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The Crystal Structure and Hydrogen Bonding of Magnesium Sulfate Hexahydrate*

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Crystals of $MgSO_4.6H_2O$ are monoclinic, space group $C2/c$, with 8 formula units in the unit cell with dimensions

$$a = 10{\cdot}110,\ b = 7{\cdot}212,\ c = 24{\cdot}41\ \text{Å};\ \beta = 98{\cdot}30°.$$

The atomic parameters were refined by least squares, X-ray counter data being used for 2576 independent reflections. The positions of the hydrogen atoms found by least squares and by Fourier methods confirm the assignment of hydrogen bonds made previously for the isomorphous $CoSO_4.6H_2O$. Average interatomic distances are: water coordinated to Mg, Mg–O = 2·06 Å; hydrogen bonds, O–O = 2·82 Å; sulfate ion, S–O = 1·473 Å (uncorrected), 1·486 Å (corrected for thermal motion).

Introduction

The crystal structure of $CoSO_4.6H_2O$ has been described in a previous paper (Zalkin, Ruben & Templeton, 1962). Hydrogen bonds were assigned on the basis of the positions of the heavier atoms, but the X-ray diffraction data did not give direct evidence of the hydrogen atom positions. The present paper describes a study of the isomorphous substance $MgSO_4.6H_2O$ for which we have obtained more accurate diffraction data. These better data permit a more accurate description of the structure and indicate hydrogen atom positions in agreement with those deduced in the earlier work.

Crystals of $MgSO_4.6H_2O$ are known in nature as the mineral hexahydrite (Dana & Ford, 1948). Other substances which have the same structure are $MgSeO_4 \cdot 6H_2O$, $NiSO_4.6H_2O$, $CoSeO_4 \cdot 6H_2O$, and $ZnSO_4 \cdot 6H_2O$ (Groth, 1908).

Experimental

Crystals of $MgSO_4.6H_2O$ were grown from saturated aqueous solution at 50–55 °C. Once grown and separated from the mother liquor, the exposed crystals could be kept at room temperatures for several months with no visible deterioration. A small single crystal with diameter 0·02–0·08 mm was mounted on a glass fiber and oriented for rotation about its b axis with the use of a Weissenberg camera. It was then transferred to a goniostat mounted on a General Electric XRD5 apparatus, which was equipped with a molybdenum X-ray tube, a scintillation counter, and a pulse-height discriminator, for further alignment and data taking.

Cell dimensions and crystal settings were calculated with $\lambda(K\alpha_1) = 0{\cdot}70926$ Å. Intensities were measured for 2576 reflections of which 289 were recorded as zero. All of the reciprocal lattice points with positive h and k, out to a limit of $\sin\theta/\lambda = 0{\cdot}705$ $(2\theta = 60°)$ were measured. Step scanning of the angle θ was done for selected reflections to verify the settings for peak intensity. For the typical reflection, a single count was made for a fixed time of 20 seconds.

The absorption coefficient μ for this crystal with Mo $K\alpha$ X-rays is estimated as 5·0 cm^{-1}. The resulting μR is less than 0·02. No correction was made for absorption or extinction.

An accurate trial structure was provided by the structure of $CoSO_4.6H_2O$ (Zalkin, Ruben & Templeton, 1961); corresponding coordinates for atoms other than hydrogen differ by 0·01 or less in the two structures. Least-square refinements were started with the programs of Busing & Levy (1959) on the IBM 704 computer. We then used the IBM 709 computer with the programs of Gantzel, Sparks & Trueblood (1961). Each program utilizes the full matrix, and each minimizes the function $\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2$, where w is the weighting factor and F_o and F_c are the observed and calculated structure factors. In this calculation, each independent reflection was assigned unit weight. The atomic scattering factors used in the refinements were Mg^{2+} and neutral S (Tomiie & Stam, 1958), neutral O (Hoerni & Ibers, 1954), and neutral H (*International Tables for X-ray Crystallography*, 1962).

Space group and cell dimensions

The space group is $C2/c$ (C^6_{2h}). Reflections are absent with $h+k$ odd, and $h0l$ reflections are absent with l odd. The cell dimensions are:

* Work done under the auspices of the U.S. Atomic Energy Commission.

$a = 10{\cdot}110 \pm 0{\cdot}005$, $b = 7{\cdot}212 \pm 0{\cdot}004$, $c = 24{\cdot}41 \pm 0{\cdot}01$ Å
(10·06) (7·16) (24·39)
$\beta = 98{\cdot}30 \pm 0{\cdot}05°$ $Z = 8$ $U = 1761{\cdot}2$ Å^3.
(98·57)

The values in parentheses are those reported by Ide (1938), changed from kX. The calculated X-ray density is 1·723 g.cm^{-3}. The $a:b:c$ ratios found in this and in previous work are:

1·402:1:3·384, $\beta = 98{\cdot}30°$ (this work),
1·404:1:3·404, $\beta = 98{\cdot}57°$ (Ide, 1938),
1·404:1:3·337, $\beta = 98{\cdot}57°$ (Marignac, 1855).

The eight Mg atoms occupy two sets of positions 4(a) and 4(e) (notation of *International Tables for X-ray Crystallography*, 1952):

$$4(a):\quad (0, 0, 0;\ 0, 0, \tfrac{1}{2}) + C \text{ centering},$$
$$4(e):\quad (0, y, \tfrac{1}{4};\ 0, -y, \tfrac{3}{4}) + C \text{ centering}.$$

The sulfur atoms, ten sets of oxygen atoms, and twelve sets of hydrogen atoms occupy general positions:

$$8(f):\quad \pm(x, y, z;\ -x, y, \tfrac{1}{2}-z) + C \text{ centering}.$$

Refinement procedure

At first hydrogen was omitted from the calculations. With isotropic temperature factors of the form $\exp(-B\sin^2\theta/\lambda^2)$ for each atom, $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ was reduced to 0·120 by three cycles of least squares. After correction of a few blunders in the original measurements, two more cycles reduced R to 0·118.

A three-dimensional Fourier synthesis with $(F_o - F_c)$ as coefficients was calculated to seek out the hydrogen atoms. The resulting maps showed a peak wherever a hydrogen atom was expected, as well as many others of about the same size which resulted from the anisotropic motion of oxygen atoms. A final isotropic refinement which included the hydrogen atoms but did not refine them resulted in $R = 0{\cdot}115$.

Anisotropic temperature factors of the form

$$\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$$

were introduced for Mg, S and O. Three cycles with hydrogen atoms included but not refined gave $R = 0{\cdot}056$.

A set of $(F_o - F_c)$ values was calculated with the resulting parameters for Mg, S, and O, but omitting hydrogen. Another difference-Fourier synthesis was calculated with these numbers as coefficients. The twelve largest peaks in this function (Fig. 1) were near the predicted locations of the twelve hydrogen atoms.

As our least-square programs at this time could not handle at one time a refinement of all the atoms including hydrogen with all the anisotropic thermal parameters, we ran a series of calculations refining parts of the structure separately. Temperature factors

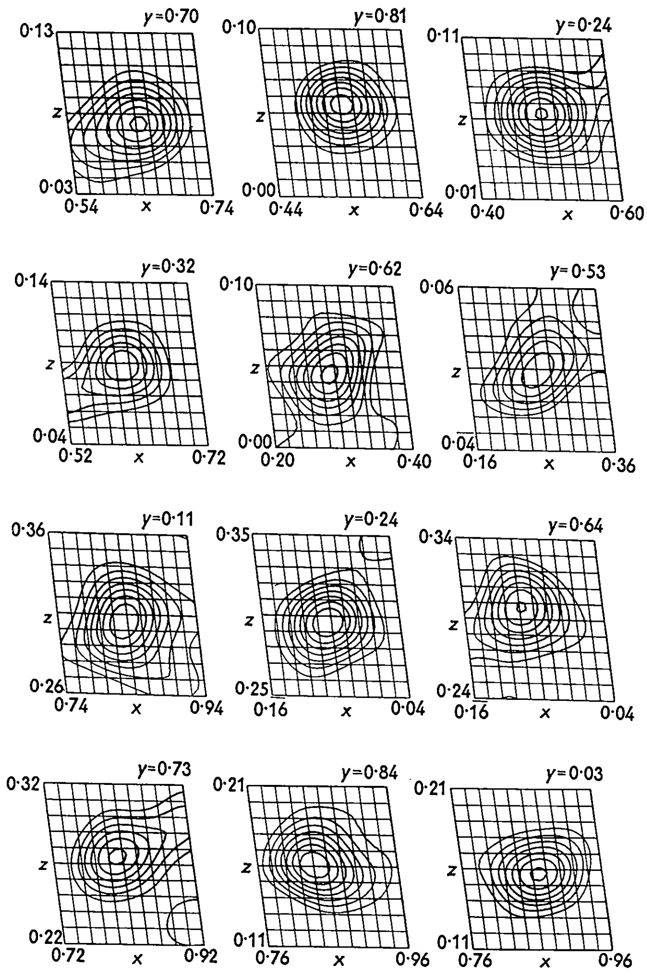

Fig. 1. Electron density sections through hydrogen atoms, with other atoms subtracted out. Contour interval approx. 0·1 e.Å^{-3}; zero and negative contours omitted. Grid lines and contours were reproduced photographically from cathode ray tube of computer.

were isotropic for H and anisotropic for the other atoms. First the H atoms were included but not refined, while all other parameters were refined. Next H atoms were refined with the other atoms fixed. Then Mg atoms were held fixed while all other atoms were refined; in this series only the 923 reflections with $\sin\theta/\lambda$ less than 0·5 were included. Finally the H atoms were fixed and all other atoms were refined. All this calculation reduced R to 0·053 for the 2576 reflections, including those with zero intensity. In the final cycle no coordinate or thermal parameter shifted more than 0·000001.

