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X-ray Crystal Analysis of the Substrates of Aconitase. VI. The Structures of Sodium and Lithium Dihydrogen Citrates*

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Anhydrous lithium and sodium dihydrogen citrates form an isomorphous pair crystallizing in the space group $P2_1/a$. There are four formula units in cells of dimensions $a=9{\cdot}143$, $b=11{\cdot}354$, $c=7{\cdot}475$ Å, $\beta=107°\ 45'$ for the lithium salt and $a=9{\cdot}668$, $b=11{\cdot}682$, $c=7{\cdot}484$ Å, $\beta=105°\ 20'$ for the sodium salt. The structure was solved directly from the Patterson map of the sodium salt. The central carboxyl group of the citrate ion is the one that is ionized in these crystals. Each metal ion is surrounded by six oxygen atoms, two centrosymmetrically related octahedra sharing one edge. Each citrate ion forms two bidentate chelate rings to two centrosymmetrically related metal ions.

This paper is a continuation of the studies on the substrates of the enzyme aconitase which catalyzes the transformation of citrate to isocitrate in the Krebs cycle (Paper V, Johnson, 1965). In a survey of various citrates (Love & Patterson, 1960) it was found that anhydrous lithium and sodium dihydrogen citrates form an isomorphous pair crystallizing in the space group $P2_1/a$ with four formula units $MC_6O_7H_7$(M = Li or Na) in the unit cell. The crystals were prepared from solutions composed of alkali carbonate and citric acid mixed in calculated proportions. Lithium dihydrogen citrate crystals were grown in an oven at 90°C by evaporation of a solution of the dihydrogen citrate. Sodium dihydrogen citrate crystals were grown from aqueous solutions of disodium hydrogen citrate layered under propanol at 70°C on a steam bath. The cell dimensions of the two compounds were measured from precession photographs. Values for the sodium salt were redetermined from some high angle reflections using the General Electric Goniostat. Unit-cell dimensions for both salts are given in Table 1. The differences in cell dimensions ($5\frac{1}{2}\%$ in *a* and 3% in *b*) are larger than are usually found in isomorphous pairs and are in the direction of a smaller cell for the lithium salt. The density of the lithium salt is greater than that of the sodium salt.

Data collection

Crystals of these two salts were ground to spheres (Bond, 1951) in order to simplify the absorption geometry. Spheres of sodium dihydrogen citrate used varied in diameter from 0·33 to 0·41 mm and the sphere of the lithium salt used measured 0·25 mm in diameter. Data were collected with an integrating Weissenberg camera with Cu $K\alpha$ radiation and an integrating precession camera with Mo $K\alpha$ radiation. All data for the lithium salt and some of the data for the sodium salt were measured from films by the use of a photometer. The remainder were estimated visually.

During the last stages of refinement of the structure a General Electric Goniostat was used to collect counter data for sodium dihydrogen citrate to complete the copper sphere and to remeasure those reflections with $|F|$ greater than 25·0. In all, 419 counter data were obtained and were used to correlate the Weissenberg and precession data. The total number of data for sodium dihydrogen citrate was 2004 of which 214 were below the threshold of the particular technique of measurement used. The three principal zones for lithium dihydrogen citrate have 345 reflections within the range of films and, of these, 84 were too weak to be measured. Corrections for Lorentz and polarization factors and for absorption were made.

Table 1. *Cell dimensions and densities*

	a	*b*	*c*	*β*	Density (obs.)	Density (calc.)
Li salt	9·143 Å	11·354 Å	7·475 Å	107°45′	1·783 g.cm^{-3}	1·780 g.cm^{-3}
Na salt	9·668	11·682	7·484	105°20′	1·747	1·744

(Standard errors: axial lengths 0·13 %; axial angles 0·10°; observed densities 1 %).
There is no apparent cleavage.

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Determination of a trial structure

The position of the sodium atom was found by a comparison of the two-dimensional $|F|^2$ maps of the sodium and lithium salts for each projection. It was hoped that the correct structure could be determined by using signs obtained from the isomorphous replacement technique. Unfortunately, the isomorphism is not exact and the Fourier maps for the sodium salt calculated on these signs showed many spurious peaks. None of the trial structures based on such maps was correct.

From the three-dimensional data of the sodium salt, a sharpened $|F|^2$ synthesis was calculated. The sharpening function was of the form $(\sum_i Z_i/\sum_i f_i)^2$. The parameters of the sodium atom found by two-dimensional methods were reaffirmed by the $|F|^2$ synthesis.

A correlation of all peaks on the $|F|^2$ map lying near the surface of a sphere of radius 2·3 Å from each of the four sodium–sodium peaks yielded the positions of five different oxygen atoms. These represent six oxygen atoms around sodium in a distorted octahedral arrangement with two of these oxygen atoms shared between two sodium atoms. The relative positions of three of these oxygen atoms resembled the oxygen positions of a carboxyl group together with an α-hydroxyl group. The probable positions of the two carbon atoms connecting those two groups were confirmed by their vectors from the sodium and from the three best-determined oxygen atoms on the $|F|^2$ synthesis. The remaining atoms were located in the same way. Only five of the 406 vectors calculated from this trial structure were in negative regions on the $|F|^2$ map. A correlation of the positions of the vectors yielded the parameters given in Table 2 which correspond closely with the final parameters listed in Table 3.

Table 2. *Initial fractional coordinates determined from the three-dimensional* $|F|^2$ *map*

	x	y	z
Na	0·09	0·13	0·10
O(1)	0·35	0·32	0·48
O(2)	0·19	0·17	0·43
O(3)	−0·04	0·55	0·78
O(4)	0·19	0·57	0·93
O(5)	0·13	0·31	1·01
O(6)	0·36	0·35	1·13
O(7)	0·40	0·43	0·82
C(1)	0·27	0·25	0·52
C(2)	0·28	0·24	0·72
C(3)	0·26	0·36	0·80
C(4)	0·14	0·43	0·70
C(5)	0·10	0·53	0·81
C(6)	0·25	0·33	1·00

Refinement of the structure of the sodium salt

Initially shifts were determined from difference Fourier syntheses computed for the projections. Least-squares calculations were then carried out on the three-dimensional data. The first three-dimensional structure factor calculation resulted in an R value of 18·8% which was reduced to 13·2% after two cycles of block-diagonal least-squares refinement. At this stage a three-dimensional difference Fourier synthesis was computed, from which all seven hydrogen atoms were located as shown in Fig. 1.

The atomic scattering factors of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) were used for carbon, oxygen and sodium and those of Ibers (1962) for hydrogen. The anomalous dispersion correction for sodium of $\Delta f' = 0{\cdot}1$ was also applied (Templeton, 1962). For the ionized oxygen atoms of the carboxyl groups the average of the scattering factors for oxygen as O and O^-, listed by Ibers (1962), were used.

Further block-diagonal least-squares refinements were made.* After each cycle 70% of the indicated parameter shifts were used. When the R value was 8·3% for the observed data only a correction for secondary extinction (Zachariasen, 1963*a*) was made. The extinction correction was applied in the form

$$F_{\text{corr}} = KF_{\text{obs}}\,(1 + \alpha\beta_{(2\theta)}\ \text{Lp}\ |F_{\text{obs}}|^2)$$

Values of $\beta_{(2\theta)}$ for $\mu r = 0{\cdot}36$ were obtained by interpolation in a table (Zachariasen, 1963*b*). The value of α was determined graphically to be $2{\cdot}05 \times 10^{-5}$. The present R values are 6·6% for the observed data and 7·4% for all data with unobserved reflections included at half their threshold intensity values.

The parameters of the atoms are given in Table 3 with the anisotropic temperature coefficients for the 14 heavier atoms. Since, in the least-squares refinement, the temperature factors for the hydrogen atoms

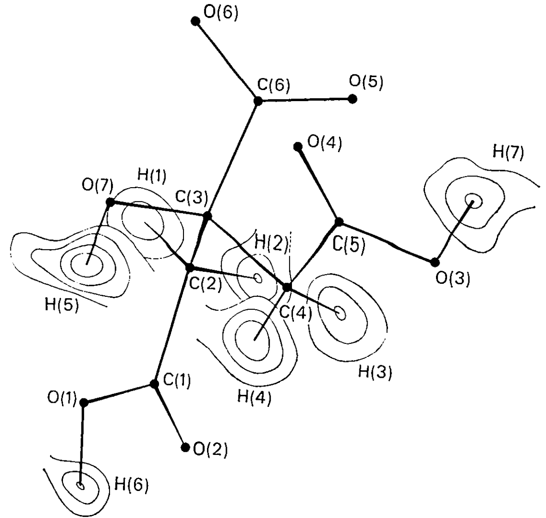

Fig. 1. The location of hydrogen atoms from a three-dimensional difference Fourier map for the sodium salt. Composite map: first contour 0·2 e.Å^{-3}; contour interval 0·2 e.Å^{-3}.

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* The weights assigned were $1/F_o^2$ for each observed reflection and $1/(3F_{\text{min}})^2$ for unobserved reflections. The program used omits reflections for which F_o is small and F_c is also small, and also omits near-in reflections with large $|F_o - F_c|$. Details are given in the write-up of the program, ICR No. 4, obtainable from the authors.

Table 3. *Parameters of atoms for sodium dihydrogen citrate*

Positional parameters are expressed as fractions of cell edges. Anisotropic temperatures factors are expressed as:

$$h^2b_{11}+k^2b_{22}+l^2b_{33}+hkb_{12}+klb_{23}+lhb_{31}$$

The hydrogen atoms were given the following isotropic temperature factors (Å^2): H(1)–H(4), 1·840; H(5), 1·705; H(6) and H(7), 2·882. Standard errors, determined from the inverted block-diagonal matrices, are listed in parentheses (for the last decimal place given).

Atom	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{23}	b_{31}
Na	0·0903 (2)	0·1251 (1)	0·1024 (2)	0·0053 (2)	0·0036 (1)	0·0102 (3)	−0·0001 (2)	−0·0004 (3)	0·0032 (4)
O(1)	0·3549 (4)	0·3212 (4)	0·4777 (4)	0·0100 (5)	0·0079 (3)	0·0066 (6)	−0·0044 (6)	−0·0012 (6)	0·0068 (8)
O(2)	0·2033 (5)	0·1735 (3)	0·4092 (5)	0·0145 (6)	0·0057 (3)	0·0084 (6)	−0·0043 (6)	−0·0045 (6)	0·0039 (10)
O(3)	−0·0391 (4)	0·5487 (3)	0·7558 (5)	0·0056 (4)	0·0073 (3)	0·0108 (6)	0·0044 (5)	−0·0041 (7)	0·0023 (7)
O(4)	0·1835 (4)	0·5741 (3)	0·9328 (5)	0·0064 (4)	0·0041 (2)	0·0123 (6)	−0·0009 (4)	−0·0041 (6)	0·0056 (8)
O(5)	0·1312 (3)	0·3123 (2)	1·0186 (4)	0·0053 (3)	0·0035 (2)	0·0080 (5)	−0·0003 (4)	−0·0002 (5)	0·0057 (7)
O(6)	0·3637 (4)	0·3495 (3)	1·1414 (4)	0·0053 (3)	0·0060 (2)	0·0061 (5)	−0·0008 (5)	0·0001 (6)	0·0027 (6)
O(7)	0·4005 (3)	0·4197 (2)	0·8221 (4)	0·0043 (3)	0·0040 (2)	0·0072 (5)	−0·0014 (4)	0·0003 (5)	0·0039 (6)
C(1)	0·2714 (5)	0·2414 (4)	0·5185 (6)	0·0069 (5)	0·0037 (2)	0·0070 (6)	0·0020 (6)	−0·0022 (6)	0·0035 (9)
C(2)	0·2725 (5)	0·2405 (3)	0·7218 (5)	0·0072 (5)	0·0032 (2)	0·0065 (6)	0·0002 (5)	−0·0006 (6)	0·0049 (9)
C(3)	0·2691 (4)	0·3594 (3)	0·8087 (5)	0·0049 (4)	0·0030 (2)	0·0069 (6)	−0·0004 (5)	−0·0010 (6)	0·0048 (8)
C(4)	0·1393 (4)	0·4278 (3)	0·6979 (5)	0·0054 (4)	0·0038 (2)	0·0076 (7)	0·0004 (5)	−0·0006 (6)	0·0034 (8)
C(5)	0·0984 (4)	0·5246 (3)	0·8079 (5)	0·0052 (4)	0·0035 (2)	0·0075 (6)	0·0007 (5)	0·0011 (6)	0·0048 (8)
C(6)	0·2544 (4)	0·3390 (3)	1·0083 (5)	0·0047 (4)	0·0029 (2)	0·0062 (6)	0·0005 (5)	−0·0002 (6)	0·0043 (8)
H(1)	0·361 (8)	0·195 (6)	0·783 (11)						
H(2)	0·192 (8)	0·194 (7)	0·737 (10)						
H(3)	0·064 (9)	0·371 (6)	0·662 (11)						
H(4)	0·156 (8)	0·460 (6)	0·580 (10)						
H(5)	0·423 (8)	0·420 (7)	0·721 (10)						
H(6)	0·335 (10)	0·328 (8)	0·358 (12)						
H(7)	−0·074 (10)	0·610 (8)	0·828 (13)						

tended to very small values, these atoms were given the equivalent isotropic temperature factor of the atom to which they were attached. The principal axes of the thermal ellipsoids were determined with respect to the orthonormal vector **E** system defined by Patterson (1952). For sodium dihydrogen citrate the γ matrix defining the **E** system is:

$$\begin{bmatrix} 9{\cdot}6023 & 0 & -1{\cdot}1254 \\ 0 & 11{\cdot}6820 & 0 \\ -1{\cdot}1254 & 0 & 7{\cdot}3989 \end{bmatrix}$$

Values of the root-mean-square vibrational displacements and their direction cosines relative to the axes of the orthonormal **E** system are given in Table 4. A graphical representation of the thermal ellipsoids is given in Fig. 2.

