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The Crystal and Molecular Structure of Diaquo Zinc Glycylglycylglycinato Hemisulfate Dihydrate*

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The crystal structure of diaquo zinc glycylglycylglycinato hemisulfate dihydrate,

$$Zn(N_3C_6O_4H_{10})\,(H_2O)_4\,(SO_4)_{1/2},$$

has been determined and refined by three-dimensional least-squares techniques, using 2095 counter data. The crystals are orthorhombic, space group *Pbcn*, with $a=25\cdot86$, $b=8\cdot011$ and $c=13\cdot59$ Å. The final R value for 2095 reflections is 0·065. The standard deviations are about 0·005 Å for the light atom positions. The zinc surrounding is better described as a trigonal bipyramid than as a square pyramid. The peptide molecules form infinite chains held together by zinc ions.

Introduction

The investigation of the structure of the zinc complex of glycylglycylglycine prepared at neutral pH was undertaken as a part of a project designed to elucidate the factors which determine the bonding geometry of metal ions in biological systems. The compound was interesting to us because it offered the chance to compare the zinc surrounding with that of a similar copper compound, glycylglycylglycinato copper(II) chloride sesquihydrate (Freeman, Robinson & Schoone, 1964). Similar comparisons of the bonding geometry of copper(II) and zinc in complexes with biological ligands had either yielded closely similar structures in the case of glutamic acid (Gramaccioli, 1966; Gramaccioli & Marsh, 1966) or, with L-serine, structures with small but distinct differences (Van der Helm & Franks, 1969; Van der Helm, Nicholas & Fisher, 1970). In addition metal replacement studies show that copper(II) often inactivates zinc enzymes (Vallee, Riordan & Colman, 1964; Linskog & Nyman, 1964; Plocke & Vallee, 1962).

Experimental

A solution of the zinc complex with glycylglycylglycine can be prepared by dissolving equimolar quantities of the peptide and zinc sulfate in distilled water. The excess sulfate is precipitated by the addition of a half molar quantity of barium hydroxide. Crystals of diaquozinc glycylglycylglycinato hemisulfate dihydrate appear within 24 hours when a layer of ethanol is placed on top of a 0·1 M solution of the peptide chelate. The crystals are long boat-shaped plates. The plate face is perpendicular to the a axis and the direction of elongation coincides with the b axis. The reciprocal space symmetry shows the space group to be orthorhombic; the systematic absence of reflections $0kl$, for $k=2n+1$, $h0l$, for $l=2n+1$, and $hk0$ for $h+k=2n+1$ uniquely determines the space group *Pbcn*.

The cell dimensions were fitted by least-squares to the 2θ values, observed at room temperature, of 34 reflections sampled from four octants of reciprocal space. The cell dimensions with standard deviations are $a=25\cdot86\pm0\cdot03$, $b=8\cdot011\pm0\cdot006$ and $c=13\cdot59\pm0\cdot01$ Å. The formula weight for $Zn(N_3C_6O_4H_{10})(H_2O)_4(SO_4)_{1/2}$ is 373·65, yielding $\varrho_c=1\cdot762$ g.cm^{-3}, with $Z=8$, while $\varrho_o=1\cdot763$ g.cm^{-3}, measured by the flotation method. The integrated intensities were taken a General Electric XRD-5 diffraction unit using the θ–2θ scan technique and Ni-filtered Cu $K\alpha$ radiation. The diffraction unit was equipped with an SPG Single Crystal Orienter, a scintillation counter and pulse-height analyzer. The crystal dimensions were $0\cdot3\times0\cdot2\times0\cdot01$ mm. All reflections with a 2θ value below 120° were measured. Of the 2095 independent reflections within this limit, no peak could be distinguished from the background for 317 and these were therefore considered to be unobserved. The intensity recorded for each unobserved reflection was one-tenth of the total background intensity measured at its location. Lorentz, polarization, and absorption ($\mu=37\cdot1$ cm^{-1}, using the actual shape of the data crystal) corrections were applied to the data.

Solution and refinement of the structure

A three-dimensional sharpened Patterson synthesis was calculated. Because the y coordinate of the zinc atom is close to $\frac{1}{2}$, two possible positions for this atom

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were found. This ambiguity could have been resolved with the use of Zn–S vectors but the presence of the sulfate ion in the structure was initially not known. Using three-dimensional Fourier maps and the heavy-atom technique two trial structures were pursued until it became possible to choose one as the correct structure. At the same time the presence of the sulfate ion was recognized and a check of the Zn–S vectors in the Patterson synthesis showed unambiguously that the chosen trial structure was correct. In addition four molecules of water were located. A structure factor calculation of the trial structure in which all non-hydrogen atoms were included yielded an $R(=\sum||kF_o|-|F_c||/\sum|kF_o|)$ of 0·23.

The structure was refined by block-diagonal least-squares computations initially using individual isotropic temperature factors and later anisotropic parameters. When all shifts were smaller than the standard deviations a difference Fourier map was calculated using only those structure factors with $\sin^2\theta<0{\cdot}45$. It was possible to locate 14 of the 18 hydrogen atoms from this map. These hydrogens were included in subsequent structure factor computations and were assigned isotropic temperature factors which were 0·5 Å^2 larger than the atoms to which they are bonded. The parameters of the hydrogen atoms were not changed in the further refinement. A second difference map revealed the approximate locations of the remaining 4 hydrogen atoms. At the same time the observed amplitudes were corrected for anomalous dispersion (Patterson, 1963). The least-squares refinement was continued until the coordinate shifts were less than $0{\cdot}1\sigma$ and the temperature parameter shifts were smaller than $0{\cdot}3\sigma$.

The R value for all 2095 amplitudes is 0·065. In a final Fourier map the Zn atom has a peak height of 60 e.Å^{-3} while the maximum electron densities at the hydrogen atom locations vary between 0·7 and 1·4 e.Å^{-3}. A final difference map, calculated from a structure factor computation without hydrogen atom contributions, shows no spurious peaks.

The final weights were $\sqrt{w}=|kF_o|/P$ if $|kF_o|\le P$, and $\sqrt{w}=P/|kF_o|$) if $P<|kF_o|$, with $P=29$ electrons. The scale factor k is a parameter in the least-squares refinement and brings the observed amplitudes on the absolute scale as determined by the calculated amplitudes. The quantity minimized in the least-squares calculations was $\sum w(|kF_o|-|F_c|)^2$. As with other structures determined in this laboratory, a logical routine was used to decide which structure amplitudes should be used in difference Fouriers or least-squares computations. An amplitude was excluded from either of these calculations when one of the following conditions prevailed:

$$|\Delta F|/|kF_o|<2, \text{ for } |kF_o|<F_{\min};$$

$$1{\cdot}5\ |F_c|<|kF_o| \text{ for } F_{\min}<|kF_o|<2F_{\min};$$

$$1{\cdot}8\ |F_c|<|kF_o| \text{ for } 2F_{\min}<|kF_o|<3F_{\min};$$

$$2{\cdot}0\ |F_c|<|kF_o| \text{ for } 3F_{\min}<|kF_o|<4F_{\min};$$

$$2{\cdot}5\ |F_c|<|kF_o| \text{ for } 4F_{\min}<|kF_o|; \text{ and}$$

$$|F_c|-|kF_o|>12{\cdot}5 \text{ while } \sin^2\theta/\lambda^2<0{\cdot}012\,.$$

The value of 5·0 was used for $F_{\min}$, which is close to the amplitude value of those observed reflections with the lowest intensities. The first condition results in the fact that the unobserved reflections are included only when $|F_c|$ is more than twice as large as the assigned $|kF_o|$ value, while the last condition is an attempt to exclude reflections which are affected by extinction. This condition did not have to be used for the present data set. The other conditions were all used to exclude reflections from a least-squares computation during the initial stages of the refinement. In the least-squares computation 344 reflections were excluded: 310 of the 317 unobserved reflections as a result of the first condition and another 34 due to the second condition.

The final parameters are given in Tables 1 and 2. The atomic scattering factors used were from *International Tables for X-ray Crystallography* (1962), for Zn^{2+}, O, N, C, O$^-$ [for O(4)] and $(\mathrm{O}+\mathrm{O}^-)/2$ [for O(5) and O(6)]. The scattering factors used for the hydrogen atoms were those of Stewart, Davidson & Simpson (1965). A list of the observed and calculated structure factors is given in Table 3.

Table 1. *Atomic positions*

Standard deviations for last digit are given in parentheses.

	x	*y*	*z*	
Zn	0·37186 (2)	0·49433 (9)	0·06174 (4)	
N(1)	0·42408 (16)	0·3213 (5)	0·0155 (3)	
C(1)	0·40092 (21)	0·1534 (7)	0·0028 (4)	
C(2)	0·35280 (17)	0·1378 (6)	0·0645 (3)	
O(1)	0·33002 (12)	0·2639 (4)	0·0950 (2)	
N(2)	0·33577 (15)	−0·0158 (5)	0·0775 (3)	
C(3)	0·28629 (19)	−0·0512 (6)	0·1229 (4)	
C(4)	0·28811 (17)	−0·0767 (6)	0·2329 (3)	
O(2)	0·32249 (14)	−0·0229 (5)	0·2851 (3)	
N(3)	0·24725 (17)	−0·1589 (5)	0·2684 (3)	
C(5)	0·23857 (18)	−0·1875 (7)	0·3727 (4)	
C(6)	0·19473 (18)	−0·0911 (6)	0·4184 (5)	
O(3)	0·16721 (16)	0·0015 (6)	0·3711 (3)	
O(4)	0·18912 (12)	−0·1116 (4)	0·5103 (2)	
W(1)	0·37096 (15)	0·5742 (7)	0·1996 (3)	
W(2)	0·41891 (13)	0·7054 (5)	0·0302 (3)	
W(3)	0·47527 (13)	0·3182 (6)	0·3672 (3)	
W(4)	0·42189 (18)	0·1154 (5)	0·2426 (4)	
S	0·50000	0·7412 (3)	0·2500	
O(5)	0·46026 (17)	0·6379 (6)	0·2953 (3)	
O(6)	0·47674 (17)	0·8443 (6)	0·1738 (3)	
H(1)N(1)	0·433	0·343	−0·044	3·85 Å^2
H(2)N(1)	0·444	0·310	0·059	3·85
H(1)C(1)	0·429	0·065	0·039	4·37
H(2)C(1)	0·387	0·160	−0·067	4·37
H N(2)	0·355	−0·106	0·052	3·37
H(1)C(3)	0·264	0·040	0·113	3·47
H(2)C(3)	0·274	−0·140	0·092	3·47
H N(3)	0·225	−0·190	0·225	3·64
H(1)C(5)	0·267	−0·160	0·408	3·70
H(2)C(5)	0·226	−0·300	0·385	3·70
H(1)*W*(1)	0·395	0·620	0·220	4·51
H(2)*W*(1)	0·353	0·550	0·255	4·51

Table 1 (*cont.*)

	x	*y*	*z*	*B*
H(1)*W*(2)	0·435	0·690	−0·030	4·07
H(2)*W*(2)	0·443	0·740	0·063	4·07
H(1)*W*(3)	0·444	0·275	0·334	5·04
H(2)*W*(3)	0·485	0·485	0·337	5·04
H(1)*W*(4)	0·416	0·097	0·319	7·10
H(2)*W*(4)	0·445	0·005	0·225	7·10

Description and discussion of the structure

Zinc surrounding

The six atoms coordinated to the zinc ion are the oxygens of two water molecules *W*(1) and *W*(2), the amine nitrogen and carbonyl oxygen of the NH_2-terminal glycyl residue, N(1) and O(1), forming a 5-membered chelate ring, and the oxygen atoms of the carboxylate group from a symmetry related peptide chain, O(3*a*) and O(4*a*) (Figs. 1 and 2).

