Submitted to Inorganic Chemistry

UCRL-18900
Preprint

cy. 2

RECEIVED
LAWRENCE
RADIATION LABORATORY
MAY 1969
LIBRARY AND
DOCUMENTS SECTION

THE CRYSTAL STRUCTURE OF $CsCr[B_9C_2H_9(CH_3)_2]_2 \cdot H_2O$, A HYDRATE OF A CHROMIUM METALLOCARBORANE SALT

David St. Clair, Allan Zalkin and David H. Templeton

April 1969

AEC Contract No. W-7405-eng-48

TWO-WEEK LOAN COPY

This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 5545

UCRL-18900
cy. 2

LAWRENCE RADIATION LABORATORY
UNIVERSITY of CALIFORNIA BERKELEY

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

Submitted to Inorganic Chemistry

UCRL-18900
Preprint

UNIVERSITY OF CALIFORNIA

Lawrence Radiation Laboratory
Berkeley, California 94720

AEC Contract No. W-7405-eng-48

CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY, UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA 94720

The Crystal Structure of $CsCr[B_9C_2H_9(CH_3)_2]_2 \cdot H_2O$, a Hydrate of a Chromium Metallocarborane Salt[1]

By David St. Clair, Allan Zalkin and David H. Templeton

---

(1) Work done under the auspices of the U. S. Atomic Energy Commission.

Abstract

The crystal structure of the monohydrate of the cesium salt of $Cr[B_9C_2H_9(CH_3)_2]_2^-$, 3,3'-commo-bis[nonahydro-1,2-dimethyl-1,2-dicarba-3-chroma-closo-dodecaborate](1-), has been determined from 1957 independent X-ray data obtained from a single-crystal using counter methods and the θ-2θ scan technique. The crystals are tetragonal with a = 9.938 ± 0.004 Å and c = 22.739 ± 0.007 Å, and space group $P4_12_12$. There are four molecules of $CsCr[B_9C_2H_9(CH_3)_2]_2 \cdot H_2O$ in the unit cell and the density is calculated to be 1.55 g/cc. The model was refined by least squares to a conventional R value of 0.047. The metallocarborane anion consists of two icosahedra linked by a chromium atom which simultaneously occupies an apex position in each cage. Each icosahedron is composed of the chromium atom, nine boron atoms each having a hydrogen pointing out radially from the cage, and two carbon atoms each having a methyl group attached. The carbon atoms in the cage are bonded to each other and to the chromium atom. The pair of methyl groups in one cage are as far as possible from the pair of methyl groups in the other cage in a single molecule. The anion has point symmetry 2/m (C_{2h}) to within ±0.02 Å.

Introduction

Ruhle and Hawthorne have recently reported[2] the preparation of

(2) H. W. Ruhle and M. F. Hawthorne, Inorg. Chem., 7, 2279(1968).

$Cr[B_9C_2H_9(CH_3)_2]_2^-$, another in the series of transition metal derivitives of the dicarbollide ion, $(B_9C_2H_{11})^{-2}$. In this anion, 3,3'-commo-bis [nonahydro-1,2-dimethyl-1,2-dicarba-3-chroma-closo-dodecaborate](1-),[3]

(3) This name comes from the ACS-approved nomenclature rules given in Inorg. Chem., 7, 1945(1968). The name given by Ruhle and Hawthorne in reference (2) is bis[π-(3)-1,2-dimethyl-1,2-dicarbollyl]chromium(III).

the chromium is in a formal +3 valence state and is bonded to two icosahedral dicarbollide ions in which the hydrogen atoms on the carbons of the $(B_9C_2H_{11})^{-2}$ have been replaced by methyl groups. It was expected that the complex would have a sandwich-type structure in which the chromium would simultaneously occupy an apex in each of two icosahedra.

The crystal structure determination of the monohydrate of the cesium salt of the metallocarborane anion, $CsCr[B_9C_2H_9(CH_3)_2]_2 \cdot H_2O$, is reported here. The sandwich nature of the anion structure, shown in Figure 1, is confirmed and it is shown that the cages are staggered across the chromium with the carbon atoms of one cage rotated as far as possible from those in the other.

Experimental

The dark red crystals of $CsCr[B_9C_2H_9(CH_3)_2]_2 \cdot H_2O$ were very well formed in the shape of square prisms elongated along the c axis. The largest faces were the rectangular shpaed (110) faces. The crystal used in this work was cut perpendicular to the c axis to dimensions 0.10 x 0.10 x 0.09 mm and was glued to the tip of a glass fiber in the open air, with its c axis as the rotation axis.

Oscillation, Weissenberg and precession photographs were taken using copper radiation (Cu $K\alpha_1$; λ = 1.5405 Å) and from them approximate cell dimensions and space group possibilities were determined. The crystal was then transfered to a manually operated General Electric XRD-5 diffractometer where, using copper radiation, space group absences were checked and accurate cell dimensions were obtained from measurements on the $h00$, $0k0$ and 00ℓ axis row reflections. An automated General Electric XRD-5 diffractometer equipped with a quarter circle, Eulerian cradle goniostat was used for collecting intensity data. Machine instructions were read in on IBM cards and the output data were punched automatically into cards. Copper $K\alpha$ radiation was used after the $K\beta$ radiation was effectively removed by a 0.003 inch thick nickel filter placed in front of the receiving slit. The scattered radiation was detected by a scintillation counter equipped with a pulse height discriminator. The integrated intensity of each reflection was measured by scanning in 2θ across the peak beginning 0.65° below the 2θ value at which the $K\alpha_1$ was diffracted, scanning at a rate of 1°/min until the 2θ reached 0.65° above the 2θ value at which the $K\alpha_2$ was diffracted. Two ten second background counts were taken with the apparatus stationary, one 0.5° below the 2θ value at which scanning was

begun and one 0.5° above the 2θ value at which the scan stopped. Before every reflection, the φ angle was first set 5.0° below the desired value so that the final phi value was approached from the same direction for all reflections thus minimizing the error due to the backlash in the φ gears. In order to check for possible systematic variations in the intensity data during data taking, four standard reflections were rechecked periodically. However, no significant systematic variation was observed.

All calculations were done using a CDC 6600 computer. The raw data were converted into intensities, $\underline{I}$, and standard deviations, $\sigma(\underline{I})$, according to the equations:

$$\underline{I} = \underline{C} - \frac{\underline{t_c}}{2\underline{t_b}}(\underline{B}_1 + \underline{B}_2) \quad \text{and} \quad \sigma^2(\underline{I}) = \underline{C} + \frac{\underline{t_c}^2}{4\underline{t_b}^2}(\underline{B}_1 + \underline{B}_2)$$

where $\underline{C}$ is the total count accumulated while scanning across a reflection for a time, $\underline{t_c}$, and $\underline{B}_1$ and $\underline{B}_2$ are the background counts obtained in time, $\underline{t_b}$. When a reflection was measured more than once, the intensities were averaged and its standard deviation was set equal to the greater of $\frac{1}{\underline{n}}(\Sigma\sigma_{\underline{i}}^2)^{\frac{1}{2}}$ or $\frac{1}{\underline{n}-1}(\Sigma\Delta_{\underline{i}}^2)^{\frac{1}{2}}$, where $\sigma_{\underline{i}}$ and $\Delta_{\underline{i}}$ are the standard deviation of the $\underline{i}^{th}$ measurement and the deviation of the $\underline{i}^{th}$ measurement from the average, respectively, and $\underline{n}$ is the number of measurements of the $\underline{i}^{th}$ reflection. To reduce the weight given to the large intensity reflections, an additional term, $(0.08\underline{I})^2$, was included in the calculation of $\sigma^2(\underline{F}^2)$, given by $\sigma^2(\underline{F}^2) = (\underline{Lp})^2[\sigma^2(\underline{I}) + (0.08\underline{I})^2]$, where $\underline{Lp}$ is the Lorentz-polarization correction.

The intensities of all the reflections of the indices $\underline{hk\ell}$ and $\underline{kh\ell}$, which in point group 422 are equal except for anomalous dispersion effects, were collected out to a limit of $2\theta = 147^{\circ}$ ($\sin\theta/\lambda = 0.622$).

This gave a total of 2233 data, excluding the systematic space group absences, of which 274 had intensities less than their estimated standard deviations.

The absorption coefficient for copper radiation is $\mu = 167\ cm^{-1}$. An absorption correction was attempted taking twelve slices through the crystal with a 9 x 9 grid using a version of Hamilton's GONO9 program. The absorption correction factors varied from 2.8 to 5.2. When this correction was applied there was no improvement in either the structure or the reliability indices. Therefore, we prefer to use and report the data uncorrected for absorption.

