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THE STRUCTURE DETERMINATIONS OF ETHYLENE TRITHIOCARBONATE OXIDE AND PHOTOCARYOPHYLLENE D BY X-RAY DIFFRACTION

by

Glen Alan Wolfe

A Dissertation Submitted to the Faculty of the

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In Partial Fulfillment of the Requirements
For the Degree of

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THE UNIVERSITY OF ARIZONA

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THE UNIVERSITY OF ARIZONA

GRADUATE COLLEGE

I hereby recommend that this dissertation prepared under my direction by Glen Alan Wolfe

entitled The Structure Determinations of Ethylene Trithiocarbonate Oxide and Photocaryophyllene D by X-ray Diffraction

be accepted as fulfilling the dissertation requirement of the degree of Doctor of Philosophy

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SIGNED: Glen Alan Wolfe

To My Parents

ACKNOWLEDGMENTS

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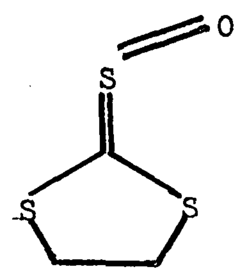
ABSTRACT

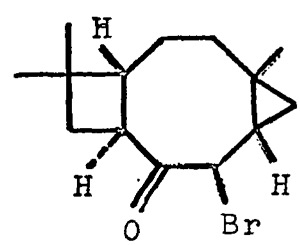
The structure of ethylene trithiocarbonate oxide (I) has been determined by X-ray analysis, providing the first experimental values for bond parameters in a substance containing the thiocarbonyl oxide grouping. The carbon-



I

sulfur-oxygen-angle is 109.4(7)°. The carbon-sulfur and sulfur-oxygen bond distances for this group are 1.64(1) Å and 1.50(1) Å, respectively. The oxygen is 0.25 Å out of the plane formed by the three sulfurs and the carbon between them. The dihedral angle between the oxygen and adjacent ring sulfur, looking down the carbon sulfur double bond, is 9°. The carbon-sulfur bonds of the trithiocarbonate group in the five membered ring have lengths of 1.73(1) Å and 1.72(1) Å, and thus show considerable double bond character. The final R value was 12.7.

The crystal structure of the alpha-bromoketone from photocaryophyllene D (II), one of the four isomeric products from the photolysis of caryophyllene and isocaryophyllene, has been determined by x-ray diffraction.



II

From its spectral and chemical properties this compound was believed to be a stereoisomer of photocaryophyllene A. This proved to be the case: it differs only in the configuration of the methyl group on the three-membered ring which is cis-fused to the eight-membered ring. The dihedral angle of the four membered ring is 28°. The final R value was 11.3.

INTRODUCTION I

The stereochemistry of bonding about sulfur has long been of interest. Phillips (1925) isolated the first optically active sulfinate. The source of the optical activity of sulfinates and sulfoxides is the asymmetric pyramidal sulfur atom. The carbon-sulfur-oxygen bond angle in sulfoxides and sulfinates is remarkably constant, $107(1)^{\circ}$, while the carbon-sulfur-carbon bond angles show erratic variations (Abrahams, 1956).

Interest in isomerism due to sulfur led to the discovery of geometrical isomerism about a carbon-sulfur "double bond" by King and Durst (1963), who isolated two isomers of oxythiobenzoyl chloride. By a combination of U. V., I. R., N. M. R., and dipole moment measurements, these isomers were hypothesized to contain the oxythiocarbonyl group and to be cis-trans isomers. The isomer with the larger dipole moment (mu = 3.97 D) was considered to have the oxygen cis to the chlorine, and the isomer with the smaller dipole moment (mu = 2.63 D) was assigned the trans configuration. The cis isomer is transformed into the trans on heating; the half-life of the cis isomer is 0.75 hours in refluxing toluene and about 8 hours in refluxing carbon tetrachloride.

The postulation of cis-trans isomerism about a carbon-sulfur double bond, requiring that the oxythiocarbonyl group is not linear but bent at some angle, led to the decision to attempt the determination of the geometry of this system by X-ray analysis. The oxide of ethylene trithiocarbonate was chosen for the structure determination because of the simplicity of the structure, the ease of crystallization of the compound, and its availability.

PRELIMINARY INVESTIGATION I

Ethylene trithiocarbonate oxide, m.p. 61°, was obtained by the oxidation of ethylene trithiocarbonate by lead tetraacetate according to the method of Doane, et al. (1965). It was found that crystals suitable for X-ray analysis could be obtained by recrystallization from an ethanol n-pentane solution. The yellow crystals of ethylene trithiocarbonate oxide are unstable to the atmosphere. It was discovered, however, that spraying the crystals with Teflon preserved them for about three days of X-ray analysis.

The oscillation photograph taken with the needle axis perpendicular to the X-ray beam showed no symmetry, nor did the corresponding 0-level Weissenberg. This lack of symmetry on both oscillation and 0-level Weissenberg photographs around an axis indicates triclinic geom. In the search for other axes, no symmetry was found and the crystal system was assumed to be triclinic.

It was necessary at this point to determine a, b, c, α, β, and γ, for a triclinic cell. This was done by shooting around three axes of the crystal in which each of the 0-level Weissenbergs showed two axes, one of which was present on a 0-level Weissenberg around a different axis, and the other on the third 0-level photograph.

The direct axial cell lengths a, b, and c were determined from the oscillation photograph perpendicular to the corresponding axis to be 7.20(1) Å, 5.41(1) Å, and 8.16(1) Å, respectively. α^*, β^*, and γ^* were obtained from the 0-level Weissenbergs around these axes and were determined to be 80.0(1)°, 83.0(1)°, and 105.1(1)°, respectively. These reciprocal angles were then converted to real angles (α = 102.4°, β = 100.1°, and γ = 73.3°). The reciprocal axial lengths a^*, b^*, and c^* were determined from the 0-level Weissenberg photographs and were found to be 0.1465 $Å^{-1}$, 0.1964 $Å^{-1}$, and 0.1269 $Å^{-1}$. All measurements on the photographs were made using a traveling microscope graduated to one hundredth of a millimeter.

The volume of the unit cell was calculated to be 294(1) $Å^3$, and the density was observed to be 1.69(1) gm/cm^3 by flotation in a tetrachloromethane-iodobenzene solution. From the volume of the unit cell, the density, and the molecular weight (152), the number of molecules of ethylene trithiocarbonate oxide per unit cell was determined to be 2.00(2). There are two space groups in the triclinic crystal system, $P\bar{1}$ and P1. $P\bar{1}$ contains two asymmetric units per unit cell related by a center of symmetry while P1 contains only one asymmetric unit and is noncentrosymmetric. The space group was presumed to be $P\bar{1}$ with two molecules per unit cell related by a center of symmetry.

In addition to determining the space group in the triclinic system it is necessary to decide whether the unit cell chosen is triobtuse or triacute. The decision was made on the basis of the volumes of each of these unit cells compared to the volume obtained independent of any real angles. A volume of 302 $\AA^3$ was calculated for the triobtuse cell while the triacute cell had a volume of 294 $\AA^3$. The volume obtained independent of any real angles was 294 $\AA^3$, indicating a triacute cell. This assignment was verified by comparison of the intensities of common reflections on upper level photographs taken around two different axes.

EXPERIMENTAL I

Collection of Intensity Data

All the intensity data were collected using a fine-focus copper target operated at forty kilovolts and twenty milliamperes. A nickel filter provided filtration of the X-beta radiation. The intensity data were collected by the multiple film technique, in which a Weissenberg photograph was taken with a pack of three films for a period of twelve to sixteen hours and another was taken for one hour with a pack of three films. Zero through fourth level Weissenberg photographs around the needle axis (b) were taken in this manner. Intensity data were also collected around the c-axis in order to provide inter-layer scaling of the needle axis data.