This coordination can be compared with the one observed in bis(glycylglycinato)zinc(II) (unpublished results quoted by Freeman, 1967). A more instructive comparison is possible with the results of the structure of glycylglycylglycinatocopper(II) chloride sesquihydrate (gggCuCl) (Freeman, Robinson & Schoone, 1964) in which the atoms coordinated to the copper are very similar to the ones found in the present structure. The difference is that a chloride ion has replaced one of the water molecules. The distances from the zinc ion to the six atoms fall into three groups (Fig. 2). The distances to N(1), *W*(1) and O(4*a*) are approximately 2·0 Å. These three atoms and the zinc ion are nearly coplanar (Table 4, Plane 1). The atoms O(1) and *W*(2) are at approximately 2·15 Å from the zinc. The bonds from the zinc ion to these atoms are almost perpendicular to the plane of the first group of atoms [Table 5(*a*)]. The Zn–O(3*a*) distance is the longest (2·782 Å).

The bonding geometry of the nearest five atoms is intermediate between that of a trigonal bipyramid and a square pyramid (Table 4, Plane 2). A comparison of bond angles for the 'ideal' trigonal bipyramid and square pyramid and the metal surroundings in gggCuCl and the present structure is given in Table 5(*a*). From the average and individual deviations it is concluded that the present structure is closer to a trigonal bipyramid and probably should be considered a distortion from that geometry, while the geometry in gggCuCl on the other hand is a square pyramid. The sixth atom completes a badly distorted octahedron in both cases.

Other angles occurring in the metal coordination are given in Table 5(*b*). There have been several structures reported in which a metal ion was located similarly with respect to the two oxygens of a carboxylate group (Freeman, Robinson & Schoone, 1964; Gramaccioli, 1966; Gramaccioli & Marsh, 1966). Freeman (1967) assigns a fractional bond order to this type of metal–ligand distance (2·8 Å). It is interesting to note that the four atoms Zn, O(4*a*), O(3*a*), and C(6*a*) are approximately planar. The sum of the four bond angles which are involved (Table 5(*b*) and Fig. 4) is 359·9°.

The bonding of the five nearest ligands is similar to that of the pentacoordination found in the zinc complexes of L-serine (Van der Helm, Nicholas & Fisher, 1970) and glutamic acid (Gramaccioli, 1966). However, the bonding in these two compounds is closer to that of a square pyramid than the trigonal bipyramid. Indeed when the bonding of the zinc complexes is compared with that of the copper(II) complexes of L-serine (Van der Helm & Franks, 1969), glutamic acid (Gramaccioli & Marsh, 1966), and glycylglycylglycine

Table 2. *Anisotropic temperature parameters*

Standard deviation for last digit is given in parentheses.

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

	b_{11}	b_{22}	b_{33}	b_{23}	b_{13}	b_{12}
Zn	0·00120 (1)	0·01206 (9)	0·00351 (3)	0·00070 (9)	−0·00015 (3)	0·00021 (6)
N(1)	0·00129 (7)	0·0156 (8)	0·0041 (2)	0·0017 (7)	0·0003 (2)	0·0001 (4)
C(1)	0·00176 (10)	0·0134 (9)	0·0056 (3)	−0·0001 (9)	0·0013 (3)	−0·0008 (5)
C(2)	0·00106 (7)	0·0118 (8)	0·0041 (3)	0·0012 (8)	0·0000 (2)	0·0002 (4)
O(1)	0·00115 (5)	0·0102 (5)	0·0052 (2)	−0·0003 (6)	0·0008 (2)	0·0010 (3)
N(2)	0·00128 (7)	0·0101 (7)	0·0044 (2)	−0·0014 (6)	0·0013 (2)	0·0008 (4)
C(3)	0·00132 (9)	0·0110 (9)	0·0045 (3)	−0·0021 (9)	−0·0002 (3)	−0·0014 (4)
C(4)	0·00114 (8)	0·0107 (8)	0·0038 (3)	−0·0015 (8)	−0·0002 (2)	0·0003 (4)
O(2)	0·00149 (6)	0·0192 (7)	0·0049 (2)	−0·0016 (7)	−0·0010 (2)	−0·0022 (4)
N(3)	0·00133 (6)	0·0171 (8)	0·0034 (2)	−0·0013 (7)	0·0004 (2)	−0·0010 (4)
C(5)	0·00137 (9)	0·0153 (9)	0·0036 (3)	0·0003 (8)	0·0010 (2)	0·0005 (4)
C(6)	0·00118 (8)	0·0115 (8)	0·0036 (3)	0·0006 (8)	−0·0005 (2)	−0·0010 (4)
O(3)	0·00222 (8)	0·0335 (10)	0·0041 (2)	0·0032 (8)	−0·0001 (2)	0·0085 (5)
O(4)	0·00145 (6)	0·0138 (6)	0·0036 (2)	−0·0004 (7)	0·0003 (2)	0·0006 (3)
W(1)	0·00212 (8)	0·0301 (9)	0·0033 (2)	−0·0027 (7)	0·0004 (2)	−0·0079 (5)
W(2)	0·00163 (6)	0·0168 (7)	0·0047 (2)	−0·0014 (6)	0·0007 (2)	−0·0029 (4)
W(3)	0·00189 (7)	0·0264 (9)	0·0045 (2)	−0·0001 (8)	0·0004 (2)	−0·0006 (4)
W(4)	0·00235 (9)	0·0227 (10)	0·0105 (4)	−0·0058 (10)	−0·0013 (3)	0·0006 (5)
S	0·00131 (3)	0·0181 (3)	0·0048 (1)	0	0·0004 (1)	0
O(5)	0·00211 (8)	0·0272 (10)	0·0066 (3)	0·0026 (9)	0·0005 (2)	−0·0030 (5)
O(6)	0·00266 (9)	0·0191 (8)	0·0060 (3)	0·0015 (8)	−0·0024 (3)	0·0011 (4)

Table 3. *Observed and calculated structure factors*

The listing shows for each reflection the index k, $|10F_o|$ and $10F_c$. The reflections for which the intensity could not be distinguished from the background are indicated by an asterisk.

