Lawrence Berkeley National Laboratory
Recent Work

Title

MOLECULAR STRUCTURE AND DISORDER IN CRYSTALS OF OCTAPHENYLURANOCENE, U(C8H4(C6H5)4)2

Permalink

https://escholarship.org/uc/item/9z5338t1

Author

Templeton, Lieselotte K.

Publication Date

1976-06-01

000045005732

MOLECULAR STRUCTURE AND DISORDER IN CRYSTALS OF OCTAPHENYLURANOCENE, $U(C_8H_4(C_6H_5)_4)_2$

Lieselotte K. Templeton, David H. Templeton, and
Roger Walker

June 1976

For Reference

Not to be taken from this room

DISCLAIMER

Molecular Structure and Disorder in Crystals of Octaphenyluranocene,

$U(C_8H_4(C_6H_5)_4)_2$[1]

Lieselotte K. Templeton, David H. Templeton[*] and Roger Walker

Abstract

Bis-π-(1,3,5,7-tetraphenylcyclooctatetraene)uranium crystallizes
with a novel disorder of the molecular packing which is described as
frequent twinning of a monoclinic structure, space group P2/c, a =
24.86(2)Å, b = 7.587(6)Å, c = 27.78(3)Å, β = 116.58°, Z = 4, d_x =
1.495 g cm^{-3} at 23.0(5)°C. The completely disordered structure has
orthorhombic symmetry, space group Pccn, a = 24.84(3)Å, b = 12.43(1)Å,
c = 7.587(6)Å, Z = 2. Refinement by least squares of x-ray diffracto-
meter data reduced the weighted R to 0.033 for 723 reflections. The
molecule is a sandwich compound with the C_8 rings in a nearly eclipsed
configuration and with phenyl rings, tilted an average of 42° from
the plane of the C_8 ring, in a staggered configuration. The C-C-C
angles in the C_8 ring alternate between average values of 140° at

carbon bonded to hydrogen and 130° at carbon bonded to phenyl. A
similar alternation between 138° and 132° occurs in the analogous
methyl-substituted compound. The phenyl compound in crystalline form
is stable in air, with no significant change in x-ray diffraction
intensities in three months.

Introduction

As part of a program to extend knowledge of the chemical and structural properties of compounds of actinide elements, we undertook a study of the structure of bis-π-(1,3,5,7-tetraphenylcyclooctatetraene) uranium when it was synthesized by Streitwieser and Walker.[2] The immediate objective was a description of the molecular geometry and in particular the positions and orientations of the phenyl rings. In view of the discovery of two different rotomeric configurations in crystals of the analogous methyl-substituted compound,[3] there was some doubt what to expect. The x-ray diffraction data soon showed the existence of an unexpected and novel disorder problem concerning the molecular packing which made the analysis of the structure more involved than is ordinarily the case.

Experimental

Bis-π-(1,3,5,7-tetraphenylcyclooctatetraene)uranium, $U(C_8H_4(C_6H_5)_4)_2$ was prepared by the reaction of UCl_4 with the dianion of 1,3,5,7-tetraphenylcyclooctatetraene.[2] The polycrystalline product, in a dry box, was loaded into a Pyrex tube fitted with a stopcock. The tube was pumped to about 10^{-6} torr; then one end was plunged into a hot salt bath. Clusters of small dark brown single crystals condensed 1 or 2 cm above the bath. Various conditions were tried, and the best crystals were obtained with the bath at 400°C. No solvent has been found good enough to permit crystal growth from solution.

When the x-ray work was started it was believed that the compound decomposed significantly in a few days on exposure to air. The crystals were very thin needles, and we anticipated correctly that the diffraction intensities would be weak. In order to avoid the higher background associated with capillaries we glued crystals with epoxy to Pyrex glass fibers for x-ray diffraction work. As it turned out, the crystals were stable indefinitely.

The diffraction patterns consist of sharp reflections and diffuse streaks. We describe them by reference to a primitive orthorhombic lattice which corresponds to the sharp reflections. An oscillation pattern ($\underline{c}$-axis rotation) showed the odd layers as continuous streaks superimposed on a few sharp reflections, while the even layers appeared as normal sharp reflections. Weissenberg photographs taken with long exposure times (up to 72 hours) indicated orthorhombic symmetry and showed streaks which were continuous in the $\underline{a}^*$ direction at $\underline{\ell}$ odd and $\underline{k}$ half-integral.

Diffractometer measurements were made first considering only the sharp reflections, using a crystal with dimensions approximately 0.030 × 0.056 × 0.174 mm. Unit cell dimensions were determined from 6 hand centered reflections with a Picker FACS-I automatic diffractometer equipped with a graphite monochromator and molybdenum radiation ($\lambda = 0.7107$ Å for Kα). The same instrument was used to collect the intensity data. The integrated intensity of each reflection was measured using a $\theta - 2\theta$ scan technique with a scanning rate of 1°/min from 0.5° below the 2θ angle at which Kα_1 was diffracted to 0.5° above the 2θ angle at which Kα_2 was diffracted. Two 20 sec

background counts were taken with the apparatus stationary and offset
0.5° from each end of the scan. Three reflections ($00\bar{2}$, 510, 220) were
remeasured at intervals of 200 reflections to check for crystal decom-
position and/or instrument malfunction. The data show an average decay
of about 4% from beginning to end, but this change is almost obscured
by random variations. Since the decay rate is small and poorly
established, and since averaging of measurements made early and late
tends to compensate for this effect, no correction was applied for decay.
Measurements were made of most of the reflections in the hemisphere
$\pm h$, $+ k$, $\pm l$, $2\theta < 45°$. Absorption corrections calculated by an
analytical integration[4] ranged from 1.098 to 1.215. Their validity
was justified by multiple measurements of three reflections at various
azimuthal angles. Equivalent reflections among the 4860 measured were
averaged to give 1617 which are unique. For 800 of them $F^2 > s(F^2)$, where
s is the standard deviation based on counting statistics. For derivation
of weights in least squares calculations, the variance of F^2 was taken
as $\sigma^2(F^2) = s^2(F^2) + (pF^2)^2 + q^2$, where p and q were chosen by trial
and error to give a flat distribution of $\langle w(\Delta F)^2 \rangle$ as a function of
magnitude of F. Zero weight was assigned when $F < \sigma(F)$.

The same crystal was used to take a data set on the streaks. The
same $\theta - 2\theta$ scan technique was employed as for the sharp features.
Measurements were made at both integral and half-integral values of
$\underline{h}$, with $\underline{k}$ half-integral and $\underline{l}$ odd. These reflections were measured
in the hemisphere $\pm h$, $\pm k$, $- l$ up to 2θ of 25°. Of the 1196 reflections
measured, 599 were unique according to monoclinic symmetry, and for

508 $F^2 > \sigma(F^2)$. There was no decay for the 3 standards within the
experimental accuracy of the experiment.

Crystal Data

For the ideal ordered structure described below, the symmetry is
monoclinic, space group P2/c; at 23.0 ± 0.5°C, a = 24.86(2)Å, b =
7.587(6)Å, c = 27.78(3)Å, β = 116.58, Z = 4, mol. wt. = 1055.12,
d_x = 1.496 g cm^{-3}, V = 4684 Å^3, μ(MoKα) = 33.3 cm^{-1}. For the
completely disordered structure the symmetry is orthorhombic; space
group Pccn, a = 24.84(3)Å, b = 12.43(1)Å, c = 7.587(6)Å, Z = 2.

Structure Determination

The data were analyzed in two stages. Consideration of reflections
corresponding to the orthorhombic lattice (the sharp reflections), with-
out any attention to the streaks, gave a result which represents the
average of the structure over all the orthorhombic unit cells. It was
determined and refined by conventional methods. Subsequent analysis
of the streaks revealed a monoclinic and orderly pattern of repetition
which, with frequent twinning, is our model for the actual structure.
The data are not sufficient for independent determination of all the
atomic parameters in the monoclinic symmetry, and the molecular dimen-
sions which are reported below came from the refinement of the sharp
reflections alone.

The sharp reflections exhibit orthorhombic Laue symmetry. A Patterson function calculated from these data shows four heavy atom sites which each must be half occupied, since the volume corresponds to only two molecules of $UC_{64}H_{48}$. The Patterson function also showed resolved U-C peaks among which one could recognize all the carbon atoms of the molecular structure. Considerations of packing lead uniquely to one pattern of relative molecular orientations. Up to this point no particular assumption was necessary about the space group. The structure which was found corresponded to space group Pccn, for which reflections are absent for the classes: 0kl, l $\neq$ 2n; h0l, l $\neq$ 2n; hk0, h+k $\neq$ 2n. All such reflections were either weak or absent, but for 17 of them (out of 239) the structure factor was measured to be greater than its standard deviation. We ignored these reflections and chose Pccn as the space group for refinement of the disordered structure.

This model includes 16 independent carbon atoms in the asymmetric unit, along with one fractional uranium atom. After four cycles of least squares refinement of these seventeen atoms with isotropic thermal parameters and using p = 0.05 and q = 0, R = $\Sigma|\Delta F|/\Sigma|F_o|$ = 0.13 for 800 reflections with $F^2 > \sigma(F^2)$. Four more cylces, using anisotropic thermal parameters for the uranium atom but isotropic temperature factors for the carbon atoms, reduced R to 0.107 and $R_2 = (\Sigma w(\Delta F)^2/\Sigma w F_o^2)^{1/2}$, the quantity minimized, to 0.068. Using anisotropic thermal parameter also for the carbon atoms gave several carbon atom thermal tensors that were not positive definite. Therefore, the data do not warrant making the carbon atoms anisotropic. Further refinements were made with q = 8.

This reduced the number of reflections with $F^2 > \sigma(F^2)$ to 740, and deleting the reflections forbidden by space group Pccn reduced that number to 723. Refinement of the 17 atoms in the asymmetric unit, using anisotropic parameters only for uranium resulted in R = 0.088 for 723 reflections and R_2 = 0.058. Hydrogen atom positions were calculated assuming planar trigonal geometry at carbon atoms and a C-H bond length of 0.95 Å. The hydrogen contributions to the structure factors were calculated with the phenyl hydrogen atoms fixed in their calculated positions. The isotropic thermal parameters were constrained to be equal, respectively, for ortho, meta, and para hydrogen atoms. For hydrogen atoms on the cyclooctatetraene (COT) ring, each thermal parameter was constrained to be equal to that of the carbon atom bonded to it, and each C-H bond was held constant in length and direction. With all these constraints, only three more independent parameters were added. When further refinement shifted the carbon atoms, the hydrogen atom positions were calculated a second time. With p = 0.015 and q = 8, the final R = 0.081 and R_2 = 0.033 for 723 reflections. The standard deviation of observation of unit weight was 0.981. The scattering factors used were those of Doyle and Turner[5] for the neutral uranium and carbon atoms, those of Stewart, Davidson, and Simpson[6] for spherical hydrogen, and the dispersion corrections from Cromer and Liberman.[7] In the last cycle no parameter changed more than 0.004σ. Final parameters are listed in Table I.

The data from the streaks were used to calculate a Patterson function, without including the sharp reflections. This was done

separately because the continuous nature of the streaks causes a problem about scaling which we did not attempt to solve. This function, if scaled properly, should be added to the previous Patterson function to give the function for the entire data set. Thus it can be examined to determine corrections to be made in the disordered structure determined before.