Table 5 gives a list of values of observed and calculated structure factors for sodium dihydrogen citrate.

Table 4. *Principal axes of anisotropic temperature factors and their direction cosines relative to the axes of the orthonormal* E *system for the sodium salt*

The root-mean-square vibrational displacements (r.m.s.d.) are given in Å together with their direction cosines (l_1, l_2 and l_3) with respect to the three axes (E_1, E_2 and E_3) of the orthonormal E system defined by the γ matrix in the text.

	r.m.s.d.	l_1	l_2	l_3
Na	0·167	0·269	0·256	−0·929
	0·157	−0·290	0·941	0·175
	0·152	0·919	0·222	0·327
O(1)	0·250	0·544	−0·836	0·073
	0·190	0·824	0·549	0·141
	0·124	−0·158	−0·017	0·987
O(2)	0·264	0·946	−0·321	−0·032
	0·201	0·271	0·844	−0·462
	0·133	0·176	0·428	0·886
O(3)	0·246	0·377	0·863	−0·335
	0·161	0·283	0·237	0·929
	0·138	0·882	−0·445	−0·155
O(4)	0·199	0·160	−0·615	0·772
	0·165	0·987	0·106	−0·121
	0·146	−0·007	0·782	0·624
O(5)	0·161	0·702	−0·455	0·547
	0·154	0·321	0·889	0·327
	0·130	−0·635	−0·054	0·771
O(6)	0·204	0·121	−0·993	−0·018
	0·153	0·990	0·122	−0·073
	0·127	−0·075	−0·009	0·997
O(7)	0·172	−0·331	0·943	0·037
	0·141	0·536	0·155	0·830
	0·125	0·777	0·294	−0·557
C(1)	0·188	0·778	0·595	−0·202
	0·155	−0·597	0·599	−0·533
	0·122	−0·196	0·536	0·821
C(2)	0·178	0·988	0·047	0·146
	0·151	−0·016	0·978	−0·209
	0·126	−0·153	0·204	0·967
C(3)	0·154	0·697	−0·506	0·509
	0·141	0·552	0·831	0·071
	0·123	−0·459	0·231	0·858
C(4)	0·164	−0·395	−0·900	0·183
	0·152	0·912	−0·361	0·195
	0·140	−0·109	0·244	0·964
C(5)	0·162	−0·534	−0·730	−0·427
	0·146	0·724	−0·656	0·215
	0·131	−0·437	−0·194	0·878
C(6)	0·148	0·789	0·532	0·308
	0·140	−0·408	0·827	−0·386
	0·119	−0·460	0·179	0·870

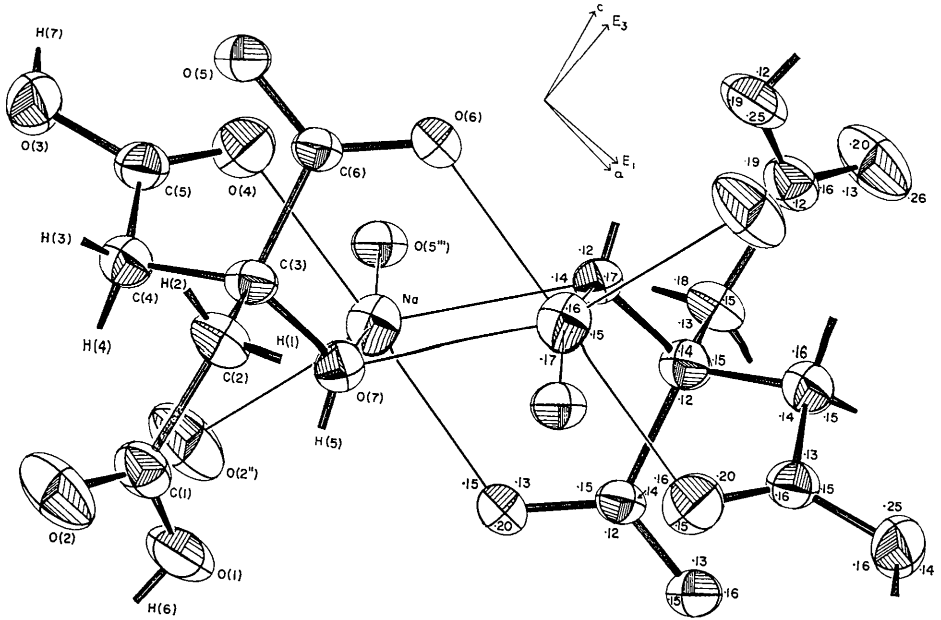

Fig. 2. A representation of the principal axes of the thermal ellipsoids for the sodium salt, plotted in parallel projection and viewed along the b (E_2) axis. The ellipsoid scale is for 67 % probability. The center of symmetry between the two sodium ions is at $\frac{1}{2}$, $\frac{1}{2}$, 0.

Table 5. *Values of observed and calculated structure factors for sodium dihydrogen citrate*

The observed values given are corrected for extinction (see text). An asterisk indicates that the reflection was too weak for measurement. The value listed corresponds to an intensity of half the threshold intensity of the technique of measurement used.

