65	-56
4	36*	42
5	305	-312
6	290	-306
7	61	-56
8	37*	-51
9	214	-225
10	93	-86
H,-13,-2		
3	165	172
4	532	535
5	106	101
6	163	-166
7	334	339
8	389	398
H,0,3		
1	408	-412
2	419	-431
3	146	-155
4	530	-530
5	418	-421
6	438	-432
7	256	-255
8	204	-201
9	143	-153
10	327	-332
11	122	-112
12	37*	-24
13	210	-209
H,1,3		
8	605	599
9	110	-117
10	335	-330
11	469	474
12	349	356
H,-1,3		
-6	513	-506
-5	586	568
-4	1198	1219
-3	284	254
-2	585	-567
-1	1248	1266
0	610	596
1	243	-250
2	190	-160
3	920	955
4	336	316
5	579	-591
6	654	646
7	609	604
8	397	-397
9	58	-41
10	453	451
11	33*	-3
12	299	-286
13	66	49
H,-2,3		
-12	77	98
-11	732	741
-10	129	124
-9	223	-222
-8	742	708
-7	827	819
-6	271	-257
-5	254	238
-4	1465	1555
-3	361	359
-2	364	-374
-1	661	674
0	1081	1124
1	116	65
2	199	181
3	2282	2564
4	1080	1082
5	217	-213
6	542	532
7	1235	1272
8	284	-275
9	97	-82
10	891	897
11	703	702
12	35*	-40
13	230	226
14	612	606
H,-3,3		
-12	190	-193
-11	61	8
-10	89	-90
-9	457	-451
-8	127	-130
-7	98	91
-6	527	-512
-5	53	-58
-4	299	284
-3	24*	20
-2	637	-650
-1	354	-321
0	170	185
1	140	-157
2	704	-706
3	674	637
4	1248	1239
5	668	-677
6	26*	-48
7	577	570
8	354	-333
9	390	-393
10	334	340
11	538	534
12	78	-80
13	58	56
14	447	437
H,-4,3		
-11	159	-146
-10	194	-198
-9	161	172
-8	75	-77
-7	406	-406
-6	88	-87
-5	77	-74
-4	393	-405
-3	223	-228
-2	75	44
-1	215	-209
0	719	-701
1	138	-152
2	500	-498
3	297	-305
4	645	-647
5	202	-206
6	481	-471
7	251	-264
8	492	-495
9	266	-265
10	49	-49
11	167	-166
12	172	-159
13	167	-163
14	83	-80
H,-5,3		
-11	39*	22
-10	37*	-30
-9	659	647
-8	634	643
-7	257	-249
-6	341	340
-5	1186	1203
-4	120	-114
-3	280	-274
-2	827	807
-1	913	902
0	224	-244
1	127	-115
2	1222	1242
3	340	325
4	759	-752
5	549	532
6	772	774
7	238	-239
8	121	-116
9	586	583
10	46	-14
11	209	-191
12	90	-60
13	338	325
14	96	-96
H,-6,3		
-10	193	-190
-9	474	467
-8	635	627
-7	204	-209
-6	32*	37
-5	960	970
-4	519	506
-3	387	-390
-2	650	652
-1	1187	1226
0	89	74
1	52	36
2	1328	1337
3	1160	1150
4	412	-410
5	804	789
6	1282	1333
7	91	85
8	55	-33
9	938	944
10	808	800
11	109	-127
12	152	144
13	752	735
14	238	248
H,-7,3		
-9	123	-117
-8	35*	-4
-7	146	-147
-6	220	-221
-5	121	128
-4	56	-58
-3	338	-322
-2	602	-588
-1	103	116
0	189	-186
1	513	-506
2	923	855
3	914	857
4	555	-553
5	105	-109
6	306	320
7	318	-303
8	533	-537
9	351	346
10	497	486
11	121	93
12	112	-110
13	505	493
14	196	195
H,-8,3		
-8	304	-302
-7	96	-90
-6	181	181
-5	231	-234
-4	188	-179
-3	30*	-21
-2	176	-166
-1	459	-457
0	281	-277
1	169	-157
2	362	-357
3	125	-137
4	365	-374
5	129	-138
6	492	-482
7	546	-535
8	197	-185
9	191	-191
10	261	-257
11	32*	-56
12	165	-165
13	112	-117
14	155	-147
H,-9,3		
-7	222	221
-6	645	642
-5	132	128
-4	218	-208
-3	613	621
-2	667	676
-1	179	-182
0	30*	6
1	865	857
2	379	350
3	543	-530
4	472	457
5	647	639
6	239	-227
7	198	-199
8	416	407
9	376	379
10	270	-261
11	33*	33
12	350	342
13	36*	-34
14	297	-291
H,-10,3		
-6	648	653
-5	287	273
-4	223	-220
-3	379	372
-2	800	805
-1	171	164
0	176	-175
1	932	935
2	703	686
3	180	-172
4	283	266
5	1169	1175
6	317	300
7	30*	19
8	749	741
9	737	725
10	89	-76
11	74	52
12	734	725
13	332	331
14	223	-226
H,-11,3		
-5	65	65
-4	288	-286
-3	300	-297
-2	168	152
-1	111	-115
0	246	-249
1	222	202
2	350	332
3	332	-330
4	31*	-59
5	246	239
6	142	-133
7	288	-276
8	111	104
9	488	475
10	62	-66
11	89	-96
12	362	346
13	229	233
H,-12,3		
-4	38*	34
-3	110	-131
-2	401	-408
-1	202	-197
0	75	-76
1	33*	51
2	145	-143
3	96	-97
4	112	-109
5	431	-427
6	462	-469
7	201	-190
8	220	-213
9	198	-204
10	34*	2
11	152	-149
12	117	-118
13	118	-110
H,-13,3		
-2	72	-53
-1	37*	-24
0	567	563
1	217	217
2	196	-187
3	212	204
4	469	454
5	51	-43
6	265	-269
7	410	407
8	311	311
9	194	-176
10	36*	-5
11	391	400
12	38*	-46
H,-14,3		
0	449	465
1	512	513
2	140	-123
3	178	174
4	739	732
5	248	247
6	92	-82
7	433	427
8	650	652
9	36*	-1
10	38*	46
11	610	604
H,-15,3		
3	158	-153
4	116	116
5	82	81
6	146	-144
7	103	98
8	225	231
9	39*	-56
H,0,-3		
0	162	-177
1	294	-299
2	320	-338
3	918	-896
4	24*	-12
5	26*	-14
6	408	-405
7	45	-36
8	73	61
9	87	-86
10	282	-290
11	34*	-28
12	36*	28
13	143	-147
H,1,-3		
7	753	743
8	879	893
9	51	42
10	33*	-12
11	609	625
12	451	474
13	223	-224
H,-1,-3		
-7	29*	-22
-6	386	-391
-5	560	558
-4	379	387
-3	393	-392
-2	370	-388
-1	438	417
0	89	-96
1	300	-317
2	87	-84
3	953	935
4	589	-581
5	632	-621
6	112	116
7	46	-50
8	524	-523
9	232	-236
10	113	126
11	216	-226
12	230	-240
13	38*	7
H,-2,-3		
-12	467	480
-11	668	658
-10	78	-79
-9	213	-200
-8	776	775
-7	622	619
-6	318	-304
-5	1025	1027
-4	1609	1717
-3	300	298
-2	171	-183
-1	1076	1131
0	938	967
1	446	-474
2	174	198
3	1613	1663
4	603	567
5	292	-280
6	785	780
7	1041	1038
8	345	-345
9	155	147
10	700	712
11	282	296
12	310	-325
13	284	288
H,-3,-3		
-11	335	343
-10	128	-107
-9	364	-361
-8	315	302
-7	305	302
-6	286	-298
-5	75	66
-4	925	930
-3	255	247
-2	405	-417
-1	365	386
0	573	582
1	156	-152
2	79	-87
3	1213	1226
4	516	498
5	322	-317
6	464	463
7	931	923
8	164	155
9	72	-66
10	781	837
11	552	564
12	297	-308