This correction function was calculated using monoclinic symmetry because deviations from orthorhombic symmetry were observed in the intensity measurements. It has periodicity according to the monoclinic lattice with C-centering and with the $\underline{c}$-axis doubled, because of the manner of sampling of the streaks. However, its main features repeat with periodicity $\underline{c}$ rather than 2 $\underline{c}$. It is antisymmetric in relation to planes at $y = 1/4$ because of the absence of k-even terms (l-even in the orthorhombic description). By far the most prominent features are the origin peak and the peaks related to it by the above symmetries. These vectors and the absence of diffuse scattering except in the direction of $\underline{c}^*$ (orthorhombic $\underline{a}^*$) show that the uranium structure must be ordered in any layer parallel with $\underline{a}$ and $\underline{b}$ (orthorhombic $\underline{b}$ and $\underline{c}$) according to the pattern shown in Figure 1. Furthermore, this structure must repeat with considerable probability at a displacement equal to $\underline{c}$. A considerably smaller peak at 0, 0, 1/2 and equivalent points shows that the relation to a layer with displacement $\underline{c}/2$ is nearly random between two choices of the uranium positions. These facts are explained by a model with space group P2/c and uranium in special positions 2(e), $\pm(0, y, 1/4)$, $y \sim 0$, and 2(f) $\pm(1/2, y, 1/4)$, $y \sim 1/2$. This structure

is highly twinned with two distinct orientations of the monoclinic c axis as seen in Figure 2. This twinning constitutes simply a change in the stacking of adjacent layers. The deviations from orthorhombic symmetry in the streaks show that the specimen studied contains unequal amounts of the two orientations.

A remarkable feature of this structure is that the carbon and hydrogen atom positions need not change much regardless of the uranium positions. The space between adjacent molecules in a stack in the direction of b (orthorhombic c) is nearly the same as the space occupied within the molecule by the uranium atom, and the parallel COT rings with their attached phenyl rings have the same geometrical relation (to a good approximation) with either site (see Figure 1). A description of the structure in space group P2/c requires 64 carbon atoms in the asymmetric unit. The available data are quite insufficient to determine so many parameters, even without the formidable problems connected with the twinning and the large amount of pseudosymmetry. We made no attempts to refine the structure in the monoclinic symmetry.

Discussion

The method of refinement makes no distinction between the site occupied by uranium and the similar site between two molecules. The plane-to-plane distance between COT rings, averaged for these two sites, is 3.793(3)Å. The ring-to-ring distance is 3.847(10)Å in the unsubstituted uranocene molecule[8] and 3.836(9)Å in the octamethyl derivative.[3]

A similar value could occur in the octaphenyl compound if carbon atoms are shifted less than 0.03 Å from the average positions reported here. Such shifts are small in comparison to the root mean square amplitudes of thermal motion estimated for carbon atoms, which average about 0.21 Å, and therefore are quite consistent with our diffraction data. Because of this disorder, the U-C distances listed in Table II are subject to a systematic error. If the ring-to-ring distance in the molecule is 3.84 Å, each U-C distance in Table II should be increased about 0.02 Å. The disorder is not expected to have any significant effect on the C-C distances and C-C-C angles discussed below.

The molecule, Figures 1 and 3, is a sandwich compound with the COT rings rotated 6° from the eclipsed configuration found in unsubstituted uranocene.[8] The phenyl rings, attached to alternate carbon atoms, are staggered. They are also tilted, the four on one C_8 ring like a right-handed propeller and the four on the other ring in a left-handed manner. The dihedral angles between the planes of the C_8 ring and the phenyl rings are 43.8° (atoms C(3) - C(8)) and 40.3° (atoms C(11) - C(16)). The differences from the average value of 42° are not significant.

The C-C bond lengths (Table II) average 1.42(2)Å in the COT ring and 1.39(2)Å in the phenyl rings. This value for phenyl rings is well accepted. The COT-ring value is in agreement with the average 1.41 Å found in $U[C_8H_4(CH_3)_4]_2$,[3] and with 1.407(6)Å in a potassium salt of $C_8H_4(CH_3)_4$ dianion.[9] For these crystals the substituents on the rings prevent excessive torsional motion. Values near 1.39 Å are reported

for several complexes of unsubstituted C_8H_8 dianion,[8,10] but in each

of these cases the effects of thermal motion are appreciable and explain

the lower result. For example, a correction calculated according to

a rigid-body-torsional model with the data for either uranocene or

thorocene[8] gives a corrected average C-C = 1.42 Å. For this model,

which probably is a good approximation here, the corrected distance

is $d_{corr} = [d^2 + 4u^2\sin^2(\pi/n)]^{1/2}$, where d is the uncorrected distance,

u is the root-mean-square amplitude of motion tangential to the ring,

and n is the number of atoms equally spaced in the ring (8 in this

case).

The inclusion of hydrogen atoms in the calculations was crucial

for the good agreement of these C-C distances and also it made the

bond angles more consistent with each other. Without the hydrogen

atoms, the C-C distances averaged 1.44(6)Å in the COT ring and 1.42(2)Å

in the phenyl rings.

The C-C-C angles in the COT ring show an interesting alternation

(Figure 4) from about 140°, for angles at carbon atoms bonded to

hydrogen, to 130° at those bonded to phenyl. A similar alternation,

between 138° and 132° occurs in tetramethylcyclooctatetraene dianion

in the uranium complex[3] and the potassium salt.[9] For some of the

angles the observed differences are less than the estimated errors,

but with a total of 36 different angle determinations in the two

structures the trend is unmistakable.[11] We are unsure whether this

angle alternation is simply a steric effect (the sign is right to

allow the bulky substituent groups to be more distant from the center

of the molecule) or whether there is a deeper electronic explanation.

As shown in Figure 1, the molecules stack in columns. These columns, viewed end-on in Figure 5, pack in a triangular fashion. The intermolecular contacts involve the phenyl rings approaching each other in the nearly perpendicular fashion which is so widespread in crystal packing of aromatic compounds. The same kind of phenyl-phenyl proximity occurs within the molecule (Fig. 3). The 6° rotation from the eclipsed configuration is attributed to an effect of this packing; the rotation relieves the crowding of phenyl groups in molecules adjacent in the orthorhombic b direction (Fig. 5) without having much effect on other intermolecular contacts. We expect that isolated molecules in the gas phase would have ideal $\bar{8}$ symmetry.

In contrast to other known uranocene-type compounds, which are sensitive to the atmosphere, the octaphenyl compound in crystalline form is remarkably stable. There was no significant decrease in the diffraction intensities for the crystal when it was reexamined three months after the original measurements, and during all this time it was exposed to the air. The dense packing of the phenyl rings around the molecule and the efficient manner in which molecules are packed together may both contribute to this lack of reactivity.

Supplementary Material Available: A listing of structure factors (7 pages). Ordering information is given on any current masthead page.

REFERENCES AND NOTES

1. This work was done in part with support from the U.S. Energy Research and Development Administration.

2. A. Streitwieser, Jr. and R. Walker, J. Organomet. Chem., 97, C 41 (1975).

3. K. O. Hodgson and K. N. Raymond, Inorg. Chem., 12, 458 (1973).

4. L. K. Templeton and D. H. Templeton, Abstracts, American Crystallographic Association Proceedings, Series 2, Vol. 1, 1973, p143.

5. P. A. Doyle and P. S. Turner, Acta Crystallogr., Sect. A, 24, 390 (1968).

6. R. F. Stewart, E. R. Davidson and W. T. Simpson, J. Chem. Phys., 42, 3175 (1965).

7. D. T. Cromer and D. Liberman, J. Chem. Phys., 53, 1891 (1970).

8. A. Avdeef, K. N. Raymond, K. O. Hodgson and A. Zalkin, Inorg. Chem., 11, 1083 (1972).

9. S. Z. Goldberg, K. N. Raymond, C. A. Harmon and D. H. Templeton, J. Am. Chem. Soc., 96, 1348 (1974).

10. K. O. Hodgson and K. N. Raymond, Inorg. Chem., 11, 171 and 3030 (1972).

11. The angle alternation, which escaped notice at the time, was partially obscured by inconsistent designation of atoms in the lists of distances and angles for the fourth ring, Reference 3.

Table I.   Atomic Parameters and Esd's[a] (Orthorhombic Coordinate System).

Atom	x	y	z	B
U	1/4	1/4	.0100(4)	--[b]
C(1)	.2826(7)	.387(2)	.285(3)	2.9(5)
C(2)	.3184(6)	.297(1)	.260(3)	2.9(4)
C(3)	.3050(4)	.4985(8)	.260(3)	2.9(2)
C(4)	.3501(5)	.5217(9)	.362(2)	3.0(3)
C(5)	.3747(5)	.622(1)	.349(2)	4.9(3)
C(6)	.3565(5)	.6994(9)	.235(3)	4.5(3)
C(7)	.3113(5)	.678(1)	.133(2)	4.6(3)
C(8)	.2866(5)	.579(1)	.144(2)	3.3(3)
C(9)	.3187(6)	.185(1)	.260(3)	2.0(4)
C(10)	.2752(6)	.111(1)	.256(3)	2.2(4)
C(11)	.3751(3)	.1415(7)	.249(3)	2.5(2)
C(12)	.4146(5)	.1863(9)	.150(2)	3.6(3)
C(13)	.4667(5)	.141(1)	.142(2)	5.0(3)
C(14)	.4782(5)	.050(1)	.231(3)	4.7(3)
C(15)	.4395(5)	.004(1)	.343(2)	4.7(3)
C(16)	.3885(5)	.051(1)	.353(2)	3.8(3)
H(1)	.3638	.4691	.4412	2.8(10)
H(2)	.4050	.6364	.4218	7.4(18)
H(3)	.3750	.7661	.2237	13.0(42)
H(4)	.2971	.7318	.0569	7.4
H(5)	.2561	.5643	.0710	2.8
H(6)	.3546	.3224	.2604	2.9
H(7)	.4073	.2504	.0842	2.8
H(8)	.4938	.1743	.0735	7.4
H(9)	.5126	.0173	.2201	13.0
H(10)	.4476	-.0584	.4097	7.4
H(11)	.3626	.0231	.4319	2.8
H(12)	.2881	.0394	.2564	2.2

[a]Estimated standard deviation of last digit is in parentheses; if none is given, the parameter is subject to a constraint.

[b]For U, $B_{11} = 2.20(5)$, $B_{22} = 3.52(6)$, $B_{33} = 1.88(5)$, $B_{12} = 1.7(2)$, $B_{13} = B_{23} = 0$.

