Acta Cryst. (1966). **21**, 726

Refinement of the Crystal Structure of Taurine, 2-Aminoethylsulfonic Acid An Example of Computer-Controlled Experimentation

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(*Received* 18 *February* 1966)

The crystal structure of taurine, 2-aminoethylsulfonic acid, was refined from three-dimensional intensity data collected on a computer-controlled diffractometer operated by an IBM 1620 machine under a stored data-collection program. The crystal belongs to the monoclinic system with $a=5{\cdot}27_9$, $b=11{\cdot}64_5$, $c=7{\cdot}93_1$ Å, $\beta=94{\cdot}1°$; the space group is $P2_1/c$. The positions of all the hydrogen atoms were obtained by a three-dimensional (F_o-F_c) synthesis.

The molecule possesses the zwitterion configuration with the formula NH_3^+–CH_2–CH_2–SO_3^-; the amino and sulfonate groups assume a *gauche* configuration around the central methylene linkage. All bond distances and angles in the zwitterion are of normal values.

The molecules are held in the structure by a three-dimensional network of N–H---O hydrogen bonds of the usual strength. A brief discussion on the program for computer-controlled data collection is also presented.

Introduction

This work is part of the project currently undertaken by the author on systematic studies on the precise molecular configurations of aliphatic amino acids with acidic groups other than carboxyl groups. Taurine, 2-aminoethylsulfonic acid, is commonly found in animals, and the knowledge of its configuration is important in understanding the metabolism of some amino acids in such a physiological environment. The crystal structure of taurine has been studied by Sutherland & Young (1963) based on three-dimensional diffraction data obtained by the photographic method. Although there is little doubt about the heavy-atom positions deduced from such data, it is difficult to accept some of the conclusions given by the authors based on the hydrogen atom positions obtained by a difference electron-density synthesis; this is especially so in view of the presence of rather heavy sulfur atoms in the structure and a relatively high R index (11% for observed reflections only). Since these authors, on the basis of their data, have not located three hydrogen atoms around the amino nitrogen atom, they have concluded that taurine might not be a zwitterion. In view of the bond distances and angles found in the taurine molecule it is quite difficult to accept the non-zwitterion structure; moreover it has been established by X-ray and neutron diffraction methods that many other similar amino acids with varieties of acidic groups possess zwitterion configurations.

Since the knowledge of the correct nature of taurine is important in understanding the biological role of the molecule, it seemed worthwhile to reinvestigate its crystal structure by more accurate and comprehensive intensity data. The present paper deals with three-dimensional refinement of the structure using intensity data collected on our computer-controlled diffractometer (Cole, Okaya & Chambers, 1963). The work might also be used as an example of continuous flow of computer usage in crystallography. After the definition of the problem and manipulation of specimens, the computers play crucial roles in all the subsequent phases of the crystallographic study; they include data collection, data analyses, and finally presentation of the results.

A needle-shaped single crystal of taurine was cut and ground into an approximately spherical shape of about 0·3 mm in diameter and mounted on a General Electric goniostat which was placed on a Picker biplane diffractometer. The diffractometer is part of the CCXD, a computer-controlled diffractometer system which is operated in a closed-loop manner by an IBM 1620 machine under a stored data collection program. As the first step of automatic* data collection processes, the cell constants were studied by using Mo $K\alpha$ radiation on the diffractometer. The cell dimensions thus obtained are: $a=5{\cdot}27_9$, $b=11{\cdot}64_5$, $c=7{\cdot}93_1$ Å, $\beta=94{\cdot}1°$; $a:b:c=0{\cdot}4533:1:0{\cdot}6811$. These axial ratios are in relatively good agreement with those listed by Groth (1910) (0·4539:1:0·6827 by exchanging a and c). The observed cell constants were so chosen that the 2θ values of reflections can be calculated within the tolerance of the diffractometer system in the range of 2θ employed in the present study. The space group $P2_1/c$ as given by Sutherland & Young is employed without further checking the systematic absences.

* Work supported in part by AFCRL 19(628)-2469.

* We should draw a clear distinction between a simply automated system and a computer-controlled system; however, the adjective *automatic* is used here in such a way as to describe processes which require minimum participation of human beings in repetitive operations including combinations of hardware manipulations as well as logical decisions.

Computer-controlled data collection

Three-dimensional integrated intensity data within the range of sin $\theta/\lambda \leq 0{\cdot}91$ were obtained from the specimen by again using Mo $K\alpha$ radiation. Since the computer-controlled diffractometer system has been used successfully in the past two years on the structure analyses of more than two dozen crystals, it might be justifiable to explain briefly the actual computer program used in the data collection.

The program is divided into two parts. The first part deals with the initialization of the task; from the cell constants and orientation of the crystal are calculated the corresponding values for the reciprocal lattice. If requested, the upper limits of the Miller indices are established for the range of sin θ/λ given as an input parameter and other necessary housekeeping work. After this initialization has been performed, this part of the program can be overlaid by the main data collection program.

The second part concerns the actual data collection which can be divided into two sub-parts, one which is relatively independent of the crystals being examined or the methods of data collection and the other part, a subroutine for manipulating the diffractometer, which might have to be modified according to the mode of data collection. The former generates all possible points in reciprocal space within the specified sin θ/λ range in the order given by input data. In order to shorten the time required in moving through reciprocal space, the reflections were generated as shown in Fig. 1. This method of generating the reciprocal lattice points may not make the traveling time minimum; however as discussed later, the data collection scheme employed presently spends more time around each reciprocal lattice point than that required for traveling from one point to the next, and this method of generating reciprocal lattice points does not seem too unreasonable. The reciprocal lattice points must of course be tested for the systematic absences or any other conditions imposed by the experimental need. These restrictions can be expressed by rewriting this portion of the subroutine for each problem.

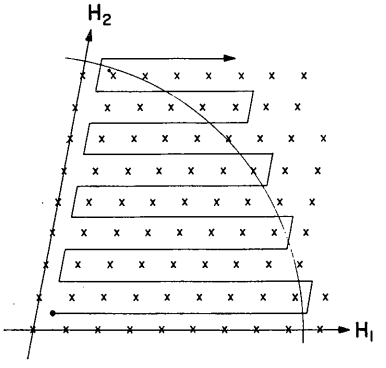

Fig. 1. The method of generating reflections used by the present program in a constant H_3 section. H_1 is the most frequently changing index, usually along the longest real cell dimension (or the shortest for reciprocal lattice). H_3 (not shown in the figure) is the least frequently changing Miller index. The negative sides of H_1 and/or H_2 can also be generated in the same manner.

The subroutine used in manipulating the diffractometer may have to be modified but not necessarily be rewritten for each crystal. The basic concept in programming for such a hardware control operation is to ensure steady flow of manipulation with maximum possible care in checking the validity of obtained data. It should be emphasized here that by having a computer-controlled diffractometer system, it is not possible to speed up the data collection task into the millisecond realm. The advantage is gained only by continuous, steady operation, elimination of clerical mistakes and use of logical ability of computers to test the obtained results at the time they are obtained.

From the programming standpoint, the diffractometer can be treated as an I/O (input-output) unit, from and to which information exchange can be made by the computer. The output from the computer is considered as commands to the diffractometer; they include new angular settings, presetting values of the counting devices, new attenuator settings and others. Input to the computer is usually the experimental conditions of the diffractometer such as present angular settings, contents of the counting devices, the filter settings and the status of the hardware. The computer also performs the following control tasks: to start and stop motors, preset, reset and start the counting devices, place the filters and attenuators in the X-ray beam, and others. For actual detailed discussion of the diffractometer system, refer to our previous article (Cole, Okaya & Chambers, 1963). In order to achieve the most important merit of computer control, it becomes necessary to program the subroutine in such a way that the experiments can be performed with the same kind of attention as given by a human operator; it is rather remarkable that, during the performance of an experiment, many logical decisions are actually made by the human operator, either consciously or subconsciously. In Fig. 2, a flow diagram of the logical decisions made by the computer after each measurement (count, in the case of diffraction experiments) is demonstrated.

The three-dimensional data used in the present analysis of the structure of taurine were obtained under the principle outlined above by using filtered Mo $K\alpha$ radiation. For each reflection, the alignment of the crystal and the instrument were first studied by optimizing the ω setting on the biplane diffractometer*. The integrated intensity was then recorded by step-scanning around the 2θ axis; the number of steps used is 24. The intervals of the 2θ step scanning had been

* If intensity measurements are made at large 2θ values (usually when a longer wavelength is employed), it might also become necessary to study the χ setting. For certain experimental conditions, the effect of missetting the χ arc becomes more important than that of the ω direction for reflections with large 2θ values.

determined by checking the profile of several reflections in various 2θ ranges; they were 9/100° for reflections below 40° in 2θ and 11/100° thereabove. These intervals were determined in such a way that the first three and the last three steps out of the 24 2θ step-scan data for a reflection do represent the background at its 2θ value. These values are also dependent on the experimental conditions – such as the speed of data collection, use of a monochromator, and others. In the course of optimizing the ω setting of a reflection, the maximum and minimum counts were recorded and if the difference between them was less than the statistical fluctuation, the reflection was treated as a non-observed one. The detailed data collection program, written in the IBM 1620 SPS (Symbolic Programming System) language, is explained elsewhere (Okaya, 1964). In order to facilitate the programming tasks, the programming system had also been modified to accomodate additional instruction sets required for the hardware manipulation. Out of the 3124 non-equivalent reflections accessible within the region of $\sin\theta/\lambda \leq 0{\cdot}91$, 2639 reflections were recorded as observed. This relatively large number of non-observed reflections is due to the 2θ limit value, which is high for reflections from such an organic compound, and also the rather stringent condition in accepting the intensity data.

The integrated intensity data of reflections were calculated from the data on count, time, and attenuator settings for the 24 steps on the (θ–2θ) step scanning. The computation was done as a time-shared program on the 1620. On account of the small linear absorption coefficient of the sample for the Mo radiation, no absorption correction was made.