TABLE II (*Continued*)

```
   H,-3,-3
13 98 102

   H,-4,-3
-10 86 -79
-9 103 -96
-8 277 -256
-7 102 -112
-6 314 -317
-5 123 -120
-4 362 -370
-3 358 -373
-2 327 -323
-1 275 -272
0 293 -291
1 346 -325
2 452 -460
3 76 -91
4 149 -164
5 348 -350
6 204 -206
7 185 174
8 167 -158
9 241 -235
10 173 -175
11 126 121
12 263 -261
13 198 -213

   H,-5,-3
-9 448 445
-8 103 112
-7 326 -324
-6 197 186
-5 316 307
-4 100 -113
-3 343 -351
-2 434 428
-1 444 435
0 626 -629
1 107 97
2 335 333
3 375 -379
4 473 -492
5 310 297
6 29* 46
7 455 -441
8 245 -239
9 175 180
10 211 -222
11 363 -380
12 62 -40
13 37* 45

   H,-6,-3
-8 408 407
-7 59 -53
-6 423 416
-5 1028 1029
-4 206 206
-3 166 -172
-2 727 746
-1 752 778
0 144 -151
1 206 201
2 1435 1484
3 458 451
4 366 -362
5 661 652
6 849 842
7 109 -97
8 141 -138
9 790 782
10 355 365
11 241 -242
12 256 251
13 559 585

   H,-7,-3
-7 168 -174
-6 93 71
-5 587 593
-4 193 165
-3 246 -249
-2 128 138
-1 543 546
0 243 -245
1 105 -93
2 558 547
3 613 596
4 260 -258
5 263 266
6 849 845
7 143 159
8 144 -129
9 667 668
10 498 513
11 210 -220
12 116 106
13 580 593

   H,-8,-3
-6 106 -77
-5 175 -182
-4 183 -181
-3 211 -198
-2 240 -238
-1 361 -360
0 161 -142
1 231 -229
2 73 -95
3 110 -117
4 247 -265
5 206 -195
6 125 -108
7 33* -51
8 289 -282
9 76 55
10 35* 40
11 248 -243
12 219 -230

   H,-9,-3
-5 66 -59
-4 313 -323
-3 398 388
-2 250 237
-1 364 -354
0 33* -27
1 338 337
2 231 -230
3 304 -316
4 111 116
5 145 145
6 192 -191
7 193 -190
8 191 199
9 183 -179
10 407 -402
11 36* -59
12 38* 2

   H,-10,-3
-3 412 427
-2 653 670
-1 36* -63
0 128 142
1 768 817
2 285 281
3 147 -144
4 396 399
5 726 723
6 80 -70
7 99 -94
8 559 554
9 423 435
10 251 -256
11 208 215

   H,-11,-3
-1 59 -70
0 130 -121
1 421 414
2 397 400
3 179 -184
4 110 108
5 532 532
6 119 119
7 157 -155
8 419 416
9 377 405

10 106 -109

   H,-12,-3
2 102 -95
3 115 -123
4 177 -182
5 92 -31
6 75 -83
7 111 -124

   H,0,4
1 462 -455
2 72 0
3 611 601
4 91 71
5 507 -512
6 206 203
7 264 200
8 324 -323
9 133 -126
10 56 60
11 82 57
12 243 -243

   H,1,4
8 116 122
9 78 -82
10 293 -292
11 105 -99

   H,-1,4
-5 351 -334
-4 1280 1297
-3 723 704
-2 318 -326
-1 1144 1134
0 1821 1900
1 73 -56
2 55 21
3 1342 1400
4 845 643
5 353 -367
6 482 480
7 1001 1017
8 272 278
9 163 -215
10 680 669
11 580 569
12 37* -49

   H,-2,4
-12 250 -266
-11 146 149
-10 35* -1
-9 245 -240
-8 119 100
-7 374 370
-6 322 -324
-5 522 -519
-4 168 179
-3 553 532
-2 491 -502
-1 45 -44
0 1309 1320
1 195 -197
2 455 -451
3 549 526
4 746 734
5 231 -243
6 28* -11
7 616 630
8 160 165
9 374 -378
10 379 393
11 747 745
12 37* 51
13 38* 32

   H,-3,4
-11 242 -234
-10 164 -156
-9 149 -149
-8 107 -115
-7 334 -346
-6 42 -18
-5 203 -201
-4 284 -276
-3 284 -295
-2 176 -180
-1 509 -501
0 577 -574
1 388 -390
2 603 -604
3 264 -254
4 179 -178
5 430 -435
6 259 -263
7 95 -98
8 180 -187
9 498 -490
10 84 -93
11 33* 34
12 138 -136
13 161 -153

   H,-4,4
-11 70 56
-10 128 -132
-9 554 577
-8 507 512
-7 218 -210
-6 67 46
-5 826 827
-4 54 42
-3 624 -621
-2 537 514
-1 607 602
0 72 42
1 335 -331
2 862 885
3 48 -37
4 470 -476
5 46 32
6 387 387
7 203 -202
8 206 -208
9 210 195
10 136 123
11 355 -348
12 162 -158
13 137 140
14 113 -112

   H,-5,4
-10 76 -76
-9 364 380
-8 847 867
-7 93 -95
-6 178 -182
-5 987 997
-4 649 643
-3 230 -246
-2 750 738
-1 1399 1452
0 26* 39
1 115 95
2 1174 1199
3 1090 1082
4 189 -190
5 390 393
6 1380 1416
7 205 199
8 106 -97
9 612 611
10 824 838
11 127 -119
12 58 -27
13 616 604
14 305 305

   H,-6,4
-9 37* -1
-8 139 146
-7 143 -158
-6 510 -523
-5 62 68
-4 351 354
-3 473 -482
-2 192 176
-1 777 741
0 202 160

1 557 -543
2 533 534
3 580 572
4 278 -284
5 303 -311
6 656 640
7 375 371
8 419 -422
9 392 386
10 785 788
11 68 69
12 34* -24
13 501 496
14 320 310

   H,-7,4
-9 70 -45
-8 177 -195
-7 155 -151
-6 71 -65
-5 311 -320
-4 193 -202
-3 72 -79
-2 416 -412
-1 203 -206
0 359 -350
1 492 -491
2 264 -265
3 321 -296
4 204 -195
5 286 -287
6 182 -173
7 539 -548
8 301 -302
9 298 -299
10 78 76
11 94 -92
12 109 -109
13 36* 6
14 63 -47

   H,-8,4
-8 167 -174
-7 86 85
-6 591 615
-5 131 136
-4 262 -269
-3 361 369
-2 609 615
-1 292 -291
0 190 -191
1 511 497
2 329 314
3 431 -427
4 35* -16
5 565 565
6 157 -154
7 305 -311
8 215 212
9 192 181
10 284 -290
11 122 -124
12 211 206
13 147 -158
14 251 -236

   H,-9,4
-7 108 -105
-6 686 712
-5 410 411
-4 225 -229
-3 334 337
-2 876 894
-1 391 384
0 30* -50
1 840 852
2 965 968
3 195 -193
4 301 289
5 1058 1040
6 475 479
7 181 -178
8 678 675
9 769 770
10 32* -28
11 80 -86
12 677 674
13 357 362
14 222 -218

   H,-10,4
-6 38* 7
-5 155 165
-4 162 -173
-3 34* -36
-2 411 413
-1 32* 40
0 271 -264
1 106 102
2 511 495
3 196 -201
4 240 -232
5 653 630
6 259 259
7 313 -315
8 269 264
9 616 609
10 93 87
11 206 -206
12 470 446
13 337 340

   H,-11,4
-5 154 -164
-4 105 -106
-3 108 -100
-2 155 -154
-1 177 -171
0 234 -240
1 119 -116
2 250 -249
3 273 -263
4 236 -229
5 416 -402
6 266 -261
7 279 -274
8 199 -194
9 33* -31
10 34* -13
11 136 -137
12 54 -46
13 86 -76

   H,-12,4
-3 335 344
-2 71 -57
-1 195 -207
0 331 333
1 234 224
2 272 -272
3 84 72
4 527 532
5 58 -58
6 394 -385
7 174 173
8 152 141
9 218 -217
10 35* -16
11 187 178
12 38* 30

   H,-13,4
-2 211 217
-1 85 -85
0 477 484
1 692 701
2 35* -31
3 91 53
4 783 787
5 334 336
6 95 -84
7 416 407
8 718 723
9 36* 43
10 36* -8
11 550 548
12 322 320

   H,-14,4
0 38* 21
1 292 309

2 115 -129
3 152 -132
4 334 326
5 247 224
6 162 -161
7 158 147
8 388 371
9 37* 22
10 177 -175

   H,-15,4
4 193 -163
5 196 -193
6 144 -148
7 161 -163
8 65 -33

   H,0,-4
0 672 665
1 409 400
2 391 -373
3 42 1
4 732 699
5 28* -2
6 341 -331
7 590 582
8 688 703
9 33* -43
10 34* 41
11 535 543
12 265 286

   H,1,-4
6 43 -17
7 723 728
8 771 748
9 32* -4
10 301 292
11 740 749
12 75 73

   H,-1,-4
-7 426 -429
-6 277 -286
-5 160 -166
-4 358 -355
-3 401 -424
-2 426 -418
-1 148 -139
0 269 -263
1 447 -440
2 419 -404
3 494 -477
4 195 -193
5 233 -240
6 204 -204
7 286 -274
8 52 -51
9 216 -203
10 267 -255
11 67 -63
12 37* -35

   H,-2,-4
-11 351 372
-10 314 -312
-9 107 115
-8 605 613
-7 121 115
-6 198 -211
-5 479 473
-4 816 817
-3 495 -498
-2 27* -1
-1 658 662
0 65 63
1 623 -623
2 595 560
3 457 439
4 262 -278
5 194 -194
6 461 455
7 173 163
8 477 -477
9 115 -117
10 298 288
11 309 -316
12 274 -270
13 122 129

   H,-3,-4
-10 94 -85
-9 36* 15
-8 689 678
-7 380 381
-6 218 -216
-5 562 560
-4 1001 1014
-3 59 37
-2 146 -146
-1 1563 1631
0 926 939
1 247 -248
2 791 777
3 945 947
4 45 28
5 337 -333
6 823 805
7 980 960
8 83 100
9 106 106
10 926 918
11 130 135
12 339 -346
13 399 387

   H,-4,-4
-9 200 -211
-8 151 159
-7 92 91
-6 284 -303
-5 123 -118
-4 489 487
-3 176 -191
-2 254 -250
-1 351 348
0 552 541
1 306 -294
2 78 -77
3 521 505
4 35* 2
5 264 -265
6 258 253
7 674 677
8 111 98
9 49 -38
10 618 630
11 249 248
12 299 -308
13 157 157

   H,-5,-4
-8 256 -261
-7 173 -167
-6 136 -130
-5 161 -156
-4 295 -300
-3 226 -215
-2 59 -61
-1 220 -228
0 413 -406
1 270 -275
2 156 -156
3 517 -511
4 216 -212
5 368 -366
6 87 -73
7 125 -120
8 63 -76
9 213 -197
10 100 -86
11 105 -107
12 202 -195

   H,-6,-4
-7 206 -202
-6 372 380
-5 481 472
-4 206 -210
-3 173 -155
-2 608 605

-1 198 197
0 419 -408
1 222 214
2 426 426
3 83 -97
4 224 -231
5 475 478
6 214 206
7 458 -453
8 87 -78
9 249 237
10 190 -187
11 319 -320
12 212 228

   H,-7,-4
-6 287 288
-5 730 745
-4 72 68
-3 35* -39
-2 826 836
-1 520 527
0 95 -98
1 339 341
2 873 888
3 214 203
4 82 -92
5 584 584
6 949 943
7 135 -125
8 34* 24
9 844 836
10 169 162
11 269 -266
12 357 361

   H,-8,-4
-5 142 141
-4 60 -74
-3 237 -234
-2 273 271
-1 355 353
0 99 -107
1 61 -31
2 253 245
3 33* 15
4 345 -340
5 81 57
6 623 617
7 120 117
8 38* -33
9 447 450
10 270 269
11 295 -306

   H,-9,-4
-3 38* 16
-2 154 -151
-1 243 -248
0 97 -98
1 188 -181
2 329 -326
3 262 -263
4 128 -124
5 158 -138
6 116 -124
7 63 -61
8 99 -86
9 85 -99
10 156 -149

   H,-10,-4
-1 273 -279
0 121 106
1 401 417
2 113 -108
3 245 -244
4 278 270
5 210 214
6 248 -241
7 62 -38
8 305 304
9 107 -108

   H,-11,-4
3 106 -88
4 485 494
5 623 628
6 96 -84

   H,0,5
2 314 -324
3 864 871
4 624 625
5 285 -284
6 272 277
7 752 763
8 323 332
9 134 -126
10 348 328
11 453 448

   H,1,5
8 158 -171
9 216 -215
10 105 -93

   H,-1,5
-5 586 -585
-4 271 266
-3 770 750
-2 190 -193
-1 170 165
0 1341 1353
1 289 278
2 579 -577
3 511 504
4 344 358
5 83 -68
6 30* -23
7 957 984
8 540 557
9 87 -84
10 293 288
11 669 685

   H,-2,5
-11 200 -189
-10 90 -110
-9 142 -140
-8 183 -171
-7 157 -155
-6 78 -98
-5 235 -221
-4 514 -505
-3 130 -142
-2 563 -543
-1 495 -475
0 27* 10
1 161 -154
2 552 -546
3 108 -115
4 53 -26
5 415 -432
6 279 -278
7 31* 9
8 143 142
9 313 -307
10 222 -227
11 224 213
12 123 -118

   H,-3,5
-10 255 -260
-9 354 351
-8 248 234
-7 208 -206
-6 114 -97
-5 442 443
-4 169 165
-3 415 -400
-2 98 89
-1 376 366
0 361 -337
1 185 -201
2 140 126
3 95 89
4 467 -476
5 28* 0
6 43 48
7 110 -117

8 413 -400
9 33* -9
10 48 -2
11 315 -316
12 207 -195

   H,-4,5
-10 149 -154
-9 365 369
-8 767 774
-7 34* 44
-6 77 -78
-5 843 847
-4 707 713
-3 87 -92
-2 384 359
-1 1448 1497
0 182 175
1 144 -157
2 1131 1137
3 924 918
4 178 -182
5 129 139
6 868 873
7 444 444
8 279 -273
9 397 401
10 691 693
11 35* 9
12 151 -166
13 521 524

   H,-5,5
-9 79 3
-8 374 380
-7 65 16
-6 443 -456
-5 215 216
-4 649 639
-3 305 -316
-2 155 135
-1 1119 1118
0 385 359
1 461 -447
2 160 161
3 633 620
4 161 -159
5 31* -12
6 812 815
7 848 850
8 153 -151
9 256 254
10 805 816
11 52 35
12 167 -168
13 529 535

   H,-6,5
-9 91 -97
-8 136 -132
-7 55 -56
-6 386 -390
-5 387 -394
-4 196 -201
-3 163 -154
-2 350 -350
-1 174 164
0 274 -272
1 438 -433
2 357 -366
3 433 -417
4 320 -312
5 351 -315
6 29* -22
7 241 222
8 349 -351
9 178 -178
10 160 168
11 134 -123
12 334 -338
13 117 107

   H,-7,5
-8 106 -114
-7 71 -58
-6 463 479
-5 108 94
-4 310 -318
-3 32* -12
-2 364 364
-1 207 -202
0 322 -332
1 278 266
2 139 159
3 296 -298
4 263 -268
5 349 354
6 231 -232
7 271 -268
8 66 -25
9 33* -60
10 344 -334
11 223 -217
12 36* -23
13 84 -51

   H,-8,5
-7 61 -73
-6 510 532
-5 689 717
-4 149 -157
-3 273 268
-2 956 997
-1 336 342
0 104 -97
1 751 763
2 871 876
3 76 -87
4 154 152
5 858 871
6 512 517
7 273 -270
8 362 360
9 838 850
10 69 -71
11 102 -106
12 492 489
13 321 321

   H,-9,5
-6 143 153
-5 324 323
-4 89 -93
-3 62 -58
-2 568 579
-1 272 282
0 371 -363
1 279 285
2 604 602
3 109 114
4 300 -301
5 698 697
6 595 600
7 200 -186
8 133 131
9 812 833
10 155 150
11 164 -166
12 501 510
13 464 466

   H,-10,5
-5 106 -97
-4 179 -169
-3 76 -83
-2 34* 20
-1 108 -117
0 251 -251
1 478 -477
2 347 -334
3 306 -300
4 299 -304
5 71 -64
6 179 173
7 172 -172
8 204 -205
9 141 130
10 234 -239
11 255 -246
12 37* -27
13 89 82

   H,-11,5
-4 79 76
-3 317 319
-2 36* -26
-1 213 -221
0 149 156
1 214 215
2 446 -455
3 99 -95
4 283 278
5 77 -65
6 255 -246
7 33* -12
8 33* -9
9 222 -216
10 223 -216
11 37* -33
12 38* -12

   H,-12,5
-3 623 641
-2 267 274
-1 112 -112
0 433 440
1 682 687
2 108 95
3 34* -7
4 734 724
5 476 476
6 180 -177
7 362 359
8 608 588
9 55 41
10 149 -147
11 463 455
12 336 338

   H,-13,5
-1 147 -150
0 88 94
1 470 476
2 36* 17
3 151 -149
4 410 403
5 420 426
6 189 -179
7 94 76
8 637 631
9 159 149
10 111 -108
11 381 379

   H,-14,5
1 161 -170
2 134 -132
3 251 -252