The resulting observed and calculated structure factors are shown in Table 1. The corresponding coordinates are listed in Table 2 and the thermal parameters in Tables 3 and 4.

By a special patch, the program was modified to allow simultaneous refinement of all 159 variable parameters. Two cycles yielded no significant improve-

Table 1. *Observed and calculated structure factors, multiplied by 10*

H,K= 0, 0		
L	FOBS	FCAL
2	50	42
4	453	-459
6	73	62
8	525	513
10	597	-582
12	710	-724
14	480	423
16	1161	1159
18	468	495
20	657	-671
22	0	-27
24	522	540
26	146	-174
28	0	42
30	121	-107
32	340	338
H,K= 2, 0		
L	FOBS	FCAL
32	0	46
30	102	-117
28	119	131
26	229	231
24	0	7
22	479	-464
20	401	397
18	1492	1519
16	368	370
14	326	-339
12	1930	1964
10	436	-435
8	46	40
6	2511	-2634
4	1447	1436
2	1574	1560
0	903	822
-2	1620	-1637
-4	1255	1218
-6	1513	1552
-8	2195	-2256
-10	383	-362
-12	323	292
-14	674	693
-16	140	151
-18	672	-657
-20	645	655
-22	190	205
-24	505	512
-26	206	-208
-28	304	333
-30	193	176
-32	184	158
-34	187	-191
H,K= 4, 0		
L	FOBS	FCAL
30	256	278
28	180	186
26	105	-92
24	119	-135
22	0	-29
20	683	699
18	416	-408
16	140	144
14	409	396
12	1197	1182
10	1312	-1289
8	199	216
6	221	208
4	73	28
2	683	-668
0	736	-741
-2	1841	1853
-4	662	687
-6	832	813
-8	1000	979
-10	616	598
-12	1284	1303
-14	571	-541
-16	188	186
-18	156	-200
-20	863	854
-22	203	-194
-24	157	164
-26	0	9
-28	257	253
-30	81	72
-32	204	-190
-34	0	1
H,K= 6, 0		
L	FOBS	FCAL
28	0	16
26	111	-128
24	339	331
22	77	-65
20	0	9
18	479	-466
16	473	471
14	204	176
12	537	543
10	549	569
8	638	635
6	953	944
4	294	-245
2	227	-234
0	434	447
-2	36	49
-4	650	641
-6	230	209
-8	883	885
-10	351	-340
-12	729	749
-14	1054	-1057
-16	275	268
-18	394	346
-20	34	49
-22	208	-185
-24	82	95
-26	224	218
-28	0	-5
-30	157	-123
-32	123	122
H,K= 8, 0		
L	FOBS	FCAL
24	422	458
22	84	-87
20	121	-112
18	55	77
16	124	92
14	108	103
12	206	196
10	259	249
8	632	608
6	240	269
4	498	-490
2	466	-484
0	872	879
-2	635	-634
-4	377	370
-6	218	-210
-8	667	630
-10	146	-144
-12	120	-136
-14	123	116
-16	682	706
-18	293	296
-20	131	-140
-22	274	267
-24	244	247
-26	135	-133
-28	42	-37
-30	0	29
H,K=10, 0		
L	FOBS	FCAL
20	88	105
18	225	244
16	226	-239
14	237	-251
12	244	236
10	201	163
8	344	342
6	511	-522
4	405	403
2	436	444
0	446	420
-2	419	-409
-4	610	608
-6	451	454
-8	70	-86
-10	238	-190
-12	176	177
-14	510	511
-16	0	50
-18	86	-121
-20	256	259
-22	41	43
-24	0	8
-26	241	-243
H,K=12, 0		
L	FOBS	FCAL
14	147	159
12	407	423
10	244	-281
8	0	13
6	200	174
4	497	516
2	98	-97
0	158	-120
-2	286	304
-4	245	252
-6	142	-138
-8	295	-268
-10	143	151
-12	284	273
-14	223	-209
-16	0	-14
-18	42	-1
-20	212	259
-22	140	-150
H,K=14, 0		
L	FOBS	FCAL
0	193	182
-2	88	107
-4	44	-78
-6	62	-66
-8	285	298
-10	99	125
H,K= 1, 1		
L	FOBS	FCAL
33	88	96
32	61	-41
31	59	64
30	41	25
29	98	-71
28	368	343
27	239	-230
26	296	-296
25	63	35
24	35	-44
23	0	2
22	455	463
21	203	-187
20	524	519
19	118	-100
18	325	-324
17	432	434
16	68	-57
15	243	233
14	659	641
13	56	-54
12	1348	1396
11	502	-511
10	333	-299
9	313	326
8	707	667
7	54	-71
6	1285	1292
5	94	-106
4	1712	1734
3	586	-563
2	1280	-1254
1	358	342
0	169	128
-1	426	391
-2	1246	1249
-3	380	385
-4	2056	2252
-5	815	-793
-6	1142	-1162
-7	404	384
-8	416	-431
-9	1088	1089
-10	896	898
-11	385	370
-12	269	-225
-13	296	-274
-14	907	-909
-15	83	-78
-16	225	227
-17	0	6
-18	209	189
-19	30	-41
-20	640	649
-21	443	-448
-22	157	-118
-23	112	-125
-24	35	-43
-25	228	229
-26	132	130
-27	155	143
-28	351	341
-29	163	-132
-30	210	-208
-31	59	50
-32	104	104
-33	61	55
-34	108	111
H,K= 3, 1		
L	FOBS	FCAL
32	213	224
31	43	-12
30	159	155
29	42	37
28	41	-51
27	40	29
26	39	-7
25	0	-20
24	570	556
23	233	-225
22	182	200
21	34	-6
20	66	-128
19	0	24
18	300	-305
17	233	235
16	961	946
15	50	-38
14	549	562
13	101	114
12	98	-85
11	206	179
10	193	-211
9	384	374
8	1672	1665
7	427	-410
6	1352	-1308
5	234	219
4	1073	-1062
3	375	373
2	366	-376
1	92	87
0	352	326
-1	433	-432
-2	1042	1030
-3	1074	-1091
-4	465	429
-5	609	-616
-6	501	-532
-7	542	532
-8	1186	1230
-9	194	-166
-10	0	4
-11	159	-144
-12	502	-471
-13	557	571
-14	283	277
-15	515	520
-16	1066	1068
-17	349	-335
-18	223	241
-19	322	-317
-20	510	542
-21	191	-186
-22	227	-241
-23	152	148
-24	312	328
-25	95	-107
-26	176	178
-27	84	-80
-28	0	15
-29	68	86
-30	99	-92
-31	199	188
-32	232	235
-33	0	-40
-34	117	102
H,K= 5, 1		
L	FOBS	FCAL
30	148	-169
29	97	95
28	121	119
27	0	30
26	319	326
25	0	-6
24	55	59
23	38	-54
22	413	-392
21	0	5
20	101	99
19	115	-125
18	0	-19
17	100	113
16	587	588
15	167	-160
14	321	-304
13	43	-35
12	199	210
11	0	17
10	804	799
9	215	-214
8	231	256
7	206	-208
6	965	-933
5	545	531
4	754	759
3	171	173
2	671	687
1	0	-21
0	730	746
-1	74	-77
-2	937	918
-3	91	-85
-4	500	508
-5	341	-350
-6	242	235
-7	399	-412
-8	1025	1040
-9	432	-438
-10	593	-602
-11	656	667
-12	321	325
-13	400	414
-14	438	415
-15	71	81
-16	667	680
-17	30	-8
-18	265	-263
-19	244	251
-20	73	-73
-21	34	-61
-22	34	-32
-23	136	-144
-24	270	272
-25	152	-163
-26	189	-180
-27	102	98
-28	0	-28
-29	0	18
-30	180	190
-31	0	-18
-32	198	200
-33	0	-30
H,K= 7, 1		
L	FOBS	FCAL
27	0	-18
26	44	-22
25	0	-29
24	119	-120
23	0	-2
22	81	58
21	56	56
