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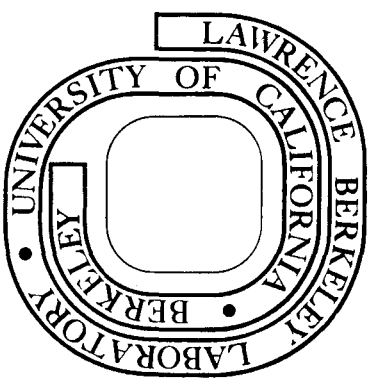

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PREPARATION AND CRYSTAL STRUCTURE OF URANIUM(IV) BOROHYDRIDE DI-TETRAHYDROFURAN, $U(BH_4)_4 \cdot 2(OC_4H_8)$[1]

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Abstract

Uranium (IV) borohydride reacts quantitatively with tetrahydrofuran to form $U(BH_4)_4 \cdot 2(OC_4H_8)$, a pale green solid which has been characterized by single crystal x-ray diffractometry. This air and moisture sensitive compound can be sublimed at 50-60°C and 10^{-5}mm. $U(BH_4)_4 \cdot 2(OC_4H_8)$ is orthorhombic with $\underline{a}$ = 7.134(4) Å, $\underline{b}$ = 11.311(6) Å, $\underline{c}$ = 10.422(7) Å and Z = 2, ($d_{\underline{x}}$ = 1.74 g cm^{-3}). The space group is Pnc2 or Pncm. For 789 data where $I > 2\sigma$ the structure refined to R = 0.027, and R_w = 0.032 in space group Pncm, with disorder in both the THF and boron positions. The complex is a monomer with a distorted octahedral arrangement about the uranium of four

borohydride groups and two THF molecules. The U-O distance is 2.47(1) Å. The U-B distance of 2.56(4) Å is characteristic of the triple hydrogen bridge bonds found in uranium (IV) borohydride. A configuration is assigned the hydrogen atoms which places 12 of them at the corners of an hexagonal antiprism, capped by the two oxygen atoms of THF, giving the uranium atom 14 nearest neighbors. This compound is the only known uranium borohydride complex which is monomeric in the solid state.

Introduction

Interest in the preparation and characterization of new volatile actinide compounds prompted us to reinvestigate the chemistry of uranium (IV) borohydride. In a study of Lewis base derivatives of $U(BH_4)_4$,[2] we synthesized a number of new volatile compounds. The methyl and ethyl etherates of $U(BH_4)_4$ have been characterized as monoetherates which form linear polymers in the crystalline state.[3] We report here on the tetrahydrofuran complex of $U(BH_4)_4$ which is a dietherate and monomeric in the crystalline state.

Experimental Section

Materials and Chemical Techniques. $U(BH_4)_4$ was prepared by the method of Schlesinger and Brown[4] and purified by sublimation at 30-40°C and 10^{-5} mm. Tetrahydrofuran (Aldrich) was doubly distilled from Na/benzophenone under argon. All manipulations were performed in mercury, oil and grease-free Pyrex high vacuum lines or in argon-

filled dry boxes.[5]

Preparation of $U(BH_4)_4 \cdot 2THF$. In a typical preparation, 0.206g $U(BH_4)_4$ (0.69 mmole) was sublimed into a 50 ml trap at -78°. THF(1.76g) was condensed on top of the $U(BH_4)_4$. The trap was sealed and warmed to 20°C with shaking. The $U(BH_4)_4$ first lightened in color and then dissolved to give a green solution. The excess THF was removed by pumping at -45° for 4 hrs. The remaining green solid was quantitatively recovered by sublimation in bulk at 50-60°C to a -78° cold finger. The pale green crystals (mp. 122 dec., sealed capillary) decompose slowly in air but explode into flames when touched with a drop of water. It has been stored under argon or in vacuum for several months with only minor decomposition. Additional physical and spectroscopic data will appear elsewhere.[2]

Crystal Growth. Crystals taken directly from the sublimer and sealed in capillaries were found to be unsuitable for study. Instead, several crystals were ground in a mortar and pestle, and poured into a capillary drawn from a 14/35 quartz joint. An adapter containing a stopcock to trap argon over the sample was placed on the loaded joint. This assembly was removed from the dry box and connected to a high vacuum line. The capillary was cooled to -78°, evacuated, and sealed. Crystals were grown inside the capillary by slow sublimation (6-24 h) using a microscope lamp focused on a colored card beneath the capillary as the heat source. After several dozen attempts a pale green transparent tabular crystal was obtained.

Data Collection, Reduction, and Refinement. The crystal was placed on a Picker FACS-I automatic diffractometer equipped with graphite monochromated MoKα radiation, (λ $K\alpha_1$ 0.70926 Å) for study. Cell dimensions obtained from carefully centered settings on the $K\alpha_1$ peaks of the 800, $\bar{8}00$, 0 12 0, 0 $\overline{12}$ 0, 0 0 12 and 00 $\overline{12}$ reflections are $\underline{a}$ = 7.134(4) Å, $\underline{b}$ = 11.311(6) Å and $\underline{c}$ = 10.442(7) Å; for two molecules in the unit cell the density is 1.74 g cm^{-2}. The widths of the omega scans at half-height were typically 0.1°. The pattern of intensities showed a very pronounced pseudo A-centering which was indicative of the uranium atom on the origin. Intensity data were collected with the θ-2θ scan method where each reflection was scanned from 0.55° before the $K\alpha_1$ to 0.55 beyond the $K\alpha_2$ peak; 4 second backgrounds were measured at each end of the scan. All of the data with zero and positive $\underline{k}$ indices was collected to a 2θ angle of 30°, and only the A-centered data were collected from 30° to 60°; beyond 30° the weak non A-centered reflections were unobservable. The temperature was 23.5 ± 1.0°C. Three standards were measured after each 200th reflection, and no observable decay in these standards was noted. The formulas used to process the data were presented in the Supplementary Materials. An ignorance factor of p = 0.04 was applied. The 3763 measured intensities resulted in 915 unique reflections; 160 of these were the weak non-A centered type, half of which had intensities less than σ(I).

An absorption correction was made,[6] $\mu = 92\ cm^{-1}$, and transmission factors varied from 3.3 to 6.4. The crystal was described by 6 crystal faces, 011, $0\bar{1}\bar{1}$, $01\bar{1}$, $0\bar{1}1$, 100 and $\bar{1}00$. The crystal dimensions were .22 × .27 × 0.13 mm in the 011, $01\bar{1}$ and 100 directions respectively; the crystal volume was 0.0077 mm^3. Several azimuthal scans in diverse regions of reciprocal space were performed to test the validity of the absorption correction and to make minor adjustments on the crystal dimensions.