4 84 -74
5 57 19
6 174 -168
7 140 -136
8 38* -20
9 85 -83

   H,0,-5
-1 352 345
0 1420 1460
1 801 772
2 395 -381
3 864 840
4 893 868
5 34* 27
6 87 -75
7 962 932
8 614 608
9 200 -186
10 331 306
11 676 647

   H,1,-5
6 78 -29
7 593 577
8 94 92
9 236 -219
10 172 149
11 287 283

   H,-1,-5
-7 33* 16
-6 179 -180
-5 297 -309
-4 150 -146
-3 298 300
-2 160 -172
-1 417 -413
0 371 355
1 97 78
2 260 -260
3 29* -12
4 343 334
5 73 -48
6 367 -351
7 263 252
8 312 309
9 195 -185
10 36* -4
11 524 526

   H,-2,-5
-10 242 -241
-9 37* 17
-8 75 105
-7 258 -267
-6 252 -262
-5 172 181
-4 122 -122
-3 463 -471
-2 167 -160
-1 97 97
0 618 -619
1 303 -302
2 281 -281
3 125 -123
4 379 -354
5 189 -192
6 122 -118
7 250 -236
8 243 -241
9 185 -182
10 194 -169
11 145 -132

   H,-3,-5
-9 370 365
-8 631 687
-7 102 93
-6 251 -243
-5 507 519
-4 568 563
-3 335 -334
-2 232 223
-1 1046 1044
0 144 137
1 346 -340
2 510 497
3 372 364
4 318 -319
5 62 50
6 550 540
7 293 291
8 405 -401
9 260 261
10 345 328
11 241 -229

   H,-4,-5
-8 617 633
-7 226 232
-6 191 -201
-5 289 298
-4 524 529
-3 153 -158
-2 80 62
-1 1141 1151
0 589 583
1 31* -39
2 593 588
3 866 872
4 97 -86
5 151 -137
6 882 864
7 671 665

8 140 -122
9 247 239
10 741 719
11 114 99

   H,-5,-5
-7 38* -41
-6 261 -278
-5 132 -124
-4 82 65
-3 159 -171
-2 213 -211
-1 283 275
0 180 165
1 269 -284
2 162 -157
3 218 217
4 88 -89
5 234 -235
6 142 142
7 405 404
8 208 -202
9 36* -3
10 305 295
11 106 96

   H,-6,-5
-6 150 158
-5 99 -131
-4 281 -278
-3 35* -30
-2 50 -48
-1 253 -262
0 320 -332
1 33* -44
2 64 -52
3 308 -302
4 255 -257
5 59 -44
6 287 -278
7 322 -330
8 135 -132
9 88 -77
10 137 -127

   H,-7,-5
-4 190 -185
-3 128 130
-2 665 671
-1 78 75
0 239 -236
1 254 249
2 474 470
3 130 -104
4 113 -120
5 556 571
6 250 248
7 296 -293
8 133 131
9 351 350
10 183 -181

   H,-8,-5
-2 618 633
-1 500 496
0 120 -115
1 320 326
2 554 556
3 82 71
4 57 -65
5 628 620
6 556 556
7 93 -92
8 183 184
9 660 651

   H,-9,-5
0 103 -102
1 74 -79
2 134 116
3 96 -99
4 247 -246
5 125 125
6 234 226
7 38* -41

   H,0,6
2 253 -247
3 394 400
4 827 845
5 32* -1
6 34* -9
7 811 827
8 639 656
9 111 -101
10 217 219

   H,1,6
8 229 225
9 160 -167

   H,-1,6
-5 409 -397
-4 254 -238
-3 31* 22
-2 405 -402
-1 385 -378
0 261 255
1 73 65
2 293 -302
3 320 -314
4 221 230
5 241 -237
6 379 -378
7 161 167
8 335 345
9 140 -145
10 85 -93

   H,-2,6
-10 176 -199
-9 62 71
-8 177 196
-7 266 -283
-6 115 -92
-5 72 92
-4 50 42
-3 299 -302
-2 30* -10
-1 68 -20
0 314 -313
1 267 -265
2 58 -69
3 43 2
4 296 -298
5 237 -238
6 54 -65
7 233 -237
8 301 -310
9 175 -171
10 75 -85
11 172 -176

   H,-3,6
-9 231 240
-8 637 636
-7 89 81
-6 34* -41
-5 782 784
-4 763 769
-3 94 -91
-2 251 237
-1 925 916
0 377 368
1 443 -443
2 693 681
3 656 676
4 74 38
5 130 -143
6 660 667
7 356 357
8 269 -273
9 193 185
10 472 492
11 71 -61

   H,-4,6
-9 117 -109
-8 568 611
-7 187 176
-6 155 -152

-5 394 382
-4 714 698
-3 31* -60
-2 186 -183
-1 813 829
0 583 580
1 276 -275
2 271 271
3 930 944
4 109 115
5 57 -42
6 663 668
7 695 702
8 33* -31
9 123 121
10 823 806
11 117 118

   H,-5,6
-8 88 -85
-7 36* 7
-6 307 -305
-5 193 -190
-4 32* -13
-3 284 -274
-2 276 -269
-1 105 -109
0 81 78
1 463 -468
2 225 -239
3 30* 20
4 135 -136
5 443 -434
6 209 209
7 443 448
8 181 -186
9 185 -175
10 182 186
11 37* -63
12 352 -366

   H,-6,6
-8 146 -148
-7 157 -154
-6 167 175
-5 53 -26
-4 275 -261
-3 198 -196
-2 123 110
-1 32* -37
0 277 -271
1 111 98
2 30* -7
3 455 -453
4 233 -246
5 157 -151
6 200 -180
7 298 -294
8 33* -19
9 176 -185
10 183 -172
11 301 -310
12 164 -160

   H,-7,6
-7 63 -47
-6 458 472
-5 668 700
-4 34* 17
-3 139 118
-2 874 867
-1 261 258
0 238 -250
1 464 453
2 867 872
3 45 -31
4 31* -23
5 719 731
6 404 393
7 304 -297
8 130 135
9 554 563
10 35* 25
11 148 -146
12 296 295

   H,-8,6
-6 223 231
-5 528 538
-4 36* 47
-3 198 -199
-2 610 618
-1 545 519
0 325 -330
1 276 278
2 847 843
3 197 199
4 119 -105
5 555 542
6 798 810
7 120 -119
8 161 154
9 789 799
10 304 309
11 124 -128
12 459 474

   H,-9,6
-5 38* -31
-4 126 -122
-3 331 -322
-2 120 -109
-1 51 -25
0 255 -246
1 388 -390
2 32* 26
3 117 -133
4 261 -258
5 101 104
6 300 303
7 168 -169
8 241 -240
9 155 161
10 54 -45
11 364 -368
12 92 93

   H,-10,6
-4 124 -131
-3 220 241
-2 72 -68
-1 237 -237
0 62 50
1 34* 37
2 393 -404
3 239 -241
4 59 -52
5 142 -136
6 83 -68
7 116 -112
8 62 -44
9 239 -243
10 335 -338
11 223 -233

   H,-11,6
-3 520 543
-2 385 392
-1 149 -162
0 404 429
1 632 644
2 34* 33
3 64 -63
4 543 536
5 385 376
6 159 -152
7 149 156
8 513 500
9 79 77
10 202 -208
11 222 226

   H,-12,6
-1 175 -169
0 97 94
1 577 582
2 210 206
3 127 -117
4 463 459
5 590 589
6 36* -43

7 36* -13
8 693 710
9 294 294
10 73 -56

   H,-13,6
1 38* 5
2 72 -40
3 209 -197
4 100 -96
5 142 140
6 147 -152
7 189 -188
8 110 108
9 60 9

   H,-14,6
4 38* 52
5 95 -109
6 39* -45

   H,0,-6
-1 442 431
0 834 831
1 30* -33
2 78 -76
3 631 615
4 715 696
5 332 -325
6 306 295
7 695 686
8 88 -92
9 325 -319
10 370 351

   H,1,-6
6 114 -111
7 52 -41
8 325 -307
9 125 -136
10 38* -4

   H,-1,-6
-7 495 492
-6 35* 26
-5 116 -115
-4 509 528
-3 405 411
-2 167 -165
-1 185 187
0 910 904
1 218 217
2 185 -175
3 647 638
4 838 821
5 32* -7
6 35* 56
7 867 837
8 319 319
9 327 -320
10 368 361

   H,-2,-6
-8 78 -71
-7 146 -141
-6 195 -217
-5 133 -126
-4 122 -115
-3 82 -79
-2 249 -245
-1 359 -357
0 73 -23
1 77 -53
2 247 -238
3 73 -52
4 270 278
5 153 -136
6 237 -225
7 34* 0
8 36* 27
9 242 -233
10 39* -33

   H,-3,-6
-7 263 -276
-6 129 -121
-5 199 208
-4 35* -20
-3 353 -363
-2 161 160
-1 108 100
0 318 -325
1 312 -313
2 152 151
3 123 -123
4 482 -457
5 161 -157
6 34* 18
7 310 -288
8 195 -188
9 38* -25
10 38* -25

   H,-4,-6
-6 64 -58
-5 616 626
-4 302 305
-3 147 -142
-2 301 293
-1 836 825
0 163 164
1 253 -253
2 583 585
3 551 547
4 237 -226
5 123 116
6 704 691
7 36* -36
8 192 -186
9 345 326

   H,-5,-6
-5 241 249
-4 403 404
-3 122 -127
-2 103 94
-1 521 530
0 242 225
1 184 -182
2 413 409
3 750 744
4 35* 2
5 100 95
6 745 741
7 251 238
8 224 -220
9 260 255

   H,-6,-6
-3 150 -136
-2 227 -221
-1 37* -26
0 54 -27
1 255 -256
2 36* 16
3 80 66
4 36* -50
5 229 -225
6 38* 57
7 37* -4
8 215 -211

   H,-7,-6
-1 226 -235
0 227 -227
1 122 119
2 37* -25
3 236 -240
4 68 -82
5 77 77
6 269 -266
7 307 -307

   H,0,7
2 213 -215
3 146 -147
4 381 389
5 186 195
6 371 -377
7 211 207
8 269 282

   H,-1,7
-5 61 -74
-4 168 -163
-3 127 -128
-2 147 -150
-1 33* -9
0 258 -265
1 258 -266
2 151 -157
3 189 -184
4 230 -229
5 209 -212
6 237 -238
7 186 -195
8 94 -94

   H,-2,7
-8 544 564
-7 216 217
-6 172 -173
-5 560 571
-4 572 575
-3 145 -137
-2 54 17
-1 560 550
0 236 237
1 230 -231
2 133 128
3 572 575
4 48 -34
5 98 -93
6 360 362
7 235 238
8 279 -281
9 60 57

   H,-3,7
-8 530 542
-7 362 369
-6 142 -152
-5 406 406
-4 727 746
-3 113 101
-2 182 -178
-1 644 649
0 497 499
1 33* -46
2 262 252
3 1010 1019
4 347 350
5 125 -127
6 656 655
7 658 674
8 154 -162
9 140 132

   H,-4,7
-7 110 83
-6 194 -186
-5 35* 13
-4 190 177
-3 200 -206
-2 420 -414
-1 176 -179
0 210 204
1 187 -186
2 168 -162
3 596 598
4 157 160
5 388 -389
6 71 66
7 369 380
8 205 -208
9 125 -138
10 285 294

   H,-5,7
-7 166 -178
-6 38* -57
-5 55 23
-4 209 -216
-3 92 -89
-2 73 -75
-1 175 -178
0 322 -320
1 32* -21
2 284 -284
3 264 -258
4 181 -177
5 286 -291
6 192 -190
7 160 -174
8 228 -226
9 94 -98
10 115 -125

   H,-6,7
-6 352 385
-5 464 474
-4 36* -3
-3 35* -2
-2 622 629
-1 305 305
0 283 -284
1 237 222
2 605 620
3 32* 34
4 240 -244
5 451 456
6 200 199
7 177 -174
8 35* -39
9 337 349
10 60 41

   H,-7,7
-5 638 687
-4 96 81
-3 119 -113
-2 458 467
-1 634 634
0 120 -128
1 111 109
2 876 897
3 361 368
4 86 -69
5 615 614
6 738 765
7 55 -51
8 102 110
9 650 659
10 286 294

   H,-8,7
-5 39* 49
-4 37* -28
-3 389 -394
-2 163 -166
-1 153 141
0 205 -198
1 161 -139
2 342 341
3 210 217
4 372 -377
5 139 134
6 250 251
7 138 -152
8 238 -238
9 280 293
10 212 207

   H,-9,7
-4 145 -167
-3 37* -15
-2 271 -285
-1 247 -254
0 70 -56
1 125 -119
2 88 -75
3 159 -164
4 222 -221
5 213 -203
6 219 -228
7 211 -214
8 164 -156
9 156 -164
10 225 -231

   H,-10,7
-3 421 463
-2 324 350

-1 153 -163
0 216 222
1 508 534
2 61 -63
3 117 -118
4 273 264
5 319 319
6 154 -158
7 54 -17
8 400 401
9 109 123
10 199 -208

   H,-11,7
-1 86 -105
0 38* 41
1 657 682
2 347 354
3 110 -96
4 444 451
5 612 628
6 53 45
7 38* 43
8 531 535
9 421 451

   H,-12,7
1 143 155
2 64 79
3 174 -175
4 37* -22
5 234 243
6 71 -83
7 169 -171
8 176 185

   H,-13,7
5 230 -234

   H,0,-7
-1 210 204
0 82 62
1 341 -336
2 189 -190
3 322 318
4 53 6
5 371 -359
6 57 35
7 65 68
8 420 -408

   H,1,-7
6 181 -176
7 231 -222
8 59 17

   H,-1,-7
-7 469 468
-6 106 -112
-5 231 237
-4 898 909
-3 424 432
-2 95 -89
-1 499 494
0 534 531
1 96 -60
2 177 -167
3 805 790
4 545 536
5 80 -78
6 366 353
7 604 590
8 137 -133

   H,-2,-7
-6 90 -99
-5 37* 10
-4 364 366
-3 176 185
-2 146 -143
-1 57 21
0 428 422
1 34* 20
2 128 -122
3 523 518
4 537 519
5 94 -93
6 144 139
7 603 580
8 84 79

   H,-3,-7
-5 75 -83
-4 157 -158
-3 184 -183
-2 222 -234
-1 104 -92
0 212 -209
1 105 -97
2 236 -228
3 131 -129
4 55 -39
5 116 -112
6 183 -178
7 38* -4

   H,-4,-7
-3 209 -212
-2 128 139
-1 160 164
0 206 -200
1 117 -114
2 359 369
3 37* -13
4 423 -424
5 97 77
6 38* 0
7 278 -272

   H,-5,-7
-1 545 546
0 38* -41
1 92 -99
2 621 626
3 517 515
4 132 -126
5 237 240

   H,0,8
2 195 -200
3 292 -293
4 39* -42
5 76 -73

   H,-1,8
-4 336 333
-3 165 -165
-2 80 -83
-1 305 316
0 234 237
1 161 -159
2 64 26
3 266 259
4 152 -138
5 139 -135
6 160 153

   H,-2,8
-5 188 197
-4 696 703
-3 142 135
-2 81 -69
-1 517 532
0 585 597
1 85 -80
2 254 253
3 753 765
4 400 405
5 174 -187
6 450 455

   H,-3,8
-5 165 -159
-4 348 366
-3 37* 42
-2 373 -362
-1 52 78
0 369 368
1 72 -70
2 116 -106
3 512 524

4 340 344
5 269 -271
6 37* 25
7 317 335

   H,-4,8
-5 100 -85
-4 125 -136
-3 74 -60
-2 153 -149
-1 361 -364
0 190 -196
1 240 -243
2 207 -210
3 99 -102
4 38* 3
5 256 -257
6 178 -189
7 135 -133
8 243 -256

   H,-5,8
-5 336 363
-4 38* -45
-3 115 -120
-2 403 424
-1 223 224
0 225 -233
1 36* 24
2 277 278
3 35* -38
4 156 -162
5 122 102
6 178 194
7 167 -156
8 80 -66

   H,-6,8
-4 141 124
-3 112 -112
-2 464 479
-1 615 632
0 98 95
1 85 82
2 754 778
3 336 347
4 100 -94
5 437 445
6 640 652
7 37* 31
8 38* 21

   H,-7,8
-3 261 -268
-2 37* 35
-1 277 275
0 75 -67
1 140 -138
2 404 403
3 349 359
4 265 -278
5 36* 11
6 422 437
7 38* -66
8 111 -111

   H,-8,8
-2 256 -272
-1 183 -196
0 95 -90
1 207 -201
2 36* -4
3 77 -79
4 205 -217
5 300 -300
6 232 -230
7 259 -272
8 199 -197

   H,-9,8
-1 180 -195
0 38* -31
1 294 299
2 37* 52
3 142 -133
4 135 141
5 216 240
6 163 -164
7 206 -216
8 209 203

   H,-10,8
0 39* 68
1 538 561
2 412 416
3 103 -102
4 316 314
5 600 609
6 113 135
7 38* 14

   H,-11,8
3 191 -195
4 72 54
5 334 345

   H,0,-8
-1 204 -208
0 204 -199
1 183 -170
2 121 -114
3 131 -108
4 161 -156
5 99 -96

   H,1,-8
5 132 116

   H,-1,-8
-3 39* -32
-2 128 -129
-1 287 295
0 119 124
1 348 -353
2 159 150
3 338 341
4 38* -40
5 244 -242

   H,-2,-8
-2 38* -43
-1 470 469
0 528 531
1 96 -90
2 93 79
3 686 684
4 266 270

   H,-3,9
0 39* 3
1 203 -211
2 260 -251

   H,-4,9
0 159 -171
1 67 -40
2 66 44
3 38* -12

   H,-5,9
0 79 72
1 113 100
2 520 526
3 289 293

   H,-6,9
1 126 -132
2 320 334
3 332 338
```