Indexing of the Weissenberg Photographs

Indexing of the 0-level Weissenberg photographs presented no difficulty, since the axes are linear and symmetrical. However, indexing the upper level Weissenberg photographs was a problem because the axes are bent, non-symmetrical, and shifted. The technique which was found to be successful in indexing these photographs was the use of 0-level photographs taken around other axes. Comparison of the intensities of reflections common to both the 0-level and upper level photograph was made in attempts to locate the axes on the upper level photograph. Once the axes were found, the non-axial spots were readily indexed.

Determination of the Observed Structure Factors

The relative intensity of each reflection was determined visually by comparison to a calibration film, which contained a series of exposures linearly increasing in density. Inter-film scale factors were determined, and a raw intensity for each reflection was calculated. The Lorentz-Polarization factor (a function of the Bragg angle) was then applied to each reflection in order to obtain the relative observed structure factors. An inter-layer scale factor was then determined for each level by comparison with common reflections on the 0-level shot perpendicular to the c-axis. The intensities of the reflections in each level were then multiplied by the appropriate scale factor in order to put all of the intensities on a common scale.

SOLUTION OF STRUCTURE I

Although $P\bar{1}$ is a centrosymmetric space group and a solution to the structure might have been achieved by direct methods, it was decided that solution of the Patterson map might be the most straightforward approach. Since the ethylene trithiocarbonate oxide molecule has three sulfurs and there are two molecules per unit cell, related by a center of symmetry, there are intramolecular vectors giving the orientation of the three sulfurs in the unit cell as well as intermolecular vectors which represent the location of the three sulfurs in the unit cell (Table 1). Since three of the intramolecular vectors coincide with three others, there must be three double strength vector peaks close to the origin. Since three of the nine intermolecular vectors coincide with three others, there are three double strength vectors and three normal strength vectors not necessarily close to the origin.

A two-dimensional sharpened Patterson map was calculated using the one-hundred and five reflections of the 0-level Weissenberg perpendicular to the needle axis (b). This gave a projection down the b-axis of the nine vectors plus the symmetry related ones as shown in Figure 1. Three double strength vector peaks were close to the origin and

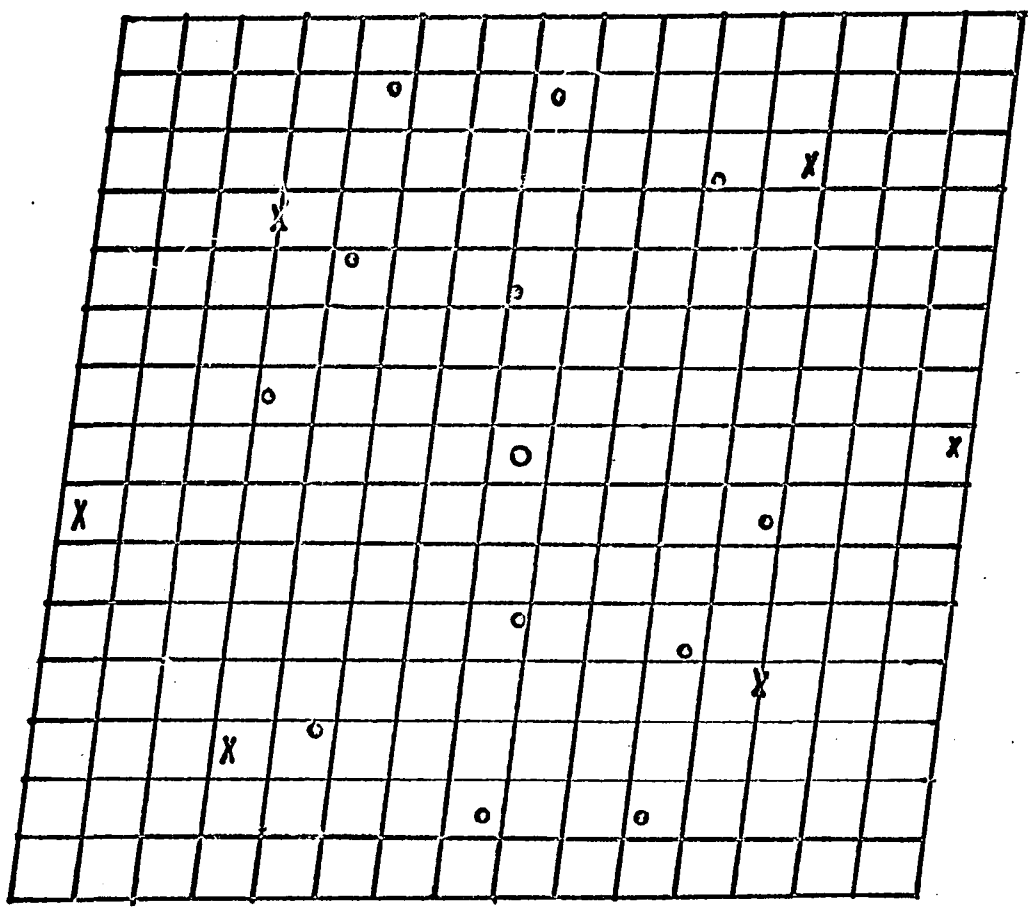


Figure 1

Two Dimensional Patterson I

X = Intramolecular Vector Peaks

o = Intermolecular Vector Peaks

gave an acceptable orientation of the molecule. These were chosen as the intramolecular vectors. The coordinates of one of these peaks were assigned to the $x_1 - x_2$, $z_1 - z_2$ vector and the coordinates of another peak were assigned to the $x_1 - x_3$, $z_1 - z_3$ vector. Expressions for x_2, x_3, z_2, and z_3 in terms of x_1 and z_1 were thus obtained from the equations resulting from these vector assignments. These expressions for x_2, x_3, z_2, and z_3 were then substituted in the intermolecular vector expressions $x_1 + x_2$, $z_1 + z_2$ and $x_1 + x_3$, $z_1 + z_3$ to give the vectors $2x_1 - 7$, $2z_1 - 5.5$ and $2x_1 + 12.5$, $2z_1 - 1$, respectively. Each of the remaining intermolecular vector peaks was then considered to be each of these vectors. Two solutions for x_1 and z_1 were thus obtained from the equations resulting from each vector peak. The two peaks which gave identical solutions for x_1 and z_1 were identified as the $x_1 + x_2$, $z_1 + z_2$ and $x_1 + x_3$, $z_1 + z_3$ vectors. The coordinates of the second and third sulfur atoms were then obtained by substitutions in the appropriate sets of equations. Once the coordinates were known, it was easy to calculate where each of the remaining peaks should lie in the Patterson map. These calculated peaks checked with those observed in the Patterson.

Structure factor calculations were made using the x and z coordinates of the three sulfur atoms and an R value of 35.7 was found. A two-dimensional Fourier map projected down the b-axis was calculated, revealing the x and z coordinates of the three carbons and oxygen. The R value of 18.6 was obtained when all seven atoms were included in the calculation of the structure factors.

The y coordinates of the sulfur atoms were determined by use of a three-dimensional Patterson map. Having solved the structure in two dimensions, it was possible to calculate the x and z coordinates of each of the nine vectors. The nine vectors were thus located and the y coordinate of each sulfur atom was determined. At this point it was possible to estimate the y coordinates of the three carbons and the oxygen by knowing their x and z coordinates and the x, y, and z coordinates of the three sulfurs. Structure factors were calculated taking into account the positions of all seven atoms and the R factor of 36.6 was obtained with poorly scaled data.

After the data had been cross axis scaled and three cycles of least squares isotropic refinement had been carried out, the R value of 21.3 was obtained. Three cycles of anisotropic refinement dropped the R factor to 13.9. Allowing the interlayer scale factors to vary while the temperature factors were held constant gave the R value of 13.0. Three cycles of anisotropic refinement were then run, giving a final R value of 12.7.