Values of $\underline{F}_o^2$ and $\sigma(\underline{F}^2)$ were calculated from $\underline{I}$ and $\sigma(\underline{I})$. Standard deviations, $\sigma(\underline{F})$, of the observed structure factors were calculated as follows: if $\underline{I} \leq \sigma(\underline{I})$, then $\sigma(\underline{F}) = [\sigma(\underline{F}^2)]^{\frac{1}{2}}$, and if $\underline{I} > \sigma(\underline{I})$, then $\sigma(\underline{F}) = \underline{F} - [\underline{F}^2 - \sigma(\underline{F}^2)]^{\frac{1}{2}}$. Refinements were done using our unpublished version of a full-matrix least-squares program which minimizes $\Sigma\underline{w}(|\underline{F}_o| - |\underline{F}_c|)^2/\Sigma\underline{w}|\underline{F}_o|^2$, where $|\underline{F}_o|$ and $|\underline{F}_c|$ are the magnitudes of the observed and calculated structure factors, respectively, and where $\underline{w} = [\sigma(\underline{F})]^{-2}$. Temperature factors for all atoms except hydrogens, which were given isotropic temperature factors, were of the form $\exp(-\underline{h}^2\beta_{11}-\underline{k}^2\beta_{22}-\underline{\ell}^2\beta_{33} -2\underline{hk}\beta_{12}-2\underline{h\ell}\beta_{13}-2\underline{k\ell}\beta_{23})$. All atoms were considered to be in their neutral valence state. The scattering factors of Cromer and Waber[4]

(4) D. T. Cromer and J. T. Waber, Acta Cryst., **18**, 104(1965).

were used for all atoms except hydrogen whose scattering factors are those of Stewart, Davidson and Simpson[5]. Cromer's corrections[6]

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

(6) D. T. Cromer, Acta Cryst., 18, 17(1965).

---

for anomalous dispersion applied to the scattering factors of the cesium and chromium atoms are $\Delta f' = -1.40$ and $\Delta f' = -0.13$, respectively for the real part and $\Delta f'' = +7.75$ and $\Delta f'' = +2.59$, respectively for the imaginary part. The stereoscopic pair drawings were prepared using Johnson's ORTEP program[7].

---

(7) C. K. Johnson, "ORTEP, A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations," Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1965.

Crystal Data

The unit cell is tetragonal with cell dimensions $\underline{a}$ = 9.938 ± 0.004 Å and $\underline{c}$ = 22.739 ± 0.007 Å. With four formula units, $CsCr[B_9C_2H_9(CH_3)_2]_2 \cdot H_2O$, in the unit cell, the density is calculated to be 1.55 g/cc.

Weissenberg photographs, confirmed by goniostat measurements, showed the systematic absences, $00\underline{\ell}$, $\underline{\ell} = 4\underline{n}$ and $\underline{h}00$, $\underline{h} = 2\underline{n}$. These rules are characteristic of two space groups, $P4_12_12$ and $P4_32_12$. These two space groups are enantiomorphic and are equivalent in intensity calculations except for the effect of anomalous dispersion. The correct space group for this particular crystal was found to be $P4_12_12$. The general symmetry equivalent positions are $\underline{x},\underline{y},\underline{z}$; $\bar{\underline{x}},\bar{\underline{y}},\frac{1}{2}+\underline{z}$; $\frac{1}{2}-\underline{y},\frac{1}{2}+\underline{x},\frac{1}{4}+\underline{z}$; $\frac{1}{2}+\underline{y},\frac{1}{2}-\underline{x},\frac{3}{4}+\underline{z}$; $\underline{y},\underline{x},\bar{\underline{z}}$; $\bar{\underline{y}},\bar{\underline{x}},\frac{1}{2}-\underline{z}$; $\frac{1}{2}-\underline{x},\frac{1}{2}+\underline{y},\frac{1}{4}-\underline{z}$; $\frac{1}{2}+\underline{x},\frac{1}{2}-\underline{y},\frac{3}{4}-\underline{z}$.

Determination of the Structure

The structure was determined under the arbitrary assumption that $P4_12_12$ was the correct space group. The enantiomorphic space group, $P4_32_12$, was tested by an attempt to refine in $P4_32_12$ the enantiomorph of the structure found in $P4_12_12$. The best agreement with the observed data was obtained in space group $P4_12_12$.

There are only four molecules of $CsCr[B_9C_2H_9(CH_3)_2]_2^-\cdot H_2O$ in the unit cell of this eight-fold general equivalent position space group. Therefore, the cesium, chromium and oxygen atoms must lie on special positions and the remainder of the asymmetric unit is one complete $B_9C_2H_9(CH_3)_2$ group.

The $\underline{x}$ and $\underline{y}$ coordinates of the cesium and chromium, whose $\underline{z}$ coordinates are fixed by symmetry, were readily found from a three dimensional Patterson function. Several cycles of least squares refinement of their coordinates and isotropic thermal parameters gave a conventional $\underline{R}$ value of 0.24, where $\underline{R} = \Sigma(|\underline{F}_o| - |\underline{F}_c|)/\Sigma|\underline{F}_o|$. These atoms were used to phase a three dimensional Fourier on which appeared all the atoms in the cage except the hydrogens. It was expected that the methyl groups would be attached to the carbon atoms in the cage so the methyl carbons and the atoms to which they were attached were given the scattering power of carbon while the other atoms were given boron atomic scattering power. Several cycles of full matrix least squares refinement of the coordinates and isotropic thermal parameters of these fifteen atoms gave $\underline{R} = 0.14$ for all 2233 data. A bond distance calculation indicated that the correct choice had been made for the positions of the carbon atoms in the cage. Giving the cesium and chromium atoms anisotropic thermal parameters and including the anomalous dispersion corrections,

further least squares refinement using only the data with $\underline{I} \geq \sigma(\underline{I})$ gave $\underline{R} = 0.063$ for 1959 data.

To test the enantiomorphic structure in space group $P4_32_12$, the coordinates of all atoms were inverted through the origin ($\underline{x},\underline{y},\underline{z}$ to $\bar{\underline{x}},\bar{\underline{y}},\bar{\underline{z}}$). A refinement in $P4_32_12$ gave $\underline{R} = 0.095$ compared to 0.063 for refinement in $P4_12_12$ under identical conditions. All further refinements were done in space group $P4_12_12$. This choice of configuration is confirmed by excellent agreement in magnitude and sign of observed and calculated values of $|\underline{F}_{hk\ell}| - |\underline{F}_{kh\ell}|$ after the final refinement.

Another series of refinement was done in which all atoms were given anisotropic temperature factors. A three dimensional difference Fourier phased by these atoms was prepared. The largest peak had a magnitude of 4 electrons/Å^3 and corresponds to a water of crystallization. Nine of the next largest twelve peaks corresponded to hydrogens attached to the boron atoms. Indications for the presence of the methyl group hydrogens were found but they were not nearly as clear as the peaks of the hydrogens on the borons. The hydrogen atoms of the water molecule did not appear on this difference Fourier.

Anisotropic temperature factors were applied to all the non-hydrogen atoms. All of the hydrogen atoms except those of the water molecule were included with two isotropic temperature factors, one for the hydrogens attached to the boron cage and another for the methyl group hydrogens. The refinement gave $\underline{R} = 0.052$ for the 1959 data having $\underline{I} \geq \sigma(\underline{I})$. It was noticed that the two maximum intensity reflections were both observed much too low, most likely because their counting rates exceeded the capacity of our scintillation counter, so they were

deleted from the calculations. Further refinement then gave $R = 0.047$ for 1957 data and $R = 0.062$ for all 2233 data. The weighted R value, $R_2 = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{\frac{1}{2}}$, was 0.058. The standard deviation of observation of unit weight, given by $[\Sigma w(|F_o| - |F_c|)^2/(n - p)]^{\frac{1}{2}}$, where n is the number of data and p is the number of parameters, was 1.1. Root-mean-square averages of $[w^{\frac{1}{2}}(|F_o| - |F_c|)]$ taken over small intensity intervals approximated the value 1.1 over the entire intensity range. On the final cycle of refinement, no positional parameter shifted by more than 10 % of its estimated standard deviation. The largest shifts were about 20 % of sigma for the thermal parameters on the hydrogen atoms.

Results and Discussion

Final atomic coordinates for all atoms except hydrogens are given in Table I and their anisotropic thermal parameters are given in Table II. Atomic coordinates and isotropic thermal parameters for the hydrogen atoms are given in Table III. Observed and calculated structure factors are listed in Table IV.

The structure of the $Cr[B_9C_2H_9(CH_3)_2]_2^-$ anion skeleton is shown without hydrogen atoms in Figure 1. The complete anion including hydrogen atoms is shown by the stereoscopic pair drawing in Figure 2. The anion consists of two complete icosahedra linked through the chromium atom which simultaneously occupies an apex position in each cage. Each of the carbon atoms in the cage has a methyl group attached and each boron has a hydrogen pointing out radially from the cage. The anion has an approximate mirror plane passing through atoms Cr, B(6), B(8) and B(10) in each cage. The only symmetry operation required by the space group relating the single cage in the asymmetric unit to its equivalent cage opposite the chromium atom is a 2-fold rotation about an axis passing through atom Cr and normal to the approximate mirror plane. Thus, point symmetry 2 is required for the anion by the space group but the anion is observed to have C_{2h} (2/m) point symmetry to within ±0.02 Å.