Table II.   Bond Distances (Å)

COT Ring

C(1) -C(2)	1.43(2)		
C(2) -C(9)	1.40(1)	C(1) -C(3)	1.49(2)
C(9) -C(10)	1.42(2)	C(9) -C(11)	1.51(2)
C(10)-C(1')	1.44(1)		
mean C-C	1.42(2)		

Phenyl bonded carbons		Hydrogen bonded carbons	
U-C(1)	2.66(2)	U-C(2)	2.62(2)
U-C(9)	2.68(2)	U-C(10)	2.62(2)
U-C(1')	2.69(2)	U-C(2')	2.61(2)
U-C(9')	2.67(2)	U-C(10')	2.66(2)
mean U-C	2.68(1)	mean U-C	2.63(2)

Phenyl Rings

C(3)-C(4)	1.39(2)	C(11)-C(12)	1.36(2)
C(4)-C(5)	1.39(2)	C(12)-C(13)	1.41(2)
C(5)-C(6)	1.38(2)	C(13)-C(14)	1.34(2)
C(6)-C(7)	1.39(2)	C(14)-C(15)	1.41(2)
C(7)-C(8)	1.37(2)	C(15)-C(16)	1.40(2)
C(8)-C(3)	1.41(2)	C(16)-C(11)	1.41(2)
mean C-C	1.39(1)	mean C-C	1.39(3)

Table III.   Bond Angles (°)

C(8) -C(3) -C(4)	117(1)
C(3) -C(4) -C(5)	120(1)
C(4) -C(5) -C(6)	122(1)
C(5) -C(6) -C(7)	119(1)
C(6) -C(7) -C(8)	120(1)
C(7) -C(8) -C(3)	122(1)
C(16)-C(11)-C(12)	118(1)
C(11)-C(12)-C(13)	121(1)
C(12)-C(13)-C(14)	121(1)
C(13)-C(14)-C(15)	120(1)
C(14)-C(15)-C(16)	118(1)
C(15)-C(16)-C(11)	121(1)
C(10')-C(1) -C(3)	111(2)
C(2) -C(1) -C(3)	119(1)
C(8) -C(3) -C(1)	122(2)
C(1) -C(3) -C(4)	120(2)
C(2) -C(9) -C(11)	111(2)
C(10)-C(9) -C(11)	119(1)
C(9) -C(11)-C(12)	124(1)
C(9) -C(11)-C(16)	118(2)

FIGURE CAPTIONS

Figure 1.   Structure of molecules whose centers are at $x = 1/4$. Two
            unit cells of the orthorhombic lattice which describes
            the periodicity of the hydrocarbon structure are outlined.
            The monoclinic cell, with $a_M = 2 b_0$ and $b_M = c_0$, is required
            to describe the uranium structure, which is ordered within
            each layer.

Figure 2.   Twinning and relation of the monoclinic cell (solid lines)
            to the orthorhombic lattice (broken lines). The numbers
            indicate z coordinates (x 100) of uranium atoms (orthorhombic).
            The uranium atoms just right of center are at the twin
            boundary and have coordinates appropriate to either
            orientation.

Figure 3.   The $U(C_8H_4(C_6H_5)_4)_2$ molecule viewed down the orthorhombic
            $\underline{c}$ axis. One may see that the COT rings are not quite eclipsed.

Figure 4.   One ligand showing the angles in the COT ring and the
            numbering of the atoms. For each of these angles, $\sigma = 2°$.

Figure 5.   Molecular packing viewed down the orthorhombic $\underline{c}$ axis.
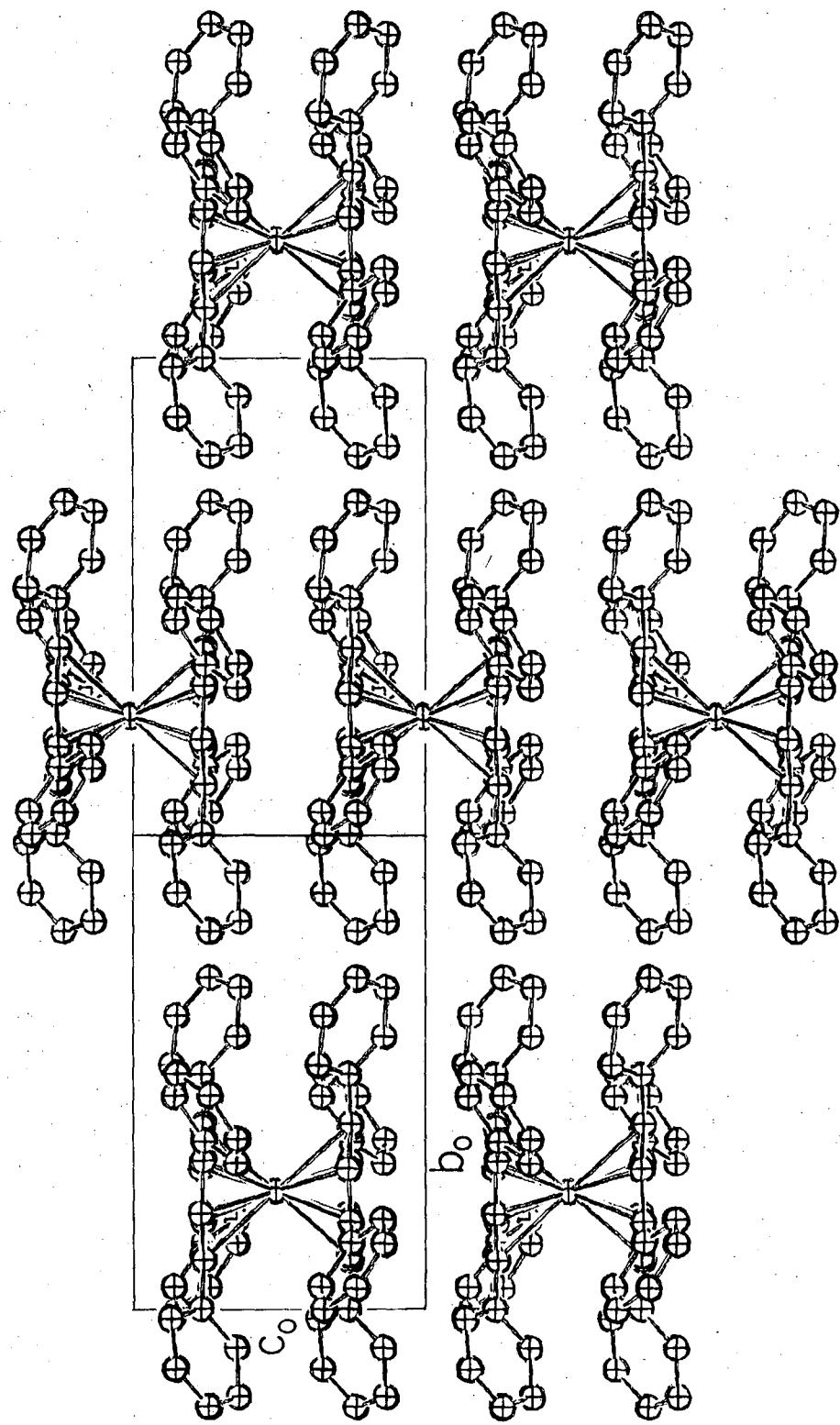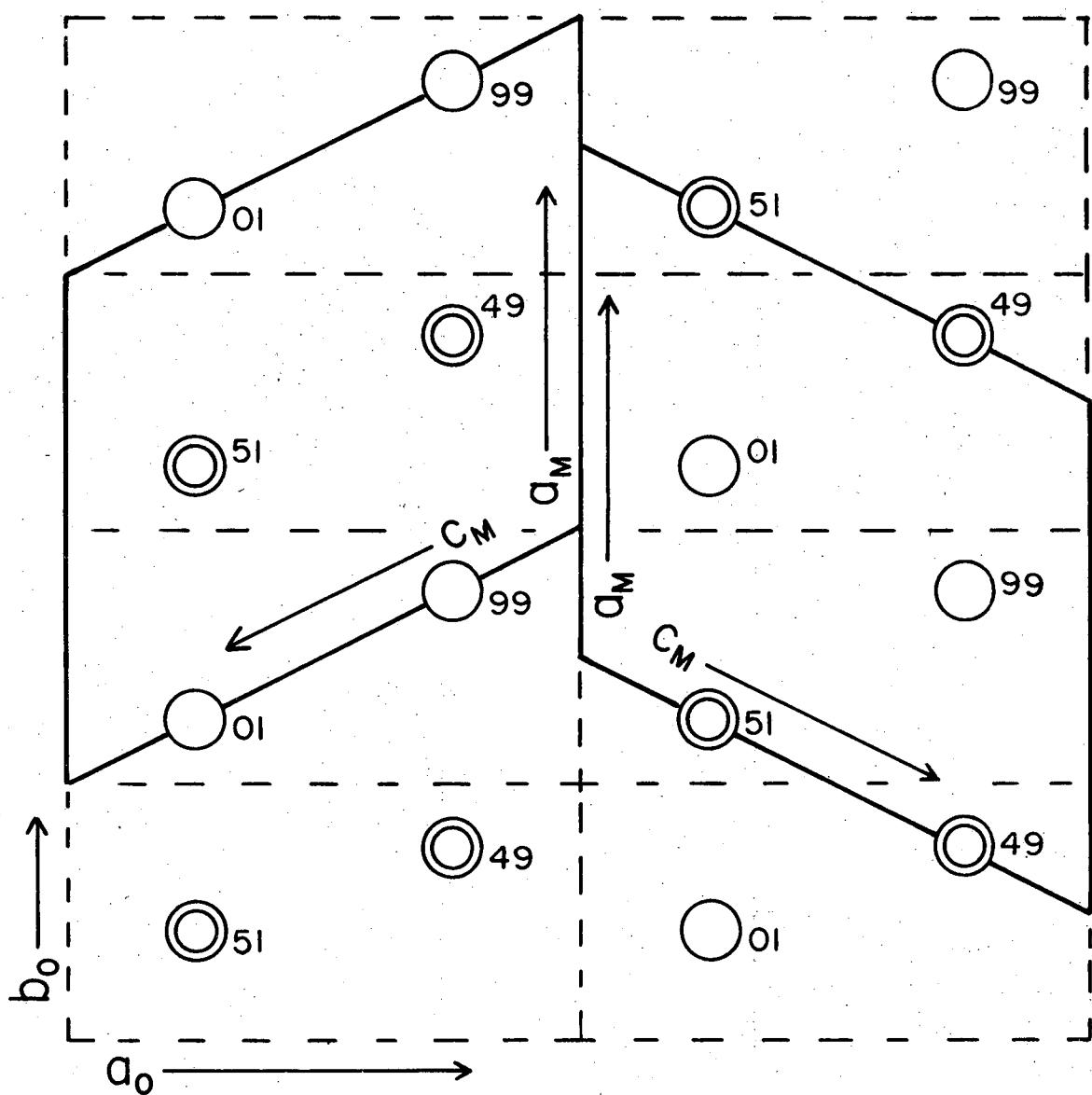
Fig. 1