The final coordinates of the atoms are given in Table 2, and the temperature factors are shown in Table 3.

The bond distances and bond angles calculated from the coordinates are listed in Table 4 and Table 5, respectively, with their standard deviations. The final observed and calculated structure factors are given in Table 7. The estimated standard deviations in the bond distances and bond angles were determined by the Oak Ridge error program ORFFE.

DISCUSSION OF RESULTS I

The crystal structure reveals that the atoms in ethylene trithiocarbonate oxide are arranged in parallel planes related by a center of symmetry. The distance between the planes of the atoms calculated by the method of least squares is 3.9 Å. No atom deviates from the least squares plane calculated for all the atoms more than 0.3 Å, except carbon-2 which is 0.63 Å from the plane. The most planar part of the molecule is the trithiocarbonate group. The deviations of atoms from this plane are shown in Table 6. The atoms of the five-membered ring deviated as much as ± 0.3 Å from the least squares plane calculated from them. The bond distances and bond angles are shown on the ORTEP plots in Figure 2 and Figure 3, respectively. The ellipsoid of vibration for each atom of the ORTEP plots were calculated from the temperature factors of the atom by the Oak Ridge program.

The parameters of the oxythiocarbonyl group, although never determined before, are consistent with those of analogous compounds. The sulfur-oxygen bond length of 1.50(1) Å compares with values of 1.47(4) Å and 1.43(1) Å for dimethyl sulfoxide and sulfur dioxide, respectively. The carbon-sulfur "double bond" length is 1.64(1) Å

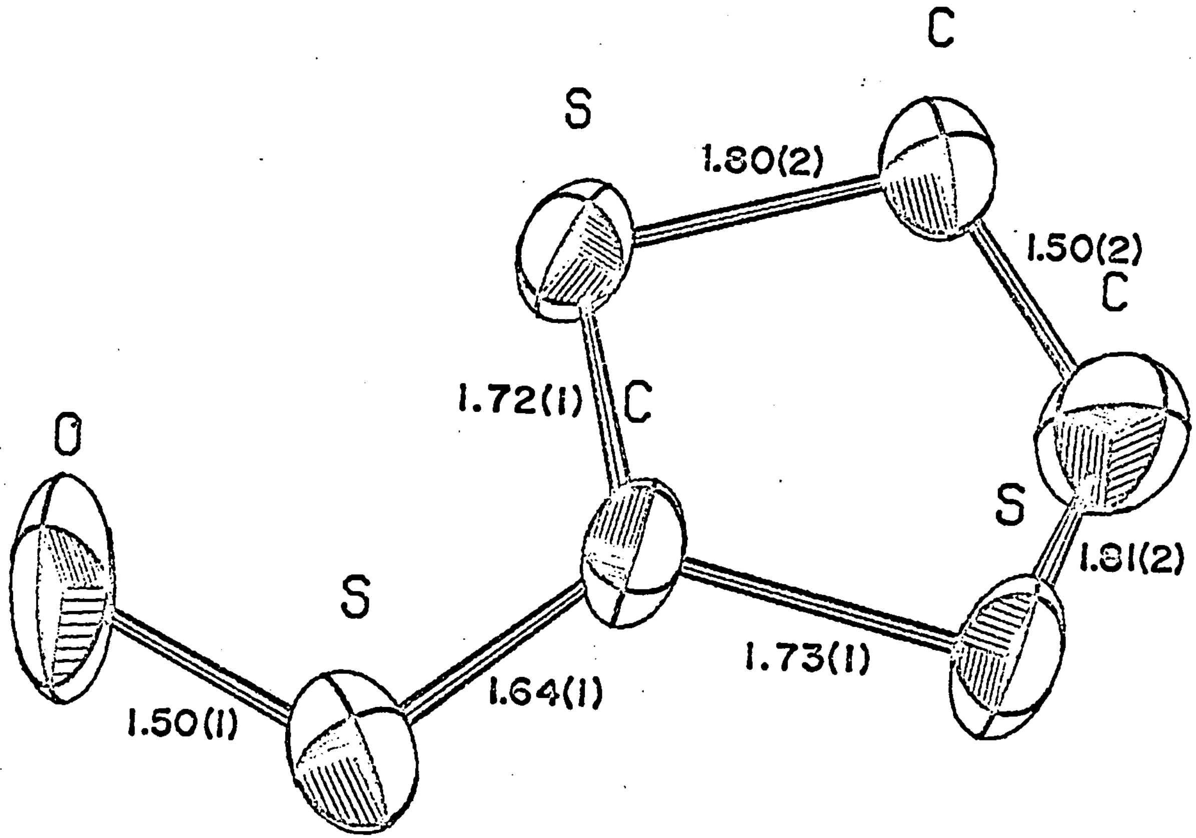


Figure 2. Bond Distances I (Å)

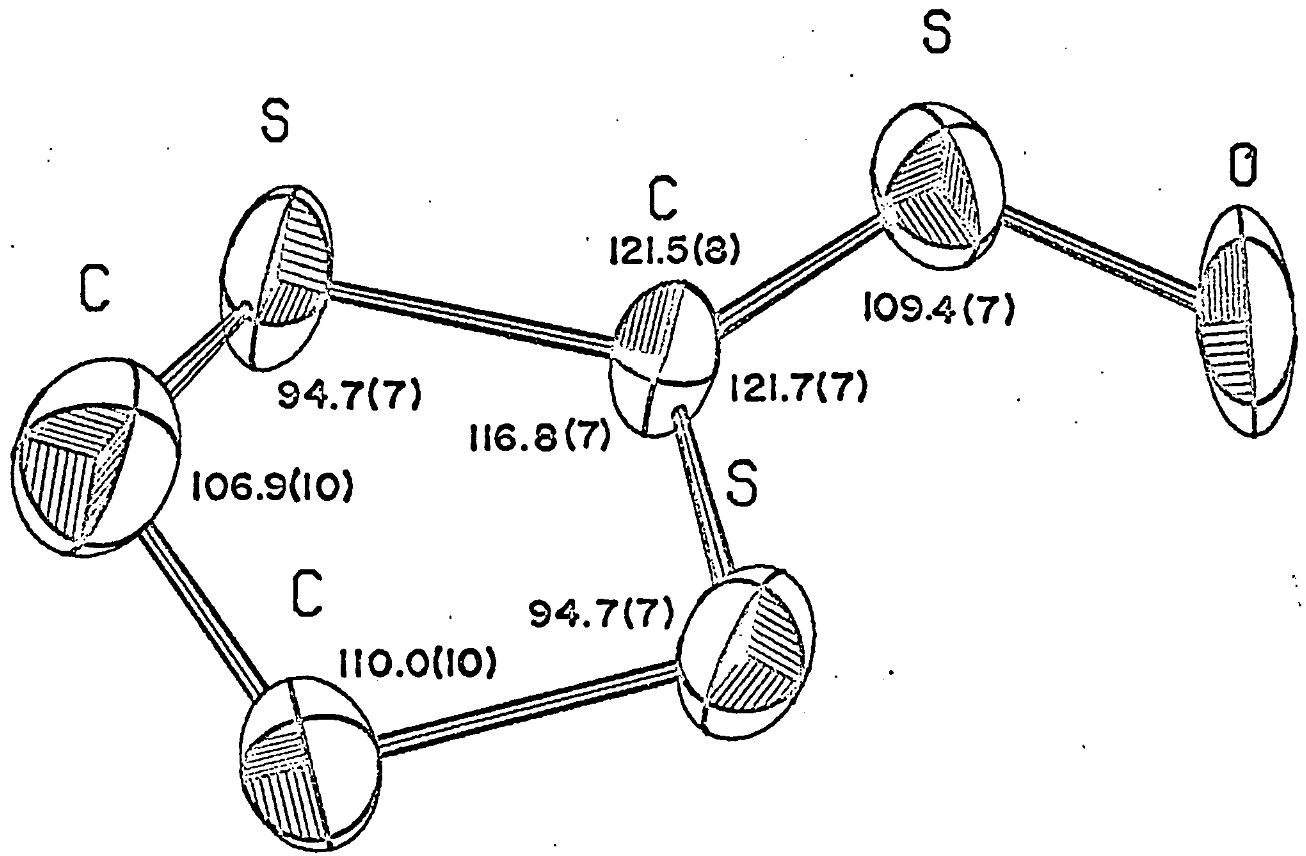


Figure 3. Bond Angles I (degrees)

corresponding to that of 1.68(2) Å for α-thiopyridone. The value of 1.64 Å gives the carbon-sulfur "double bond" of the oxy-thiocarbonyl approximately 90% double bond character (Abrahams, 1956).