A complete list of interatomic bond distances is given in Table V. Average bond angles involving the atoms in the skeleton of the icosahedron are given in Table VI. Two estimates of the error in an average of two or more values were calculated. One estimate was based on the root-mean-scatter of individual values, α_i, around the average, $\bar{\alpha}$, of $\underline{n}$ equivalent angles, given by $[\Sigma(\alpha_i - \bar{\alpha})^2]^{\frac{1}{2}}/(\underline{n} - 1)$. The other estimate

Table I

Positional Parameters for all Atoms except Hydrogen[a,b]

Atom	x	y	z
Cs	0.13791(5)	0.13791(5)	0.00000(0)[c]
Cr	0.52992(9)	0.52992(9)	0.00000(0)[c]
C(1)	0.3266(6)	0.6165(6)	-0.0239(2)
C(2)	0.4413(7)	0.7344(7)	-0.0222(3)
C(3)	0.2776(8)	0.5576(9)	-0.0819(3)
C(4)	0.5068(10)	0.7858(8)	-0.0783(3)
B(4)	0.3231(7)	0.5302(8)	0.0410(3)
B(5)	0.1987(8)	0.6531(8)	0.0254(3)
B(6)	0.2740(8)	0.7802(8)	-0.0158(3)
B(7)	0.5222(7)	0.7339(7)	0.0430(3)
B(8)	0.4453(8)	0.6056(8)	0.0864(3)
B(9)	0.2741(7)	0.6477(8)	0.0954(3)
B(10)	0.2457(8)	0.8050(8)	0.0595(3)
B(11)	0.3990(8)	0.8567(9)	0.0282(3)
B(12)	0.3989(7)	0.7762(8)	0.0968(3)
O(1)	0.9064(13)	0.9064(13)	0.0000(0)[c]

(a) Standard deviations of the least significant digits estimated by least squares are given in parentheses.

(b) The numbering system used for the anion is shown in Figure 1.

(c) Atoms on special four-fold positions, $z = 0$.

Table II

Thermal Parameters for all Atoms except Hydrogen[a]

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cs[b]	4.14(2)	4.14(2)	2.11(2)	0.70(2)	-0.16(2)	0.16(2)
Cr[b]	2.17(3)	2.17(3)	1.69(5)	0.01(4)	0.18(3)	-0.18(3)
C(1)	2.6(3)	2.6(3)	1.5(2)	0.6(2)	0.0(2)	-0.3(2)
C(2)	3.3(3)	2.1(3)	2.0(2)	-0.2(2)	0.4(2)	0.2(2)
C(3)	3.5(4)	4.2(4)	2.6(3)	0.7(3)	-0.9(2)	-0.9(2)
C(4)	4.8(4)	3.2(3)	2.7(3)	0.7(3)	1.0(3)	0.7(2)
B(4)	2.1(3)	2.4(3)	1.9(3)	0.1(2)	0.5(2)	0.0(2)
B(5)	2.2(3)	2.9(3)	2.3(3)	-0.1(3)	0.0(2)	-0.1(3)
B(6)	3.1(3)	2.2(3)	2.5(3)	0.8(2)	0.4(2)	0.2(2)
B(7)	2.0(3)	2.0(3)	2.2(3)	-0.5(2)	0.3(2)	-0.6(2)
B(8)	2.7(3)	3.5(4)	1.4(2)	0.7(2)	0.2(2)	0.0(2)
B(9)	2.2(3)	2.2(3)	2.4(3)	0.2(2)	0.2(2)	0.2(2)
B(10)	3.0(3)	2.3(3)	2.1(3)	0.4(3)	0.3(2)	-0.4(2)
B(11)	3.0(3)	2.6(3)	2.5(3)	-0.1(3)	0.6(2)	0.0(3)
B(12)	2.3(3)	2.8(3)	2.3(3)	0.0(2)	0.2(2)	-1.0(2)
O(1)[b]	12.5(8)	12.5(8)	15.8(15)	-2.2(11)	-0.7(9)	0.7(9)

(a) The anisotropic thermal parameters, B, in units of A^2 are given by $B_{ij} = 4\beta_{ij}/a_i^* a_j^*$, where a_i^* is the length of the ith reciprocal cell dimension.

(b) Due to symmetry of the special position of this atom, $B_{22} = B_{11}$ and $B_{23} = -B_{13}$.

Table III

Positional Parameters for Hydrogen Atoms[a]

Atom	x	y	z
H(4)	0.283(7)	0.419(7)	0.038(3)
H(5)	0.095(7)	0.615(7)	0.012(3)
H(6)	0.239(7)	0.835(7)	-0.056(3)
H(7)	0.641(7)	0.763(6)	0.047(3)
H(8)	0.497(7)	0.547(7)	0.127(3)
H(9)	0.222(7)	0.619(7)	0.139(3)
H(10)	0.173(6)	0.871(7)	0.078(3)
H(11)	0.428(7)	0.969(7)	0.015(3)
H(12)	0.427(7)	0.838(7)	0.136(3)
H(31)	0.32(1)	0.60(1)	-0.112(6)
H(32)	0.31(1)	0.46(1)	-0.089(5)
H(33)	0.17(1)	0.55(1)	-0.085(5)
H(41)	0.60(1)	0.81(1)	-0.064(5)
H(42)	0.44(1)	0.81(1)	-0.103(6)
H(43)	0.55(1)	0.72(1)	-0.095(6)

(a) The atom number for H(4) through H(12) refers to the position of the boron in Figure 1 to which the hydrogen is bonded. The refined common temperature parameter for these atoms is $\underline{B} = 2.2 \pm 0.6$ Å^2. H(31), H(32), H(33) and H(41), H(42), H(43) are bonded to the methyl carbons, C(3) and C(4), respectively. The refined common thermal parameter for these atoms is $\underline{B} = 8 \pm 1$ Å^2.

Table IV

Observed and Calculated Structure Factors. Each datum given zero weight in the final refinement are indicated with an asterisk.