XBL 766-8387

Fig. 2

XBL 766-8385

Fig. 3

Fig. 4

XBL 766-8386

b_o

a_o
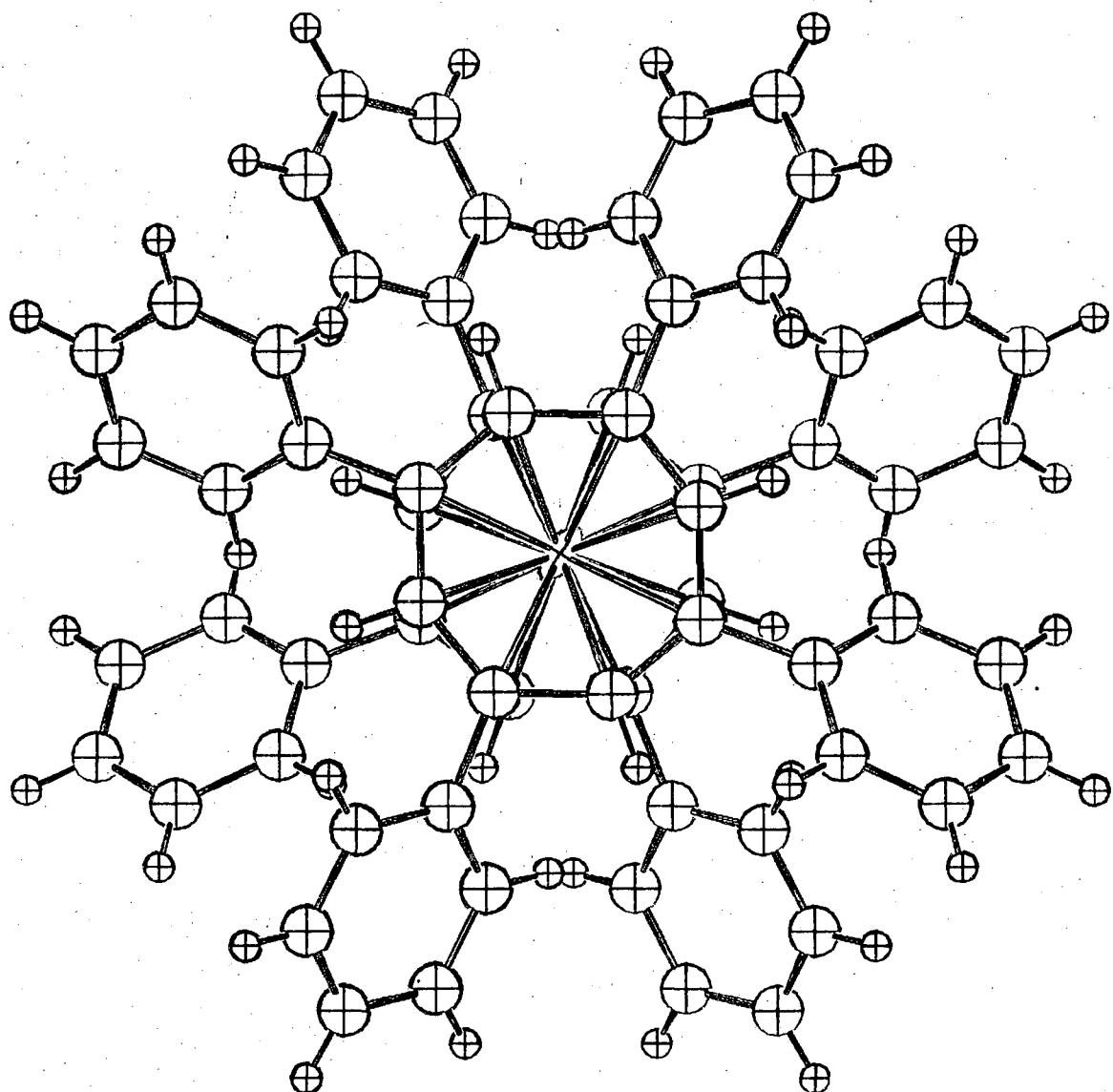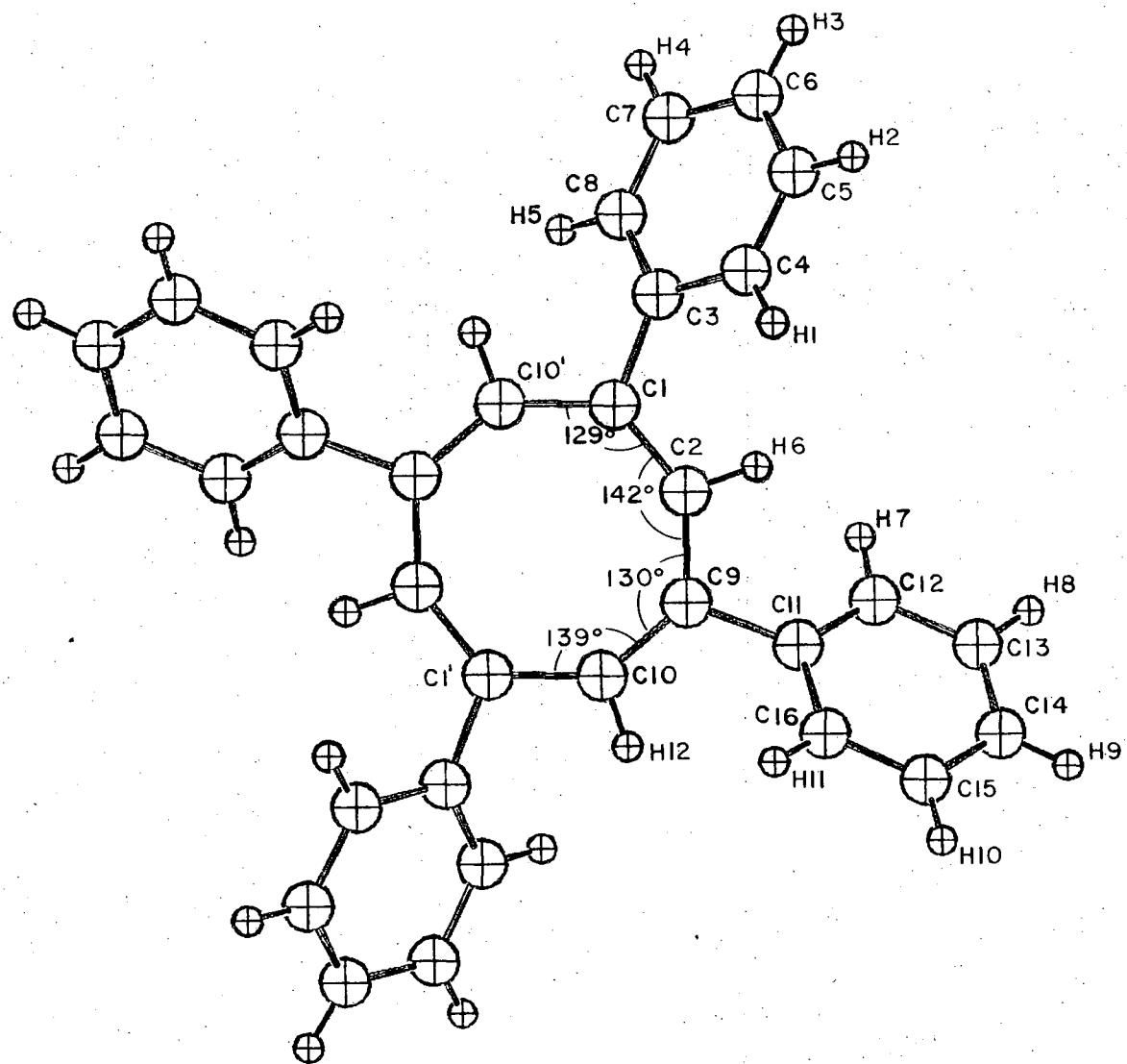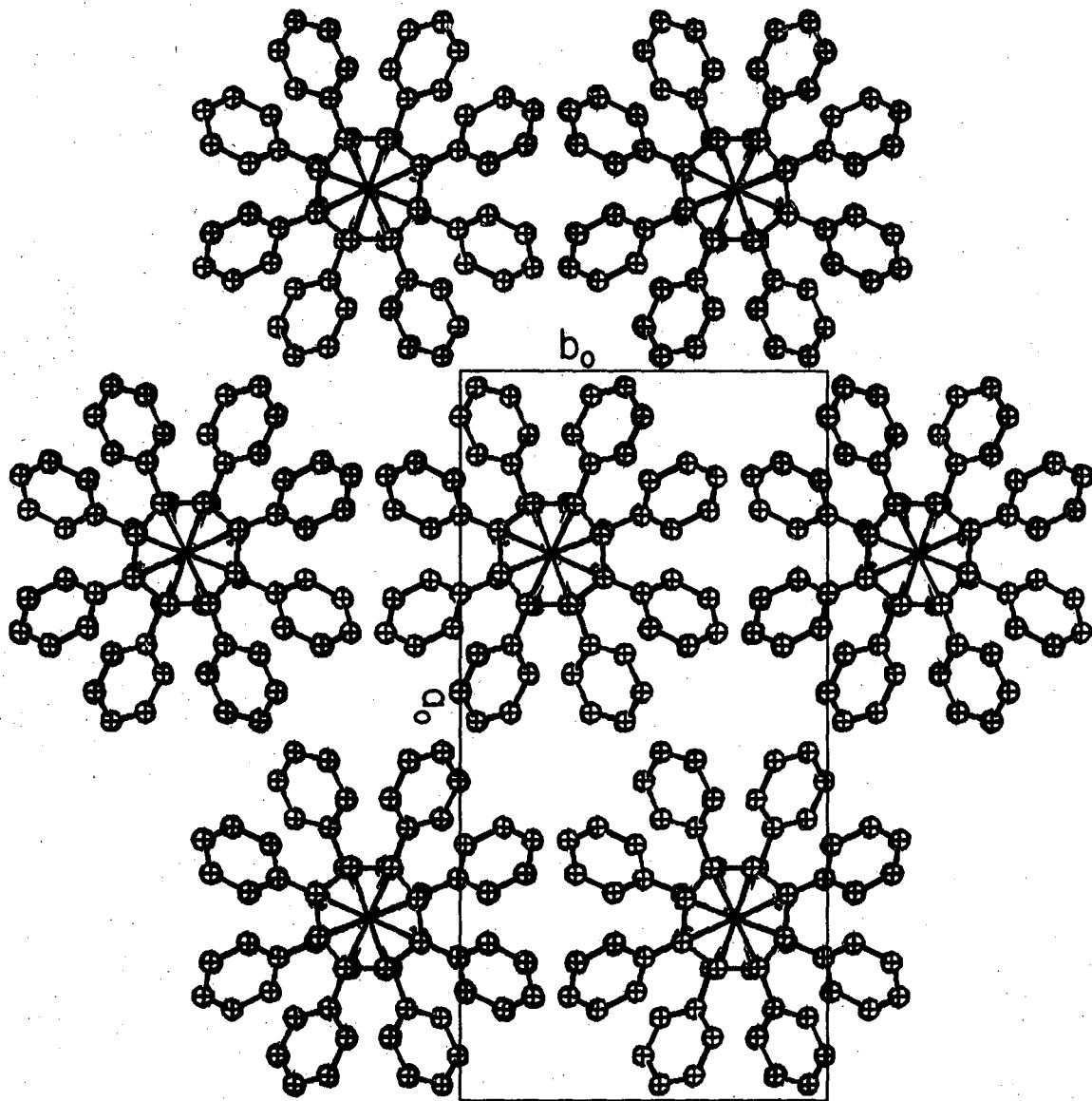
XBL 766-8388

Fig. 5

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL X 3.0)