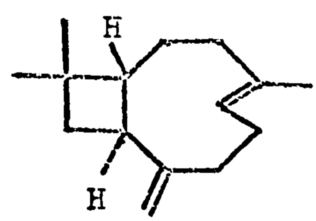
The carbon-sulfur-oxygen angle of $109.4(7)^\circ$ is consistent with the use of atomic orbitals for the sulfur with a 19° expansion of the expected 90° angle due to electrostatic repulsion between the oxygen and adjacent ring sulfur. This repulsion is probably also responsible for the oxygen being 0.25 Å out of the plane formed by the three sulfurs and the carbon between them. The dihedral angle between the oxygen and the adjacent ring sulfur, looking down the sulfur-carbon "double bond", is 9°. The oxygen-sulfur-oxygen angle of $119.0(5)^\circ$ in sulfur dioxide (Abrahams, 1956), which probably has the most similar sulfur atom, may be greater due to even more electrostatic repulsion.

The carbon-sulfur-carbon bond angle of $94.7(7)^\circ$ can be explained in terms of the use of atomic orbitals on sulfur plus 5° of expansion due to strain caused by deviation from 108° (the interior angle of a regular pentagon) and also to the driving force to have the hydrogens staggered. Compression of the ring is also indicated by carbon-2 being 0.731 Å out of the plane of the thiocarbonate group, causing a carbon-carbon-sulfur angle of $106.9(10)^\circ$. The hybridization on the carbon of the trithiocarbonate group corresponds to sp^2. The sulfur-carbon-sulfur bond angle of $116.8(7)^\circ$ in the five-membered ring is probably due to the compression of the ring forcing this carbon slightly out of the plane of the five-membered ring. The sulfur-carbon-sulfur bond angles of $121.7(7)^\circ$ and $121.5(8)^\circ$ (exterior to the ring) correspond very closely to a sp^2 hybridized carbon.

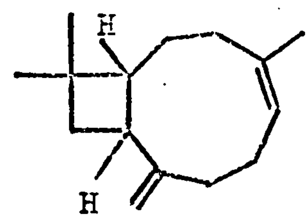
The carbon-sulfur bonds of the trithiocarbonate group in the ring have lengths of 1.73(1) Å and 1.72(1) Å. These values correspond to approximately 60% double bond character and compare with thiophene where the carbon-sulfur bond lengths are 1.74(3) Å. The carbon-sulfur bonds of the 1,2-dithioethane grouping in the ring have lengths of 1.81(2) Å and 1.80 Å, that of a carbon-sulfur single bond.

INTRODUCTION II

Caryophyllene (III), a major constituent of clove oil, was irradiated with ultraviolet light by Ohloff (1967) and was found to give a mixture of four isomeric products, also $C_{15}H_{24}$, which were named photocaryophyllene A, B, C, and D. These were obtained in a yield of 27%, 5%, 2%, and 2%, respectively. Irradiation of isocaryophyllene (IV) gives the same four isomers, but in yields of 8%, 1%, 23%, and 19%, respectively.



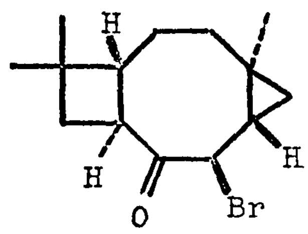
III



IV

Since the molecular formulas of these substances were unchanged, a photochemical rearrangement must be involved. Preliminary investigation of these compounds showed that of the two double bonds in the starting material only the one in the exocyclic methylene group remained in the product.

Finding no other possible means of determining the structure of the photocaryophyllenes, it was decided to use x-ray analysis. Photocaryophyllene A ($C_{15}H_{24}$) was converted to $C_{14}H_{22}O$ by ozonolysis and then brominated to give the α-bromoketone (V), a suitable heavy atom derivative for X-ray analysis. The structure was subsequently determined by Bates and Forsythe (1967).



V

From the spectra of the photocaryophyllenes and the structure of photocaryophyllene A, it was thought that the three isomeric photocaryophyllenes, B, C, and D are probably stereoisomers of A differing only in configuration at the two asymmetric centers formed during the reaction. It was decided to attempt the X-ray crystal structure of a derivative from photocaryophyllene D, one of the major products in the photolysis of isocaryophyllene, in order to confirm this as well as to contribute information as to the mechanism of the photochemical reaction.

PRELIMINARY INVESTIGATION II

Photocaryophyllene D, a major product from the photolysis of isocaryophyllene, was converted to the bromoketone which was considered to be a suitable derivative for x-ray analysis. The crystals which were needles showed extinction every 90° under the polarizing microscope. The oscillation photograph perpendicular to the needle axis (b) showed symmetry. In addition, two screw axes observed on the 0-level Weissenberg photograph were also symmetry axes. This combination of evidence determines an orthorhombic crystal system with either the space group $P2_12_12$ or $P2_12_12_1$. The 0-level Weissenberg photograph around the a-axis showed b^* also to be a screw axis, defining the space group $P2_12_12_1$. There are four equivalent positions in this space group related by three screw axes.

The axial lengths a, b, and c were determined to be 14.34(2) Å, 9.15(2) Å, and 10.66(2) Å, respectively, by means of measurement of a^*, b^*, and c^* on the Weissenberg photographs using a traveling microscope. Values for the lengths of a and b were also obtained from measurement of the oscillation photographs around those axes, providing a very good check on the cell lengths.

The volume of the unit cell was calculated to be 1398.7 Å^3. The density was observed to be 1.35 gm/cm^3 by the flotation method. From the volume of the unit cell, the density, and the molecular weight (285), the number of molecules of photocaryophyllene D in the unit cell was determined to be 4.00(2). Since there are four equivalent positions in $P2_12_12_1$, it was concluded that there is one molecule per asymmetric unit.

EXPERIMENTAL II

The intensity data were collected on a Buerger Automated Diffractometer. The goniometer, which was built by Charles H. Supper Co., New York, employs equi-inclination Weissenberg geometry with a phi scan, which is analogous to a horizontal scan through a spot on the Weissenberg film. A Philips Electronic Instruments generator equipped with voltage and current stabilization provided the x-rays. All the data were collected using a fine-focus copper target tube operated at forty kilovolts and twenty milliamperes. A nickel filter provided filtration of the K-beta radiation. The counting equipment consisted of a Philips transistorized scintillator with a cesium iodide crystal coupled to a Philips control panel. The control panel provided a voltage stabilizer for the scintillation tube and a pulse height analyzer. During the collection of data, the pulse height analyzer was set with a fifteen volt baseline and window.

The b-axis (needle) mounting of the crystal used on the photographs was also used in this collection of the intensity data. The automated equipment was programmed to measure the background for 45 seconds, scan through the reflection for 3° at 1.5°/min., and count the background again for 45 seconds. Measurements were completed through the eighth level, which had a mu angle of 42.4. On this level, over half the possible reflections were still observed.