20	359	351
19	0	-18
18	0	15
17	0	19
16	253	254
15	86	-103
14	0	-21
13	75	80
12	602	605
11	126	-107
10	448	461
9	196	-214
8	141	-136
7	30	24
6	0	-30
5	275	256
4	865	851
3	76	62
2	183	193
1	415	426
0	157	163
-1	257	263
-2	453	-453
-3	97	104
-4	443	424
-5	366	-369
-6	40	-71
-7	111	-116
-8	64	-70
-9	175	-182
-10	411	-436
-11	73	-65
-12	727	738
-13	157	-158
-14	261	274
-15	186	198
-16	33	-45
-17	133	127
-18	285	-292
-19	84	73
-20	339	341
-21	72	-93
-22	237	235
-23	0	-5
-24	66	-62
-25	0	-45
-26	0	-30
-27	40	-15
-28	380	381
-29	84	-76
-30	86	96
-31	0	5
H,K= 9, 1		
L	FOBS	FCAL
23	63	71
22	251	247
21	0	3
20	133	144
19	41	-43
18	153	-167
17	114	111
16	39	36
15	123	120
14	143	162
13	38	38
12	185	195
11	168	-158
10	316	-313
9	71	-74
8	50	43
7	130	-129
6	281	286
5	34	-32
4	498	515
3	115	-118
2	446	-449
1	164	165
0	33	-49
-1	103	104
-2	280	265
-3	203	203
-4	536	548
-5	103	-87
-6	451	-442
-7	0	-41
-8	293	290
-9	194	-206
-10	646	645
-11	96	-102
-12	397	404
-13	69	-88
-14	185	-190
-15	0	-12
-16	62	58
-17	0	6
-18	274	265
-19	145	151
-20	288	302
-21	0	13
-22	171	-174
-23	40	25
-24	100	98
-25	0	15
-26	178	187
-27	0	23
-28	115	122
-29	44	-44
H,K=11, 1		
L	FOBS	FCAL
17	125	133
16	295	308
15	0	-6
14	42	13
13	0	0
12	149	-173
11	0	31
10	70	47
9	69	58
8	596	604
7	203	-190
6	67	83
5	128	-135
4	0	36
3	137	-158
2	53	-69
1	135	133
0	487	493
-1	112	119
-2	203	199
-3	37	43
-4	139	-134
-5	53	50
-6	37	43
-7	186	187
-8	597	604
-9	0	-2
-10	0	8
-11	93	-103
-12	248	-252
-13	94	-91
-14	67	-71
-15	56	61
-16	245	255
-17	0	12
-18	40	-38
-19	0	-24
-20	0	5
-21	42	-39
-22	43	-41
-23	86	97
-24	239	245
-25	0	3
H,K=13, 1		
L	FOBS	FCAL
9	0	-2
8	44	68
7	87	-103
6	238	-248
5	0	10
4	121	134
3	0	-8
2	127	126
1	42	-49
0	42	21
-1	94	-105
-2	167	-181
-3	59	49
-4	72	87
-5	144	157
-6	236	228
-7	42	32
-8	59	81
-9	146	-125
-10	267	-268
-11	42	42
-12	95	93
-13	0	44
-14	105	104
-15	43	-27
-16	138	130
-17	132	-131
H,K= 0, 2		
L	FOBS	FCAL
0	1170	1153
-1	434	-418
-2	343	324
-3	546	-553
-4	434	-451
-5	589	573
-6	1272	1273
-7	220	251
-8	838	841
-9	238	218
-10	128	108
-11	601	584
-12	1030	1042
-13	522	-484
-14	290	-257
-15	822	841
-16	822	822
-17	430	-396
-18	348	-341
-19	270	-252
-20	257	-250
-21	80	-32
-22	139	-140
-23	360	379
-24	326	318
-25	115	-92
-26	53	-10
-27	66	-78
-28	39	47
-29	40	43
-30	109	104
-31	119	108
-32	183	186
-33	76	-85
H,K= 2, 2		
L	FOBS	FCAL
32	44	56
31	43	13
30	84	-82
29	131	127
28	171	170
27	142	-153
26	159	152
25	0	-8
24	426	421
23	344	-353
22	413	-416
21	342	334
20	322	315
19	0	12
18	590	598
17	68	-66
16	243	258
15	29	-17
14	406	-384
13	415	417
12	418	426
11	186	-187
10	548	563
9	58	80
8	656	666
7	361	340
6	1093	1041
5	204	-179
4	111	103
3	1301	1308
2	1350	1339
1	171	165
0	317	277
-1	1210	-1170
-2	2079	-2246
-3	795	749
-4	324	-300
-5	256	-271
-6	856	-825
-7	248	218
-8	767	775
-9	796	-804
-10	138	-173
-11	806	804
-12	1079	1106
-13	124	102
-14	285	305
-15	110	-105
-16	413	463
-17	704	-711
-18	288	-277
-19	135	124
-20	32	53
-21	126	127
-22	255	272
-23	239	-258
-24	180	190
-25	149	-146
-26	83	81
-27	148	158
-28	103	104
-29	144	143
-30	164	167
-31	59	-64
-32	135	122
-33	158	-141
H,K= 4, 2		
L	FOBS	FCAL
30	44	58
29	61	84
28	256	268
27	82	-83
26	40	-13
25	39	50
24	67	51
23	38	-62
22	164	146
21	254	243
20	742	743
19	59	-136
18	94	110
17	156	-164
16	648	-647
15	338	336
14	90	-76
14	90	-76
13	147	139
12	152	153
11	262	-258
10	297	-279
9	239	-239
8	58	-58
7	0	16
6	466	454
5	581	584
4	1731	1739
3	735	-735
2	219	194
1	82	-88
0	198	186
-1	368	-345
-2	974	-951
-3	650	629
-4	995	998
-5	1276	-1293
-6	23	-37
-7	269	-252
-8	527	531
-9	186	166
-10	263	236
-11	589	600
-12	1042	1067
-13	315	-311
-14	533	543
-15	175	-170
-16	310	-309
-17	543	548
-18	184	176
-19	64	62
-20	421	412
-21	337	-322
-22	182	177
-23	0	-5
-24	81	68
-25	111	110
-26	53	-70
-27	164	148
-28	194	214
-29	151	-152
-30	41	-46
-31	60	53
-32	43	-70
-33	0	34
H,K= 6, 2		
L	FOBS	FCAL
27	43	-49
26	135	-150
25	84	93
24	217	224
23	57	53
22	411	412
21	185	-188
20	75	90
19	165	-179
18	130	-145
17	154	191
16	278	279
15	76	78
14	223	236
13	173	-163
12	55	66
11	469	-479
10	562	-550
9	441	441
8	726	724
7	92	-86
6	790	798
5	135	-133
4	771	773
3	391	-390
2	655	-657
1	1200	1200
0	957	951
-1	0	-17
-2	601	588
-3	231	-231
-4	270	293
-5	0	14
-6	61	-45
-7	146	157
-8	39	-33
-9	40	42
-10	540	544
-11	277	-263
-12	29	13
-13	52	47
-14	635	-652
-15	483	486
-16	32	-11
-17	98	89
-18	453	470
-19	179	-175
-20	249	247
-21	132	-138
-22	247	-236
-23	205	204
-24	284	289
-25	221	-232
-26	78	84
-27	0	13
-28	135	144
-29	118	-112
-30	195	-204
-31	97	103
-32	88	82
H,K= 8, 2		
L	FOBS	FCAL
24	237	253
23	220	-215
22	74	-86
21	42	-18
20	0	30
19	209	200
18	336	342
17	155	143
16	488	494
15	252	-264
14	195	-196
13	269	244
12	168	177
11	0	-2
10	0	-29
9	208	218
8	544	542
7	446	-443
6	278	-276
5	32	-39
4	496	-516
3	439	434
2	243	232
1	208	207
0	704	699
-1	277	-273
-2	103	-110
-3	152	151
-4	170	-181
-5	168	160
-6	504	498
-7	339	-349
-8	401	401
-9	298	-304
-10	121	-134
-11	46	-44
-12	216	-218
-13	75	-81
-14	0	7
-15	243	235
-16	746	754
-17	262	-257
-18	88	-80
-19	137	133
-20	74	62
-21	65	-64
-22	94	100
-23	78	90
-24	380	371
-25	152	-136
-26	41	-74
-27	0	17