H	OBS	CALC
(H,0,0)		
2	74.5	-78.3
4	55.0	-56.4
6	29.0	-30.4
8	21.3	21.1
10	3.3	-0.3
12	1.8*	0.9
(H,1,0)		
1	7.2	-10.5
2	9.5	11.1
3	12.3	-14.0
4	33.3	-35.8
5	27.0	-27.0
6	4.1	4.0
7	12.4	-12.2
8	3.2	-1.8
9	13.9	13.7
10	10.2	9.4
11	5.9	5.0
12	8.9	-7.9
13	7.7	7.2
(H,2,0)		
0	29.9	-33.4
1	49.7	54.0
2	77.8	80.5
3	22.5	21.2
4	17.2	-17.7
5	17.0	-17.6
6	2.3	1.5
7	11.2	-11.5
8	17.0	-16.4
9	39.1	38.2
10	9.0	9.0
11	3.0	-2.8
12	1.9*	1.5
13	4.0	-7.3
(H,3,0)		
1	71.1	-73.2
2	5.3	5.2
3	42.3	42.1
4	21.5	-20.8
5	1.4*	2.5
6	12.2	-12.2
7	11.3	11.8
8	1.2*	-0.4
9	5.1	-4.4
10	7.6	-7.4
11	1.3*	-0.4
12	9.1	8.6
(H,4,0)		
0	59.7	-62.4
1	24.9	-26.9
2	1.6*	-0.2
3	7.6	8.1
4	30.6	32.9
5	30.5	30.7
6	10.8	10.8
7	6.8	6.8
8	3.4	-3.6
9	6.9	-6.8
10	3.2	-2.7
11	5.1	5.1
12	7.4	-6.4
(H,5,0)		
1	25.1	-27.8
2	16.4	18.3
3	10.1	-10.6
4	1.1*	1.6
5	41.1	41.7
6	7.7	6.3
7	11.0	12.2
8	32.0	-32.1
9	13.7	-13.9
10	9.8	-9.6
11	5.4	-4.5
12	11.7	10.6
(H,6,0)		
0	10.6	10.8
1	2.8	-3.2
2	11.7	-11.1
3	46.9	48.0
4	9.3	-9.8
5	3.6	-1.8
6	2.1*	2.3
7	1.8*	-0.9
8	10.3	10.2
9	1.3*	-1.0
10	8.4	-8.3
11	1.2*	1.8
12	1.5*	-0.5
(H,7,0)		
1	29.2	29.5
2	1.9	3.5
3	27.7	-28.8
4	16.4	16.9
5	11.4	11.4
6	18.8	-19.9
7	1.8*	0.7
8	1.8*	-2.4
9	1.3*	1.9
10	9.3	-9.5
11	10.5	-9.6
(H,8,0)		
0	15.0	-15.4
1	18.5	18.6
2	2.1	1.8
3	20.4	-19.8
4	16.2	16.1
5	5.1	5.9
6	29.1	-29.0
7	4.4	4.5
8	2.3*	-1.5
9	12.9	13.0
10	5.6	5.7
11	7.5	-6.7
(H,9,0)		
1	10.7	11.0
2	2.0	-0.3
3	9.1	9.5
(H,9,0)		
4	17.6	-18.1
5	10.5	-10.0
6	1.3*	-0.9
7	1.3*	0.5
8	1.8*	0.1
9	5.6	-4.4
10	17.0	15.8
(H,10,0)		
0	20.9	-21.6
1	19.3	-19.9
2	15.4	14.9
3	30.1	-29.6
4	18.1	-18.7
5	1.8*	1.0
6	7.0	6.9
7	23.5	23.6
8	5.0	-4.7
9	3.3	-3.3
10	7.5	6.4
(H,11,0)		
1	1.7*	-2.7
2	15.4	-15.6
3	4.9	5.3
4	4.1	3.7
5	7.8	7.7
6	3.5	-3.0
7	3.6	-3.5
8	7.6	7.8
9	1.5*	-1.5
(H,12,0)		
0	12.4	12.2
1	2.0	-2.2
2	18.7	-19.6
3	3.2	-3.5
4	5.2	5.3
5	2.8	-2.7
6	3.9	3.9
7	3.8	3.1
8	5.4	5.0
(H,13,0)		
1	1.3*	1.3
2	9.2	9.2
3	2.8	2.4
4	8.6	8.5
5	5.4	-5.6
6	2.9	-1.9
7	3.4	3.3
8	2.9	-4.1
(H,14,0)		
0	0.8	0.4
1	0.8*	-0.8
2	0.9*	-0.1
3	6.8	6.5
4	1.7*	-2.4
5	1.6*	0.4
6	1.3*	2.1
(H,15,0)		
1	2.8	2.5
2	12.9	12.9
3	3.7	2.8
4	4.9	4.5
5	5.4	-4.5
(H,16,0)		
0	7.8	7.7
1	6.7	6.3
2	1.0*	-0.1
3	2.2	-3.6
(H,0,1)		
-12	7.0	7.7
-10	11.5	11.8
-8	16.8	-16.7
-6	11.2	9.2
-4	6.2	-5.5
-2	28.1	30.8
0	75.8	68.7
2	8.9	9.9
4	65.4	-67.4
6	49.9	-51.5
8	25.2	26.8
10	3.6	2.4
(H,1,1)		
-12	2.7	-2.6
-11	2.5	1.7
-10	2.6	1.4
-9	9.4	-9.2
-8	4.2	2.8
-7	33.7	-33.8
-6	31.3	-30.3
-5	36.2	-37.8
-4	43.6	44.1
-3	38.4	41.1
-2	1.3	1.7
-1	60.7	59.0
0	8.4	-8.6
1	47.2	-46.0
2	49.5	-52.0
3	4.4	2.3
4	7.1	6.3
5	17.6	-20.2
6	7.1	7.3
7	16.6	17.7
8	9.2	8.6
9	19.2	21.3
10	4.7	-4.3
11	1.8	-1.2
(H,2,1)		
-12	4.5	4.7
-11	1.0*	0.6
-10	4.8	5.4
-9	16.1	-16.4
-8	2.6	1.3
-7	1.1*	0.0
-6	0.8*	0.9
-5	4.3	3.1
-4	39.9	40.8
-3	62.8	62.9
(H,2,1)		
-2	35.0	-36.4
-1	20.8	-22.4
0	38.7	40.6
1	14.2	15.3
2	13.4	-13.9
3	6.9	-7.4
4	16.4	-16.4
5	18.6	19.5
6	4.1	-1.4
7	17.2	-18.8
8	16.7	-18.8
9	16.3	17.8
10	6.9	7.4
11	1.3	1.8
(H,3,1)		
-11	5.3	-5.3
-10	8.4	-8.0
-9	2.9	1.1
-8	5.7	5.4
-7	12.6	11.8
-6	2.0	1.2
-5	19.8	-19.0
-4	10.0	12.0
-3	13.6	-13.2
-2	39.7	-40.8
-1	6.2	7.3
0	24.5	-25.7
1	1.0	0.2
2	31.5	30.3
3	37.7	-37.0
4	46.8	-47.3
5	41.8	41.2
6	3.3	3.1
7	18.7	19.8
8	9.7	9.9
9	8.5	-9.0
10	6.4	6.7
11	5.4	-5.8
(H,4,1)		
-11	5.4	4.7
-10	5.0	4.5
-9	6.3	6.7
-8	13.4	13.9
-7	8.8	-9.4
-6	28.0	28.8
-5	6.5	-6.1
-4	18.4	-19.0
-3	23.6	-26.5
-2	2.8	2.0
-1	113.1	103.7
0	59.7	-60.3
1	9.6	-9.2
2	23.1	26.0
3	14.5	14.5
4	20.0	22.5
5	2.4*	0.2
6	15.8	17.0
7	4.9	-6.4
8	20.8	-23.4
9	2.7	-2.4
10	6.3	-6.9
11	3.7	4.0
(H,5,1)		
-11	2.4	1.9
-10	13.8	13.3
-9	6.4	-6.4
-8	7.9	7.8
-7	4.2	4.4
-6	11.7	11.5
-5	29.0	28.8
-4	43.3	-43.1
-3	4.4	5.4
-2	44.9	-45.7
-1	71.5	-69.9
0	25.2	27.5
1	13.2	-13.0
2	9.9	11.3
3	37.6	38.6
4	14.0	-13.8
5	21.4	23.2
6	17.4	-18.6
7	10.8	-11.6
8	9.6	9.6
9	7.0	-7.3
10	7.3	-7.7
(H,6,1)		
-11	4.6	4.8
-10	11.6	-12.2
-9	5.6	5.2
-8	25.4	26.6
-7	5.6	5.8
-6	8.6	-9.2
-5	23.2	-25.8
-4	9.9	-9.6
-3	12.3	-13.0
-2	18.1	-18.5
-1	28.2	28.9
0	0.6*	1.7
1	35.7	37.4
2	9.4	10.6
3	21.6	23.9
4	22.1	23.8
5	10.7	-12.3
6	19.1	-20.2
7	10.1	-9.9
8	9.6	9.7
9	7.1	-7.2
10	5.4	-6.1
(H,7,1)		
-10	3.9	3.6
-9	3.5	-3.0
-8	13.6	13.2
-7	4.3	-2.7
-6	11.8	-12.0
-5	21.3	22.4
-4	9.1	-8.9
-3	23.7	-25.5
-2	6.3	6.3
-1	14.1	15.3
0	30.4	-31.1
1	17.2	18.4
2	5.8	5.8
3	5.7	-4.5
4	2.3	-2.3
5	23.3	-25.0
(H,7,1)		
6	15.4	16.1
7	2.1	1.6
8	16.5	-17.3
9	2.4	2.0
(H,8,1)		
-10	2.9	2.7
-9	3.1	-3.1
-8	10.5	-10.9
-7	6.0	-6.0
-6	6.6	-5.7
-5	1.2*	1.2
-4	16.1	-17.3
-3	19.6	20.4
-2	16.4	18.1
-1	12.7	-13.9
0	4.8	4.8
1	12.2	12.7
2	5.2	5.2
3	3.9	-3.7
4	2.6	-2.6
5	4.4	4.3
6	5.7	-5.7
7	4.8	-4.3
8	3.5	3.7
9	5.6	5.7
(H,9,1)		
-9	0.6*	1.1
-8	10.7	-10.8
-7	5.0	-4.7
-6	18.1	18.9
-5	3.9	4.2
-4	25.7	27.7
-3	5.0	4.5
-2	17.5	-18.5
-1	10.1	10.6
0	13.1	-14.9
1	14.5	15.6
2	22.1	-23.9
3	24.5	-26.2
4	23.3	24.4
5	3.0	-2.9
6	4.2	-4.3
7	5.1	-5.2
8	6.2	6.2
(H,10,1)		
-9	5.5	-6.0
-8	2.7	1.6
-7	8.8	-8.0
-6	11.9	-12.3
-5	11.6	11.4
-4	2.8	-2.0
-3	12.9	12.8
-2	4.0	4.1
-1	1.0*	0.8
0	2.4	1.3
1	11.3	-11.3
2	2.7	-2.5
3	14.6	-16.2
4	2.6	-2.1
5	2.2	-1.4
6	12.0	12.4
7	15.7	16.7
8	4.1	-3.8
(H,11,1)		
-8	5.4	-4.8
-7	4.4	4.3
-6	8.6	8.3
-5	1.0*	0.4
-4	6.4	6.8
-3	15.1	16.2
-2	13.1	13.9
-1	9.9	-10.1
0	13.1	-14.6
1	15.6	-16.8
2	14.5	-15.9
3	12.0	12.1
4	6.9	7.1
5	2.7	1.9
6	13.5	14.4
7	4.1	-4.4
(H,12,1)		
-7	2.9	-2.9
-6	4.9	4.5
-5	0.7*	0.0
-4	0.9*	0.5
-3	8.1	-8.2
-2	6.5	-6.8
-1	5.6	5.2
0	8.2	-8.1
1	2.3	1.7
2	3.5	3.0
3	1.1*	-0.5
4	5.8	5.7
5	11.5	-12.8
6	2.7	2.9
(H,13,1)		
-5	4.0	-4.0
-4	6.2	-5.9
-3	2.9	-3.1
-2	3.5	3.6
-1	3.5	3.7
0	0.9*	0.1
1	1.7	1.4
2	5.0	5.2
3	5.2	-5.5
4	6.6	6.6
5	1.0	-0.6
(H,14,1)		
-4	6.9	7.7
-3	0.6*	0.9
-2	9.1	-9.8
-1	2.9	-3.4
0	8.1	8.3
1	10.8	11.9
2	4.7	-4.4
3	1.7	-2.2
(H,15,1)		
-4	2.1*	-0.9
-3	0.9*	-0.5
-2	9.0	-9.0
(H,15,1)		
-1	10.6	9.9
0	4.9	4.4
1	4.1	-4.3
2	14.4	13.8
(H,16,1)		
-4	2.0	-3.9
-3	7.4	-7.0
-2	8.7	8.7
-1	6.6	6.1
0	3.1	0.4
1	3.1	-2.8
2	1.7*	0.4
(H,0,2)		
-12	5.0	5.5
-10	7.0	-6.7
-8	11.7	-11.3
-6	4.2	-4.1
-4	39.4	41.2
-2	34.6	-37.6
0	10.4	10.1
2	63.5	-64.9
4	52.1	-52.9
6	22.4	24.0
8	13.6	14.3
10	1.1*	0.2
(H,1,2)		
-12	0.6*	-0.6
-11	2.5	-1.9
-10	4.2	4.1
-9	23.4	-25.3
-8	17.9	-19.0
-7	14.8	-13.6
-6	3.9	-2.7
-5	9.8	9.0
-4	35.4	36.8
-3	44.0	44.3
-2	2.6	-1.2
-1	48.9	49.5
0	31.8	-32.6
1	6.3	5.9
2	22.6	-26.6
3	6.4	-7.3
4	17.1	17.7
5	14.7	-15.2
6	12.9	12.6
7	12.6	12.8
8	8.0	6.9
9	13.4	13.0
10	3.5	-1.9
11	6.3	-5.5
(H,2,2)		
-12	1.1	0.2
-11	0.9*	1.7
-10	10.4	10.5
-9	11.9	-10.7
-8	15.8	-15.2
-7	4.7	4.6
-6	9.4	8.9
-5	49.6	50.1
-4	21.1	19.9
-3	9.8	-9.7
-2	9.0	-9.3
-1	15.0	14.4
0	74.7	-73.3
1	13.8	-13.8
2	20.5	-19.9
3	26.2	-29.7