Refinement of the structure

The structure was refined in a straightforward fashion by using the three-dimensional data thus obtained. Starting from the values given by Sutherland & Young (1963), the atomic coordinates of the seven non-hydrogen atoms were refined by using a full-matrix least-squares program on an IBM 7094 computer (Okaya, 1963); the isotropic temperature factors given by them were converted into the corresponding anisotropic temperature factors and also used as the starting parameters. The positions of hydrogen atoms were then studied by the usual three-dimensional ($F_o - F_c$) synthesis method. All the four hydrogen atoms on the two carbon atoms were located at positions close to those given by Sutherland & Young (1963). However, contrary to their results, three peaks were located around the nitrogen; these three peaks are at distances of about 0·9 Å from the nitrogen and, together with the carbon atom bonded to it, form a tetrahedral arrangement around the nitrogen. Two of these peaks are near the positions of the two hydrogen atoms, H(5) and H(6), given in the previous article; no electron density was found around its last hydrogen atom, H(7). A composite diagram of the difference synthesis is shown in Fig. 3. The atomic coordinates of these seven hydrogen atoms were then subjected to the least-squares treatment by assigning isotropic temperature

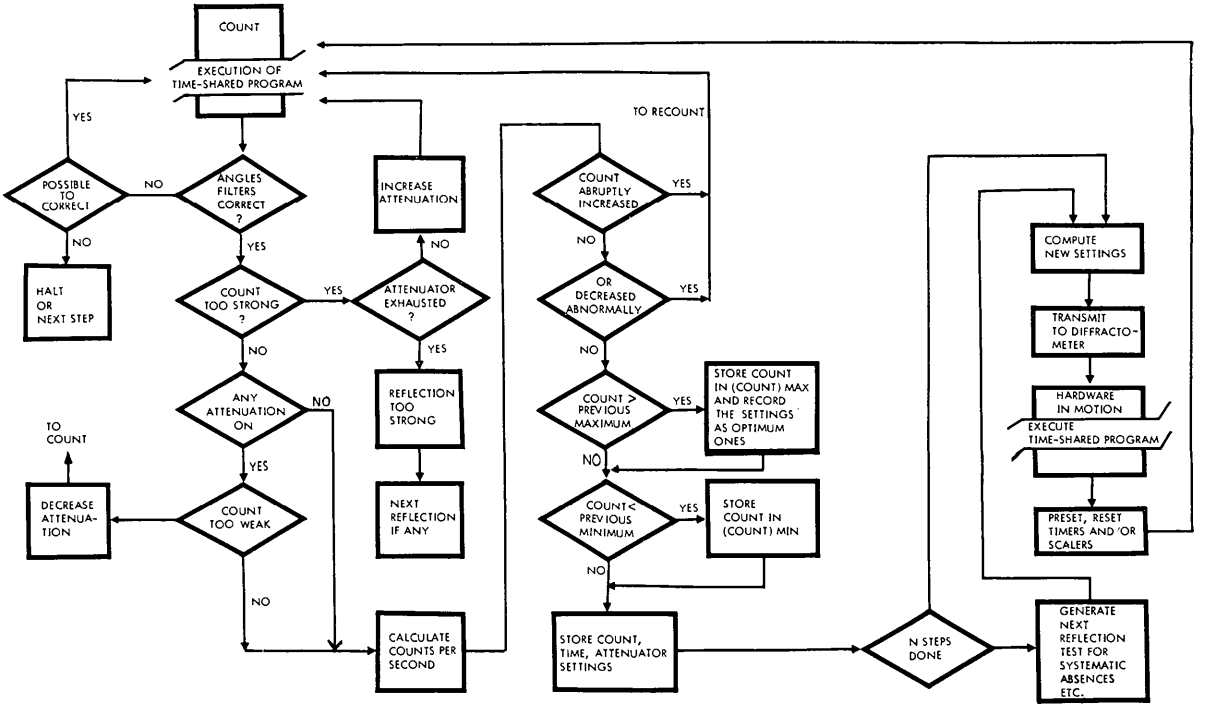

Fig. 2. A flow chart of the data collection scheme stressing logical decisions made by the computer to ensure the validity of the obtained data. After each measurement (or count), the computer tests for any abnormal condition which invalidates the results. This decision-making routine is, as programming technique, a copy of decisions made by a *conscientious* human operator.

factors to account for their thermal motions. The final conventional error factor $R=\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$ was reduced to 0·040 for the observed reflections (or 0·059 when all accidentally absent reflections were introduced as $F_o\equiv 0$). The atomic coordinates, their standard deviations and thermal parameters at the final stage are listed in Table 1. The shifts of parameters at this stage are quite negligible. To confirm further our basis for rejecting the previous H(7) position, the similar calculation was performed by replacing the coordinates of H(7) in Table 1 by the values for H(7) in the previous report. This gave an R of 0·044 (for the observed reflections only) with subsequent abnormal increase in the temperature factor of this hydrogen atom*.Except for this seventh hydrogen atom, the coordinates given in the previous paper are correct within reasonable multiples of their standard deviations.. The following are some of the computational details for the data-analysis stage. All the necessary computation was done on an IBM 7094 machine at the IBM Research Center; the atomic scattering factors used in the structure factor calculations were obtained from the values in *International Tables for X-ray Crystallography*. The weighting scheme used in the least-squares analysis is $\omega=1\cdot0$ for $|F_{obs}|\leq 20\cdot0$ and $\omega=20\cdot0/|F_{obs}|$ for $|F_{obs}|\geq 20\cdot0$ and unobserved reflections were given zero weight; this weighting scheme is employed to represent errors in observed structure factors rather realistically, and not those in observed intensities. Comparison between the observed and calculated structure factors is given in Table 2. Interatomic distances and bond angles were calculated from the atomic coordinates in Table 1(*a*).

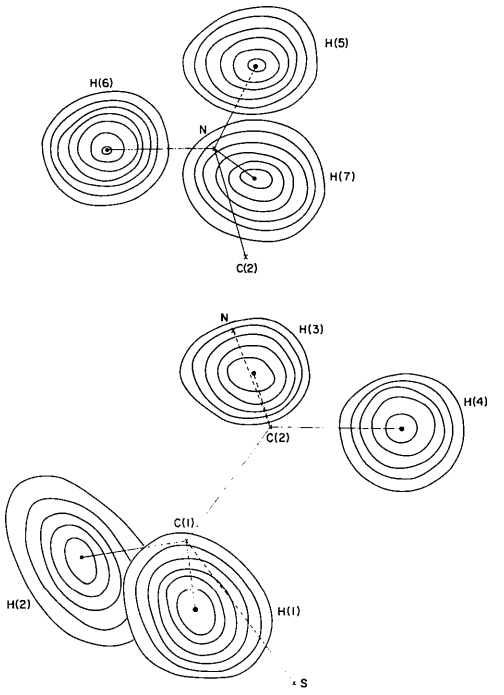

Fig. 3. A composite diagram of the electron-density function associated with the hydrogen atoms; the contours are drawn at equal intervals of 0·1 e.Å^{-3} starting from 0·2 e.Å^{-3}.

Table 1(*a*). *Atomic coordinates in fractions of cell edges and their standard deviations*, σ (in 10^{-4} Å)

The standard deviations of the hydrogen atoms are 0·02–0·03 Å.

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
S	0·29666	2	0·15131	2	0·14910	3
O(1)	0·56437	8	0·16291	9	0·20700	13
O(2)	0·15802	10	0·25869	10	0·14570	12
O(3)	0·26797	9	0·08903	10	−0·01156	9
C(1)	0·16033	11	0·06153	11	0·30006	12
C(2)	0·28932	11	−0·05490	12	0·31863	12
N	0·23581	9	−0·12925	10	0·16840	10
H(1)	0·175		0·105		0·403	
H(2)	−0·015		0·056		0·260	
H(3)	0·234		−0·090		0·417	
H(4)	0·466		−0·044		0·342	
H(5)	0·290		−0·200		0·191	
H(6)	0·086		−0·129		0·139	
H(7)	0·305		−0·094		0·090	

Table 1(*c*). *Isotropic temperature factors for hydrogen atoms* (*in* 10^{-16} cm^2)

H(1)	H(2)	H(3)	H(4)	H(5)	H(6)	H(7)
1·2	1·6	0·9	2·1	1·1	0·6	2·1

Discussion of the results

The present study on the crystal structure of taurine has established unambiguously that taurine possesses the zwitterion configuration and its structural formula is written as NH_3^+–CH_2–CH_2–SO_3^-. As shown in Fig. 4, the zwitterion possesses a *gauche* configuration around the central methylene linkage. Fig. 5 shows bond distances in the taurine molecule, and in Table 3 are listed bond angles.

The configuration of the sulfonate group is rather interesting. O(1) and O(3), which are involved in rela-

* There is hardly any need to consult the table of Hamilton (1965) in deciding which of the two positions is the correct one for the seventh hydrogen atom.

Table 1(*b*). *Anisotropic temperature factors*

β's are used in the usual formula $\exp\{-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)\}$.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S	0·01275	0·00230	0·00899	0·00080	0·00324	−0·00036
O(1)	0·01450	0·00406	0·01814	−0·00375	−0·00227	−0·00212
O(2)	0·02862	0·00334	0·01503	0·00878	0·01138	0·00190
O(3)	0·02184	0·00424	0·00832	0·00017	0·00503	−0·00090
C(1)	0·01793	0·00350	0·00807	0·00015	0·00537	−0·00061
C(2)	0·02011	0·00354	0·00850	−0·00056	−0·00235	0·00163
N	0·01720	0·00293	0·00924	0·00083	0·00396	0·00049