-28	0	-35
-29	0	10
-30	99	76
H,K=10, 2		
L	FOBS	FCAL
19	88	-71
18	130	130
17	95	-101
16	0	-28
15	0	52
14	41	-28
13	235	244
12	381	393
11	162	-160
10	78	99
9	148	-151
8	120	-128
7	163	-154
6	217	-212
5	0	-5
4	163	166
3	0	34
2	477	476
1	164	-169
0	0	-12
-1	0	9
-2	61	-64
-3	260	270
-4	608	607
-5	71	-89
-6	269	276
-7	0	-10
-8	62	45
-9	201	-204
-10	251	-248
-11	203	202
-12	513	517
-13	53	-65
-14	195	202
-15	93	95
-16	77	100
-17	129	-143
-18	229	-223
-19	187	183
-20	180	178
-21	58	60
-22	59	57
-23	0	-19
-24	95	-88
-25	43	-50
-26	76	-71
H,K=12, 2		
L	FOBS	FCAL
12	187	195
11	62	-71
10	0	-44
9	43	24
8	42	19
7	73	55
6	220	225
5	0	15
4	239	241
3	141	-151
2	107	-106
1	57	41
0	80	87
-1	69	76
-2	57	75
-3	250	247
-4	211	227
-5	165	-163
-6	283	-307
-7	57	70
-8	40	21
-9	0	31
-10	81	100
-11	158	148
-12	287	281
-13	210	-202
-14	93	-78
-15	73	-76
-16	0	-19
-17	85	83
-18	121	114
-19	0	31
-20	44	42
-21	77	-95
H,K= 1, 3		
L	FOBS	FCAL
32	44	-40
31	106	98
30	85	70
29	72	68
28	163	151
27	264	-250
26	95	-79
25	228	223
24	181	167
23	51	48
22	259	265
21	34	20
20	342	320
19	550	-558
18	335	-347
17	189	196
16	0	15
15	390	-392
14	103	-99
13	234	251
12	607	610
11	292	-282
10	562	582
9	25	-24
8	939	969
7	175	152
6	1205	1218
5	198	200
4	550	568
3	696	661
2	37	-10
1	303	-259
0	880	-850
-1	540	541
-2	303	-265
-3	1184	1184
-4	857	877
-5	580	-596
-6	209	212
-7	172	203
-8	645	654
-9	249	-237
-10	303	314
-11	418	416
-12	401	410
-13	589	-593
-14	443	-448
-15	377	353
-16	30	33
-17	96	-124
-18	0	-28
-19	495	497
-20	439	425
-21	310	-311
-22	130	120
-23	188	-190
-24	73	-81
-25	197	208
-26	179	179
-27	39	-31
-28	150	167
-29	309	-308
-30	73	-33
-31	0	15
-32	44	36
H,K= 3, 3		
L	FOBS	FCAL
30	44	38
29	61	-63
28	42	52
27	82	-69
26	0	20
25	345	347
24	457	443
23	125	-116
22	211	218
21	51	44
20	149	154
19	174	-172
18	164	-165
17	484	484
16	185	179
15	309	303
14	292	284
13	473	-449
12	183	-143
11	40	-52
10	123	124
9	498	464
8	649	647
7	0	5
6	498	459
5	175	-179
4	156	158
3	633	-593
2	326	-294
1	759	762
0	178	183
-1	492	468
-2	234	230
-3	0	-15
-4	395	387
-5	1545	-1581
-6	818	-808
-7	1312	1314
-8	831	853
-9	587	-591
-10	397	405
-11	787	-773
-12	61	71
-13	288	-302
-14	168	163
-15	320	313
-16	546	540
-17	69	-66
-18	470	473
-19	211	-208
-20	133	124
-21	59	-61
-22	0	-47
-23	345	356
-24	142	143
-25	92	83
-26	149	148
-27	162	-168
-28	40	-20
-29	41	-29
-30	59	-60
-31	196	218
-32	180	189
H,K= 5, 3		
L	FOBS	FCAL
28	44	47
27	106	120
26	153	158
25	59	-49
24	115	137
23	191	-208
22	0	-21
21	54	77
20	65	68
19	251	256
18	237	238
17	198	204
16	305	307
15	517	-518
14	161	-162
13	174	-183
12	319	-325
11	634	636
10	304	317
9	115	-111
8	446	434
7	1433	-1427
6	441	-402
5	531	534
4	629	661
3	61	-80
2	637	655
1	332	334
0	1087	1093
-1	975	-984
-2	53	43
-3	183	208
-4	0	4
-5	753	742
-6	201	208
-7	448	465
-8	426	432
-9	701	-703
-10	194	-183
-11	268	251
-12	29	31
-13	633	635
-14	637	641
-15	238	-236
-16	329	339
-17	389	-387
-18	100	-94
-19	131	131
-20	69	-61
-21	200	199
-22	63	59
-23	165	163
-24	196	198
-25	250	-259
-26	147	-143
-27	213	203
-28	92	89
-29	0	22
-30	74	83
-31	0	21
H,K= 7, 3		
L	FOBS	FCAL
25	233	-241
24	173	-158
23	165	151
22	102	102
21	148	151
20	348	339
19	230	-209
18	272	274
17	126	-151
16	159	170
15	37	-35
14	51	-53
13	685	695
12	392	390
11	305	-305
10	0	-13
9	260	-270
8	189	-192
7	165	171
6	78	-75
5	948	959
4	913	920
3	671	-671
2	384	379
1	347	-351
0	0	27
-1	265	268
-2	0	9
-3	472	471
-4	236	223
-5	129	-131
-6	43	40
-7	315	-303
-8	313	-324
-9	396	410
-10	71	75
-11	440	447
-12	467	483
-13	314	-331
-14	180	177
-15	254	-260
-16	34	-2
-17	218	-222
-18	159	-165
-19	268	266
-20	288	297
-21	286	-291
-22	76	70
-23	78	-56
-24	125	102
-25	90	-71
-26	71	42
-27	229	223
-28	229	244
-29	87	-95
-30	44	61
H,K= 9, 3		
L	FOBS	FCAL
21	133	-137
20	144	152
19	227	-217
18	60	-35
17	330	339
16	235	243
15	57	13
14	164	176
13	39	-56
12	86	76
11	244	-260
10	322	-320
9	474	474
8	64	45
7	193	189
6	279	266
5	250	-247
4	170	174
3	320	-298
2	60	-53
1	92	100
0	109	54
-1	69	94
-2	321	318
-3	424	-420
-4	124	113
-5	396	-393
-6	147	-140
-7	336	339
-8	331	328
-9	50	57
-10	371	383
-11	0	2
-12	322	311
-13	281	-293
-14	122	-133
-15	306	288
-16	113	115
-17	0	-34
-18	0	23
-19	117	114
-20	168	166
-21	279	-259
-22	41	-43
-23	72	89
-24	73	80
-25	121	124
-26	174	165
-27	44	-2
H,K=11, 3		
L	FOBS	FCAL
15	269	-274
14	44	36
13	97	-104
12	60	-55
11	158	160
10	125	136
9	41	33
8	232	233
7	163	-161
6	57	68
5	0	24
4	0	24
3	112	121
2	88	86
1	135	136
0	210	220
-1	129	-143
-2	55	67
-3	39	-54
-4	39	42
-5	0	-46
-6	0	19
-7	348	351
-8	382	381
-9	288	-289
-10	0	9
-11	68	61
-12	40	-4
-13	69	62
-14	57	-46
-15	264	257
-16	82	106
-17	59	-54
-18	42	25
-19	60	-81
-20	0	-32
-21	0	10
-22	44	23
H,K=13, 3		
L	FOBS	FCAL
4	76	84
3	0	20
2	0	35
1	97	123
0	87	95
-1	156	-154
-2	86	-89
-3	122	144
-4	43	66
-5	0	-5
-6	86	81
-7	61	-54
-8	97	85
-9	260	-259
-10	87	-90
-11	62	84
-12	0	29
-13	76	83
H,K= 0, 4		
L	FOBS	FCAL
0	1211	1207
-1	1170	-1208
-2	596	589
-3	350	340
-4	178	201
-5	207	188
-6	0	-10
-7	210	228
-8	180	175
-9	130	-141
-10	117	119
-11	113	129
-12	0	-29
-13	59	-61
-14	0	-15
-15	658	662
-16	384	392
-17	0	-17
-18	315	310
-19	233	-232
-20	60	-53
-21	213	207