OBSERVED AND CALCULATED STRUCTURE FACTORS OF CR-GLOB

FCA(0,0,0) = 2040

L	FOB	FCA
H,K= 0, 0		
4	508	740*
8	416	456
12	431	440
16	182	171
20	190	182
24	164	166
28	90	96
H,K= 1, 0		
1	187	177
2	157	147
3	77	76
4	12	10
5	213	198
6	211	202
7	185	179
8	19	8
9	145	139
10	54	50
11	153	143
12	6	3*
13	95	93
14	110	100
15	162	155
16	26	21
17	45	42
18	116	115
19	139	132
20	0	14*
21	69	68
22	16	20*
23	45	50
24	10	4*
25	62	61
26	20	25
27	60	61
28	0	5*
H,K= 1, 1		
0	351	439*
1	246	286
2	76	77
3	222	254
4	275	316
5	151	162
6	211	229
7	154	168
8	251	272
9	177	187
10	118	128
11	135	142
12	222	231
13	114	119
14	159	169
15	160	167
16	157	161
17	50	52
18	153	158
19	140	139
20	143	138
21	77	76
22	107	109
23	50	55
24	113	117
25	73	75
26	105	109
27	42	42
H,K= 1, 2		
1	155	164
2	39	42
3	96	96
4	253	272
5	97	96
6	189	198
7	174	177
8	282	290
9	36	33
10	131	133
11	158	157
12	217	216
13	58	56
14	127	131
15	83	85
16	93	91
17	49	51
18	116	117
19	69	68
20	102	98
21	43	45
22	70	70
23	44	43
24	73	76
25	61	59
26	44	49
27	33	36
H,K= 1, 3		
1	179	185
2	153	160
3	144	143
4	81	80
5	147	150
6	141	146
7	217	218
8	64	62
9	80	83
10	94	95
11	167	164
12	47	42
13	142	137
14	124	119
15	80	81
16	36	38
17	123	123
18	116	115
19	88	86
20	40	35
21	72	78
22	45	47
23	77	78
24	12	25*
25	61	61
26	34	35
27	40	43
H,K= 1, 4		
1	270	278
2	309	328
3	200	205
4	77	80
5	159	164
6	235	241
7	148	145
8	82	84
9	136	137
10	227	227
11	157	152
12	67	65
13	164	159
14	170	165
15	107	104
16	72	73
17	111	107
18	136	128
19	81	74
20	49	49
21	75	74
22	115	117
23	74	76
24	31	33
25	56	60
26	83	84
H,K= 1, 5		
1	10	10*
2	165	170
3	0	8*
4	45	47
5	51	47
6	133	130
7	64	59
8	32	31
9	51	50
10	146	141
11	58	59
12	38	37
13	54	53
14	126	127
15	27	20
16	16	21
17	51	46
18	96	93
19	15	20
20	0	8*
21	30	27
22	67	68
23	11	23*
24	0	3*
25	20	19
H,K= 1, 6		
1	128	127
2	20	19
3	165	165
4	165	159
5	172	172
6	54	53
7	168	167
8	141	136
9	121	119
10	81	77
11	87	80
12	105	100
13	94	93
14	22	27
15	93	90
16	98	91
17	116	112
18	0	10*
19	84	86
20	81	79
21	72	73
22	17	16
23	50	49
24	41	42
H,K= 1, 7		
1	72	68
2	9	11*
3	72	69
4	131	131
5	114	111
6	33	24
7	69	67
8	133	131
9	88	87
10	8	5*
11	57	54
12	108	104
13	87	84
14	21	12
15	84	81
16	88	88
17	80	79
18	0	5*
19	54	52
20	67	68
21	47	48
22	17	20
23	44	45
H,K= 1, 8		
1	61	61
2	71	68
3	68	64
4	62	65
5	79	70
6	56	55
7	89	87
8	45	43
9	71	63
10	48	51
11	49	48
12	33	34
13	49	45
14	55	52
15	0	22*
16	36	33
17	32	30
18	58	55
19	24	24
20	10	23*
21	30	27
H,K= 1, 9		
1	111	108
2	94	88
3	102	99
4	6	12*
5	113	105
6	83	82
7	93	87
8	13	14*
9	75	72
10	82	80
11	62	64
12	34	29
13	51	51
14	73	70
15	51	51
16	0	8*
17	52	48
18	61	60
19	56	55
H,K= 1, 10		
1	42	41
2	0	16*
3	45	42
4	26	26
5	26	28
6	28	21
7	40	38
8	28	30
9	32	34
10	18	20
11	32	31
12	26	31
13	23	23
14	29	27
15	0	3*
16	26	25
H,K= 1, 11		
1	46	42
2	0	5*
3	47	45
4	55	55
5	46	44
6	13	6
7	49	43
8	54	53
9	43	42
10	17	13
11	46	44
12	64	64
H,K= 1, 12		
1	49	47
2	10	6*
3	32	34
4	34	31
5	25	27
H,K= 2, 0		
0	102	91
1	257	253
2	19	17
3	216	215
4	40	42
5	239	233
6	35	35
7	318	306
8	88	91
9	178	174
10	27	22
11	250	239
12	63	65
13	201	193
14	0	5*
15	194	188
16	61	58
17	133	128
18	21	24
19	175	165
20	39	37
21	112	108
22	0	2*
23	105	103
24	69	67
25	108	110
26	10	13*
27	66	67
H,K= 2, 1		
0	415	461
1	172	178
2	67	71
3	108	109
4	262	274
5	111	110
6	186	193
7	158	162
8	270	276
9	9	15*
10	113	118
11	147	148
12	210	208
13	67	64
14	125	129
15	86	90
16	83	82
17	61	60
18	117	115
19	74	73
20	97	93
21	51	50
22	68	68
23	34	44
24	82	80
25	47	52
26	48	47
27	20	24
H,K= 2, 2		
0	13	20
1	60	61
2	339	380
3	67	67
4	155	168
5	65	62
6	380	405
7	41	42
8	172	181
9	0	15*
10	263	273
11	20	22
12	94	98
13	13	11
14	213	216
15	18	9
16	118	125
17	36	35
18	186	185
19	0	7*
20	74	76
21	5	7*
22	126	126
23	0	1*
24	13	13*
25	0	11*
26	98	100
27	25	30
H,K= 2, 3		
1	156	171
2	15	11
3	253	268
4	158	168
5	150	159
6	83	91
7	247	257
8	112	111
9	148	153
10	108	111
11	195	199
12	109	107
13	135	134
14	43	40
15	175	174
16	95	97
17	120	120
18	26	27
19	110	110
20	59	58
21	110	109
22	0	13*
23	56	59
24	58	61
25	80	82
26	0	2*
H,K= 2, 4		
1	137	139
2	44	43
3	170	179
4	90	93
5	149	160
6	40	37
7	159	161
8	123	124
9	123	125
10	38	42
11	134	134
12	83	87
13	110	107
14	39	43
15	109	110
16	43	42
17	116	119
18	16	20
19	83	83
20	54	52
21	73	76
22	30	30
23	56	62
24	37	37
25	56	58
26	21	19
H,K= 2, 5		
1	118	121
2	90	93
3	112	113
4	252	264
5	54	52
6	76	79
7	86	85
8	215	214
9	62	65
10	34	33
11	94	94
12	171	167
13	63	63
14	29	29
15	36	36
16	171	168
17	35	33
18	35	33
19	47	41
20	114	114
21	37	38
22	16	13
23	36	36
24	84	82
25	28	31
H,K= 2, 6		
1	141	140
2	153	153
3	115	116
4	21	18
5	175	174
6	119	114
7	113	110
8	69	69
9	114	111
10	127	122
11	78	76
12	0	6*
13	51	48
14	99	94
15	75	70
16	6	17*
17	62	59
18	78	77
19	57	55
20	8	12*
21	31	30
22	69	67
23	33	33
24	14	20
H,K= 2, 7		
1	100	97
2	18	14
3	87	86
4	64	62
5	100	100
6	53	47
7	101	96
8	83	76
9	116	114
10	66	63
11	128	121
12	74	68
13	108	102
14	50	44
15	87	83
16	45	46
17	79	76
18	37	33
19	75	76
20	38	38
21	59	60
22	17	22
H,K= 2, 8		
1	37	30
2	84	83
3	58	56
4	0	1*
5	48	50
6	88	84
7	85	81
8	0	11*
9	68	63
10	85	84
11	70	64
12	0	4*
13	80	78
14	84	85
15	76	73
16	0	18*
17	65	64
18	61	61
19	64	59
20	12	8
H,K= 2, 9		
1	50	45
2	0	2*
3	75	68
4	92	85
5	51	51
6	0	8*
7	59	57
8	72	72
9	36	35
10	0	11*
11	37	31
12	61	62
13	50	48
14	12	14*
15	32	34
16	42	43
17	49	47
18	19	14
H,K= 2, 10		
1	50	52
2	65	70
3	53	57
4	0	8*
5	44	45
6	78	73
7	56	55
8	0	6*
9	51	49
10	60	59
11	48	51
12	11	23*
13	46	46
14	51	50
15	35	38
H,K= 2, 11		
1	23	30
2	11	20*
3	35	32
4	0	13*
5	51	50
6	35	29
7	48	44
8	17	19
9	35	33
10	31	30
11	47	47
H,K= 2, 12		
1	36	32
2	29	28
3	11	7
4	21	17
H,K= 3, 0		
1	44	41
2	432	443
3	26	23
4	12	16
5	78	70
6	316	317
7	103	90
8	25	24
9	52	47
10	286	287
11	35	32
12	19	19
13	70	66
14	312	300
15	27	26
16	34	34
17	69	68
18	220	210
19	31	35
20	9	3*
21	19	17
22	161	159
23	27	24
24	0	4*
25	0	20*
26	131	134
27	6	6*
H,K= 3, 1		
0	70	70
1	185	186
2	144	151
3	156	155
4	77	76
5	157	156
6	149	152
7	221	221
8	89	84
9	91	93
10	114	112
11	157	157
12	47	43
13	142	136
14	114	112
15	89	89
16	24	29
17	120	122
18	111	110
19	100	96
20	28	38
21	77	72
22	54	51
23	71	72
24	17	26
25	62	64
26	26	29
27	38	45
H,K= 3, 2		
0	138	150
1	142	153
2	10	7
3	247	258
4	166	173
5	143	154
6	77	86
7	243	251
8	90	90
9	173	179
10	100	104
11	200	203
12	97	95
13	142	138
14	34	37
15	162	166
16	103	103
17	113	111
18	30	30
19	104	104
20	60	60
21	117	112
22	7	15*
23	62	66
24	59	59
25	78	80
26	0	12*
H,K= 3, 3		
0	238	263
1	60	61
2	110	124
3	147	149
4	223	233
5	36	32
6	99	103
7	56	48
8	244	257
9	60	61
10	139	141
11	72	70
12	248	250
13	47	50
14	69	63
15	57	58
16	201	196
17	26	31
18	50	45
19	14	18*
20	141	134
21	35	38
22	42	44
23	14	20*
24	99	95
25	19	27
26	27	28
H,K= 3, 4		
1	82	86
2	102	101
3	86	92
4	93	94
5	107	112
7	54	55
8	58	56
9	117	116
10	119	116
11	105	103
12	44	50
13	72	70
14	65	65
15	98	99
16	45	55
17	67	67
18	80	76
19	63	61
20	34	27
21	51	54
22	71	73
23	74	71
24	17	10
25	43	46
H,K= 3, 5		
1	165	167
2	41	42
3	178	185
4	140	143
5	158	158
6	20	13
7	166	167
8	118	117
9	147	145
10	74	74
11	176	172
12	81	76
13	110	108
14	32	37
15	130	121
16	71	68
17	100	95
18	32	34
19	85	82
20	47	44
21	89	84
22	40	44