Table 2. *Comparison between the observed and calculated structure factors*

The values in the Table have been multiplied by 10

	FO	FC
H,K= 0, 0		
L=2	512	587
4	249	259
6	449	459
8	0	8
10	50	52
12	32	31
14	27	23
H,K= 1, 0		
L=0	196	197
2	373	400
4	148	143
6	241	254
8	180	181
10	27	28
12	83	79
H,K= 2, 0		
L=0	767	875
2	43	28
4	223	217
6	331	351
8	0	6
10	60	56
12	58	58
H,K= 3, 0		
L=0	39	60
2	324	311
4	276	284
6	45	46
8	90	90
10	66	64
12	18	17
H,K= 4, 0		
L=0	222	200
2	176	163
4	104	104
6	143	141
8	20	16
10	38	39
12	60	61
H,K= 5, 0		
L=0	207	203
2	9	13
4	137	133
6	98	97
8	32	34
10	56	55
H,K= 6, 0		
L=0	0	10
2	73	72
4	59	54
6	102	101
8	51	50
10	0	4
H,K= 7, 0		
L=0	205	198
2	91	89
4	57	54
6	97	99
8	25	21
H,K= 8, 0		
L=0	74	74
2	20	14
4	29	28
6	18	22
H,K= 9, 0		
L=0	38	37
2	29	28
4	0	9
H,K= 0, 1		
L=1	377	399
2	502	552
3	13	5
4	267	265
5	53	51
6	149	146
7	196	196
8	103	101
9	20	16
10	13	10
11	32	30
12	60	59
13	47	48
14	0	4
H,K= 1, 1		
L=0	438	486
1	631	741
2	18	25
3	101	93
4	47	49
5	193	197
6	106	102
7	84	82
8	100	103
9	0	0
10	61	63
11	52	50
12	0	6
13	0	2
14	31	29
H,K= 2, 1		
L=0	178	154
1	291	278
2	298	284
3	69	67
4	92	89
5	110	104
6	107	106
7	154	155
8	115	116
9	59	58
10	40	41
11	0	15
12	29	24
13	30	29
H,K= 3, 1		
L=0	241	226
1	408	386
2	115	114
3	112	101
4	72	75
5	121	119
6	97	94
7	101	100
8	11	11
9	0	1
10	9	8
11	51	50
12	15	13
13	0	11
H,K= 4, 1		
L=0	217	216
1	28	29
2	63	69
3	48	44
4	77	74
5	154	153
6	61	62
7	23	22
8	28	25
9	22	24
10	37	37
11	19	24
12	0	2
H,K= 5, 1		
L=0	110	116
1	68	60
2	51	48
3	0	3
4	98	101
5	64	63
6	47	45
7	62	63
8	37	37
9	0	8
10	0	5
11	38	42
H,K= 6, 1		
L=0	133	130
1	43	41
2	47	47
3	39	37
4	88	84
5	128	123
6	95	96
7	33	31
8	0	5
9	0	2
10	21	30
H,K= 7, 1		
L=0	58	60
1	12	11
2	83	84
3	17	17
4	118	117
5	0	1
6	0	5
7	23	24
8	28	30
9	0	0
H,K= 8, 1		
L=0	80	78
1	45	51
2	80	78
3	28	27
4	11	9
5	34	33
6	29	33
7	16	16
H,K= 9, 1		
L=0	38	35
1	12	10
2	12	13
3	0	3
4	39	38
H,K= 0, 2		
L=0	82	128
1	342	378
2	163	132
3	104	90
4	271	255
5	153	151
6	37	33
7	32	33
8	57	56
9	57	59
10	82	78
11	30	32
12	0	0
13	42	49
14	0	13
H,K= 1, 2		
L=0	45	43
1	118	109
2	335	342
3	84	72
4	128	118
5	81	75
6	39	36
7	199	199
8	65	59
9	52	52
10	0	3
11	7	8
12	0	6
13	29	40
14	0	5
H,K= 2, 2		
L=0	63	56
1	135	138
2	97	86
3	58	60
4	207	203
5	130	127
6	24	22
7	53	53
8	11	8
9	30	29
10	43	42
11	44	43
12	0	2
13	12	20
H,K= 3, 2		
L=0	72	67
1	146	130
2	105	97
3	195	186
4	76	72
5	245	250
6	11	14
7	67	67
8	27	25
9	63	68
10	9	9
11	26	36
12	0	4
13	0	20
H,K= 4, 2		
L=0	55	54
1	187	181
2	80	82
3	229	234
4	35	38
5	59	62
6	16	17
7	36	37
8	34	33
9	0	2
10	4	2
11	13	26
12	0	4
H,K= 5, 2		
L=0	9	12
1	78	76
2	51	53
3	22	19
4	0	0
5	138	139
6	0	8
7	13	11
8	29	31
9	31	43
10	0	21
11	0	39
H,K= 6, 2		
L=0	10	12
1	76	73
2	95	95
3	136	135
4	56	56
5	41	42
6	0	0
7	52	62
8	27	42
9	21	42
10	0	15
H,K= 7, 2		
L=0	27	26
1	143	137
2	40	38
3	54	53
4	0	9
5	76	78
6	0	8
7	31	41
8	0	7
9	0	3
H,K= 8, 2		
L=0	30	30
1	27	27
2	31	32
3	49	49
4	30	32
5	34	43
6	0	10
7	0	20
H,K= 9, 2		
L=0	17	11
1	46	46
2	0	3
3	36	37
4	0	6
H,K= 0, 3		
L=1	460	516
2	214	209
3	590	655
4	22	22
5	205	188
6	37	42
7	132	133
8	6	7
9	78	79
10	29	28
11	16	22
12	18	20
13	27	29
14	0	3
H,K= 1, 3		
L=0	6	2
1	381	398
2	19	16
3	20	20
4	332	326
5	197	196
6	23	26
7	56	54
8	44	46
9	94	94
10	30	27
11	80	79
12	0	7
13	7	1
14	25	25
H,K= 2, 3		
L=0	126	117
1	193	180
2	35	34
3	363	359
4	124	125
5	138	136
6	13	8
7	142	142
8	49	55
9	127	128
10	59	61
11	0	11
12	0	1
13	31	33
H,K= 3, 3		
L=0	91	85
1	418	425
2	97	94
3	159	161
4	193	201
5	157	161
6	28	28
7	153	159
8	35	38
9	0	7
10	7	12
11	48	49
12	3	0
13	15	12
H,K= 4, 3		
L=0	95	90
1	167	166
2	104	103
3	127	122
4	97	98
5	138	142
6	18	22
7	39	36
8	0	38
9	77	81
10	34	38
11	12	16
12	0	3
H,K= 5, 3		
L=0	9	11
1	121	114
2	14	14
3	105	111
4	82	82
5	52	52
6	17	16
7	99	104
8	17	17
9	26	27
10	0	11
11	26	27
H,K= 6, 3		
L=0	50	51
1	54	54
2	28	27
3	72	75
4	29	25
5	105	109
6	26	27
7	0	10
8	0	0
9	49	45
10	14	7
H,K= 7, 3		
L=0	18	16
1	59	60
2	0	4
3	123	124
4	43	42
5	49	48
6	0	4
7	29	30
8	0	2
9	26	26
H,K= 8, 3		
L=0	31	30
1	56	56
2	24	21
3	32	33
4	18	17
5	14	14
6	5	12
7	0	6
H,K= 9, 3		
L=0	0	7
1	11	13
2	0	3
3	26	28
H,K= 0, 4		
L=0	554	623
1	307	323
2	80	68
3	63	66
4	47	42
5	134	127
6	185	179
7	69	70
8	26	26
9	70	70
10	52	50
11	32	30
12	30	27
13	19	12
14	18	16
H,K= 1, 4		
L=0	299	289
1	420	425
2	129	129
3	104	106
4	239	232
5	58	60
6	40	36
7	5	6
8	118	121
9	16	19
10	58	56
11	57	55
12	36	35
13	12	15
14	20	18
H,K= 2, 4		
L=0	364	340
1	48	58
2	169	165
3	120	117
4	60	54
5	219	222
6	245	256
7	141	142
8	33	35
9	23	25
10	51	49
11	50	50
12	50	48
13	32	31
H,K= 3, 4		
L=0	258	248
1	214	219
2	215	208
3	175	171
4	305	309
5	79	79
6	58	56
7	50	51
8	102	101
9	26	24
10	68	66
11	27	27
12	18	17
13	0	11
H,K= 4, 4		
L=0	218	213
1	155	154
2	223	216
3	111	110
4	48	46
5	71	71
6	143	144
7	98	97
8	40	36
9	8	12
10	23	25
11	29	28
12	38	37
H,K= 5, 4		
L=0	139	134
1	184	186
2	66	65
3	0	4
4	127	122
5	19	19
6	31	28
7	19	18
8	62	63
9	37	35
10	49	48
11	8	1
H,K= 6, 4		
L=0	73	71
1	67	67
2	99	96
3	82	82
4	45	43
5	16	22
6	69	67
7	60	57
8	44	40
9	26	27
10	0	5
H,K= 7, 4		
L=0	79	77
1	108	107
2	29	28
3	12	8
4	31	25
5	35	35
6	39	35
7	39	39
8	0	5
H,K= 8, 4		
L=0	37	38
1	35	36
2	0	2
3	30	29
4	34	31
5	18	19
6	0	8
H,K= 9, 4		
L=0	0	4
1	18	14
2	18	16
3	0	5
H,K= 0, 5		
L=1	199	188
2	138	133
3	131	118
4	112	105
5	56	49
6	6	8
7	86	94
8	41	39
9	5	0
10	50	48
11	18	16
12	42	43
13	12	15
14	0	3
H,K= 1, 5		
L=0	12	5
1	175	156
2	214	213
3	41	36
4	103	99
5	76	83
6	133	122
7	11	13
8	12	9
9	32	30
10	95	94
11	15	12
12	38	36
13	0	4
H,K= 2, 5		
L=0	352	342
1	201	193