Space group and disorder. Our data are ambiguous with respect to the space group. The diffraction symmetry and the reflections which are systematically absent are consistent with either Pncm[7] (centric) or Pnc2 (non-centric). A search for Bijvoet differences revealed none which were significantly larger than the differences between equivalent reflections, but for this crystal the differences are expected to be small. For the reflections with k+l even, which constitute most of the data, the centric uranium structure and the at least nearly centric tetrahydrofuran structure dominate the structure factor. For the reflections with k+l odd, uranium makes no contribution, and its anomalous dispersion cannot give rise to a Bijvoet difference.

As the structure determination proceeded, it became clear that there was disorder of the conformation of the tetrahydrofuran ring, as is generally true of this molecule in crystals.[8-15] In space group Pncm, the THF ring lies in the mirror plane, and the disorder consists of displacements of atoms from this plane equally in either direction. Space group Pnc2 permits unsymmetrical displacements, but

several attempts at refinement of various models failed to yield any significant improvement over centric models.

Fourier maps calculated for Pncm require the boron atoms to be distributed between pairs of sites, about 0.76 Å apart. The non-centric space group permits assignment of boron atoms to half these sites in an ordered manner, described below, which corresponds to a molecular structure with very plausible packing of the hydrogen atoms. For this reason we prefer Pnc2 as a description of the structure. However, we carried out the final refinement in space group Pncm to avoid the several ambiguities inherent in the unsymmetrical disorder models and to gain a much better rate of convergence. The resulting coordinates for boron atoms should be nearly the same for either space group because the high-angle data are exclusively of the type k+l even. For these reflections, the magnitudes of a structure factor, calculated for a non-centric structure and for a superposition of that structure and its inverse, are rarely different by 1% and never by as much as 2% for the models which we refined. Several non-centric refinements, with different models for the THF, gave coordinates for boron within one standard deviation of those reported here.

Structure refinement. A trial structure, obtained from a three dimensional Patterson map, was refined by full-matrix least squares in the centric space group Pncm. Anisotropic thermal parameters were applied first to the uranium atom only. Because the observed values of the larger intensities were consistently below the calculated values, an empirical extinction correction was applied where $F_{cor} = F_{obs}(1 + 5 \times 10^{-7} I)$; F_{corr} and F_{obs} are the corrected and

observed structure factors and I is the observed intensity. The largest correction factor was 1.19. The $R = \Sigma||F_o|-|F_c||/\Sigma|F_o$ at this stage was 0.037 for 789 data with $F^2 > 2\sigma$. The planar THF molecule had chemically unreasonable bond distances which suggested disorder. A difference Fourier map showed disorder not only in the THF molecule but also in the boron positions. Efforts to resolve the disorder into a double set of discrete atoms for the THF molecule failed and the THF molecule was refined with anisotropic thermal parameters. The boron atoms were refined as two half atoms with isotropic thermal parameters. Hydrogen atoms were not observed and not included in the refinement. The final R factor was 0.027 for 789 data where $I > 2\sigma$, and 0.037 for all 915 data. $R_w = (\Sigma w|F_o|-|F_c|)^2/\Sigma wF_o^2)^{1/2}$ was 0.032. The standard deviation of a reflection of unit weight was 1.16.

The atomic parameters are given in Table I, and the distances and angles in Tables II and III.

DISCUSSION

The structure consists of monomeric units of $U(BH_4)_4 \cdot 2(OC_4H_8)$. The borohydride and THF ligands form a distorted octohedron about the uranium atom; see Fig. 1. The U-B distance is characteristic of the "terminal" borohydride found in $U(BH_4)_4$[16] where three hydrogen atoms from the borohydride ion contact the uranium atom. There are fourteen neighbors to the uranium atom, consisting of twelve hydrogen and two oxygen atoms. Fourteen coordination is also observed in $U(BH_4)_4$,[16] $U(BH_4)_4 \cdot O(CH_3)_2$,[3] and $U(BH_4)_4 \cdot O(C_2H_5)_2$.[3]

The BH_4^- bonding in this molecule is similar to that found for $U(BH_4)_4$ in the gaseous phase by analysis of infrared spectra[17,18] in that each boron has triple hydrogen bridges to a single uranium atom. Upon subliming, $U(BH_4)_4$ converts from the 14-coordinate polymeric solid-state structure to the 12-coordinate monomeric structure. $U(BH_4)_4 \cdot 2(OC_4H_8)$ is the only uranium borohydride complex known to us which is monomeric in the solid state. However, $U(BH_4)_4$ has the monomeric structure in dilute mixed crystals with $Zn(BH_4)_4$ or $Hf(BH_4)_4$, which themselves are monomeric.[19]

The hydrogen atoms can be fit around the uranium atom very neatly in the configuration shown in Fig. 2. This arrangement places the 12 hydrogen neighbors at the corners of an hexagonal antiprism, with oxygen atoms capping each hexagonal face (Fig. 3). If these figures are constructed with tetrahedral BH_4^- ions and with oxygen equidistant from its six nearest hydrogen neighbors, one calculates O-U-B angles of 83° and 97°, with boron atoms alternately above and below the equatorial plane (oxygen atoms at the poles). The calculations in space group Pnc2 give just this conformation, with O-U-B angles 81°, 82°, 99°, and 98°. The O-U-B angles can be fit exactly with very minor adjustments of the hydrogen positions. One calculates other angles O-U-H = 68°, H-U-H = 49° (hydrogen atoms in the same borohydride ion) and H-U-H = 57° or 58° (hydrogen atoms in neighboring ions). These values are well within the ranges for corresponding ones observed in the structure of the diethylether complex of uranium borohydride.[3] The bicapped hexagonal antiprism which we propose here is the same

polyhedron which was chosen as an approximation to describe the 14 hydrogen neighbors of uranium in the structure of $U(BH_4)_4$.[16]

The above structure for the complex is that corresponding to space group Pnc2. In the centric group these complexes are mixed randomly with others which are inverted through the uranium atom.

The disorder of the THF molecule seems to be independent of the possible disorder of the borohydride configuration. The bond lengths are calculated shorter than is chemically reasonable, and large thermal parameters indicate significant displacements from the planar structure imposed by the model. Both of these characteristics are commonplace of this molecule as observed in many other crystals,[8-15] and we have been unable to find an example of a crystal structure in which it exists with good bond lengths and small amplitudes of motion. It is a flexible molecule with many conformations of nearly equal energy, and it has a mode of motion, referred to as "pseudorotation," in which a flexing of the ring carries angular momentum.[20] We made several attempts to describe it as a mixture of two conformations, both in the centric and non-centric space groups, without finding any significantly better model. Evidently more conformations are important, but the diffraction data do not justify a more elaborate model.

The monomer units pack in the crystal in a manner which corresponds nearly to a simple cubic lattice. Each molecule has six neighbors, two at 7.13 Å and four at 7.69 Å (measured between uranium atoms), in directions parallel to a and to the b c diagonals.

