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The Crystal Structure of Tutton's Salts.
VI. Vanadium(II), Iron(II) and Cobalt(II) Ammonium Sulfate Hexahydrates

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The crystal structures of the isomorphous vanadium(II), iron(II), and cobalt(II) ammonium sulfate hexahydrates, $V(NH_4)_2(SO_4)_2.6H_2O$, $Fe(NH_4)_2(SO_4)_2.6H_2O$, and $Co(NH_4)_2(SO_4)_2.6H_2O$, have been determined by three-dimensional X-ray analysis. The crystals are monoclinic, space group $P2_1/a$, with 2 molecules per cell. Cell dimensions are:

V: $a=9{\cdot}42$, $b=12{\cdot}76$, $c=6{\cdot}22$ Å; $\beta=107{\cdot}2°$
Fe: $a=9{\cdot}32$, $b=12{\cdot}65$, $c=6{\cdot}24$ Å; $\beta=106{\cdot}8°$
Co: $a=9{\cdot}25$, $b=12{\cdot}52$, $c=6{\cdot}24$ Å; $\beta=107{\cdot}1°$

The water molecules are arranged about the metal ions in almost regular octahedra, the average metal–oxygen distances being:

V–O, 2·15 Å; Fe–O, 2·13 Å; Co–O, 2·09 Å.

Introduction

As a part of a comparative study of the isomorphous series of ammonium Tutton's salts,

$$M(NH_4)_2(SO_4)_2.6H_2O$$

(*cf.* Montgomery, Chastain & Lingafelter, 1966), we have determined the crystal structures of the vanadium(II), iron(II) and cobalt(II) compounds.

Experimental

The vanadium(II) ammonium sulfate was prepared by a modification of the method of Kranz (1963). V_2O_5 was reduced to $VOSO_4$ with SO_2 in sulfuric acid solution. $(NH_4)_2SO_4$ was added, and the solution was cooled and reduced electrolytically with a mercury cathode. The reduced solution was concentrated in a vacuum desiccator over alkaline pyrogallol solution, forming flat, diamond-shaped amethyst-colored crystals. These were recrystallized under nitrogen from dilute aqueous sulfuric acid. The dried crystals are moderately stable in air (up to several weeks).

A crystal was cut into a rod to reduce absorption effects ($\mu=94{\cdot}6$ cm^{-1}). The axis of the rod was found to be the $[20\bar{1}]$ direction of the monoclinic cell. All data were collected from rotation and equi-inclination Weissenberg photographs taken about this axis with copper radiation. Intensities were recorded by multiple-film techniques for levels 0 through 7 about the $[20\bar{1}]$ axis on a Nonius equi-inclination integrating Weissenberg camera, integrating in one direction only, and were measured with photometer and planimeter. In all, 833 reflections were surveyed, of which 189 were below the minimum observable intensity and 19 were given zero weight in refinements because of secondary extinction effects. The intensities of the remainder varied from 1 to 1800.

Crystals of iron(II) ammonium sulfate hexahydrate were grown by cooling an aqueous solution, and a nearly equi-dimensional crystal (thickness ~0·2 mm) was selected for collection of data. The cell dimensions were determined from rotation and zero-level *hk*0 Weissenberg photographs calibrated with sodium chloride ($a_0=5{\cdot}6387$ Å) and taken with copper radiation ($\lambda=1{\cdot}5418$ Å); the angle β was measured on a precession photograph of the a^*c^* net. Intensities were recorded with iron radiation by multiple-film techniques for levels *hk*0 through *hk*3 on a Nonius equi-inclination integrating Weissenberg camera, integrating in one direction only, and were measured with photometer and planimeter. In all, 539 reflections were surveyed, of which 45 were below the minimum observable intensity and 6 were given zero weight in refinements because of secondary extinction effects. The intensities of the remainder varied from 1 to 5000.

Crystals of cobalt(II) ammonium sulfate hexahydrate were grown by cooling an aqueous solution, and a crystal with dimensions $0{\cdot}24\times0{\cdot}18\times0{\cdot}16$ mm was selected for collection of data. All data were collected with zirconium-filtered Mo $K\alpha$ radiation ($\lambda=0{\cdot}71069$ Å) on a Picker X-ray diffractometer equipped with a General Electric goniostat, a scintillation counter and a pulse-height discriminator. The cell dimensions were

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calculated by least squares from sixteen measured 2θ values. The intensity data were collected by the ω–2θ scan method (Furnas, 1957). A total of 1587 unique reflections were examined, out to $2\theta = 55^\circ$, of which 145 were considered unobserved, giving intensities less than twice their standard deviations; 34 reflections were given zero weight in refinement because of secondary extinction effects. The intensities of the 1442 observed reflections ranged from 1 to 9000.

The intensities for all three compounds were corrected for Lorentz and polarization factors but not for absorption. The resulting structure factors for the vanadium and iron compounds were then scaled by levels to correspond to the calculated values, using the parameters of the isomorphous zinc salt (Montgomery & Lingafelter, 1964*a*). (It should be noted that this scaling procedure, together with the lack of absorption correction, precludes physical interpretation of the anisotropic thermal parameters.)

The cell dimensions for the three compounds are: Vanadium(II) ammonium sulfate hexahydrate: $a = 9{\cdot}42(3)$, $b = 12{\cdot}76(3)$, $c = 6{\cdot}22(2)$ Å; $\beta = 107{\cdot}2(2)^\circ$. Iron(II) ammonium sulfate hexahydrate: $a = 9{\cdot}32(2)$, $b = 12{\cdot}65(2)$, $c = 6{\cdot}24(1)$ Å; $\beta = 106{\cdot}8(1)^\circ$. Cobalt(II) ammonium sulfate hexahydrate: $a = 9{\cdot}254(2)$, $b = 12{\cdot}521(4)$, $c = 6{\cdot}244(1)$ Å; $\beta = 107{\cdot}06(4)^\circ$. Systematic absences: $h0l$ when h is odd; $0k0$ when k is odd; space group $P2_1/a$; $Z = 2$.

Refinement

For all three compounds, the initial atomic parameters for the heavy atoms were taken from the zinc salt and

Table 1. *Final parameters* (*with least-squares standard errors in parentheses*)