2	35	28
3	69	55
4	180	172
5	69	65
6	27	26
7	53	54
8	74	75
9	17	13
10	7	4
11	19	16
12	38	34
13	13	9
H,K= 3, 5		
L=0	249	250
1	33	24
2	352	354
3	46	46
4	61	62
5	12	14
6	212	211
7	21	17
8	111	109
9	7	3
10	41	41
11	0	13
12	39	37
13	0	1
H,K= 4, 5		
L=0	396	411
1	142	141
2	149	148
3	7	10
4	110	111
5	35	34
6	72	71
7	38	39
8	18	19
9	0	6
10	3	12
11	0	14
12	10	15
H,K= 5, 5		
L=0	4	14
1	32	34
2	144	145
3	20	25
4	0	0
5	6	7
6	104	99
7	0	1
8	77	77
9	0	3
10	11	11
11	0	7
H,K= 6, 5		
L=0	142	140
1	26	26
2	71	67
3	20	21
4	83	83
5	12	13
6	83	84
7	0	14
8	13	9
9	18	10
10	26	25
H,K= 7, 5		
L=0	47	46
1	0	1
2	89	90
3	37	37
4	92	91
5	15	17
6	19	16
7	0	8
8	31	30
H,K= 8, 5		
L=0	48	49
1	9	16
2	80	80
3	0	6
4	0	0
5	16	15
6	38	38
H,K= 9, 5		
L=0	63	61
1	7	11
2	0	4
3	0	6
H,K= 0, 6		
L=0	0	17
1	114	115
2	242	239
3	57	60
4	172	171
5	124	125
6	34	28
7	0	3
8	13	11
9	52	51
10	92	92
11	35	33
12	0	3
13	0	7
H,K= 1, 6		
L=0	316	312
1	59	49
2	37	37
3	6	0
4	154	150
5	14	12
6	22	24
7	39	41
8	102	100
9	24	16
10	0	8
11	41	37
12	18	16
13	22	16
H,K= 2, 6		
L=0	90	81
1	108	100
2	211	214
3	37	37
4	185	182
5	132	134
6	148	144
7	68	66
8	68	67
9	16	14
10	84	81
11	37	34
12	31	25
13	0	8
H,K= 3, 6		
L=0	201	205
1	113	113
2	126	132
3	21	22
4	266	278
5	53	52
6	75	76
7	18	14
8	83	81
9	19	18
10	38	37
11	22	18
12	11	10
H,K= 4, 6		
L=0	69	66
1	71	70
2	284	293
3	44	43
4	16	19
5	41	43
6	61	59
7	58	59
8	69	69
9	13	14
10	35	32
11	21	17
12	0	13
H,K= 5, 6		
L=0	104	102
1	133	128
2	18	19
3	48	48
4	86	84
5	49	48
6	0	7
7	28	29
8	60	59
9	0	2
10	42	40
11	0	7
H,K= 6, 6		
L=0	28	25
1	33	31
2	151	145
3	72	73
4	41	39
5	22	23
6	26	27
7	24	27
8	64	65
9	21	20
H,K= 7, 6		
L=0	56	57
1	88	90
2	68	66
3	40	38
4	18	22
5	35	36
6	28	30
7	32	29
8	0	1
H,K= 8, 6		
L=0	68	70
1	14	15
2	16	20
3	32	34
4	34	37
5	28	28
6	23	17
H,K= 9, 6		
L=0	11	15
1	35	34
2	21	24
H,K= 0, 7		
L=1	230	229
2	134	137
3	514	562
4	91	94
5	82	83
6	30	33
7	83	87
8	57	56
9	92	94
10	0	9
11	27	25
12	0	3
13	13	15
H,K= 1, 7		
L=0	84	76
1	221	214
2	214	212
3	76	80
4	10	10
5	206	209
6	21	18
7	62	64
8	16	19
9	107	109
10	35	35
11	67	69
12	0	0
13	0	10
H,K= 2, 7		
L=0	27	22
1	61	66
2	111	112
3	345	369
4	132	132
5	69	70
6	0	7
7	80	78
8	109	104
9	115	113
10	48	45
11	0	7
12	0	4
13	22	27
H,K= 3, 7		
L=0	90	89
1	240	240
2	238	232
3	82	79
4	9	11
5	141	139
6	42	42
7	126	122
8	49	47
9	0	7
10	24	21
11	36	37
12	0	1
H,K= 4, 7		
L=0	94	94
1	133	128
2	47	47
3	132	128
4	109	112
5	60	62
6	12	14
7	24	23
8	54	51
9	79	81
10	28	26
11	0	5
H,K= 5, 7		
L=0	22	21
1	96	92
2	60	59
3	111	111
4	26	24
5	34	37
6	20	17
7	85	86
8	24	22
9	30	30
10	18	16
H,K= 6, 7		
L=0	18	17
1	73	71
2	42	41
3	54	53
4	62	62
5	61	58
6	11	15
7	0	11
8	17	16
9	46	47
H,K= 7, 7		
L=0	0	5
1	37	36
2	42	42
3	112	114
4	19	21
5	43	41
6	19	10
7	24	23
8	15	15
H,K= 8, 7		
L=0	0	5
1	47	45
2	30	28
3	0	13
4	15	11
5	9	11
H,K= 9, 7		
L=0	0	11
1	13	10
H,K= 0, 8		
L=0	72	64
1	317	332
2	22	16
3	59	59
4	168	171
5	165	160
6	44	47
7	51	51
8	49	50
9	97	94
10	73	73
11	24	22
12	23	19
13	7	10
H,K= 1, 8		
L=0	70	65
1	55	51
2	279	281
3	265	281
4	154	164
5	37	40
6	0	3
7	97	96
8	53	54
9	89	88
10	22	22
11	37	36
12	0	1
13	29	24
H,K= 2, 8		
L=0	102	88
1	157	153
2	5	2
3	22	27
4	101	101
5	155	162
6	34	36
7	95	95
8	0	3
9	49	44
10	27	30
11	32	34
12	0	10
H,K= 3, 8		
L=0	10	11
1	47	45
2	50	50
3	244	266
4	32	31
5	91	96
6	15	13
7	11	9
8	33	33
9	82	82
10	13	15
11	0	1
12	0	7
H,K= 4, 8		
L=0	23	18
1	126	126
2	28	27
3	144	153
4	20	22
5	6	2
6	33	35
7	53	56
8	32	34
9	0	3
10	0	1
11	19	19
H,K= 5, 8		
L=0	0	1
1	125	124
2	15	11
3	60	62
4	25	27
5	55	54
6	0	5
7	0	11
8	29	31
9	61	59
10	18	20
H,K= 6, 8		
L=0	19	18
1	45	45
2	75	73
3	126	122
4	40	40
5	23	16
6	0	3
7	56	53
8	18	22
9	38	37
H,K= 7, 8		
L=0	16	15
1	131	127
2	0	9
3	23	19
4	0	9
5	42	42
6	13	15
7	43	42
H,K= 8, 8		
L=0	12	14
1	37	34
2	36	37
3	50	51
4	18	17
5	23	25
H,K= 0, 9		
L=1	57	59
2	221	232
3	116	117
4	85	85
5	70	70
6	61	65
7	93	87
8	83	84
9	7	12
10	34	33
11	56	52
12	26	25
13	30	28
H,K= 1, 9		
L=0	94	89
1	212	220
2	113	109
3	43	44
4	172	179
5	85	81
6	36	38
7	46	44
8	13	9
9	36	35
10	67	64
11	0	16
12	0	1
H,K= 2, 9		
L=0	44	36
1	24	28
2	90	91
3	96	95
4	79	78
5	32	34
6	14	15
7	77	75
8	55	54
9	50	47
10	0	4
11	0	13
12	13	13
H,K= 3, 9		
L=0	35	16
1	157	162
2	139	139
3	67	71
4	32	34
5	54	54
6	23	25
7	58	60
8	47	45
9	0	5
10	24	22
11	18	18
12	0	2
H,K= 4, 9		
L=0	123	119
1	124	123
2	74	72
3	22	24
4	52	52
5	53	54
6	37	36
7	29	28
8	8	17
9	14	12
10	16	15
11	0	1
H,K= 5, 9		
L=0	61	59
1	36	33
2	117	115
3	26	16
4	45	43
5	17	18
6	0	13
7	32	30
8	58	59
9	0	3
10	0	2
H,K= 6, 9		
L=0	96	96
1	83	81
2	80	81
3	24	21
4	47	47
5	78	78
6	55	54
7	57	56
8	10	6
H,K= 7, 9		
L=0	60	58
1	18	16
2	69	71
3	46	47
4	56	58
5	23	26
6	9	11
7	0	8
H,K= 8, 9		
L=0	39	36
1	74	74
2	51	50
3	32	34
4	0	13
H,K= 0, 10		
L=0	384	404
1	168	171
2	219	246
3	28	33
4	204	222
5	69	75
6	154	155
7	32	32
8	10	13
9	0	8
10	62	65
11	0	11
12	0	2
H,K= 1, 10		
L=0	48	55
1	82	88
2	151	167
3	22	24
4	55	58
5	12	15
6	107	110
7	66	69
8	114	121
9	19	21
10	15	9
11	9	11
12	39	37
H,K= 2, 10		
L=0	176	182
1	31	32
2	154	155
3	38	37
4	153	153
5	49	48
6	136	132
7	0	5
8	40	42
H,K= 3, 10		
L=0	82	77
1	143	140
2	79	82
3	78	77
4	146	142
5	49	49
6	35	33
7	39	36
8	57	54
9	19	19
10	23	23
11	19	19
H,K= 4, 10		
L=0	17	15
1	26	27
2	157	154
3	24	14
4	0	11
5	17	16
6	53	51
7	13	13
8	44	43
9	11	13
10	36	38
H,K= 5, 10		
L=0	123	122
1	50	48
2	46	46
3	0	6
4	95	96
5	5	11
6	69	69
7	23	24
8	0	14
9	0	11
H,K= 6, 10		
L=0	37	37
1	0	3
2	85	86
3	16	14
4	32	30
5	0	11
6	31	32
7	11	13
8	46	47
H,K= 7, 10		
L=0	113	109
1	0	2
2	82	80
3	0	3
4	37	38
5	12	15
6	57	58
H,K= 8, 10		
L=0	57	58
1	0	8
2	0	9
3	0	11