K	FO	FC
H= 0, L= 0		
2	1551	1474
4	1137	1104
6	1187	1157
8	657	630
H= 1, L= 0		
1	1899	1703
3	456	468
5	1205	1259
7	91*	116
9	324	341
H= 2, L= 0		
0	544	-549
2	2181	-2158
4	289	-286
6	114	123
8	77*	39
H= 3, L= 0		
1	1221	-1187
3	1136	-1159
5	410	-396
7	361	-367
H= 4, L= 0		
0	1779	-1512
2	3224	-3182
4	1649	-1597
6	854	-862
8	562	-630
H= 5, L= 0		
1	1532	-1355
3	1945	-1837
5	1103	-1141
7	299	-323
H= 6, L= 0		
0	1502	1207
2	1409	1351
4	1133	1161
6	186	-207
8	429	461
H= 7, L= 0		
1	2044	1972
3	2095	2165
5	525	519
7	206	276
H= 8, L= 0		
0	845	743
2	987	895
4	1093	1171
6	846	890
8	563	404
H= 9, L= 0		
1	853	789
3	1384	1385
5	665	678
7	394	442
H= 10, L= 0		
0	325	-327
2	287	-326
4	200	-193
6	593	-640
8	113	-108
H= 11, L= 0		
1	1524	-1482
3	1265	-1246
5	749	-780
7	653	-692
H= 12, L= 0		
0	164	-16
2	2091	-2087
4	253	-248
6	380	-410
8	151	-169
H= 13, L= 0		
1	1410	-1386
3	717	-684
5	253	275
7	376	-407
H= 14, L= 0		
0	212	216
2	137	126
4	217	223
6	148	-166
H= 15, L= 0		
1	1361	1309
3	954	976
5	589	611
7	408	444
H= 16, L= 0		
0	1170	1173
2	892	891
4	815	848
6	323	327
H= 17, L= 0		
1	715	687
3	159	182
5	182	182
7	164	148
H= 18, L= 0		
0	611	-618
2	114	-118
4	552	382
6	290	-282
H= 19, L= 0		
1	1269	-1294
3	439	-427
5	510	-510
H= 20, L= 0		
0	1117	-1151
2	872	-900
4	756	-788
6	470	-494
H= 21, L= 0		
1	56*	-12
3	190	-188
5	377	-389
H= 22, L= 0		
0	273	287
2	157	176
4	338	324
H= 23, L= 0		
1	462	463
3	423	438
5	436	459
H= 24, L= 0		
0	982	955
2	442	463
4	687	743
H= 25, L= 0		
1	416	409
3	73	-42
H= 26, L= 0		
0	198	-225
2	404	-409
4	255	-261
H= 27, L= 0		
1	463	-461
3	395	-382
H= 28, L= 0		
0	143	-148
2	416	-426
H= 0, L= 1		
2	521	537
4	238	-263
6	476	471
8	211	203
H= 1, L= 1		
1	762	-729
2	1035	-1056
3	97	-82
4	272	274
5	182	-154
6	462	448
7	79	34
8	195	181
H= 2, L= 1		
1	298	258
2	1297	-1232
3	307	-310
4	293	-301
5	101	-104
6	134	-127
7	201	186
8	73	54
H= 3, L= 1		
1	956	-946
2	78	92
3	938	953
4	127	127
5	200	198
6	59	-51
7	256	295
8	55*	57
H= 4, L= 1		
1	88	136
2	1568	1599
3	1302	1233
4	201	-208
5	260	-244
6	114	-128
7	58*	18
8	58*	29
H= 5, L= 1		
1	683	-669
2	403	357
3	538	-463
4	87	76
5	239	-218
6	466	-466
7	62*	12
8	68	-33
H= 6, L= 1		
1	1103	-1075
2	249	299
3	582	-550
4	296	298
5	71	22
6	71	58
7	162	-135
8	75*	19
H= 7, L= 1		
1	665	-711
2	36	29
3	39	75
4	453	-441
5	65*	48
6	166	126
7	199	197
8	64*	35
H= 8, L= 1		
1	279	255
2	330	398
3	160	-123
4	527	-555
5	338	347
6	172	-188
7	171	-158
8	168	156
H= 9, L= 1		
1	598	-598
2	319	317
3	186	191
4	290	302
5	321	-343
6	61*	-14
7	290	271
8	57*	-35
H= 10, L= 1		
1	346	-309
2	138	152
3	343	338
4	399	374
5	83	109
6	130	-146
7	61*	22
8	55*	-40
H= 11, L= 1		
1	40*	-7
2	117	-158
3	536	592
4	98	81
5	168	-190
6	58*	78
7	132	-128
8	106	100
H= 12, L= 1		
1	367	-362
2	99	88
3	350	-360
4	178	-192
5	158	172
6	58*	-23
7	61	86
8	68	98
H= 13, L= 1		
1	236	251
2	294	-306
3	446	474
4	60	17
5	323	-314
6	150	-152
7	48	76
8	165	-131
H= 14, L= 1		
1	78	51
2	348	-330
3	186	182
4	332	346
5	111	-118
6	58*	6
7	292	-319
H= 15, L= 1		
1	423	-394
2	160	167
3	434	439
4	156	155
5	216	-224
6	83	-69
7	127	131
H= 16, L= 1		
1	328	-340
2	110	-75
3	105	89
4	141	-135
5	136	-123
6	126	-140
7	147	144
H= 17, L= 1		
1	75	-51
2	244	-247
3	91	81
4	89	-70
5	105	-128
6	56*	27
7	51*	32
H= 18, L= 1		
1	154	-156
2	53*	3
3	55*	7
4	247	264
5	333	347
6	55*	-1
7	106	103
H= 19, L= 1		
1	393	-367
2	388	386
3	94	64
4	57*	-6
5	114	-130
6	53*	15
H= 20, L= 1		
1	206	210
2	56*	9
3	123	-128
4	131	113
5	56*	-65
6	99	-96
H= 21, L= 1		
1	145	-139
2	137	-142
3	343	357
4	65	-58
5	55*	-31
6	49*	-21
H= 22, L= 1		
1	104	-121
2	307	-301
3	57*	-17
4	75	94
5	75	-102
H= 23, L= 1		
1	339	-324
2	213	-222
3	93	-91
4	55*	-17
5	103	-108
H= 24, L= 1		
1	143	119
2	56*	-9
3	56*	30
4	96	88
5	47*	54
H= 25, L= 1		
1	56*	-51
2	191	210
3	194	203
4	125	126
H= 26, L= 1		
1	55*	-53
2	227	-207
3	51*	-3
H= 27, L= 1		
1	150	-168
2	49	-64
3	164	162
H= 28, L= 1		
1	49*	-46
2	47*	-2
H= 0, L= 2		
0	676	724
2	547	585
4	947	983
6	867	841
8	318	343
H= 1, L= 2		
0	1497	-1482
1	1308	1295
2	810	-905
3	429	451
4	864	-868
5	713	702
6	231	-236
7	238	231
8	356	-345
H= 2, L= 2		
0	655	-763
1	450	-407
2	198	187
3	949	-961
4	119	146
5	795	-777
6	280	283
7	251	-264
8	321	-298
H= 3, L= 2		
0	1669	-1655
1	1454	-1412
2	476	-482
3	990	-1035
4	537	-551
5	431	-456
6	510	-521
7	187	-169
8	212	-214
H= 4, L= 2		
0	3370	-3563
1	741	-667
2	114	-25
3	647	-648
4	917	-956
5	76	68
6	776	-795
7	59	65
8	439	-434
H= 5, L= 2		
0	2168	2162
1	42	-8
2	125	106
3	359	-377
4	622	617
5	938	-958
6	442	468
7	230	-233
8	319	344
H= 6, L= 2		
0	335	-346
1	2442	2486
2	839	889
3	639	679
4	522	-577
5	660	686
6	53*	-31
7	304	323
8	145	149
H= 7, L= 2		
0	1012	1024
1	379	-435
2	1530	1519
3	1380	1369
4	428	458
5	660	671
6	343	354
7	500	513
8	142	135
H= 8, L= 2		
0	289	-294
1	475	482
2	2028	2068
3	448	-492
4	863	843
5	138	166
6	702	735
7	214	223
8	285	303
H= 9, L= 2		
0	715	-731
1	314	323
2	1585	-1563
3	896	861
4	518	-523
5	188	191
6	344	-350
7	250	262
8	327	-358
H= 10, L= 2		
0	143	103
1	963	-941
2	266	-223
3	1772	-1756
4	749	-764
5	465	-449
6	80	95
7	473	-477
8	56*	-120
H= 11, L= 2		
0	401	-369
1	100	75
2	1059	-1000
3	1114	-1147
4	341	-350
5	645	-651
6	321	-331
7	273	-294
8	64	-24
H= 12, L= 2		
0	589	-637
1	197	220
2	989	-952
3	81	-101
4	724	-782
5	58*	92
6	375	-380
7	63	34
8	374	-405
H= 13, L= 2		
0	1394	1393
1	586	-553
2	1225	1243
3	712	-765
4	471	505
5	152	-150
6	467	486
7	230	-247
H= 14, L= 2		
0	355	367
1	1412	1427
2	254	265
3	755	786
4	87	83
5	705	741
6	527	576
7	459	491
H= 15, L= 2		
0	178	-177
1	1145	1137
2	219	228
3	329	308
4	415	429
5	383	390
6	90	88
7	378	394
H= 16, L= 2		
0	481	517
1	248	-255
2	903	911
3	376	-381
4	164	172
5	288	-290
6	277	282
7	146	-149
H= 17, L= 2		
0	509	-519
1	328	328
2	631	-643
3	382	394
4	315	-327
5	152	167
6	363	-386
7	243	280
H= 18, L= 2		
0	517	-499
1	883	-908
2	53*	-37
3	655	-674
4	316	-316
5	305	-324
6	40	54
H= 19, L= 2		
0	511	-495
1	865	-891
2	139	-124
3	227	-240
4	208	-223
5	524	-541
6	52*	-26
H= 20, L= 2		
0	603	-620
1	432	441
2	55*	-60
3	465	491
4	649	-648
5	179	184
6	237	-251
H= 21, L= 2		
0	552	591
1	345	-323
2	353	357
3	156	160
4	366	384
5	179	-186
6	179	171
H= 22, L= 2		
0	92	83
1	417	437
2	248	229
3	150	158
4	57	-69
5	258	270
H= 23, L= 2		
0	228	246
1	425	457
2	149	164
3	406	410
4	225	252
5	419	340
H= 24, L= 2		
0	57*	17
1	310	-308
2	371	384
3	85	-84
4	464	465
H= 25, L= 2		
0	523	-532
1	56*	65
2	293	-309
3	53*	-65
4	329	-351
H= 26, L= 2		
0	65	-26
1	231	-246
2	53*	-20
3	149	-158
H= 27, L= 2		
0	52*	-9
1	198	-203
2	66	-79
3	236	-271
H= 28, L= 2		
0	472	-475
1	74	92
2	232	-236
H= 0, L= 3		
2	896	958
4	287	250
6	326	323
8	42*	3
H= 1, L= 3		
1	1199	1250
2	330	-342
3	165	-172
4	46	-34
5	255	288
6	209	222
7	59	-5
8	92	110
H= 2, L= 3		
1	369	382
2	721	765
3	374	352
4	177	-196
5	115	-94
6	182	-170
7	230	236
8	48*	32
H= 3, L= 3		
1	115	-67
2	132	-124
3	472	-502
4	591	579
5	232	-249
6	93	86
7	60	5
8	49*	-15
H= 4, L= 3		
1	588	611
2	722	-225
3	205	219
4	501	-504
5	198	-207
6	139	-125
7	52*	12
8	134	-131
H= 5, L= 3		
1	1336	1379
2	190	-182
3	379	-418
4	480	-464
5	175	150
6	95	-99
7	361	-366
8	70	-64
H= 6, L= 3		
1	41	-77
2	51	17
3	566	-571
4	240	-206
5	58	-52
6	276	294
7	115	96
8	75	-89
H= 7, L= 3		
1	1107	1149
2	707	-741
3	124	-133
4	90	-93
5	236	246
6	159	148
7	56*	-18
8	53*	5
H= 8, L= 3		
1	108	60
2	206	-178
3	250	-250
4	208	230
5	80	-83
6	358	375
7	58*	-44
8	86	83
H= 9, L= 3		
1	87	23
2	532	535
3	112	95
4	198	180
5	191	226
6	191	180
7	61*	26
8	62*	-72
H= 10, L= 3		
1	906	929
2	200	161
3	303	314
4	88	-94
5	179	194
6	155	-162
7	171	122
8	61*	-6
H= 11, L= 3		
1	55	-11
2	593	-569
3	613	-660
4	56*	75
5	255	251
6	122	123
7	212	-212
8	61	-70
H= 12, L= 3		
1	70	-30
2	265	-272
3	317	-307
4	150	-162
5	201	-204
6	90	-64
7	59*	68
8	192	-203
H= 13, L= 3		
1	52*	-18
2	169	-180
3	513	-562
4	58*	8
5	79	67
6	88	96
7	87	-112
H= 14, L= 3		
1	381	378
2	52*	44
3	82	82
4	172	164
5	395	-394
6	66	75
7	53*	5
H= 15, L= 3		
1	48*	-19
2	77	-47
3	198	210
4	63	-59
5	220	238