	Fractional positional parameters × 10^4			Anisotropic thermal parameters × 10^4					
	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{33}	β_{12}	β_{23}
			Vanadium(II) ammonium sulfate hexahydrate						
V	0	0	0	17 (3)	20 (1)	222 (14)	−2 (1)	40 (6)	2 (3)
S	4077 (3)	1370 (2)	7406 (6)	6 (3)	19 (1)	168 (15)	−5 (1)	25 (7)	−2 (3)
O(3)	4104 (9)	2273 (5)	5891 (15)	108 (14)	21 (4)	295 (44)	−16 (5)	61 (23)	17 (9)
O(4)	5470 (8)	789 (6)	7872 (17)	7 (10)	53 (5)	361 (51)	16 (5)	−24 (21)	−17 (12)
O(5)	2798 (7)	668 (5)	6234 (14)	40 (10)	28 (4)	228 (40)	−15 (5)	44 (19)	−14 (8)
O(6)	3842 (8)	1765 (5)	9483 (14)	63 (11)	34 (4)	186 (41)	−14 (5)	25 (21)	−3 (9)
O(7)	1749 (8)	1071 (5)	1702 (15)	46 (11)	34 (3)	285 (42)	−26 (5)	61 (20)	−20 (10)
O(8)	−1632 (7)	1135 (4)	321 (15)	12 (9)	21 (3)	324 (45)	6 (4)	16 (18)	9 (9)
O(9)	−12 (8)	−711 (5)	3072 (16)	49 (11)	28 (4)	297 (42)	12 (5)	64 (20)	21 (9)
N	1339 (9)	3483 (6)	3589 (19)	28 (13)	39 (5)	247 (54)	1 (6)	16 (25)	1 (12)
			Iron(II) ammonium sulfate hexahydrate						
Fe	0	0	0	36 (2)	25 (1)	184 (14)	−2 (1)	−1 (4)	3 (2)
S	4068 (2)	1372 (1)	7417 (4)	26 (2)	25 (1)	165 (17)	−5 (1)	4 (4)	0 (3)
O(3)	4083 (5)	2275 (4)	5896 (10)	132 (8)	39 (3)	290 (33)	−23 (4)	81 (11)	5 (7)
O(4)	5462 (5)	783 (4)	7853 (10)	40 (6)	60 (3)	394 (35)	4 (4)	20 (11)	−20 (8)
O(5)	2799 (4)	673 (3)	6284 (9)	54 (6)	41 (3)	221 (33)	−7 (3)	−1 (10)	−6 (6)
O(6)	3848 (5)	1769 (4)	9532 (11)	88 (7)	48 (3)	165 (36)	−4 (4)	56 (11)	−2 (9)
O(7)	1742 (5)	1079 (3)	1722 (10)	78 (7)	44 (3)	237 (31)	−19 (3)	49 (10)	4 (7)
O(8)	−1637 (4)	1129 (3)	289 (10)	42 (6)	35 (3)	307 (30)	3 (3)	26 (10)	2 (6)
O(9)	−1 (4)	−702 (3)	3027 (10)	63 (7)	36 (3)	260 (33)	8 (3)	55 (10)	17 (6)
N	1331 (6)	3500 (4)	3609 (13)	90 (9)	44 (4)	306 (43)	4 (4)	40 (14)	21 (8)
			Cobalt(II) ammonium sulfate hexahydrate						
Co	0	0	0	46 (1)	27 (1)	120 (2)	−2 (1)	18 (1)	1 (1)
S	4077 (1)	1369 (1)	7401 (2)	59 (1)	35 (1)	157 (3)	−6 (1)	24 (1)	−6 (1)
O(3)	4119 (4)	2272 (3)	5911 (6)	144 (6)	48 (2)	220 (10)	−21 (3)	56 (6)	−7 (4)
O(4)	5473 (4)	769 (4)	7866 (7)	75 (4)	76 (3)	367 (14)	10 (3)	27 (6)	−13 (5)
O(5)	2810 (4)	665 (3)	6241 (6)	80 (4)	51 (2)	222 (10)	−9 (2)	34 (5)	−17 (4)
O(6)	3843 (4)	1763 (3)	9489 (6)	127 (5)	50 (2)	207 (10)	−7 (3)	59 (6)	−8 (4)
O(7)	1712 (4)	1080 (3)	1652 (6)	86 (4)	53 (2)	187 (9)	−13 (3)	34 (5)	−6 (4)
O(8)	−1622 (4)	1114 (3)	345 (6)	79 (4)	41 (2)	266 (11)	6 (2)	41 (5)	6 (4)
O(9)	−12 (4)	−689 (3)	3009 (5)	97 (4)	44 (2)	197 (9)	5 (2)	52 (5)	6 (4)
N	1335 (5)	3479 (4)	3575 (8)	97 (6)	52 (3)	222 (13)	−3 (3)	53 (7)	0 (5)
	Parameters × 10^3				Isotropic parameter, B				
H(11)	61 (9)	323 (6)	208 (15)		7 (2)				
H(12)	218 (9)	288 (6)	417 (12)		6 (2)				
H(13)	69 (9)	341 (6)	446 (13)		6 (2)				
H(14)	168 (9)	415 (7)	362 (12)		6 (2)				
H(15)	204 (7)	81 (6)	319 (12)		5 (2)				
H(16)	241 (6)	119 (4)	64 (9)		3 (1)				
H(17)	−256 (10)	87 (7)	−54 (14)		7 (2)				
H(18)	−148 (6)	188 (5)	4 (9)		3 (1)				
H(19)	−144 (14)	−47 (11)	306 (21)		14 (3)				
H(20)	28 (5)	−144 (4)	341 (8)		2 (1)				

Table 2. *Observed and calculated structure factors for vanadium*(II) *ammonium sulfate hexahydrate*

Columns are k, $10F_o$ and $10F_c$. Unobserved reflections are marked with *, and those omitted because of secondary extinction are marked with *E*.