Table 2 (*cont.*)

L	FO	FC
H,K= 0, 11		
L=1	16	15
2	260	268
3	99	101
4	147	146
5	67	65
6	83	81
7	39	42
8	99	99
9	43	42
10	0	5
11	28	27
12	31	30
H,K= 1, 11		
L=0	237	238
1	159	160
2	113	114
3	13	15
4	72	75
5	96	99
6	82	81
7	91	91
8	35	34
9	15	13
10	49	49
11	20	19
12	0	7
H,K= 2, 11		
L=0	30	30
1	60	57
2	196	197
3	139	136
4	127	124
5	23	22
6	48	51
7	0	8
8	108	108
9	54	53
10	46	46
11	0	7
H,K= 3, 11		
L=0	130	131
1	119	117
2	148	143
3	57	60
4	34	25
5	65	64
6	62	60
7	68	67
8	19	19
9	0	10
10	20	18
11	25	27
H,K= 4, 11		
L=0	105	106
1	59	57
2	22	18
3	105	103
4	103	101
5	46	44
6	40	40
7	0	5
8	42	42
9	46	45
10	38	38
H,K= 5, 11		
L=0	51	48
1	61	63
2	62	61
3	40	39
4	43	40
5	48	47
6	38	37
7	44	46
8	27	30
9	0	1
H,K= 6, 11		
L=0	71	70
1	56	57
2	0	5
3	42	40
4	79	79
5	34	33
6	62	61
7	24	20
H,K= 7, 11		
L=0	33	29
1	0	5
2	56	55
3	0	5
4	55	56
5	19	19
H,K= 8, 11		
L=0	38	39
1	22	19
2	40	40
H,K= 0, 12		
L=0	140	142
1	126	129
2	16	15
3	27	24
4	16	12
5	73	76
6	35	31
7	18	16
8	60	55
9	45	46
10	9	8
11	0	8
H,K= 1, 12		
L=0	48	47
1	20	20
2	32	31
3	145	147
4	12	9
5	1	11
6	48	48
7	71	77
8	10	15
9	60	68
10	0	3
11	0	2
H,K= 2, 12		
L=0	19	19
1	151	147
2	68	68
3	89	86
4	0	10
5	38	41
6	33	30
7	49	48
8	32	31
9	19	18
10	0	0
11	18	16
H,K= 3, 12		
L=0	21	19
1	26	26
2	31	32
3	161	162
4	42	42
5	100	95
6	20	19
7	24	24
8	0	1
9	71	70
H,K= 4, 12		
L=0	16	17
1	97	98
2	28	26
3	122	119
4	85	85
5	8	4
6	0	9
7	35	38
8	0	5
9	0	2
H,K= 5, 12		
L=0	50	48
1	58	55
2	39	40
3	84	82
4	1	4
5	79	81
6	0	13
7	0	7
8	0	8
H,K= 6, 12		
L=0	16	13
1	37	39
2	32	33
3	85	81
4	54	54
5	5	14
6	0	11
7	30	30
H,K= 7, 12		
L=0	56	57
1	69	67
2	37	38
3	0	5
4	0	0
5	29	30
H,K= 0, 13		
L=1	103	106
2	33	36
3	101	103
4	46	46
5	15	14
6	10	10
7	83	84
8	18	20
9	22	16
10	12	3
11	0	19
H,K= 1, 13		
L=0	57	59
1	124	128
2	57	58
3	30	29
4	19	15
5	69	68
6	34	35
7	21	22
8	0	14
9	33	32
10	10	0
11	22	27
H,K= 2, 13		
L=0	22	23
1	22	18
2	18	16
3	107	108
4	45	45
5	71	72
6	16	16
7	67	66
8	28	26
9	53	50
10	18	13
H,K= 3, 13		
L=0	63	60
1	160	160
2	0	66
3	107	102
4	34	33
5	38	39
6	49	47
7	63	62
8	13	6
9	0	2
10	12	9
H,K= 4, 13		
L=0	23	23
1	81	80
2	49	47
3	48	51
4	50	47
5	90	87
6	0	1
7	16	14
8	22	23
9	33	33
H,K= 5, 13		
L=0	0	14
1	61	57
2	44	45
3	68	67
4	23	19
5	14	12
6	24	25
7	52	49
8	0	4
H,K= 6, 13		
L=0	13	11
1	55	53
2	17	14
3	21	21
4	0	4
5	69	68
6	0	3
H,K= 7, 13		
L=0	29	29
1	13	15
2	0	0
3	52	52
H,K= 0, 14		
L=0	120	119
1	127	130
2	52	49
3	0	2
4	52	54
5	97	101
6	64	62
7	33	32
8	0	0
9	18	13
10	23	21
H,K= 1, 14		
L=0	32	32
1	0	5
2	82	83
3	31	33
4	75	73
5	47	47
6	30	28
7	48	47
8	64	68
9	0	7
10	19	20
H,K= 2, 14		
L=0	117	116
1	104	100
2	124	125
3	0	10
4	0	9
5	122	122
6	77	76
7	84	80
8	20	22
9	0	2
10	22	21
H,K= 3, 14		
L=0	84	83
1	25	24
2	56	54
3	60	60
4	99	94
5	96	96
6	0	8
7	0	12
8	50	48
9	14	12
H,K= 4, 14		
L=0	52	50
1	119	113
2	78	77
3	47	50
4	0	4
5	50	49
6	50	52
7	58	59
8	21	18
H,K= 5, 14		
L=0	58	55
1	39	39
2	24	24
3	13	9
4	65	65
5	48	45
6	26	28
7	22	16
H,K= 6, 14		
L=0	0	1
1	61	59
2	31	33
3	28	27
4	17	17
5	20	20
H,K= 7, 14		
L=0	32	30
1	31	29
2	24	22
H,K= 0, 15		
L=1	23	12
2	105	107
3	13	3
4	68	68
5	40	37
6	44	43
7	28	26
8	41	39
9	21	24
10	0	0
H,K= 1, 15		
L=0	164	156
1	56	53
2	61	63
3	0	5
4	52	52
5	29	27
6	120	117
7	31	32
8	0	0
9	0	10
10	36	35
H,K= 2, 15		
L=0	111	108
1	28	25
2	59	62
3	24	23
4	105	106
5	23	21
6	0	4
7	0	4
8	45	44
9	0	7
H,K= 3, 15		
L=0	138	136
1	19	18
2	103	106
3	28	25
4	12	13
5	0	0
6	112	112
7	0	11
8	29	27
H,K= 4, 15		
L=0	130	130
1	52	53
2	2	2
3	34	30
4	66	68
5	0	12
6	25	23
7	0	2
H,K= 5, 15		
L=0	48	47
1	6	5
2	55	56
3	14	12
4	0	6
5	6	3
6	64	61
H,K= 6, 15		
L=0	69	67
1	0	4
2	0	16
3	0	1
4	35	34
H,K= 0, 16		
L=0	62	64
1	52	51
2	14	20
3	23	25
4	32	32
5	61	59
6	25	27
7	10	11
8	48	46
9	17	20
H,K= 1, 16		
L=0	74	75
1	49	46
2	39	42
3	41	41
4	72	70
5	5	7
6	0	4
7	32	33
8	24	23
9	0	3
H,K= 2, 16		
L=0	41	43
1	69	67
2	26	27
3	8	3
4	25	20
5	39	37
6	74	70
7	0	18
8	0	1
H,K= 3, 16		
L=0	101	101
1	10	15
2	26	28
3	9	3
4	83	82
5	13	9
6	24	23
7	0	8
8	18	21
H,K= 4, 16		
L=0	38	38
1	18	17
2	71	70
3	0	0
4	36	35
5	19	20
6	34	35
7	0	20
H,K= 5, 16		
L=0	62	59
1	31	30
2	15	12
3	0	3
4	22	21
5	32	31
H,K= 6, 16		
L=0	0	8
1	0	3
2	50	47
H,K= 0, 17		
L=1	96	96
2	72	68
3	99	102
4	64	60
5	37	34
6	12	1
7	58	56
8	22	20
H,K= 1, 17		
L=0	37	39
1	58	58
2	34	35
3	33	34
4	15	14
5	73	76
6	32	31
7	0	6
8	0	1
H,K= 2, 17		
L=0	25	24
1	41	37
2	49	50
3	74	74
4	59	58
5	48	50
6	14	6
7	36	35
8	40	37
H,K= 3, 17		
L=0	65	64
1	63	60
2	61	59
3	20	19
4	0	10
5	31	29
6	45	45
7	20	22
H,K= 4, 17		
L=0	47	44
1	20	17
2	19	15
3	0	12
4	35	32
5	31	32
H,K= 5, 17		
L=0	0	12
1	41	40
2	0	14
3	48	48
4	0	9
H,K= 0, 18		
L=0	11	5
1	103	100
2	35	33
3	26	29
4	0	18
5	89	92
6	14	13
7	13	17
H,K= 1, 18		
L=0	15	16
1	57	58
2	63	59
3	99	98
4	48	42
5	29	26
6	0	1
7	59	57
H,K= 2, 18		
L=0	26	26
1	89	87
2	0	9
3	0	9
4	0	4
5	83	81
6	0	6
H,K= 3, 18		
L=0	0	7
1	0	6
2	0	4
3	61	59
4	0	1
5	43	42
6	0	6
H,K= 4, 18		
L=0	0	9
1	59	55
2	0	4
3	33	30
4	0	4
H,K= 5, 18		
L=0	0	11
1	25	24
H,K= 0, 19		
L=1	23	23
2	66	64
3	38	37
4	38	38
5	18	21
6	26	28
H,K= 1, 19		
L=0	74	73
1	59	59
2	0	2
3	0	2
4	39	39
5	34	36
6	56	51
H,K= 2, 19		
L=0	38	37
1	0	1
2	29	29
3	29	27
4	36	37
5	0	5
H,K= 3, 19		
L=0	39	35
1	43	41
2	27	19
3	18	19
4	0	1
H,K= 4, 19		
L=0	45	43
1	44	43
2	0	4
H,K= 0, 20		
L=0	78	75
1	43	45
2	20	18
3	0	0
4	48	46
5	20	17
H,K= 1, 20		
L=0	16	15
1	0	5
2	38	37
3	31	28
4	22	20
H,K= 2, 20		
L=0	53	51
1	0	8
2	35	36
3	7	9
H,K= 3, 20		
L=0	50	49
1	23	23
2	0	4
H,K= 0, 21		
L=1	0	7
2	68	68
H,K= 1, 21		
L=0	58	59
1	19	18
2	39	40
H,K= −1, 0		
L=2	582	629
4	250	248
6	337	356
8	190	191
10	0	1
12	107	105
14	21	19
H,K= −2, 0		
L=2	5	8
4	198	202
6	287	300
8	60	58
10	58	56
12	18	20
14	10	13
H,K= −3, 0		
L=2	181	181
4	150	151
6	153	155
8	0	5
10	44	47
12	78	77
14	0	4
H,K= −4, 0		
L=2	226	234
4	150	150
6	87	86
8	59	60
10	16	8
12	0	5
H,K= −5, 0		
L=2	0	0
4	96	96
6	132	131
8	57	55
10	33	31
12	18	17
H,K= −6, 0		
L=2	129	131
4	29	31
6	108	108
8	72	74
10	21	22
12	29	27
H,K= −7, 0		
L=2	33	33
4	45	44
6	46	43
8	55	56
10	0	3
H,K= −8, 0		
L=2	83	86
4	39	40
6	89	86
8	30	30
H,K= −9, 0		
L=2	40	39
4	25	30
6	0	3
H,K= −1, 1		
L=1	473	532
2	241	242
3	115	114
4	336	334
5	335	350
6	217	220
7	99	99
8	105	106
9	23	21
10	75	71
11	38	36
12	0	5
13	0	11
14	36	33
H,K= −2, 1		
L=1	226	225
2	20	23
3	135	124
4	8	4
5	82	83
6	210	207
7	175	177
8	53	53
9	35	30
10	52	48
11	60	58
12	66	65
13	42	42
14	0	5
H,K= −3, 1		
L=1	148	134
2	72	76
3	56	57
4	72	71
5	170	173
6	28	26
7	13	14
8	109	114
9	0	1
10	36	38
11	22	23
12	23	22
13	0	14
14	26	25
H,K= −4, 1		
L=1	258	265
2	64	62
3	5	10
4	56	56
5	51	47
6	113	110
7	76	76
8	39	40
9	43	44
10	53	53
11	35	33
12	26	28
13	0	7
H,K= −5, 1		
L=1	95	97
2	259	264
3	76	77
4	57	55
5	44	44
6	99	99
7	84	83
8	114	112
9	16	15
10	0	6
11	0	10
12	28	28
H,K= −6, 1		
L=1	124	124
2	41	42
3	15	3
4	119	118
5	20	21
6	50	50
7	0	7
8	89	88
9	28	30
10	56	52
11	0	4
H,K= −7, 1		
L=1	65	66
2	153	154
3	0	6
4	21	20
5	21	24
6	85	84
7	63	62
8	57	54
9	12	8
10	17	20
H,K= −8, 1		
L=1	38	38
2	55	53
3	30	29
4	94	94
5	24	21
6	38	37
7	0	0
8	47	46
H,K= −9, 1		
L=1	18	23
2	37	39
3	37	33
4	29	27
5	41	39
6	53	54
H,K= −1, 2		
L=1	558	562
2	570	583
3	33	27
4	301	293
5	67	67
6	0	4
7	232	234
8	103	102
9	10	10
10	14	16
11	51	53
12	0	6
13	35	34
14	0	0
H,K= −2, 2		
L=1	273	263
2	171	158
3	311	304
4	103	95
5	193	190
6	20	13
7	161	165
8	65	64
9	109	114
10	72	70
11	31	32
12	9	3
13	56	54
14	15	18
H,K= −3, 2		
L=1	317	317
2	127	120
3	36	35
4	90	88
5	96	94
6	31	33
7	94	94
8	46	45
9	113	113
10	0	5
11	83	83
12	14	12
13	14	12
14	0	0
H,K= −4, 2		
L=1	250	260
2	5	7
3	312	321
4	41	40
5	155	154
6	27	25
7	162	162
8	0	11
9	84	81
10	0	5
11	0	8
12	0	15
13	46	46
H,K= −5, 2		
L=1	153	153
2	43	44
3	141	140
4	100	99
5	164	161
6	12	13
7	40	39
8	9	4
9	104	103
10	18	19
11	54	53
12	13	9
H,K= −6, 2		
L=1	165	165
2	40	39
3	116	115
4	17	11
5	24	24
6	41	42
7	158	159
8	0	6
9	38	39
10	19	18
11	16	14
H,K= −7, 2		
L=1	102	104
2	19	22
3	77	79
4	69	68
5	79	76
6	0	0
7	14	15
8	27	28
9	58	61
10	31	29
H,K= −8, 2		
L=1	134	132
2	51	51
3	43	45
4	25	25
5	43	45
6	0	13
7	85	84
8	0	5
H,K= −9, 2		
L=1	0	9
2	19	21
3	74	74
4	31	26
5	36	34
6	0	10
H,K= −1, 3		
L=1	116	127
2	483	481
3	382	371
4	102	92
5	331	339
6	18	17
7	10	13
8	76	77
9	126	123
10	29	25
11	64	59
12	0	12
13	20	19
14	17	18
H,K= −2, 3		
L=1	279	274
2	125	116
3	177	177
4	100	95
5	116	113
6	106	109
7	182	182
8	76	76
9	29	29
10	35	30
11	45	42
12	36	35
13	15	15
14	18	16
H,K= −3, 3		
L=1	179	172
2	384	380
3	156	149
4	117	117
5	114	114
6	17	21
7	93	91
8	114	114
9	99	96
10	19	16
11	0	5
12	0	5
13	35	36
H,K= −4, 3		
L=1	363	351
2	27	30
3	111	106
4	2	2
5	136	134
6	83	83
7	105	104
8	73	71
9	24	22
10	36	37
11	33	34
12	29	29
13	0	5
H,K= −5, 3		
L=1	108	110
2	187	182
3	189	188
4	87	87
5	32	30
6	14	3
7	106	105
8	90	92
9	51	51
10	35	35
11	22	19
12	7	0
H,K= −6, 3		
L=1	75	74
2	11	6
3	58	57
4	27	23
5	91	91
6	29	30
7	0	4
8	42	42
9	62	61
10	0	8
11	24	24
H,K= −7, 3		
L=1	81	80
2	62	62
3	86	86
4	0	6
5	11	4
6	18	19
7	40	39
8	34	39
9	0	9
10	0	13
H,K= −8, 3		
L=1	0	11
2	29	27
3	82	84
4	0	8
5	49	48
6	10	16
7	29	27
8	18	19
H,K= −9, 3		
L=1	41	39
2	0	12
3	0	7
4	0	17
5	44	44
H,K= −1, 4		
L=1	279	266
2	343	345
3	302	288
4	145	134
5	163	156
6	142	146
7	83	79
8	87	88
9	39	39
10	22	19
11	50	47
12	39	38
13	0	5
14	0	2
H,K= −2, 4		
L=1	83	83
2	275	259
3	118	110
4	172	173
5	24	29
6	52	56
7	61	60
8	121	118
9	105	102
10	53	51
11	0	8
12	0	0
13	0	13
14	20	18
H,K= −3, 4		
L=1	167	176
2	258	266
3	123	122
4	24	27
5	183	182
6	169	171
7	82	81
8	19	16
9	12	5
10	31	33
11	45	46
12	37	36
13	10	3
H,K= −4, 4		
L=1	45	44
2	201	207
3	138	141
4	194	199
5	10	7
6	34	35
7	35	35
8	95	96
9	40	39
10	24	23
11	54	53
12	19	20
13	23	25
H,K= −5, 4		
L=1	83	78
2	85	83
3	64	65
4	47	47
5	207	205
6	130	130
7	78	75
8	0	6
9	17	15
10	28	29
11	40	36
12	17	19
H,K= −6, 4		
L=1	15	17
2	67	69
3	38	40
4	38	38
5	59	61
6	24	22
7	30	33
8	44	45
9	4	4
10	0	2
11	51	50
H,K= −7, 4		
L=1	44	43
2	0	3
3	35	35
4	22	21
5	70	72
6	22	22
7	0	1
8	32	32
9	29	29
10	0	6
H,K= −8, 4		
L=1	61	60
2	47	46
3	45	45
4	17	16
5	34	33
6	33	31
7	16	13
8	0	2
H,K= −9, 4		
L=1	15	21
2	44	45
3	29	32
4	13	18
5	0	7