6	151	-171
7	52*	37
H= 16, L= 3		
1	199	205
2	51*	54
3	223	218
4	136	135
5	53*	26
6	56*	61
7	51*	51
H= 17, L= 3		
1	218	221
2	229	240
3	400	-394
4	79	-52
5	347	376
6	107	104
7	61	-19
H= 18, L= 3		
1	75	93
2	53*	-46
3	174	-112
4	246	-249
5	68	-43
6	90	73
H= 19, L= 3		
1	213	197
2	200	199
3	205	-197
4	57*	15
5	56*	-49
6	52*	-21
H= 20, L= 3		
1	85	108
2	202	-200
3	149	145
4	56*	27
5	55*	32
6	49*	11
H= 21, L= 3		
1	143	127
2	79	-41
3	56*	17
4	85	-99
5	53*	-5
H= 22, L= 3		
1	56*	67
2	56*	54
3	158	-161
4	55*	-15
5	85	-98
H= 23, L= 3		
1	164	139
2	139	-140
3	120	-124
4	82	54
5	180	196
H= 24, L= 3		
1	55	-62
2	86	73
3	107	97
4	51*	53
H= 25, L= 3		
1	67	79
2	53*	67
3	86	-85
4	67	75
H= 26, L= 3		
1	82	-52
2	110	107
3	80	91
H= 27, L= 3		
1	49*	26
2	48*	-4
H= 28, L= 3		
1	132	-136
H= 0, L= 4		
0	1274	1410
2	352	-396
4	722	-718
6	171	-128
8	140	114
H= 1, L= 4		
0	2419	-2527
1	225	-226
2	1030	-1098
3	227	-251
4	1282	-1352
5	98	94
6	895	-888
7	67	-85
8	519	-503
H= 2, L= 4		
0	1005	976
1	998	-1032
2	396	-414
3	805	-810
4	614	582
5	1211	-1235
6	170	-153
7	519	-503
8	60	35
H= 3, L= 4		
0	1540	-1628
1	231	207
2	704	-751
3	234	242
4	801	-861
5	236	245
6	509	-505
7	51*	-56
8	279	-272
H= 4, L= 4		
0	585	-585
1	1033	-1067
2	38	-25
3	362	388
4	356	381
5	121	-127
6	280	-282
7	98	-112
8	214	193
H= 5, L= 4		
0	1546	1582
1	57	-53
2	1562	1608
3	222	-224
4	1247	1257
5	437	-440
6	680	707
7	175	159
8	503	512
H= 6, L= 4		
0	1941	-1474
1	918	935
2	237	245
3	1939	2018
4	167	189
5	1035	1055
6	314	-319
7	491	508
8	68	41
H= 7, L= 4		
0	31*	110
1	80	-64
2	529	545
3	146	158
4	316	329
5	311	-339
6	462	449
7	55*	11
8	110	128
H= 8, L= 4		
0	405	-391
1	87	-55
2	97	-104
3	147	120
4	522	524
5	78	-55
6	213	-217
7	81	-62
8	131	109
H= 9, L= 4		
0	1072	-1096
1	347	365
2	1251	-1269
3	530	537
4	588	-623
5	175	182
6	532	-550
7	54	-50
8	379	-384
H= 10, L= 4		
0	882	888
1	1795	-1825
2	285	-318
3	1349	-1395
4	257	285
5	846	-883
6	114	-107
7	655	-686
8	100	113
H= 11, L= 4		
0	520	477
1	421	-409
2	669	-688
3	370	-366
4	432	-478
5	57*	30
6	272	-287
7	171	-141
H= 12, L= 4		
0	442	462
1	202	210
2	520	-545
3	574	586
4	118	-125
5	266	265
6	70*	20
7	64*	-5
H= 13, L= 4		
0	412	447
1	157	-114
2	759	755
3	86	-113
4	474	483
5	297	304
6	587	618
7	75	-57
H= 14, L= 4		
0	498	514
1	1012	1032
2	532	-546
3	1144	1172
4	288	288
5	703	720
6	168	-183
7	535	570
H= 15, L= 4		
0	556	574
1	344	330
2	536	512
3	166	-190
4	371	382
5	297	298
6	266	273
7	60	-32
H= 16, L= 4		
0	745	749
1	412	-417
2	60	-51
3	446	-462
4	201	210
5	385	-404
6	55*	26
7	128	-132
H= 17, L= 4		
0	576	-591
1	52*	-32
2	636	-662
3	159	-160
4	649	-648
5	191	-181
6	552	-489
H= 18, L= 4		
0	313	-328
1	726	-746
2	155	-155
3	507	-508
4	49	61
5	545	-558
6	64	-85
H= 19, L= 4		
0	324	-343
1	91	-48
2	388	-410
3	106	117
4	500	-509
5	67	83
6	49*	-86
H= 20, L= 4		
0	229	253
1	291	293
2	281	-291
3	278	265
4	95	106
5	278	290
6	48*	-44
H= 21, L= 4		
0	808	815
1	99	94
2	578	590
3	56*	-6
4	552	562
5	88	-90
H= 22, L= 4		
0	333	337
1	630	639
2	99	-70
3	282	266
4	137	149
5	322	331
H= 23, L= 4		
0	292	276
1	55*	-24
2	121	133
3	98	-115
4	198	206
H= 24, L= 4		
0	214	199
1	190	-199
2	200	-215
3	277	-270
4	48*	-4
H= 25, L= 4		
0	545	-549
1	53*	67
2	411	-419
3	65	-56
H= 26, L= 4		
0	236	226
1	343	-372
2	173	-174
3	273	-269
H= 27, L= 4		
0	83	-72
1	67	91
2	47*	-60
H= 28, L= 4		
0	241	265
H= 0, L= 5		
2	649	-654
4	635	642
6	211	184
8	175	149
H= 1, L= 5		
1	372	-401
2	505	-500
3	695	713
4	287	-283
5	285	-270
6	179	-166
7	154	127
8	101	-97
H= 2, L= 5		
1	306	-288
2	871	-915
3	111	98
4	188	203
5	232	-251
6	55	-40
7	227	229
8	47*	27
H= 3, L= 5		
1	541	-559
2	134	132
3	316	366
4	205	-178
5	252	-243
6	107	-106
7	49*	42
8	100	80
H= 4, L= 5		
1	776	792
2	33*	-77
3	86	-61
4	40*	21
5	286	-300
6	353	-394
7	175	-203
8	146	-130
H= 5, L= 5		
1	454	-501
2	769	810
3	75	-66
4	50	25
5	172	-196
6	161	159
7	60	21
8	47*	31
H= 6, L= 5		
1	357	386
2	154	-149
3	297	306
4	293	308
5	146	146
6	178	172
7	105	109
8	48*	-47
H= 7, L= 5		
1	81	-138
2	473	502
3	138	136
4	224	-222
5	115	-135
6	161	-176
7	154	149
8	77	75
H= 8, L= 5		
1	376	-360
2	109	101
3	149	132
4	134	141
5	66	101
6	53*	-14
7	53*	25
8	100	117
H= 9, L= 5		
1	393	-435
2	438	475
3	346	354
4	289	-301
5	52*	5
6	92	-86
7	225	232
8	49*	51
H= 10, L= 5		
1	274	271
2	44*	-35
3	492	-504
4	79	106
5	53	1
6	113	-104
7	171	-185
H= 11, L= 5		
1	208	-247
2	173	162
3	162	183
4	86	-102
5	252	-263
6	58*	9
7	72	-62
H= 12, L= 5		
1	74	-85
2	167	-187
3	102	88
4	53*	-91
5	58*	-6
6	97	-108
7	68*	17
H= 13, L= 5		
1	147	-158
2	93	-70
3	318	327
4	95	74
5	384	-382
6	73*	11
7	57*	-36
H= 14, L= 5		
1	54	43
2	186	-168
3	257	278
4	68*	17
5	70*	2
6	63*	-13
7	118	140
H= 15, L= 5		
1	65*	-17
2	281	286
3	336	338
4	65*	28
5	63*	-11
6	57*	-28
7	212	213
H= 16, L= 5		
1	58*	-4
2	299	291
3	237	-218
4	59*	6
5	87	99
6	53*	80
H= 17, L= 5		
1	179	-196
2	106	-97
3	61	73
4	122	-130
5	56*	28
6	116	114
H= 18, L= 5		
1	136	-129
2	171	160
3	169	-174
4	91	-96
5	55*	-39
6	49*	-36
H= 19, L= 5		
1	169	-145
2	84	-98
3	107	-98
4	132	-148
5	243	-190
6	86	-104
H= 20, L= 5		
1	55*	-24
2	52	55
3	109	128
4	129	-136
5	56	33
H= 21, L= 5		
1	125	-127
2	56*	55
3	163	173
4	155	165
5	91	-98
H= 22, L= 5		
1	87	93
2	55*	-10
3	135	-123
4	56	64
5	111	123
H= 23, L= 5		
1	130	-127
2	98	-106
3	68	84
4	50	29
H= 24, L= 5		
1	111	-113
2	139	-156
3	51*	-1
4	45*	20
H= 25, L= 5		
1	146	-139
2	148	-144
3	110	139
H= 26, L= 5		
1	49*	-16
2	116	-118
H= 27, L= 5		
1	47*	-51
H= 0, L= 6		
0	1786	-1830
2	1142	-1198
4	1251	-1305
6	150	-144
8	532	-473
H= 1, L= 6		
0	1434	-1571
1	281	-282
2	655	-697
3	569	-558
4	392	-409
5	454	-446
6	404	-413
7	253	-236
8	195	-191
H= 2, L= 6		
0	1082	1088
1	2319	-1927
2	33*	50
3	1227	-1249
4	183	-164
5	914	-927
6	293	304
7	284	-298
8	130	-102
H= 3, L= 6		
0	1625	-1674
1	33*	90
2	674	-703
3	607	637
4	604	-613
5	492	473
6	430	-420
7	124	120
8	152	-158
H= 4, L= 6		
0	824	868
1	747	802
2	1245	1308
3	565	562
4	644	667
5	62	-57
6	650	654
7	197	178
8	332	330
H= 5, L= 6		
0	934	969
1	755	781
2	1432	1474
3	867	900
4	325	332
5	282	292
6	353	363
7	359	373
8	345	328
H= 6, L= 6		
0	679	-736
1	1089	1131
2	284	309
3	864	892
4	809	-821
5	369	381
6	52*	33
7	359	363
8	110	-118
H= 7, L= 6		
0	860	872
1	868	-929
2	508	517
3	268	-266
4	539	563
5	519	-515
6	189	189
7	143	-123
8	169	142
H= 8, L= 6		
0	1109	-1139
1	129	-132
2	425	-405
3	508	-510
4	614	-644
5	93	-66
6	482	-500
7	97	-72
H= 9, L= 6		
0	910	-926
1	877	-902
2	984	-988
3	336	-354
4	649	-670
5	423	-433
6	369	-378
7	381	-393
H= 10, L= 6		
0	486	486
1	731	-735
2	215	243
3	659	-674
4	91	-100
5	438	-452
6	405	421
7	253	-240
H= 11, L= 6		
0	387	-401
1	711	752
2	365	-340
3	690	713
4	275	-277
5	453	455
6	121	-129
7	338	330
H= 12, L= 6		
0	230	229
1	47*	-9
2	876	919
3	138	139
4	262	273
5	125	124
6	423	422
7	62*	-14
H= 13, L= 6		
0	728	726
1	624	649
2	706	727
3	205	223
4	398	413
5	265	271
6	201	194
7	177	198
H= 14, L= 6		
0	454	-496
1	676	665
2	157	-171
3	697	723
4	71	14
5	205	203
6	64*	20
7	301	301
H= 15, L= 6		
0	156	-159
1	553	-578
2	546	548
3	827	-839
4	336	335
5	491	-446
6	57*	38
H= 16, L= 6		
0	777	-760
1	122	-104
2	176	-184
3	83	99
4	496	-514
5	136	-126
6	278	-292
H= 17, L= 6		
0	904	-914
1	237	-255
2	334	-342
3	59*	-58
4	271	-286
5	140	-171
6	227	-218
H= 18, L= 6		
0	294	-274
1	859	-873
2	415	430
3	318	-316
4	146	-151
5	389	-407
6	149	157
H= 19, L= 6		
0	257	-259
1	422	442
2	278	-287
3	593	629
4	132	-142
5	235	260
H= 20, L= 6		
0	148	142
1	145	170
2	533	568
3	100	83
4	335	362
5	144	145
H= 21, L= 6		
0	496	506
1	52	-29
2	249	264
3	64	54
4	288	287
5	47*	36
H= 22, L= 6		
0	167	-164
1	517	526
2	53*	20
3	390	402
4	229	-247
H= 23, L= 6		
0	226	218
1	241	-238
2	178	203
3	259	-263
4	196	214
H= 24, L= 6		
0	610	-631
1	229	-228
2	181	-190
3	311	-259
H= 25, L= 6		
0	182	-190
1	51*	-48
2	409	-416
H= 26, L= 6		
0	131	157
1	265	-275
H= 0, L= 7		
2	546	-525
4	39*	16
6	91	-72
8	154	125
H= 1, L= 7		
1	430	-440
2	45	26
3	201	-191
4	71	75
5	320	299
6	101	-96
7	42*	29
H= 2, L= 7		
1	275	-290
2	672	686
3	33	23
4	40*	-24
5	44*	-67