	0,K,0	
8	319	324
10	43	45
12	144	135
14	303	322
16	207	187
	1,K,0	
1	72	64
2	364	404
3	734E	1059
4	41*	50
5	52*	53
6	110	95
7	45*	57
8	64*	-25
9	309	285
10	74*	16
11	414	408
12	146	-131
13	8*	3
14	67*	-31
	2,K,0	
0	351E	619
1	455E	-572
2	273	290
3	167	175
4	381	390
5	213	196
6	297	291
7	54*	13
8	188	201
9	259	-238
10	77	71
11	70*	-28
12	172	174
13	184	173
14	269	269
15	49*	23
	3,K,0	
1	293	384
2	155	148
3	274	301
4	518	-571
5	539	612
6	176	-156
7	207	202
8	174	140
9	160	-139
10	307	304
11	64	94
12	136	-126
13	303	315
14	55*	-52
15	200	206
	4,K,0	
0	137	-147
1	232	-283
2	665E	842
3	212	204
4	563	628
5	347	330
6	221	-186
7	103	66
8	169	-169
9	293	-278
10	292	275
11	63	-58
12	265	279
13	114	101
14	126	146
15	67	-55
	5,K,0	
1	620E	772
2	121	124
3	333	-321
4	73	-82
5	99	96
6	125	-125
7	460	459
8	64	66
9	288	296
10	130	122
11	31*	-18
12	89	-100
13	160	192
14	41*	-33
	6,K,0	
0	132	133
1	193	195
2	427	466
3	59*	62
4	234	237
5	78	-76
6	25*	16
7	69	-70
8	59*	65
9	70*	-32
10	367	365
11	64*	22
12	203	194
13	47*	42
	7,K,0	
1	526	586
2	351	-383
3	95	130
4	59*	54
5	50*	-52
6	77	107
7	115	117
8	61*	-70
9	201	226
10	55*	-67
11	2*	-2
12	142	140
	0,K,1	
1	444E	563
2	519	401
3	432	446
4	86	-61
5	261	253
6	443	456
7	53*	15
8	324	330
9	63*	52
10	395	403
11	208	-216
12	118	132
13	262	-254
14	57*	-64
15	71	97
16	132	133
	1,K,1	
2	396	-390
3	86	-71
4	403	409
5	309	336
6	303	279
7	430	430
8	176	-161
9	291	274
10	153	-133
11	168	-145
12	68*	22
13	227	238
14	58*	28
15	193	193
	-1,K,1	
1	157	179
2	456E	-567
3	431	512
4	228	-245
5	137	146
6	231	212
7	170	179
8	174	193
9	524	550
10	296	-299
11	232	238
12	123	147
13	67	58
14	92	76
15	36*	-25
	2,K,1	
0	342E	-629
1	465E	651
2	548E	698
3	497	564
4	716	805
5	402	-393
6	108	105
7	50*	-30
8	221	-176
9	59*	-14
10	335	316
11	63*	-3
12	308	315
13	58*	18
14	35*	30
	-2,K,1	
0	820E	1197
1	236	288
2	157	189
3	291	300
4	50	-39
5	353	383
6	478	545
7	327	-337
8	328	352
9	55*	17
10	73	-72
11	60*	-35
12	73	93
13	150	-131
14	276	291
15	101	104
	3,K,1	
1	44	50
2	167	153
3	124	-99
4	191	177
5	612	627
6	50*	-44
7	642	644
8	237	-222
9	207	191
10	175	172
11	58*	-53
12	64*	-34
13	158	169
14	50*	-17
15	171	175
	-3,K,1	
1	275	-302
2	474	536
3	810E	1036
4	233	216
5	594	606
6	66	-76
7	160	-145
8	64*	49
9	16*	8
10	185	-174
11	488	482
12	76*	30
13	115	111
14	68	-73
15	31*	25
	4,K,1	
0	101	138
1	136	-152
2	419	448
3	323	318
4	422	420
5	315	278
6	33*	-26
7	142	123
8	71	75
9	288	-266
10	245	247
11	144	-141
12	244	265
13	82	81
14	61	74
	-4,K,1	
0	15	-10
1	123	140
2	119	-116
3	248	256
4	378	433
5	277	276
6	563	582
7	97	-99
8	597	602
9	196	-198
10	64	-56
11	70*	-25
12	21*	-11
13	61*	45
14	270	273
15	37*	-5
	5,K,1	
1	289	323
2	304	344
3	185	185
4	182	-173
5	147	140
6	62	-50
7	274	244
8	170	140
9	216	201
11	195	177
12	203	-230
13	5*	3
13	5*	3
	-5,K,1	
1	401	465
2	159	147
3	212	237
4	150	-133
5	183	197
6	253	-263
7	184	191
8	128	105
9	51*	47
10	201	231
11	326	338
12	117	-136
13	63	86
14	85	-94
	-6,K,1	
0	253	277
1	206	-193
2	279	301
3	53*	-45
4	306	315
5	270	267
6	6*	-4
7	263	272
8	263	265
9	311	-314
10	316	314
11	148	-130
12	40*	44
13	143	126
	-7,K,1	
1	506	564
2	797	789
3	118	-107
4	158	162
5	44*	-36
6	66*	-39
7	441	473
8	66*	-29
9	233	245
10	60*	41
11	20*	14
12	260	-251
13	136	131
	-8,K,1	
0	260	-282
1	109	-106
2	148	130
3	70*	-21
4	488	523
5	71*	24
6	260	270
7	63*	61
8	22*	13
9	103	-99
10	281	296
11	47*	-18
	-9,K,1	
1	283	280
2	62*	37
3	123	149
4	64	73
5	147	150
6	58*	67
7	227	243
8	134	-118
9	100	95
10	37*	-52
	0,K,2	
0	80	-110
1	218	-235
2	6	-8
3	275	-279
4	618	692
5	360	-359
6	351	371
7	295	264
8	110	99
9	59*	17
10	380	361
11	63*	-29
12	208	194
13	121	87
14	50*	-111
	1,K,2	
1	129	141
2	56	-59
3	179	181
4	38*	-27
5	178	168
6	240	-236
7	597	617
8	59*	-42
9	266	260
10	327	314
11	51*	43
12	72	-68
13	43*	35
14	68	-64
15	210	202
	-1,K,2	
1	560E	964
2	539E	-754
3	26*	43
4	251	-254
5	191	-157
6	420	446
7	402	417
8	60*	-49
9	261	269
10	67*	-36
11	88	-41
12	67*	-33
13	208	216
14	87	92
15	231	246
	2,K,2	
1	190	187
2	68	62
3	257	247
4	540	511
5	226	205
6	287	262
7	59*	-50
8	251	239
9	342	-329
10	102	95
11	70*	46
12	139	133
13	217	220
14	129	118
15	125	-105
	-2,K,2	
0	353E	-481
1	170	-210
2	196	233
3	215	220
4	491	531
5	174	-179
6	410	464
7	58*	-19
8	149	151
9	238	233
10	332	365
11	71*	-49
12	162	170
13	193	-197
14	76	-79
15	44	45
	3,K,2	
1	185	224
2	240	252
3	374	354
4	301	-273
5	292	278
6	536	-495
7	101	81
8	217	207
9	42*	35
10	317	292
11	327	312
12	57*	-28
13	44*	61
14	102	-100
	0,K,3	
0	474E	690
	-3,K,2	
1	110	137
2	59	67
3	206	239
4	217	-229
5	431	470
6	81	98
7	207	216
8	161	-166
9	256	291
10	132	-115
11	111	124
12	252	263
13	179	184
14	116	129
15	27*	23
	-4,K,2	
0	47	47
1	94	85
2	300	385
3	43*	39
4	89	126
5	293	-310
6	408	480
7	328	-361
8	318	336
9	309	317
10	15*	-9
11	237	242
12	44*	47
13	104	-108
14	224	225
	-5,K,2	
1	88	99
2	84	88
3	443	478
4	164	137
5	448	494
6	71*	5
7	182	-161
8	75*	-8
9	183	161
10	109	-123
11	365	358
12	81	65
	-6,K,2	
0	381	372
1	59*	-22
2	107	109
3	410	-388
4	166	-130
5	66*	15
6	418	434
7	164	149
8	616	635
9	70*	38
10	51*	45
11	63*	-37
	-7,K,2	
1	84	86
2	178	-165
3	326	340
4	66*	-39
5	264	272
6	153	-151
7	67	91
8	141	135
9	200	198
10	61	80
11	242	304
12	62	-83
	-8,K,2	
0	376	386
1	181	-179
2	184	186
3	319	-335
4	195	185
5	156	122
6	100	111
7	175	155
8	171	176
9	154	-157
10	138	161
11	42*	7
	-9,K,2	
1	127	114
2	355	367
3	140	130
4	65*	69
6	247	-239
7	364	387
8	54*	27
9	89	98
10	115	122
	-10,K,2	
0	125	-128
1	136	-143
2	79	111
3	64*	-82
4	416	452
5	59*	23
6	144	134
7	50*	-62
	-11,K,2	
1	242	248
2	47*	51
3	11*	-9
4	132	-128
5	96	96
	0,K,3	
1	98	-114
2	220	241
3	422	-413
4	102	112
5	348	336
6	261	256
7	164	153
8	93	97
9	220	-230
10	38*	21
11	69*	-23
12	150	141
13	160	157
14	172	163
	1,K,3	
1	331E	443
2	280	265
3	378	384
4	41*	20
5	157	132
6	479	-452
7	208	-194
8	113	92
9	145	120
10	169	186
11	268	268
12	56*	-13
13	127	134
14	53	-61
	-1,K,3	
1	149	210
2	40*	15
3	396	434
4	56	70
5	43	60
6	81	-89
7	233	250
8	230	215
9	168	181
10	66*	-5
11	58	70
12	164	-180
13	120	113
14	84	-83
	-2,K,3	
0	289	336
1	297	-348
2	95	105
3	477	-558
4	482	549
5	47*	-18
6	111	124
7	148	135
8	52	-54
9	62*	50
10	233	242
11	62*	23
12	282	288
13	55	54
14	20*	15
	-3,K,3	
1	529E	737
2	125	136
3	103	117
4	53*	-30
5	123	-106
6	60*	7
7	345	360
8	72	-73
9	44*	31
10	68*	-15
11	21*	-10
12	77	75
13	165	186
14	42*	-3
	-4,K,3	
0	129	142
1	70	82
2	311	387
3	323	374
4	425	518
5	201	-203
6	156	-153
7	197	-194
8	15*	-7
9	145	150
10	289	329
11	71	93
12	193	221
13	106	-119
14	32*	36
	-5,K,3	
1	333	351
2	64*	-4
3	62	-50
4	68*	-10
5	210	238
6	165	159
7	375	428
8	188	-208
9	116	128
10	75*	-72
11	67*	70
12	111	137
13	94	98
14	93	105
	-6,K,3	
0	361	359
1	53*	-32
2	402	418
3	73	-52
4	124	128
5	349	-360
6	44*	48
7	58*	-4
8	129	126
9	235	235
10	118	108
11	53	70
12	113	104
13	126	-111
	-7,K,3	
1	172	162
2	413	-360
3	418	391
4	77*	19
5	337	292
6	294	290
7	89	-115
8	75*	-20
9	83	93
10	111	-122
11	224	242
12	73	-59
	-8,K,3	
0	585	563
1	70*	50
2	265	231
3	71*	-21
4	205	-162
5	70*	-40
6	280	272
7	67*	72
8	253	257
9	58*	5
10	15*	11
11	106	-81
	-9,K,3	
1	44*	-32
2	67*	-21
3	423	440
4	66*	-43
5	267	270
6	144	-129
7	120	-90
8	32*	-1
9	180	179
10	124	100
	-10,K,3	
0	499	495
1	57*	-14
2	237	230
3	55*	-25
4	25*	-19
5	52	81
6	132	139
7	74	-85
8	114	120
	-11,K,3	
1	171	182
2	245	267
3	150	155
4	43*	-51
5	120	128
6	206	-219
	-1,K,4	
1	90	-75
2	61	67
3	408	469
4	50*	-11
5	357	361
6	57*	-31
7	60	-62
8	61*	30
9	157	134
10	61*	27
11	165	168
12	51*	-115
13	134	146
	-2,K,4	
0	371E	469
1	207	-234
2	246	274
3	131	-131
4	29*	-18
5	335	346
6	294	302
7	68*	76
8	48*	31
9	215	-195
10	35*	-15
11	65*	-76
12	165	151
13	49*	71
	-3,K,4	
1	9*	-5
2	388	443
3	277	304
4	58*	26
5	246	257
6	155	-151
7	128	124
8	171	167
9	232	257
10	64*	-16
11	73	82
12	158	-158
13	97	85
	-4,K,4	
2	234	245
3	74	-47
4	129	151
5	228	240
6	62	92
7	156	-126
8	140	144
9	131	-136
10	201	220
11	55*	30
12	168	175
13	96	97
	-5,K,4	
2	191	189
3	112	-95
4	65*	28
5	45*	34
6	140	-163
7	305	344
8	68*	30
9	143	148
10	62*	9
11	2*	1
	-6,K,4	
2	277	284
3	189	184
4	420	480
5	106	93
6	69	-99
7	70*	-15
8	9*	4
9	65*	-61
10	199	237
11	95	-97
12	157	178
	-7,K,4	
2	263	-234
3	69*	-61
4	99	106
5	178	200
6	172	178
7	410	455
8	71*	-47
9	56*	60
10	125	-125
	-8,K,4	
0	43*	41
1	151	142
2	172	160
3	49	84
4	386	362
5	168	-180
6	115	92
7	60	38
8	31*	29
9	85	94
10	167	134
	-9,K,4	
1	192	175
2	185	-196
3	314	275
4	66	85
5	64	89
6	220	193
7	38*	30
8	102	-105
9	94	93
	-10,K,4	
0	417	418
1	136	175
2	83	86
3	317	285
4	63	-79
5	52	-55
6	294	262
7	99	-82
	-11,K,4	
1	15*	-13
2	44*	45
3	334	338
	-3,K,5	
1	122	-126
2	67	-57
3	405	368
4	233	-198
5	305	254
6	62*	12
7	48*	37
8	61*	21
9	107	97
10	53*	59
11	174	188
	-4,K,5	
0	411	425
1	228	-213
2	168	156
3	69*	22
4	84	-95
6	233	209
7	70*	-37
8	259	231
9	90	-88
10	41*	27
	-5,K,5	
2	303	298
3	161	167
4	136	-121
5	150	-154
6	127	-116
7	52*	43
8	87	96
9	244	229
10	82	72
11	222	178
	-10,K,5	
0	36	-38
1	125	115
2	100	91
3	39*	-2
4	309	265
5	183	-162
	-11,K,5	
1	227	215
	-5,K,6	
2	158	-142
3	174	180
4	125	-93
5	54*	78
6	80	70
7	26*	13

Table 3. *Observed and calculated structure factors for iron*(II) *ammonium sulfate hexahydrate*

Columns are k, $10F_o$ and $10F_o$. Unobserved reflections are marked with *, and those omitted because of secondary extinction are marked with *E*.