Table 2 (*cont.*)

	FO	FC
H,K= -1, 5		
L=1	180	176
2	187	189
3	93	95
4	274	283
5	49	63
6	27	26
7	10	11
8	82	82
9	46	45
10	77	76
11	22	20
12	18	11
13	0	4
14	20	20
H,K= -2, 5		
L=1	127	120
2	182	175
3	15	12
4	238	245
5	125	120
6	261	267
7	37	42
8	69	72
9	0	0
10	85	85
11	17	15
12	43	43
13	14	12
H,K= -3, 5		
L=1	13	12
2	71	68
3	47	44
4	161	162
5	74	79
6	106	107
7	21	18
8	144	145
9	39	41
10	23	26
11	32	31
12	66	64
13	12	1
H,K= -4, 5		
L=1	15	3
2	242	253
3	28	28
4	191	191
5	76	76
6	249	251
7	10	7
8	54	53
9	0	0
10	86	85
11	14	11
12	36	33
13	0	4
H,K= -5, 5		
L=1	14	12
2	143	142
3	63	61
4	101	105
5	35	37
6	101	100
7	0	3
8	104	104
9	31	29
10	20	5
11	22	23
12	62	59
H,K= -6, 5		
L=1	35	30
2	20	20
3	6	11
4	157	157
5	39	40
6	103	106
7	0	5
8	17	18
9	16	10
10	71	71
11	0	12
H,K= -7, 5		
L=1	0	7
2	104	102
3	35	35
4	29	28
5	C	8
6	55	57
7	14	14
8	26	24
9	3	11
10	25	29
H,K= -8, 5		
L=1	17	15
2	57	54
3	0	12
4	103	101
5	18	18
6	19	19
7	0	0
8	33	35
H,K= -9, 5		
L=1	0	1
2	43	41
3	0	10
4	32	31
5	8	10
H,K= -1, 6		
L=1	217	204
2	393	391
3	61	60

	FO	FC
H,K= -1, 6		
4	43	40
5	66	58
6	76	70
7	91	93
8	107	106
9	0	12
10	22	21
11	41	42
12	0	3
13	0	12
H,K= -2, 6		
L=1	57	53
2	101	91
3	40	41
4	245	247
5	40	43
6	93	98
7	66	67
8	126	130
9	55	55
10	86	87
11	0	4
12	28	24
13	29	28
H,K= -3, 6		
L=1	181	181
2	296	305
3	0	1
4	75	70
5	129	128
6	107	104
7	98	102
8	48	48
9	13	16
10	73	72
11	41	41
12	0	5
13	18	9
H,K= -4, 6		
L=1	25	23
2	134	129
3	47	47
4	244	247
5	0	3
6	36	38
7	73	73
8	87	88
9	20	20
10	32	28
11	28	27
12	37	41
H,K= -5, 6		
L=1	89	89
2	74	74
3	87	85
4	160	162
5	136	138
6	106	110
7	54	56
8	26	26
9	32	32
10	67	66
11	33	35
12	0	4
H,K= -6, 6		
L=1	63	62
2	41	38
3	40	37
4	53	54
5	35	34
6	53	54
7	60	60
8	35	37
9	0	2
10	0	9
11	33	31
H,K= -7, 6		
L=1	34	36
2	0	7
3	74	75
4	79	79
5	76	79
6	0	4
7	8	3
8	21	19
9	39	37
H,K= -8, 6		
L=1	71	70
2	59	60
3	32	31
4	0	6
5	30	28
6	30	34
7	40	39
H,K= -9, 6		
L=1	0	5
2	45	45
3	34	35
4	44	44
H,K= -1, 7		
L=1	156	157
2	85	76
3	229	229
4	196	187
5	230	231
6	0	5
7	20	18
8	0	1
9	146	146
10	33	29
11	53	52

	FO	FC
H,K= -1, 7		
12	10	6
13	0	6
H,K= -2, 7		
L=1	185	175
2	52	53
3	298	307
4	97	96
5	77	74
6	45	43
7	119	121
8	30	27
9	63	63
10	40	35
11	29	28
12	0	0
13	0	3
H,K= -3, 7		
L=1	145	148
2	30	30
3	137	132
4	150	149
5	68	69
6	22	26
7	46	47
8	42	42
9	104	106
10	28	27
11	0	1
12	8	10
13	24	21
H,K= -4, 7		
L=1	182	181
2	155	157
3	101	105
4	11	12
5	117	119
6	25	22
7	70	68
8	20	16
9	0	4
10	38	35
11	24	24
12	0	7
H,K= -5, 7		
L=1	33	33
2	69	71
3	131	136
4	100	102
5	0	2
6	14	17
7	49	51
8	46	44
9	42	42
10	27	25
11	31	32
12	0	1
H,K= -6, 7		
L=1	26	26
2	37	34
3	55	55
4	0	2
5	88	92
6	21	22
7	0	11
8	0	3
9	47	46
10	19	19
H,K= -7, 7		
L=1	60	60
2	0	11
3	83	83
4	14	18
5	19	19
6	12	4
7	21	17
8	0	0
9	0	6
H,K= -8, 7		
L=1	27	25
2	0	15
3	76	76
4	23	22
5	44	44
6	14	14
7	35	33
H,K= -9, 7		
L=1	32	33
2	0	10
3	16	13
H,K= -1, 8		
L=1	200	200
2	255	266
3	332	335
4	228	234
5	106	105
6	32	30
7	130	128
8	66	69
9	66	63
10	38	36
11	52	51
12	7	10
13	17	17
H,K= -2, 8		
L=1	76	77
2	101	102
3	214	214
4	52	56
5	106	104
6	37	37

	FO	FC
H,K= -2, 8		
7	39	39
8	23	22
9	133	131
10	52	53
11	0	7
12	13	11
13	16	17
H,K= -3, 8		
L=1	132	134
2	94	97
3	152	155
4	90	92
5	110	114
6	13	11
7	49	52
8	58	59
9	21	18
10	39	39
11	59	57
12	17	16
H,K= -4, 8		
L=1	55	52
2	40	41
3	234	234
4	5	9
5	46	46
6	0	6
7	50	52
8	13	3
9	80	80
10	6	6
11	25	25
12	5	2
H,K= -5, 8		
L=1	55	54
2	0	4
3	52	51
4	11	10
5	143	147
6	8	4
7	19	19
8	0	5
9	48	46
10	0	4
11	31	33
H,K= -6, 8		
L=1	65	65
2	23	23
3	97	100
4	30	30
5	63	63
6	0	7
7	69	70
8	14	14
9	33	33
10	12	12
H,K= -7, 8		
L=1	45	44
2	28	29
3	53	53
4	39	40
5	66	68
6	0	2
7	21	23
8	15	17
9	41	44
H,K= -8, 8		
L=1	84	84
2	0	12
3	53	55
4	18	20
5	55	55
6	7	7
H,K= -1, 9		
L=1	140	145
2	71	68
3	126	124
4	215	219
5	185	181
6	27	27
7	71	70
8	46	48
9	35	29
10	72	73
11	0	0
12	13	15
13	0	8
H,K= -2, 9		
L=1	0	0
2	163	165
3	41	31
4	14	9
5	161	164
6	82	85
7	93	92
8	77	78
9	25	25
10	43	45
11	71	69
12	23	19
13	27	27
H,K= -3, 9		
L=1	84	91
2	0	7
3	73	72
4	114	116
5	56	55
6	47	46
7	10	9
8	62	62
9	34	35

	FO	FC
H,K= -3, 9		
10	37	38
11	13	12
12	30	28
H,K= -4, 9		
L=1	119	118
2	67	67
3	13	11
4	108	108
5	91	92
6	63	65
7	30	29
8	19	19
9	40	39
10	54	54
11	36	38
12	0	4
H,K= -5, 9		
L=1	100	102
2	105	102
3	93	94
4	55	55
5	30	30
6	88	89
7	70	68
8	58	56
9	36	36
10	0	1
11	16	15
H,K= -6, 9		
L=1	50	48
2	0	2
3	30	32
4	126	127
5	48	49
6	37	38
7	16	16
8	25	24
9	38	39
10	54	53
H,K= -7, 9		
L=1	70	70
2	96	94
3	37	37
4	30	28
5	53	54
6	63	65
7	48	50
8	36	35
H,K= -8, 9		
L=1	19	20
2	21	19
3	45	45
4	77	78
5	45	42
6	9	13
H,K= -1, 10		
L=1	104	108
2	246	252
3	56	55
4	28	24
5	37	37
6	160	156
7	68	63
8	131	127
9	22	20
10	43	43
11	0	10
12	53	50
H,K= -2, 10		
L=1	39	44
2	68	72
3	0	6
4	214	213
5	77	74
6	87	87
7	25	24
8	24	28
9	0	6
10	57	56
11	23	22
12	0	5
H,K= -3, 10		
L=1	0	2
2	147	144
3	31	31
4	45	42
5	78	78
6	86	85
7	0	9
8	38	38
9	0	9
10	69	68
11	0	5
12	32	33
H,K= -4, 10		
L=1	0	5
2	95	90
3	12	12
4	137	135
5	64	63
6	18	13
7	26	25
8	42	44
9	13	16
10	0	14
11	19	20
H,K= -5, 10		
L=1	11	12
2	43	41
3	0	12