Supplementary Material Available: Data processing formulas and listing of observed structure factors (5 pages). Ordering information is given on any current masthead page.

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(7) Space group Pncm is a non-standard setting of space group No. 53. The general positions are: $\pm(x,y,z;\ \bar{x},\ 1/2 + y,\ 1/2 + z;\ x,\ 1/2 - y,\ 1/2 + z;\ \bar{x},\bar{y},z)$.

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Table I. Positional and Thermal Parameters[a]

Atom	x	y	z
U	0	0	0
O	.1618(9)	.1931(6)	0
C(1)	.077(2)	.313(1)	0
C(2)	.223(3)	.399(1)	0
C(3)	.397(2)	.340(1)	0
C(4)	.362(2)	.217(1)	0
B(1)	.242(3)	-.046(2)	.179(2)
B(2)	.197(2)	-.106(1)	.164(2)

Atom	B_{11} or B	B_{22}	B_{33}	B_{12}
U	3.13(1)	2.93(1)	3.79(2)	-.04(5)
O	3.6(3)	2.8(2)	6.1(4)	-.1(2)
C(1)	5.5(5)	3.6(5)	11.9(12)	1.0(4)
C(2)	6.4(8)	3.6(5)	21.5(24)	-.2(5)
C(3)	5.6(7)	6.2(7)	11.5(12)	-2.6(6)
C(4)	3.9(6)	4.4(6)	30.8(31)	-1.5(5)
B(1)	5.0(3)			
B(2)	4.3(3)			

[a]The anisotropic temperature factor has the form $\exp(-0.25(B_{11}h^2a^{*2}+2B_{12}hka^*b^*+\ldots))$. Because of the mirror plane, $B_{13} = B_{23} = 0$.

The isotropic temperature factor has the form $\exp(-B(\sin\theta/\lambda)^2)$.

Table II. Interatomic Distances (Å)

U	- 2 O	2.47(1)
U	- 4 B(1)[a]	2.60(2)
U	- 4 B(2)[a]	2.52(2)
B(1)	- B(2)	.76(2)
O	- C(1)	1.48(2)
O	- C(4)	1.45(2)
C(1)	- C(2)	1.43(2)
C(2)	- C(3)	1.41(2)
C(3)	- C(4)	1.42(2)

[a] B(1) and B(2) are the two positions occupied by the disordered boron atoms in the structure.

Table III. Angles (deg.)

O	- U	- O^{i}	180.0
O	- U	- B(1)[a]	82.4(5)
O	- U	- B(2)[a]	80.8(4)
B(1)	- U	- $B(1)^{iii}$	87.9(9)
$B(2)^{i}$	- U	- $B(2)^{ii}$	94.2(8)
B(1)	- U	- $B(2)^{ii}$	91.3(6)
B(1)	- U	- $B(2)^{i}$	163.0(5)
U	- O	- C(1)	128.1(7)
U	- O	- C(4)	128.4(7)
C(1)	- O	- C(4)	103.5(9)
O	- C(1)	- C(2)	109(1)
C(1)	- C(2)	- C(3)	108(1)
C(2)	- C(3)	- C(4)	108(1)
C(3)	- C(4)	- O	111(1)

Symmetry operations

i	$\bar{x}\ \bar{y}\ \bar{z}$
ii	$x\ y\ \bar{z}$
iii	$\bar{x}\ \bar{y}\ z$

[a] See footnote a in Table II.

FIGURE CAPTIONS

Fig. 1. Structure of the $U(BH_4)_4 \cdot 2THF$ molecule. The tetrahydrofuran rings, which are shown as planar, represent the average portions of the atoms. The boron atoms are shown in one conformation of the disorder model.

Fig. 2. Structure proposed for the hydrogen atoms around each uranium atom. A boron atom (not shown) is at the center of each tetrahedron of hydrogen atoms; the view on the right shows how the boron atoms are alternately to either side of the central plane.

Fig. 3. Coordination polyhedron for uranium. Hydrogen atoms, as shown in Fig. 2, are at the corners of the central hexagonal antiprism, while oxygen atoms are at the top and bottom vertices.