4	19.1	18.9
5	15.2	14.8
6	5.1	4.7
7	7.4	-7.5
8	9.2	-8.0
9	8.6	8.5
10	1.5*	0.3
(H,3,2)		
-12	0.5*	-0.1
-11	2.8	-2.6
-10	5.2	-4.8
-9	11.5	10.5
-8	11.8	-12.2
-7	10.4	8.8
-6	19.9	-19.1
-5	25.8	-26.4
-4	36.4	36.9
-3	5.3	4.2
-2	10.8	-10.8
-1	32.6	32.3
0	34.3	-34.6
1	3.3	-1.9
2	3.1	1.5
3	12.8	14.7
4	9.5	7.8
5	7.5	7.4
6	4.8	3.2
7	4.9	-2.2
8	19.8	21.2
9	2.8*	-2.1
10	2.1*	1.2
(H,4,2)		
-12	0.4*	-6.2
-11	4.9	4.2
-10	2.7	2.4
-9	15.4	16.2
-8	28.2	29.0
-7	8.9	7.7
-6	5.0	5.6
-5	32.4	-32.1
-4	25.9	-28.2
-3	17.6	18.5
-2	23.9	-28.2
-1	77.2	76.6
0	17.1	-18.0
1	28.1	-28.8
2	16.8	18.8
3	33.0	-33.0
4	2[illegible].9	25.1
5	[illegible].1	-3.6
6	5.0	3.7
7	5.1	4.0
8	16.8	-16.3
9	11.5	-11.7
10	3.9	-3.7
(H,5,2)		
-11	4.0	3.4
-10	2.2	-2.3
-9	12.3	11.6
-8	2.3	-1.0
-7	3.6	-4.0
-6	22.6	-24.8
-5	1.7	1.8
-4	25.5	-26.4
-3	17.7	-19.7
-2	16.5	16.6
-1	20.2	-22.0
0	4.2	-2.3
1	30.6	31.5
2	44.9	44.2
3	5.7	5.7
4	25.3	-27.4
5	6.6	7.6
6	3.3*	1.1
7	12.6	-11.6
8	8.5	8.8
9	10.0	-9.0
10	6.9	6.8
(H,6,2)		
-11	5.8	5.7
-10	10.4	-9.7
-9	5.8	5.8
-8	18.6	18.2
-7	14.0	14.5
-6	11.9	11.1
-5	9.9	-10.5
-4	21.1	-21.5
-3	27.5	-28.2
-2	19.5	-21.1
-1	18.3	20.0
0	35.3	34.9
1	44.9	48.3
2	13.2	14.6
3	10.1	-8.8
4	4.4	-2.4
5	26.0	-27.0
6	12.5	-12.5
7	14.4	-15.5
8	8.2	8.3
9	2.3*	1.6
10	3.6	-3.3
(H,7,2)		
-10	5.5	4.8
-9	0.8*	-0.9
-8	10.4	-9.7
-7	14.6	-13.4
-6	13.2	-12.6
-5	4.0	1.5
-4	3.7	-4.1
-3	3.1	-3.2
-2	9.7	-8.3
-1	2.5	1.3
0	17.6	17.4
1	11.8	12.2
2	7.7	-7.3
3	14.0	-14.7
4	11.2	10.2
5	14.3	-14.4
6	3.2*	3.3
7	3.0*	0.3
8	2.6*	-2.0
9	3.0	2.6
(H,8,2)		
-10	5.8	-5.4
-9	2.6	1.8
-8	14.3	-13.6
-7	2.7	2.3
-6	5.2	5.0
-5	10.4	10.5
-4	8.9	7.6
-3	4.3	3.6
-2	24.3	27.1
-1	16.9	-17.1
0	11.0	-11.6
1	3.4	3.2
2	11.4	10.7
3	9.8	10.1
4	1.5*	0.4
5	2.4	1.0
6	13.6	-13.0
7	12.7	-13.7
8	5.0	4.6
9	1.4*	0.5
(H,9,2)		
-9	2.6	2.2
-8	4.2	-3.9
-7	10.9	-10.0
-6	16.2	15.5
-5	4.8	-4.4
-4	8.6	7.9
-3	5.4	5.2
-2	6.6	-6.0
-1	12.5	12.0
0	19.9	-20.1
1	15.4	-16.3
2	13.6	-14.0
3	10.6	-10.0
4	17.8	18.4
5	7.0	-7.0
6	10.8	10.8
7	4.7	3.9
8	1.8*	0.2
(H,10,2)		
-9	6.5	7.1
-8	2.4	2.3
-7	0.7*	-1.7
-6	1.0*	-0.5
-5	11.8	11.2
-4	5.4	-5.1
-3	11.3	10.7
-2	13.0	12.5
-1	17.1	-16.9
0	7.7	7.9
1	17.7	-18.2
2	2.5	-1.7
3	15.2	15.2
4	4.5	-4.3
5	2.5	2.2
6	7.5	8.0
7	4.9	5.6
8	0.4*	1.8
(H,11,2)		
-8	3.0	2.9
-7	6.1	5.4
-6	10.5	9.9
-5	0.8*	-0.4
-4	2.9	1.8
-3	0.8*	-0.5
-2	2.2	2.1
-1	15.8	-15.9
0	6.2	-5.7
1	1.1*	-1.0
2	17.1	-16.9
3	3.9	-3.9
4	5.4	5.0
5	11.7	11.4
6	12.4	12.7
7	3.5	-3.8
(H,12,2)		
-7	6.1	6.0
-6	4.5	-4.5
-5	1.9	1.4
-4	10.8	11.2
-3	9.4	-8.9
-2	15.1	-15.5
-1	9.1	-9.3
0	6.4	5.9
1	14.5	15.2
2	0.6*	0.0
3	7.2	6.4
4	8.1	8.0
5	10.2	-10.0
6	4.6	5.1
(H,13,2)		
-6	0.5*	-0.3
-5	3.1	2.7
-4	3.7	-3.6
-3	9.5	-9.1
-2	5.4	5.9
-1	2.5	-2.5
0	5.1	5.3
1	3.9	-4.0
2	0.6*	-0.7
3	5.8	5.5
4	7.1	-6.9
(H,14,2)		
-5	13.7	-13.0
-4	4.6	4.2
-3	1.7	0.7
-2	4.9	5.1
-1	4.5	4.8
0	7.3	-7.3
1	10.1	9.7
2	0.5*	1.0
3	0.4*	-3.9
(H,15,2)		
-5	1.8*	0.3
-4	3.7	3.9
-3	3.7	3.8
-2	1.7*	1.7
-1	1.0*	1.7
0	2.7	1.9
1	5.5	-5.1
(H,16,2)		
-2	6.6	8.9
-1	9.4	8.2
0	4.0	3.6
1	5.3	-3.1
(H,0,3)		
-12	0.9*	-0.2
-10	17.1	-18.9
-8	24.7	-25.3
-6	2.7	1.8
-4	41.5	44.5
-2	11.7	-11.2
0	46.7	-45.9
2	16.7	-18.2
4	9.3	8.3
6	27.6	27.8
8	14.9	14.1
10	3.6	-3.2
(H,1,3)		
-12	9.0	-9.7
-11	6.9	6.6
-10	10.5	10.2
-9	16.7	-17.2
-8	4.0	2.0
-7	7.7	7.1
-6	17.8	-17.3
-5	32.7	33.5
-4	5.7	4.6
-3	40.6	43.9
-2	96.7	93.4
-1	18.0	-17.3
0	87.1	-82.4
1	46.5	-46.9
2	17.7	-20.6
3	23.6	20.9
4	29.0	28.0
5	4.3	-3.8
6	19.9	22.0
7	17.8	-18.0
8	5.7	-5.2
9	6.1	4.9
10	11.6	12.5
(H,2,3)		
-12	6.0	-6.2
-11	1.6*	0.9
-10	6.2	-5.1
-9	10.4	-10.1
-8	9.5	-9.3
-7	20.1	21.2
-6	3.4	-1.0
-5	7.3	8.0
-4	7.1	7.9
-3	2.9	3.3
-2	23.8	-24.0
-1	26.6	-28.4
0	9.7	-9.0
1	48.7	-50.7
2	3.5	-2.7
3	1.7*	0.3
(H,2,3)		
4	4.3	3.9
5	20.3	21.5
6	22.6	23.5
7	8.5	7.5
8	4.6	2.9
9	5.4	-4.9
10	7.0	-6.9
(H,3,3)		
-12	5.1	-5.4
-11	9.0	-8.8
-10	6.2	-5.6
-9	26.1	26.7
-8	3.5*	-0.2
-7	21.2	22.5
-6	5.2	-5.6
-5	31.2	-31.0
-4	32.4	32.4
-3	34.0	-35.6
-2	21.1	21.3
-1	35.1	37.2
0	20.2	-20.8
1	41.3	42.7
2	2.4*	-2.1
3	22.0	-22.4
4	13.2	13.0
5	10.1	-10.3
6	12.1	12.4
7	3.4*	1.1
8	5.7	4.7
9	7.9	-8.6
(H,4,3)		
-11	4.6	4.3
-10	5.6	4.8
-9	6.2	-5.3
-8	12.1	12.4
-7	7.7	-7.6
-6	4.8	-4.9
-5	30.2	31.5
-4	32.0	-34.5
-3	36.8	-38.9
-2	11.2	-10.8
-1	1.9	-1.3
0	15.3	16.0
1	4.0	-4.4
2	13.9	14.7
3	29.5	-29.6
4	3.1*	2.2
5	3.3*	2.0
6	5.9	3.2
7	6.2	6.0
8	6.5	-6.8
9	2.4*	1.9
(H,5,3)		
-11	9.2	8.6
-10	5.5	-5.1
-9	11.1	11.3
-8	11.6	-10.9
-7	10.1	-10.9
-6	13.7	-13.7
-5	3.4	-2.9
-4	12.1	13.1
-3	16.9	-17.2
-2	8.2	9.0
-1	1.6	1.3
0	38.0	38.9
1	16.3	17.9
2	13.3	14.4
3	18.5	18.9
4	16.7	-17.8
5	8.4	-8.5
6	7.8	-8.9
7	13.8	-14.4
8	9.0	10.5
9	2.2*	0.1
(H,6,3)		
-11	4.1	4.0
-10	2.7*	1.7
-9	3.1*	-1.4
-8	5.9	-3.6
-7	3.5*	0.9
-6	3.0	-2.3
-5	7.4	-7.8
-4	2.3	2.5
-3	13.8	-14.4
-2	9.5	10.7
-1	0.7*	-0.2
0	2.3	-1.6
1	16.3	17.6
2	10.0	-9.5
3	12.8	-12.8
4	15.0	16.5
5	16.5	-16.5
6	20.6	-23.7
7	8.0	-8.3
9	10.6	10.7
(H,7,3)		
-10	5.7	5.4
-9	5.0	-3.2
-8	3.3*	-1.2
-7	10.6	-9.4
-6	29.3	-30.8
-5	7.0	-7.9
-4	2.3	-1.2
-3	10.9	11.9
-2	40.4	41.6
-1	0.8*	1.8
0	9.3	9.9
1	11.1	11.5
2	5.7	-5.2
3	15.6	-17.4
4	3.5*	-1.0
5	7.0	-6.6
6	3.3*	0.6
7	12.1	11.5
8	6.0	-4.2
(H,8,3)		
-10	5.3	-4.9
-9	8.8	8.0
-8	9.9	-8.9
-7	10.3	10.6
-6	4.6	4.0
-5	15.3	-15.7
-4	17.7	19.4
-3	8.1	8.3
(H,8,3)		
-2	21.9	23.8
-1	10.8	-12.5
0	21.3	-22.4
1	11.5	-12.8
2	5.8	-3.9
3	12.9	13.1
4	11.5	-12.2
5	3.3*	2.9
6	3.0*	2.6
7	4.9	-5.0
8	1.9*	-1.7
(H,9,3)		
-9	2.2*	-0.3
-8	6.5	-6.5
-7	5.3	-3.3
-6	22.4	23.3
-5	5.9	-6.0
-4	9.4	-11.1
-3	1.8*	-1.1
-2	14.0	-14.6
-1	7.9	8.8
0	4.9	4.2
1	5.9	4.3
2	8.4	9.0
3	18.5	-19.1
4	3.3*	0.0
5	5.3	2.2
6	1.4	0.8
7	15.8	15.8
(H,10,3)		
-9	10.6	11.9
-8	5.1	5.1
-7	6.1	5.8
-6	7.0	6.9
-5	10.6	11.8
-4	3.9	-3.6
-3	8.6	-8.4
-2	3.6	3.3
-1	13.3	-15.3
0	7.3	6.7
1	9.5	-9.9
2	7.7	-8.1
3	16.2	15.9
4	5.2	-3.2
5	9.2	8.9
6	2.3*	0.0
7	1.4*	-0.1
(H,11,3)		
-8	1.6*	-0.1
-7	3.9	-2.4
-6	7.8	7.1
-5	3.5	3.3
-4	1.1*	1.2
-3	10.0	-9.9
-2	16.0	-17.4
-1	6.9	-6.6
0	9.1	8.1
1	5.5	-3.9
2	5.4	-4.8
3	14.1	15.4
4	2.7*	1.5
5	8.0	7.9
6	1.6*	0.7
(H,12,3)		
-7	5.8	6.5
-6	2.2*	0.1
-5	1.1*	1.1
-4	2.3	1.7
-3	1.7*	-0.5
-2	11.3	-11.8
-1	6.4	-6.8
0	4.4	4.2
1	5.5	5.2
2	4.9	4.4
3	8.0	8.7
4	7.0	6.5
5	2.9	-3.4
(H,13,3)		
-6	9.0	-7.9
-5	1.2*	-1.7
-4	2.5	-2.7
-3	5.2	-4.6
-2	9.0	9.4
-1	4.5	-4.5
0	3.8	1.6
1	4.0	-2.2
2	3.6	-3.0
3	10.4	9.5
(H,14,3)		
-6	1.9*	1.5
-5	7.2	-7.0
-4	2.5	3.0
-3	9.9	9.5
-2	1.3*	-1.7
-1	3.5	3.2
0	2.6	-1.9
1	4.4	5.2
(H,15,3)		
-5	1.4	1.7
-4	2.1	1.4
-3	9.8	9.0
-2	4.5	4.3
-1	5.5	-5.7
0	1.3*	1.6
(H,16,3)		
0	1.1*	2.9
(H,0,4)		
-12	6.2	-6.2
-10	20.2	-21.5
-8	16.9	17.2
-6	17.5	-18.5
-4	50.7	53.7
-2	17.4	-19.8
0	20.5	21.2
2	12.3	-13.0
4	3.2	3.5
6	17.8	16.7
8	9.3	8.8