---

[a] The columns are, respectively, h, $10sF_o$, and $10F_c$, where $s = 0.51007$. An asterisk designated an unobservable reflection which has been assigned the estimated minimum observable F_o.

cell dimensions and a wavelength of 0.710688 Å. Background readings were taken at $2\theta - {}^1/_2\Delta 2\theta$ and at $2\theta + {}^1/_2\Delta 2\theta$ where $\Delta 2\theta = 1.80 + 1.0 \tan \theta$ is the scan range recommended by Alexander and Smith.[16] The counting time was 10 sec for a peak and each background. Two standard reflections were measured every 4 hr. The constancy of the standard reflection intensity indicated no deterioration of the crystal during the period of data collection. The scatter of the standards was within ±1% for a 13,000-cps peak and ±2% for a 3000-cps peak from their mean values.

To convert the observed peak intensities (I_p) to integrated intensities (I_i), 21 strong reflections spanning the 2θ range were measured by the 2θ scan method. A curve of I_i/I_p *vs.* 2θ was prepared and utilized to obtain the conversion for each reflection.

A total of 3976 independent lattice points were measured resulting in a final set of data consisting of 3683 observed reflections ($I_p > 2\sigma(I)$) and 293 unobserved reflections. The unobserved reflections were assigned intensities equal to $2\sigma(I)$ where $\sigma(I) = [P + (B_L + B_H)/4]^{1/2}$. P, B_L, and B_H are the counts at the peak, low-background, and high-background positions, respectively.

The intensity data were corrected for Lorentz and polarization factors but not for absorption. Based on a calculated linear absorption coefficient of 32.13 cm^{-1} the maximum error due to absorption for the crystal size and shape used would be about 4% of the intensities, which was considered negligible.

Structure Determination and Refinement

The structure was readily solved by conventional Patterson and heavy-atom techniques. The assumption of $P\bar{1}$ symmetry and the limitation of one unit of $Na_3(CrMo_6O_{24}H_6) \cdot 8H_2O$ per unit cell require that the Cr atom and at least one Na atom must lie on centers of symmetry. Therefore, the Cr atom was placed at the origin. Approximate coordinates of the Mo atoms were readily derived from a three-dimensional Patterson synthesis. A structure factor calculation based on the Cr and Mo atoms supplied enough correct phases to compute a three dimensional electron density map which revealed all of the Na and O atoms. The structure was then refined by full-matrix least-squares techniques. The initial refinement using individual isotropic temperature factors converged to a conventional R value of 0.060 (computed for observed data only). A difference synthesis at this point gave no indication of misplaced atoms, but there were suggestions of appreciable anisotropic motion for some of the atoms. Consequently, the refinement was continued using individual anisotropic temperature factors for all atoms to a final R value of 0.033 (observed data only).

The least-squares program minimized the quantity $\Sigma w(|F_o| - |F_c|)^2$. Throughout the refinement the observed reflections were weighted as $w = 25.5/F_o$ for $F_o > 25.5$ and $w = 1.0$ for $F_o \leq 25.5$ where F_o is the scaled F_o obtainable from Table II. The values quoted in Table II are 10 times the scaled F_o. The unobserved reflections were weighted as $w = 1.0$ if $F_c > F(\min)$ and $w = 0.0$ if $F_c \leq F(\min)$ where $F(\min)$ is the structure factor derived from the assigned threshold intensity. All parameter shifts in the final cycle were less than the standard error. The atomic scattering factors used were O^-, Na^+, neutral Cr, and neutral Mo.[17]

An electron density difference map was computed based on the final anistropic parameters. It was essentially zero everywhere except for small areas of negative electron density (−2 electrons/Å^3) at the Cr and Mo positions which could indicate that these atoms should have been chosen to have some positive charge.

An examination of the final list of structure factors reveals that the very strong reflections all tend to have F_o lower than F_c which could indicate extinction effects. However, since these reflections received very small weights in the refinement, the application of an extinction correction would have negligible effects on the parameters. Therefore, the refinement was terminated at this point.

Results and Discussion

The final parameters resulting from the anisotropic least-squares refinement are listed in Table III (positional) and Table IV (thermal). The oxygen atoms have been numbered so that chemically similar atoms are grouped together. Anion oxygen atoms are O(1) through O(12) and the water molecules are O(13) through O(16). The anion oxygens are grouped so that O(1)–O(3) are each coordinated to one Cr and two Mo atoms, O(4)–O(6) are each coordinated to two Mo atoms, and O(7)–O(12) are each coordinated to

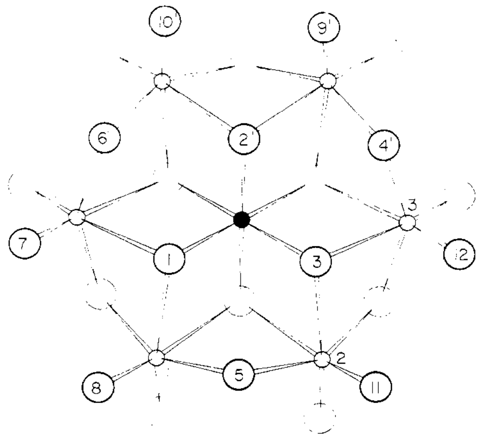

Figure 2.—View of the anion as projected onto the least-squares best plane fitted to the Cr and Mo atoms. Large circles are oxygen atoms, small open circles are molybdenum atoms, and the small solid circle is the chromium atom. The oxygen atoms of the upper layer have been numbered to conform to the designations used in the present work.