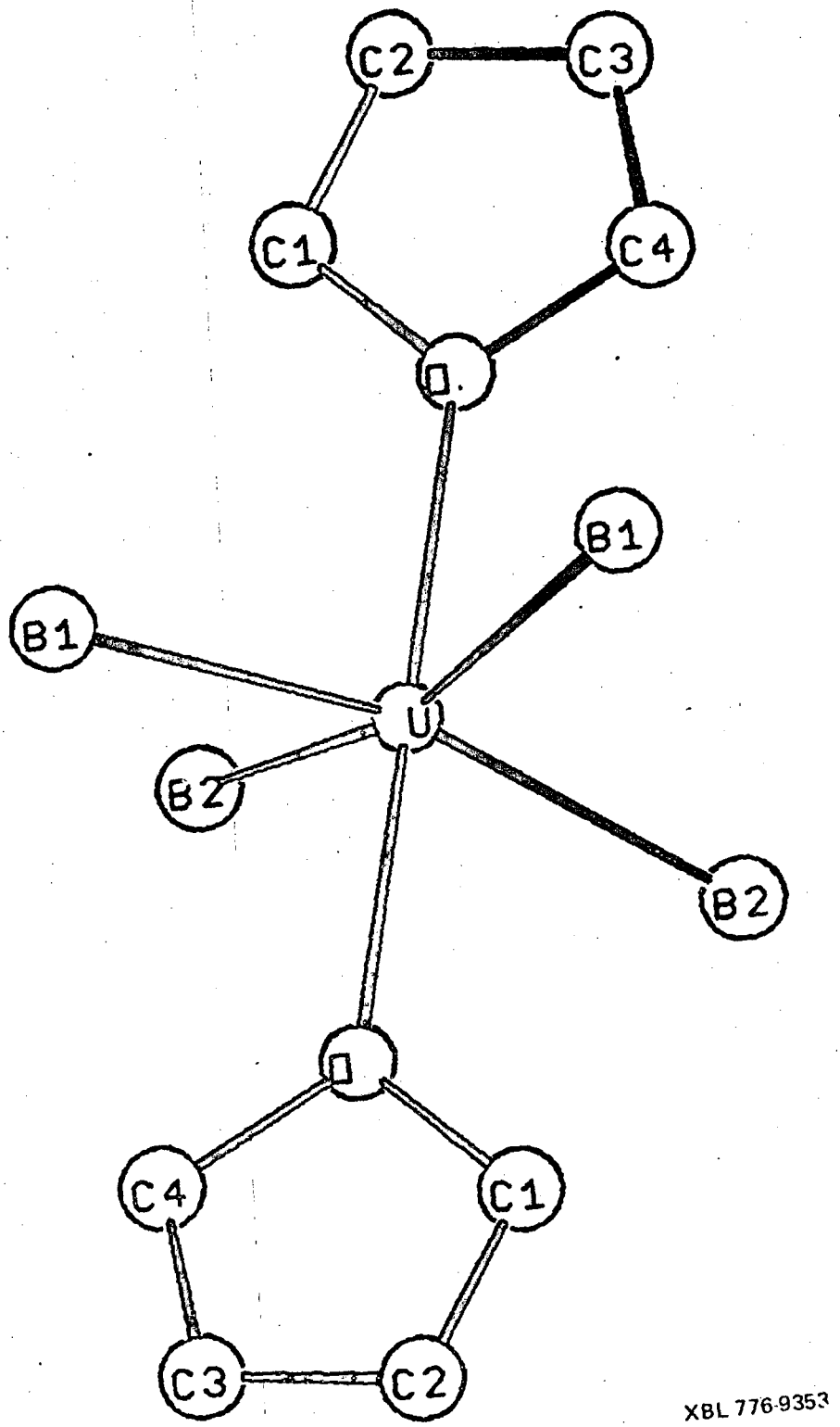

Fig. 1

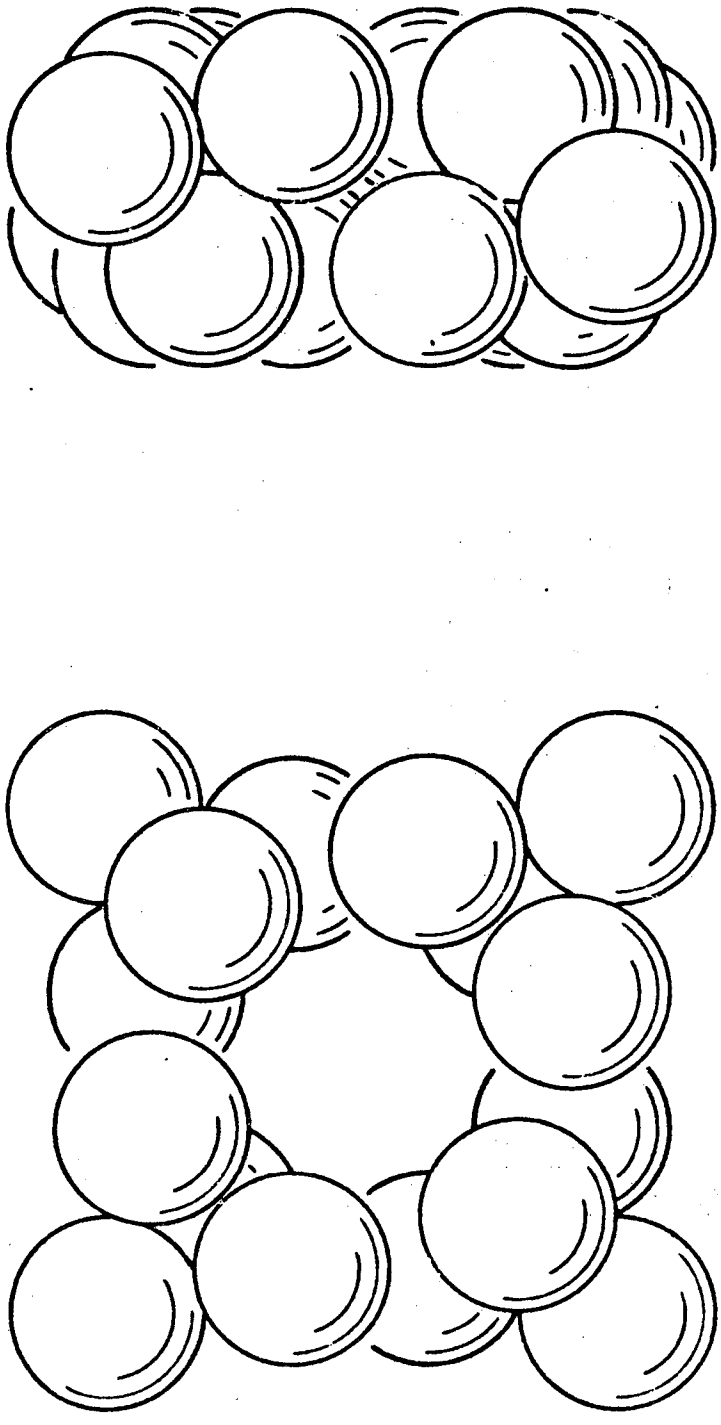

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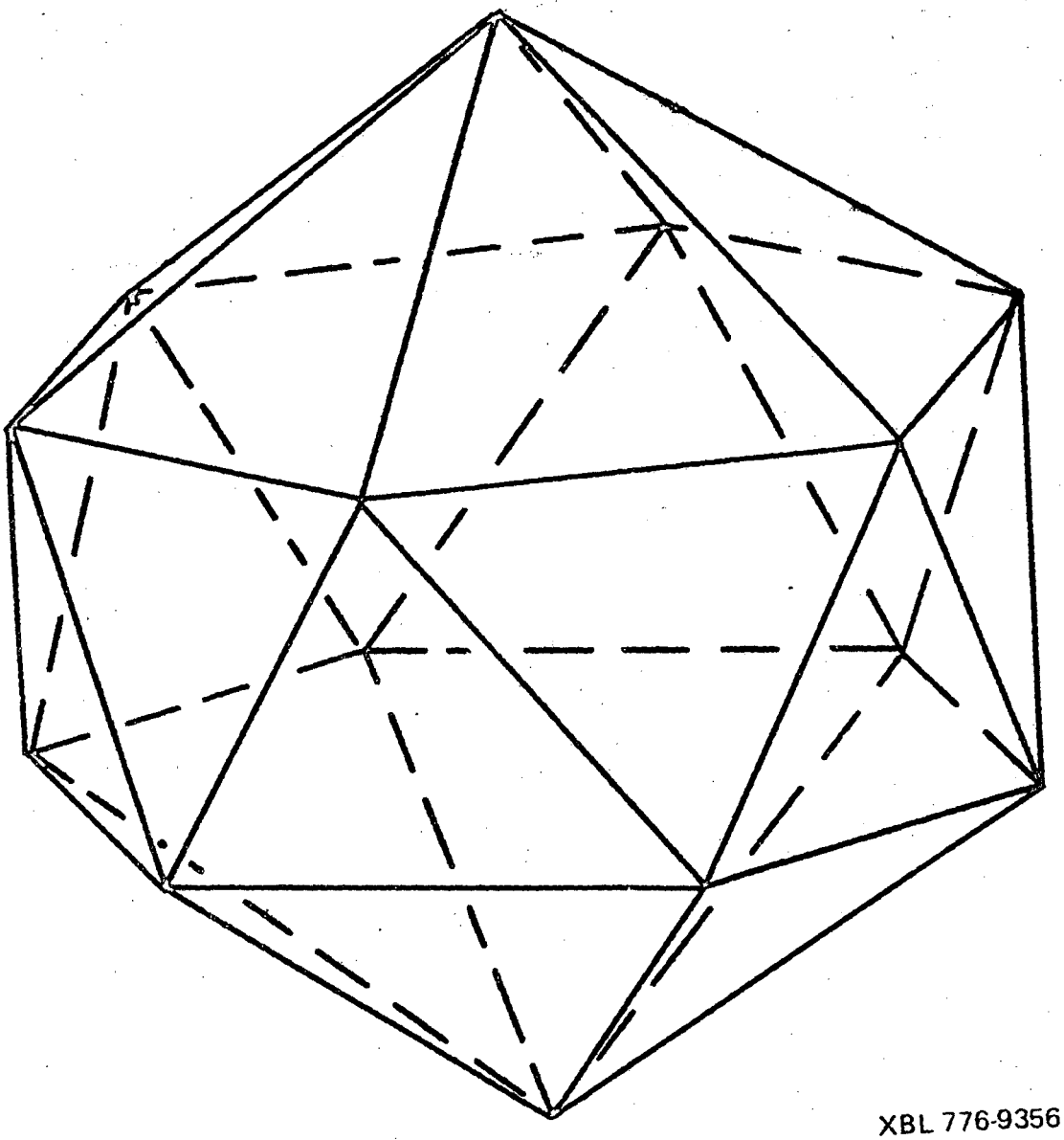
Fig. 2