In order to determine an observed structure factor, the raw intensity was first determined by subtracting the sum of the background counts from the scan count. A reflection was considered observed if its raw intensity was greater than twice the square root of the sum of both backgrounds and the scan count. The Lorentz-Polarization factor (a function of the Bragg angle) was then applied to each observed reflection. This had been calculated previously in the diffractometer setting program, but could not be applied until the reflection had been measured.

The data were scaled by shooting a 0-level around another axis to determine inter-layer scale factors. The intensities of the reflections in each level were then multiplied by the appropriate scale factor in order to put all the intensities on a common scale.

SOLUTION OF THE STRUCTURE II

The structure of photocaryophyllene D bromoketone was solved by the use of the "x-ray system" programs from Sandia Corporation. The bromine was located in the unit cell by calculation of a three-dimensional unsharpened Patterson map using the intensities of 872 reflections. The equivalent positions for $P2_12_12_1$ indicate that there are three bromine-bromine vector peaks each located on a different Harker section. Since there was only one large peak on the Harker section z = 1/2, it was convenient to locate the 1/2 + 2x, 2y, 1/2 vector and to determine the x and y fractional coordinate of the bromine both to be approximately 0.48. Knowing the y coordinate, it was possible to identify the 1/2, 1/2 + 2y, 2z vector on the x = 1/2 Harker section and to find the z fractional coordinate to be 0.445. The third bromine-bromine vector at 2x, 1/2, 1/2 + 2z was a successful check on these coordinates. Structure factors were then calculated using the coordinates of the bromine atom and the R value of 39.9 was found.

A three-dimensional F-observed Fourier map was constructed by putting in the coordinates of the bromine. The electron density map was transferred to clear plastic sheets, which were then stacked to allow visualization of the three-dimensional map. The electron density map clearly showed the location of each of the atoms in the unit cell and the structure was determined. Using the coordinates of all the atoms and an overall temperature factor of 3.0, the R value dropped from 39.9 with just the bromine to 28.3. Three cycles of anisotropic refinement gave the R value of 13.2. Inter-layer scaling of the observed structure factors to the calculated structure factors followed by three cycles of anisotropic refinement gave the final R value of 11.3.

The final coordinates of the atoms are given in Table 8, and the temperature factors are shown in Table 9. The bond distances and bond angles calculated from the coordinates are listed in Table 10 and Table 11, respectively. The estimated standard deviations in the bond distances and bond angles were determined by the Oak Ridge error program ORFFE. The final observed and calculated structure factors are given in Table 12.

DISCUSSION OF RESULTS II

The crystal structure of the bromoketone from photocaryophyllene D shows the four molecules per unit cell to be related by three screw axes. The molecules lie in the plane of the x and z axes. A projection down the y-axis shows considerable overlap of molecules in this plane. It was found that photocaryophyllene D is related to photocaryophyllene A and differs only in the configuration of the methyl group on the three-membered ring, which is cis-fused to the eight-membered ring. The molecule has the configuration shown on the three-dimensional ORTEP plot in Figure 4. The absolute configuration was derived from caryophyllene (Robertson and Todd, 1955), since it is unlikely that any change in the trans-juncture of the cyclobutane ring of caryophyllene would occur under the conditions of the photolysis. The intramolecular bond distances and bond angles are given in Figure 5 and Figure 6, respectively.

The cis-ring juncture of the three-membered ring prevents the atoms of the cyclooctane ring from taking the stable crown configuration as was the case for photocaryophyllene A. However, it was observed that the conformation of the eight-membered ring was probably the most stable possible as far as alleviation of interactions between the