(gggCuCl), one is struck by the greater variation in the bonding geometry which one finds in the zinc complexes.

The elongation of the axial bonds in the present structure is noteworthy. The elongation of the Zn–O(1) bond could be attributed to the strain imposed by

Table 3 (*cont.*)

K	FO	FC
H= 2, L= 7		
6	72	-7
7	44*	-30
8	39*	-8
H= 3, L= 7		
1	270	270
2	101	-84
3	455	-471
4	45*	-1
5	284	264
6	39	-64
7	314	-308
8	149	-133
H= 4, L= 7		
1	147	121
2	149	-150
3	36*	-75
4	85	-86
5	47	13
6	48*	-25
7	61	34
H= 5, L= 7		
1	72	45
2	253	-271
3	79	-81
4	90	101
5	315	327
6	133	132
7	111	-81
H= 6, L= 7		
1	451	-475
2	66	51
3	44*	-28
4	210	-211
5	185	184
6	128	154
7	48*	34
H= 7, L= 7		
1	241	239
2	325	-338
3	257	-272
4	171	152
5	99	90
6	128	121
7	88	-81
H= 8, L= 7		
1	571	-581
2	42*	-25
3	113	-124
4	90	102
5	189	179
6	115	117
7	49*	-4
H= 9, L= 7		
1	368	386
2	44*	-17
3	176	-187
4	51*	-28
5	308	307
6	267	-263
7	144	-130
H= 10, L= 7		
1	153	-180
2	91	79
3	80	66
4	231	-241
5	88	84
6	53*	-19
7	258	-252
H= 11, L= 7		
1	47*	37
2	337	-357
3	176	-179
4	202	198
5	71	-59
6	94	113
7	52*	-13
H= 12, L= 7		
1	81	-88
2	118	128
3	68	61
4	258	271
5	56*	-19
6	105	-109
7	58*	65
H= 13, L= 7		
1	168	176
2	208	-165
3	53*	22
4	156	151
5	58*	50
6	62*	73
H= 14, L= 7		
1	185	-182
2	41*	76
3	81	89
4	172	-210
5	64*	-31
6	63*	-30
H= 15, L= 7		
1	408	429
2	96	96
3	311	-318
4	105	-93
5	116	144
6	107	-110
H= 16, L= 7		
1	71	84
2	69*	-5
3	70*	-41
4	65*	70
5	76	53
6	171	173
H= 17, L= 7		
1	152	140
2	251	242
3	132	-141
4	102	-94
5	53*	21
6	32	-55
H= 18, L= 7		
1	79	-93
2	59*	87
3	58*	10
4	105	-113
5	51*	-63
H= 19, L= 7		
1	41*	421
2	56*	8
3	56*	12
4	53*	21
5	36	59
H= 20, L= 7		
1	54*	82
2	149	-149
3	53*	58
4	51*	-35
5	47*	-23
H= 21, L= 7		
1	151	140
2	89	92
3	264	-270
4	46	-51
H= 22, L= 7		
1	53*	-15
2	52*	28
3	51*	-4
4	47*	43
H= 23, L= 7		
1	140	136
2	51*	-45
3	73	62
H= 24, L= 7		
1	49*	-2
2	47*	-52
H= 25, L= 7		
1	47*	7
H= 0, L= 8		
0	334	-388
2	1557	-1625
4	729	-775
6	662	-663
H= 1, L= 8		
0	183	-195
1	720	-762
2	374	-14
3	883	-914
4	237	236
5	256	-240
6	158	-151
7	299	-286
H= 2, L= 8		
0	199	-202
1	90	-109
2	269	-281
3	241	224
4	313	304
5	44*	19
6	76	-76
7	41	-18
H= 3, L= 8		
0	142	-144
1	716	737
2	251	236
3	539	531
4	124	-117
5	508	529
6	47	-25
7	40*	125
H= 4, L= 8		
0	1939	2036
1	47	-25
2	949	974
3	130	112
4	1009	1044
5	318	-314
6	467	477
7	48*	9
H= 5, L= 8		
0	582	-591
1	1119	1189
2	360	355
3	418	420
4	47	2
5	555	563
6	75	-66
7	430	428
H= 6, L= 8		
0	495	-487
1	276	-282
2	180	-189
3	135	116
4	284	303
5	265	289
6	315	-322
7	84	94
H= 7, L= 8		
0	40*	6
1	478	-508
2	399	-402
3	749	-770
4	267	275
5	567	-564
6	56	38
7	304	-320
H= 8, L= 8		
0	1088	-1139
1	270	258
2	1421	-1499
3	194	-190
4	505	-507
5	78	76
6	512	-505
7	131	-133
H= 9, L= 8		
0	294	312
1	1120	-1172
2	52	51
3	807	-870
4	84	88
5	365	-365
6	88	-36
7	281	-296
H= 10, L= 8		
0	742	779
1	47	49
2	48*	36
3	347	-360
4	69	60
5	53*	30
6	51*	-18
7	47*	-14
H= 11, L= 8		
0	48*	-17
1	1093	1145
2	85	-68
3	894	928
4	56	-62
5	508	525
6	71	69
H= 12, L= 8		
0	1509	1602
1	154	137
2	534	543
3	52*	-5
4	637	672
5	181	203
6	461	475
H= 13, L= 8		
0	208	-238
1	618	626
2	116	-132
3	296	323
4	167	162
5	421	445
6	59*	-44
H= 14, L= 8		
0	53*	-107
1	127	98
2	241	-234
3	93	-104
4	50	68
5	105	-90
6	280	-282
H= 15, L= 8		
0	293	178
1	992	-1008
2	280	291
3	736	-747
4	102	-83
5	505	-504
6	51*	31
H= 16, L= 8		
0	638	-664
1	167	-148
2	975	-957
3	72*	34
4	545	-569
5	87	-85
H= 17, L= 8		
0	461	-460
1	169	-176
2	66*	54
3	171	-173
4	82	-56
5	177	-193
H= 18, L= 8		
0	349	358
1	153	174
2	206	224
3	76	108
4	263	270
5	48	0
H= 19, L= 8		
0	265	276
1	451	459
2	91	-106
3	684	700
4	59	-25
H= 20, L= 8		
0	600	611
1	55*	-12
2	432	435
3	145	-163
4	556	496
H= 21, L= 8		
0	53*	69
1	224	226
2	75	86
3	136	126
4	78	85
H= 22, L= 8		
0	217	-214
1	52*	-10
2	269	-273
3	48*	-42
H= 23, L= 8		
0	49*	-47
1	572	-572
2	93	-115
H= 24, L= 8		
0	229	-217
1	128	150
H= 0, L= 9		
2	117	154
4	117	-121
6	77	-49
H= 1, L= 9		
1	37*	42
2	241	-246
3	374	401
4	42*	21
5	321	-288
6	42*	-4
7	176	164
H= 2, L= 9		
1	101	119
2	39*	56
3	328	-316
4	60	49
5	122	-119
6	56	-63
7	52	-34
H= 3, L= 9		
1	268	284
2	43	5
3	196	191
4	283	-292
5	207	-202
6	98	-97
7	40*	-51
H= 4, L= 9		
1	362	385
2	139	153
3	298	293
4	169	-161
5	220	-220
6	120	108
7	45*	1
H= 5, L= 9		
1	285	-293
2	39*	-65
3	184	-192
4	364	369
5	233	-229
6	174	171
7	288	222
H= 6, L= 9		
1	58	67
2	73	-101
3	47*	6
4	67	-39
5	51*	28
6	114	-114
7	90	108
H= 7, L= 9		
1	107	-123
2	100	114
3	382	378
4	73	-54
5	76	86
6	58	63
H= 8, L= 9		
1	77	92
2	406	437
3	99	79
4	80	97
5	190	202
6	48*	-7
H= 9, L= 9		
1	82	-73
2	139	-148
3	54	-39
4	43	43
5	131	-144
6	252	-263
H= 10, L= 9		
1	238	-251
2	362	-370
3	83	-88
4	129	124
5	274	-281
6	82	87
H= 11, L= 9		
1	523	-533
2	62	-79
3	175	195
4	140	132
5	106	-93
6	49*	25
H= 12, L= 9		
1	147	133
2	216	-211
3	57	83
4	94	-102
5	53*	-1
6	119	-141
H= 13, L= 9		
1	155	-160
2	344	-361
3	269	291
4	123	126
5	84	-97
6	57*	72
H= 14, L= 9		
1	195	175
2	56*	52
3	125	141
4	211	196
5	112	113
H= 15, L= 9		
1	276	-297
2	255	263
3	77	-34
4	64*	79
5	59*	-8
H= 16, L= 9		
1	65*	-54
2	94	-117
3	140	-138
4	77	-93
5	141	-155
H= 17, L= 9		
1	188	-130
2	268	-249
3	204	194
4	42*	-98
5	39*	-18
H= 18, L= 9		
1	59*	10
2	58*	9
3	56*	14
4	96	129
H= 19, L= 9		
1	269	-272
2	53*	21
3	52*	20
4	48*	-80
H= 20, L= 9		
1	90	92
2	52*	-13
3	49*	-52
H= 21, L= 9		
1	116	-118
2	49*	-8
3	47*	-15
H= 22, L= 9		
1	110	-118
2	68	-63
H= 23, L= 9		
1	161	-169
H= 0, L= 10		
0	457	-510
2	428	-455
4	432	-470
6	341	-354
H= 1, L= 10		
0	685	730