XBL 776-9356

Fig. 3

DATA PROCESSING FORMULAE

$$I = C - (t_c/2t_b)(B_1+B_2)$$

$$\sigma(B) = \text{Max}[(t_c/2t_b)(B_1+B_2)^{\frac{1}{2}}, (t_c/2t_b)|B_1-B_2|]$$

$$\sigma(I) = [C + \sigma^2(B)]^{\frac{1}{2}}$$

$$F^2 = (D \cdot A/Lp)I$$

$$\sigma(F^2) = (D \cdot A/Lp)\sigma(I)$$

$$F_a^2 = \Sigma F^2/n$$

$$\sigma(F_a^2) = [\Sigma\sigma^2(F^2)/n]^{\frac{1}{2}}$$

When $S(F_a^2) > 4\sigma(F_a^2)$, $\sigma(F_a^2)$ is replaced by $S(F_a^2)$.

$$S(F_a^2) = [\Sigma|F^2-F_a^2|^2/n(n-1)]^{\frac{1}{2}}$$

$$\sigma(F_o^2) = [\sigma^2(F_a^2) + (pF_a^2)^2 + q^2]^{\frac{1}{2}}$$

$$F_o = (F_a^2)^{\frac{1}{2}}$$

$$\sigma(F) = F_o - [F_a^2 - \sigma(F_o^2)]^{\frac{1}{2}} \text{ when } \sigma(F_o^2) \leq F_a^2 \text{ or } [\sigma(F_a^2)]^{\frac{1}{2}} \text{ when } \sigma(F_a^2) > F_a^2$$

$$Lp = [\cos^2 2\theta_m + \cos^2 2\theta]/[\sin 2\theta\ (1 + \cos^2 2\theta_m)]$$

$$wtg = 1/\sigma^2(F)$$

C = counts recorded during a scan

I = individual raw intensity, background removed.

t_c = scan count time

t_b = background count time

B_1 = individual background count

$\sigma(B)$ = estimated standard deviation of the total background count

F = structure factor

D = decay correction; an empirically applied correction obtained from the fluctuations of the standard reflections.

A = absorption correction

Lp = Lorentz and polarization corrections

θ_m = monochromater angle

θ = crystal diffraction angle

S = scatter

a = average

q = additional uncertainty that affects the weak intensities

p = estimate of non-statistical errors

wtg = weighting factors in least squares

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL X 5.0)
U(8H4)4.2(OC4H8) F(0,0,0) = 1656

FOB AND FCA ARE THE OBSERVED AND CALCULATED STRUCTURE FACTORS.
SG = ESTIMATED STANDARD DEVIATION OF FOB. DEL = /FOB/ - /FCA/.
* INDICATES ZERO WEIGHTED DATA.