ment in R nor any substantial change in coordinates. The results are not reported because parameters were oscillating; further cycles to attain convergence (at 0·9 hours per cycle with IBM 7090) seemed unjustified.

Standard deviations of coordinates estimated by least-squares, with neglect of the possible error in the hydrogen parameters, are reported in Table 2. The similar estimates of standard deviations of the anisotropic thermal parameters range from 2 to 4% of the diagonal elements of the β matrices. They are not reported in detail because the effects of systematic errors in the data and of coupling with hydrogen parameters are of unknown and possibly considerably larger magnitude.

The standard deviations of hydrogen coordinates correspond to about 0·05 Å when estimated by least squares with neglect of errors in heavy atom parame-

Table 1 (*cont.*)

-22	51	-42
-23	286	270
-24	198	203
-25	309	-302
-26	164	163
-27	128	-121
-28	0	-12
-29	85	-79
-30	0	5
-31	193	184

H,K= 2, 4

L	FOBS	FCAL
30	44	24
29	87	91
28	60	82
27	0	21
26	0	-1
25	0	3
24	68	-49
23	226	-237
22	53	35
21	312	310
20	343	344
19	195	205
18	374	367
17	385	-384
16	236	233
15	383	-386
14	177	188
13	631	643
12	341	330
11	283	-277
10	0	-38
9	585	585
8	0	-29
7	360	-363
6	60	26
5	440	447
4	368	398
3	533	490
2	619	612
1	321	330
0	399	392
-1	948	-936
-2	171	-166
-3	831	864
-4	135	122
-5	559	545
-6	456	479
-7	298	-299
-8	227	217
-9	544	-537
-10	62	-58
-11	0	-47
-12	184	200
-13	0	34
-14	428	447
-15	279	-285
-16	71	93
-17	549	-561
-18	220	-222
-19	447	449
-20	120	114
-21	146	144
-22	346	345
-23	0	-5
-24	227	203
-25	346	-347
-26	40	17
-27	162	160
-28	124	115
-29	0	-8
-30	149	147
-31	44	-54

H,K= 4, 4

L	FOBS	FCAL
28	0	14
27	106	-108
26	0	-18
25	42	24
24	71	68
23	126	113
22	78	83
21	491	500
20	225	213
19	370	-361
18	157	168
17	132	-137
16	151	160
15	201	195
14	137	132
13	492	504
12	178	135
11	621	-630
10	0	25
9	0	10
8	137	119
7	51	48
6	294	291
5	372	357
4	400	382
3	823	-846
2	0	-3
1	807	-761
0	214	-205
-1	242	240
-2	153	175
-3	622	590
-4	465	452
-5	314	-298
-6	314	309
-7	233	-258
-8	341	334
-9	148	148

-10	599	603
-11	493	490
-12	575	568
-13	887	-889
-14	122	-123
-15	240	-234
-16	224	-234
-17	238	243
-18	113	118
-19	541	534
-20	256	247
-21	314	-299
-22	0	-2
-23	192	178
-24	94	57
-25	88	88
-26	150	145
-27	153	180
-28	125	120
-29	154	-165
-30	43	-49
-31	108	-105

H,K= 6, 4

L	FOBS	FCAL
25	240	244
24	43	33
23	0	-2
22	59	74
21	158	-151
20	69	78
19	341	-323
18	0	-17
17	360	375
16	37	42
15	0	-8
14	161	144
13	253	-268
12	184	181
11	291	-301
10	200	189
9	581	570
8	373	394
7	282	283
6	429	434
5	658	-656
4	89	93
3	513	-503
2	111	-100
1	724	727
0	159	170
-1	86	[illegible]
-2	175	-163
-3	0	[illegible]
-4	0	41
-5	247	244
-6	881	879
-7	496	507
-8	136	136
-9	206	198
-10	[illegible]	[illegible]
-11	[illegible]	[illegible]
-12	46	-40
-13	618	-616
-14	75	-78
-15	514	511
-16	159	182
-17	35	-41
-18	72	95
-19	258	-266
-20	0	22
-21	131	-144
-22	77	94
-23	263	261
-24	169	172
-25	41	-7
-26	41	27
-27	84	-83
-28	0	-11
-29	116	-118

H,K= 8, 4

L	FOBS	FCAL
21	0	16
20	61	-30
19	268	274
18	59	56
17	0	-6
16	114	112
15	311	-314
14	56	58
13	164	158
12	93	98
11	299	301
10	193	191
9	322	317
8	277	263
7	281	-296
6	101	103
5	61	-38
4	0	37
3	374	357
2	77	96
1	178	174
0	137	118
-1	914	-914
-2	48	-28
-3	201	205
-4	167	182
-5	108	119
-6	149	162
-7	154	147
-8	60	64
-9	357	-369
-10	35	14
-11	61	67

-12	156	161
-13	81	77
-14	146	149
-15	133	135
-16	167	162
-17	403	-407
-18	167	155
-19	135	-149
-20	177	166
-21	113	116
-22	71	68
-23	72	70
-24	0	7
-25	121	-126
-26	43	5
-27	0	27

H,K=10, 4

L	FOBS	FCAL
16	0	-1
15	0	13
14	0	-15
13	175	203
12	94	89
11	223	-223
10	41	58
9	0	-29
8	57	53
7	98	-100
6	0	14
5	366	366
4	160	139
3	55	-67
2	196	187
1	245	-239
0	209	221
-1	312	-313
-2	114	120
-3	284	282
-4	76	91
-5	194	-193
-6	54	51
-7	148	-149
-8	66	72
-9	0	24
-10	39	20
-11	344	349
-12	68	44
-13	153	139
-14	120	131
-15	90	-92
-16	168	158
-17	59	51
-18	173	184
-19	0	-9
-20	43	-11
-21	0	22
-22	0	-60
-23	[illegible]	[illegible]

H,K=12, 4

L	FOBS	FCAL
8	44	-16
7	139	124
6	0	59
5	179	191
4	161	170
3	96	-105
2	128	131
1	60	75
0	42	52
-1	127	124
-2	60	47
-3	42	38
-4	111	94
-5	206	-227
-6	42	34
-7	60	-27
-8	85	-68
-9	175	174
-10	0	12
-11	121	130
-12	167	158
-13	216	-201
-14	62	80
-15	0	14
-16	44	-28

H,K= 1, 5

L	FOBS	FCAL
29	0	49
28	75	-61
27	316	-318
26	42	1
25	130	134
24	120	122
23	289	293
22	149	144
21	278	-275
20	52	62
19	367	-378
18	138	137
17	0	27
16	249	253
15	342	345
14	195	192
13	32	32
12	0	-23
11	639	-637
10	219	211
9	537	553
8	94	108
7	102	102
6	104	102
5	322	315
4	113	-114

3	1287	-1309
2	375	403
1	618	618
0	287	298
-1	505	498
-2	499	493
-3	702	732
-4	460	464
-5	766	-767
-6	468	475
-7	283	292
-8	29	-29
-9	875	868
-10	187	-188
-11	351	-369
-12	0	38
-13	362	-368
-14	33	27
-15	47	40
-16	0	7
-17	277	278
-18	255	275
-19	172	168
-20	323	328
-21	522	-523
-22	215	195
-23	87	-73
-24	40	21
-25	99	101
-26	82	75
-27	133	134
-28	74	41
-29	368	-371

H,K= 3, 5

L	FOBS	FCAL
27	98	103
26	105	101
25	158	173
24	0	-13
23	129	-124
22	0	-19
21	175	-177
20	38	60
19	0	5
18	212	214
17	506	511
16	123	131
15	344	-251
14	327	340
13	34	[illegible]
12	408	414
11	33	53
10	375	375
9	1087	1091
8	132	-160
7	781	-799
6	0	-29
5	30	-1
4	202	-207
3	42	34
2	248	236
1	307	309
0	0	-5
-1	[illegible]	[illegible]
-2	593	608
-3	783	-800
-4	546	554
-5	0	-10
-6	351	353
-7	543	542
-8	177	183
-9	370	-371
-10	70	79
-11	666	-676
-12	111	-120
-13	149	151
-14	0	-5
-15	554	558
-16	249	244
-17	394	-394
-18	312	315
-19	0	19
-20	0	5
-21	281	-281
-22	127	128
-23	442	444
-24	80	79
-25	152	-141
-26	41	37
-27	152	-144
-28	0	-23
-29	0	8