	0,K,0	
2	495	417
4	806	-814
6	38	-30
8	301	307
10	26*	20
12	106	103
	1,K,0	
1	73	71
2	500	-404
3	1048	1051
4	34	-34
5	76	63
6	91	-78
7	44	41
8	25*	16
9	236	254
10	43	-23
11	314	357
12	107	111
	2,K,0	
0	540	612
1	625	577
2	382	297
3	157	-171
4	423	385
5	207	-190
6	288	278
7	24*	-16
8	172	168
9	199	210
10	53	57
11	21*	20
12	143	141
	3,K,0	
1	367	382
2	159	-148
3	292	294
4	580	551
5	583	567
6	166	152
7	192	190
8	148	-129
9	126	-123
10	248	-266
11	70	61
12	96	106
	4,K,0	
0	134	-139
1	236	264
2	738	804
3	193	-190
4	565	584
5	313	-312
6	176	-167
7	65	-59
8	156	-164
9	217	246
10	230	242
11	63	59
	5,K,0	
1	656	715
2	101	-95
3	293	-299
4	74	66
5	102	96
6	112	104
7	386	417
8	51	-52
9	223	237
10	91	-102
	6,K,0	
0	111	109
1	178	-191
2	403	422
3	26*	-19
4	193	204
5	82	94
6	25*	-6
7	57	53
8	51	48
9	16*	3
	7,K,0	
1	498	518
2	337	355
3	106	108
4	62	-50
5	35	-40
6	90	-94
7	79	93
8	57	63
	8,K,0	
0	192	188
1	317	-299
2	124	115
3	179	-177
4	184	182
5	107	114
6	251	226
	0,K,1	
1	477	563
2	440	406
3	443	451
4	62	-49
5	260	256
6	436	430
7	25*	20
8	298	288
9	59	49
10	145	160
11	196	-196
12	106	116
	1,K,1	
1	489E	715
2	344	-388
3	52	-49
4	366	400
5	292	303
6	262	277
7	382	404
8	152	-149
9	246	262
10	129	-129
11	131	-123
12	31	29
	-1,K,1	
1	200	190
2	616	-549
3	577	504
4	237	-233
5	147	131
6	229	206
7	166	169
8	166	179
9	483	492
10	260	-261
11	199	213
12	123	117
	2,K,1	
0	511E	-621
1	480E	633
2	567E	682
3	461	552
4	699E	813
5	348	-377
6	94	103
7	33	-24
8	168	-178
9	27*	-4
10	291	289
11	21*	-1
12	270	272
	-2,K,1	
0	838E	1190
1	281	268
2	182	175
3	311	281
4	52	-51
5	387	375
6	571	505
7	311	-304
8	341	341
9	27*	15
10	72	-68
11	41	-34
12	61	63
	3,K,1	
1	52	56
2	137	156
3	107	-98
4	161	171
5	552	592
6	63	-41
7	584	619
8	185	-184
9	171	173
10	153	141
11	62	-61
	-3,K,1	
1	261	-274
2	518	533
3	940	990
4	218	191
5	570	563
6	84	-74
7	147	-152
8	53	49
9	27*	-7
10	143	-139
11	336	417
	4,K,1	
0	127	136
1	128	-142
2	397	427
3	303	302
4	403	403
5	277	284
6	38	-27
7	119	110
8	91	79
9	273	-256
10	219	212
11	114	-115
	-4,K,1	
0	29	-15
1	114	121
2	113	-115
3	251	269
4	396	397
5	256	271
6	508	511
7	100	-89
8	521	530
9	179	-187
10	59	-56
11	25	-24
	5,K,1	
1	308	326
2	314	320
3	172	166
4	163	-151
5	144	132
6	66	-53
7	227	213
8	143	143
9	181	172
10	62	61
	-5,K,1	
1	410	426
2	144	147
3	196	202
4	128	-119
5	163	164
6	213	-218
7	163	152
8	91	93
9	38	34
10	178	188
	6,K,1	
0	536	556
1	428	-438
2	154	126
3	271	-269
4	27*	-3
5	313	295
6	158	144
7	76	67
8	329	300
	-6,K,1	
0	217	229
1	181	-171
2	258	260
3	27*	-25
4	268	264
5	231	223
6	27*	-8
7	225	225
8	192	203
9	252	-271
	7,K,1	
1	123	-120
2	368	362
3	378	351
4	58	46
5	261	250
6	86	-81
7	118	119
	-7,K,1	
1	479	484
2	227	232
3	109	-102
4	133	152
5	41	-47
6	29	-18
7	372	373
8	18	-20
	8,K,1	
0	417	431
1	94	86
2	105	101
3	85	89
4	29	-3
	-8,K,1	
4	409	412
5	19*	4
6	211	201
	0,K,2	
0	120	-120
1	197	-239
2	12*	-21
3	266	-265
4	653	684
5	351	-347
6	347	360
7	245	239
8	92	95
9	25*	27
10	347	337
11	19*	-25
12	183	174
	1,K,2	
1	110	129
2	15*	-14
3	168	189
4	19*	-21
5	167	176
6	216	-241
7	540	584
8	44	-40
9	234	237
10	285	276
	-1,K,2	
1	889	948
2	744	-756
3	48	41
4	276	-260
5	165	-157
6	447	437
7	419	394
8	61	-48
9	255	249
10	56	-52
11	101	-86
	2,K,2	
0	148	169
1	153	184
2	85	70
3	246	226
4	478	504
5	195	210
6	260	254
7	54	-44
8	228	220
9	307	-307
10	91	97
	-2,K,2	
0	420	-477
1	203	-220
2	247	220
3	216	213
4	527	496
5	181	-170
6	471	440
7	24*	-26
8	144	140
9	220	216
10	320	321
11	41	-46
12	132	134
	3,K,2	
1	185	227
2	229	240
3	331	343
4	248	-255
5	246	267
6	460	-477
7	102	89
8	191	192
9	65	55
10	286	270
	-3,K,2	
1	122	119
2	86	83
3	229	207
4	215	-196
5	472	435
6	109	102
7	220	198
8	160	-154
9	247	254
10	119	-116
11	97	99
	4,K,2	
0	209	203
1	212	-239
2	112	121
3	306	-311
4	84	83
5	287	274
6	291	285
7	24*	1
8	462	429
9	132	-133
10	65	55
	-4,K,2	
0	47	-17
1	94	89
2	336	340
3	20*	29
4	100	108
5	295	-283
6	452	433
7	320	-333
8	325	308
9	297	270
10	20*	-15
11	202	198
	5,K,2	
1	41	-50
2	144	-127
3	301	293
4	152	-143
5	368	359
6	63	58
7	97	79
8	217	188
	-5,K,2	
1	75	79
2	105	104
3	431	403
4	144	133
5	437	435
6	25*	-15
7	157	-155
8	24*	-13
9	135	123
10	100	-100
	6,K,2	
0	401	426
1	54	49
2	236	223
3	23*	32
4	64	-60
5	20*	2
6	211	190
	-6,K,2	
0	307	294
1	24*	-16
2	88	79
3	331	-350
4	123	-114
5	25*	25
6	361	357
7	127	122
8	496	523
	7,K,2	
1	19*	16
2	278	-269
3	224	221
4	43	-43
5	194	217
	-7,K,2	
1	25*	41
2	142	-125
3	268	259
4	24*	-22
5	214	212
6	125	-130
7	74	71
8	113	116
	0,K,3	
1	109	-119
2	201	247
3	387	-429
4	110	113
5	295	307
6	238	239
7	134	134
8	79	74
9	207	-207
11	11*	-1
	1,K,3	
1	373	431
2	238	263
3	150	398
4	19*	2
5	127	133
6	440	-464
7	168	-164
8	93	93
9	125	115
10	205	194
	-1,K,3	
1	194	211
2	31	21
3	453	435
4	51	48
5	59	54
6	93	-88
7	225	232
8	205	203
9	155	147
10	17*	1
11	54	51
	2,K,3	
1	103	106
2	279	276
3	94	-98
4	97	-90
5	47	-58
6	283	292
7	72	-62
8	406	402
9	27	-28
10	56	46
	-2,K,3	
0	284	319
1	372	-374
2	105	87
4	550	534
5	24	-11
6	121	110
7	123	120
8	70	-52
9	54	49
10	205	212
11	23	14
	3,K,3	
1	299	298
2	39	-31
3	227	248
4	85	83
5	260	269
6	63	65
7	147	-141
8	58	-49
9	63	58
	-3,K,3	
1	626	674
2	115	108
3	118	98
4	63	-61
5	97	-94
6	29	28
7	354	335
8	78	-73
9	19*	19
10	19	-25
11	31	-24
	4,K,3	
1	21*	32
2	318	312
3	64	49
4	92	90
5	212	-198
6	129	134
7	39	21
8	155	151
	-4,K,3	
0	134	135
1	64	53
2	366	364
3	359	342
4	460	459
5	219	-222
6	163	-160
7	184	-185
8	20*	-21
9	150	153
10	269	261
	5,K,3	
1	200	195
2	437	-436
3	176	185
4	146	-136
5	175	168
6	302	273
7	50	46
	-5,K,3	
1	308	306
2	19*	4
3	61	-69
4	21*	-7
5	202	202
6	150	142
7	359	348
8	174	-195
9	112	95
10	54	-54
	6,K,3	
1	88	94
2	102	99
3	185	164
4	244	224
	-6,K,3	
0	299	286
1	21*	-27
2	347	360
3	50	-54
4	79	75
5	307	-329
6	35	25
7	18*	5
8	99	92
9	177	198
	-7,K,3	
1	104	104
2	300	-322
3	308	301
4	20*	4
5	243	240
6	213	212
7	111	-116
	-8,K,3	
0	462	435