F(0,0,0) = 3081

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

L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL	L	FOB	SG	DEL
H,K=	0,	0		H,K=	0,	6		0	674	25	-31	0	285	6	4	0	542	5	-4
1	0	3	0*	0	77	34	-3	1	30	3	1*	1	13	3	-35*	1	66	20	53
2	593	10	-2	1	0	3	0*	2	126	40	-0	2	232	6	-2	2	306	10	-4
3	37	3	37*	2	412	6	5	3	35	3	12*	3	10	3	-19*	3	25	3	7*
4	398	6	5	3	55	3	55*	4	523	5	-1	4	219	10	-4	4	333	5	-10
5	20	3	20*	4	200	13	-28	5	30	3	22*	5	0	3	-10*	5	25	3	24*
6	106	24	-8	5	34	3	34*	6	119	18	4	6	128	15	8	6	202	8	7
7	0	3	0*	6	173	16	-5	7	59	3	50*	H,K=	1,	8		7	74	49	67
8	283	9	-25	7	0	3	0*	8	192	9	8	0	23	3	23*	8	149	12	16
H,K=	0,	1		H,K=	0,	7		H,K=	1,	2		1	51	3	-42*	H,K=	2,	3	
1	15	3	15*	0	36	3	36*	0	0	3	0*	2	77	30	14	0	34	3	34*
2	79	3	-14*	1	40	3	40*	1	498	10	-6	3	3	3	-7*	1	20	3	14*
3	41	3	41*	2	67	3	11*	2	261	12	-14	4	80	41	2	2	120	14	-8
4	110	18	7	3	39	3	39*	3	26	3	17*	5	29	3	23*	3	63	22	10
5	31	3	31*	4	73	31	16	4	30	3	-13*	H,K=	1,	9		4	64	23	20
6	72	26	34	5	0	3	0*	5	76	26	23	0	220	7	4	5	62	33	27
7	92	28	92*	H,K=	0,	8		6	53	39	3	1	0	3	-3*	6	92	23	11
H,K=	0,	2		0	218	8	-4	7	44	3	20*	2	197	10	-2	7	79	33	47
0	253	7	-6	1	40	3	40*	H,K=	1,	3		3	0	3	-11*	H,K=	2,	4	
1	38	3	38*	2	220	13	4	0	352	8	2	4	161	25	15	0	283	10	-2
2	402	9	-11	3	52	3	52*	1	146	33	-9	5	70	35	57	1	126	19	-8
3	24	3	24*	4	203	18	-0	2	507	6	2	6	124	15	6	2	442	5	3
4	394	8	-2	5	78	3	78*	3	133	9	6	H,K=	1,	10		3	139	10	-15
5	52	3	52*	6	129	17	13	4	157	8	2	0	0	3	0*	4	228	6	1
6	167	16	8	H,K=	0,	9		5	45	3	29*	1	33	3	25*	5	20	3	8*
7	0	3	0*	0	27	3	27*	6	256	7	-7	2	92	63	75	6	216	16	9
8	135	15	1	1	36	3	36*	7	86	3	40*	3	33	3	27*	7	40	3	3*
H,K=	0,	3		2	64	46	-44	H,K=	1,	4		H,K=	1,	11		H,K=	2,	5	
0	40	3	40*	3	84	3	84*	0	29	3	29*	0	100	76	6	0	0	3	0*
1	0	3	0*	4	45	3	36*	1	0	3	-9*	1	31	3	25*	1	22	3	-40*
2	71	3	1*	H,K=	0,	10		2	34	3	-9*	2	198	11	7	2	114	12	20
3	42	3	42*	0	102	20	27	3	44	3	22*	4	65	44	3	3	100	13	14
4	81	29	-8	1	35	3	35*	4	74	21	-11	H,K=	1,	13		4	38	3	-9*
5	74	26	74*	2	260	12	1	5	64	27	61	0	85	61	3	5	55	45	-25
6	79	3	21*	3	0	3	0*	6	124	26	18	H,K=	2,	0		6	71	32	25
7	88	3	88*	4	108	30	33	H,K=	1,	5		1	0	3	0*	7	0	3	-41*
H,K=	0,	4		5	48	3	48*	0	230	7	-13	2	117	41	-7	H,K=	2,	6	
0	323	7	2	H,K=	0,	11		1	0	3	-20*	3	42	3	42*	0	350	5	-3
1	0	3	0*	0	31	3	31*	2	421	5	7	4	506	5	1	1	0	3	-2*
2	416	7	-8	1	0	3	0*	3	18	3	1*	5	53	3	53*	2	302	5	-4
3	21	3	21*	2	85	55	60	4	182	8	-2	6	105	22	9	3	25	3	12*
4	242	12	4	H,K=	0,	12		5	48	3	35*	7	48	3	48*	4	166	13	3
5	23	3	23*	0	95	33	9	6	202	17	-14	8	206	9	-5	5	37	3	26*
6	232	27	12	2	156	15	-2	7	65	54	61	H,K=	2,	1		6	196	17	-6
7	24	3	24*	H,K=	1,	0		H,K=	1,	6		0	0	3	0*	7	47	3	27*
H,K=	0,	5		1	11	3	11*	0	41	3	41*	1	29	3	20*	H,K=	2,	7	
0	10	3	10*	2	0	3	-32*	1	50	3	-15*	2	221	18	-3	0	55	3	55*
1	0	3	0*	3	35	3	35*	2	74	20	2	3	46	3	18*	1	0	3	-38*
2	98	16	8	4	137	9	-1	3	36	3	20*	4	132	12	-10	2	35	3	-16*
3	23	3	23*	5	32	3	32*	4	49	3	1*	5	43	3	38*	3	54	43	-2
4	95	22	12	6	34	3	-6*	5	53	3	39*	6	96	24	6	4	66	35	20
5	11	3	11*	7	21	3	21*	6	43	3	-26*	7	0	3	-9*	5	51	3	3*
6	98	3	-12*	H,K=	1,	1		H,K=	1,	7		H,K=	2,	2		H,K=	2,	8	

STRUCTURE FACTORS CONTINUED FOR

L FOB SG DEL	L FOB SG DEL	L FOB SG DEL	L FOB SG DEL	L FOB SG DEL
0 234 6 -2	1 131 30 -23	2 0 3 -25*	2 57 33 -5	7 48 3 48*
1 31 3 11*	2 389 6 5	3 25 3 12*	3 58 29 32	H,K= 5, 1
2 192 9 15	3 101 15 6	H,K= 3, 11	4 47 3 14*	0 653 5 4
3 29 3 -17*	4 224 6 -11	0 63 3 13*	5 37 3 29*	1 73 3 40*
4 238 10 -10	5 34 3 23*	1 33 3 21*	6 69 34 -16	2 333 8 1
5 46 3 41*	6 228 15 0	2 200 21 -6	H,K= 4, 6	3 45 3 37*
6 108 16 13	7 23 3 -2*	4 89 20 6	0 455 5 5	4 224 7 -12
H,K= 2, 9	H,K= 3, 4	H,K= 3, 13	1 0 3 -6*	5 35 3 5*
0 0 3 0*	0 29 3 29*	0 115 19 -7	2 189 7 0	6 239 8 5
1 38 3 -59*	1 53 3 -25*	H,K= 4, 0	3 52 3 23*	7 81 29 53
2 52 3 -14*	2 73 24 -18	0 231 6 -7	4 255 8 -3	8 152 11 15
3 72 23 32	3 115 14 -6	1 13 3 13*	5 59 3 43*	H,K= 5, 2
4 51 3 30*	4 74 21 28	2 414 8 -10	6 137 35 7	0 43 3 43*
H,K= 2, 10	5 102 18 19	3 41 3 41*	7 94 42 88	1 0 3 -26*
0 86 27 31	6 34 3 -37*	4 397 6 1	H,K= 4, 7	2 0 3 -10*
1 44 3 -17*	H,K= 3, 5	5 58 40 58*	0 53 3 53*	3 65 22 26
2 261 18 -7	0 256 7 -8	6 173 12 2	1 0 3 -15*	4 56 29 -8
3 70 3 19*	1 29 3 26*	7 57 3 57*	2 57 43 46	5 57 3 46*
4 60 3 -4*	2 316 5 -2	8 132 12 4	3 76 31 72	6 95 31 2
5 61 3 51*	3 42 3 -2*	H,K= 4, 1	4 55 3 -18*	7 23 3 16*
H,K= 2, 11	4 259 9 6	0 0 3 0*	5 0 3 -13*	H,K= 5, 3
0 0 3 0*	5 42 3 30*	1 511 5 0	H,K= 4, 8	0 287 12 -2
1 38 3 2*	6 170 23 -1	2 0 3 -11*	0 267 6 2	1 131 15 -30
2 0 3 -5*	7 51 3 39*	3 50 41 41	1 32 3 25*	2 461 5 -11
H,K= 2, 12	H,K= 3, 6	4 77 19 -3	2 171 9 1	3 145 10 13
0 108 30 -22	0 42 3 42*	5 87 18 12	3 39 3 -11*	4 232 7 1
2 125 20 1	1 0 3 -17*	6 95 16 -9	4 230 10 24	5 27 3 9*
H,K= 3, 0	2 38 3 -33*	7 57 3 42*	5 0 3 -9*	6 240 11 23
0 0 3 0*	3 63 28 -4	H,K= 4, 2	6 104 17 -10	7 5 3 -23*
1 26 3 26*	4 62 33 20	0 266 5 6	H,K= 4, 9	H,K= 5, 4
2 261 16 0	5 61 3 1*	1 40 3 28*	0 52 3 52*	0 15 3 15*
3 46 3 46*	6 58 3 7*	2 607 6 4	1 16 3 -31*	1 60 3 -11*
4 76 20 -13	H,K= 3, 7	3 21 3 10*	2 39 3 3*	2 29 3 6*
5 42 3 42*	0 419 6 -2	4 141 9 -8	3 52 3 -6*	3 55 32 -12
6 49 3 30*	1 35 3 -27*	5 45 3 29*	4 42 3 1*	4 99 18 -2
7 0 3 0*	2 180 9 4	6 261 9 -6	H,K= 4, 10	5 49 3 20*
H,K= 3, 1	3 51 47 3	7 57 3 55*	0 156 13 3	6 97 21 7
0 382 6 -1	4 190 13 -6	H,K= 4, 3	1 44 3 0*	H,K= 5, 5
1 35 3 10*	5 0 3 -23*	0 0 3 0*	2 196 22 16	0 295 5 5
2 292 14 -6	6 141 12 -3	1 24 3 -16*	3 0 3 -8*	1 22 3 2*
3 26 3 13*	H,K= 3, 8	2 82 27 6	4 108 17 -6	2 243 7 -2
4 456 5 -2	0 40 3 40*	3 83 20 3	5 52 3 44*	3 60 34 31
5 40 3 34*	1 13 3 -14*	4 43 3 -9*	H,K= 4, 11	4 310 7 -6
6 156 10 6	2 57 40 18	5 77 26 33	0 0 3 0*	5 22 3 18*
7 0 3 -4*	3 29 3 22*	6 89 34 10	1 0 3 -27*	6 136 34 -1
8 133 14 -10	4 75 37 47	H,K= 4, 4	2 47 3 -1*	7 64 3 46*
H,K= 3, 2	5 29 3 19*	0 262 8 -1	H,K= 4, 12	H,K= 5, 6
0 26 3 26*	H,K= 3, 9	1 0 3 -39*	0 149 21 1	0 60 38 60*
1 217 12 3	0 196 10 -6	2 353 5 3	2 101 18 -12	1 23 3 -7*
2 230 13 7	1 6 3 -31*	3 58 44 28	H,K= 5, 0	2 34 3 -5*
3 2 3 2*	2 221 8 3	4 282 7 -5	0 0 3 0*	3 35 3 -10*
4 125 13 -11	3 56 3 -26*	5 0 3 -11*	1 73 3 73*	4 48 3 -3*
5 55 31 29	4 88 26 -5	6 181 16 -9	2 282 10 -4	5 56 3 16*
6 126 17 2	5 49 3 47*	7 66 3 35*	3 59 32 59*	6 71 3 10*
7 54 3 29*	H,K= 3, 10	H,K= 4, 5	4 26 3 -22*	H,K= 5, 7
H,K= 3, 3	0 22 3 22*	0 0 3 0*	5 58 42 58*	0 280 6 -9
0 419 10 -8	1 28 3 2*	1 189 7 -7	6 53 3 16*	1 7 3 -43*