Table 5 (*cont.*)

H	OBS	CALC
(H,1,4)		
-12	2.5	-2.7
-11	4.0	-3.5
-10	3.6	-2.6
-9	1.6*	-0.3
-8	19.4	20.2
-7	20.4	21.8
-6	3.9	-2.7
-5	2.5	-0.2
-4	6.4	6.3
-3	5.5	5.0
-2	9.1	-9.6
-1	33.8	-35.1
0	7.7	-7.6
1	34.3	-35.0
2	1.5*	1.2
3	1.8*	-0.3
4	3.9	-3.4
5	19.9	20.9
6	21.2	23.1
7	10.1	-9.7
8	13.4	-13.7
9	4.5	-4.3
(H,2,4)		
-12	1.0*	-0.6
-11	1.6*	-0.4
-10	3.1	-0.2
-9	12.2	-12.7
-8	21.1	-21.5
-7	20.7	21.1
-6	14.8	15.7
-5	16.5	17.0
-4	8.9	8.9
-3	28.5	-29.1
-2	9.5	9.0
-1	43.9	-45.2
0	15.8	-16.1
1	16.8	17.4
2	35.1	36.5
3	3.0	-1.1
4	4.6	-4.0
5	27.2	27.9
6	3.3	1.1
7	4.6	3.9
8	3.1	-2.7
9	8.6	-8.3
(H,3,4)		
-12	0.8*	-10.1
-11	1.6*	1.0
-10	1.9*	-1.0
-9	14.4	14.7
-8	13.7	14.4
-7	9.2	7.9
-6	23.2	24.4
-5	3.7	-5.1
-4	11.9	-12.5
-3	52.0	-54.8
-2	13.4	12.7
-1	21.9	22.1
0	23.4	-24.6
1	6.6	6.6
2	4.9	4.0
3	9.8	9.0
4	9.5	8.3
5	16.7	-16.2
6	2.1*	-1.9
7	4.5	-4.2
8	1.7*	1.3
9	1.1*	1.4
(H,4,4)		
-11	2.8	1.5
-10	10.5	10.6
-9	7.3	-6.9
-8	7.2	-8.0
-7	21.6	-22.8
-6	7.4	-8.5
-5	10.5	11.5
-4	20.7	-21.3
-3	9.8	10.9
-2	6.6	6.1
-1	8.9	-8.4
0	41.5	41.4
1	37.9	-39.3
2	2.7	-1.2
3	19.3	17.4
4	3.6	-1.1
5	23.4	22.5
6	17.3	-14.5
7	1.9*	1.5
8	5.0	4.8
(H,5,4)		
-11	6.2	5.8
-10	11.1	-10.2
-9	4.4	3.6
-8	9.5	9.0
-7	5.2	-4.7
-6	7.9	-8.7
-5	4.8	-6.1
-4	19.7	21.8
-3	1.2*	1.2
-2	12.7	13.9
-1	10.8	-9.5
0	2.9	2.4
1	21.7	20.2
2	3.6	-2.9
3	6.7	6.7
4	12.3	-10.6
5	13.8	-13.3
6	18.0	-16.1
7	1.8*	0.3
8	6.2	4.9
(H,6,4)		
-11	2.2	-2.6
-10	5.5	-4.7
-9	12.9	-12.8
-8	6.4	6.3
-7	18.8	-19.5
-6	19.2	-20.5
-5	13.5	14.7
-4	7.6	-8.7
-3	9.7	-10.0
-2	33.5	34.4
-1	6.8	5.2
0	2.9	-3.3
1	7.2	5.3
2	23.2	-21.3
3	3.6	-2.6
(H,6,4)		
4	9.8	-9.4
5	7.4	-6.8
6	13.2	12.3
7	4.2	4.1
(H,7,4)		
-10	13.5	13.1
-9	4.2	-3.6
-8	9.3	-8.9
-7	8.4	-6.8
-6	5.5	-5.4
-5	9.5	11.0
-4	4.3	3.4
-3	24.3	25.4
-2	14.1	15.2
-1	16.1	-14.8
0	19.7	20.3
1	5.9	5.2
2	11.3	-9.6
3	12.9	-12.4
4	18.0	-16.1
5	9.3	8.3
6	5.6	-5.5
7	5.5	4.6
8	1.8	1.8
(H,8,4)		
-10	3.6	-3.0
-9	2.9	-2.7
-8	11.2	-10.8
-7	8.9	8.3
-6	1.1*	-0.8
-5	18.0	-19.5
-4	21.5	23.3
-3	5.1	6.1
-2	2.0	0.9
-1	6.7	-6.2
0	19.0	-18.2
1	10.8	10.1
2	28.9	-30.1
3	13.3	-11.8
4	17.0	15.7
5	9.8	9.1
6	2.8	1.0
7	2.5	1.7
(H,9,4)		
-9	7.1	-6.2
-8	10.0	9.1
-7	1.9*	1.2
-6	1.1*	0.5
-5	10.8	12.2
-4	10.6	-10.6
-3	2.4	1.3
-2	5.6	-5.0
-1	3.6	2.1
0	7.2	6.2
1	2.2*	-0.3
2	12.7	11.7
3	2.1*	1.4
4	6.5	-5.6
5	3.3	-2.0
6	2.4	0.6
(H,10,4)		
-8	2.4	1.9
-7	9.8	8.7
-6	10.1	10.1
-5	6.8	-7.3
-4	20.4	-20.9
-3	5.9	-5.8
-2	6.7	6.4
-1	3.6	1.8
0	6.6	-5.9
1	18.9	-17.5
2	4.8	4.9
3	18.4	18.0
4	11.9	-12.1
5	1.4*	-0.6
6	3.0	3.3
(H,11,4)		
-7	2.3	-1.2
-6	3.5	3.9
-5	0.9*	-0.3
-4	6.3	-6.3
-3	9.0	-9.0
-2	5.0	-5.8
-1	7.6	6.1
0	1.4*	1.2
1	7.1	6.2
2	6.8	5.5
3	10.3	9.5
4	9.2	8.3
5	2.1	-1.3
(H,12,4)		
-7	5.4	5.0
-6	5.2	-5.9
-5	7.1	7.7
-4	1.3*	0.0
-3	4.1	-4.4
-2	16.0	-16.8
-1	8.9	-8.1
0	9.7	9.4
1	9.4	8.5
2	6.0	5.8
3	2.1	-1.7
4	0.6*	2.5
(H,13,4)		
-7	5.2	3.8
-6	9.6	-9.8
-5	11.6	-11.7
-4	7.3	6.6
-3	0.8*	0.9
-2	3.4	-3.4
-1	5.4	4.7
0	10.4	9.3
1	2.6	2.2
2	3.4	-3.5
(H,14,4)		
-6	3.7	3.4
-5	2.5	2.8
-4	5.3	4.8
-3	8.3	8.6
-2	7.3	-6.8
(H,14,4)		
-1	4.1	3.8
0	7.5	-6.7
(H,15,4)		
-3	3.7	3.3
-2	8.3	8.0
-1	1.8*	1.0
0	2.5	-1.3
(H,0,5)		
-10	16.1	15.8
-8	14.0	14.4
-6	7.1	-6.1
-4	20.3	21.8
-2	1.5*	-0.2
0	3.2	-3.0
2	11.7	-11.4
4	7.5	7.3
6	6.8	-7.0
8	1.1*	-0.3
(H,1,5)		
-11	7.8	-8.0
-10	9.4	8.3
-9	4.8	-4.0
-8	13.8	13.9
-7	6.8	6.3
-6	1.9	-1.0
-5	7.8	7.5
-4	20.6	-23.3
-3	20.1	-23.1
-2	4.3	3.8
-1	12.3	-13.1
0	8.9	-8.6
1	10.3	-9.5
2	7.7	-8.1
3	5.1	5.5
4	22.9	23.2
5	23.6	23.3
6	6.9	-6.5
7	3.0	-2.4
8	4.1	-3.9
(H,2,5)		
-11	2.3	-2.2
-10	3.3	3.6
-9	3.5	2.0
-8	10.6	10.0
-7	29.9	30.7
-6	9.2	-10.1
-5	18.2	-19.3
-4	13.6	15.8
-3	14.4	-15.4
-2	5.0	-4.1
-1	7.5	-7.1
0	7.1	6.8
1	29.5	25.0
2	11.1	10.9
3	4.9	4.7
4	9.3	-9.3
5	16.3	16.5
6	10.3	-10.3
7	7.3	-7.3
8	5.1	-4.9
(H,3,5)		
-11	1.8	-1.8
-10	12.5	-11.5
-9	4.9	-4.4
-8	25.6	25.3
-7	11.9	13.1
-6	12.8	13.6
-5	24.8	-26.6
-4	20.2	-21.3
-3	16.1	-16.9
-2	27.2	-28.2
-1	18.0	18.7
0	20.9	-23.0
1	10.2	9.7
2	32.0	32.9
3	0.7	0.8
4	3.8	-3.8
5	14.7	-14.3
6	3.7	-2.8
7	12.9	12.8
8	7.4	-9.2
(H,4,5)		
-11	4.4	-4.7
-10	10.3	10.2
-9	4.9	4.7
-8	17.7	-17.2
-7	17.8	-19.3
-6	11.0	12.3
-5	23.7	25.4
-4	32.2	-33.1
-3	0.8*	-0.6
-2	30.4	32.6
-1	1.3*	2.1
0	5.1	4.1
1	3.6	2.8
2	21.8	22.2
3	4.2	4.2
4	25.4	-26.3
5	10.7	10.5
6	5.9	-5.2
7	4.2	4.2
(H,5,5)		
-11	6.1	6.6
-10	1.3*	0.6
-9	5.9	-5.9
-8	8.3	-8.9
-7	17.9	-18.2
-6	9.5	10.8
-5	13.5	13.8
-4	12.6	14.4
-3	16.0	16.2
-2	21.2	-22.2
-1	20.2	-19.9
0	6.2	-6.6
1	1.6*	0.5
2	8.0	-8.0
3	7.5	7.1
4	7.0	6.8
5	0.6*	1.1
6	19.0	-18.5
7	7.6	-7.3
(H,6,5)		
-10	8.6	-9.1
-9	13.1	-12.8
-8	6.6	6.7
-7	12.9	-13.7
-6	14.5	-16.5
-5	12.8	12.7
-4	13.5	14.7
-3	16.9	17.7
-2	12.5	-11.9
-1	15.8	16.0
0	26.6	28.5
1	9.9	-9.8
2	31.9	-33.0
3	5.6	-5.1
4	11.7	11.7
5	0.6*	0.4
6	2.3	1.8
7	5.9	5.8
(H,7,5)		
-10	3.9	3.9
-9	6.0	6.6
-8	1.6*	1.2
-7	4.7	-5.0
-6	8.5	-8.9
-5	12.5	13.3
-4	1.8	-1.4
-3	16.8	18.3
-2	23.5	23.0
-1	13.1	-11.8
0	15.0	-15.2
1	20.6	-20.6
2	9.9	-10.3
3	1.5	-0.8
4	7.4	-7.4
5	12.5	12.0
6	6.2	6.3
(H,8,5)		
-9	6.9	-7.5
-8	6.2	-6.1
-7	0.2*	1.3
-6	13.9	15.4
-5	11.7	-11.9
-4	5.6	-6.4
-3	9.5	10.4
-2	4.7	5.0
-1	10.8	10.8
0	18.0	-18.0
1	1.7*	0.4
2	3.9	-3.6
3	6.5	-6.2
4	8.1	7.5
5	0.2*	0.4
6	9.7	9.5
(H,9,5)		
-8	10.6	9.5
-7	7.4	7.8
-6	3.6	3.7
-5	6.0	6.4
-4	11.3	-11.9
-3	6.8	7.6
-2	15.5	-15.9
-1	10.6	-10.2
0	17.3	17.0
1	3.2	-2.3
2	4.6	4.5
3	1.5	-2.0
4	6.4	-6.2
5	1.8	1.3
(H,10,5)		
-8	2.3*	-0.2
-7	0.8*	-0.4
-6	1.0*	-0.3
-5	8.4	-8.4
-4	7.8	-8.3
-3	4.0	3.8
-2	4.6	-4.5
-1	7.4	-7.4
0	3.5	3.1
1	7.9	7.7
2	7.0	6.5
3	1.4	1.4
4	4.5	-4.3
(H,11,5)		
-8	5.9	4.8
-7	0.9*	-0.2
-6	3.8	-4.3
-5	3.4	3.4
-4	1.1*	-0.7
-3	5.0	-5.0
-2	4.6	-4.6
-1	5.6	5.6
0	5.6	3.5
1	5.3	4.8
2	4.2	4.4
3	5.7	5.6
(H,12,5)		
-8	5.5	-2.7
-7	8.7	-8.3
-6	8.4	-8.4
-5	10.1	9.0
-4	5.2	-6.0
-3	6.4	-6.6
-2	2.8	2.0
-1	5.5	5.3
0	2.4	2.5
1	10.2	-9.8
2	6.4	7.0
(H,13,5)		
-7	0.9	2.1
-6	1.5*	2.6
-5	11.6	-10.9
-4	0.7*	-0.7
-3	4.0	-3.6
-2	5.2	5.7
0	1.6*	1.7
(H,14,5)		
-4	8.8	8.8
-3	12.2	11.5
-2	6.3	-6.5
0	1.2*	-1.1
(H,0,6)		
-10	2.8	2.5
-8	49.7	48.9
-6	24.7	-25.6
-4	25.9	-27.3
-2	4.5	2.3
0	8.1	9.5
2	18.6	-19.7
4	3.4	-3.1
6	6.5	5.9
(H,1,6)		
-11	2.0	-1.6
-10	1.2*	-0.4
-9	6.6	-6.3
-8	10.5	9.7
-7	3.3	1.6
-6	4.9	-4.5
-5	7.7	6.9
-4	17.1	-17.2
-3	6.7	-5.8
-2	8.5	-7.1
-1	13.4	13.2
0	2.0	1.1
1	2.6	-2.3
2	23.3	24.9
3	4.9	4.4
4	9.3	-9.0
5	7.9	7.4
6	2.8	2.2
7	2.0	1.9
(H,2,6)		
-11	1.8	-1.8
-10	12.9	14.6
-9	7.4	7.7
-8	7.1	-7.1
-7	20.6	22.0
-6	4.0	3.9
-5	6.8	-7.5
-4	1.5	-2.6
-3	32.0	-31.9
-2	8.6	-8.2
-1	13.4	-14.1
0	10.7	-10.8
1	59.2	60.6
2	8.9	9.1
3	12.2	-11.3
4	4.0	-3.6
5	12.5	-13.0
6	8.6	-9.0
7	6.5	-7.4
(H,3,6)		
-11	5.6	6.6
-10	1.1*	-0.4
-9	12.0	-11.8
-8	12.7	13.7
-7	12.9	-14.4
-6	9.9	-10.7
-5	5.0	4.4
-4	14.5	-15.4
-3	13.2	14.0
-2	17.2	-19.5
-1	3.6	2.0
0	12.0	12.0
1	11.6	12.6
2	7.2	6.4
3	6.8	-6.2
4	17.7	19.1
5	3.6	-3.0
6	11.5	-12.8
(H,4,6)		
-10	1.1*	-0.6
-9	12.3	13.0
-8	3.8	-4.1
-7	7.5	-7.9
-6	4.7	-4.6
-5	3.6	4.7
-4	5.9	6.4
-3	1.9	1.1
-2	2.3	-1.6
-1	15.4	16.7
0	2.8	1.4
1	11.4	-12.6
2	9.7	9.2
3	3.7	3.3
4	12.4	-12.9
5	3.9	-2.7
6	1.6*	-2.0
(H,5,6)		
-10	1.5	0.3
-9	6.2	-6.4
-8	8.0	-8.4
-7	3.2	-3.7
-6	4.2	4.1
-5	3.9	-4.2
-4	8.4	-8.8
-3	13.0	13.4
-2	17.0	18.7
-1	3.8	2.8
0	30.5	-30.5
1	8.5	-8.5
2	2.3*	0.3
3	2.3*	1.2
4	12.5	13.8
5	3.3	-2.3
6	3.0	3.2
(H,6,6)		
-10	1.9	-2.6
-9	3.6	-3.7
-8	7.0	6.8
-7	0.9*	1.1
-6	11.1	10.5
-5	15.5	16.4
-4	12.0	-13.1
-3	6.9	6.3
-2	3.2	3.0
-1	5.4	5.1
0	8.6	8.9
1	5.2	-4.7
2	10.1	-10.2
3	8.2	-8.3
4	9.5	9.4
5	8.7	-8.0
6	1.8	-1.6
(H,7,6)		
-9	9.2	9.8
-8	4.1	-4.3
-7	6.7	7.3
-6	2.4	-1.7
-5	3.5	-3.4
-4	12.5	14.0
-3	1.5*	0.0
-2	9.7	-9.3
-1	8.8	-8.4
0	8.0	-5.4
1	10.3	-10.2
2	7.2	-6.2
3	3.4	-2.8
4	1.8*	1.0
5	8.9	9.5
(H,8,6)		
-9	9.6	-8.2
-8	2.3	-2.9
-7	5.5	5.8
-6	14.1	14.5
-5	5.3	-5.6
-4	14.0	14.2
-3	11.4	12.2
-2	20.2	-22.7
-1	1.4*	-1.2
0	5.9	-5.1
1	8.5	8.3
2	9.5	8.7
3	11.6	-12.1
4	14.2	14.2
(H,9,6)		
-9	7.1	5.1
-8	3.1	2.9
-7	1.6	-1.3
-6	5.7	5.1
-5	8.3	8.6
-4	5.3	-5.0
-3	7.6	-7.6
-2	10.8	-11.8
-1	8.5	-8.2
0	5.4	5.3
1	5.7	-6.2
2	7.8	7.1
3	3.4	3.7
4	5.3	-6.2
(H,10,6)		
-9	9.6	7.8
-8	6.2	-6.1
-7	12.9	-13.1
-6	4.7	4.6
-5	5.7	-6.7
-4	6.7	-7.0
-3	4.2	4.0
-2	8.9	9.5
-1	11.1	13.0
0	1.1*	-1.1
1	9.4	-9.8
2	6.9	6.6
(H,11,6)		
-8	0.8*	-0.4
-7	1.5	1.4
-6	1.8*	-0.2
-5	7.0	-7.6
-4	2.3	2.2
-3	6.9	7.3
-2	6.6	-6.2
-1	3.0	-3.4
0	5.1	4.0
1	5.0	5.3
(H,12,6)		
-7	0.8*	-1.1
-6	18.2	-16.0
-5	4.2	-4.6
-4	2.0	2.0
-3	2.2*	2.0
0	8.5	8.0
(H,13,6)		
-5	0.8*	-1.5
-4	1.0*	0.6
-3	7.4	-5.5
0	9.1	-7.7
(H,0,7)		
-10	3.0	-2.7
-8	12.1	10.8
-6	3.2	-2.7
-4	10.3	-9.2
-2	22.3	-24.0
0	7.4	7.6
2	7.2	6.3
4	3.3	2.3
(H,1,7)		
-10	8.2	8.2
-9	1.1*	0.9
-8	3.6	-2.7
-7	5.0	-3.8
-6	13.2	-13.5
-5	9.5	8.6
-4	4.1	-2.2
-3	2.5*	1.7
-2	8.5	7.0
-1	6.6	5.7
0	11.9	11.6
1	1.9*	0.8
2	5.5	-4.6
3	6.2	-5.7
4	9.1	8.5
5	1.4*	-0.1
(H,2,7)		
-10	5.9	-6.1
-9	6.4	6.8
-8	2.3	-2.2
-7	9.3	-8.7
-6	0.8*	1.3
-5	4.4	-4.9
-4	7.1	-5.7
-3	16.2	-15.7
-2	3.0	0.6
-1	3.0	1.0
(H,2,7)		
0	12.2	-11.8
1	20.7	20.5
2	8.0	7.1
3	1.8*	0.7
4	2.5	2.2
5	12.1	-13.6
(H,3,7)		
-10	2.3*	0.2
-9	1.1*	1.2
-8	3.0	3.3
-7	6.3	-6.0
-6	3.0	-3.3
-5	8.8	-9.8
-4	19.0	-19.8
-3	17.6	17.9
-2	10.7	11.0
-1	21.5	21.9
0	20.0	19.7
1	12.4	-12.5
2	5.3	5.6
3	11.2	-10.8
4	3.5	-3.2
5	0.8	-0.9
(H,4,7)		
-10	2.3*	-0.8
-9	9.7	10.8
-8	6.4	-6.5
-7	9.4	-9.7
-6	2.6	-2.1
-5	4.7	-4.6
-4	6.7	6.2
-3	1.3	0.2
-2	4.1	4.1
-1	6.3	-6.3
0	6.8	-5.6
1	4.3	3.7
2	4.0	-3.5
3	2.6	-2.3
4	2.5	2.2
(H,5,7)		
-10	11.3	-10.2
-9	7.0	-7.2
-8	6.2	6.8
-7	4.6	-4.4
-6	0.9*	0.2
-5	5.7	6.0
-4	1.0	-0.9
-3	8.4	8.4
-2	11.3	10.6
-1	9.8	-9.9
0	1.7*	0.5
1	9.0	8.6
2	4.8	-3.7
3	0.6*	-0.1
4	1.1	-0.5
(H,6,7)		
-10	3.3	2.5
-9	1.0*	0.9
-8	7.7	8.4
-7	7.0	7.0
-6	2.2	2.4
-5	3.4	3.8
-4	1.0	1.0
-3	1.5	-1.7
-2	14.1	-14.2
-1	8.6	-8.5
0	9.9	8.0
1	6.9	-6.3
2	1.8	-1.7
3	3.6	-3.7
4	2.2	2.6
(H,7,7)		
-10	2.7	-2.3
-9	6.8	6.6
-8	9.0	-9.4
-7	9.5	8.6
-6	4.2	4.0
-5	5.3	-5.5
-4	11.7	11.5
-3	17.0	-16.3
-2	7.9	7.5
-1	0.6*	0.3
0	14.5	-13.7
1	8.6	7.5
2	5.6	-5.6
3	6.0	5.5
(H,8,7)		
-10	5.2	5.3
-9	1.0*	-0.1
-8	3.2	3.5
-7	6.6	6.6
-6	14.1	14.8
-5	1.6	1.1
-4	10.4	-9.6
-3	3.9	-4.1
-2	4.7	-4.5
-1	3.1	-2.8
0	5.4	-4.7
1	0.7	0.1
2	3.2	3.7
(H,9,7)		
-9	0.7*	0.6
-8	4.0	-4.6
-7	5.2	4.5
-6	3.2	3.3
-5	7.0	-7.5
-4	8.3	7.6
-3	5.3	-4.9
-2	13.6	-12.8
-1	1.5	-1.8
0	4.8	3.5
1	0.9	-0.6
(H,10,7)		
-7	1.6*	-0.2
-6	0.8*	-0.5
-5	4.4	-4.8
-4	3.8	3.3
-3	5.1	-4.7
-2	7.0	6.8
-1	9.9	9.9
(H,10,7)		
0	5.5	-4.2
(H,11,7)		
-6	3.6	-3.9
-5	5.1	5.7
-4	6.1	5.8
0	5.8	-4.1
(H,12,7)		
-5	3.1	5.7
-4	12.2	10.3
(H,0,8)		
-10	9.2	-10.0
-8	9.1	8.1
-6	2.6	1.1
-4	6.9	-6.3
-2	4.4	4.2
0	2.9	3.2
2	1.2*	0.0
(H,1,8)		
-11	6.2	4.6
-10	0.9*	1.5
-9	1.2*	0.2
-8	9.2	-9.4
-7	10.3	10.1
-6	1.8	-1.9
-5	6.2	-5.0
-4	1.6*	1.0
-3	13.7	-13.9
-2	9.8	9.3
-1	2.3	1.0
0	1.7*	0.0
1	4.4	3.9
2	4.7	4.0
3	5.2	-5.1
(H,2,8)		
-11	9.0	6.6
-10	6.1	-6.3
-9	5.2	-5.6
-8	11.9	-11.4
-7	7.6	-8.1
-6	4.6	4.0
-5	9.0	-9.0
-4	16.0	15.5
-3	4.7	3.9
-2	2.7	1.6
-1	4.2	3.4
0	17.3	-15.8
1	8.8	8.9
2	10.8	10.7
3	1.2*	-1.0
4	1.9*	3.7
(H,3,8)		
-11	1.6*	0.8
-10	3.3	3.1
-9	8.4	9.0
-8	11.4	-10.5
-7	11.4	-11.6
-6	3.6	3.7
-5	3.9	3.2
-4	4.5	-4.2
-3	6.2	6.0
-2	12.8	12.7
-1	1.4	1.1
0	11.0	9.5
1	5.0	-4.7
2	4.5	-3.7
3	8.2	-7.9
(H,4,8)		
-11	2.9	2.3
-10	1.8	-0.3
-9	10.5	11.0
-8	7.9	-7.7
-7	19.9	-20.6
-6	3.9	-3.7
-5	5.7	-5.6
-4	14.1	13.9
-3	9.0	8.3
-2	1.8	1.7
-1	3.1	2.8
0	1.7*	0.4
1	8.6	-8.7
2	12.0	-10.8
(H,5,8)		
-10	6.3	-6.2
-9	0.9*	-0.4
-8	10.1	9.5
-7	0.8*	0.6
-6	1.5	-0.4
-5	1.8	-1.4
-4	5.8	5.4
-3	2.8	2.4
-2	1.1	-0.1
-1	4.2	3.8
0	9.1	7.9
1	2.7	-2.7
2	10.3	-10.2
(H,6,8)		
-9	1.9	2.3
-8	8.8	8.2
-7	9.9	10.0
-6	1.6	1.5
-5	1.6	-1.9
-4	16.6	-15.8
-3	9.8	-9.8
-2	1.7	1.7
-1	9.0	-8.4
0	11.0	9.6
1	1.9	1.8
(H,7,8)		
-9	2.6	2.7
-8	1.5*	-1.6
-7	7.1	7.6
-6	6.9	6.8
-5	10.4	-10.5
-4	3.0	3.2
-3	3.6	-3.5
-2	4.4	4.5
(H,7,8)		
-1	0.5	-0.5
0	10.8	-9.0
(H,8,8)		
-8	2.7	-0.2
-7	8.5	8.7
-6	4.4	4.6
-5	2.3*	0.9
-4	5.6	-5.0
-3	6.5	-6.2
-2	12.3	-12.7
0	3.4	2.2
(H,9,8)		
-7	4.3	-3.8
-6	3.0	-3.0
-5	2.1*	-2.1
0	1.2*	1.1
(H,10,8)		
-6	0.9	-1.3
-5	4.6	3.8
0	2.1	-2.5
(H,0,9)		
-6	2.4	-2.6
-4	3.1	2.7
-2	4.8	4.5
0	5.4	-4.7
(H,1,9)		
-9	6.3	-5.3
-8	18.1	-18.3
-7	3.1	3.6
-6	5.7	-5.6
-5	4.1	3.6
-4	10.9	11.2
-3	10.4	-10.6
-2	10.1	10.4
-1	16.2	-18.6
0	8.3	-8.9
1	7.4	16.5
(H,2,9)		
-9	9.4	-8.5
-8	3.4	3.0
-7	5.6	-5.6
-6	2.2	-1.6
-5	2.3	-2.1
-4	7.1	6.8
-3	16.4	17.9
-2	4.5	4.1
-1	1.7	-0.9
0	1.8	1.8
1	0.5*	0.3
(H,3,9)		
-9	10.2	8.2
-8	6.8	-6.7
-7	0.8*	0.1
-6	1.3	-1.2
-5	2.9	-3.0
-4	2.3	2.0
-3	9.4	-8.8
-2	9.2	8.8
-1	15.4	14.7
0	7.3	-6.7
(H,4,9)		
-8	10.6	-10.9
-7	1.5	-2.0
-6	10.9	9.1
-5	5.3	5.0
-4	3.8	-3.4
-3	0.8	0.0
-2	17.2	16.5
-1	6.5	6.2
0	10.0	-8.1
(H,5,9)		
-8	4.3	4.3
-7	0.9*	-0.2
-6	5.9	5.0
-3	4.4	3.9
0	8.3	6.6
(H,6,9)		
-7	6.0	5.6
-6	12.8	-11.1
0	12.5	10.7
(H,7,9)		
-7	3.3	3.3
-6	8.0	-6.9
0	7.8	-6.9
(H,8,9)		
-6	2.9	2.2
(H,0,10)		
0	14.4	14.6
(H,1,10)		
-7	4.8	-3.9
0	10.8	-11.3
(H,2,10)		
-7	9.3	8.5
0	3.0	3.3
(H,3,10)		
-7	5.5	4.6