K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
H,L=	0,	0		H,L=	0,	6		3	79	18	-2	4	26	6	9	4	11	30	-12*
2	594	12	38	0	583	12	-9	5	82	16	6	5	511	10	14	5	290	6	-1
4	569	12	0	2	392	8	5	7	71	17	9	6	67	3	-2	6	22	50	-1*
6	570	12	37	4	352	8	-2	H,L=	0,	14		7	398	8	3	7	232	5	0
8	317	7	8	6	323	7	-3	0	63	15	-5	8	18	25	1*	9	180	4	1
10	332	7	-3	8	208	5	-8	2	62	16	0	9	265	6	-5	11	131	4	-3
12	173	5	-6	10	204	5	2	4	98	10	37	10	16	29	8*	13	91	5	1
14	120	9	8	12	125	5	2	H,L=	1,	0		11	204	5	-1	H,L=	1,		8
H,L=	0,	1		14	90	9	10	0	776	52	-65	13	146	4	2	0	299	6	-7
1	763	15	22	H,L=	0,	7		1	286	7	9	15	101	6	5	1	51	10	15
3	746	15	38	1	363	8	-9	2	735	19	26	H,L=	1,	4		2	272	6	-1
5	667	14	12	3	319	7	-5	3	160	11	33	0	596	12	-8	3	49	50	43*
7	382	8	15	5	304	6	3	4	631	13	32	1	138	4	-21	4	263	6	1
9	335	7	-8	7	221	5	-3	5	52	4	-14	2	534	11	20	5	0	55	-2*
11	221	5	7	9	193	5	1	6	487	10	-12	3	0	18	-3*	6	220	5	-3
13	144	5	-5	11	136	6	-3	7	39	9	9	4	505	10	13	8	166	4	2
15	119	10	5	13	85	9	-2	8	341	7	5	5	16	19	-7*	10	135	4	-3
H,L=	0,	2		H,L=	0,	8		9	59	9	5	6	403	8	-2	12	99	7	6
01	127	36	-8	0	325	7	2	10	300	6	-3	7	21	23	1*	H,L=	1,		9
2	415	9	-14	2	250	5	-3	12	183	6	-4	8	281	6	5	1	232	7	-6
4	583	12	15	4	266	6	5	14	121	6	6	9	15	36	-17*	2	0	55	-23*
6	577	12	36	6	241	5	-1	H,L=	1,	1		10	240	5	-3	3	220	5	3
8	299	6	-1	8	158	6	-6	1	769	16	8	12	157	5	2	4	20	32	18*
10	310	7	-4	10	145	7	-1	2	147	3	38	14	95	8	-7	5	202	5	-2
12	180	5	-4	12	112	6	15	3	646	13	9	H,L=	1,	5		7	155	6	-5
14	116	6	-3	H,L=	0,	9		4	137	4	11	1	520	11	12	9	120	5	-3
H,L=	0,	3		1	223	7	-2	5	523	11	-9	2	27	11	-0*	11	93	5	-1
1	474	11	-27	3	220	6	-1	6	91	3	-0	3	434	9	4	H,L=	1,		10
3	584	12	-9	5	217	6	-2	7	458	9	11	4	58	5	-1	0	198	5	1
5	610	12	13	7	165	6	4	8	27	8	11	5	395	8	2	2	180	4	-1
7	356	7	0	9	121	5	-7	9	297	6	-2	6	34	9	8	4	176	4	3
9	299	6	-2	11	102	7	4	10	6	41	3*	7	311	6	-3	6	147	6	-1
11	203	5	-2	H,L=	0,	10		11	225	5	-2	8	13	42	1*	8	105	5	-6
13	149	7	2	0	212	5	3	13	153	4	-2	9	227	5	-6	10	96	5	5
15	117	7	8	2	176	7	-3	15	98	8	-4	11	171	4	-2	H,L=	1,		11
H,L=	0,	4		4	182	5	3	H,L=	1,	2		13	123	5	5	1	153	5	-0
0	785	16	-13	6	162	5	4	0	724	16	-12	H,L=	1,	6		3	141	6	4
2	445	9	17	8	107	7	-2	1	337	7	37	0	463	9	2	5	127	5	0
4	478	10	-9	10	88	12	-4	2	649	13	13	1	43	6	2	7	102	5	-1
6	462	9	6	H,L=	0,	11		3	33	35	14*	2	402	8	11	9	81	6	-1
8	252	5	-9	1	159	8	-5	4	609	12	25	3	40	14	-10*	H,L=	1,		12
10	262	6	4	3	147	6	4	5	25	7	9	4	368	8	3	0	120	6	-2
12	158	5	-2	5	128	6	-3	6	476	10	-3	5	10	28	-18*	2	115	6	3
14	110	7	5	7	104	9	3	7	31	9	1	6	302	6	-5	4	103	6	-0
H,L=	0,	5		9	86	19	4	8	318	6	2	7	39	48	16*	6	97	6	8
1	549	15	20	H,L=	0,	12		9	47	13	4	8	225	5	-1	8	81	9	10
3	465	9	-5	0	127	6	-10	10	280	6	-3	10	191	4	-3	H,L=	1,		13
5	428	9	2	2	106	8	-10	12	171	4	-8	12	122	5	-1	1	97	6	6
7	261	6	-6	4	110	7	7	14	112	5	-2	14	91	5	9	3	77	10	-5
9	244	5	-2	6	101	7	10	H,L=	1,	3		H,L=	1,	7		5	68	10	-6
11	165	5	-6	8	65	12	-7	1	622	13	-8	1	362	8	-2	7	67	14	5
13	113	6	2	H,L=	0,	13		2	68	9	22	2	24	25	19*	H,L=	1,		14
15	93	22	8	1	86	13	-6	3	561	11	6	3	316	6	3	0	75	8	8

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K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
2	70	9	6	H,L=	2,	4		6	200	4	-1	5	397	8	2	11	136	4	-2
4	63	16	3	0	529	11	13	8	159	4	-3	6	26	11	17*	13	104	6	-7
H,L=	2,	0		1	61	5	6	10	118	4	-0	7	342	7	-3	H,L=	3,	6	
0	452	12	-44	2	513	11	4	12	82	10	-6	8	53	20	14*	0	298	6	-2
1	0	71	-10*	3	26	8	14*	H,L=	2,	9		9	275	6	-3	1	44	8	10
2	543	11	-35	4	454	9	14	1	225	5	-11	11	176	4	1	2	348	7	-1
3	34	38	2*	5	12	27	6*	2	21	51	4*	13	139	6	-8	3	0	29	-10*
4	586	13	-7	6	369	8	-1	3	220	5	2	15	76	9	-1	4	314	6	5
5	46	5	31	7	18	27	-2*	5	170	4	1	H,L=	3,	2		5	35	26	34*
6	504	10	1	8	287	6	4	7	143	4	-9	0	510	11	-5	6	250	6	-2
7	50	6	1	9	31	36	-5*	9	114	4	-7	1	75	3	6	7	14	38	-2*
8	328	7	-10	10	201	4	-2	11	84	6	-1	2	535	11	-4	8	202	4	-11
9	42	50	-6*	12	152	4	2	H,L=	2,	10		3	71	3	-5	10	134	4	-1
10	241	5	4	14	106	5	6	0	179	4	-2	4	469	9	10	12	117	5	5
12	185	6	-1	H,L=	2,	5		2	178	4	1	5	32	15	-12*	H,L=	3,	7	
14	132	5	3	1	427	9	9	4	151	5	-3	6	360	7	8	1	284	6	8
H,L=	2,	1		2	20	21	-2*	6	136	6	3	7	45	11	2	2	37	19	33*
1	594	12	-34	3	445	9	4	8	114	5	0	8	317	7	-9	3	303	6	4
2	14	38	-2*	4	42	10	2	10	92	5	7	9	0	43	-16*	4	0	52	-12*
3	688	15	28	5	326	7	0	H,L=	2,	11		10	202	4	4	5	222	5	-4
4	44	4	-7	6	25	27	12*	1	125	6	-7	12	152	4	-1	6	28	38	22*
5	393	8	-13	7	307	6	3	3	135	5	1	14	113	6	1	7	200	4	-3
6	24	12	24*	8	0	34	-7*	5	117	5	2	H,L=	3,	3		9	147	4	-3
7	420	9	9	9	233	5	1	7	103	5	2	1	511	10	6	11	115	4	9
8	46	8	12	11	160	4	0	9	81	9	2	2	40	5	-12	13	92	6	7
9	309	7	-2	13	129	5	9	H,L=	2,	12		3	504	10	3	H,L=	3,	8	
10	22	33	4*	H,L=	2,	6		0	98	7	-3	4	8	25	-5*	0	233	6	-3
11	208	5	-1	0	351	7	9	2	99	6	-6	5	327	7	-1	1	0	41	-5*
13	157	4	2	1	17	26	-6*	4	106	6	6	6	39	29	18*	2	249	6	-4
15	96	6	7	2	360	7	-2	6	93	8	7	7	315	6	-2	3	13	39	-1*
H,L=	2,	2		3	22	24	-10*	8	66	17	-3	8	0	50	-29*	4	207	4	-3
0	647	15	19	4	345	7	-2	H,L=	2,	13		9	259	5	-2	6	176	4	1
1	19	38	-36*	5	33	24	31*	1	84	7	2	11	167	5	1	8	156	5	-2
2	617	13	-7	6	299	6	2	3	88	8	6	13	125	5	-4	10	111	6	5
3	0	21	-1*	7	32	46	7*	5	61	10	-10	H,L=	3,	4		12	94	6	11
4	542	11	3	8	226	5	0	H,L=	2,	14		0	421	9	-6	H,L=	3,	9	
5	16	20	5*	10	158	5	-5	0	55	15	-11	1	43	5	2	1	200	4	-4
6	444	9	-1	12	127	5	2	2	57	19	-7*	2	454	9	-9	3	195	4	-2
7	40	9	11	14	96	5	10	H,L=	3,	0		3	37	8	-1	5	143	4	-5
8	317	6	-1	H,L=	2,	7		0	496	10	4	4	378	8	-1	7	135	4	-6
9	18	51	-28*	1	317	6	1	1	126	5	2	5	20	25	-5*	9	113	6	1
10	223	5	-3	2	0	36	-12*	2	538	11	-7	6	307	6	3	11	78	7	-0
12	172	4	3	3	332	7	5	3	38	6	10	7	45	28	13*	H,L=	3,	10	
14	114	5	-0	4	36	41	11*	4	525	11	3	8	271	6	-10	0	153	6	-5
H,L=	2,	3		5	252	6	-3	5	11	31	-2*	10	179	4	3	2	162	5	-2
1	648	13	7	6	0	50	-13*	6	395	8	6	12	133	4	-3	4	133	5	-5
2	65	4	2	7	220	5	-4	7	71	8	9	14	100	8	4	6	116	5	-1
3	608	12	20	9	167	4	-1	8	321	7	-12	H,L=	3,	5		8	108	5	2
4	37	6	5	11	116	5	-2	9	0	45	-12*	1	376	8	3	10	81	7	7
5	365	7	10	13	85	7	-7	10	197	5	2	2	25	26	24*	H,L=	3,	11	
6	31	12	27*	H,L=	2,	8		12	151	5	-9	3	402	8	-6	1	126	5	4
7	363	7	5	0	286	6	-1	14	123	6	-1	4	31	10	-7*	3	126	5	-0
8	31	34	22*	1	32	48	11*	H,L=	3,	1		5	290	6	-2	5	101	6	-2
9	279	6	-2	2	285	6	4	1	532	11	3	6	44	26	43*	7	99	5	7
11	182	4	-2	3	32	60	25*	2	18	22	17*	7	265	6	-3	9	63	13	-7
13	136	4	3	4	235	6	-6	3	590	12	15	8	0	33	-10*	H,L=	3,	12	
15	79	8	-0	5	0	32	-1*	4	71	3	0	9	205	5	3	0	89	6	-2