23	71	71
24	38	39
H,K= 3, 6		
1	38	39
2	85	85
3	55	55
4	127	131
5	39	36
6	96	96
7	40	43
8	130	128
9	12	14*
10	105	100
11	51	49
12	108	103
13	21	17
14	87	81
15	55	54
16	91	86
17	34	30
18	64	65
19	0	21*
20	64	63
21	0	21*
22	50	50
23	35	36
H,K= 3, 7		
1	98	95
2	30	24
3	129	125
4	139	134
5	97	92
6	24	18
7	130	124
8	113	107
9	77	75
10	49	40
11	76	74
12	75	67
13	62	57
14	16	21
15	62	59
16	68	63
17	50	49
18	0	16*
19	34	35
20	58	57
21	11	16*
22	0	3*
H,K= 3, 8		
1	118	109
2	104	97
3	110	106
4	16	15
5	102	100
6	78	71
7	92	88
8	0	12*
9	83	80
10	81	74
11	93	89
12	23	17
13	83	77
14	71	66
15	74	72
16	0	2*
17	44	40
18	53	52
19	43	39
20	19	19
H,K= 3, 9		
1	41	37
2	70	66
3	28	30
4	23	7
5	21	20
6	69	70
7	17	25
8	1	4*
9	23	16
10	71	73
11	16	16
12	25	21
13	22	21
14	55	56
15	19	20
16	13	10
17	24	24
H,K= 3, 10		
1	61	58
2	68	67
3	44	45
4	0	10*
5	68	66
6	83	80
7	46	43
8	0	11*
9	54	52
10	51	52
11	44	42
12	10	13*
13	42	44
14	43	46
H,K= 3, 11		
1	43	38
2	30	28
3	36	31
4	25	22
5	0	4*
6	27	29
7	24	26
8	17	20
9	21	26
10	19	19
H,K= 4, 0		
0	116	112
1	62	64
2	14	17
3	15	15
4	200	195
5	49	48
6	8	8*
7	47	46
8	225	215
9	30	30
10	0	4*
11	6	5*
12	129	124
13	0	13*
14	23	14
15	0	1*
16	90	85
17	10	6*
18	12	7
19	4	2*
20	74	75
21	8	2*
22	5	2*
23	0	12*
24	44	51
25	13	6*
26	0	4*
H,K= 4, 1		
0	18	18
1	282	286
2	319	338
3	187	191
4	73	75
5	165	168
6	234	240
7	148	146
8	85	85
9	144	145
10	228	229
11	144	140
12	51	54
13	151	148
14	196	188
15	91	87
16	59	61
17	97	96
18	142	136
19	73	74
20	47	50
21	79	79
22	113	113
23	69	75
24	28	35
25	62	63
26	81	84
H,K= 4, 2		
0	124	136
1	152	156
2	49	47
3	174	184
4	84	88
5	147	157
6	30	32
7	148	152
8	118	122
9	137	139
10	45	43
11	137	139
12	87	89
13	126	126
14	54	54
15	131	131
16	52	56
17	113	115
18	34	32
19	88	87
20	55	55
21	73	72
22	28	26
23	57	60
24	34	35
25	59	60
26	21	17
H,K= 4, 3		
0	94	98
1	76	81
2	108	109
3	90	96
4	96	95
5	115	119
6	117	116
7	78	78
8	50	50
9	121	123
10	114	112
11	101	100
12	53	54
13	82	77
14	72	73
15	90	90
16	51	52
17	67	67
18	71	70
19	53	59
20	16	19
21	45	47
22	73	75
23	65	65
24	19	18
25	49	47
H,K= 4, 4		
0	294	306
1	109	115
2	134	136
3	178	184
4	309	316
5	38	40
6	120	121
7	117	117
8	222	220
9	42	39
10	63	61
11	118	117
14	61	57
15	137	138
16	173	165
17	66	67
18	0	28*
19	32	31
20	92	90
21	80	79
22	24	15
23	36	39
24	97	97
H,K= 4, 5		
1	57	57
2	36	33
3	122	123
4	0	5*
5	66	65
6	59	58
7	93	85
8	60	59
9	84	86
10	61	63
11	85	85
12	27	26
13	37	38
14	41	38
15	84	80
16	25	23
17	47	48
18	33	30
19	43	45
20	13	11*
21	36	34
22	0	9*
23	30	33
24	21	22
H,K= 4, 6		
1	117	117
2	72	69
3	131	132
4	71	70
5	97	96
6	82	80
7	109	105
8	75	72
9	126	121
10	92	89
11	127	116
12	38	38
13	98	92
14	64	59
15	94	91
16	21	25
17	91	84
18	67	62
19	87	82
20	0	3*
21	80	78
22	54	56
H,K= 4, 7		
1	44	45
2	144	138
3	51	48
4	51	47
5	32	38
6	109	100
7	61	51
8	41	36
9	26	26
10	121	117
11	56	51
12	58	56
13	22	22
14	106	104
15	33	35
16	35	32
17	25	24
18	85	82
19	34	34
20	14	23
21	20	15
H,K= 4, 8		
1	0	9*
2	24	28
3	0	12*
4	93	88
5	25	14
6	10	7*
7	10	18*
8	71	68
9	12	8*
10	24	21
11	24	20
12	57	57
13	9	10*
14	11	16*
15	0	4*
16	63	62
17	3	14*
18	0	10*
19	20	20
H,K= 4, 9		
1	85	84
2	3	11*
3	78	77
4	49	47
5	86	83
6	0	11*
7	70	70
8	54	51
9	80	78
10	16	5
11	73	73
12	53	49
13	59	57
14	0	4*
15	66	63
16	35	33
H,K= 4, 10		
1	11	18*
2	22	27
3	18	21
4	13	21*
5	35	28
6	16	16
7	33	27
8	18	21
9	24	26
10	0	7*
11	16	17
12	33	33
13	14	17
H,K= 4, 11		
1	26	33
2	69	66
3	20	12
4	45	44
5	40	37
6	66	64
7	12	10
8	16	13
H,K= 5, 0		
1	218	222
2	155	156
3	216	218
4	45	49
5	250	249
6	176	174
7	194	186
8	19	20*
9	199	193
10	122	109
11	144	138
12	39	37
13	183	176
14	105	97
15	118	112
16	47	48
17	169	162
18	102	97
19	119	114
20	0	1*
21	103	105
22	83	81
23	105	108
24	13	19
25	88	92
H,K= 5, 1		
0	12	11
1	22	19
2	170	170
3	34	32
4	45	45
5	63	60
6	126	124
7	45	46
8	31	33
9	40	40
10	147	143
11	65	65
12	38	37
13	45	46
14	130	128
15	33	32
16	25	20
17	48	48
18	87	84
19	10	12*
20	8	9*
21	26	29
22	71	72
23	26	26
24	0	3*
25	20	21
H,K= 5, 2		
0	214	229
1	129	131
2	89	95
3	92	98
4	247	260
5	64	66
6	75	78
7	74	76
8	205	210
9	63	66
10	35	36
11	86	90
12	145	142
13	49	48
14	38	43
15	35	29
16	158	159
17	38	33
18	38	33
19	32	40
20	115	116
21	38	36
22	13	11*
23	39	39
24	76	79
25	15	18
H,K= 5, 3		
0	126	128
1	163	167
2	26	28
3	175	183
4	138	144
5	144	143
6	31	36
7	159	159
8	133	136
9	148	149
10	85	86
11	156	155
12	91	89
13	97	96
14	34	37
15	124	118
16	76	73
17	90	85
18	38	36
19	83	83
20	41	42
21	103	97
22	45	43
23	67	69
24	36	36
H,K= 5, 4		
0	12	12*
1	77	77
2	30	30
3	141	139
4	19	10
5	74	76
6	44	44
7	94	91
8	68	66
9	81	79
10	54	54
11	87	88
12	27	27
13	65	62
14	32	40
15	94	88
16	19	21
17	48	51
18	24	28
19	37	41
20	13	11*
21	36	34
22	0	14*
23	22	25
24	26	19
H,K= 5, 5		
0	49	48
1	124	124
2	181	178
3	153	154
4	40	40
5	78	82
6	152	151
7	39	36
8	48	48
9	103	100
10	152	147
11	35	31
12	33	31
13	69	63
14	134	126
15	76	71
16	38	42
17	48	49
18	141	137
19	39	38
20	69	67
21	42	45
22	130	127
23	65	66
H,K= 5, 6		
1	67	64
2	17	16
3	91	92
4	87	86
5	52	51
6	28	24
7	84	80
8	74	69
9	43	43
10	32	29
11	65	63
12	49	48
13	52	45
14	0	24*
15	43	43
16	30	33
17	40	39
18	39	40
19	33	31
20	22	18
21	38	38
H,K= 5, 7		
1	80	74
2	75	75
3	100	99
4	22	25
5	63	63
6	68	66
7	84	80
8	29	19
9	79	77
10	74	69
11	70	68
12	1	16*
13	57	57
14	54	53
15	59	59
16	26	27
17	51	52
18	36	33
19	42	41
20	15	22
H,K= 5, 8		
1	71	67
2	31	30
3	59	58
4	109	101
5	69	64
6	41	43
7	47	45
8	94	89
9	43	43
10	32	35
11	50	50
12	92	85
13	36	36
14	21	24
15	50	47
16	70	67
17	42	40
18	22	21
H,K= 5, 9		
1	0	2*
2	28	31
3	20	6
4	13	16*
5	0	16*
6	46	45
7	0	2*
8	18	23
9	22	22
10	44	44
11	0	7*
12	23	23
13	13	7
14	24	30
15	3	1*
H,K= 5, 10		
1	62	56
2	9	13*
3	56	57
4	77	77
5	58	56
6	0	3*
7	44	46
8	60	55
9	45	40
10	17	19
11	33	35
H,K= 5, 11		
1	34	31
2	50	51
3	0	12*
4	17	21
5	22	27
H,K= 6, 0		
0	89	90
1	122	115
2	46	45
3	52	48
4	140	139
5	51	48
6	5	11*
7	58	53
8	108	108
9	80	76
10	12	12
11	90	89
12	71	68
13	98	96
14	47	51
15	36	31
16	93	93
17	39	33
18	9	22*
19	20	25
20	64	67
21	34	37
22	20	12
23	14	17*
24	58	60
H,K= 6, 1		
0	172	179
1	133	139
2	0	11*
3	162	167
4	153	154
5	170	173
6	61	62
7	183	183
8	139	138
9	135	134
10	84	85
11	92	88
12	113	110
13	93	97
14	32	31
15	95	94
16	109	102
17	110	111
18	9	16*
19	83	84
20	83	85
21	73	75
22	18	15
23	45	48
24	40	42
H,K= 6, 2		
0	0	2*
1	140	142
2	169	172
3	114	111
4	16	19
5	164	169
6	115	115
7	121	121
8	66	65
9	115	111
10	104	108
11	79	77
12	0	6*
13	50	46
14	92	95
15	77	74
16	0	15*
17	54	54
18	61	65
19	63	62
20	0	9*
21	24	27
22	65	66
23	23	23
24	19	23
H,K= 6, 3		
0	96	97
1	52	50
2	89	89
3	57	55
4	121	127
5	41	40
6	101	99
7	40	41
8	127	128
9	30	23
10	110	108
11	57	53
12	103	99
13	26	24
14	95	91
15	62	60
16	92	90
17	23	30
18	61	61
19	14	17
20	70	71
21	18	19
22	54	56
23	43	42
H,K= 6, 4		
0	46	42
1	103	109
2	71	69
3	121	126
4	63	63
5	92	91
6	62	59
7	116	113
8	57	59
9	138	135
10	70	71
11	120	117
13	96	93
14	63	60
15	95	91
16	28	25
17	87	85
18	69	65
19	86	82
20	16	12
21	82	81
22	65	62
H,K= 6, 5		
0	60	56
1	63	66
2	23	25
3	84	85
4	99	96
5	41	41
6	37	36
7	75	76
8	83	81
9	39	32
10	37	32
11	57	57
12	47	48
13	44	39
14	14	22*
15	43	43
16	30	35
17	38	38
18	40	39
19	27	27
20	5	6*
21	43	43
H,K= 6, 6		
0	52	48
1	25	27