Refinement of the structure of the lithium salt

Initially a block-diagonal least-squares refinement of the lithium salt was begun using parameters of the citrate ion from the sodium salt. The lithium ion parameters were then determined from a difference Fourier map. Full-matrix least-squares refinement was continued, first for the separate zones, and then for all the observed data for the three principal zones together. Extinction coefficients α were $2{\cdot}5\times10^{-5}$ for $0kl$ and $h0l$ data and 5×10^{-6} for $hk0$ data. The final R values, after correcting the data for extinction, are 8·4% for observed data only and 10·6% for all data with unobserved reflections included at half their threshold intensity values. Parameters from this refinement are given in Table 6 and are compared with those listed for the sodium salt. All atoms were considered to have isotropic temperature factors. The hydrogen atoms were included at calculated positions with temperature factors of $B=2{\cdot}5$ Å^2 and their positions were not refined. A list of calculated and observed structure factors is given in Table 7.

Discussion of the structure

The covalent bond lengths and angles for the sodium salt are given in Fig. 3, and some oxygen–oxygen and hydrogen–oxygen distances are shown in Fig. 4. For the sodium salt the standard deviations of carbon–carbon and carbon–oxygen bonds are about 0·005 Å and of angles between such bonds about 0·4°. For carbon–hydrogen and oxygen–hydrogen bonds the

Table 6. *Parameters of atoms for lithium dihydrogen citrate and a comparison with values for the sodium salt*

Positional parameters are expressed as fractions of cell edges and isotropic temperature factors in Å^2. Standard errors for the last decimal place given are listed in parentheses. The values Δx, Δy and Δz represent the differences in positional parameters between the lithium and the sodium salt.

	x	y	z	B	Δx	Δy	Δz
Li	0·0894 (23)	0·1314 (19)	0·1053 (32)	2·04 (34)	−0·0009	0·0063	0·0029
O(1)	0·3685 (10)	0·3229 (8)	0·4752 (11)	2·14 (17)	0·0136	0·0017	−0·0025
O(2)	0·2007 (10)	0·1741 (7)	0·3899 (12)	1·87 (14)	−0·0026	0·0006	−0·0193
O(3)	−0·0393 (9)	0·5532 (7)	0·7637 (12)	2·20 (16)	−0·0002	0·0045	0·0079
O(4)	0·2045 (9)	0·5759 (8)	0·9381 (10)	1·73 (14)	0·0210	0·0018	0·0053
O(5)	0·1359 (8)	0·3051 (7)	1·0129 (10)	1·08 (13)	0·0047	−0·0072	−0·0057
O(6)	0·3885 (10)	0·3389 (7)	1·1408 (10)	1·51 (16)	0·0248	−0·0106	−0·0006
O(7)	0·4229 (8)	0·4169 (7)	0·8305 (11)	1·44 (13)	0·0224	−0·0028	0·0084
C(1)	0·2763 (13)	0·2415 (17)	0·5027 (14)	1·50 (18)	0·0049	−0·0001	−0·0158
C(2)	0·2764 (14)	0·2339 (12)	0·7100 (13)	1·45 (20)	0·0039	−0·0066	−0·0118
C(3)	0·2796 (12)	0·3555 (10)	0·8063 (14)	1·39 (18)	0·0105	−0·0039	−0·0024
C(4)	0·1461 (11)	0·4321 (9)	0·6951 (13)	1·35 (16)	0·0068	0·0043	−0·0028
C(5)	0·1091 (11)	0·5285 (9)	0·8166 (14)	1·10 (17)	0·0107	0·0039	0·0087
C(6)	0·2680 (14)	0·3337 (10)	1·0056 (19)	1·23 (19)	0·0136	−0·0053	−0·0027

standard deviations are about 0·08 Å. For the lithium salt standard deviations of carbon–carbon and carbon–oxygen bonds are about 0·015 Å.

Of the three carboxyl groups in the sodium salt, the two end carboxyls have carbon–oxygen distances which are non-equivalent (1·203, 1·321 and 1·216, 1·313 Å), while the central carboxyl has two equivalent carbon–oxygen distances (1·253, 1·252 Å). The corrections to be applied to these distances on the assumption that the carboxyl oxygens are 'riding' on the adjacent carbon atoms (Busing & Levy, 1964) are small. (The corrected carbon–oxygen distances are 1·336, 1·225, 1·329, 1·226, 1·257, 1·263, and 1·436 Å for O(1) to O(7) in order.) These facts, together with the locations of hydrogen atoms attached to O(1) and O(3), demonstrate that it is the central carboxyl group which is ionized in this crystalline form of sodium dihydrogen citrate. (A preliminary report of this result has been given by Glusker, van der Helm, Love, Dornberg & Patterson, 1960.) For the lithium salt the correspond-

Table 7. *Observed and calculated structure factors for lithium dihydrogen citrate*

The observed values given are corrected for extinction (see text). An asterisk indicates that the reflection was too weak for measurement. The value listed corresponds to an intensity of half the threshold intensity of the technique of measurement used.