H,K= 5, 5

L	FOBS	FCAL
25	62	-59
24	61	14
23	154	-164
22	42	31
21	267	262
20	114	-107
19	105	-99
18	55	40
17	207	190
16	146	124
15	596	-605
14	361	365
13	157	168
12	283	277
11	254	260
10	209	204
9	225	-234
8	111	99
7	467	-465
6	33	-10

5	399	403
4	128	-137
3	247	239
2	84	102
1	438	-470
0	233	253
-1	169	-160
-2	163	176
-3	0	-11
-4	307	310
-5	241	221
-6	365	369
-7	189	190
-8	0	-2
-9	684	-675
-10	123	75
-11	649	667
-12	126	126
-13	118	110
-14	261	264
-15	222	169
-16	36	3
-17	407	-419
-18	52	45
-19	345	324
-20	54	53
-21	145	134
-22	68	80
-23	40	19
-24	115	-65
-25	212	-214
-26	84	50
-27	0	18
-28	124	142

H,K= 7, 5

L	FOBS	FCAL
22	0	-34
21	185	198
20	43	-39
19	190	-196
18	72	70
17	41	-55
16	0	20
15	120	-128
14	142	131
13	190	190
12	0	-8
11	14	166
10	340	-339
9	355	364
8	179	177
7	448	439
6	122	125
5	178	-185
4	104	102
3	0	-25
2	0	5
1	[illegible]	[illegible]
0	[illegible]	[illegible]
-1	34	-39
-2	146	154
-3	527	512
-4	0	3
-5	366	-387
-6	0	13
-7	130	146
-8	93	76
-9	176	-166
-10	375	374
-11	379	398
-12	125	127
-13	361	-366
-14	104	105
-15	205	-205
-16	125	125
-17	38	-51
-18	160	146
-19	163	155
-20	0	-23
-21	0	-4
-22	0	-18
-23	201	-189
-24	112	110
-25	75	61
-26	124	121

H,K= 9, 5

L	FOBS	FCAL
17	89	98
16	0	-3
15	156	158
14	0	24
13	73	-97
12	0	14
11	206	-205
10	122	111
9	156	152
8	187	182
7	131	128
6	180	191
5	95	84
4	102	81
3	515	-504
2	199	186
1	194	198
0	0	25
-1	54	-53
-2	0	-15
-3	100	106
-4	53	-52
-5	456	-447
-6	221	220
-7	232	243
-8	133	147
-9	149	133

-10	67	78
-11	110	-159
-12	111	94
-13	189	-190
-14	164	158
-15	57	-52
-16	41	43
-17	159	169
-18	0	-38
-19	0	28
-20	43	55
-21	114	-114
-22	115	133
-23	146	128

H,K=11, 5

L	FOBS	FCAL
9	257	267
8	44	48
7	281	-285
6	143	156
5	0	-23
4	85	103
3	104	-110
2	60	77
1	279	278
0	0	-31
-1	72	-77
-2	93	85
-3	204	-198
-4	132	128
-5	172	158
-6	42	57
-7	310	298
-8	42	19
-9	59	-27
-10	84	95
-11	170	-182
-12	60	46
-13	96	98
-14	61	-71
-15	163	160
-16	44	-16
-17	117	-111

H,K= 0, 6

L	FOBS	FCAL
0	372	-353
-1	894	-886
-2	514	508
-3	224	-211
-4	420	442
-5	105	-106
-6	449	457
-7	368	-380
-8	212	-242
-9	188	177
-10	58	-33
-11	346	336
-12	[illegible]	[illegible]
-13	[illegible]	[illegible]
-14	375	386
-15	419	422
-16	73	-73
-17	369	-375
-18	164	163
-19	288	-301
-20	151	158
-21	119	125
-22	81	3
-23	281	269
-24	151	-133
-25	185	-200
-26	106	132
-27	0	-1

H,K= 2, 6

L	FOBS	FCAL
26	0	9
25	137	-138
24	60	-64
23	272	-278
22	189	184
21	286	281
20	56	-47
19	203	199
18	77	-86
17	387	-379
16	278	275
15	151	-163
14	473	470
13	610	614
12	167	-168
11	0	-34
10	0	11
9	67	-73
8	119	134
7	167	-157
6	391	408
5	628	625
4	0	9
3	231	230
2	109	112
1	361	-380
0	357	380
-1	593	-594
-2	609	633
-3	661	684
-4	283	-284
-5	106	102
-6	150	-148
-7	341	-347
-8	219	235
-9	136	-127
-10	367	373
-11	486	491

-12	34	38
-13	0	22
-14	202	209
-15	250	-266
-16	189	174
-17	433	-419
-18	293	286
-19	241	250
-20	55	-52
-21	56	-61
-22	0	21
-23	108	-106
-24	42	41
-25	175	-176
-26	136	138
-27	158	159

H,K= 4, 6

L	FOBS	FCAL
24	63	72
23	130	129
22	60	72
21	188	185
20	41	-47
19	307	-320
18	233	230
17	56	-49
16	155	160
15	267	269
14	0	-18
13	153	168
12	0	-26
11	461	-468
10	409	421
9	231	228
8	70	67
7	236	250
6	34	56
5	148	140
4	106	95
3	532	-545
2	408	415
1	128	-113
0	93	96
-1	104	96
-2	47	39
-3	0	-20
-4	491	-496
-5	349	355
-6	0	-24
-7	0	71
-8	76	92
-9	76	-76
-10	134	129
-11	342	333
-12	0	-71
-13	335	-338
-14	173	174
-15	52	71
-16	74	63
-17	242	242
-18	[illegible]	[illegible]
-19	233	221
-20	39	-21
-21	238	-243
-22	141	155
-23	0	-31
-24	60	78
-25	0	18
-26	62	80
-27	77	83

H,K= 6, 6

L	FOBS	FCAL
21	117	-106
20	0	43
19	149	-154
18	120	58
17	204	200
16	71	-76
15	122	-131
14	120	130
13	142	-139
12	78	81
11	176	-180
10	147	173
9	238	252
8	53	70
7	0	43
6	172	141
5	166	-187
4	0	7
3	95	-95
2	87	91
1	507	507
0	71	-58
-1	0	13
-2	271	267
-3	0	-11
-4	162	170
-5	133	-121
-6	262	247
-7	160	168
-8	88	103
-9	89	-74
-10	73	85
-11	37	-24
-12	149	-129
-13	271	-258
-14	157	165
-15	378	374
-16	0	15
-17	197	191
-18	105	57
-19	114	-125
-20	164	147

-21	138	-133
-22	227	217
-23	86	74
-24	0	16
-25	62	-63

H,K= 8, 6

L	FOBS	FCAL
17	77	77
16	0	1
15	106	-137
14	96	104
13	60	50
12	94	93
11	0	26
10	278	278
9	141	154
8	40	25
7	211	-207
6	0	-15
5	130	-145
4	68	60
3	236	235
2	122	106
1	374	365
0	86	-89
-1	294	-292
-2	245	261
-3	54	75
-4	223	217
-5	38	41
-6	243	237
-7	0	23
-8	0	0
-9	365	-356
-10	39	49
-11	209	-214
-12	56	55
-13	57	48
-14	99	98
-15	191	204
-16	41	-50
-17	172	-170
-18	179	173
-19	43	-38
-20	129	131
-21	131	123
-22	44	22

H,K=10, 6

L	FOBS	FCAL
9	146	-149
8	0	-15
7	267	-269
6	178	178
5	148	158
4	5	-8
3	42	59
2	94	-77
1	178	-169
0	0	-21
-1	162	-163
-2	283	276
-3	364	354
-4	[illegible]	[illegible]
-5	59	65
-6	0	13
-7	162	-168
-8	59	46
-9	119	-123
-10	112	117
-11	225	209
-12	0	-26
-13	0	38
-14	0	30
-15	131	-125
-16	76	69

-12	117	-118
-13	314	-297
-14	522	536
-15	230	246
-16	55	12
-17	39	-36
-18	113	124
-19	0	20
-20	116	-130
-21	177	-198
-22	159	170
-23	0	-43
-24	170	164

H,K= 1, 7

L	FOBS	FCAL
24	63	82
23	75	86
22	0	-1
21	42	59
20	200	-204
19	340	-333
18	274	282
17	69	69
16	78	-71
15	102	118
14	38	-55
13	100	108
12	118	-124
11	303	-370
10	548	553
9	36	-39
8	433	431
7	326	325
6	86	-113
5	35	-34
4	0	-34
3	201	-200
2	215	227
1	59	68
0	168	179
-1	414	425
-2	235	-244
-3	119	112
-4	278	-286
-5	429	-435
-6	669	674
-7	211	209
-8	215	221
-9	147	136
-10	63	74
-11	0	15