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K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
2	101	5	3	11	149	4	-2	7	130	5	5	1	307	6	-2	5	133	4	6
4	99	6	8	13	117	4	2	9	96	6	3	2	34	17	3*	7	105	4	7
6	89	9	12	H,L=		4,	4	H,L=		4,	10	3	304	6	-1	9	70	7	-6
H,L=		3,	13	0	320	7	-4	0	137	4	2	4	71	17	20	H,L=		5,	10
1	77	9	4	1	51	9	17	2	138	5	-4	5	303	6	-3	0	118	5	2
3	81	10	6	2	402	8	2	4	131	4	5	6	12	36	-13*	2	111	4	0
5	49	17	-14*	3	35	19	24*	6	102	5	-3	7	214	5	5	4	110	5	3
H,L=		3,	14	4	315	6	-9	8	89	6	-0	9	165	4	0	6	91	5	-2
0	56	16	-1*	5	38	22	30*	H,L=		4,	11	11	127	4	-2	8	68	8	-2
H,L=		4,	0	6	253	6	0	1	117	5	2	13	84	5	0	H,L=		5,	11
0	423	9	9	7	0	39	-5*	3	109	6	0	H,L=		5,	4	1	96	5	1
1	49	8	34	8	234	5	-2	5	93	6	2	0	327	7	8	3	97	6	11
2	549	11	-8	10	158	4	-4	7	88	7	10	1	47	11	24	5	78	7	0
3	21	40	10*	12	125	4	4	H,L=		4,	12	2	287	6	2	7	59	8	-5
4	390	9	3	H,L=		4,	5	0	93	8	5	3	40	50	24*	H,L=		5,	12
5	45	9	30	1	341	7	-5	2	95	6	7	4	265	6	-1	0	83	7	4
6	292	6	2	2	44	12	37	4	79	11	2	5	17	38	14*	2	73	6	2
7	58	34	32*	3	330	7	5	6	57	11	-8	6	231	5	-3	4	64	9	2
8	287	6	2	4	56	13	23	H,L=		4,	13	8	168	4	-8	H,L=		6,	0
10	183	4	-4	5	256	6	-5	1	76	10	11	10	132	4	-4	0	287	7	-3
12	140	5	-1	6	0	46	-25*	3	80	8	18	12	102	4	3	2	253	6	-2
14	80	9	-8	7	234	5	-1	H,L=		5,	0	H,L=		5,	5	4	254	5	-9
H,L=		4,	1	9	156	4	-3	0	436	9	-10	1	275	6	-3	5	0	49	-5*
1	444	9	-12	11	122	4	1	1	51	13	3	2	34	52	7*	6	203	5	-4
2	17	23	11*	13	88	5	-4	2	355	8	-2	3	245	6	-3	8	165	5	-12
3	462	10	12	H,L=		4,	6	3	31	36	7*	4	0	42	-18*	10	139	6	-3
4	66	9	6	0	279	6	-6	4	296	6	-0	5	231	5	-1	12	100	6	11
5	348	7	5	1	29	23	22*	5	44	48	24*	7	172	4	-3	H,L=		6,	1
6	31	17	-14*	2	317	7	2	6	273	6	6	9	146	4	1	1	298	7	-7
7	324	7	-10	3	24	32	6*	7	0	53	-25*	11	110	5	1	3	212	6	-2
8	25	36	17*	4	258	6	-1	8	209	5	-8	H,L=		5,	6	4	0	36	-10*
9	203	4	6	5	22	44	19*	10	169	4	-6	0	265	6	-3	5	262	5	-2
11	154	4	-5	6	203	4	3	12	127	5	13	1	20	33	3*	7	173	5	-4
13	134	4	13	8	178	4	-3	H,L=		5,	1	2	225	5	-9	9	149	4	-2
H,L=		4,	2	10	128	4	1	1	367	8	-3	3	31	19	30*	11	123	5	-0
0	362	7	12	12	99	5	6	2	48	15	-6*	4	198	4	-1	H,L=		6,	2
1	38	11	-3	H,L=		4,	7	3	321	7	-6	6	174	4	-3	0	283	7	-5
2	482	10	0	1	247	6	-2	4	58	10	11	8	146	4	-0	2	256	6	-3
3	31	24	9*	2	0	51	-3*	5	313	7	-10	10	115	4	0	3	0	52	-8*
4	371	8	5	3	236	5	-1	6	26	43	-26*	12	85	6	6	4	256	5	-5
5	0	27	-11*	4	0	30	-19*	7	227	5	3	H,L=		5,	7	6	202	4	0
6	287	6	1	5	192	4	-5	9	193	4	3	1	203	4	-8	8	159	4	-1
7	32	50	25*	7	178	4	1	11	142	4	-1	3	181	4	-5	10	130	4	4
8	282	6	3	9	133	4	3	13	87	6	1	5	176	4	5	12	86	7	4
10	185	4	-3	11	97	6	1	H,L=		5,	2	7	138	4	2	H,L=		6,	3
12	135	4	-5	H,L=		4,	8	0	383	8	1	9	117	4	3	1	276	6	-7
14	93	5	4	0	182	4	-6	1	0	30	-34*	11	86	6	1	3	211	4	-2
H,L=		4,	3	1	0	37	-14*	2	323	7	-5	H,L=		5,	8	4	21	30	15*
1	374	8	-3	2	202	5	-4	3	40	15	6*	0	177	4	2	5	243	5	-3
2	27	38	-18*	4	183	5	-2	4	300	6	-2	2	162	4	-2	7	159	4	-2
3	395	10	11	6	152	4	-1	5	40	47	35*	4	154	4	-1	9	129	4	1
4	45	9	15	8	138	4	2	6	273	6	4	6	141	4	5	11	106	5	-2
5	312	6	-5	10	104	5	6	7	0	37	-7*	8	103	4	-2	H,L=		6,	4
6	47	30	23*	H,L=		4,	9	8	198	4	-4	10	79	8	-3	0	246	5	1
7	298	6	-5	1	166	4	1	10	158	4	-2	H,L=		5,	9	2	220	5	-4
8	35	31	28*	3	162	5	0	12	117	6	5	1	141	4	4	3	41	42	33*
9	192	4	-0	5	142	4	2	H,L=		5,	3	3	139	4	4	4	216	5	-6