1	321	-336
2	471	490
3	520	-523
4	586	585
5	495	-512
6	570	558
H= 2, L= 10		
0	333	-351
1	479	520
7	200	214
3	682	697
4	221	-213
5	365	345
6	192	180
H= 3, L= 10		
0	286	256
1	404	428
2	741	753
3	644	666
4	252	257
5	457	456
6	42*	28
H= 4, L= 10		
0	243	250
1	45*	37
2	833	892
3	48*	22
4	349	358
5	175	-185
6	455	441
H= 5, L= 10		
0	711	-740
1	397	408
2	877	-869
3	227	232
4	612	-643
5	154	132
6	243	-250
H= 6, L= 10		
0	45*	-4
1	530	-554
2	81	88
3	499	-514
4	114	-126
5	327	-344
6	94	-113
H= 7, L= 10		
0	47*	3
1	522	-553
2	200	-209
3	439	-472
4	51*	12
5	310	-306
6	47*	-44
H= 8, L= 10		
0	1023	-1065
1	98	-89
2	318	-328
3	51*	-29
4	544	-559
5	154	137
6	264	-270
H= 9, L= 10		
0	1328	1440
1	320	-332
2	175	183
3	70	-40
4	51*	46
5	252	-267
6	320	324
H= 10, L= 10		
0	98	74
1	787	840
2	283	-286
3	390	396
4	128	-137
5	458	452
6	191	181
H= 11, L= 10		
0	62	-47
1	332	365
2	128	-115
3	347	377
4	141	140
5	286	283
H= 12, L= 10		
0	281	284
1	314	-324
2	522	521
3	151	-163
4	459	461
5	51*	7
H= 13, L= 10		
0	195	176
1	144	170
2	230	-232
3	232	226
4	215	-212
5	208	211
H= 14, L= 10		
0	551	-564
1	379	-395
2	51	19
3	409	-422
4	134	-129
5	333	-338
H= 15, L= 10		
0	402	-409
1	203	-197
2	180	-174
3	386	-397
4	247	-254
5	331	-347
H= 16, L= 10		
0	425	-444
1	93	100
2	192	-176
3	354	372
4	444	-443
H= 17, L= 10		
0	219	230
1	273	-272
2	269	237
3	135	-127
4	210	220
H= 18, L= 10		
0	57*	49
1	229	225
2	117	112
3	198	207
H= 19, L= 10		
0	274	239
1	276	261
2	204	225
3	319	318
H= 20, L= 10		
0	194	174
1	71	-23
2	411	398
H= 21, L= 10		
0	196	-214
1	127	137
2	364	-378
H= 0, L= 11		
2	247	-239
4	228	-207
6	39*	2
H= 1, L= 11		
1	41*	22
2	42*	-73
3	375	-377
4	357	-358
5	53	3
6	60	65
H= 2, L= 11		
1	133	124
2	42*	18
3	286	-309
4	80	61
5	55	-44
6	39*	-47
H= 3, L= 11		
1	360	369
2	102	81
3	151	152
4	82	79
5	145	136
6	39*	3
H= 4, L= 11		
1	94	87
2	45*	4
3	116	117
4	128	125
5	82	-99
6	256	241
H= 5, L= 11		
1	223	221
2	272	304
3	226	-232
4	204	-205
5	141	150
6	44*	-11
H= 6, L= 11		
1	48*	60
2	71	-95
3	49*	-7
4	78	-88
5	135	152
H= 7, L= 11		
1	163	164
2	49*	-26
3	213	-210
4	65	-13
5	109	112
H= 8, L= 11		
1	116	-107
2	51*	1
3	152	-179
4	126	-143
5	47*	19
H= 9, L= 11		
1	193	209
2	322	342
3	115	-115
4	51*	-73
5	61	-74
H= 10, L= 11		
1	51*	-16
2	61	-86
3	177	178
4	49*	42
5	47*	-39
H= 11, L= 11		
1	200	185
2	291	-292
3	52*	-28
4	77	-108
5	54	60
H= 12, L= 11		
1	53	-55
2	210	215
3	114	-116
4	51*	62
5	50	-25
H= 13, L= 11		
1	235	240
2	255	271
3	53*	23
4	53*	32
H= 14, L= 11		
1	245	260
2	138	156
3	56*	31
4	57*	-39
H= 15, L= 11		
1	58*	14
2	77	78
3	206	-205
4	55*	-30
H= 16, L= 11		
1	105	-97
2	126	-116
3	56*	34
H= 17, L= 11		
1	171	185
2	55*	55
3	98	-123
H= 18, L= 11		
1	52*	6
2	94	80
H= 19, L= 11		
1	126	135
2	47*	41
H= 0, L= 12		
0	45*	-19
2	272	-293
4	122	-124
H= 1, L= 12		
0	234	230
1	248	-267
2	596	625
3	156	135
4	376	379
5	39*	-67
H= 2, L= 12		
0	110	110
1	480	484
2	69	-39
3	798	807
4	211	207
5	477	478
H= 3, L= 12		
0	412	414
1	52	-41
2	504	515
3	240	235
4	299	306
5	109	128
H= 4, L= 12		
0	136	115
1	87	75
2	59	53
3	60	-38
4	90	116
5	189	-179
H= 5, L= 12		
0	652	-687
1	224	238
2	381	-383
3	49*	36
4	311	-308
5	91	-87
H= 6, L= 12		
0	221	212
1	697	-726
2	264	-273
3	567	-574
4	114	121
5	444	-423
H= 7, L= 12		
0	445	-460
1	258	-263
2	370	-398
3	141	-151
4	359	-356
5	44*	30
H= 8, L= 12		
0	51*	-64
1	114	129
2	120	-108
3	49*	41
4	153	151
H= 9, L= 12		
0	564	580
1	51*	21
2	444	459
3	122	129
4	401	413
H= 10, L= 12		
0	336	341
1	623	636
2	149	-162
3	432	438
4	84	-107
H= 11, L= 12		
0	384	417
1	155	157
2	229	-230
3	80	-69
4	296	295
H= 12, L= 12		
0	254	263
1	51*	-66
2	65	-70
3	196	-202
4	70	77
H= 13, L= 12		
0	739	-788
1	74	-112
2	340	-343
3	52*	-22
H= 14, L= 12		
0	98	115
1	478	-492
2	63	-44
3	358	-370
H= 15, L= 12		
0	127	-124
1	141	170
2	108	-116
3	87	-81
H= 16, L= 12		
0	76	84
1	58*	49
2	127	-138
H= 17, L= 12		
0	176	185
1	51*	-6
H= 0, L= 13		
2	54*	-20
4	72	-72
H= 1, L= 13		
1	181	-177
2	121	-126
3	106	108
4	39*	22
H= 2, L= 13		
1	131	128
2	87	91
3	42*	-51
4	39*	26
H= 3, L= 13		
1	201	-213
2	74	76
3	222	219
4	39*	16
H= 4, L= 13		
1	169	168
2	45*	-44
3	89	114
4	112	131
H= 5, L= 13		
1	222	-223
2	54	-41
3	68	74
4	67	-51
H= 6, L= 13		
1	135	-136
2	49*	21
3	150	-217
4	66	-46
H= 7, L= 13		
1	218	-208
2	86	-77
3	55*	-24
4	56	-85
H= 8, L= 13		
1	68	-50
2	48*	3
3	62	75
4	65	80
H= 9, L= 13		
1	246	-251
2	48*	52
3	169	171
H= 10, L= 13		
1	49*	0
2	106	-110
3	125	117
H= 11, L= 13		
1	56	-19
2	48*	-15
3	69	77
H= 12, L= 13		
1	66	-81
2	60	-40
3	100	-110
H= 13, L= 13		
1	90	-86
2	80	-110
H= 14, L= 13		
1	100	114
H= 0, L= 14		
0	544	557
2	646	626
H= 1, L= 14		
0	159	173
1	285	280
2	337	354
3	186	181
H= 2, L= 14		
0	264	-274
1	375	390
2	40*	1
3	447	453
H= 3, L= 14		
0	167	179
1	194	-186
2	326	343
3	266	-252
H= 4, L= 14		
0	585	-558
1	42*	-16
2	351	-341
3	43	52
H= 5, L= 14		
0	395	-402
1	157	-158
2	192	-204
3	364	-360
H= 6, L= 14		
0	146	162
1	471	-490
2	262	280
3	295	-299
H= 7, L= 14		
0	477	-489
1	199	199
2	277	-267
H= 8, L= 14		
0	166	160
1	45*	-10
2	266	260
H= 9, L= 14		
0	444	463
1	260	252
2	254	259
H= 10, L= 14		
0	174	-164
1	421	446
2	45*	15
H= 11, L= 14		
0	258	271
1	302	-309
H= 1, L= 15		
1	124	122
H= 2, L= 15		
1	37*	29
2	37*	5
H= 3, L= 15		
1	114	108
H= 4, L= 15		
1	127	114
H= 5, L= 15		
1	97	82
H= 0, L= 16		
0	622	620

Table 4. *Least-squares planes*

Equations are expressed in the form $Ax+By+Cz=D$, where D is expressed in Å, and x, y and z are fractional coordinates. The method of Schomaker, Waser, Marsh & Bergman (1959) was used to calculate the least-squares planes.