H,K= 3, 7

L	FOBS	FCAL
22	125	133
21	144	-151
20	74	79
19	0	18
18	72	91
17	338	347
16	210	-200
15	80	54
14	68	-54
13	295	-302
12	435	431
11	126	123
10	209	203
9	257	260
8	127	-128
7	203	-197
6	284	292
5	160	-164
4	333	335
3	100	106
2	247	249
1	384	369
0	136	-137
-1	86	-96
-2	244	258
-3	122	-124
-4	35	48
-5	375	-383
-6	458	477
-7	329	321
-8	486	-505
-9	277	-273
-10	137	145
-11	105	-97
-12	257	275
-13	53	64
-14	236	225
-15	145	155

H,K= 5, 7

L	FOBS	FCAL
19	181	177
18	43	38
17	0	3
16	0	11
15	182	-183
14	170	170
13	41	43
12	98	102
11	164	175
10	118	-137
9	183	-182
8	116	-106
7	450	-457
6	451	456
5	309	297
4	155	-167
3	37	48
2	306	317
1	148	155
0	83	82
-1	276	-273
-2	383	379
-3	0	10
-4	0	-4
-5	64	58
-6	0	-47
-7	65	88
-8	258	-256
-9	204	-208
-10	153	168
-11	54	60
-12	123	137
-13	337	330
-14	40	-37
-15	89	71
-16	151	148
-17	153	-143
-18	203	206
-19	119	-93
-20	95	81
-21	61	65
-22	0	-4
-23	63	60

11	164	-160
10	42	-14
9	124	-132
8	41	-29
7	82	-8
6	243	226
5	362	367
4	170	-158
3	98	-99
2	235	242
1	125	-111
0	219	220
-1	39	48
-2	239	232
-3	219	226
-4	39	20
-5	0	-8
-6	56	-59
-7	305	-300
-8	178	168
-9	0	12
-10	121	136
-11	226	226
-12	41	-28
-13	41	-23
-14	59	81
-15	178	-186
-16	104	108
-17	113	-102
-18	188	190
-19	223	231
-20	132	-131

H,K= 9, 7

L	FOBS	FCAL
9	99	120
8	88	72
7	98	119
6	0	-40
5	106	-102
4	0	-44
3	227	-218
2	165	173
1	0	25
0	180	187
-1	243	252
-2	85	-72
-3	104	-101
-4	0	-5
-5	296	-274
-6	275	264
-7	120	114
-8	85	90
-9	128	114
-10	0	23
-11	75	-87
-12	61	-74
-13	255	-265
-14	197	195
-15	172	174

H,K= 7, 7

L	FOBS	FCAL
15	0	-0
14	222	231
13	240	234
12	171	-160

-9	129	-126
-10	544	546
-11	97	89
-12	89	74
-13	40	-56
-14	41	54
-15	92	-98
-16	59	-62
-17	73	-75
-18	196	199
-19	0	27
-20	0	-14
-21	77	67

H,K= 0, 8

L	FOBS	FCAL
0	328	-321
-1	254	-216
-2	150	153
-3	234	-226
-4	296	287
-5	113	86
-6	0	[illegible]
-7	85	53
-8	102	-109
-9	129	-122
-10	260	253
-11	112	88
-12	40	37
-13	114	108
-14	318	330
-15	143	143
-16	270	-270
-17	146	-150
-18	121	93
-19	61	-29
-20	152	147
-21	44	57

H,K= 2, 8

L	FOBS	FCAL
19	98	112
18	97	-95
17	96	-97
16	0	17
15	162	-175
14	293	285
13	92	93
12	146	-143
11	40	26
10	0	-29
9	118	-136
8	55	70
7	0	-34
6	306	306
5	192	202
4	331	322
3	255	250
2	66	-78
1	65	-63
0	93	-92
-1	151	-140
-2	480	487
-3	178	163
-4	165	-170
-5	0	-28
-6	0	31
-7	38	-66
-8	0	0

H,K= 4, 8

L	FOBS	FCAL
17	0	-46
16	287	278
15	189	194
14	43	-46
13	42	51
12	237	-221
11	161	-161
10	136	136
9	82	-85
8	295	292
7	90	92
6	271	274
5	79	56
4	177	-177
3	222	-213
2	271	264
1	55	-44
0	135	133
-1	39	38
-2	110	172
-3	170	169
-4	434	-435
-5	78	-66
-6	167	167
-7	39	-37
-8	254	235
-9	0	6
-10	251	246
-11	81	80
-12	168	-167
-13	71	-77
-14	42	45
-15	94	-160
-16	220	229
-17	43	51
-18	43	-32
-19	98	102

H,K= 6, 8

L	FOBS	FCAL
13	44	-65
12	0	-21
11	0	-50
10	353	344
9	142	126
8	95	-81
7	42	-28
6	73	-67
5	156	-153
4	41	23
3	189	-180
2	504	513
1	193	180
0	209	-204
-1	91	100
-2	142	-120
-3	0	-22
-4	82	82
-5	41	-32
-6	263	266
-7	92	93
-8	83	72
-9	177	164
-10	103	-124
-11	94	-82
-12	42	40
-13	165	-160
-14	317	328
-15	75	79
-16	0	-7
-17	77	84

H,K= 8, 8

L	FOBS	FCAL
5	88	-78
4	275	279
3	124	130
2	0	-9
1	75	79
0	168	-168
-1	168	-163
-2	274	293
-3	123	-107
-4	264	252
-5	106	112
-6	61	-61
-7	44	32
-8	124	-117
-9	158	-157
-10	132	129
-11	44	-37

9	0	23
8	0	45
7	110	117
6	101	100
5	92	-88
4	213	-220
3	142	-141
2	227	232
1	41	48
0	71	89
-1	195	207
-2	153	157
-3	0	-0
-4	169	-180
-5	92	-91
-6	350	351
-7	41	19
-8	246	253
-9	73	57
-10	60	-68
-11	74	-81
-12	121	-110
-13	106	-101
-14	75	87
-15	0	29

H,K= 3, 9

L	FOBS	FCAL
12	153	142
11	98	98
10	363	369
9	74	30
8	234	-234
7	85	-89
6	42	30
5	73	-92
4	42	52
3	72	102
2	256	257
1	72	82
0	0	-5
-1	41	-43
-2	138	-133
-3	72	-48
-4	239	221
-5	42	41
-6	490	488
-7	0	-1
-8	119	-116
-9	73	-83
-10	135	-143
-11	61	-70
-12	184	186
-13	0	25
-14	152	163
-15	44	40

H,K= 5, 9

L	FOBS	FCAL
8	166	-152
7	44	35
6	347	354
5	43	31
4	115	106
3	0	13
2	178	-167
1	43	-40
0	142	-133
-1	0	-25
-2	219	201
-3	43	18
-4	227	218
-5	0	17
-6	86	-96
-7	0	-1
-8	242	-237
-9	44	34
-10	409	410
-11	0	18

H,K= 1, 9

L	FOBS	FCAL
15	77	77
14	0	-29
13	0	-8
12	183	-180
11	74	-76
10	240	240

H,K= 0,10

L	FOBS	FCAL
0	334	-331
-1	76	66
-2	271	268
-3	76	-81
-4	159	154
-5	125	66

H,K= 2,10

L	FOBS	FCAL
2	44	-46
1	147	-141
0	44	-10
-1	63	28
-2	388	408
-3	0	-14

ters. The least-square results and the Fourier synthesis results (Fig. 1) disagree in several cases by about 0·1 Å. The best evidence of the accuracy of these coordinates comes from the O–H distances which are expected to be somewhere in the range 0·9 to 1·0 Å, depending on the magnitude of the thermal motion effect. We find values 0·7 to 1·1 Å for eleven such bonds and 0·5 Å for O(7)–H(6). This scatter suggests 0·2 Å as the standard deviation of position of hydrogen, including the effects of systematic errors in the data.