OKL PROJECTION

L	OBS	CALC
(0,0,L)		
1	53.8	45.1
2	5.1	3.7
3	25.0	-27.4
4	26.5	27.1
5	12.8	13.7
6	27.8	29.8
7	6.7	7.0
8	5.2	3.5
9	12.9	-10.1
10	11.9	13.3
(0,1,L)		
1	2.1*	-0.8
2	10.1	-9.4
3	64.0	-62.6
4	3.9	3.4
5	8.7	-9.0
6	7.9	-6.7
7	8.5	6.8
8	2.3*	-0.1
9	17.0	-15.4
10	2.8	-5.2
(0,2,L)		
0	42.5	-41.8
1	34.5	37.0
2	79.9	-68.5
3	15.0	-15.2
4	15.7	-16.3
5	7.9	6.0
6	16.1	-14.9
7	11.7	-10.0
8	20.9	-19.6
9	2.3*	-0.9
(0,3,L)		
1	11.0	-12.7
2	14.9	-16.5
3	2.8*	1.8
4	18.0	-18.9
5	23.4	-23.2
6	2.7*	-2.4
7	12.7	11.5
8	6.1	5.5
9	2.2*	-0.7
(0,4,L)		
0	27.5	-26.9
1	32.7	-34.0
2	4.6	-6.4
3	10.5	9.8
4	25.2	26.9
5	10.1	-10.0
6	2.2*	-0.5
7	5.9	-2.9
8	2.3*	0.8
9	2.1*	-4.0
(0,5,L)		
1	15.7	17.7
2	21.3	-21.7
3	25.0	24.2
4	5.3	6.2
5	15.6	-16.3
6	27.6	-26.3
7	2.3*	0.2
8	13.8	11.1
9	7.7	6.1
(0,6,L)		
0	10.0	10.1
1	2.3*	-1.3
2	25.6	30.3
3	2.4*	-2.3
4	9.6	-9.8
5	26.4	26.1
6	7.8	8.3
7	8.1	8.3
8	16.5	14.3
9	12.1	10.5
(0,7,L)		
1	36.3	-44.3
2	2.5*	-0.4
3	2.5*	-3.1
4	18.1	17.5
5	2.2*	0.2
6	3.2*	-1.6
7	8.1	-6.8
8	6.1	-7.5
9	5.1	-5.8

OKL PROJECTION

L	OBS	CALC
(0,8,L)		
0	37.7	-45.2
1	13.2	-12.6
2	12.7	-12.7
3	16.4	-15.9
4	8.9	-8.2
5	13.4	-13.0
6	5.2	-2.8
7	6.0	-6.1
8	5.1	-3.0
(0,9,L)		
1	5.4	-6.6
2	11.9	-11.7
3	10.4	9.4
4	12.5	11.0
5	17.2	17.8
6	5.6	7.3
7	2.2*	-0.3
8	2.0*	-1.5
(0,10,L)		
0	18.3	-20.7
1	5.2	5.5
2	14.6	15.2
3	12.4	12.1
4	2.8*	-2.6
5	5.5	6.3
6	2.7*	0.1
7	2.5*	3.1
(0,11,L)		
1	5.9	-5.3
2	2.8*	1.3
3	13.3	12.7
4	2.8*	2.0
5	5.3	4.0
6	2.6*	0.7
7	12.0	-10.5
(0,12,L)		
0	21.4	23.7
1	2.8*	-2.5
2	8.8	9.0
3	2.3*	-3.9
4	2.7*	-2.4
5	6.6	-4.3
6	6.2	7.8
(0,13,L)		
1	5.5	-5.6
2	2.2*	4.0
3	4.5	-4.7
4	5.6	5.2
5	5.2	4.4
(0,14,L)		
0	3.7*	-2.3
1	7.5	9.8
2	7.9	-9.4
3	2.0*	2.7
4	4.7	-3.9
(0,15,L)		
1	2.7*	6.8
2	2.5*	-1.5
3	4.4	-3.1

HOL PROJECTION

L	OBS	CALC
(0,0,L)		
1	41.5	45.1
2	5.1	3.7
3	26.6	-27.4
4	28.3	27.1
5	14.2	13.7
6	28.0	29.8
7	6.4	7.0
8	5.5	3.5
9	9.7	-10.1
(2,0,L)		
-9	7.6	9.0
-8	6.3	5.2
-7	13.8	-10.9
-6	15.6	13.7
-5	3.7	4.0
-4	19.7	-20.9
-3	53.0	-57.0
-2	56.5	-62.6
-1	7.4	7.3
0	76.2	-76.2
1	12.8	12.6
2	40.6	-45.2
3	19.5	-19.5
4	6.8	-7.7
5	2.4*	-0.4
6	24.1	-22.7
7	12.7	10.8
8	5.5	-3.7
(4,0,L)		
-9	1.7*	-1.6
-8	2.3*	-3.4
-7	9.9	-9.8
-6	25.1	-25.0
-5	21.0	20.2
-4	25.4	27.9
-3	18.8	18.6
-2	23.6	24.3
-1	2.5	0.8
0	27.7	-27.4
1	24.5	-24.5
2	33.0	-34.8
3	19.9	19.4
4	2.5	2.1
5	4.9	-7.0
6	12.9	-11.7
7	12.6	-12.8
(6,0,L)		
-9	5.1	1.6
-8	2.1*	0.4
-7	2.4*	-3.5
-6	28.1	-27.4
-5	16.9	-18.2
-4	28.3	-28.0
-3	12.2	12.1
-2	2.2*	1.7
-1	32.6	31.3
0	25.6	-24.6
1	54.0	-57.8
2	12.1	13.2
3	31.4	32.5
4	23.5	24.1
5	3.8	5.2
6	1.5*	-2.5
(8,0,L)		
-8	7.4	5.0
-7	7.6	8.6
-6	31.0	31.6
-5	6.3	5.9
-4	19.2	19.8
-3	2.5*	1.4
-2	25.6	27.1
-1	6.0	-4.2
0	13.5	12.5
1	11.3	11.0
2	2.4*	-2.0
3	2.1*	-3.0
4	9.3	10.2
(10,0,L)		
-7	11.0	-11.6
-6	3.5	1.1
-5	11.4	11.9
-4	13.6	-12.8
-3	23.6	-23.1
-2	9.5	-9.8
-1	2.1*	1.8
0	6.1	5.9
1	5.7	4.4
2	6.3	5.8

HKO PROJECTION

K	OBS	CALC
(0,K,0)		
2	43.8	-41.8
4	33.4	-26.9
6	11.3	10.1
8	44.1	-45.2
10	21.4	-20.7
12	23.2	23.7
14	3.6*	-2.3
(1,K,0)		
1	18.3	-16.9
2	53.2	56.9
3	67.9	-66.2
4	32.1	-32.0
5	8.2	-8.1
6	6.4	-7.0
7	27.1	25.8
8	20.1	18.5
9	3.6*	-4.2
10	18.0	-19.5
11	11.4	12.5
12	3.1*	1.5
13	8.6	9.8
14	8.0	-6.1
15	4.5	-5.8
(2,K,0)		
0	72.7	-76.2
1	32.9	34.4
2	72.4	73.2
3	15.6	13.4
4	3.1*	-4.0
5	8.9	-7.6
6	12.5	-9.7
7	3.3*	-0.1
8	3.4*	-2.7
9	10.4	9.3
10	10.9	9.0
11	11.2	-12.0
12	16.9	-15.8
13	14.6	16.5
14	3.7*	1.4
15	8.3	10.0
(3,K,0)		
1	2.2*	0.2
2	65.7	63.9
3	42.1	39.9
4	5.5	2.7
5	15.1	-13.9
6	30.6	30.2
7	31.7	-32.0
8	23.4	-21.9
9	19.1	19.5
10	22.8	-24.9
11	4.3	-1.2
12	6.9	-5.3
13	3.0*	1.5
14	4.6	4.3
15	6.1	7.7
(4,K,0)		
0	29.1	-27.4
1	7.5	-6.1
2	3.6*	-2.6
3	2.6*	-0.8
4	9.1	11.2
5	14.0	-13.8
6	26.0	-24.3
7	15.5	12.7
8	20.0	19.5
9	17.0	-15.2
10	13.3	-14.0
11	8.2	6.0
12	7.6	4.1
13	6.2	7.5
14	3.7*	1.5
15	1.9*	2.1
(5,K,0)		
1	18.2	-16.7
2	2.7*	-3.9
3	3.1*	-0.1
4	28.1	30.4
5	22.5	22.6
6	11.8	-11.3
7	19.4	17.5
8	7.7	9.0
9	4.0*	-2.1
10	3.8*	2.2
11	4.3*	0.5
12	3.7*	-1.8
13	12.8	-13.5
14	3.7*	3.0

HKO PROJECTION

K	OBS	CALC
(6,K,0)		
0	25.2	-24.6
1	8.6	7.5
2	4.2*	2.3
3	5.7	-4.2
4	4.9	6.7
5	15.6	15.6
6	3.2*	2.0
7	25.4	-26.5
8	10.1	-11.9
9	5.6	-6.4
10	7.8	7.7
11	6.1	-5.1
12	5.5	-6.7
13	4.2*	-7.0
(7,K,0)		
1	14.8	-14.5
2	14.5	-15.9
3	14.7	14.1
4	24.3	26.0
5	4.5*	0.6
6	5.2	7.8
7	5.3*	-3.1
8	4.6*	-3.7
9	9.2	8.4
10	17.5	18.2
11	5.4	-6.5
12	3.7*	-1.6
13	2.2*	2.5
(8,K,0)		
0	12.2	12.5
1	27.4	-27.5
2	14.7	-14.9
3	11.0	-11.2
4	4.3	4.9
5	18.1	-17.7
6	16.5	17.2
7	4.7*	-2.2
8	4.1*	-1.9
9	4.0*	-2.5
10	3.6*	-2.3
11	12.7	12.4
12	1.9*	-1.1
(9,K,0)		
1	7.6	4.6
2	19.3	20.3
3	15.5	-17.4
4	7.1	-9.2
5	3.9*	0.5
6	11.0	11.9
7	13.1	13.0
8	4.7	6.3
9	14.8	-15.2
10	8.9	-9.8
(10,K,0)		
0	6.0	5.9
1	13.2	14.0
2	3.9*	0.7
3	9.3	-8.4
4	4.2*	-3.4
5	8.9	-7.3
6	3.4*	4.0
7	4.0*	1.2
8	7.2	5.3
9	16.1	14.8
(11,K,0)		
1	6.8	4.8
2	5.9	-6.0
3	3.7*	2.1
4	9.1	-9.7
5	4.3*	-0.5
6	6.3	-6.7
7	9.0	-14.0
(12,K,0)		
0	9.5	-15.2
1	3.0*	-6.0
2	2.7*	4.9
3	3.7	6.3

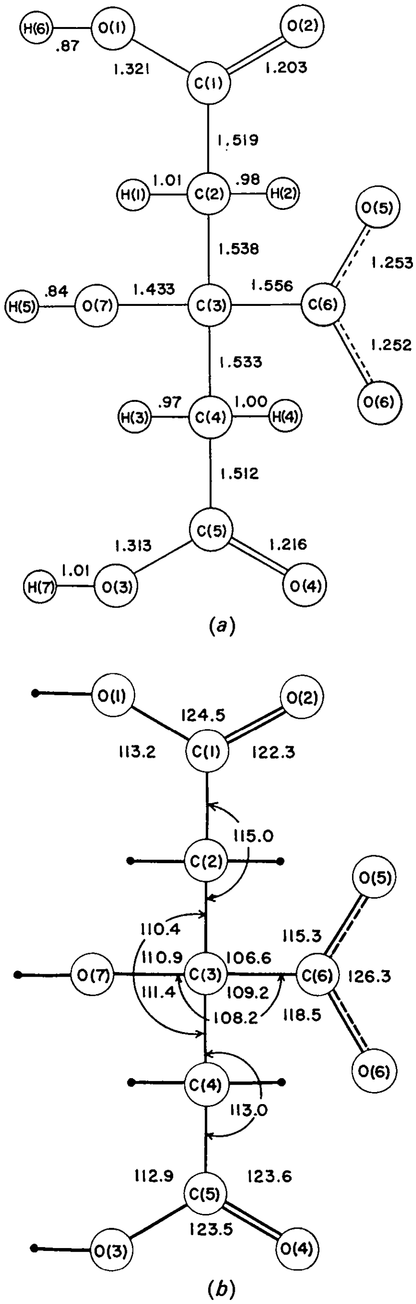

Fig. 3. (*a*) Covalent bond lengths and (*b*) angles for the dihydrogen citrate ion in the sodium salt (Å). The 'riding' correction has not been applied to these figures.

ing carbon–oxygen distances are 1·19, 1·31 and 1·18, 1·32 Å for the end carboxyl groups and 1·27, 1·25 Å for the central carboxyl group.

Nuclear magnetic resonance measurements (Loewenstein & Roberts, 1960) indicate that in solution the singly ionized citrate is predominantly ionized on the end carboxyl groups. Martin (1961) deduced from titration studies that in solution 60% of the singly ionized citrate is ionized on the central carboxyl group and 40% on the two terminal groups. He also suggested that the nuclear magnetic resonance data may be reinterpreted to agree with these results by the use of more accurate *p*K values. We do not, however, wish to infer that the species which crystallizes is necessarily the one present in greatest concentration in solution.

The carbon–carbon bond of the central carboxyl group is long (1·556 Å). This same bond is 1·555 Å in the triionized citrate ion (Johnson, 1965). Long bonds have been reported in ammonium oxamate (Beagley & Small, 1963) and lithium oxalate (Beagley & Small, 1964). The short distance O(7)–O(6) (2·638 Å) is characteristic of the citrates (Nordman, Weldon & Patterson, 1960*a*, 1960*b*; Johnson, 1965; Glusker, Patterson, Love & Dornberg, 1963). The hydroxyl group lies 0·387 Å from the plane of the central carboxyl group (see Table 8). The carbon–carbon bonds adjacent to the terminal carboxyl groups are shorter (average 1·516 Å) than those adjacent to the central saturated carbon atom (average 1·536 Å), a difference of 4σ. All covalent bond distances for the citrate ion in the lithium salt agree (within 2·5σ for the lithium salt) with those found for the ion in the sodium salt.

Table 8 shows some least-squares planes calculated through the molecule with the deviations of some atoms from these planes. The hydrogen atom H(1) lies very close to the plane defined by C(2)C(3)C(4). The carbon atom C(5) lies 0·489 Å above and C(1) 1·136 Å below this plane.

Some data on hydrogen bonds are given in Table 9. An internal hydrogen bond is formed from the hydroxyl group O(7) to one of the end carboxyl oxygen atoms, O(1), through the hydrogen, H(5), of the hydroxyl group. The oxygen–hydrogen–oxygen angle is 133°. The oxygens of the ionized carboxyl group, O(5) and O(6), are hydrogen bonded to adjacent molecules (as shown in Fig. 4). The oxygen atom O(6) lies 2·562 Å from O(1″) which lies almost in the plane of the carboxyl group C(3)C(6)O(5)O(6) (see Table 8). The oxygen atom O(5) is hydrogen bonded to O(3′) which does not lie in this plane. This oxygen–oxygen distance is 2·660 Å.