STRUCTURE FACTORS CONTINUED FOR
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K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL	K	FOB	SG	DEL
6	180	4	1	H,L=	7,	2		3	146	4	-1	8	89	13	14				
8	138	4	-1	0	201	4	-3	5	142	4	1	H,L=	9,	3					
10	119	4	6	2	206	4	1	7	102	5	2	1	97	5	-1				
12	78	7	4	4	180	4	-3	9	86	6	-1	3	104	5	4				
H,L=	6,	5		6	160	4	-3	H,L=	8,	2		5	90	5	9				
1	216	5	-5	8	125	4	4	0	152	4	-1	7	87	13	10				
3	168	4	-4	10	98	5	2	2	146	4	-1	H,L=	9,	4					
5	196	4	1	H,L=	7,	3		4	130	4	0	0	87	6	-0				
7	138	4	-3	1	206	5	-5	6	115	4	2	2	102	6	3				
9	119	4	3	3	175	4	4	8	100	7	-1	4	89	5	4				
11	99	7	4	5	159	4	-1	H,L=	8,	3		6	57	60	-12*				
H,L=	6,	6		7	135	4	3	1	143	4	-0	8	69	19	4				
0	191	4	-3	9	109	5	3	3	127	4	-2	H,L=	9,	5					
2	173	4	-0	H,L=	7,	4		5	119	4	-0	1	85	7	-1				
4	168	4	2	0	178	4	-6	7	99	5	4	3	87	5	2				
6	141	4	-2	2	176	4	1	9	89	7	4	7	81	15	17				
8	122	4	6	4	160	4	1	H,L=	8,	4		H,L=	9,	6					
10	99	5	2	6	142	4	1	0	133	4	-2	0	73	7	0				
H,L=	6,	7		8	108	5	0	2	131	4	-1	2	86	6	6				
1	166	4	-5	10	96	7	9	4	121	4	7	6	56	25	-0*				
3	137	4	-1	H,L=	7,	5		6	101	5	2	H,L=	10,	0					
5	148	4	2	1	164	4	-1	8	95	7	6	0	79	9	5				
7	111	4	3	3	144	4	0	H,L=	8,	5		2	79	13	-1				
9	90	6	2	5	140	4	-2	1	124	4	3	4	78	15	16				
H,L=	6,	8		7	115	6	4	3	116	4	-0	6	69	17	7				
0	143	4	-4	9	97	6	9	5	110	6	5	H,L=	10,	1					
2	135	4	1	H,L=	7,	6		7	84	7	3	1	72	6	-2				
4	124	5	-3	0	133	5	-9	H,L=	8,	6		3	91	11	13				
6	108	5	1	2	142	4	7	0	109	5	5	5	53	25	-5*				
8	84	8	3	4	135	4	1	2	105	4	-3	H,L=	10,	2					
H,L=	6,	9		6	112	6	-4	4	106	6	8	0	65	18	-6				
1	120	4	1	8	90	7	4	6	91	6	7	2	76	31	-2*				
3	110	4	6	H,L=	7,	7		H,L=	8,	7		6	42	47	-18*				
5	105	5	1	1	125	4	-1	1	96	5	6	H,L=	10,	3					
7	82	6	5	3	117	5	4	3	89	6	-1	1	68	16	0				
H,L=	6,	10		5	119	4	8	5	79	7	-1	3	79	16	5				
0	103	6	8	7	82	6	-4	H,L=	8,	8		H,L=	10,	4					
2	92	5	4	H,L=	7,	8		0	70	8	-7	0	80	13	17				
4	91	6	6	0	118	5	6	2	73	7	-5	2	99	10	32				
6	72	10	-2	2	105	4	2	4	63	11	-6	H,L=	10,	5					
H,L=	6,	11		4	97	5	1	H,L=	9,	0		1	76	15	18				
1	79	6	5	6	90	5	7	0	100	7	-5								
3	63	7	-3	H,L=	7,	9		2	117	7	-3								
H,L=	7,	0		1	93	5	-0	4	102	6	2								
0	198	6	4	3	77	7	-2	6	77	8	-1								
2	208	5	-2	5	76	11	1	8	67	19	-11								
4	201	5	4	H,L=	7,	10		H,L=	9,	1									
6	176	5	-3	0	80	10	3	1	113	4	4								
8	133	5	8	2	68	8	-2	3	106	7	-5								
10	97	6	-1	H,L=	8,	0		5	96	5	7								
H,L=	7,	1		0	156	5	-5	7	84	8	1								
1	215	5	-5	2	156	4	-1	9	62	22	1*								
3	195	4	5	4	143	7	-3	H,L=	9,	2									
5	181	4	2	6	128	6	7	0	96	8	-2								
7	149	4	1	8	109	6	9	2	113	4	1								
9	111	4	-0	H,L=	8,	1		4	104	4	8								
11	98	6	3	1	155	4	-1	6	82	5	6								

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