Table 4. *Observed and calculated structure factors for cobalt*(II) *ammonium sulfate hexahydrate*

Columns are k, $10F_o$ and $10F_c$. Unobserved reflections are marked with *, and those omitted because of secondary extinction are marked with *E*.

0,K,0		
2	415E	492
4	549E	-685
6	26*	31
8	302	320
10	70	63
12	129	124
14	258	252
16	170	148
1,K,0		
1	149	135
2	343E	-410
3	716E	1104
4	30	-47
5	109	113
6	66	-58
7	98	95
8	15*	-0
9	277	285
10	19	-27
11	349	359
12	95	96
13	39	40
14	44	34
15	47	50
16	33	-32
2,K,0		
0	533E	676
1	453E	565
2	304	328
3	138	-154
4	400	422
5	169	-188
6	302	341
7	20	-21
8	208	213
9	181	184
10	79	84
11	25	22
12	154	152
13	119	-115
14	226	216
15	20*	-11
16	111	104
3,K,0		
1	398	414
2	139	-135
3	320	338
4	480	523
5	537E	588
6	143	151
7	230	240
8	113	-111
9	74	-81
10	238	-234
11	101	96
12	83	90
13	252	252
14	40	38
15	171	160
4,K,0		
0	67	-74
1	244	265
2	698E	801
3	185	-176
4	565E	619
5	287	-293
6	85	-89
7	68	-70
8	84	-93
9	221	226
10	232	228
11	55	48
12	215	212
13	71	-75
14	133	120
15	38	32
5,K,0		
1	637E	710
2	98	-91
3	195	-206
4	65	69
5	147	142
6	99	90
7	400	406
8	38	-37
9	243	239
10	89	-87
11	19*	6
12	68	60
13	156	151
14	25	21
6,K,0		
0	147	157
1	136	-144
2	422	437
3	29*	-32
4	231	229
5	66	63
6	47	49
7	37	39
8	82	70
9	19*	5
10	285	284
11	20*	-10
12	166	167
13	21*	-13
7,K,0		
1	494	513
2	267	277
3	137	136
4	42	-48
5	18*	1
6	72	-74
7	105	106
8	51	54
9	182	184
10	44	45
11	20*	27
12	88	-78
8,K,0		
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2	151	145
3	142	-152
4	184	188
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6	234	231
7	19*	23
8	68	69
9	106	-98
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11	79	68
9,K,0		
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2	225	221
3	300	296
4	26	-27
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6	111	-109
7	20*	18
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9	67	68
10	94	82
10,K,0		
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1	50	-46
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3	20*	-5
4	73	67
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6	168	156
7	36	32
8	137	132
11,K,0		
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2	58	-42
3	205	189
4	21*	-3
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1	454E	558
2	394E	491
3	364E	439
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6	416	445
7	46	45
8	312	320
9	85	64
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11	166	-168
12	143	141
13	215	-202
14	27	-13
15	64	56
16	113	106
1,K,1		
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2	335E	-402
3	14*	-6
4	355	379
5	324	349
6	252	285
7	402	424
8	125	-126
9	288	299
10	119	-125
11	56	-60
12	19*	19
13	214	205
14	25	25
15	156	153
16	48	-44
2,K,1		
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1	517E	599
2	635E	749
3	454E	506
4	686E	841
5	314	-337
6	144	147
7	16*	-8
8	103	-107
9	18*	-6
10	286	292
11	18*	-5
12	273	268
13	19*	0
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15	24	-9
3,K,1		
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2	151	162
3	48	-49
4	164	162
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6	34	-39
7	575	615
8	168	-178
9	205	200
10	133	131
11	23	-16
12	36	-28
13	150	149
14	24	-19
15	156	154
4,K,1		
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1	122	-129
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3	285	309
4	415	439
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6	46	40
7	104	101
8	105	103
9	207	-206
10	212	208
11	101	-95
12	217	216
13	54	54
14	83	76
5,K,1		
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2	295	302
3	203	203
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5	156	154
6	39	-41
7	228	223
8	106	115
9	195	198
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11	162	159
12	176	-162
13	28	34
14	34	-19
6,K,1		
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3	226	-231
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5	244	256
6	160	157
7	73	70
8	283	282
9	122	-121
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11	20*	-8
12	83	82
13	77	72
7,K,1		
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2	285	295
3	329	333
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5	232	234
6	48	-47
7	148	138
8	64	57
9	161	153
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12	91	-74
8,K,1		
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2	121	123
3	63	57
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5	54	55
6	198	190
7	48	-37
8	159	160
9	21	-17
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9,K,1		
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2	22	-19
3	270	264
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5	185	186
6	21	15
7	20*	22
8	89	-82
9	49	47
10,K,1		
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1	138	130
2	89	92
3	20*	30
4	74	72
5	136	-123
6	150	136
-1,K,1		
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2	436E	-548
3	459E	577
4	213	-248
5	171	175
6	192	185
7	194	189
8	166	173
9	479	499
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11	228	234
12	103	99
13	80	77
14	57	56
15	20*	14
16	47	-43
-2,K,1		
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2	210	233
3	258	287
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6	495	540
7	258	-280
8	341	353
9	42	38
10	18*	-16
11	36	-33
12	100	97
13	124	-114
14	237	233
15	60	56
-3,K,1		
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2	440E	505
3	777E	1019
4	191	204
5	527E	590
6	64	-52
7	66	-72
8	41	49
9	53	40
10	130	-140
11	388	393
12	20	15
13	114	113
14	62	-48
15	45	37
-4,K,1		
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1	126	144
2	47	-47
3	215	237
4	390	413
5	232	256
6	493	533
7	72	-71
8	503	532
9	150	-156
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11	36	-27
12	32	25
13	38	35
14	214	208
15	21*	-5
-5,K,1		
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2	126	147
3	257	264
4	96	-100
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6	198	-207
7	178	185
8	72	73
9	62	64
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11	274	271
12	97	-92
13	92	93
14	71	-64
-6,K,1		
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1	154	157
2	276	276
3	29	33
4	295	301
5	208	-212
6	38	35
7	202	-206
8	233	230
9	222	222
10	255	253
11	95	87
12	64	65
13	82	-77
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-7,K,1		
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2	212	-216
3	34	-39
4	114	-113
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7	364	367
8	27	-2
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10	29	-34
11	45	39
12	182	159
13	102	98
-8,K,1		
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2	138	131
3	21	18
4	374	375
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7	44	-41
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9	61	53
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11	20*	8
12	137	131
-9,K,1		
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2	33	-32
3	128	122
4	52	-51
5	127	115
6	51	-42
7	182	180
8	78	69
9	87	92
10	36	31
-10,K,1		
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2	132	123
3	119	-112
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5	79	76
6	177	170
7	43	39
8	20*	16
-11,K,1		
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2	68	71
3	124	118
4	85	-75
5	123	114
6	128	-113
0,K,2		
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1	231	-248
2	50	39
3	264	-274
4	620E	705
5	317	-323
6	358	371
7	244	249
8	140	143
9	18*	17
10	359	367
11	29	-36
12	211	209
13	62	60
14	72	-62
15	21*	11
1,K,2		