The octahedral coordination of the sodium ion is shown in Fig. 5. Some distances and angles for the sodium and lithium salts are given in Table 10. The changes in cell dimensions are fully accounted for by the difference in sizes of the coordination octahedra (see Table 10, part 3). The octahedra occur in centrosymmetrically related pairs with one edge shared. Two bidentate chelate rings are formed. One is five-membered, MO(6)C(6)C(3)O(7), subtending an angle of 67·3° at the sodium ion and distorting the octahedron as a result of the short distance between O(6) and O(7). The other is six-membered, MO(4)C(5)C(4)C(3)O(7), subtending an angle of 78·5° at the sodium ion. The six sodium–oxygen distances are very similar and range from 2·305–2·461 Å. Of the lithium–oxygen distances, five lie between 1·96 and 2·22 Å, and one lithium–oxygen distance is 2·49 Å. The oxygen atoms O(1) and

Table 8. *Equations of some planes in the citrate ion and perpendicular deviations of atoms from these planes*

The equations are expressed in the form:

$$lX+mY+nZ=D$$

where distances are expressed in Å. X, Y, Z are coordinates in the orthonormal E system, using the γ matrix given in the text. The deviations, Δ, from these planes, are also expressed in Å.

Designation of plane	Atoms in plane	l	m	n	D
(1)	O(1), O(2), C(1), C(2)	0·76494	−0·63384	0·11448	0·17308
(2)	O(3), O(4), C(4), C(5)	−0·27908	−0·66134	0·69624	0·02584
(3)	O(5), O(6), C(3), C(6)	−0·23168	0·96764	0·09995	4·24336
(4)	O(7), C(3), C(6)	−0·49128	0·87080	0·01867	2·93981
(5)	C(2), C(3), C(4)	−0·65720	−0·36803	0·65775	1·09119
(6)	C(1), C(2), C(3), C(6)	0·98054	−0·00949	0·19608	2·68621

Atoms	Δ(1)	Atoms	Δ(2)	Atoms	Δ(3)	Atoms	Δ(4)	Atoms	Δ(5)	Atoms	Δ(6)
O(1)	0·003	O(3)	0·001	O(5)	−0·001	O(7)	0·000	C(2)	0·000	C(1)	−0·038
O(2)	0·004	O(4)	0·001	O(6)	−0·001	C(3)	0·000	C(3)	0·000	C(2)	0·043
C(1)	−0·009	C(5)	−0·004	C(6)	0·003	C(6)	0·000	C(4)	0·000	C(3)	0·029
C(2)	0·003	C(4)	0·001	C(3)	−0·001					C(6)	0·035
H(6)	−0·190	C(1)	0·003	O(7)	0·387	H(5)	−0·177	C(1)	−1·136		
		H(7)	0·038	H(5)	0·232			C(5)	0·489		
						O(5)	0·319				
				H(7′)	1·160	O(6)	−0·319	O(1)	−2·296		
				O(3′)	2·011			O(2)	−0·976		
								O(3)	1·063		
				H(6″)	0·041			O(4)	0·376		
				O(1″)	0·036						
								H(1)	−0·087		
								H(2)	0·852		
								H(3)	0·572		
								H(4)	−0·911		

Table 9. *Hydrogen bonds in sodium dihydrogen citrate*

Donor (O_d)	Hydrogen	Acceptor (O_a)	$O_d\cdots O_a$ (Å)	$H-O_d$ (Å)	$H\cdots O_a$ (Å)	$\angle O_a\cdots H-O_d$ (degrees)	$\angle H-O_d\cdots O_a$ (degrees)	$\angle H-O_d-C$ (degrees)
O(7)	H(5)	O(1)	2·750	0·84	2·11	133	34	112
O(1)	H(6)	O(6′)	2·562	0·87	1·73	159	14	109
O(3)	H(7)	O(5″)	2·660	1·01	1·67	167	9	116

O(6′) at $x, y, z-1$

O(5″) at $-x, 1-y, 2-z$

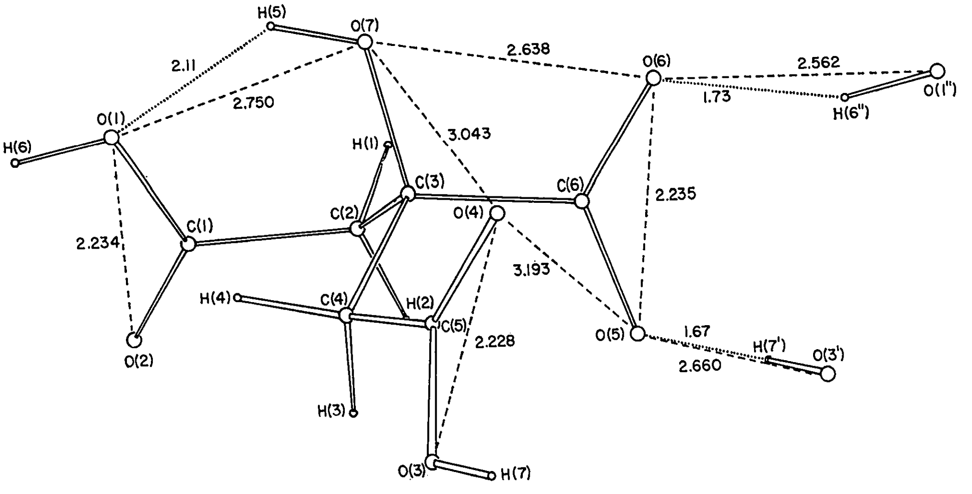

Fig. 4. Some oxygen–oxygen and hydrogen–oxygen distances (Å) in sodium dihydrogen citrate.

O(3), of the terminal carboxyl groups, do not take part in the packing around the metal.

Computations

The reduction of the original data and the first attempts at a solution of the structure were carried out on an IBM 602A computer.

The three-dimensional $|F|^2$ map was calculated on the IBM 650 computer at Princeton University through the courtesy of Dr U. van Wijk.

The first three-dimensional least-squares refinements and the difference Fourier synthesis used to locate hydrogen atoms were computed on the IBM 650 computer at the University of Indiana. We are indebted to Professor Lynne L. Merritt, Jr. for the use of this computer.

The full-matrix least-squares refinement of the lithium dihydrogen citrate data was made on a CDC 1604 computer at the Oak Ridge National Laboratory, using a modification of the ORFLS program by Dr W. R. Busing, Mrs K. O. Martin and Dr H. A. Levy. Fig. 2 was plotted at Oak Ridge National Laboratory with the program ORTEP (Johnson, 1964). We are indebted to Oak Ridge National Laboratory for these uses of the computer.

The final refinement of the sodium salt and all other calculations were made on an IBM 1620 computer installed in this laboratory. (For a complete list of programs see Johnson, 1965.)

We are grateful to Miss Evelyn Wydro and Miss Helen Loughran for assistance in the preparation of data tapes.

Table 10. *Some angles and distances in the octahedra of oxygen atoms surrounding the metal ions*

With respect to the coordinates in Table 3 the oxygens and sodium ions of the octahedra in Figs. 2 and 5, with a center of symmetry at $\frac{1}{2}, \frac{1}{2}, 0$, are

Na′	$\frac{1}{2}+x, \frac{1}{2}-y, z$
O(4), O(7)	$x, y, z-1$
O(6′), O(7′)	$1-x, 1-y, 1-z$
O(2″), Na	$\frac{1}{2}-x, \frac{1}{2}+y, -z$
O(5‴)	$\frac{1}{2}-x, \frac{1}{2}+y, 1-z$

(1) Sodium—oxygen and lithium—oxygen distances (Å)

	Na	Li
O(6′)	2·305	1·96
O(2″)	2·336	2·12
O(5‴)	2·337	2·17
O(4)	2·348	2·10
O(7′)	2·451	2·22
O(7)	2·461	2·49

(2) Metal ion—metal ion distances (Å)

Na · · · · Na	3·539
Li · · · · Li	3·53

(3) Oxygen–oxygen distances (Å)

Sodium salt

	O(7′)	O(7)	O(4)	O(2″)	O(5‴)
O(6′)	2·638	3·495	4·643	3·374	3·516
O(5‴)	3·570	4·769	3·277	3·253	
O(2″)	4·718	3·446	3·253		
O(4)	3·955	3·043			
O(7)	3·406				

Lithium salt

	O(7′)	O(7)	O(4)	O(2″)	O(5‴)
O(6′)	2·59	3·24	4·06	2·90	3·17
O(5‴)	3·22	4·63	2·95	3·08	
O(2″)	4·30	3·37	3·05		
O(4)	3·32	2·98			
O(7)	3·13				

(4) Oxygen–sodium–oxygen angles (°)

O(6′) –Na–O(7′)	67·3
O(7) –Na–O(4)	78·5
O(7) –Na–O(7′)	87·8
O(4) –Na–O(2″)	88·0
O(5‴)–Na–O(2″)	88·2
O(5‴)–Na–O(4)	88·8
O(7) –Na–O(2″)	91·8
O(2″) –Na–O(6′)	93·3
O(7) –Na–O(6′)	94·3
O(5‴)–Na–O(7′)	96·4
O(5‴)–Na–O(6′)	98·5
O(7′) –Na–O(4)	111·0
O(7′) –Na–O(2″)	160·5
O(7) –Na–O(5‴)	167·2
O(6′) –Na–O(4)	172·7

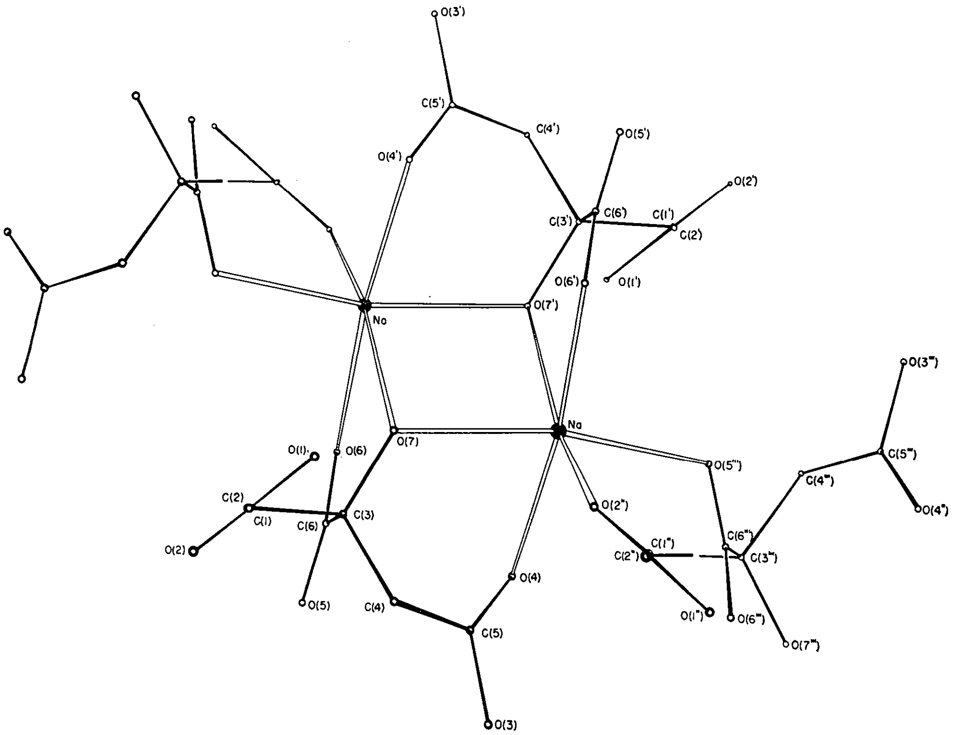

Fig. 5. Octahedral coordination of oxygen atoms around metal ions. (The projection down the *c* axis with the *b* axis horizontal).

References

Beagley, B. & Small, R. W. H. (1963). *Proc. Roy. Soc.* A, **275**, 469.

Beagley, B. & Small, R. W. H. (1964). *Acta Cryst.* **17**, 783.

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.

Bond, W. L. (1951). *Rev. Sci. Instrum.* **22**, 344.

Busing, W. R. & Levy, H. A. (1964). *Acta Cryst.* **17**, 142.

Glusker, J. P., van der Helm, D., Love, W. E., Dornberg, M. L. & Patterson, A. L. (1960). *J. Amer. Chem. Soc.* **82**, 2964.

Glusker, J. P., Patterson, A. L., Love, W. E. & Dornberg, M. L. (1963). *Acta Cryst.* **16**, 1102.

Ibers, J. A. (1962). *International Tables for X-ray Crystallography*, Vol. III, p. 201. Birmingham: Kynoch Press.

Johnson, C. K. (1964). Paper A-11, Amer. Cryst. Assn. Meeting, Montana State College, Bozeman, Mont., July 26–31.

Johnson, C. K. (1965). *Acta Cryst.* **18**, 1004.

Loewenstein, A. & Roberts, J. D. (1960). *J. Amer. Chem. Soc.* **82**, 2705.

Love, W. E. & Patterson, A. L. (1960). *Acta Cryst.* **13**, 426.

Martin, R. B. (1961). *J. Phys. Chem.* **65**, 2053.

Nordman, C. E., Weldon, A. S. & Patterson, A. L. (1960*a*). *Acta Cryst.* **13**, 414.

Nordman, C. E., Weldon, A. S. & Patterson, A. L. (1960*b*). *Acta Cryst.* **13**, 418.

Patterson, A. L. (1952). *Acta Cryst.* **5**, 829.

Templeton, D. H. (1962). *International Tables for X-ray Crystallography*, Vol. III, p. 213. Birmingham: Kynoch Press.

Zachariasen, W. H. (1963*a*). *Acta Cryst.* **16**, 1139.

Zachariasen, W. H. (1963*b*). Paper G-2, Amer. Cryst. Assn. Meeting, M.I.T., Cambridge, Mass., March 28–30.