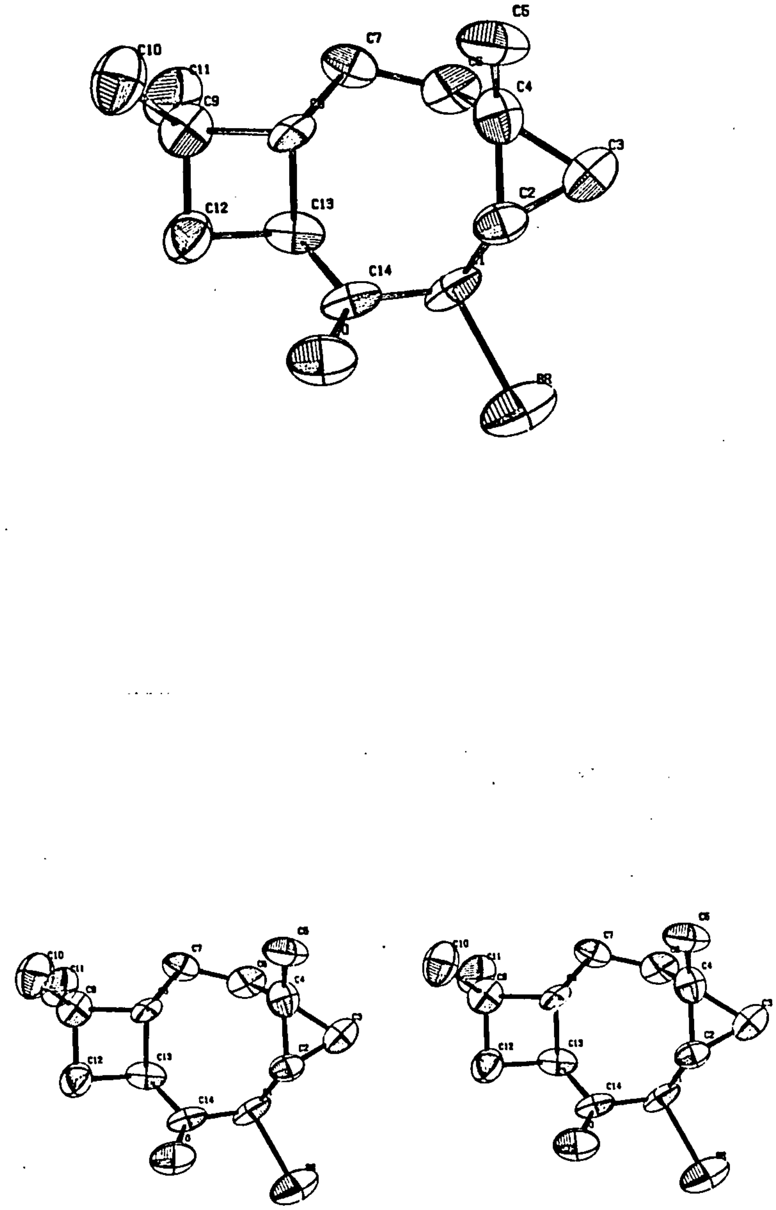


Figure 4. Three Dimensional ORTEP Plot II

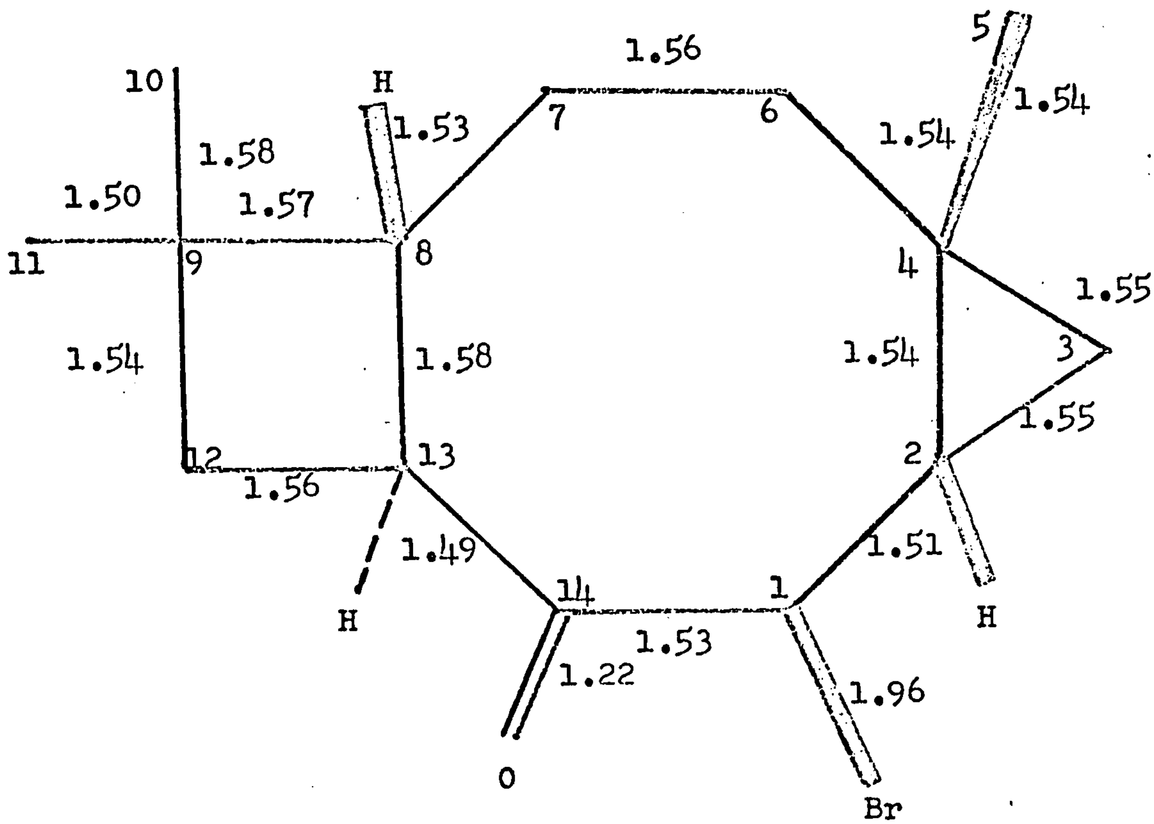


Figure 5

Bond Distances II (Å)

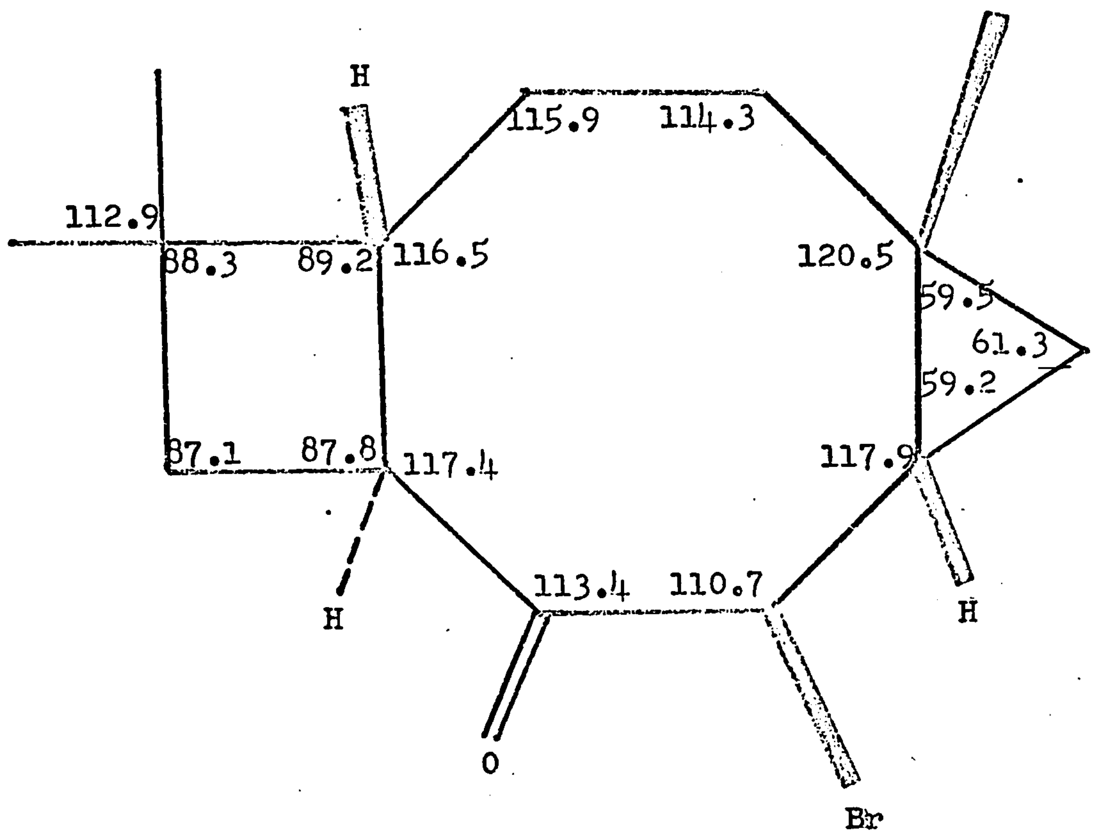


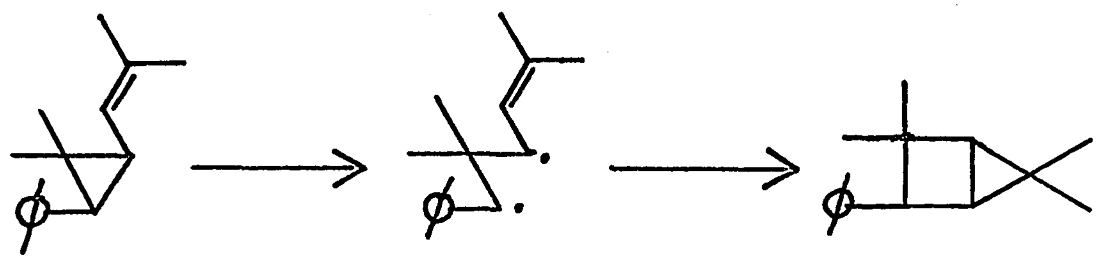
Figure 6

Bond Angles II (degrees)

hydrogens were concerned. The shortest intermolecular atomic distance is between the carbonyl oxygen and the bromine carbon and was determined to be 3.53 Å. It is expected that this is a result of a dipole-dipole interaction, giving added stability to this arrangement. The cyclobutane ring of photocaryophyllene D bromoketone has a dihedral angle of about 28° compared to the value of 23° reported for the A-isomer. The larger dihedral angle in the D-isomer is probably due to increased torsion about the carbon(8)-carbon(13) bond caused by the cis-fusion of the cyclopropane ring.

It is interesting to note that the structures of photocaryophyllene A and D possess much of the original ring strain and seem to add strain by formation of the new cyclopropane ring. At least two mechanisms can be used to explain this rearrangement. In one mechanism, the electronically excited olefin undergoes a 1,2-hydrogen shift to give a 1,3-diradical which can then combine to form the cyclopropane ring. This mechanism was suggested by Golub and Stephens (1968) for a corresponding reaction of 1,4-cis-polyisoprene.

A second mechanism involves opening of the nine-membered ring of caryophyllene with the formation of two allylic radicals, one of which closes to a cyclopropyl radical and couples with the other allylic radical to form the photocaryophyllenes. This is somewhat analogous to the mechanism implied by Kristinsson and Hammond (1967) for the reaction shown below.