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2	50	-64
3	223	226
4	32	-24
5	213	219
6	16*	-184
7	562	596
8	44	-46
9	273	277
10	255	251
11	81	77
12	56	-55
13	62	62
14	44	-46
15	170	160
2,K,2		
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1	167	171
2	98	105
3	231	244
4	502	538
5	190	204
6	293	302
7	39	-40
8	256	256
9	278	-277
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11	26	35
12	152	145
13	177	170
14	109	110
15	83	-73
3,K,2		
1	243	253
2	250	268
3	374	400
4	222	-230
5	288	294
6	424	-455
7	141	137
8	143	143
9	95	88
10	237	235
11	274	268
12	20*	-13
13	68	68
14	64	-61
4,K,2		
0	272	287
1	201	-213
2	146	148
3	259	-275
4	121	109
5	244	253
6	285	291
7	19*	-17
8	419	427
9	98	-99
10	106	96
11	24	28
12	29	27
13	78	76
14	160	155
5,K,2		
1	24	-0
2	113	-118
3	302	305
4	132	-134
5	348	351
6	46	39
7	132	124
8	176	176
9	165	155
10	69	-60
11	247	240
12	30	-25
13	44	53
6,K,2		
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1	31	30
2	235	233
3	18*	-10
4	21	-1
5	22	21
6	199	198
7	19	-7
8	217	218
9	64	59
10	71	75
11	56	-45
12	23	21
7,K,2		
1	48	43
2	227	-228
3	218	216
4	32	-22
5	229	228
6	66	65
7	110	110
8	77	-69
9	152	141
10	35	-33
11	104	100
8,K,2		
0	111	109
1	192	194
2	131	127
3	150	150
4	104	108
5	197	-183
6	174	172
7	74	-69
8	113	112
9	128	111
9,K,2		
1	103	110
2	64	-70
3	72	73
4	77	71
5	127	122
6	68	65
7	178	165
10,K,2		
0	51	44
1	21*	5
2	143	136
3	21*	-8
-1,K,2		
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2	595E	-729
3	89	86
4	226	-239
5	89	-87
6	371	398
7	414	434
8	44	-41
9	276	291
10	35	-39
11	34	-39
12	31	-22
13	197	186
14	73	62
15	203	191
-2,K,2		
0	395E	-478
1	187	-215
2	239	267
3	217	236
4	497E	564
5	153	-171
6	448	488
7	38	-35
8	194	194
9	187	196
10	342	345
11	27	-23
12	160	164
13	154	-147
14	40	-31
15	37	31
-3,K,2		
1	178	182
2	69	69
3	247	267
4	192	-195
5	443	478
6	90	103
7	244	254
8	140	-141
9	266	274
10	104	-106
11	126	128
12	201	191
13	167	161
14	105	92
15	41	48
-4,K,2		
0	75	82
1	76	81
2	366	394
3	43	49
4	145	149
5	260	-271
6	431	450
7	300	-315
8	320	331
9	234	234
10	50	43
11	185	180
12	60	67
13	69	-70
14	179	173
15	28	-25
-5,K,2		
1	125	117
2	88	95
3	430	458
4	134	137
5	440	459
6	18*	12
7	83	-87
8	24	-27
9	164	167
10	97	-96
11	297	297
12	49	51
13	81	75
14	21*	-1
-6,K,2		
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1	16*	-5
2	142	131
3	298	-316
4	55	-59
5	18*	-4
6	372	382
7	99	103
8	483	499
9	25	28
10	64	67
11	22	-23
12	20*	7
13	21*	8
-7,K,2		
1	104	97
2	133	-144
3	289	292
4	19	-28
5	245	242
6	106	-107
7	100	95
8	97	94
9	172	171
10	58	57
11	227	226
12	59	-55
13	35	40
-8,K,2		
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1	119	-123
2	174	170
3	238	-243
4	164	159
5	82	79
6	110	106
7	100	100
8	146	149
9	102	-98
10	123	120
11	21*	-2
12	88	82
-9,K,2		
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2	237	241
3	116	113
4	52	48
5	156	154
6	154	-150
7	280	266
8	37	19
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10	71	64
-10,K,2		
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1	78	-82
2	86	93
3	43	-44
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7	53	-37
8	31	28
9	54	-45
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2	23	-32
3	28	26
4	75	63
5	80	74
6	39	34
-12,K,2		
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0,K,3		
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1	121	-140
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3	424	-464
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5	286	301
6	256	309
7	135	144
8	127	125
9	184	-193
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12	151	145
13	134	125
14	156	153
15	49	-33
1,K,3		
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2	239	248
3	434	457
4	17*	-2
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6	417	-440
7	124	-127
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9	135	138
10	165	160
11	251	253
12	20*	-8
13	134	124
14	45	-38
2,K,3		
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1	107	119
2	327	332
3	84	-91
4	35	-45
5	54	-51
6	305	312
7	88	-88
8	413	419
9	25	-28
10	88	88
11	125	118
12	20*	4
13	79	70
14	166	159
3,K,3		
1	345	355
2	22	5
3	261	258
4	67	74
5	298	297
6	39	31
7	78	-71
8	46	-45
9	89	88
10	20*	-19
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12	40	40
13	147	140
4,K,3		
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2	342	352
3	52	50
4	117	-113
5	179	-185
6	158	154
7	19*	16
8	175	175
9	156	155
10	49	53
11	44	-34
12	44	51
13	106	-95
5,K,3		
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2	397	-407
3	207	204
4	139	-130
5	191	195
6	241	239
7	74	74
8	44	-43
9	71	75
10	69	-67
11	108	105
12	104	87
6,K,3		
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1	56	67
2	124	126
3	131	131
4	237	237
5	125	-125
6	171	164
7	20*	-16
8	82	78
9	113	108
10	132	126
11	22*	-19
7,K,3		
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2	27	-26
3	92	88
4	20*	-3
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6	43	44
7	171	164
8	48	-41
9	108	108
8,K,3		
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1	20*	8
2	146	140
3	36	33
4	197	196
5	21*	-6
6	21*	26
7	68	-57
9,K,3		
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2	48	48
3	36	34
-1,K,3		
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2	16*	1
3	471	507
4	34	31
5	110	114
6	91	-93
7	269	273
8	199	210
9	196	196
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11	81	89
12	155	-145
13	116	111
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-2,K,3		
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6	168	176
7	108	94
8	19*	-11
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12	257	252
13	61	56
14	41	43
15	22*	14
-3,K,3		
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2	122	124
3	160	159
4	35	-43
5	44	-42
6	26*	0
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8	66	-71
9	63	65
10	25	-10
11	21	7
12	69	67
13	165	164
14	22*	2
-4,K,3		
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1	54	56
2	383	416
3	318	327
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5	184	-195
6	90	-93
7	183	-183
8	38	27
9	123	123
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11	85	80
12	190	183
13	71	-68
14	51	44
-5,K,3		
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2	17*	7
3	17*	-13
4	25	-30
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6	125	129
7	381	386
8	160	-165
9	129	129
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11	91	84
12	106	104
13	94	98
14	77	65
-6,K,3		
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3	37	-41
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5	289	-296
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7	19*	-12
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10	103	105
11	55	50
12	97	99
13	74	-69
-7,K,3		
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2	283	-288
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6	208	210
7	43	-44
8	20*	-18
9	96	96
10	74	-77
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12	52	-30
-8,K,3		
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3	33	-27
4	76	-80
5	40	-34
6	229	231
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8	187	194
9	35	10
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11	56	-46
-9,K,3		
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2	36	-32
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4	43	-34
5	148	203
6	88	-84
7	33	-26
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10	76	61
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3	20*	-13
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6	46	103
7	58	-48
8	64	89
-11,K,3		