Plane		A	B	C	D
1	W(1), N(1), O(4a)	−15·565	−6·002	3·751	−8·471
2	N(1), O(1), O(4a), W(2)	2·619	1·360	13·321	2·121
3	O(1), N(2), C(1), C(2)	14·132	−0·420	11·361	5·639
4	O(2), N(3), C(3), C(4)	12·806	−6·890	−1·664	3·818
5	O(3), O(4), C(5), C(6)	16·102	6·103	2·423	3·598

	$\Delta(1)$		$\Delta(2)$		$\Delta(3)$		$\Delta(4)$		$\Delta(5)$
Zn	−0·052 Å	Zn	0·348 Å	O(1)	−0·008 Å	O(2)	−0·005 Å	O(3)	0·002 Å
N(1)	0·000	N(1)	−0·367	N(2)	−0·007	N(3)	−0·004	O(4)	0·002
O(4a)	0·000	O(1)	0·367	C(1)	−0·006	C(3)	−0·003	C(5)	0·001
W(1)	0·000	O(4a)	−0·338	C(2)	0·021	C(4)	0·012	C(6)	−0·005
O(1)	2·107	W(2)	0·337	C(3)	−0·176	C(5)	−0·099		
W(2)	−2·168	W(1)	2·290	Zn	0·110				
O(3a)	−0·184			N(1)	0·395				

chelation. However, no such simple explanation is available in the case of the Zn–W(2) bond. The elongations are approximately 7 and 9 per cent. Molecular orbital calculations by Craig, Maccol, Nyholm, Orgel & Sutton (1954) predict an elongation of this magnitude for d^0 and d^{10} trigonal bipyramids.

The chelate ring Zn, N(1), C(1), C(2), O(1) is nonplanar and has the envelope conformation (Table 4, plane 3).

Bond distances and bond angles

The bond distances and bond angles in the peptide are shown in Figs. 3 and 4. These can be compared

Table 5. *Bond angles in metal environment*

(*a*) Comparison of metal coordinations in Zn(ggg) $(H_2O)_4(SO_4)_{1/2}$ and Cu(ggg)Cl$(H_2O)_{1\,1/2}$ (Freeman, Robinson & Schoone, 1964) with values for the 'ideal trigonal bipyramid and square pyramid (Gillespie, 1963).

Trigonal bipyramid	Observed Zn(ggg) $(H_2O)_4(SO_4)_{1/2}$		Observed Cu(ggg)Cl$(H_2O)_{1\,1/2}$		Square pyramid
120°	*W*(1) Zn N(1)	121·3°	*W*(5) Cu N(1)	90·9°	100°
90	*W*(1) Zn O(1)	94·1	*W*(5) Cu O(1)	93·6	100
90	*W*(1) Zn *W*(2)	86·6	*W*(5) Cu Cl	104·5	100
120	*W*(1) Zn O(4*a*)	100·0	*W*(5) Cu O(3)′	90·8	100
90	N(1) Zn O(1)	79·5	N(1) Cu O(1)	84·2	88·4
90	N(1) Zn *W*(2)	95·7	N(1) Cu Cl	91·8	88·4
90	*W*(2) Zn O(4*a*)	90·4	Cl Cu O(3)′	92·9	88·4
90	O(1) Zn O(4*a*)	94·6	O(1) Cu O(3)′	90·4	88·4
120	N(1) Zn O(4*a*)	138·4	N(1) Cu O(3)′	174·5	160
180	O(1) Zn *W*(2)	174·1	O(1) Cu Cl	161·5	160

Average deviation from trigonal bipyramid is 7·4° for Zn(ggg) $(H_2O)_4(SO_4)_{1/2}$ and 16·0° for Cu(ggg)Cl$(H_2O)_{1\,1/2}$.
Average deviation from square pyramid is 10·1° for Zn(ggg) $(H_2O)_4(SO_4)_{1/2}$ and 5·9° for Cu(ggg)Cl$(H_2O)_{1\,1/2}$.

(*b*) Other angles of the metal coordination.

Standard deviations are 0·3°.

Zn N(1) C(1)	112·6°	O(3*a*) Zn *W*(2)	90·6°
Zn O(1) C(2)	112·5	O(3*a*) Zn N(1)	87·8
Zn O(4*a*) C(6*a*)	112·4	O(3*a*) Zn O(1)	91·3
Zn O(3*a*) C(6*a*)	74·1	O(3*a*) Zn O(4*a*)	51·0
		O(3*a*) Zn *W*(1)	150·7

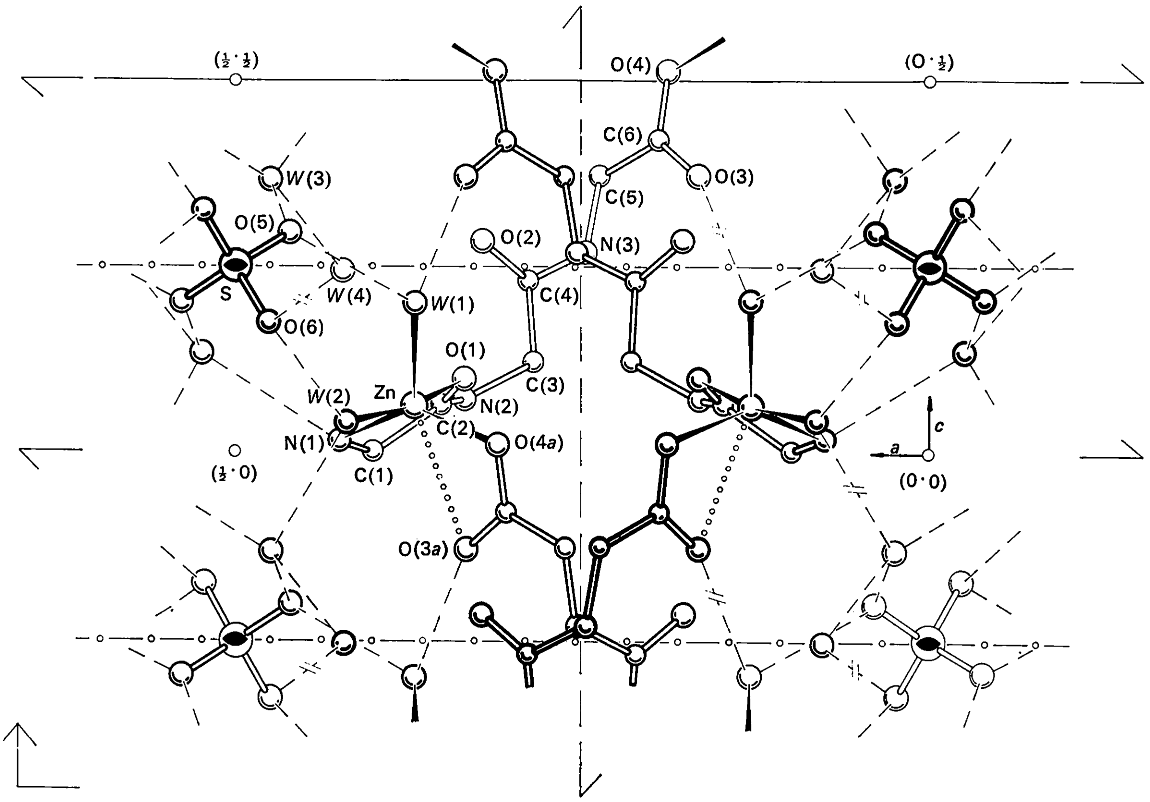

Fig. 1. Projection of the structure down the *b* axis.

with the average values for free peptides compiled from previous structure determinations (Marsh & Donohue, 1967). The N–C$^{\alpha}$–C′ angles in the second and third glycyl residue, *i.e.* the N(2)–C(3)–C(4) (114·8°) and the N(3)–C(5)–C(6) (115·9°) angles are larger than the normal value (111°). Only two other bond angles deviate by more than 3σ from the average values, C(3)–C(4)–N(3) (113·6°) and O(2)–C(4)–C(3) (123·6°) in comparison with 116° and 120·5°, respectively. It is probable that these last two deviations are related to the unusually short C(4)–O(2) distance (1·216 Å), which is the only bond distance which differs by more than 3σ from the average values. Marsh & Donohue (1967) list 1·24 Å for the C′–O distance in a peptide group. The same distance in the gggCuCl structure (Freeman, Robinson & Schoone, 1964) is also short; 1·19 Å. It is interesting to note that in both structures the O(2) atom is neither hydrogen bonded nor complexed to a metal ion.

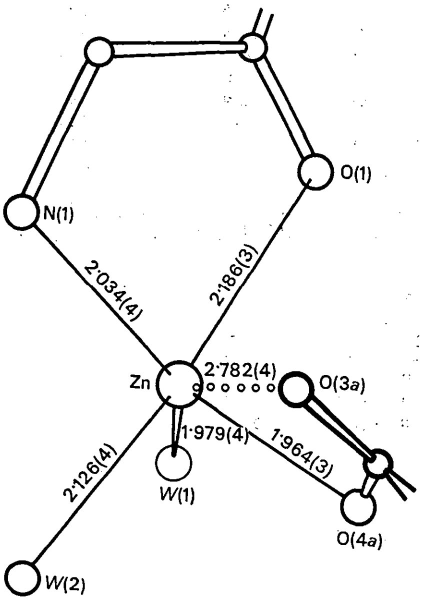

Fig. 2. Zinc surrounding. Standard deviation for the last digit is given in parentheses.

Both peptide groups are slightly non-planar, which can be seen in the deviations of C(3) and C(5) from the least-squares planes 3 and 4, respectively (Table 4). The carboxylate group is planar. A better description of the peptide backbone is possible by calculating the conformational angle for each bond (Table 6). The conventions suggested by Edsall, Flory, Liquori, Nemethy, Ramachandran & Scheraga (1966) have been used to calculate these angles. The values of φ and ψ for each residue lie in the allowed regions of the conformational maps calculated for glycyl residues (Ramachandran, 1968).