APPENDIX

TABLES OF EXPERIMENTAL VALUES FOR I AND II

Table 1

Vectors Generated by the Three Sulfurs ($P\bar{1}$)

1 - 2	$x_1 - x_2$,	$y_1 - y_2$,	$z_1 - z_2$
1 - 3	$x_1 - x_3$,	$y_1 - y_3$,	$z_1 - z_3$
2 - 3	$x_2 - x_3$,	$y_2 - y_3$,	$z_2 - z_3$
1 - $\bar{2}$	$x_1 + x_2$,	$y_1 + y_2$,	$z_1 + z_2$
1 - $\bar{3}$	$x_1 + x_3$,	$y_1 + y_3$,	$z_1 + z_3$
2 - $\bar{3}$	$x_2 + x_3$,	$y_2 + y_3$,	$z_2 + z_3$
1 - $\bar{1}$	$2x_1$,	$2y_1$,	$2z_1$
2 - $\bar{2}$	$2x_2$,	$2y_2$,	$2z_2$
3 - $\bar{3}$	$2x_3$,	$2y_3$,	$2z_3$

Table 2

Fractional Coordinates and Estimated Standard Deviations I

Atom	x/a	ESD	y/b	ESD	z/c	ESD
C(1)	.9460	.0017	.4387	.0027	.2210	.0016
C(2)	.5849	.0022	.7130	.0034	.2392	.0020
C(3)	.7080	.0019	.8341	.0030	.3840	.0018
O	.3157	.0014	.3242	.0024	.2165	.0015
S(1)	.1387	.0005	.2256	.0007	.1471	.0005
S(2)	.7187	.0004	.3741	.0007	.1794	.0005
S(3)	.9588	.0005	.7380	.0007	.3415	.0005

Table 3

Anisotropic Temperature Factors (x100) I

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	1.09	4.45	1.38	-1.05	-0.04	0.41
C(2)	1.86	5.41	2.11	-0.30	-0.08	0.02
C(3)	1.50	4.81	1.83	-0.94	0.53	-0.52
O	1.20	7.48	3.16	-1.40	0.68	0.89
S(1)	1.45	3.81	1.74	-0.30	0.48	0.17
S(2)	1.03	3.44	2.13	-0.68	-0.01	-0.18
S(3)	1.41	3.46	1.77	-0.87	0.07	-0.23

Table 4

Bond Lengths and Estimated Standard Deviations I

Atoms	Distance (Å)	ESD (Å)
S(1)-O	1.496	0.011
C(1)-S(1)	1.638	0.013
C(1)-S(2)	1.732	0.012
C(1)-S(3)	1.719	0.014
C(2)-S(2)	1.812	0.017
C(3)-S(3)	1.803	0.014
C(2)-C(3)	1.497	0.020

Table 5

Bond Angles and Estimated Standard Deviations I

Atoms	Angle (degrees)	ESD
C(1)-S(1)-O	109.36	0.66
S(1)-C(1)-S(2)	121.46	0.81
S(1)-C(1)-S(3)	121.71	0.73
S(2)-C(1)-S(3)	116.82	0.75
C(1)-S(2)-C(2)	94.72	0.71
C(1)-S(3)-C(3)	94.74	0.61
S(2)-C(2)-C(3)	106.90	1.00
S(3)-C(3)-C(2)	110.01	0.97

Table 6

Out of Plane Displacements Three Sulfur-Carbon(1) Plane

Atom	Displacement (Å)
S(1)	0.0221
S(2)	-0.0808
S(3)	-0.0051
C(1)	0.0638
C(2)	-0.7313
C(3)	0.0559
O	0.2467

Table 7

Calculated and Observed Structure Factors (x10) I

H	K	L	FOBS	FCAL
0	0	3	185	249
0	0	4	219	193
0	0	5	159	143
0	0	6	195	177
0	0	7	34	30
0	0	9	11	6
0	3	1	134	119
0	3	-1	123	163
0	3	2	43	41
0	3	-2	184	229
0	3	3	36	29
0	3	-3	111	106
0	3	-4	87	83
0	3	5	57	49
0	3	-5	48	32
0	3	6	54	46
0	3	7	59	61
0	3	8	23	25
0	5	0	54	52
0	4	0	59	60
0	4	1	26	25
0	4	-1	91	96
0	4	-2	38	40
0	4	3	99	77
0	4	-3	77	77
0	4	4	79	57
0	4	-4	56	54
0	4	5	106	83
0	4	-5	37	26
0	4	6	18	14
0	4	-6	36	40
0	4	-8	29	23
0	6	0	45	48
0	1	1	227	247
0	1	2	354	389
0	1	3	226	183
0	1	-3	114	91
0	1	4	274	215

H	K	L	FOBS	FCAL
0	1	-4	402	360
0	1	5	45	40
0	1	-5	98	117
0	1	6	25	25
0	1	-6	86	76
0	1	-7	81	61
0	1	8	47	52
0	1	-9	14	9
0	2	1	154	191
0	2	-1	166	189
0	2	2	189	208
0	2	-2	63	58
0	2	3	78	67
0	2	-3	87	60
0	2	4	20	22
0	2	-4	284	278
0	2	-5	35	16
0	2	6	55	31
0	2	-6	146	164
0	2	7	19	21
0	2	-7	28	35
1	0	2	243	228
1	0	3	90	67
1	0	4	224	211
1	0	5	74	66
1	0	6	226	210
1	0	7	27	19
1	0	8	23	18
1	1	0	273	307
1	1	1	34	32
1	1	-1	218	178
1	1	2	360	351
1	1	-3	77	100
1	1	4	249	263
1	1	-4	155	151
1	1	-5	26	16
1	1	6	52	48
1	1	-6	53	41

H	K	L	FOBS	FCAL
1	2	1	18	6
1	2	2	212	219
1	2	-2	123	117
1	2	3	32	12
1	2	-3	92	83
1	2	4	148	158
1	2	-4	303	324
1	2	-5	11	20
1	2	6	163	180
1	2	-6	131	139
1	2	7	23	20
1	2	8	33	32
1	3	0	139	183
1	3	1	55	44
1	3	2	203	219
1	3	3	57	38
1	3	4	203	219
1	3	6	19	17
1	3	7	21	15
1	3	8	31	51
1	4	0	137	165
1	4	1	57	42
1	4	2	177	142
1	4	-2	75	83
1	4	3	25	12
1	4	-3	25	9
1	4	4	49	26
1	4	-4	179	205
1	4	5	22	12
1	4	-5	51	42
1	4	6	81	70
1	4	-6	67	68
1	4	-7	36	39
1	4	-8	37	42
1	6	0	37	30
1	5	0	77	70
-1	0	1	203	213
-1	0	2	104	98
-1	0	3	116	119
-1	0	4	161	159
-1	0	5	55	47
-1	0	6	23	19
-1	0	7	47	41
-1	0	8	41	37
-1	0	9	55	60
-1	1	0	227	260
-1	1	1	157	174
-1	1	-1	145	149

H	K	L	FOBS	FCAL
-1	1	2	173	163
-1	1	-2	63	62
-1	1	3	52	44
-1	1	-3	10	9
-1	1	4	49	50
-1	1	-4	163	134
-1	1	5	138	122
-1	1	-5	61	64
-1	1	6	79	75
-1	1	-6	36	37
-1	1	7	130	126
-1	1	-7	51	41
-1	1	-8	79	81
-1	1	9	29	23
-1	2	0	35	18
-1	2	1	31	31
-1	2	2	214	205
-1	2	3	161	201
-1	2	-3	136	134
-1	2	4	152	143
-1	2	5	189	181
-1	2	-5	17	19
-1	2	-6	79	79
-1	2	7	19	16
-1	2	8	56	49
-1	3	0	241	277
-1	3	1	177	223
-1	3	-1	33	23
-1	3	2	185	189
-1	3	-2	91	89
-1	3	3	175	192
-1	3	-3	172	171
-1	3	4	23	12
-1	3	-4	84	76
-1	3	5	15	19
-1	3	-5	129	111
-1	3	6	85	87
-1	3	-6	40	29
-1	3	7	66	78
-1	3	8	29	40
-1	4	0	117	108
-1	4	1	149	122
-1	4	-1	161	163
-1	4	-2	178	175
-1	4	3	75	53
-1	4	4	74	115
-1	4	-4	59	62
-1	4	5	104	84