The standard deviations of isotropic thermal parameters of hydrogen are estimated by least squares to range from 0·7 Å^2 for H(11) to 1·5 Å^2 for H(6). The scatter from the average value of 3·4 Å^2 corresponds to σ=1·8 Å^2. This latter value may be a better esti-

Table 2. *Atomic coordinates in* $MgSO_4.6H_2O$

	Atom	x	y	z
	Mg(1)	0	0	0
	Mg(2)	0	0·9425	¼
	S	0·8659	0·4490	0·1241
Sulfate oxygens	O(1)	0·7747	0·5986	0·1361
	O(2)	0·9834	0·4442	0·1665
	O(3)	0·9069	0·4854	0·0692
	O(4)	0·7961	0·2688	0·1211
Water about Mg(1)	O(5)	0·5852	0·7125	0·0483
	O(6)	0·5393	0·3162	0·0640
	O(7)	0·3126	0·5464	0·0231
Water about Mg(2)	O(8)	0·8887	0·1486	0·2820
	O(9)	0·8884	0·7405	0·2811
	O(10)	0·8598	0·9435	0·1799
Water (5)	H(1)	0·643	0·698	0·076
	H(2)	0·539	0·806	0·045
Water (6)	H(3)	0·495	0·232	0·060
	H(4)	0·614	0·340	0·093
Water (7)	H(5)	0·304	0·650	0·055
	H(6)	0·271	0·544	0·014
Water (8)	H(7)	0·840	0·092	0·312
	H(8)	0·937	0·235	0·296
Water (9)	H(9)	0·940	0·651	0·291
	H(10)	0·820	0·733	0·275
Water (10)	H(11)	0·849	0·831	0·164
	H(12)	0·847	0·033	0·164

Estimated standard deviations are:
for Mg(2), $\sigma(y)=0·0002$;
for *S*, $\sigma(x)=0·00006$, $\sigma(y)=0·00008$, $\sigma(z)=0·00002$;
for oxygen, $\sigma(x)=0·0002$, $\sigma(y)=0·0003$, $\sigma(z)=0·0001$.

Table 3. *Anisotropic thermal parameters (each multiplied by* 10^4*) and root mean square amplitudes of vibration in* $MgSO_4.6H_2O$

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	u (Å)
Mg(1)	45	86	8·7	2	−1	−3	0·16
Mg(2)	46	72	7·8	(0)*	2	(0)*	0·15
S	46	72	9·8	0	−1	−1	0·16
O(1)	64	103	12·4	23	4	−1	0·18
O(2)	79	107	17·6	10	−17	−5	0·21
O(3)	81	120	13·2	0	10	−1	0·19
O(4)	68	85	15·4	−19	−4	6	0·19
O(5)	67	129	13·6	17	−5	−12	0·19
O(6)	87	175	19·0	−51	−15	26	0·23
O(7)	54	216	16·7	7	3	−21	0·21
O(8)	67	94	12·6	−2	6	−7	0·18
O(9)	61	113	16·0	−5	5	12	0·19
O(10)	103	103	10·7	−8	−8	2	0·20

* Zero because of twofold axis.

Table 4. *Isotropic thermal parameters for* H *in* $MgSo_4.6H_2O$

Atom	B	Atom	B	Atom	B
H(1)	1·9 Å²	H(5)	5·6 Å²	H(9)	5·9 Å²
H(2)	5·2	H(6)	1·9	H(10)	3·1
H(3)	1·9	H(7)	4·3	H(11)	2·6
H(4)	5·7	H(8)	1·3	H(12)	2·0

mate of the true accuracy, since there is no obvious reason for the thermal motions of the various hydrogen atoms to be substantially different.

Discussion

The structure of $MgSO_4.6H_2O$ differs very slightly from that already described for $CoSO_4.6H_2O$ (Zalkin, Ruben & Templeton, 1962). The hydrogen bonds which were assigned in the previous study on the basis of oxygen positions are fully confirmed by the present results.

Table 5 lists some interatomic distances and their standard deviations, with no correction for thermal motion. The S–O bond distances in the sulfate ion average 1·473 Å with an average deviation of 0·007 Å. When a librational correction is made assuming the oxygen atoms to ride on the sulfur atom, the average becomes 1·486 Å with an average deviation of 0·004

Table 5. *Interatomic distances and standard deviations in* $MgSO_4.6H_2O$

Water octahedron about Mg(1)

Atoms	Distance	σ
Mg(1)–2 O(5)	2·046 Å	0·002 Å
–2 O(6)	2·044	0·003
–2 O(7)	2·080	0·002
O(5)–O(6)	2·930	0·004
–O(6)	2·854	0·004
–O(7)	2·986	0·003
–O(7)	2·847	0·003
O(6)–O(7)	2·889	0·004
–O(7)	2·943	0·003

Water octahedron about Mg(2)

Atoms	Distance	σ
Mg(2)–2 O(8)	2·083 Å	0·002 Å
–2 O(9)	2·054	0·002
–2 O(10)	2·059	0·003
O(8)–O(8)	2·917	0·004
–O(9)	2·943	0·003
–O(10)	2·876	0·003
–O(10)	2·973	0·003
O(9)–O(9)	2·896	0·003
–O(10)	2·850	0·003
–O(10)	2·972	0·003

Sulfate ion

Atoms	Distance	σ
S–O(1)	1·476 Å	0·002 Å
–O(2)	1·460	0·003
–O(3)	1·482	0·002
–O(4)	1·475	0·002

Hydrogen bonds

Atoms	Distance	σ
O(5) –H(1) ··· O(1)	2·785 Å	0·003 Å
O(5) –H(2) ··· O(3)	2·766	0·003
O(6) –H(3) ··· O(3)	2·747	0·003
O(6) –H(4) ··· O(4)	2·785	0·004
O(7) –H(5) ··· O(4)	2·906	0·003
O(7) –H(6) ··· O(3)	2·934	0·004
O(8) –H(7) ··· O(1)	2·796	0·003
O(8) –H(8) ··· O(2)	2·707	0·003
O(9) –H(9) ··· O(2)	2·721	0·003
O(9) –H(10) ··· O(8)	3·068	0·003
O(10)–H(11) ··· O(1)	2·794	0·003
O(10)–H(12) ··· O(4)	2·777	0·003

Å. In the Tutton salt $Mg(NH_4)_2(SO_4)_2 . 6H_2O$ Margulis & Templeton (1962) also found 1·486 Å for the average S–O distance when it was corrected in the same way.

The average Mg–O distance is 2·06 Å without correction for thermal motion. Magnesium is also in water octahedra in $Mg(NH_4)_2(SO_4)_2 . 6H_2O$ with average Mg–O = 2·07 Å and in $Ce_2Mg_3(NO_3)_{12} . 24H_2O$ (Templeton, Zalkin & Forrester, 1962) with average Mg–O = 2·06 Å. In all three crystals the average Mg–O distance is 2·07 Å with thermal correction assuming in-phase motion.

The hydrogen bond O–O distances average 2·82 Å, but scatter over a considerable range as is common in complicated hydrate structures. The accuracy of the hydrogen positions, about 0·2 Å, does not justify tabulation of the many bond distances and angles involving hydrogen atoms.

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Crystal Structure and Magnetic Properties of $CoTiO_3$*

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The crystal structure of cobalt titanate, $CoTiO_3$, has been refined by least-squares analysis of X-ray and neutron-diffraction data. Magnetic susceptibility measurements on polycrystalline samples gave a nearly ideal antiferromagnetic susceptibility curve with a Néel temperature of 38 °K. The ordered magnetic structure at low temperatures is identical with that of $NiTiO_3$ in which the spin direction is perpendicular to the rhombohedral [111] direction. Within each (111) plane the Co^{2+} moments are ferromagnetically coupled, but alternate layers are antiparallel.

Cobalt titanate, $CoTiO_3$, is isomorphous with ilmenite (Barth & Posnjak, 1934) and antiferromagnetic at low temperatures (Ishikawa & Akimoto, 1958*b*). This paper reports a refinement of the crystal structure and the details of the magnetic transitions, including the low-temperature spin structure.

The lattice parameters of $CoTiO_3$ were determined from high-angle diffractometer data obtained with a polycrystalline specimen supplied by the National Lead Company.† Neutron-diffraction and magnetic-susceptibility measurements were carried out on the same material. The rhombohedral unit-cell dimensions are $a = 5{\cdot}4846 \pm 0{\cdot}0007$ Å and $\alpha = 55°01' \pm 02'$, in substantial agreement with previous results (Ishikawa & Akimoto, 1958*a*). The space group of $CoTiO_3$ is $R\bar{3}$, with oxygen in general positions $\pm(x, y, z; z, x, y; y, z, x)$ and cobalt and titanium in special positions along the threefold symmetry axes at $\pm(u, u, u)$ and $\pm(v, v, v)$, respectively. Values of the atomic coordinates were obtained from Weissenberg intensity data with Mo $K\alpha$ radiation and a small single crystal grown by the Verneuil process.‡ Intensities of 140 (hhl) reflections were estimated visually by comparison with a cal-

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