---

[16] L. E. Alexander and G. S. Smith, *Acta Crystallogr.*, **15**, 983 (1962).

[17] "International Tables for X-Ray Crystallography," Vol. III, Kynoch Press, Birmingham, England, 1962, pp 202, 210, 211.

TABLE III

POSITIONAL PARAMETERS[a]

Atom	x	y	z
Cr	0.0	0.0	0.0
Mo(1)	0.27552 (3)	−0.06622 (3)	−0.00253 (5)
Mo(2)	0.28333 (3)	0.18775 (3)	−0.18424 (5)
Mo(3)	0.00896 (3)	0.25164 (3)	−0.18745 (5)
Na(1)	0.5	0.0	0.5
Na(2)	0.4368 (2)	−0.4761 (2)	0.2798 (3)
O(1)	0.1032 (2)	−0.0218 (3)	0.2137 (4)
O(2)	0.1378 (2)	−0.0316 (3)	−0.2052 (4)
O(3)	0.1096 (2)	0.1956 (2)	0.0359 (4)
O(4)	0.1257 (3)	−0.2381 (3)	−0.0254 (4)
O(5)	0.3346 (3)	0.1285 (3)	0.0352 (4)
O(6)	0.1442 (3)	0.1840 (3)	−0.3545 (4)
O(7)	0.0745 (3)	−0.2336 (3)	0.4139 (5)
O(8)	0.3595 (3)	−0.0690 (3)	0.2036 (6)
O(9)	0.3535 (3)	−0.1189 (3)	−0.2347 (5)
O(10)	0.3737 (3)	0.1349 (4)	−0.3964 (5)
O(11)	0.3685 (3)	0.3616 (3)	−0.0824 (5)
O(12)	0.1016 (3)	0.4217 (3)	−0.0920 (5)
O(13)	0.2303 (3)	0.3739 (3)	0.3982 (5)
O(14)	0.1422 (7)	−0.4243 (5)	−0.4236 (7)
O(15)	0.3199 (5)	−0.3612 (5)	0.1805 (14)
O(16)	0.4737 (4)	−0.3616 (4)	−0.3354 (6)

[a] The standard error in parentheses after each parameter is for the last decimal place given and is derived from the full-matrix refinement.

one Mo atom. A view of the anion projected onto the least-squares best plane fitted to the Cr and Mo atoms is shown in Figure 2. The anion has the Anderson–Evans[13,14] configuration. The value of n in the formula as previously written[6] is established as 3. Arguments presented below indicate the most likely sites for the anion hydrogen atoms are on the oxygens coordinated to the central Cr atom. On this basis the formula of the anion should be written $Cr(OH)_6Mo_6O_{18}^{3-}$, although, for the present work, it is written $CrMo_6O_{24}H_6^{3-}$ to retain the analogy with the prototype anion $TeMo_6O_{24}^{6-}$.

Structure Description.—Views of the structure as projected down the c and a axes are depicted in Figures 3 and 4, respectively. The structure can be considered as consisting of columns of disk-like $CrMo_6O_{24}H_6^{3-}$ groups stacked along the c axis. The plane of each disk is tilted so that it is very nearly parallel to the (2,8,11) plane. These stacks are held together primarily through the linkage provided by the bonding of anion oxygens to Na(1). The Na(1) coordination, also, links neighboring stacks along the a direction. Additional direct bonding in the c direction is provided by hydrogen bonding utilizing two-thirds of the hydrogen atoms associated with each anion. A chain of Na(2) octahedra, formed by sharing edges, runs parallel to the c axis through the center of the ab face of the unit cell as shown in Figure 3. One of the shared edges of the chain consists of two anion oxygens, each from a different anion group, so that a linkage between stacks of anions is provided in the [110] direction. The Na(2) coordination is completed by water molecules. Further bonding along the anion stacks and between these stacks is provided by hydrogen bonding involving water molecules. One water molecule, O(14), is not coordinated to any cation and is held in the structure only through hydrogen bonds.

The resulting structure is consistent with the observed perfect cleavage parallel to (010). To cleave along this plane at $y = {}^1/_2$ only one Na–O bond and four hydrogen bonds need to be broken for each unit cell. To cleave along any other plane a minimum of three Na–O bonds and more than four hydrogen bonds must be broken for each unit cell.

The structure is, also, consistent with the easy loss

TABLE IV

THERMAL PARAMETERS[a]

Atom	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
Cr	0.00162 (6)	0.00208 (7)	0.00470 (16)	0.00040 (5)	−0.00006 (8)	0.00088 (8)
Mo(1)	0.00226 (3)	0.00313 (3)	0.00887 (8)	0.00095 (2)	−0.00042 (3)	0.00101 (4)
Mo(2)	0.00222 (3)	0.00295 (3)	0.00738 (8)	0.00048 (2)	0.00068 (3)	0.00124 (3)
Mo(3)	0.00297 (3)	0.00274 (3)	0.00657 (8)	0.00084 (2)	−0.00021 (3)	0.00139 (3)
Na(1)	0.0090 (3)	0.0215 (6)	0.0102 (6)	0.0118 (4)	0.0019 (4)	0.0053 (5)
Na(2)	0.0056 (2)	0.0060 (2)	0.0145 (5)	0.0006 (1)	0.0001 (2)	0.0038 (2)
O(1)	0.0023 (2)	0.0028 (2)	0.0042 (5)	0.0010 (2)	−0.0006 (3)	0.0008 (3)
O(2)	0.0018 (2)	0.0024 (2)	0.0036 (5)	0.0008 (2)	0.0008 (2)	0.0001 (3)
O(3)	0.0021 (2)	0.0018 (2)	0.0040 (5)	0.0004 (2)	−0.0002 (2)	0.0005 (3)
O(4)	0.0024 (2)	0.0024 (2)	0.0070 (6)	0.0007 (2)	0.0008 (3)	0.0008 (3)
O(5)	0.0022 (2)	0.0024 (2)	0.0091 (6)	0.0001 (2)	−0.0019 (3)	0.0007 (3)
O(6)	0.0022 (2)	0.0036 (2)	0.0052 (5)	0.0008 (2)	0.0005 (3)	0.0015 (3)
O(7)	0.0056 (3)	0.0044 (3)	0.0088 (7)	0.0021 (2)	−0.0020 (4)	0.0015 (3)
O(8)	0.0043 (3)	0.0053 (3)	0.00156 (8)	0.0011 (2)	−0.0042 (4)	0.0021 (4)
O(9)	0.0036 (3)	0.0057 (3)	0.0124 (7)	0.0025 (2)	0.0027 (3)	0.0017 (4)
O(10)	0.0050 (3)	0.0066 (3)	0.0120 (8)	0.0031 (3)	0.0043 (4)	0.0019 (4)
O(11)	0.0029 (2)	0.0026 (2)	0.0113 (7)	−0.0002 (2)	0.0002 (3)	0.0015 (3)
O(12)	0.0044 (3)	0.0027 (2)	0.0117 (7)	0.0005 (2)	−0.0000 (3)	0.0017 (3)
O(13)	0.0046 (3)	0.0042 (3)	0.0083 (6)	0.0005 (2)	0.0005 (3)	0.0013 (3)
O(14)	0.0258 (10)	0.0083 (5)	0.0155 (10)	0.0110 (6)	0.0060 (8)	0.0032 (6)
O(15)	0.0084 (5)	0.0086 (5)	0.0882 (35)	0.0014 (4)	−0.0098 (11)	0.0144 (11)
O(16)	0.0056 (3)	0.0058 (3)	0.0177 (9)	0.0012 (3)	−0.0014 (4)	−0.0005 (5)

[a] Anisotropic thermal parameters as defined by: $\exp(-b_{11}h^2 - b_{22}k^2 - b_{33}l^2 - 2b_{12}hk - 2b_{13}hl - 2b_{23}kl)$. The standard errors as derived from the full-matrix refinement are given in parentheses and refer to the last decimal place given.

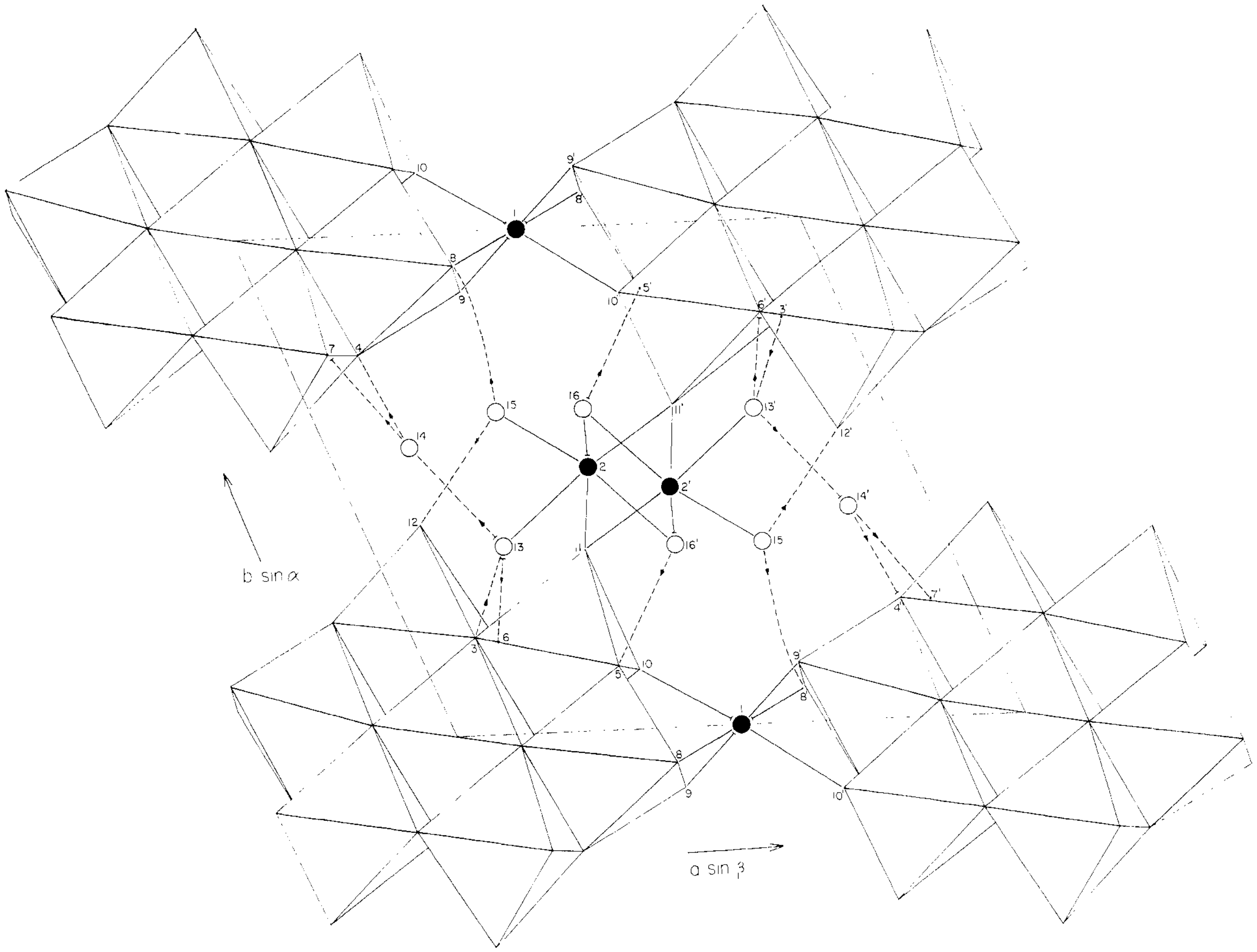

Figure 3.—View of the structure as projected down the *c* axis onto a plane normal to that axis. Open circles are water molecules and solid circles are Na atoms. Anion oxygens are at the octahedral corners or indicated by dots for hidden corners. Bonds to Na atoms are shown as solid lines. Postulated hydrogen bonds are shown as dashed lines with arrows to indicate the probable hydrogen donor (*i.e.*, O—H·····O shown as O→---O). Bond lines ending in arcs are to the indicated atom in the cell below. Bonding is indicated for unit cell contents between $z = -0.5$ and $z = 0.5$. Only atoms involved in indicated bonding have been numbered.

of water from the crystals. The first water molecules lost would probably be the O(14) molecules. These molecules are held in the crystal only by hydrogen bonds and lie in channels which are open to the (001) face. It is reasonable to expect that once the O(14) waters are gone, some rearrangement of Na coordination and hydrogen bonding takes place which permits other water molecules to escape.