	FO	FC
H,K= -5, 10		
4	121	122
5	38	36
6	61	60
7	14	14
8	11	11
9	0	4
10	58	53
H,K= -6, 10		
L=1	0	0
2	82	83
3	30	30
4	22	14
5	0	5
6	92	90
7	0	6
8	49	51
9	17	12
H,K= -7, 10		
L=1	19	19
2	29	28
3	0	11
4	74	76
5	28	28
6	18	19
7	0	14
8	19	22
H,K= -8, 10		
L=1	0	14
2	54	53
3	11	10
4	16	15
5	0	4
H,K= -1, 11		
L=1	138	142
2	7	0
3	39	37
4	219	213
5	80	82
6	147	145
7	65	64
8	33	32
9	21	25
10	51	50
11	0	4
12	0	3
H,K= -2, 11		
L=1	67	64
2	102	106
3	93	92
4	18	19
5	89	87
6	101	97
7	41	42
8	52	52
9	14	14
10	28	27
11	37	34
12	36	35
H,K= -3, 11		
L=1	45	49
2	46	45
3	109	108
4	117	120
5	53	57
6	44	43
7	22	19
8	48	49
9	38	38
10	27	30
11	6	5
H,K= -4, 11		
L=1	68	69
2	75	79
3	46	45
4	21	18
5	75	80
6	37	37
7	26	28
8	14	18
9	0	6
10	34	33
11	26	25
H,K= -5, 11		
L=1	26	25
2	146	146
3	71	69
4	66	66
5	7	7
6	50	44
7	0	7
8	63	60
9	30	29
10	0	9
H,K= -6, 11		
L=1	60	60
2	0	5
3	33	30
4	52	50
5	37	39
6	12	7
7	0	14
8	50	48
9	7	3
H,K= -7, 11		
L=1	0	3
2	80	81
3	25	23
4	12	15
5	23	22
6	46	45

	FO	FC
H,K= -7, 11		
7	0	10
H,K= -8, 11		
L=1	0	8
2	27	22
3	0	1
4	49	48
H,K= -1, 12		
L=1	94	96
2	91	93
3	100	98
4	45	43
5	69	69
6	67	67
7	78	78
8	24	24
9	0	13
10	0	0
11	37	35
12	22	18
H,K= -2, 12		
L=1	0	6
2	74	75
3	181	181
4	10	10
5	98	99
6	0	3
7	34	31
8	74	72
9	73	73
10	15	16
11	9	4
H,K= -3, 12		
L=1	118	119
2	0	2
3	40	41
4	14	9
5	94	94
6	29	28
7	34	36
8	24	24
9	48	44
10	12	12
11	54	49
H,K= -4, 12		
L=1	89	86
2	109	109
3	203	204
4	19	19
5	61	61
6	14	14
7	64	64
8	40	39
9	71	70
10	10	4
H,K= -5, 12		
L=1	83	82
2	55	56
3	0	9
4	66	66
5	80	80
6	44	41
7	12	8
8	36	36
9	50	49
H,K= -6, 12		
L=1	70	68
2	65	66
3	103	102
4	11	11
5	5	7
6	47	45
7	61	62
8	33	32
H,K= -7, 12		
L=1	43	40
2	16	14
3	23	24
4	29	32
5	39	41
6	20	18
H,K= -8, 12		
L=1	44	41
H,K= -1, 13		
L=1	33	31
2	15	10
3	114	115
4	18	19
5	114	113
6	53	52
7	15	19
8	0	8
9	48	48
10	0	3
11	11	13
H,K= -2, 13		
L=1	137	136
2	23	20
3	10	10
4	38	39
5	97	94
6	0	2
7	94	93
8	0	10
9	24	21
10	29	33
11	39	38
H,K= -3, 13		
L=1	17	15

	FO	FC
H,K= -3, 13		
2	97	97
3	130	133
4	106	105
5	63	62
6	22	18
7	75	76
8	32	34
9	53	53
10	25	23
H,K= -4, 13		
L=1	160	161
2	0	13
3	0	3
4	26	30
5	93	96
6	0	2
7	53	52
8	7	12
9	44	43
10	38	39
H,K= -5, 13		
L=1	86	87
2	41	40
3	114	116
4	59	60
5	13	14
6	0	9
7	86	84
8	33	32
9	39	38
H,K= -6, 13		
L=1	65	66
2	24	23
3	32	34
4	0	9
5	54	57
6	0	3
7	0	4
H,K= -7, 13		
L=1	62	64
2	17	19
3	47	48
4	0	7
5	0	11
H,K= -1, 14		
L=1	169	166
2	84	84
3	63	65
4	8	3
5	0	5
6	64	63
7	81	80
8	42	42
9	0	9
10	0	10
H,K= -2, 14		
L=1	40	40
2	41	39
3	38	37
4	106	107
5	55	56
6	38	38
7	39	34
8	30	30
9	24	24
10	17	17
H,K= -3, 14		
L=1	156	158
2	91	94
3	26	27
4	22	27
5	48	46
6	73	75
7	66	66
8	14	17
9	0	8
10	31	30
H,K= -4, 14		
L=2	69	67
3	50	51
4	87	87
5	46	44
6	0	1
7	33	32
8	46	47
9	0	0
H,K= -5, 14		
L=1	80	82
2	53	54
3	28	29
4	17	16
5	70	70
6	39	36
7	46	47
8	0	2
H,K= -6, 14		
L=1	0	6
2	44	45
3	0	1
4	28	27
5	0	1
6	20	17
H,K= -7, 14		
L=1	31	37
2	0	0
3	11	11
H,K= -1, 15		
L=1	90	89

	FO	FC
H,K= -1, 15		
2	23	22
3	42	43
4	87	88
5	0	12
6	73	72
7	45	47
8	34	35
9	16	15
10	28	26
H,K= -2, 15		
L=1	23	24
2	115	117
3	44	44
4	14	13
5	26	27
6	109	108
7	0	4
8	32	32
9	18	19
10	16	16
H,K= -3, 15		
L=1	25	27
2	51	52
3	0	7
4	78	76
5	28	28
6	9	12
7	53	53
8	56	57
9	0	9
H,K= -4, 15		
L=1	53	55
2	87	85
3	19	19
4	38	40
5	35	35
6	106	101
7	16	16
8	12	11
H,K= -5, 15		
L=1	31	33
2	53	53
3	24	22
4	35	36
5	0	1
6	12	14
7	28	29
H,K= -6, 15		
L=1	0	2
2	12	12
3	26	25
4	35	36
5	19	21
H,K= -1 16		
L=1	43	42
2	102	106
3	27	28
4	33	34
5	0	9
6	51	49
7	39	39
8	0	15
9	0	2
H,K= -2, 16		
L=1	50	50
2	94	92
3	19	15
4	59	57
5	47	47
6	17	22
7	0	5
8	87	83
9	0	11
H,K= -3, 16		
L=1	51	49
2	76	80
3	9	1
4	22	20
5	28	31
6	73	76
7	42	39
8	0	2
H,K= -4, 16		
L=1	0	0
2	91	90
3	13	7
4	61	60
5	0	4
6	17	19
7	13	18
H,K= -5, 16		
L=1	33	33
2	0	0
3	13	15
4	60	59
5	23	23
6	64	66
H,K= -6, 16		
L=1	37	35
2	46	47
3	21	21
H,K= -1, 17		
L=1	39	38
2	21	23
3	48	50
4	53	53
5	71	69
6	29	24

	FO	FC
H,K= -1, 17		
7	27	25
8	0	10
H,K= -2, 17		
L=1	108	109
2	52	48
3	70	70
4	0	4
5	0	0
6	32	32
7	58	57
8	13	15
H,K= -3, 17		
L=1	0	2
2	0	7
3	46	50
4	33	32
5	36	37
6	0	1
7	40	42
H,K= -4, 17		
L=1	63	61
2	54	53
3	14	9
4	13	6
5	36	36
6	26	24
H,K= -5, 17		
L=1	30	33
2	9	16
3	36	36
4	20	19
5	0	4
H,K= -1, 18		
L=1	105	106
2	40	39
3	108	106
4	52	52
5	0	7
6	0	6
7	66	65
H,K= -2, 18		
L=1	56	55
2	0	1
3	40	41
4	0	1
5	63	64
6	16	17
7	0	11
H,K= -3, 18		
L=1	84	87
2	8	12
3	51	49
4	21	22
5	15	18
6	0	6
H,K= -4, 18		
L=1	0	1
2	0	4
3	39	38
4	0	11
5	20	23
H,K= -5, 18		
L=1	40	37
2	0	0
H,K= -1, 19		
L=1	41	41
2	0	6
3	35	35
4	29	29
5	49	51
6	29	30
H,K= -2, 19		
L=1	20	20
2	63	65
3	0	4
4	24	29
5	50	47
6	34	34
H,K= -3, 19		
L=1	6	13
2	20	20
3	28	27
4	20	19
5	19	13
H,K= -4, 19		
L=1	34	33
2	10	15
3	0	8
H,K= -1, 20		
L=1	0	5
2	50	49
3	32	33
4	0	4
H,K= -2, 20		
L=1	0	14
2	13	12
3	16	14
4	46	47
H,K= -3, 20		
L=1	20	18
2	30	30
H,K= -1, 21		
L=1	0	9

tively strong N–H---O hydrogen bonds, have equivalent S–O distances, whereas O(2), which is only loosely associated with an amino group, exhibits a smaller separation of 1·448 Å. The C(1)–S distance of 1·780 Å is a normal one for such an aliphatic molecule.

The average O–S–O and C–S–O angles in the sulfonate group are 112·9 and 106·7°, respectively. These deviations from the tetrahedral angle of 109·5° are commonly found in aliphatic as well as aromatic sulfonates and also in other ions with $-X-SO_3^-$ groups, X replacing the C. Other bond distances and angles in the molecule are quite normal; as is usual, the average C–H and N–H distances of 0·95 and 0·85 Å, respectively, are much shorter than those found by the neutron diffraction method.

In the crystal structure, the molecules are held together by a three-dimensional network of N–H---O

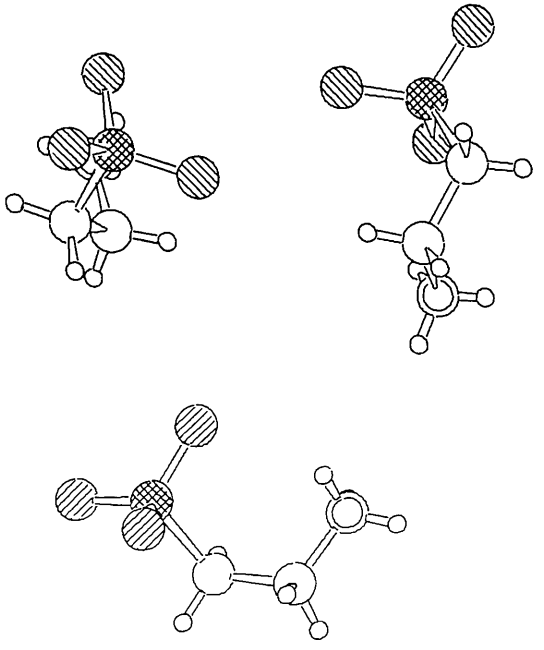

Fig. 4. Several views of the taurine molecule showing the conformation around the central methylene linkage. Open circles, carbon; double circles, nitrogen; shaded circles, oxygen; double-shaded circles, sulfur.