2	161	157
3	57	56
4	61	54
5	18	16
6	157	149
7	21	20
8	69	60
9	27	28
10	142	136
11	0	15*
12	54	52
13	0	5*
14	105	105
15	24	20
16	35	28
17	23	17
18	92	94
19	12	5
20	20	21
H,K= 6, 7		
1	62	60
2	34	33
3	91	87
4	43	39
5	58	57
6	31	27
7	70	61
8	41	35
9	86	86
10	44	36
11	65	65
12	20	24
13	53	56
14	28	30
15	53	52
16	0	8*
17	51	52
18	52	47
H,K= 6, 8		
1	58	58
2	15	9
3	62	59
4	30	27
5	62	60
6	23	17
7	27	30
8	35	31
9	71	68
10	8	11*
11	35	36
12	21	21
13	42	42
14	14	12
15	49	50
16	15	20
H,K= 6, 9		
1	11	13*
2	23	18
3	4	9*
4	99	101
5	20	14
6	10	11*
7	20	4
8	80	82
9	7	12*
10	17	19
11	24	23
12	71	69
13	0	5*
H,K= 6, 10		
1	32	30
2	10	7*
3	12	9*
4	13	19
5	34	32
6	0	11*
7	18	19
8	20	20
H,K= 7, 0		
1	83	84
2	133	136
3	59	54
4	0	4*
5	56	52
6	138	135
7	69	66
8	59	59
9	32	37
10	127	128
11	62	62
12	0	2*
13	74	71
14	99	98
15	74	70
16	0	10*
17	64	64
18	67	72
19	66	68
20	19	11*
21	52	54
22	65	70
23	28	29
H,K= 7, 1		
0	104	108
1	81	81
2	0	16*
3	61	61
4	110	112
5	108	110
6	29	29
7	67	67
8	112	113
9	71	75
10	19	5
11	59	60
12	92	94
13	79	77
14	17	11
15	93	88
16	90	93
17	65	70
18	0	9*
19	59	59
20	61	66
21	37	45
22	19	17
23	48	48
H,K= 7, 2		
0	41	41
1	113	118
2	21	19
3	86	87
4	49	48
5	81	84
6	41	41
7	112	111
8	94	91
9	102	103
10	59	56
11	124	120
12	77	76
13	98	97
14	44	44
15	92	88
16	47	48
17	66	70
18	27	30
19	68	69
20	44	47
21	65	68
22	23	20
H,K= 7, 3		
0	113	115
1	94	96
2	28	20
3	121	123
4	123	123
5	96	97
6	16	22
7	124	120
8	119	116
9	70	73
10	40	43
11	80	79
12	71	69
13	47	48
14	15	21
15	62	62
16	71	72
17	43	49
18	0	19*
19	28	30
20	62	62
21	18	19
22	13	2
H,K= 7, 4		
0	77	77
1	55	54
2	143	143
3	50	47
4	38	41
5	33	35
6	105	101
7	54	53
8	36	34
9	24	25
10	117	114
11	43	45
12	51	52
13	32	27
14	107	105
15	33	33
16	30	31
17	25	22
18	69	75
19	29	33
20	26	25
21	11	6
H,K= 7, 5		
0	17	16*
1	77	75
2	62	68
3	114	110
4	14	20*
5	70	67
6	62	61
7	82	81
8	0	20*
9	84	78
10	73	70
11	65	69
12	0	14*
13	61	61
14	50	48
15	59	61
16	20	23
17	46	46
18	33	32
19	40	39
20	25	27
H,K= 7, 6		
0	27	23
1	67	60
2	31	27
3	88	85
4	35	37
5	61	56
6	35	32
7	53	55
8	39	40
9	82	84
10	48	45
11	52	55
12	29	27
13	54	54
14	24	30
15	51	52
16	13	8
17	47	53
18	51	49
H,K= 7, 7		
0	101	102
1	14	3
2	71	69
3	0	11*
4	94	91
5	0	7*
6	52	52
7	17	21
8	65	64
9	0	18*
10	70	67
11	19	10
12	67	69
13	0	14*
14	68	70
15	0	3*
16	49	53
H,K= 7, 8		
1	26	24
2	47	50
3	33	29
4	19	24
5	39	43
6	55	57
7	0	6*
8	46	45
9	50	51
10	38	40
11	0	4*
12	56	61
13	25	25
14	31	32
H,K= 7, 9		
1	45	48
2	21	18
3	41	45
4	0	8*
5	63	62
6	36	27
7	51	50
8	6	8*
9	63	62
10	36	32
H,K= 7, 10		
1	17	21
2	34	33
3	0	8*
H,K= 8, 0		
0	126	126
1	139	142
2	0	16*
3	110	109
4	110	110
5	85	79
6	18	13
7	77	79
8	102	103
9	108	105
10	25	27
11	109	106
12	92	85
13	96	91
14	14	9
15	63	64
16	69	66
17	66	64
18	11	17*
19	57	60
20	60	63
21	67	69
H,K= 8, 1		
0	49	50
1	66	66
2	79	81
3	57	56
4	52	56
5	80	79
6	59	60
7	78	81
8	44	45
9	64	63
10	52	57
11	58	58
12	39	38
13	38	38
14	51	53
15	31	33
16	44	39
17	15	15
18	41	40
19	29	30
20	28	32
21	19	18
H,K= 8, 2		
0	11	10*
1	47	51
2	91	90
3	54	51
4	0	3*
5	50	53
6	102	99
7	83	83
8	0	8*
9	57	59
10	92	88
11	68	67
12	10	6*
13	66	70
14	79	78
15	77	79
16	8	15*
17	61	63
18	55	56
19	55	56
20	14	11
H,K= 8, 3		
0	21	21
1	116	117
2	107	105
3	98	97
4	8	15*
5	93	91
6	76	71
7	97	96
8	5	12*
9	79	76
10	73	68
11	90	90
12	18	17
13	74	75
14	63	65
15	75	74
16	0	1*
17	38	42
18	39	40
19	46	45
20	0	18*
H,K= 8, 4		
0	79	78
1	24	19
2	27	28
3	0	4*
4	82	81
5	7	16*
6	10	7*
7	13	17*
8	73	74
9	0	15*
10	28	20
11	14	17*
12	57	61
13	14	17
14	20	17
15	0	8*
16	61	64
17	0	11*
18	0	11*
19	25	22
H,K= 8, 5		
0	119	110
1	71	69
2	29	34
3	61	59
4	109	105
5	72	67
6	40	44
7	41	43
8	91	90
9	42	42
10	29	35
11	51	50
12	83	81
13	43	40
14	29	32
15	48	51
16	60	62
17	41	43
18	18	24
H,K= 8, 6		
0	32	34
1	53	54
2	0	9*
3	50	58
4	31	26
5	64	63
6	0	18*
7	25	30
8	14	21*
9	71	71
10	20	12
11	38	39
12	6	23*
13	36	42
14	0	12*
15	51	52
16	19	22
H,K= 8, 7		
0	44	43
1	30	27
2	40	47
3	29	34
4	21	25
5	42	42
6	58	64
7	12	9*
8	46	54
9	55	54
10	46	48
11	14	14
12	61	63
13	33	34
14	29	33
H,K= 8, 8		
0	11	24*
1	28	25
2	69	66
3	34	38
4	29	34
5	15	12
6	52	51
7	38	40
8	41	44
9	0	8*
10	92	94
11	36	43
H,K= 8, 9		
1	7	22*
2	20	24
3	29	29
4	0	4*
5	18	20
H,K= 9, 0		
1	10	10*
2	17	14
3	11	10*
4	0	11*
5	18	15
6	12	14*
7	30	25
8	21	15
9	15	19
10	0	11*
11	44	41
12	38	35
13	55	56
14	8	14*
15	55	56
16	13	18*
17	45	45
18	7	8*
19	31	34
H,K= 9, 1		
0	9	5*
1	113	113
2	93	96
3	101	100
4	29	12
5	98	97
6	88	87
7	92	91
8	19	14
9	72	78
10	68	68
11	52	57
12	32	29
13	57	59
14	62	68
15	50	55
16	0	8*
17	39	45
18	52	56
19	49	52
H,K= 9, 2		
0	82	82
1	55	53
2	0	7*
3	55	58
4	69	71
5	58	57
6	0	13*
7	50	53
8	70	73
9	38	36
10	1	12*
11	30	33
12	50	58
13	44	44
14	0	10*
15	40	37
16	46	50
17	41	43
18	13	13
H,K= 9, 3		
0	15	15
1	52	48
2	78	77
3	29	29
4	19	4
5	29	27
6	73	75
7	17	23
8	11	6*
9	11	16*
10	67	71
11	14	10
12	0	19*
13	21	26
14	52	58
15	20	24
16	19	11
17	0	17*
H,K= 9, 4		
0	63	58
1	73	79
2	3	13*
3	62	70
4	60	60
5	70	73
6	18	13
7	68	69
8	59	55
9	75	77
10	0	4*
11	69	73
12	57	53
13	51	55
14	0	2*
15	56	60
16	44	39
H,K= 9, 5		
0	14	21
1	0	3*
2	23	26
3	0	6*
4	7	12*
5	20	15
6	40	43
7	16	2
8	21	17
9	13	19*
10	39	44
11	0	7*
12	16	25
13	13	5
14	21	30
15	0	0*
H,K= 9, 6		
0	102	102
1	4	10*
2	8	15*
3	17	4
4	96	101
5	0	14*
7	0	4*
8	79	83
9	7	10*
10	15	20
11	23	26
12	64	67
13	0	1*
H,K= 9, 7		
0	6	7*
1	44	45
2	18	15
3	38	41
4	10	8*
5	56	57
6	33	32
7	30	36
9	47	52
10	31	36
H,K= 9, 8		
0	0	5*
1	16	22
2	21	23
3	27	32
4	0	2*
5	27	29
H,K= 10, 0		
0	73	78
1	85	88
2	0	5*
3	83	89
4	73	76
5	87	86
6	23	18
7	76	75
8	62	67
9	37	37
10	50	48
11	37	35
12	47	53
13	33	33
14	15	18
15	43	44
16	41	47
H,K= 10, 1		
0	21	28
1	47	45
2	0	15*
3	28	35
4	22	28
5	40	39
6	20	15
7	29	34
8	39	37
9	27	32
10	24	21
11	37	39
12	26	30
13	4	17*
14	25	31
15	8	11*
16	17	22
H,K= 10, 2		
0	6	0*
1	51	52
2	66	72
3	44	50
4	15	8
5	42	51
6	68	71
7	45	51
8	5	6*
9	54	53
10	66	71
11	46	49
12	26	24
13	34	40
14	60	65
15	28	36
H,K= 10, 3		
0	0	9*
1	48	57
2	59	61
3	44	52
4	0	8*
5	52	55
6	69	73
7	46	53
8	0	7*
9	56	57
10	36	39
11	32	34
12	6	12*
13	47	50
14	40	40
H,K= 10, 4		
0	30	34
1	0	12*
2	2	25*
3	20	23
4	22	25
5	24	28
6	20	22
7	27	31
8	18	29
9	22	28
10	9	12*
11	9	14*
12	30	34
13	3	19*
H,K= 10, 5		
0	75	80
1	57	57
2	8	21*
3	56	63
4	64	69
5	58	62
6	0	11*
7	44	45
8	51	57
9	32	36
10	11	19*
11	25	34
H,K= 10, 6		
0	21	22
1	26	28
2	0	11*
3	19	15
4	29	29
5	30	30
6	11	14
7	27	28
8	18	15
H,K= 10, 7		
0	18	25*
1	0	22*
2	24	31
3	0	9*
H,K= 11, 0		
1	39	42
2	32	35
3	34	37
4	15	8
5	30	29
6	40	45
7	15	18
8	10	14*
9	11	12
10	34	36
11	13	21
12	10	9*
H,K= 11, 1		
0	61	64
1	46	45
2	13	5
3	33	42
4	55	57
5	37	41
6	0	6*
7	45	47
8	42	47
9	40	42
10	3	13*
11	37	46
12	42	49
H,K= 11, 2		
0	0	13*
1	28	31
2	21	23
3	36	41
6	27	28
7	40	47
8	20	22
9	31	37
10	25	24
11	26	35
H,K= 11, 3		
0	39	43
1	25	33
2	19	20
3	29	33
4	19	20
5	18	20
6	24	24
7	13	20
8	23	29
9	18	26
10	9	16*
H,K= 11, 4		
0	28	26
1	31	31
2	59	70
3	21	24
4	35	39
5	31	34
6	53	66
7	10	9*
8	0	11*
H,K= 11, 5		
0	3	4*
1	13	30
2	36	45
3	16	24
4	0	16*
5	19	22
H,K= 12, 0		
0	52	55
1	8	3*
2	9	2*
3	11	6*
4	43	44
5	5	9*
6	5	3*
H,K= 12, 1		
0	42	46
1	34	44
2	18	6
3	30	36
4	29	33
5	14	24
H,K= 12, 2		
0	12	11
1	28	30
2	26	28
3	13	17
4	18	27