Anion Details.—The complete list of metal–oxygen, oxygen–oxygen, and metal–metal distances and all angles with a metal vertex in the anion is given in Table V. Atoms with no superscript have parameters as given in Table III. Superscripted atoms have had their original positions transformed by the operation given in Table VI.

The Mo atoms closely approximate a regular planar hexagonal configuration with the Cr located in its center. The least-squares plane fitted to these atoms, expressed in fractional unit cell coordinates, is described by the equation $0.87445x + 3.6287y + 4.9772z = 0$ The maximum deviation from this plane is 0.012 Å. The range of nearest neighbor Mo–Mo distances is 3.309–3.351 Å and the Cr–Mo distances vary from 3.303 to 3.349 Å. Each metal atom is coordinated by a distorted octahedron of oxygen atoms. The Mo atoms are all displaced from the centers of their octahedra toward the outer oxygens of the anion so that all metal–metal distances are increased over the values they would have in an undistorted configuration. The observed distortions from ideal octahedra are in accord with generally accepted structural principals[18] and have been observed in several similar compounds.[19-22]

The observed metal–oxygen distances are in good agreement with other reported values. The Cr–O distances vary from 1.968 to 1.986 Å which may be compared with the typical octahedral Cr^{3+}–O distance

---

(18) A. F. Wells, "Structural Inorganic Chemistry," 2nd ed, Clarendon Press, Oxford, 1950, p 93.
(19) J. F. Keggin, *Proc. Roy. Soc. Ser. A*, **144**, 75 (1934).
(20) N. F. Yannoni, Doctoral Dissertation, Boston University, 1961.
(21) H. T. Evans, Jr., *J. Amer. Chem. Soc.*, **90**, 3275 (1968).
(22) D. D. Dexter and J. V. Silverton, *ibid.*, **90**, 3589 (1968).

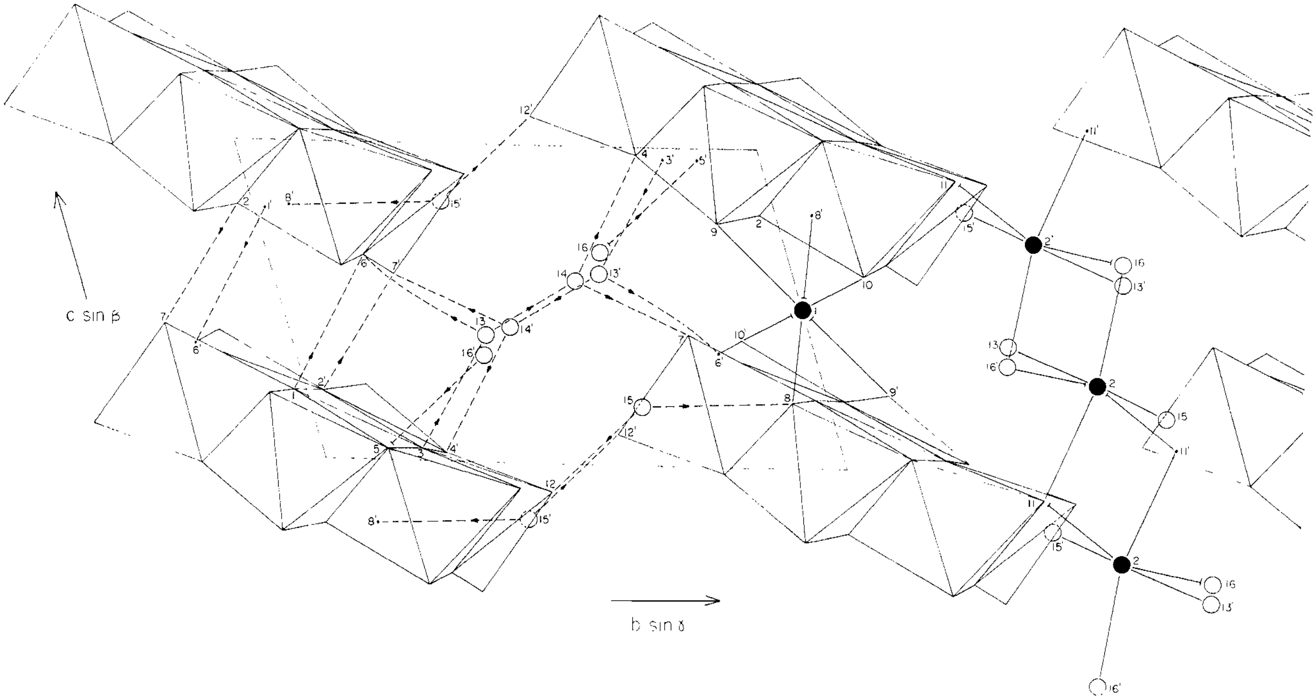

Figure 4.—View of the structure as projected down the *a* axis onto a plane normal to that axis. Atoms and bond conventions are the same as for Figure 3. The left side of the picture has been used to indicate probable hydrogen bonding and the Na atoms have not been shown. The right side has been used to indicate the Na coordination and O(14), which is not coordinated to an Na atom, has been omitted.

of 1.98 ± 0.08 Å.[23] The Mo–O distances fall into three ranges which depend on the oxygen coordination. The longest distances (2.243–2.347 Å, average 2.292 Å) are to oxygens coordinated to the Cr and two Mo atoms [O(1), O(2), O(3)]. Intermediate Mo–O distances (1.907–1.985 Å, average 1.939 Å) are to oxygens coordinated to two Mo atoms [O(4), O(5), O(6)]. The shortest distances (1.695–1.720 Å, average 1.707 Å) are to oxygens coordinated to only one Mo atom [O(7)–O(12)]. This same trend has been found in $TeMo_6O_{24}^{6-}$ ion where the comparable average Mo–O distances were found to be 2.299, 1.943, and 1.714 Å[21] and in the $CeMo_{12}O_{42}^{8-}$ ion where average values of 2.28, 1.98, and 1.68 Å have been reported.[22] Similar ranges have been reported for other molybdate structures containing octahedral MoO_6 groups joined by edge sharing, *e.g.*, 1.67–2.56 Å in molybdenum bronzes[24,25] and 1.67–2.33 Å in MoO_3.[26]

The oxygen–metal–oxygen angles in the anion deviate widely from the 90° angle expected for an ideal octahedron, but this is simply a consequence of the distortions resulting from the outward displacements of the Mo atoms and the shortening of oxygen–oxygen distances of shared edges.

Sodium Coordination.—Both types of sodium atoms may be considered to be in distorted octahedral surroundings. The pertinent distances and angles are listed in Table VII.

The sodium on a center of symmetry, Na(1), is coordinated to anion oxygens exclusively and its octahedron is isolated from all other sodium polyhedra. Oxygens from four anions are coordinated by a single Na(1) ion.

A Na(2) octahedron includes a pair of anion oxygens, each from a different anion, and four water molecules. The Na(2) octahedra are linked by edge sharing, of centrosymmetrically related O(11) atoms and O(16) atoms, to form a chain parallel to the *c* axis.

The range of Na–O distances (2.307–2.536 Å) found in this structure is well within the range 2.25–2.78 Å found in other compounds[27] containing sodium coordinated to six oxygens.

Hydrogen Sites on the Anion.—The structure determination has established that the anion contains 24 oxygen atoms. From the known valence states of the various atoms in the anion and the net charge of the anion it is clear that six hydrogen atoms must be included in the anion. As expected, the hydrogen positions could not be observed even in the electron density difference map. However, the following considerations of possible hydrogen bonds and local charge distributions strongly support the conclusion that the anion hydrogen atoms are bonded to the oxygens which are coordinated to the Cr atom.

There is no evidence to suggest that the hydrogen atoms violate the symmetry of the crystal, so it can reasonably be assumed that there are only three anion hydrogen atoms in the asymmetric unit. It would appear reasonable, also, to expect the hydrogen atoms to be bonded to oxygen atoms that would be chemically equivalent in the isolated anion. There are three such sets. In the asymmetric unit O(1), O(2), O(3) form

---

(23) See ref 17, p 266.

(24) J. Graham and A. D. Wadsley, *Acta Crystallogr.*, **20**, 93 (1966).

(25) N. C. Stephenson and A. D. Wadsley, *ibid.*, **18**, 241 (1965).

(26) L. Kihlborg, *Ark. Kemi*, **21**, 357 (1963).

(27) See ref 17, p 258.

TABLE V: ANION COORDINATION[a]

Distances		Angles	
Around Cr			
Cr–O(1)	1.986 (3)	O(1)–Cr–O(2)	85.12 (11)
Cr–O(2)	1.968 (3)	O(1)–Cr–O(2)^1	94.88 (11)
Cr–O(3)	1.972 (2)	O(1)–Cr–O(3)	95.25 (11)
O(1)–O(2)	2.674 (4)	O(1)–Cr–O(3)^1	84.75 (11)
O(1)–O(2)^1	2.913 (4)	O(2)–Cr–O(3)	83.58 (10)
O(1)–O(3)	2.924 (5)	O(2)–Cr–O(3)^1	96.42 (10)
O(1)–O(3)^1	2.667 (3)		
O(2)–O(3)	2.626 (4)		
O(2)–O(3)^1	2.937 (3)		
Around Mo(1)			
Mo(1)–O(1)	2.347 (3)	O(1)–Mo(1)–O(2)	70.46 (10)
Mo(1)–O(2)	2.289 (3)	O(1)–Mo(1)–O(4)	70.53 (10)
Mo(1)–O(4)	1.943 (2)	O(1)–Mo(1)–O(5)	81.20 (11)
Mo(1)–O(5)	1.926 (3)	O(1)–Mo(1)–O(8)	95.28 (14)
Mo(1)–O(8)	1.703 (4)	O(2)–Mo(1)–O(4)	82.45 (12)
Mo(1)–O(9)	1.708 (3)	O(2)–Mo(1)–O(5)	72.03 (11)
O(1)–O(2)	2.674 (4)	O(2)–Mo(1)–O(9)	89.01 (15)
O(1)–O(4)	2.499 (4)	O(4)–Mo(1)–O(8)	98.00 (14)
O(1)–O(5)	2.799 (4)	O(4)–Mo(1)–O(9)	98.34 (12)
O(1)–O(8)	3.024 (5)	O(5)–Mo(1)–O(8)	101.34 (14)
O(2)–O(4)	2.801 (5)	O(5)–Mo(1)–O(9)	101.74 (14)
O(2)–O(5)	2.496 (3)	O(8)–Mo(1)–O(9)	105.87 (18)
O(2)–O(9)	2.832 (5)		
O(4)–O(8)	2.757 (4)		
O(4)–O(9)	2.767 (4)		
O(5)–O(8)	2.811 (6)		
O(5)–O(9)	2.822 (4)		
O(8)–O(9)	2.722 (5)		
Around Mo(2)			
Mo(2)–O(2)	2.294 (2)	O(2)–Mo(2)–O(3)	70.23 (9)
Mo(2)–O(3)	2.270 (3)	O(2)–Mo(2)–O(5)	71.77 (10)
Mo(2)–O(5)	1.934 (4)	O(2)–Mo(2)–O(6)	81.96 (11)
Mo(2)–O(6)	1.941 (3)	O(2)–Mo(2)–O(10)	93.69 (13)
Mo(2)–O(10)	1.699 (4)	O(3)–Mo(2)–O(5)	83.79 (12)
Mo(2)–O(11)	1.716 (3)	O(3)–Mo(2)–O(6)	71.95 (11)
O(2)–O(3)	2.626 (4)	O(3)–Mo(2)–O(11)	92.03 (11)
O(2)–O(5)	2.496 (3)	O(5)–Mo(2)–O(10)	100.69 (17)
O(2)–O(6)	2.790 (5)	O(5)–Mo(2)–O(11)	98.47 (14)
O(2)–O(10)	2.941 (4)	O(6)–Mo(2)–O(10)	96.91 (16)
O(3)–O(5)	2.819 (5)	O(6)–Mo(2)–O(11)	101.20 (15)
O(3)–O(6)	2.488 (4)	O(10)–Mo(2)–O(11)	105.16 (14)
O(3)–O(11)	2.894 (4)		
O(5)–O(10)	2.801 (5)		
O(5)–O(11)	2.768 (5)		
O(6)–O(10)	2.729 (5)		
O(6)–O(11)	2.829 (4)		
O(10)–O(11)	2.712 (4)		
Around Mo(3)			
Mo(3)–O(1)^1	2.306 (3)	O(1)^1–Mo(3)–O(3)	71.80 (9)
Mo(3)–O(3)	2.243 (3)	O(1)^1–Mo(3) O(4)^1	72.05 (10)
Mo(3)–O(4)^1	1.907 (3)	O(1)^1–Mo(3)–O(6)	82.83 (10)
Mo(3)–O(6)	1.985 (3)	O(1)^1–Mo(3)–O(7)^1	90.67 (13)
Mo(3)–O(7)^1	1.720 (4)	O(3)–Mo(3)–O(4)^1	84.00 (12)
Mo(3)–O(12)	1.695 (3)	O(3)–Mo(3)–O(6)	71.82 (11)
O(1)^1–O(3)	2.667 (3)	O(3)–Mo(3)–O(12)	92.84 (14)
O(1)^1–O(4)^1	2.499 (4)	O(4)^1–Mo(3)–O(7)^1	102.48 (14)
O(1)^1–O(6)	2.848 (3)	O(4)^1–Mo(3)–O(12)	99.05 (13)
O(1)^1–O(7)^1	2.893 (5)	O(6)–Mo(3)–O(7)^1	94.50 (14)
O(3)–O(4)^1	2.788 (4)	O(6)–Mo(3)–O(12)	100.27 (13)
O(3)–O(6)	2.488 (4)	O(7)^1–Mo(3)–O(12)	105.96 (17)
O(3)–O(12)	2.877 (5)		
O(4)^1–O(7)^1	2.831 (4)		
O(4)^1–O(12)	2.744 (4)		
O(6)–O(7)^1	2.726 (5)		
O(6)–O(12)	2.830 (4)		
O(7)^1–O(12)	2.727 (4)		
Metal–Metal			
Cr–Mo(1)	3.3488 (4)	Mo(1)–Cr–Mo(2)	59.34 (1)
Cr–Mo(2)	3.3352 (3)	Mo(2)–Cr–Mo(3)	60.17 (1)
Cr–Mo(3)	3.3031 (4)	Mo(3)–Cr–Mo(1)^1	60.49 (1)
Mo(1)–Mo(2)	3.3085 (5)		
Mo(2)–Mo(3)	3.3279 (5)		
Mo(3)–Mo(1)^1	3.3510 (4)		