STRUCTURE FACTORS CONTINUED FOR

L	FOB	SG	DEL
2	205	10	0
3	0	3	-5*
4	250	9	-12
5	44	3	33*
6	52	3	-50*
H,K= 5, 8			
0	60	26	60*
1	35	3	25*
2	57	3	33*
3	0	3	-17*
4	54	3	-8*
5	19	3	-7*
H,K= 5, 9			
0	240	9	-8
1	0	3	-21*
2	182	9	6
3	0	3	-28*
4	127	16	11
5	58	3	46*
H,K= 5, 10			
0	25	3	25*
1	56	3	-9*
2	22	3	-23*
3	41	3	26*
H,K= 5, 11			
0	76	3	3*
1	0	3	-15*
2	162	14	-13
4	103	19	-13
H,K= 5, 13			
0	124	17	0
H,K= 6, 0			
0	204	12	-5
1	45	3	45*
2	585	6	9
3	49	3	49*
4	154	12	-15
5	45	3	45*
6	275	8	-8
7	16	3	16*
H,K= 6, 1			
0	40	3	40*
1	33	3	27*
2	146	9	-1
3	46	3	4*
4	32	3	-10*
5	80	19	43
6	85	22	1
7	17	3	-4*
H,K= 6, 2			
0	306	15	-7
1	238	14	15
2	448	6	-1
3	206	7	-1
4	231	7	-2
5	45	3	10*
6	229	8	5

L	FOB	SG	DEL
7	39	3	-15*
H,K= 6, 3			
0	0	3	0*
1	21	3	7*
2	173	7	-10
3	48	3	31*
4	10	3	-8*
5	60	37	59
6	80	27	5
H,K= 6, 4			
0	218	8	-9
1	0	3	-79*
2	430	5	-2
3	53	36	-5
4	192	8	17
5	24	3	-9*
6	224	15	-7
7	0	3	-18*
H,K= 6, 5			
0	32	3	32*
1	0	3	-28*
2	44	3	-8*
3	25	3	14*
4	78	24	-21
5	32	3	11*
6	89	29	5
H,K= 6, 6			
0	442	6	11
1	0	3	-23*
2	201	8	4
3	55	40	32
4	257	7	-1
5	0	3	-25*
6	119	22	11
7	72	39	63
H,K= 6, 7			
0	43	3	43*
1	30	3	14*
2	41	3	30*
3	112	17	-23
4	86	19	16
5	136	23	12
H,K= 6, 8			
0	304	6	8
1	5	3	-44*
2	130	13	1
3	45	3	39*
4	254	11	-5
5	52	3	38*
6	70	36	-0
H,K= 6, 9			
0	53	3	53*
1	0	3	-9*
2	43	3	2*
3	31	3	-31*
4	46	3	-31*
H,K= 6, 10			

L	FOB	SG	DEL
0	195	13	6
1	43	3	18*
2	102	41	-34
3	17	3	-12*
4	161	10	1
5	35	3	23*
H,K= 6, 11			
0	42	3	42*
1	0	3	-2*
2	0	3	-22*
H,K= 6, 12			
0	134	22	7
2	99	19	-10
H,K= 7, 0			
0	0	3	0*
1	18	3	18*
2	107	21	12
3	59	24	59*
4	78	23	-12
5	23	3	23*
6	147	13	15
H,K= 7, 1			
0	324	13	-14
1	171	21	-14
2	470	6	6
3	148	9	1
4	213	6	2
5	20	3	8*
6	241	9	12
7	98	27	47
H,K= 7, 2			
0	25	3	25*
1	90	25	4
2	0	3	-44*
3	117	11	24
4	39	3	-5*
5	62	3	-3*
6	73	65	-26
H,K= 7, 3			
0	330	7	3
1	69	44	22
2	433	5	7
3	88	18	-16
4	200	7	1
5	54	47	49
6	229	9	7
7	0	3	-14*
H,K= 7, 4			
0	35	3	35*
1	35	3	29*
2	169	8	0
3	59	24	16
4	37	3	13*
5	50	3	2*
6	41	3	-13*
H,K= 7, 5			
0	346	6	-3

L	FOB	SG	DEL
1	0	3	-24*
2	340	5	1
3	41	3	-7*
4	168	10	20
5	42	3	37*
6	221	11	14
7	37	3	17*
H,K= 7, 6			
0	32	3	32*
1	41	3	39*
2	8	3	-14*
3	51	3	36*
4	87	19	-7
5	62	3	34*
H,K= 7, 7			
0	252	7	-7
1	37	3	24*
2	151	10	-4
3	0	3	-64*
4	315	6	-9
5	61	3	41*
6	82	26	17
H,K= 7, 8			
0	39	3	39*
1	18	3	-23*
2	51	3	19*
3	64	32	57
4	55	3	10*
H,K= 7, 9			
0	207	9	-16
1	26	3	-24*
2	138	20	12
3	37	3	30*
4	210	11	-16
5	28	3	12*
H,K= 7, 10			
0	63	46	63*
1	75	33	-4
2	0	3	-32*
3	9	3	-11*
H,K= 7, 11			
0	155	26	-9
1	65	40	42
2	105	26	-9
4	125	14	6
H,K= 8, 0			
0	332	9	-9
1	8	3	8*
2	407	5	-10
3	29	3	29*
4	264	7	-7
5	38	3	38*
6	204	13	-6
7	58	3	58*
H,K= 8, 1			
0	0	3	0*
1	56	3	9*

L	FOB	SG	DEL
2	96	20	-29
3	70	23	36
4	36	3	-13*
5	38	3	2*
6	58	3	-17*
H,K= 8, 2			
0	274	9	-10
1	12	3	-1*
2	341	5	0
3	53	36	27
4	296	7	0
5	63	30	41
6	188	15	-8
7	34	3	20*
H,K= 8, 3			
0	36	3	36*
1	34	3	2*
2	38	3	3*
3	94	22	-7
4	79	19	-11
5	53	3	-1*
6	87	60	7
H,K= 8, 4			
0	139	10	-13
1	32	3	-7*
2	462	5	15
3	18	3	-4*
4	174	9	-7
5	83	20	77
6	207	13	1
7	56	3	37*
H,K= 8, 5			
0	29	3	29*
1	121	12	-5
2	52	3	22*
3	46	3	27*
4	36	3	-25*
5	77	26	14
6	67	3	-9*
H,K= 8, 6			
0	425	5	4
1	33	3	24*
2	164	10	-11
3	21	3	-42*
4	215	10	7
5	33	3	-17*
6	147	13	5
H,K= 8, 7			
0	53	3	53*
1	22	3	-6*
2	37	3	-2*
3	0	3	-30*
4	56	3	5*
5	27	3	19*
H,K= 8, 8			
0	282	6	6
1	28	3	5*

STRUCTURE FACTORS CONTINUED FOR

Column 1

L	FOB	SG	DEL
2	165	10	-2
3	56	3	9*
4	169	33	-9
5	30	3	4*
6	94	29	-5
H,K= 8, 9			
0	52	3	52*
1	0	3	-10*
2	31	3	7*
3	43	3	21*
4	28	3	-6*
H,K= 8, 10			
0	265	14	-17
1	0	3	-48*
2	68	43	-4
3	10	3	-4*
4	174	11	10
H,K= 8, 11			
0	57	3	57*
1	39	3	30*
H,K= 8, 12			
0	74	49	21
2	144	12	2
H,K= 9, 0			
0	0	3	0*
1	21	3	21*
2	80	3	-18*
3	58	37	58*
4	41	3	-7*
5	72	22	72*
6	134	27	-35
H,K= 9, 1			
0	281	7	-4
1	27	3	-13*
2	460	5	3
3	57	51	33
4	211	7	9
5	58	3	40*
6	195	15	-18
7	0	3	-33*
H,K= 9, 2			
0	25	3	25*
1	153	12	9
2	40	3	7*
3	40	3	28*
4	66	25	-8
5	44	3	29*
6	68	3	-4*
H,K= 9, 3			
0	173	10	-12
1	22	3	2*
2	446	5	8
3	48	3	-6*
4	201	7	10
5	60	35	31
6	217	11	3
7	69	37	65

Column 2

L	FOB	SG	DEL
H,K= 9, 4			
0	27	3	27*
1	114	11	9
2	59	25	39
3	43	3	27*
4	56	3	27*
5	53	3	29*
6	89	31	1
H,K= 9, 5			
0	261	7	-4
1	42	3	36*
2	268	6	2
3	60	28	-9
4	241	7	-10
5	68	40	48
6	196	12	36
7	61	3	52*
H,K= 9, 6			
0	25	3	25*
1	10	3	-1*
2	52	36	-2
3	26	3	17*
4	81	31	20
5	63	3	40*
H,K= 9, 7			
0	292	6	2
1	44	3	-40*
2	182	9	6
3	43	3	25*
4	190	11	-9
5	50	3	19*
6	113	18	-13
H,K= 9, 8			
0	46	3	46*
1	37	3	19*
2	59	33	-1
3	45	3	33*
4	39	3	4*
H,K= 9, 9			
0	255	7	-3
1	43	3	26*
2	110	23	6
3	72	32	3
4	202	9	-9
5	32	3	2*
H,K= 9, 10			
0	0	3	0*
1	12	3	5*
2	0	3	-43*
H,K= 9, 11			
0	207	13	-19
2	83	29	9
H,K= 10, 0			
0	202	9	-10
1	28	3	28*
2	442	6	5
3	42	3	42*

Column 3

L	FOB	SG	DEL
4	204	11	-14
5	25	3	25*
6	207	25	-13
7	46	3	46*
H,K= 10, 1			
0	11	3	11*
1	75	20	4
2	111	11	19
3	93	16	3
4	36	3	-17*
5	62	3	-6*
6	39	3	-14*
H,K= 10, 2			
0	296	6	-5
1	46	3	6*
2	234	7	-2
3	40	3	30*
4	347	6	-3
5	32	3	30*
6	159	21	12
7	0	3	-29*
H,K= 10, 3			
0	30	3	30*
1	41	3	25*
2	71	27	14
3	50	3	28*
4	63	43	-37
5	36	3	28*
6	100	22	2
H,K= 10, 4			
0	368	5	5
1	41	3	-33*
2	299	6	-1
3	46	3	32*
4	184	9	8
5	61	3	43*
6	200	12	8
7	74	35	44
H,K= 10, 5			
0	24	3	24*
1	15	3	-3*
2	65	41	-7
3	41	3	17*
4	32	3	-5*
5	0	3	-26*
H,K= 10, 6			
0	235	9	3
1	23	3	-38*
2	223	10	-6
3	42	3	22*
4	252	9	2
5	0	3	-13*
6	119	15	11
H,K= 10, 7			
0	43	3	43*
1	52	39	22
2	23	3	17*