Table 3. *Taurine*: *bond angles*

Around S			
O(1)–S–O(2)	113·7°	O(1)–S–C(1)	105·8°
O(1)–S–O(3)	110·9	O(2)–S–C(1)	106·9
O(2)–S–O(3)	113·0	O(3)–S–C(1)	105·8
Around C(1)			
S–C(1)–C(2)	112·9	H(1)–C(1)–C(2)	112
S–C(1)–H(1)	105	H(2)–C(1)–C(2)	113
S–C(1)–H(2)	104	H(1)–C(1)–H(2)	109
Around C(2)			
C(1)–C(2)–N	112·6	H(3)–C(2)–N	111
C(1)–C(2)–H(3)	107	H(4)–C(2)–N	111
C(1)–C(2)–H(4)	109	H(3)–C(3)–H(4)	105
Around N			
C(2)–N–H(5)	111	H(5)–N–H(6)	110
C(2)–N–H(6)	111	H(5)–N–H(7)	117
C(2)–N–H(7)	104	H(6)–N–H(7)	104

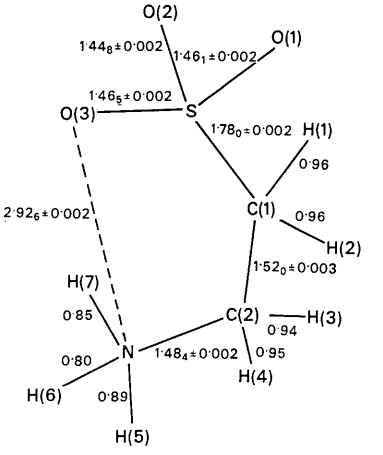

Fig. 5. Bond distances in the taurine molecule. The intramolecular N–O separation is shown by a dotted line.

Table 4

(*a*) Short N–O contacts.

No.	Atom	From N of the original coordinates to Equivalent position	Distance	Hydrogen atom involved	N–H–O angle	O–H separations
I	O(1)	$(1-x, -\frac{1}{2}+y, \frac{1}{2}-z)$	$2{\cdot}79_3$ Å	H(5)	166°	$1{\cdot}9_2$
II	O(3)	$(-x, -y, -z)$	$2{\cdot}89_3$	H(6)	162	$2{\cdot}1_2$
III	O(2)	$(-x, -\frac{1}{2}+y, \frac{1}{2}-z)$	$2{\cdot}93_9$	H(6)	110	$2{\cdot}5_6$
IV	O(3)	$(1-x, -y, -z)$	$3{\cdot}01_6$	H(7)	132	$2{\cdot}3_8$
V	O(3)	intramolecular	$2{\cdot}92_6$	H(7)	133	$2{\cdot}2_9$

(*b*) Configuration around N

Angles between		and	
	C–N	I	100·1°
	C–N	II	111·5
	C–N	IV	96·8
	C–N	V	82·5
	I	II	127·2
	I	IV	87·9
	I	V	153·1
	II	IV	127·6
	II	V	74·5

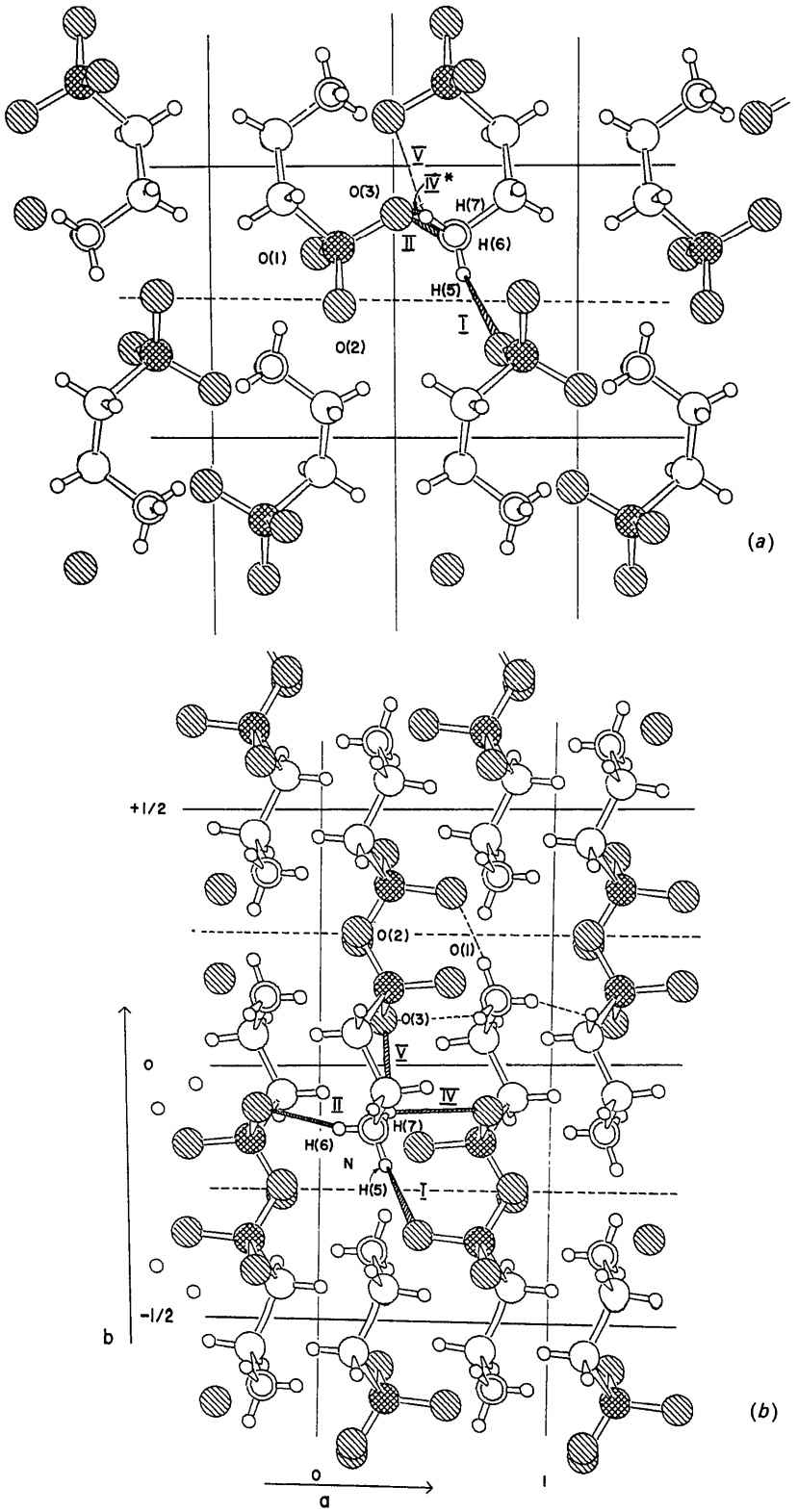

Fig. 6. (*a*) View of the structure projected down the *a* axis. The N–O contacts discussed in the text (and Table 4) are shaded. H(6) is almost under the nitrogen atom. Bond IV* goes to O(3), which is one unit cell above along the *a* axis. (*b*) Projection of the structure along the *c* axis. These two drawings and Fig. 4 were drawn on an IBM 1627 machine based on calculations done by a structure drawing program on an IBM 7094 (Okaya, 1966). Except for adding the necessary information for the hydrogen-bond system and unit-cell outlines, these drawings have not been retouched.

hydrogen bonds. The assignment of hydrogen bonds to observed short N–O contacts can be made with the help of the hydrogen positions. In Table 4(*a*), five N–O contacts which are within hydrogen bond distances are listed together with the angles around the hydrogen atoms. The contact III with a relatively sharp N–H–O angle can be eliminated as a hydrogen bond; therefore contacts I and II are considered as the hydrogen bonds formed by H(5) and H(6), respectively. For H(7), there are two choices, IV and V; although the configuration around the nitrogen atoms [Table 4 (*b*)] seems to favor contact IV as the hydrogen bond formed by H(7), the evidence is not strong enough to preclude the fifth (intramolecular) contact as a possible one. It is also possible to consider that H(7) is actually involved in a bifurcated hydrogen bond system with these two contacts. In either case, O(3) is involved in two hydrogen bonds; this is rather interesting because the S–O(1) and S–O(3) bond lengths are almost the same, although O(1) is involved in only one hydrogen bond. A similar situation was found in the structure of D-tartaric acid, where the two carboxyl groups in the molecule exhibit almost equal C=O distances, although one of these two double bonded oxygen atoms receives two hydrogen bonds whereas the other receives only one (Okaya, Stemple & Kay, 1966). As discussed before, O(2) with a short S–O bond is not involved in hydrogen bond formation. Fig. 6 shows drawings of the structure viewed down the *a* and *c* axes. The hydrogen bond system is illustrated in the drawings by shaded lines.

The author is indebted to N. R. Stemple for his help in collecting intensity data on the CCXD.

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The Structure of Decammine-μ-peroxo-dicobalt Monosulfate Tris(bisulfate)*

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(*Received* 23 *February* 1966)

Decammine-μ-peroxo-dicobalt monosulfate tris(bisulfate), $(NH_3)_5CoO_2Co(NH_3)_5SO_4(HSO_4)_3$, crystallizes in the orthorhombic space group $P2_12_12_1$ with $a=16{\cdot}36$, $b=13{\cdot}95$, and $c=9{\cdot}98$ Å; there are four formula units in the cell. The structure was determined by Patterson and trial-and-error methods and refined by three-dimensional least-squares calculations based on 1458 reflections. The final R index is 0·078 and the standard deviations in the coordinates are about 0·003, 0·005, 0·02, and 0·02 Å for the Co, S, N, and O atoms.

The coordinating ligands about the cobalt atoms form nearly regular octahedra with Co–N and Co–O distances of 1·95 and 1·89 Å. The Co–O–O–Co group is planar, with the bridging peroxide group skewed to the Co–Co axis; the O–O distance is 1·31 Å and the Co–O–O angles are 118°. The ions are held together by a very extensive network of hydrogen bonds which appears to involve all 33 hydrogen atoms.

Recently Vannerberg & Brosset (VB; 1963) have reported the results of a crystal-structure investigation of the bridged dicobalt compound decammine-μ-peroxo-dicobalt pentanitrate,

$$(NH_3)_5CoO_2Co(NH_3)_5(NO_3)_5 .$$

They reported the O–O axis of the bridging peroxide group to be perpendicular to the Co–Co axis, a configuration in agreement with that postulated earlier by Vlček (1960) for the decammine-μ-peroxo-dicobalt(5+) cation; this configuration could result, according to Vlček, from overlap of cobalt *d*-orbitals with π-electrons on the bridging O_2 group. However, VB's results were based on a relatively small number of observed reflections and their final R index was

* Contribution No. 3348 from the Gates and Crellin Laboratories of Chemistry.