Table 6. *Conformational angles*

Residue	φ	ψ	ω	N–C$^{\alpha}$–C′
1	—	341°	355°	110·1°
2	272	338	358	114·8
3	71	358	—	115·9

The sulfate ion is quite regular. The bond distances are 1·455 ± 0·004 Å for both S–O distances. The bond angles are 109·4° for O(5)–S–O(6), 110·8° for O(5)–S–O(5*b*) and 108·2° for O(6)–S–O(6*b*). The covalent bond distances involving the hydrogen atoms are not tabulated because the hydrogen atom locations were not refined. In addition the hydrogens attached to *W*(3) and *W*(4) had to be located from peaks which

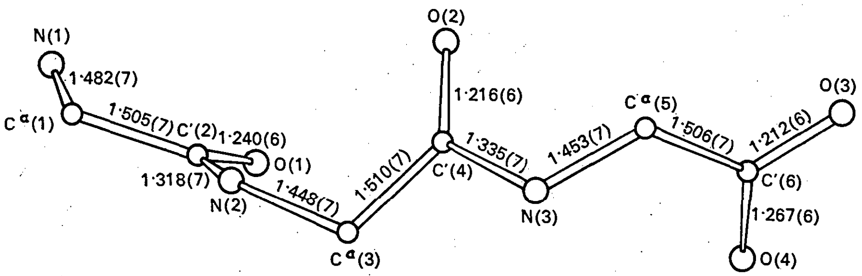

Fig. 3. Atom numbering and bond distances. Standard deviation for the last digit is given in parentheses.

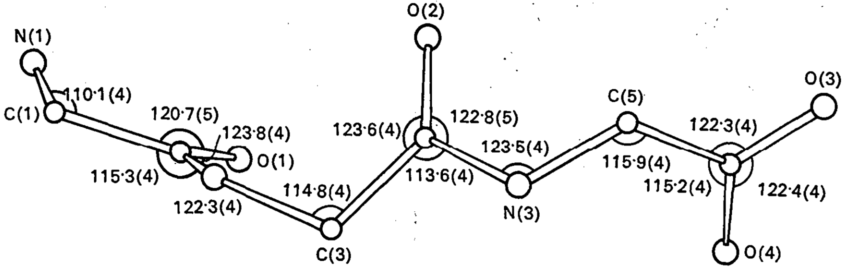

Fig. 4. Bond angles. Standard deviation for the digit is given in parentheses.

were not well defined. Consequently some of the distances involving those particular four hydrogens deviate from literature values. The worst values are a covalent bond distance of 1·4 Å for *W*(3)–H(2) and a van der Waals contact of 1·6 Å between the H(1) atoms of *W*(3) and *W*(4).

Packing and hydrogen bonding

The peptides form infinite chains through the complex bonds with the zinc ions (Fig. 1). Two separate infinite chains form an extended, two-stranded coil around the twofold screw axis parallel to the *c* axis. The coils pack along the *b* glide plane thus forming an infinite sheet of protein-like material parallel to the *bc* plane. The sulfate ion is located on the twofold axis parallel to the *b* axis. This ion and two water molecules, not bonded to the zinc ion, form an infinite column. This aqueous column does not penetrate the proteinlike sheet.

The crystallographically independent half sulfate ion and the water molecules, *W*(3) and *W*(4), form an infinite spiral of hydrogen bonds and covalent bonds (Fig. 5): O(5)–*W*(3), *W*(3)–*W*(4), *W*(4)–O(6), O(6)–S and S–O(5). This spiral is connected to the rest of the crystal structure through the hydrogen bonds: *W*(4)–O(2), O(6)–*W*(2), *W*(3)–*W*(2), O(5)–*W*(1), *W*(3)–N(1). The zinc ligand *W*(1) is the donor in another hydrogen bond to O(3). Associated with the atoms in this bond are the two largest degrees of vibrational anisotropy found in this structure. The major and minor axes of the vibration ellipsoids of *W*(1) and O(3) respectively 10·2 and 2·3, and 11·1 and 2·8 Å^2, while all other atoms show little anisotropy in their temperature movement (Nicholas, 1968). The main axis of both ellipsoids is roughly perpendicular to the hydrogen bond. These bonds are all shown in Fig. 1 and the distances and angles involved are given in Table 7. As expected *W*(1) and *W*(2) each donate two hydrogen bonds, while *W*(3) accepts two and donates two bonds.

Table 7. *Intermolecular distances and angles*

Hydrogen bond distances and angles

Donor (X)	Acceptor (Y)	Distance (X···Y)	Angle (X–H···Y)
W(3)	*W*(4)	2·723 Å	142°
W(3)	O(5)	2·768	143
W(4)	O(6*c*)	2·757	167
W(1)	O(5)	2·700	154
W(1)	O(3*d*)	2·598	167

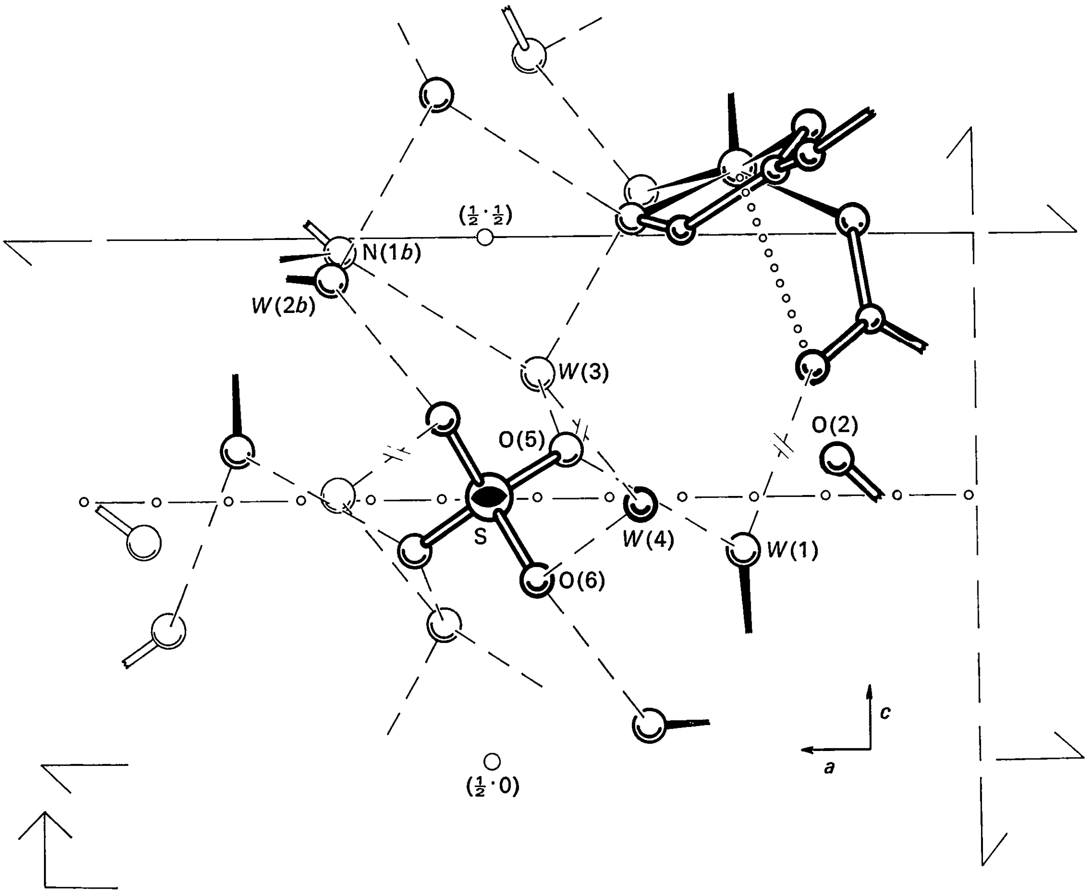

Fig. 5. The surrounding of the sulfate ion.

Table 7 (*cont.*)

Donor (X)	Acceptor (Y)	Distance (X···Y)	Angle (X–H···Y)
W(2)	O(6)	2·698	156
W(2)	*W*(3*e*)	2·659	168
N(1)	*W*(3*b*)	3·053	155
W(4)	O(2)	2·858*	90
N(1)	O(5*e*)	3·153†	171
N(2)	*W*(2*c*)	3·166†	161
N(3)	O(1*f*)	3·152†	170

* Possibly not a hydrogen bond but a van der Waals contact.

† Weak hydrogen bond.

Bond angles between hydrogen bonds or covalent bond and hydrogen bond

C(1) N(1) *W*(3*b*)	113°	O(5) *W*(1) O(3*d*)	86°
C(1) N(1) O(5*e*)	96	O(6) *W*(2) *W*(3*e*)	109
C(3) N(2) *W*(2*c*)	114	*W*(4) *W*(3) O(5)	105
C(5) N(3) O(1*f*)	127	O(2) *W*(4) O(6*c*)	103

Van der Waals contacts less than 3·50 Å

O(2) N(3*d*)	3·44 Å	C(3) O(4*h*)	3·22 Å
O(2) C(5*d*)	3·34	C(5) O(3*f*)	3·48
N(2) O(4*g*)	3·19	*W*(3) *W*(3*b*)	3·43
C(3) O(4*g*)	3·17	*W*(3) *W*(4*b*)	3·46
C(3) O(1*f*)	3·37		

The letters in parentheses indicate that one of the following operations has to be applied to the coordinates given in Table 1.

(*a*)	$-x+\frac{1}{2}$	$-y+\frac{1}{2}$	$z-\frac{1}{2}$
(*b*)	$-x+1$	y	$-z+\frac{1}{2}$
(*c*)	x	$y-1$	z
(*d*)	$-x+\frac{1}{2}$	$y+\frac{1}{2}$	z
(*e*)	x	$-y+1$	$z-\frac{1}{2}$
(*f*)	$-x+\frac{1}{2}$	$y-\frac{1}{2}$	z
(*g*)	$-x+\frac{1}{2}$	$-y-\frac{1}{2}$	$z-\frac{1}{2}$
(*h*)	x	$-y$	$z-\frac{1}{2}$

There is one incongruous feature. This is the apparent lack of hydrogen bonding of *W*(4). The atoms *W*(4) and O(2) are 2·86 Å from each other and the vector between the atoms is directed such that hydrogen bond formation could occur without disrupting the other bonds formed by *W*(4); however, the difference map shows no peak between *W*(4) and O(2). The peak identified as H(1) *W*(4) does not allow for hydrogen bond formation with O(2) (the angle *W*(4)–H(1)–O(2) is 90°), and the vector *W*(4)–H(1) is not directed to another potential hydrogen bond acceptor. It was previously stated that the hydrogens attached to *W*(3) and *W*(4) could not be located with certainty and it is therefore not possible to dismiss the existence of an hydrogen bond between *W*(4) and O(2). N(1), N(2) and N(3) each form a weak hydrogen bond (3·15 – 3·17 Å). In the latter three cases the hydrogen atoms are directed toward the acceptor atoms. These bonds are not indicated in Fig. 1. The hydrogen bonding is summarized in Table 7. All other intermolecular contacts less than 3·50 Å are also given in Table 7.

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