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2	153	139
3	113	112
4	28	-24
5	53	95
6	135	-114
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1	72	66
2	249	250
3	109	107
4	27*	-15
5	90	-89
6	299	301
7	76	84
8	304	304
9	21*	-19
10	98	94
11	29	-31
12	40	43
13	22*	-9
1,K,4		
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2	42	34
3	218	222
4	327	329
5	208	208
6	188	194
7	81	73
8	241	-241
9	149	146
10	78	-71
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12	21*	6
13	78	75
2,K,4		
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1	215	223
2	235	314
3	227	230
4	108	114
5	306	-315
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7	24	-25
8	211	208
9	160	157
10	143	141
11	21*	-11
12	68	69
13	65	-79
3,K,4		
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2	158	-162
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5	149	147
6	206	206
7	230	232
8	124	-126
9	90	92
10	59	-91
11	121	111
12	67	65
4,K,4		
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1	19*	23
2	111	111
3	64	67
4	358	394
5	20*	-7
6	133	133
7	20*	-2
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9	81	81
10	124	125
11	52	-43
5,K,4		
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2	92	84
3	108	111
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5	71	68
6	57	51
7	196	193
8	34	30
9	28	46
10	109	-90
6,K,4		
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1	82	-81
2	123	122
3	75	-69
4	242	239
5	123	113
6	83	83
7	38	-28
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7,K,4		
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2	171	161
3	54	57
4	21*	-2
5	21*	18
6	124	-110
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1	30	-27
2	101	100
3	21*	-12
-1,K,4		
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2	52	61
3	496	526
4	19*	-7
5	395	414
6	26	-30
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8	50	54
9	148	146
10	20*	22
11	175	175
12	121	-104
13	158	143
14	22*	9
-2,K,4		
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3	131	-127
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6	324	330
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8	49	54
9	175	-176
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11	68	-58
12	153	145
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14	193	176
-3,K,4		
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5	275	279
6	139	-138
7	146	140
8	144	152
9	255	248
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11	96	100
12	127	-121
13	98	88
14	94	-74
-4,K,4		
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2	290	296
3	23	-33
4	171	165
5	222	238
6	112	112
7	108	-104
8	140	143
9	115	-117
10	205	205
11	20*	17
12	163	155
13	70	64
-5,K,4		
1	501	515
2	163	177
3	41	-40
4	29	27
5	58	54
6	131	-137
7	310	318
8	22	22
9	159	150
10	20*	7
11	44	35
12	40	28
13	70	75
-6,K,4		
0	34	15
1	168	175
2	288	290
3	157	163
4	422	440
5	88	80
6	57	-55
7	20*	-7
8	37	29
9	37	-35
10	202	203
11	89	-76
12	152	140
-7,K,4		
1	224	215
2	190	-190
3	19*	-10
4	73	73
5	184	190
6	131	137
7	358	357
8	28	-23
9	58	-57
10	86	-78
11	46	37
12	22*	-8
-8,K,4		
0	76	66
1	58	96
2	151	148
3	60	63
4	307	298
5	108	-111
6	92	90
7	35	31
8	38	45
9	57	57
10	101	104
11	39	-37
-9,K,4		
1	158	153
2	128	-125
3	229	215
4	50	50
5	83	86
6	126	118
7	39	47
8	62	-61
9	70	75
-10,K,4		
0	313	289
1	107	104
2	81	80
3	192	176
4	23	-18
5	32	-29
6	185	176
7	43	-42
-11,K,4		
1	43	32
2	35	23
3	225	209
4	22*	-1
5	101	98
0,K,5		
0	284	286
1	251	256
2	159	154
3	70	73
4	213	213
5	238	-238
6	71	72
7	21*	-16
8	140	138
9	124	123
10	158	162
11	22*	-18
12	112	104
1,K,5		
1	218	216
2	209	-213
3	28	-21
4	59	58
5	99	92
6	52	60
7	360	354
8	42	-41
9	231	214
10	36	-40
11	71	67
12	78	65
2,K,5		
0	20*	2
1	57	61
2	187	185
3	20*	-8
4	261	258
5	55	54
6	65	67
7	24	-18
8	112	101
9	21*	5
10	196	192
11	22*	-5
3,K,5		
1	199	202
2	131	126
3	20*	21
4	34	-30
5	168	157
6	76	-76
7	244	239
8	84	76
9	98	94
10	25	-15
4,K,5		
0	86	-72
1	158	-153
2	152	148
3	132	-127
4	252	243
5	125	120
6	136	136
7	23	-23
8	22*	28
9	74	-69
5,K,5		
1	40	37
2	192	177
3	109	107
4	38	-27
5	186	173
6	101	-55
7	143	135
6,K,5		
0	188	177
1	82	-71
2	100	103
3	25	-30
4	22*	19
5	76	69
-1,K,5		
1	32	28
2	266	-265
3	291	297
4	142	149
5	226	229
6	175	179
7	205	201
8	135	-139
9	84	78
10	73	-70
11	131	124
12	59	57
-2,K,5		
0	170	169
1	85	87
2	22	4
3	150	157
4	223	222
5	120	-121
6	336	338
7	20*	2
8	216	217
9	57	63
10	54	57
11	26	-27
12	50	56
-3,K,5		
1	77	-88
2	43	-48
3	385	395
4	180	-180
5	275	292
6	23	19
7	90	82
8	21*	11
9	121	126
10	49	41
11	197	188
12	22*	7
-4,K,5		
0	426	436
1	202	-212
2	187	187
3	53	40
4	47	-53
5	121	129
6	226	229
7	35	-28
8	255	257
9	86	-87
10	68	66
11	22*	19
12	22*	28
-5,K,5		
1	145	141
2	266	271
3	180	180
4	106	-108
5	168	167
6	91	-93
7	72	74
8	69	77
9	243	231
10	59	53
11	181	167
12	87	-79
-6,K,5		
0	22	28
1	143	-143
2	162	154
3	71	-72
4	85	86
5	146	150
6	232	223
7	21*	8
8	245	233
9	130	-120
10	124	123
11	22*	2
-7,K,5		
1	196	187
2	36	34
3	54	54
4	49	-54
5	199	191
6	129	-126
7	159	159
8	42	47
9	106	99
10	92	80
-8,K,5		
0	182	-167
1	51	42
2	155	150
3	37	-36
4	336	331
5	42	41
6	127	116
7	53	49
8	22*	11
9	33	-35
-9,K,5		
1	155	161
2	69	-69
3	21*	-4
4	67	60
5	111	101
6	22*	8
7	252	238
8	26	-18
-10,K,5		
0	24*	-0
1	82	74
2	80	77
3	22*	-2
4	198	185
5	90	-86
6	89	80
-11,K,5		
1	162	148
2	72	-63
0,K,6		
0	25*	35
1	27	-28
2	226	225
3	53	-56
4	179	180
5	82	74
6	73	68
7	51	45
8	31	33
9	77	-66
10	161	153
1,K,6		
1	317	304
2	35	40
3	21*	1
4	120	-115
5	66	68
6	103	-98
7	128	128
8	96	92
9	120	116
2,K,6		
0	58	58
1	178	-170
2	182	172
3	113	-104
4	129	125
5	71	74
6	75	79
7	36	28
8	101	101
3,K,6		
1	37	44
2	123	125
3	142	135
4	79	-69
5	196	183
6	82	-76
7	84	76
4,K,6		
0	224	214
1	35	-37
2	103	102
3	46	25
4	47	48
5	22*	14
5,K,6		
1	23	26
-1,K,6		
1	213	217
2	121	-120
3	54	47
4	22*	-1
5	88	88
6	45	-40
7	200	202
8	22*	10
9	171	158
10	34	22
-2,K,6		
0	55	55
1	172	177
2	139	143
3	21*	1
4	271	265
5	157	-163
6	161	155
7	72	-68
8	35	-20
9	115	106
10	85	97
-3,K,6		
1	180	174
2	165	-194
3	216	217
4	42	52
5	101	103
6	78	76
7	102	104
8	63	-65
9	56	52
10	79	-70
-4,K,6		
0	212	212
1	110	109
2	79	80
3	43	38
4	137	133
5	114	-110
6	147	151
7	61	-46
8	167	161
9	151	133
10	94	86
-5,K,6		
1	157	155
2	145	-144
3	186	188
4	104	-98
5	51	88
6	71	64
7	61	61
8	23	9
9	80	86
10	54	-41
-6,K,6		
0	365	363
1	115	-103
2	142	141
3	77	-62
4	21*	-5
5	21*	-12
6	60	76
7	22*	-9
8	228	211
9	23*	12
-7,K,6		
1	83	87
2	115	118
3	218	210
4	41	-35
5	160	157
6	54	-57
7	23*	-0
8	23*	20
9	23*	36
-8,K,6		
0	106	96
1	115	-110
2	88	85
3	77	-68
4	96	93
5	26	37
6	172	160
7	32	24
-9,K,6		
1	94	94
2	74	67
3	142	129
4	57	-47
5	125	116
6	120	-109
-10,K,6		
0	83	73
1	62	-47
2	144	128
3	71	-61
0,K,7		
0	156	146
1	117	-110
2	105	59
3	91	-81
4	82	80
5	68	69
6	72	72
7	95	82
1,K,7		
1	82	78
2	23*	30
3	151	149
4	23*	11
5	123	115
2,K,7		
0	231	219
1	22*	30
2	94	89
3	52	37
4	31	28
-1,K,7		
1	209	204
2	124	114
3	101	95
4	23*	-27
5	40	48
6	88	-83
7	62	68
-2,K,7		
0	69	66
1	61	-56
2	134	142
3	33	39
4	173	172
5	97	87
6	89	84
7	26	13
8	23*	-1
-3,K,7		
1	157	158
2	146	131
3	71	70
4	24	22
5	67	69
6	44	-42
7	152	147
8	22*	-15
-4,K,7		
0	38	29
1	22*	11
2	152	146
3	23*	16
4	173	172
5	46	-40
6	102	100
7	40	-31
8	39	-19
-5,K,7		
1	229	217
2	94	-95
3	59	65
4	38	35
5	52	51
6	134	117
7	87	85
-6,K,7		
0	103	106
1	95	90
2	162	147
3	46	41
4	83	85
5	51	-47
6	44	53
7	23*	-5
-7,K,7		
1	109	102
2	113	-95
3	90	89
4	50	47
5	134	119
6	87	73
-8,K,7		
0	199	181
1	83	69
2	129	113
3	32	28
4	50	45
-2,K,8		
0	162	147
1	25*	1
-3,K,8		
1	58	63
2	134	119
-4,K,8		
0	106	92
1	76	-71
2	54	61
-5,K,8		
1	73	71