Column 4

L	FOB	SG	DEL
3	53	3	51*
4	43	3	-6*
5	0	3	-21*
H,K= 10, 8			
0	273	6	3
1	45	3	6*
2	199	10	1
3	0	3	-43*
4	84	44	4
5	23	3	-19*
H,K= 10, 9			
0	39	3	39*
1	25	3	4*
2	0	3	-40*
3	56	3	22*
H,K= 10, 10			
0	163	22	-7
1	28	3	-14*
2	126	18	-2
4	125	16	-24
H,K= 10, 12			
0	71	61	-26
H,K= 11, 0			
0	6	3	6*
1	44	3	44*
2	0	3	-46*
3	27	3	27*
4	55	52	-22
5	53	3	53*
6	111	22	8
H,K= 11, 1			
0	276	6	-5
1	33	3	30*
2	401	5	1
3	18	3	-2*
4	177	11	-2
5	59	45	36
6	207	14	-15
7	57	3	46*
H,K= 11, 2			
0	22	3	22*
1	95	26	9
2	97	21	8
3	55	48	-0
4	72	38	39
5	38	3	25*
6	0	3	-58*
H,K= 11, 3			
0	462	6	11
1	43	3	36*
2	219	11	-2
3	60	35	10
4	274	10	-2
5	73	31	54
6	135	16	-5
7	83	27	69
H,K= 11, 4			

Column 5

L	FOB	SG	DEL
0	33	3	33*
1	44	3	-14*
2	50	3	44*
3	71	64	5
4	67	38	-4
5	66	3	14*
6	64	3	-5*
H,K= 11, 5			
0	225	7	-4
1	34	3	-17*
2	270	7	7
3	65	34	54
4	237	10	-3
5	0	3	-50*
6	120	20	-11
H,K= 11, 6			
0	0	3	0*
1	74	27	30
2	85	22	17
3	57	3	34*
4	58	3	15*
5	0	3	-12*
H,K= 11, 7			
0	283	7	9
1	50	3	-31*
2	201	10	-1
3	71	39	63
4	146	23	-0
5	66	41	37
6	129	22	-15
H,K= 11, 8			
0	41	3	41*
1	37	3	13*
2	38	3	34*
3	66	3	37*
4	0	3	-43*
H,K= 11, 9			
0	123	26	27
1	0	3	-49*
2	206	17	7
3	44	3	9*
4	153	11	-6
5	33	3	-9*
H,K= 11, 10			
0	0	3	0*
1	0	3	-75*
2	67	53	34
H,K= 11, 11			
0	144	14	-6
2	124	24	13
H,K= 12, 0			
0	87	15	-7
1	32	3	32*
2	438	7	-5
3	0	3	0*
4	230	8	8
5	52	3	52*

STRUCTURE FACTORS CONTINUED FOR

L	FOB	SG	DEL		L	FOB	SG	DEL		L	FOB	SG	DEL		L	FOB	SG	DEL		L	FOB	SG	DEL
6	206	22	11		0	99	16	9		0	327	7	-0		6	127	24	-20		0	295	6	-8
7	61	3	61*		1	42	3	18*		1	69	52	51		H,K= 14, 3					1	0	3	-10*
H,K= 12, 1					2	255	13	-18		2	195	9	12		0	31	3	31*		2	203	8	-3
0	14	3	14*		3	17	3	-58*		3	61	36	-36		1	59	3	30*		3	49	3	15*
1	55	30	2		4	110	25	6		4	225	8	1		2	34	3	27*		4	276	7	-9
2	59	30	-12		5	88	27	46		5	4	3	-48*		3	111	15	-1		5	57	3	43*
3	46	3	17*		H,K= 12, 9					6	127	13	8		4	93	18	21		6	133	17	17
4	55	3	-6*		0	16	3	16*		H,K= 13, 6					5	122	34	-4		H,K= 15, 2			
5	45	3	8*		1	96	3	15*		0	43	3	43*		H,K= 14, 4					0	31	3	31*
6	64	3	6*		2	0	3	-23*		1	58	41	2		0	269	6	-3		1	0	3	-1*
H,K= 12, 2					3	34	3	-33*		2	72	22	25		1	48	3	9*		2	62	35	24
0	476	6	6		H,K= 12, 10					3	45	3	31*		2	175	10	-16		3	45	3	44*
1	39	3	-7*		0	134	35	12		4	65	3	-2*		3	56	41	17		4	42	3	-8*
2	220	7	-7		1	31	3	-31*		H,K= 13, 7					4	281	8	6		5	53	3	48*
3	73	35	64		2	154	12	0		0	121	12	1		5	53	3	24*		H,K= 15, 3			
4	243	7	2		4	118	21	-6		1	0	3	-31*		6	97	24	-7		0	318	6	2
5	53	3	49*		H,K= 13, 0					2	281	8	-17		H,K= 14, 5					1	19	3	2*
6	146	20	-17		0	0	3	0*		3	51	3	-22*		0	15	3	15*		2	144	13	-13
7	23	3	5*		1	0	3	0*		4	56	3	-35*		1	32	3	28*		3	43	3	4*
H,K= 12, 3					2	66	3	-66*		5	70	3	37*		2	0	3	-6*		4	336	7	6
0	34	3	34*		3	27	3	27*		H,K= 13, 8					3	41	3	18*		5	26	3	-2*
1	46	3	2*		4	61	31	19		0	28	3	28*		4	28	3	-31*		6	80	41	4
2	0	3	-34*		5	55	3	55*		1	59	3	7*		5	68	49	56		H,K= 15, 4			
3	70	56	-2		6	68	50	14		2	69	3	3*		H,K= 14, 6					0	43	3	43*
4	80	28	9		H,K= 13, 1					3	25	3	-23*		0	170	9	-6		1	64	36	18
5	100	36	54		0	464	5	6		H,K= 13, 9					1	32	3	-28*		2	35	3	-14*
6	32	3	-32*		1	19	3	-6*		0	75	34	-15		2	254	7	-3		3	54	3	31*
H,K= 12, 4					2	230	8	-4		1	20	3	-59*		3	64	3	23*		4	0	3	-57*
0	437	6	1		3	52	3	13*		2	211	11	-9		4	131	20	-12		5	77	35	42
1	39	3	-46*		4	211	8	-2		4	96	23	6		5	55	3	34*		H,K= 15, 5			
2	181	9	-3		5	35	3	2*		H,K= 13, 10					H,K= 14, 7					0	287	7	-7
3	52	3	46*		6	181	20	3		0	70	38	70*		0	43	3	43*		1	46	3	20*
4	208	9	-8		7	19	3	-9*		H,K= 13, 11					1	39	3	16*		2	227	9	-3
5	70	58	62		H,K= 13, 2					0	59	3	-70*		2	87	28	-3		3	55	3	-14*
6	145	13	2		0	0	3	0*		2	134	12	14		3	13	3	-5*		4	103	35	8
H,K= 12, 5					1	79	24	8		H,K= 14, 0					4	42	3	29*		5	35	3	5*
0	0	3	0*		2	39	3	22*		0	259	6	-2		H,K= 14, 8					6	186	13	7
1	50	3	32*		3	50	3	32*		1	41	3	41*		0	128	29	23		H,K= 15, 6			
2	97	16	-5		4	85	18	5		2	293	6	-3		1	0	3	-49*		0	20	3	20*
3	55	34	41		5	27	3	5*		3	43	3	43*		2	223	18	-11		1	32	3	30*
4	60	34	30*		6	0	3	-30*		4	238	8	8		3	3	3	-47*		2	22	3	10*
5	38	3	3*		H,K= 13, 3					5	62	3	62*		4	110	19	10		3	18	3	10*
H,K= 12, 6					0	336	6	1		6	158	10	5		H,K= 14, 9					4	0	3	-37*
0	205	8	7		1	46	3	1*		H,K= 14, 1					0	43	3	43*		H,K= 15, 7			
1	0	3	-51*		2	202	8	-9		0	0	3	0*		1	27	3	9*		0	112	17	-3
2	230	7	-6		3	23	3	16*		1	43	3	-12*		2	0	3	-13*		1	40	3	-28*
3	62	49	24		4	320	6	-5		2	28	3	-7*		H,K= 14, 10					2	259	13	-6
4	221	13	7		5	96	49	83		3	33	3	-24*		0	195	11	5		3	34	3	-4*
5	0	3	-28*		6	78	31	-15		4	36	3	-21*		2	106	17	-13		4	76	3	-16*
6	111	17	-2		H,K= 13, 4					5	53	3	20*		H,K= 15, 0					5	0	3	-27*
H,K= 12, 7					0	23	3	23*		H,K= 14, 2					0	0	3	0*		H,K= 15, 8			
0	49	3	49*		1	71	23	68		0	400	6	1		1	21	3	21*		0	25	3	25*
1	55	3	-14*		2	31	3	-22*		1	31	3	25*		2	0	3	-55*		1	33	3	26*
2	40	3	17*		3	58	3	42*		2	235	7	5		3	23	3	23*		2	40	3	-18*
3	73	32	46		4	83	27	26		3	66	44	4		4	107	20	-15		H,K= 15, 9			
4	0	3	-57*		5	63	3	46*		4	215	8	4		5	55	3	55*		0	143	14	4
H,K= 12, 8					H,K= 13, 5					5	0	3	-6*		H,K= 15, 1					1	63	3	8*

STRUCTURE FACTORS CONTINUED FOR

Column 1:
```
 L FOB SG  DEL
 2 181  9   -1
 H,K= 15, 11
 0  83 31  -43
 H,K= 16,  0
 0 278  7  -16
 1  43  3   43*
 2 225  7    0
 3  57  3   57*
 4 238  8  -11
 5  78  3   78*
 6 126 20    2
 H,K= 16,  1
 0   0  3    0*
 1  98 20    1
 2   5  3  -14*
 3  55 37   44
 4  37  3  -24*
 5  67  3   50*
 H,K= 16,  2
 0 315  6    3
 1  32  3  -22*
 2 196  8   -6
 3  30  3    3*
 4 243 22   19
 5  50  3   22*
 6 127 17    7
 H,K= 16,  3
 0  33  3   33*
 1  37  3   25*
 2  30  3   23*
 3  48  3   -3*
 4  58  3  -13*
 5   5  3  -38*
 H,K= 16,  4
 0 315  7   11
 1  56  3   15*
 2 172 11    2
 3  81 29   38
 4 212 16   -9
 5  37  3   10*
 6 112 22   -2
 H,K= 16,  5
 0   8  3    8*
 1  43  3   31*
 2  57  3   20*
 3  99 39   70
 4  37  3   -7*
 H,K= 16,  6
 0  96 19   -3
 1  45  3  -28*
 2 280 17   -1
 3  18  3   -1*
 4 117 21   12
 5  39  3   -5*
 H,K= 16,  7
 0  58  3   58*
 1  20  3   -5*
```