XBL 692-167

Table V

Interatomic Bond Distances(Å)[a]

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
Cr	C(1)	2.262	B(8)	B(12)	1.77
Cr	C(2)	2.272	B(9)	B(12)	1.78
Cr	B(4)	2.257	B(10)	B(5)	1.76
Cr	B(7)	2.251	B(10)	B(6)	1.75
Cr	B(8)	2.266	B(10)	B(9)	1.79
C(1)	C(3)	1.52	B(10)	B(11)	1.76
C(1)	C(2)	1.64	B(10)	B(12)	1.77
C(1)	B(4)	1.71	B(11)	B(12)	1.76
C(1)	B(5)	1.73	B(4)	H(4)	1.18
C(1)	B(6)	1.72	B(5)	H(5)	1.14
C(2)	C(4)	1.52	B(6)	H(6)	1.12
C(2)	B(6)	1.73	B(7)	H(7)	1.22
C(2)	B(7)	1.69	B(8)	H(8)	1.21
C(2)	B(11)	1.72	B(9)	H(9)	1.15
B(4)	B(5)	1.77	B(10)	H(10)	1.06
B(4)	B(8)	1.76	B(11)	H(11)	1.19
B(4)	B(9)	1.77	B(12)	H(12)	1.11
B(5)	B(6)	1.74	C(3)	H(31)	0.9
B(5)	B(9)	1.76	C(3)	H(32)	1.1
B(6)	B(11)	1.77	C(3)	H(33)	1.1
B(7)	B(8)	1.79	C(4)	H(41)	1.0
B(7)	B(11)	1.76	C(4)	H(42)	0.9
B(7)	B(12)	1.78	C(4)	H(43)	0.9
B(8)	B(9)	1.76			

Table V (continued)

(a) Standard deviations estimated from the least squares estimates of the accuracy of the coordinates are: ±0.007 Å on distances to Cr, ±0.01 Å on distances between carbon and boron, ±0.07 A on hydrogen to boron distances, and ±0.1 Å on the methyl group hydrogen atoms to C(3) and C(4).

calculated from the individual standard deviations, σ_i, calculated from the accuracy of the final atomic positions estimated by least squares, of the n independent angles included in the calculation of the average, was given by $(\Sigma\sigma_i^2)^{\frac{1}{2}}/n$. The larger of these two error estimates is reported in Table VI. In a large majority of cases, the error calculated from the scatter was larger than the error calculated from standard deviations of the individual angles and, on the average, the former was about double the latter.

The molecular packing is shown in Figure 3 by the stereoscopic pair drawing of the unit cell looking approximately down the b axis. The z coordinates of the cesium, labeled CS, the oxygen of the water molecule, labeled O, and the unlabeled chromium sandwiched between the cages, are fixed by symmetry at $z = 0, \frac{1}{4}, \frac{1}{2}, \frac{3}{4}$, and 1, progressing along the direction of the c axis from the base to the top of the unit cell. The positions of the carbon atoms in the cage are distinguished by having the methyl groups attached. All other atoms in the cage are boron atoms. Each of the dumbell-like anions is tilted out of the ab plane until its axis, B(10) in one cage to B(10) in the cage on the opposite side of the chromium, makes an angle of about 20° with the c axis. Since the hydrogen atoms of the water molecule were not determined, the nearest neighbors to the oxygen of the water molecule are given in Table VII along with the nearest neighbors to the cesium. The water molecule has no neighbors suitable for hydrogen bonding, and therefore it is not surprising that it is observed to have large amplitudes of thermal motion, nor that we failed to detect its hydrogen atoms. We cannot exclude the possibility that the large thermal parameters in part reflect incomplete occupancy of the water sites.

Table VI

Average Angles Between Atoms in the Cage[a]

Atom 1	Atom 2	Atom 3	Number	Angle (degrees)
	Angles around triangular faces			
C	Cr	C	1	42.3 ± .2
C	Cr	B	2	44.1 ± .4
B	Cr	B	2	46.2 ± .4
Cr	C	C	2	68.9 ± .5
Cr	B	C	2	68.4 ± .6
Cr	C	B	2	67.6 ± .2
Cr	B	B	4	66.9 ± .3
C	C	B	2	61.7 ± .4
B	C	B	4	61.6 ± .4
C	B	B	8	59.2 ± .3
B	B	B	30	60.0 ± .1
	Angles around pentagonal rings			
C	Cr	B	4	75.1 ± .3
B	Cr	B	1	77.8 ± .3
Cr	C	B	4	126.1 ± .8
Cr	B	B	6	122.9 ± .4
C	C	B	4	110.5 ± .2
B	C	B	2	112.5 ± .4
C	B	B	12	105.9 ± .2
B	B	B	23	107.8 ± .2

(a) Standard deviations are given by the larger of either $(\Sigma\Delta_i^2)^{\frac{1}{2}}/(n - 1)$ or $(\Sigma\sigma_i^2)^{\frac{1}{2}}/n$, where Δ_i is the difference between the ith

Table VI (continued)

measurement and the average of n measurements and where σ_i is the error of the ith value estimated from the accuracy of the atomic coordinates.