those for the hydrogen atoms from the magnesium salt (Montgomery & Lingafelter, 1964*b*). Scattering factors for the vanadium ion were taken from *International Tables for X-ray Crystallography* (1962); for the ferrous ion from Thomas & Umeda (1957); for cobalt and sulfur from Viervoll & Øgrim (1949); for oxygen and nitrogen from Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) and for hydrogen from Table II of Stewart, Davidson & Simpson (1965). Dispersion corrections were made for vanadium ($\Delta f'=0{\cdot}2$, $\Delta f''=2{\cdot}3$) and for iron ($\Delta f'=-2{\cdot}67$, $\Delta f''=0$).

The refinement of all three structures was carried out by full-matrix least squares, first in isotropic and then in anisotropic mode, where the anisotropic temperature factor was of the form

$$\exp\left[-\{\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl\}\right].$$

The calculations were carried out on an IBM 7094 computer, with programs adapted or developed at the University of Washington (Stewart, 1964). The function minimized was $\Sigma\, w(F_o-F_c)^2$, with a modified Hughes weighting scheme for the vanadium and iron compounds and all $w=1$ for the cobalt compound. In each cycle, unobserved reflections for which $F_c<F_o$ were given zero weight. The hydrogen atom positions were not refined in the vanadium and iron compounds and an average temperature factor ($B=2{\cdot}5$) was used for the hydrogen atoms in all calculations. (Because of the close agreement between cell dimensions and heavy atom fractional coordinates of the vanadium, iron, and magnesium salts, it is felt that the refined hydrogen coordinates from the magnesium salt are as valid as recalculated assumed positions for the other salts. All distances and angles involving hydrogen atoms appear to be reasonable.) Hydrogen atom positions and isotropic temperature factors were refined for the cobalt compound.

During the final cycles of refinement the average parameter shifts were $0{\cdot}02\sigma$ for the vanadium, $0{\cdot}01\sigma$ for the iron, and $0{\cdot}1\sigma$ for the cobalt compound and the maximum shifts were $0{\cdot}07\sigma$ for the vanadium, $0{\cdot}02\sigma$ for the iron, and $0{\cdot}7\sigma$ for the cobalt compound. The final values of the R index were 0·080 for the vanadium, 0·059 for the iron and 0·050 for the cobalt compound. ($R=\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$, omitting unobserved reflections, and those suspected of having large secondary extinction effects.)

The final values of the parameters are listed in Table 1, the observed and calculated structure factors in Tables 2, 3 and 4, bond lengths and angles in Table 5, and hydrogen-bond distances in Table 6.

Table 5. *Bond lengths* (Å) *and angles* (°)

	V	Fe	Co
M–O(7)	2·16 (1)	2·156 (7)	2·107 (3)
M–O(8)	2·16 (1)	2·136 (7)	2·106 (4)
M–O(9)	2·12 (1)	2·086 (7)	2·070 (4)
S—O(3)	1·49 (1)	1·487 (8)	1·472 (4)
S—O(4)	1·46 (1)	1·454 (8)	1·448 (4)
S—O(5)	1·51 (1)	1·483 (8)	1·475 (3)
S—O(6)	1·46 (1)	1·480 (8)	1·468 (4)
O(7)–M–O(8)	89·5 (3)	89·3 (2)	88·9 (1)
O(7)–M–O(9)	92·0 (3)	90·9 (2)	91·5 (1)
O(8)–M–O(9)	90·6 (3)	91·2 (2)	89·4 (1)
O(3)–S—O(4)	109·6 (4)	109·9 (4)	109·8 (3)
O(3)–S—O(5)	108·3 (4)	107·9 (4)	108·2 (2)
O(3)–S—O(6)	109·1 (4)	109·7 (4)	109·9 (2)
O(4)–S—O(5)	109·7 (4)	109·3 (4)	108·8 (2)
O(4)–S—O(6)	111·2 (4)	110·7 (4)	110·6 (2)
O(5)–S—O(6)	108·9 (4)	109·3 (4)	109·5 (2)

Discussion

The metal–oxygen distances show the same pattern as in the other Tutton's salts (with the exception of copper): two longer equatorial bonds and a slightly shorter axial bond.

The pattern for the iron compound is essentially identical with that (2·14, 2·14, 2·07 Å) observed in $FeSO_4.7H_2O$ by Baur (1964) for one iron atom, Fe(1), which has the same environment, namely six water molecules, each bonded by two hydrogen bonds to oxygen atoms of surrounding sulfate ions.

Thus there is no evidence of any special tendency for the FeO_6 octahedron in the hexa-aquoferrous ion to distort. This is contrary to the distortion reported for the FeF_6 octahedron reported in FeF_2 (Baur, 1957; Stout & Shulman, 1960) and predicted for the hexa-aquoferrous ion by Hatfield & Piper (1964).

Table 6. *Hydrogen bond distances*

Bond	Relative position of second atom*	Bond distance (Å)		
		V	Fe	Co
O(7)–O(5)	x, y, z	2·75 (2)	2·78 (2)	2·793 (6)
O(7)–O(6)	$x, y, z-1$	2·86 (2)	2·83 (2)	2·831 (6)
O(8)–O(4)	$x-1, y, z-1$	2·74 (2)	2·73 (2)	2·714 (6)
O(8)–O(6)	$x-\frac{1}{2}, \frac{1}{2}-y, z-1$	2·86 (2)	2·76 (2)	2·769 (6)
O(9)–O(3)	$\frac{1}{2}-x, y-\frac{1}{2}, 1-z$	2·73 (2)	2·72 (2)	2·707 (6)
O(9)–O(5)	$-x, -y, 1-z$	2·78 (2)	2·76 (2)	2·764 (6)
N–O(3)	x, y, z	3·00 (3)	2·99 (2)	2·974 (7)
N–O(3)	$x-\frac{1}{2}, \frac{1}{2}-y, z$	3·03 (3)	3·02 (2)	2·995 (7)
N–O(4)	$x-\frac{1}{2}, \frac{1}{2}-y, z$	3·15 (3)	3·12 (2)	3·155 (7)
N–O(5)	$\frac{1}{2}-x, \frac{1}{2}+y, 1-z$	2·90 (3)	2·86 (2)	2·842 (7)
N–O(6)	$x-\frac{1}{2}, \frac{1}{2}-y, z-1$	2·93 (3)	2·92 (2)	2·910 (7)

* This is the transformation relating each 'second atom' to the corresponding atom in the original asymmetric unit.

The general packing of the ions and the hydrogen-bond network is essentially identical with that found in the other Tutton's salts, that is, each water molecule forms two hydrogen bonds (Table 6) which range in length from 2·71 to 2·86 Å. The ammonium ion is also hydrogen-bonded to the oxygen atoms of the sulfate groups, but one bond (to O(3) and O(4) of the same sulfate ion) is 'bifurcated'. In addition, there is a fairly close approach of the water octahedra along the *c* axis, the O(9)–O(9′) distance being 3·00 Å for the vanadium, 3·03 Å for the iron, and 3·02 Å for the cobalt compound.

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The Crystal Structure of Bis-(*N*-isopropyl-3-ethylsalicylaldiminato)nickel

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The crystal structure of bis-(*N*-isopropyl-3-ethylsalicylaldiminato)nickel, $Ni(C_{12}H_{16}NO)_2$, has been determined from three-dimensional data collected on a single-crystal diffractometer with Cu $K\alpha$ radiation. The cell has dimensions $a = 26{\cdot}947$, $b = 19{\cdot}883$, and $c = 8{\cdot}820$ Å, belongs to space group *Pbca*, and contains eight molecules. The structure consists of discrete molecules in which nickel(II) exhibits a distorted-tetrahedral coordination configuration. The dihedral angle between the plane defined by the Ni–N(1)–O(1) group and the plane defined by the Ni–N(2)–O(2) group is 85·3°.

Introduction

A striking example of the small energy difference between the diamagnetic and paramagnetic states of the nickel(II) ion has been provided by a series of ring-substituted salicylaldimine chelates of nickel(II). That is, Holm & Swaminathan (1963) have reported that for 3-substituted bis-(*N*-isopropylsalisylaldiminato)nickel chelates the magnetic moments are 3·28, 0, and 3·30 Bohr magnetons for the substituents hydrogen, methyl, and ethyl, respectively. Consistent with these magnetic moments, the coordination configuration of the 3-hydrogen chelate has indeed been shown to be tetrahedral (Fox, Oriolo, Lingafelter & Sacconi, 1964) and the coordination configuration of the 3-methyl chelate has been shown to be strictly planar (Braun & Lingafelter, 1966). We have now completed the structural determination of the 3-ethyl chelate by three dimensional X-ray diffraction techniques.

Experimental

Bis-(*N*-isopropyl-3-ethylsalicylaldiminato)nickel was prepared by the method of Sacconi, Paoletti & Del Re (1957), using 3-ethylsalicylaldehyde which was prepared according to the general procedure of the Duff (1941) reaction. Final purification was accomplished by two recrystallizations from equal volumes of methanol and 2-butanol by slow evaporation at room temperature. The crystals were in the form of flat needles, lath-like on (100) and elongated along [001]. A crystal of dimensions 0·14 × 0·12 × 0·03 mm was used