Column 2:
```
 L FOB SG  DEL
 2 109 50   11
 3  14  3    2*
 H,K= 16,  8
 0 147 18  -12
 1   0  3  -64*
 2 168 17   -4
 4  77 25  -29
 H,K= 16, 10
 0 132 16    6
 2 110 17  -18
 H,K= 17,  0
 0   0  3    0*
 1  57 35   57*
 2   0  3  -41*
 3   0  3    0*
 4  44  3   -4*
 5  36  3   36*
 H,K= 17,  1
 0 220  9  -10
 1  50  3  -20*
 2 235  9   -0
 3  73 23   14
 4 223 12    5
 5  38  3   27*
 6 135 16    1
 H,K= 17,  2
 0  29  3   29*
 1  80 23   71
 2  59 35   17
 3  51  3    2*
 4  32  3  -19*
 H,K= 17,  3
 0 291  8    8
 1  24  3    7*
 2 184 11    3
 3  44  3   -9*
 4 227 13   11
 5   0  3  -16*
 6 104 28  -10
 H,K= 17,  4
 0  26  3   26*
 1  59 33   45
 2  52  3    4*
 3   0  3  -13*
 4  22  3  -20*
 5  76  3   70*
 H,K= 17,  5
 0 258 11    5
 1  39  3  -59*
 2 183 11   -6
 3  76  3   53*
 4 155 14    4
 5   0  3  -25*
 H,K= 17,  6
 0  49  3   49*
 1  67  3  -18*
 2  49  3   44*
```

Column 3:
```
 L FOB SG  DEL
 3  57  3   12*
 4  56  3   20*
 5  66  3   24*
 H,K= 17,  7
 0 124 19    8
 1  27  3   -8*
 2 237 10    2
 3   0  3  -80*
 4 111 15   28
 H,K= 17,  8
 0  51  3   51*
 1   0  3  -18*
 2   0  3  -11*
 3   0  3  -12*
 H,K= 17,  9
 0 114 24  -20
 1   0  3  -50*
 2 159 12   16
 3   0  3  -61*
 H,K= 17, 10
 0   0  3    0*
 1  78  3   77*
 H,K= 18,  0
 0 295  7    6
 1  56  3   56*
 2 209 10  -10
 3  52  3   52*
 4 148 60  -34
 5   0  3    0*
 6 125 21   -7
 H,K= 18,  1
 0   0  3    0*
 1  56  3  -35*
 2  31  3  -34*
 3  25  3  -20*
 4   0  3  -27*
 5  20  3    4*
 6   0  3  -43*
 H,K= 18,  2
 0 306  6    5
 1  76 20   28
 2 207 11   -1
 3  81 26   61
 4  89 26  -42
 5  44  3   22*
 H,K= 18,  3
 0  43  3   43*
 1  19  3    5*
 2  40  3   18*
 3  94 53   13
 4  85 29    8
 5 123 30   47
 H,K= 18,  4
 0 272  8  -12
 1  61 36   23
 2 132 17    0
 3   0  3  -40*
```

Column 4:
```
 L FOB SG  DEL
 4 252 10    3
 5  36  3   -2*
 H,K= 18,  5
 0  67  3   67*
 1  49  3   45*
 2   0  3  -45*
 3  44  3   35*
 4  61  3   32*
 5  59  3   30*
 H,K= 18,  6
 0  85  3   -8*
 1  30  3   -0*
 2 221 12  -11
 3  39  3  -39*
 4 135 17   -5
 H,K= 18,  7
 0  12  3   12*
 1   0  3  -25*
 2  48  3   39*
 3   0  3  -21*
 4  86 41   42
 H,K= 18,  8
 0  84 29  -33
 1  29  3  -49*
 2 199 12   16
 3  36  3    4*
 H,K= 18,  9
 0 100 31  100*
 1   0  3   -9*
 2   0  3  -22*
 H,K= 19,  0
 0   0  3    0*
 1  25  3   25*
 2  22  3  -67*
 3   0  3    0*
 4  37  3   23*
 5   0  3    0*
 H,K= 19,  1
 0 152 16   -4
 1   0  3  -23*
 2 296  9    2
 3  55  3   -5*
 4  64  3  -32*
 5  57  3   55*
 H,K= 19,  2
 0  26  3   26*
 1  68 31  -12
 2  27  3  -30*
 3  28  3  -23*
 4  67  3   34*
 5   0  3  -28*
 H,K= 19,  3
 0 258 11   -5
 1  50  3   20*
 2 159 12   -4
 3  23  3   -9*
 4 193 12   -2
```

Column 5:
```
 L FOB SG  DEL
 5  75 38   42
 H,K= 19,  4
 0  35  3   35*
 1  95 23   36
 2  46  3   46*
 3  67  3   45*
 4  26  3  -29*
 5   0  3  -56*
 H,K= 19,  5
 0 168 20  -16
 1  27  3  -41*
 2 172 22    8
 3  44  3   18*
 4 213 13   11
 H,K= 19,  6
 0  53  3   53*
 1  54  3    2*
 2  71 40   65
 3   0  3   -4*
 4  92 37   47
 H,K= 19,  7
 0 168 19  -13
 1  26  3  -21*
 2 161 17   -5
 3  88 66   23
 H,K= 19,  8
 0  78  3   78*
 1   0  3  -28*
 2  50  3   39*
 3   0  3   -6*
 H,K= 19,  9
 0 121 37   -6
 1   0  3  -61*
 H,K= 20,  0
 0 105 17  -18
 1  68 25   68*
 2 318 10   15
 3  54  3   54*
 4 101 41   11
 5  74  3   74*
 H,K= 20,  1
 0   0  3    0*
 1  46  3   23*
 2   0  3   -8*
 3  27  3   21*
 4   0  3  -47*
 5   0  3   -4*
 H,K= 20,  2
 0 153 12    0
 1  66 32   61
 2 223 16   -6
 3  71  3   24*
 4 166 12    2
 5  64  3   40*
 H,K= 20,  3
 0   0  3    0*
 1  66 42    5
```

STRUCTURE FACTORS CONTINUED FOR

L	FOB	SG	DEL
2	60	3	30*
3	23	3	3*
4	44	3	7*
5	97	36	80
H,K= 20, 4			
0	310	15	-17
1	16	3	-0*
2	110	39	10
3	35	3	-38*
4	199	18	18
H,K= 20, 5			
0	0	3	0*
1	58	3	-11*
2	53	3	6*
3	79	50	61
4	66	3	32*
H,K= 20, 6			
0	115	22	-30
1	51	3	13*
2	166	11	-10
3	51	3	-20*
4	144	18	7
H,K= 20, 7			
0	73	3	73*
1	62	3	59*
2	0	3	-7*
3	90	50	88
H,K= 20, 8			
0	125	27	-19
1	73	53	-6
2	130	29	-5
H,K= 21, 0			
0	0	3	0*
1	55	3	55*
2	0	3	-41*
3	0	3	0*
4	0	3	-58*
5	104	3	104*
H,K= 21, 1			
0	116	19	21
1	0	3	-5*
2	279	9	4
3	32	3	2*
4	88	40	-14
5	26	3	10*
H,K= 21, 2			
0	58	3	58*
1	50	3	30*
2	27	3	-25*
3	32	3	26*
4	52	3	33*
H,K= 21, 3			
0	171	15	-22
1	41	3	-22*
2	183	11	8
3	64	3	52*
4	171	12	5
H,K= 21, 4			
0	49	3	49*
1	92	23	41
2	12	3	1*
3	54	3	46*
4	19	3	-38*
H,K= 21, 5			
0	228	10	-1
1	0	3	-50*
2	107	28	-11
3	0	3	-44*
4	153	13	-7
H,K= 21, 6			
0	0	3	0*
1	40	3	34*
2	0	3	-58*
3	76	3	74*
H,K= 21, 7			
0	148	17	-17
1	0	3	-47*
2	132	18	-10
H,K= 21, 8			
0	95	34	95*
1	0	3	-6*
H,K= 22, 0			
0	152	19	17
1	37	3	37*
2	227	19	-18
3	0	3	0*
4	94	33	8
H,K= 22, 1			
0	12	3	12*
1	35	3	6*
2	0	3	-19*
3	60	3	38*
4	41	3	12*
H,K= 22, 2			
0	90	40	10
1	68	3	31*
2	261	9	17
3	17	3	13*
4	118	26	-15
H,K= 22, 3			
0	0	3	0*
1	71	3	41*
2	0	3	-34*
3	0	3	-7*
4	0	3	-31*
H,K= 22, 4			
0	233	15	19
1	29	3	5*
2	124	23	-8
3	63	3	6*
H,K= 22, 5			
0	0	3	0*
1	41	3	-33*
2	0	3	-6*
3	0	3	-6*
H,K= 22, 6			
0	199	15	16
1	0	3	-66*
2	130	18	-3
H,K= 22, 7			
0	0	3	0*
1	96	34	60
H,K= 23, 0			
0	0	3	0*
1	23	3	23*
2	15	3	-28*
3	0	3	0*
4	0	3	-22*
H,K= 23, 1			
0	173	18	9
1	20	3	5*
2	186	10	-5
3	37	3	30*
4	101	21	-4
H,K= 23, 2			
0	0	3	0*
1	57	3	41*
2	89	39	83
3	0	3	-4*
H,K= 23, 3			
0	187	11	11
1	0	3	-54*
2	161	12	2
3	85	81	79
H,K= 23, 4			
0	0	3	0*
1	96	31	57
2	53	3	29*
3	17	3	13*
H,K= 23, 5			
0	125	26	-20
1	72	52	16
2	152	12	8
H,K= 23, 6			
0	82	44	82*
1	100	32	68
H,K= 24, 0			
0	106	44	-4
1	0	3	0*
2	209	11	2
3	41	3	41*
H,K= 24, 1			
0	0	3	0*
1	0	3	-5*
2	0	3	-55*
3	0	3	-15*
H,K= 24, 2			
0	210	11	22
1	0	3	-28*
2	153	13	1
3	0	3	-9*
H,K= 24, 3			
0	48	3	48*
1	22	3	1*
2	40	3	7*
3	0	3	-15*
H,K= 24, 4			
0	108	24	18
1	0	3	-22*
2	169	17	-8
H,K= 24, 5			
0	0	3	0*
1	0	3	-43*
H,K= 25, 0			
0	0	3	0*
1	0	3	0*
2	0	3	-49*
H,K= 25, 1			
0	150	18	-8
1	89	37	82
2	158	15	11
H,K= 25, 2			
0	0	3	0*
1	88	37	83
2	0	3	-17*
H,K= 25, 3			
0	175	18	-12
1	39	3	17*
2	146	14	15
H,K= 25, 4			
0	0	3	0*
1	70	3	60*
H,K= 26, 0			
0	137	18	3
1	71	3	71*
H,K= 26, 1			
0	0	3	0*
1	0	3	-5*
H,K= 26, 2			
0	141	17	-12
1	61	3	37*
H,K= 26, 3			
0	0	3	0*