[a] Distances (Å) and angles (deg) within the anion. The standard deviations are given in parentheses and were computed using only the diagonal terms of the error matrix. The values refer to the last significant figure quoted. Some oxygen–oxygen distances have been repeated in order to group the complete environment around each cation. The superscripts on the atom designations refer to the operations as given in Table VI. Atoms without superscripts have the parameters listed in Table III.

TABLE VI

PARAMETER TRANSFORMATIONS[a]

Superscript	Transformation		
1	$-x$	$-y$	$-z$
2	x	y	$1+z$
3	$1-x$	$-y$	$-z$
4	x	$1+y$	z
5	$1-x$	$1-y$	$-z$
6	x	$1+y$	$1+z$
7	$1-x$	$-y$	$1-z$
8	x	y	$-1+z$
9	$-x$	$-1-y$	$-1-z$
10	$-x$	$1-y$	$-z$

[a] Transformations used in obtaining distances and angles. The x, y, z values are those listed in Table III.

TABLE VII

SODIUM COORDINATION[a]

Distances		Angles	
Around Na(1)			
Na(1)–O(8)	2.324 (4)	O(8)–Na(1)–O(9)^2	103.76 (11)
Na(1)–O(9)^2	2.536 (3)	O(8)–Na(1)–O(10)^2	75.53 (12)
Na(1)–O(10)^2	2.307 (4)	O(8)–Na(1)–O(9)^3	76.24 (11)
O(8)–O(9)^2	3.826 (5)	O(8)–Na(1)–O(10)^3	104.47 (13)
O(8)–O(10)^2	2.836 (4)	O(9)^2–Na(1)–O(10)^2	82.17 (13)
O(8)–O(9)^3	3.005 (4)	O(9)^2–Na(1)–O(10)^3	97.83 (13)
O(8)–O(10)^3	3.661 (6)		
O(9)^2–O(10)^2	3.187 (6)		
O(9)^2–O(10)^3	3.653 (5)		
Around Na(2)			
Na(2)^4–O(11)	2.453 (3)	O(11)–Na(2)^4–O(13)	85.03 (10)
Na(2)^4–O(13)	2.442 (4)	O(11)–Na(2)^4–O(11)^5	82.57 (11)
Na(2)^4–O(11)^5	2.475 (3)	O(11)–Na(2)^4–O(15)^4	84.22 (20)
Na(2)^4–O(15)^4	2.348 (9)	O(11)–Na(2)^4–O(16)^3	85.74 (14)
Na(2)^4–O(16)^6	2.419 (4)	O(13)–Na(2)^4–O(15)^4	89.78 (19)
Na(2)^4–O(16)^3	2.458 (6)	O(13)–Na(2)^4–O(16)^3	83.55 (15)
O(11)–O(13)	3.308 (4)	O(13)–Na(2)^4–O(16)^6	83.21 (12)
O(11)–O(11)^5	3.252 (3)	O(11)^5–Na(2)^4–O(15)^4	89.55 (20)
O(11)–O(15)^4	3.220 (7)	O(11)^5–Na(2)^4–O(16)^3	94.93 (15)
O(11)–O(16)^3	3.341 (6)	O(11)^5–Na(2)^4–O(16)^6	109.06 (12)
O(13)–O(15)^4	3.381 (9)	O(15)^4–Na(2)^4–O(16)^6	101.62 (23)
O(13)–O(16)^3	3.265 (6)	O(16)^3–Na(2)^4–O(16)^6	87.06 (17)
O(13)–O(16)^6	3.228 (4)		
O(11)^3–O(15)^4	3.398 (7)		
O(11)^3–O(16)^3	3.635 (6)		
O(11)^3–O(16)^6	3.986 (5)		
O(15)^4–O(16)^6	3.694 (10)		
O(16)^3–O(16)^6	3.359 (6)		

[a] Distances (Å) and angles (deg) of Na coordination. Standard deviations and superscript significance are the same as described for Table V.

one set, O(4), O(5), O(6) form a second set, and O(7)–O(12) form a third set. The last set can be eliminated because it contains too many atoms, so the problem reduces to a choice between sets 1 and 2. In Table VIII are listed all of the close oxygen–oxygen pairs which could reasonably be expected to be linked by hydrogen bonds. The pairs O(1)–O(6) and O(2)–O(7) are the only pairs not involving a water molecule. The first pair is inconclusive as it involves oxygens from both set 1 and set 2. The second pair involves oxygens from set 1 and set 3. If the original assumption discarding set 3 is valid and if these close contacts actually are hydrogen bonds, then O(2) must be the donor in the hydrogen bond to O(7). Therefore, if all the postulated assumptions are valid, it may be concluded that the anion hydrogen atoms are bonded to the oxygen atoms of set 1 which are the oxygen atoms coordinated to the Cr atom.

The same conclusion can be reached in a more satisfactory manner by applying the principle of detailed neutralization of valence set forth by Pauling,[28] who applied it to silicate structures, and extensively used by Zachariasen[29] for borate structures. Let the index i denote a crystallographic species of atoms having a formal positive chemical valence v_i, while the index j designates a crystallographic species of negative valence $-v_j$. If v_{ij} $(=v_{ji})$ represents the valence strength assigned to a chemical bond of length r_{ij} formed between atoms i and j, then the principle states that the conditions

$$\sum_j v_{ij} \approx v_i, \qquad \sum_i v_{ij} \approx v_j$$

shall hold for all crystallographic species i and j.

To apply this principle to the present structure, estimates of the bond strengths for Cr^{3+}–O^{2-} and Mo^{6+}–O^{2-} are needed. The Cr–O bond lengths are essentially equal, within 0.02 Å, so that the Cr valence can be divided equally among its six neighboring oxygens to yield a Cr–O bond strength of 0.5. The Mo–O bond lengths vary over several tenths of 1 Å and it would be quite unrealistic to assign them equal bond strengths. However, a good estimate can be obtained by assuming a linear relationship between bond strength and bond length. One point was obtained by assuming the average of all the Mo–O distances (1.979 Å) in the present structure was equivalent to the average ideal octahedral bond strength of 1.000 required for Mo^{6+}. The second point was derived in a similar fashion from the results of Ibers and Smith[30] for the compound $NaCo_{2.31}(MoO_4)_3$ in which all of the Mo is hexavalent and tetrahedrally coordinated. Here it was assumed that the average of all the Mo–O distances (1.770 Å) was equivalent to the average ideal tetrahedral bond strength of 1.500 required for Mo^{6+}. From these two points a straight-line plot of bond strength *vs.* bond length for Mo–O bonds was obtained. The summations around each Mo atom using points from this curve

$$\sum_j (\mathrm{Mo}(i)\text{–O}_j) = 5.91, 6.05, 6.06 \text{ for } i = 1, 2, 3$$

are sufficiently close to the ideal value (6.00) that the linear approximation may be accepted as a good estimate. Using the above estimates of bond strengths for Cr–O and Mo–O bonds the valence balance around the oxygens of interest (O(1)–O(6)) was computed with the following results for Σ (= 2.00 ideally): O(1), 0.84; O(2), 1.01; O(3), 1.18; O(4), 2.27; O(5), 2.23; O(6), 2.07. Within the accuracy of the approximations made O(4), O(5), and O(6) can be considered as balanced, but O(1), O(2), and O(3) clearly need the additional bond strength (~1.0) supplied by coordination to a hydrogen atom.

Hydrogen Bonding—Since the hydrogen atoms could not be located directly in the present work, any proposed hydrogen-bonding scheme is necessarily speculative. A scheme based on short oxygen–oxygen distances is generally considered highly probable if all hydrogens can be utilized and if only one hydrogen can be assigned to each short distance.[31] Short O–O distances which are part of a coordination polyhedron are excluded from consideration.

A list of all potential hydrogen-bonding distances under 3.1 Å is given in Table VIII. Using these distances and accepting the conclusion of the previous section that O(1), O(2), and O(3) are hydroxyl oxygens, a satisfactory scheme based on the above criterion can be devised for all hydroxyl oxygens and the O(13) and O(15) water oxygens. However, O(14), with five short distances, has too many close contacts, and O(16), with only one, has too few.

TABLE VIII

POSSIBLE HYDROGEN BONDS[a]

Atom pair	Distance, Å	Atom pair	Distance, Å
$O(1)–O(6)^2$	2.960(3)	$O(7)^8–O(14)$	2.949(8)
$O(2)–O(7)^8$	2.706(3)	O(8)–O(15)	3.027(8)
O(3)–O(13)	2.629(3)	$O(12)–O(14)^4$	3.015(7)
O(4)–O(14)	2.802(5)	$O(12)–O(15)^4$	2.897(6)
$O(5)–O(16)^3$	2.896(4)	$O(13)–O(14)^5$	2.652(8)
$O(6)^2–O(13)$	2.832(5)	$O(14)–O(14)^9$	3.004(10)

[a] Short oxygen–oxygen distances (<3.1 Å) which suggest possible hydrogen bonds. Standard deviations and superscript significance are the same as described for Table V. No pair of oxygens coordinated to the same cation is included.

There are two possibilities for satisfying the O(14) molecules. The three shortest O–O distances could be chosen to yield a "normal" configuration, or bifurcated hydrogen bonds could be postulated to utilize all of the short O–O distances. Even though the existence of stable bifurcated hydrogen bonds has been unambiguously established by several high-precision X-ray and neutron diffraction studies,[31] the first possibility was chosen on the basis of the extremely elongated thermal ellipsoid obtained for O(14). Use of the three shortest O–O distances around O(14) would give it a planar coordination which is essentially normal to the longest principal axis of its thermal ellipsoid.