Table VII

Distances (Å) to Cesium and Oxygen Atoms

Atom[a]	To O(1)	Atom[a]	To Cs
H(7)	3.18 ± 0.07	H(9)	2.90 ± 0.07
H(10)	3.21 ± 0.07	H(10)	3.20 ± 0.07
H(31)	3.24 ± 0.14	H(8)	3.23 ± 0.07
H(8)	3.26 ± 0.07	H(4)	3.26 ± 0.07
Cs	3.25 ± 0.02		

(a) Each hydrogen atom has a symmetry related atom with the same label at an equal distance from either O(1) or Cs.

Acknowledgements

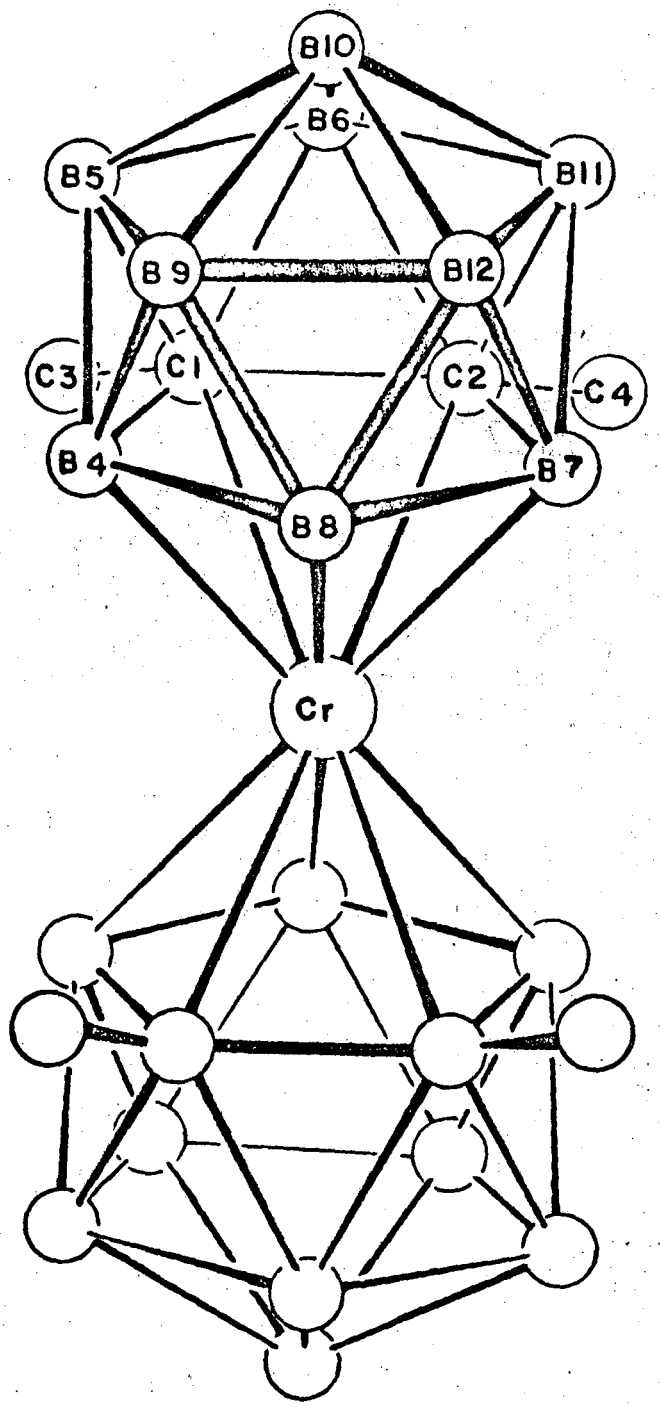
The authors wish to express thanks to Prof. M. Frederick Hawthorne for sending us the crystals used in this work.

Figure Captions

Figure 1: Skeletal drawing of the $Cr[B_9C_2H_9(CH_3)_2]_2^-$ anion (hydrogen atoms not shown) including the numbering system.

Figure 2: Stereoscopic pair drawing of the complete $Cr[B_9C_2H_9(CH_3)_2]_2^-$ anion.

Figure 3: Stereoscopic pair drawing of the unit cell of $CsCr[B_9C_2H_9(CH_3)_2]_2 \cdot H_2O$ (hydrogens not shown) looking down the b axis. The a axis is horizontal and the c axis is vertical in the plane of the paper. The sets of two $Cr[B_9C_2H_9(CH_3)_2]_2^-$ anions appearing at $z = \frac{1}{4}$ and $\frac{3}{4}$ for the chromium atom should be in both the rear and the fore of the unit cell but for clarity only one set of each pair is shown in the drawing.



XBL 692-164

Fig. 1

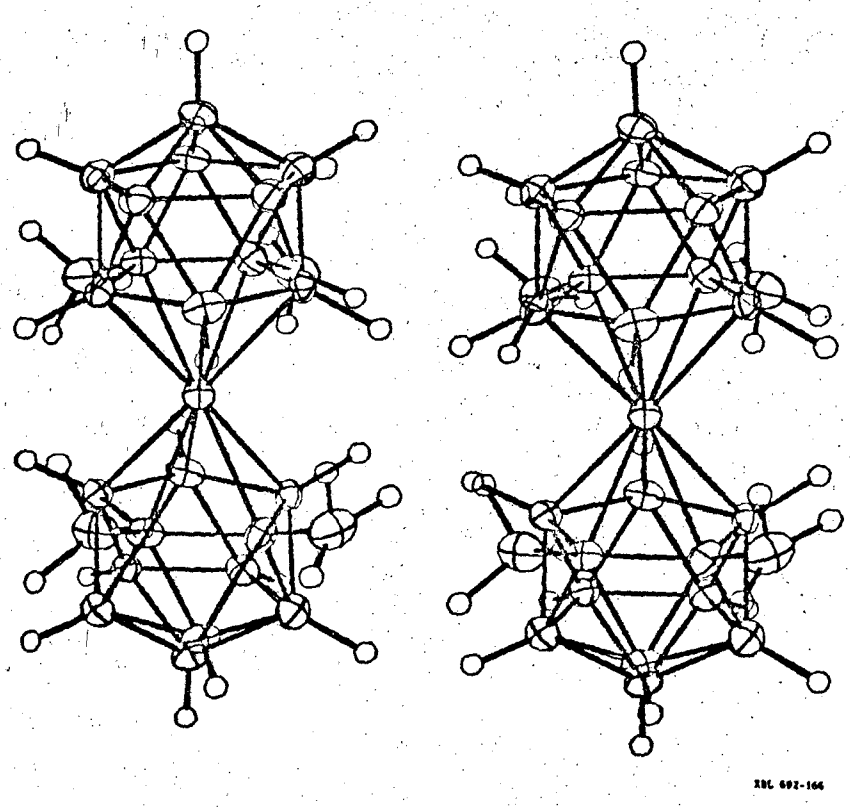


Fig. 2

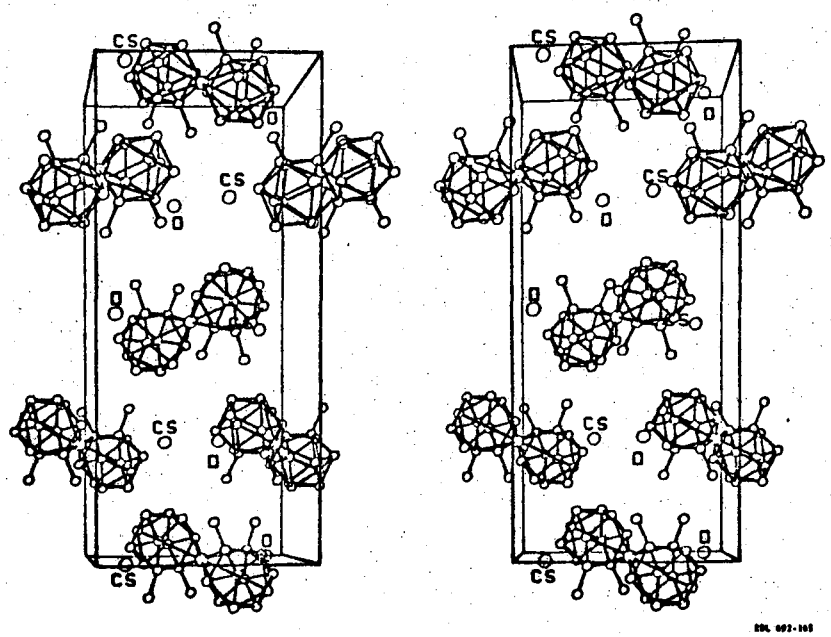


Fig. 3

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

TECHNICAL INFORMATION DIVISION
LAWRENCE RADIATION LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720