The only possibility for O(16) is to assume that one of its hydrogen atoms is not utilized for hydrogen bond formation. This situation has been found to exist in $MgSO_4 \cdot 4H_2O$.[32]

The postulated hydrogen-bonding scheme is illustrated in Figures 3 and 4. Briefly, the situation for each atom involved in the asymmetric unit is postulated as follows. Above and below are used in reference to the relative z parameter. O(1) donates its hydrogen to an O(6) in the anion above. O(2) donates its hydrogen to an O(7) in the anion below. O(3) contributes its hydrogen to the water molecule O(13). The O(13) water contributes its hydrogen atoms to an O(6) in the anion above and to an O(14) water molecule. O(14) donates one hydrogen to an

---

[28] L. Pauling, *J. Amer. Chem. Soc.*, **51**, 1010 (1929).

[29] W. H. Zachariasen, *Acta Crystallogr.*, **16**, 385 (1963).

[30] J. A. Ibers and G. W. Smith, *ibid.*, **17**, 190 (1964).

[31] W. H. Baur, *ibid.*, **19**, 909 (1965).

[32] W. H. Baur, *ibid.*, **17**, 863 (1964).

TABLE IX

SIZE AND ORIENTATION OF THERMAL ELLIPSOIDS[a]

Atom	i	$\bar{u}_i$	$\theta(X)$	$\theta(Y)$	$\theta(Z)$	Atom	i	$\bar{u}_i$	$\theta(X)$	$\theta(Y)$	$\theta(Z)$
Cr	1	0.0888	42	48	87	O(6)	1	0.0940	115	95	26
	2	0.0953	85	92	174		2	0.1112	143	115	115
	3	0.1080	132	42	95		3	0.1346	115	26	96
Mo(1)	1	0.1056	21	72	81	O(7)	1	0.1116	59	92	31
	2	0.1223	70	153	107		2	0.1415	78	164	100
	3	0.1362	93	109	19		3	0.1790	34	74	119
Mo(2)	1	0.0993	43	60	118	O(8)	1	0.1064	38	73	57
	2	0.1181	88	135	135		2	0.1633	80	161	74
	3	0.1371	133	60	122		3	0.2025	53	97	143
Mo(3)	1	0.1094	78	104	19	O(9)	1	0.1014	35	100	123
	2	0.1139	107	160	101		2	0.1654	107	161	98
	3	0.1287	21	103	105		3	0.1774	119	74	146
Na(1)	1	0.1178	17	100	77	O(10)	1	0.0950	46	103	133
	2	0.1357	79	102	163		2	0.1808	98	167	80
	3	0.3319	77	16	99		3	0.1955	45	88	45
Na(2)	1	0.1457	65	45	125	O(11)	1	0.0944	62	30	99
	2	0.1669	113	115	144		2	0.1445	119	66	39
	3	0.2021	35	125	96		3	0.1566	42	107	52
O(1)	1	0.0836	64	77	30	O(12)	1	0.1112	79	14	99
	2	0.1127	153	78	66		2	0.1509	85	100	169
	3	0.1183	85	18	107		3	0.1650	12	100	83
O(2)	1	0.0702	60	108	144	O(13)	1	0.1258	99	77	16
	2	0.1008	134	134	100		2	0.1324	116	152	82
	3	0.1225	120	50	125		3	0.1797	28	114	76
O(3)	1	0.0846	77	42	51	O(14)	1	0.1350	113	34	67
	2	0.0947	107	125	40		2	0.1781	84	112	23
	3	0.1130	22	111	85		3	0.3680	24	66	87
O(4)	1	0.1028	44	78	132	O(15)	1	0.1742	128	45	110
	2	0.1066	91	162	108		2	0.1909	138	132	92
	3	0.1361	46	103	47		3	0.4232	105	76	20
O(5)	1	0.0830	50	46	72	O(16)	1	0.1434	69	33	67
	2	0.1248	57	135	63		2	0.1829	154	78	67
	3	0.1465	57	97	146		3	0.2161	104	60	146

[a] Principal axes of anisotropic temperature factors, referred to orthogonal axes X, Y, Z. X is along the crystallographic a axis, Y is normal to the (010) plane (*i.e.*, along b^*), and Z is normal to X and Y in a direction to form a right-handed coordinate system. The root-mean-square displacements ($\bar{u}_i$) along the principal axes are in ångströms and the direction angles θ are in degrees. No errors were computed for these quantities.

O(7) in an anion below and the second to an O(4) in an anion above. O(15) donates one hydrogen to an O(8) of one anion and the other to an O(12) of a second anion. O(16) donates one hydrogen to an O(5) and apparently leaves the other hydrogen unshared.

Thermal Motion.—In Table IX are listed, for each atom, the root-mean-square displacements along the principal axes of the thermal ellipsoid derived from the anisotropic temperature factors and the orientation of each ellipsoid relative to an orthogonal coordinate system X, Y, Z. X is taken along the a axis, Y is taken normal to the (010) plane (*i.e.*, along b^*), and Z is oriented so as to form a right-handed orthogonal system. Errors for these quantities were not obtained. However, based on other X-ray structures of comparable precision, it is estimated that the errors in the displacements are in the third decimal place and the errors in the orientation angles would be of the order of several degrees.

There is always some doubt as to the reliance to be placed on the magnitudes of the temperature factors and the quantities derived from them, because errors in quantities such as the form factors and absorption, which are approximately an exponential function of $(\sin\theta)/\lambda$, can be compensated by adjustment of the temperature factors. The relative magnitudes of the thermal motion can usually be accepted as a good qualitative indication in deciding whether one atom moves more or less than another.

In the present case there are no data on strictly equivalent compounds with which a comparison can be made. However, comparison with some, not closely, related compounds provides support for the physical reasonableness of the values obtained in this study.

Anisotropic displacements for Mo in tetrahedral surroundings range from 0.087 to 0.116 Å in the compound $NaCo_{2.31}(MoO_4)_3$.[30] This may be compared to the range 0.099–0.137 Å found for Mo in the present work. The order of magnitude of the displacements is the same for the two compounds. It is reasonable that an atom in octahedral surroundings should have slightly greater freedom of motion than the same atom in tetrahedral coordination.

The anion oxygen displacements vary from a minimum value of 0.084 Å to a maximum value of 0.202 Å. No equivalent comparison data are available, but the values observed for sulfate oxygens in $MgSO_4 \cdot 7H_2O$,[33] 0.110–0.233 Å, could be considered representative val-

---

(33) W. H. Baur, *Acta Crystallogr.*, **17**, 1361 (1964).

ues for hydrated inorganic salts. More significant is the observation that the trend of the displacement magnitudes is in agreement with what would be expected from the structure. The oxygens with the most ligands (O(1)–O(3)) have the smallest displacements, O(4)–O(6) are intermediate, and O(7)–O(12), with the fewest ligands, have the largest amplitudes of motion.

No thermal motion data could be found for a salt which loses water of hydration as readily as $Na_3(CrMo_6O_{24}H_6) \cdot 8H_2O$. It is reasonable to expect the water molecules to have equal or greater freedom of motion than any anion oxygen. This is observed to be the case. In view of the generally good agreement with other structures of the magnitudes of thermal motion of the other atoms in the salt it must be concluded that the values for the displacements of the water molecules are of comparable accuracy.

The anisotropy of the computed thermal ellipsoids appears to be real. In all cases the maximum principal axis is orientated in a direction which could be predicted from the observed structure. For example, the anion oxygens which are bonded to only one Mo atom (O(7)–O(12)) would be predicted to have the maximum displacement approximately normal to the Mo–O bond and this is found to be true for all of them. On the basis of the proposed hydrogen-bonding scheme O(15) is coordinated to three atoms. This situation would predict a marked anisotropy with the maximum principal axis approximately normal to the plane defined by the three coordinating atoms and this is what is found. The O(14) atom provides the same situation, but its coordination was chosen to yield agreement. The distorted octahedron around Na(1) has one centrosymmetrically related pair of faces which are appreciably larger than the other octahedral faces. The direction defined by the centers of these large opposing faces would be predicted to be a direction of large thermal displacement. It is observed that Na(1) has a very elongated thermal ellipsoid with the maximum principal axis in the predicted direction. This consistent agreement appears to justify considerable confidence in the accuracy of the present structure determination.

Acknowledgment.—I wish to express my appreciation to Professors L. C. W. Baker and J. V. Silverton of Georgetown University for suggesting the problem and their continued interest and assistance during the solution. Special thanks are due to F. A. Mauer of the National Bureau of Standards for his assistance with the data collection and to Dr. D. E. Appleman of the U. S. Geological Survey for performing the piezoelectric tests.

CONTRIBUTION FROM THE MALLINCKRODT LABORATORY, DEPARTMENT OF CHEMISTRY,
HARVARD UNIVERSITY, CAMBRIDGE, MASSACHUSETTS 02138

The Crystal and Molecular Structure of Acenaphthylenediiron Pentacarbonyl, $C_{12}H_8Fe_2(CO)_5$

BY MELVYN R. CHURCHILL[1] AND JOHN WORMALD

Received December 18, 1969

Acenaphthylenediiron pentacarbonyl, $C_{12}H_8Fe_2(CO)_5$, crystallizes in the centrosymmetric monoclinic space group $P2_1/n$ (C_{2h}^5; no. 14) with $a = 10.056 \pm 0.007$ Å, $b = 16.089 \pm 0.010$ Å, $c = 9.376 \pm 0.006$ Å, $\beta = 91.53 \pm 0.09°$, $\rho_{obsd} = 1.74 \pm 0.03$ g cm^{-3}, and $\rho_{calcd} = 1.769$ g cm^{-3} for $Z = 4$. A single-crystal X-ray diffraction study of this complex has been completed, using counter data to $\sin \theta = 0.40$ (Mo Kα radiation). All atoms, including hydrogens, have been located, the final discrepancy index being $R_F = 6.71\%$ for 1791 independent nonzero reflections The crystal consists of distinct molecular units of $C_{12}H_8Fe_2(CO)_5$ separated by normal van der Waals distances. The molecule consists of a modified acenaphthylene ligand in which the carbon atoms of the five-membered ring form a π-cyclopentadienyl system which is bonded to an $Fe(CO)_2$ group, while the three atoms C(5), C(10), and C(6) participate in a π-allyl linkage to an $Fe(CO)_3$ group. The two iron atoms are mutually linked *via* a bond 2.769 Å in length.

Introduction

Recent structural studies on the azulene–metal–carbonyl complexes $C_{10}H_8Fe_2(CO)_5$,[2] $C_{10}H_8Mo_2(CO)_6$,[3–5] $[(i\text{-}C_3H_7)(CH_3)_2C_{10}H_5]Mo_2(CO)_6$,[3,6] $[C_{10}H_8Mo(CO)_3CH_3]_2$,[7] $C_{10}H_8Mn_2(CO)_6$,[8] $(C_{10}H_8)_2Fe_4(CO)_{10}$,[9] and $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$[10] have indicated that a dominant feature of azulene-to-metal bonding is the use of

---

(1) Research Fellow of the Alfred P. Sloan Foundation, 1968–1970.

(2) M. R. Churchill, *Inorg. Chem.*, **6**, 190 (1967); M. R. Churchill, *Chem. Commun.*, 450 (1966).

(3) M. R. Churchill and P. H. Bird, *ibid.*, 746 (1967).

(4) J. S. McKechnie and I. C. Paul, *ibid.*, 747 (1967).

(5) A. W. Schlueter and R. A. Jacobsen, *Inorg. Chim. Acta*, **2**, 241 (1968).

(6) M. R. Churchill and P. H. Bird, *Inorg. Chem.*, **7**, 1545 (1968).

(7) P. H. Bird and M. R. Churchill, *ibid.*, **7**, 349 (1968); P. H. Bird and M. R. Churchill, *Chem. Commun.*, 705 (1967).

(8) M. R. Churchill and P. H. Bird, *Inorg. Chem.*, **7**, 1793 (1968); P. H. Bird and M. R. Churchill, *Chem. Commun.*, 145 (1968).

(9) M. R. Churchill and P. H. Bird, *Inorg. Chem.*, **8**, 1941 (1969); M. R. Churchill and P. H. Bird, *J. Amer. Chem. Soc.*, **90**, 3241 (1968).

(10) M. R. Churchill, K. Gold, and P. H. Bird, *Inorg. Chem.*, **8**, 1956 (1969); M. R. Churchill and P. H. Bird, *J. Amer. Chem. Soc.*, **90**, 800 (1968).