H	K	L	FOBS	FCAL
-1	4	-5	68	76
-1	4	6	61	47
-1	4	-6	34	37
-1	4	-7	35	48
-1	4	-8	18	25
-1	5	0	73	74
-1	6	0	59	63
2	0	0	127	115
2	0	1	292	314
2	0	2	251	245
2	0	3	236	218
2	0	4	164	145
2	0	5	27	30
2	0	6	49	46
2	0	7	54	52
2	0	8	44	48
2	0	9	29	36
2	1	0	118	137
2	1	2	31	9
2	1	-2	114	108
2	1	3	87	85
2	1	-3	37	35
2	1	4	164	138
2	1	-4	262	224
2	1	5	64	53
2	1	-5	222	197
2	1	6	56	49
2	1	-6	97	116
2	1	7	56	55
2	1	-7	121	135
2	1	8	36	36
2	2	0	93	90
2	2	1	69	57
2	2	2	178	166
2	2	-2	92	79
2	2	3	99	79
2	2	-3	149	146
2	2	-4	42	21
2	2	5	101	92
2	2	-5	36	34
2	2	6	98	93
2	2	-6	68	59
2	2	7	32	28
2	2	-7	62	67
2	2	-8	20	47
2	2	-9	37	58
2	3	0	82	77
2	3	1	113	97

H	K	L	FOBS	FCAL
2	3	-1	23	27
2	3	2	155	160
2	3	-2	86	89
2	3	3	144	133
2	3	-3	89	72
2	3	4	178	190
2	3	-4	75	53
2	3	5	67	59
2	4	1	125	94
2	4	-1	37	43
2	4	3	87	58
2	4	-3	16	21
2	4	4	42	30
2	4	-4	79	80
2	4	-5	31	21
2	4	6	72	116
2	4	-6	16	0
2	4	-7	30	13
2	4	-8	35	35
2	5	0	140	123
2	6	0	91	86
-2	0	1	33	53
-2	0	2	300	335
-2	0	4	142	129
-2	0	5	244	222
-2	0	6	94	82
-2	0	7	62	54
-2	0	8	103	105
-2	0	9	76	85
-2	1	0	233	245
-2	1	1	497	505
-2	1	-1	187	190
-2	1	2	38	34
-2	1	-2	100	76
-2	1	3	251	231
-2	1	-3	96	107
-2	1	4	204	195
-2	1	-4	191	157
-2	1	5	111	113
-2	1	-5	83	89
-2	1	6	220	187
-2	1	-6	139	119
-2	1	7	129	114
-2	1	8	54	53
-2	2	0	32	25
-2	2	1	139	125
-2	2	-1	289	316
-2	2	2	215	203

H	K	L	FOBS	FCAL
-2	2	-2	183	184
-2	2	3	150	137
-2	2	-3	137	133
-2	2	4	184	204
-2	2	-4	109	86
-2	2	5	109	91
-2	2	-5	38	40
-2	2	6	59	52
-2	2	-6	59	54
-2	2	7	18	24
-2	2	8	33	29
-2	3	0	145	150
-2	3	1	157	149
-2	3	-1	14	6
-2	3	2	152	163
-2	3	-2	10	21
-2	3	3	69	49
-2	3	-3	83	128
-2	3	4	24	24
-2	3	-4	147	122
-2	3	5	54	54
-2	3	-5	73	63
-2	3	6	55	53
-2	3	-6	78	64
-2	3	7	13	12
-2	4	0	107	92
-2	4	-1	96	85
-2	4	2	16	8
-2	4	-2	90	88
-2	4	3	61	50
-2	4	-3	44	46
-2	4	4	71	43
-2	4	5	41	26
-2	4	-5	25	28
-2	4	6	33	25
-2	4	-6	26	39
3	0	0	281	260
3	0	1	147	128
3	0	2	393	383
3	0	3	27	19
3	0	4	174	174
3	0	5	145	135
3	0	6	62	55
3	0	7	105	112
3	0	8	82	-95
3	0	9	18	31
3	1	0	401	460
3	1	1	30	13

H	K	L	FOBS	FCAL
3	1	2	212	206
3	1	3	212	242
3	1	-3	16	6
3	1	4	89	106
3	1	-4	44	32
3	1	5	218	183
3	1	6	141	147
3	1	-6	76	73
3	1	7	44	48
3	1	-7	44	48
3	1	8	33	35
3	1	-9	43	37
3	2	0	109	89
3	2	1	252	245
3	2	2	187	185
3	2	-2	260	262
3	2	3	196	188
3	2	-3	168	203
3	2	4	170	185
3	2	-4	121	127
3	2	5	49	35
3	2	-5	67	62
3	2	6	31	26
3	2	-6	30	39
3	2	7	51	40
3	2	-7	59	71
3	2	8	33	32
3	2	-8	27	50
3	3	0	249	240
3	3	1	196	182
3	3	-1	231	221
3	3	2	200	201
3	3	-2	31	21
3	3	3	35	24
3	3	-3	29	25
3	3	4	24	12
3	3	-4	147	124
3	3	5	82	76
3	3	-5	144	126
3	3	6	44	36
3	3	7	57	71
3	3	-7	68	58
3	4	0	98	90
3	4	1	44	28
3	4	-1	125	130
3	4	2	32	25
3	4	-2	116	136
3	4	3	85	68

H	K	L	FOBS	FCAL
3	4	-3	132	140
3	4	4	20	12
3	4	-4	40	29
3	4	5	84	79
3	4	-5	49	36
3	4	6	46	45
3	4	-6	61	53
3	4	-7	46	44
3	4	-8	43	36
3	5	0	54	49
3	6	0	40	43
-3	0	1	131	136
-3	0	2	85	71
-3	0	3	231	228
-3	0	4	122	114
-3	0	5	19	4
-3	0	6	32	25
-3	0	7	105	94
-3	0	8	48	53
-3	0	9	20	21
-3	1	0	156	133
-3	1	1	95	82
-3	1	-1	101	96
-3	1	2	169	155
-3	1	-2	81	77
-3	1	3	162	145
-3	1	-3	61	62
-3	1	5	117	138
-3	1	-5	55	50
-3	1	6	34	29
-3	1	-6	81	87
-3	1	-7	29	26
-3	1	-8	11	13
-3	1	9	39	52
-3	2	0	110	95
-3	2	1	229	231
-3	2	-1	66	50
-3	2	2	39	26
-3	2	-2	78	74
-3	2	3	150	137
-3	2	-3	62	48
-3	2	-4	30	19
-3	2	5	83	68
-3	2	-6	64	64
-3	2	7	92	86
-3	3	0	14	12
-3	3	1	89	72
-3	3	-1	200	202
-3	3	2	19	18
-3	3	-2	27	20
-3	3	3	138	145
-3	3	-3	82	67
-3	3	-4	18	14
-3	3	5	111	123
-3	3	-5	55	42
-3	3	6	34	31
-3	3	-6	16	9
-3	3	7	12	8
-3	4	0	33	27
-3	4	1	185	164
-3	4	2	19	6
-3	4	3	139	116
-3	4	4	39	49
-3	4	5	34	24
4	0	0	53	51
4	0	1	143	123
4	0	2	79	68
4	0	3	136	126
4	0	4	22	18
4	0	5	86	87
4	0	6	63	70
4	0	8	29	46
4	1	0	185	144
4	1	1	95	110
4	1	2	44	39
4	1	4	137	150
4	1	-4	35	13
4	1	5	53	56
4	1	-5	99	95
4	1	6	83	99
4	1	-6	24	13
4	1	-7	71	74
4	1	-8	29	18
4	1	-9	52	66
4	2	0	33	30
4	2	1	148	134
4	2	2	210	197
4	2	3	120	132
4	2	4	131	132
4	2	-4	138	143
4	2	-5	102	112
4	2	-6	24	27
4	2	7	36	35
4	2	-7	65	76
4	3	0	272	238
4	3	1	180	154

H	K	L	FOBS	FCAL
4	3	-1	227	202
4	3	2	170	157
4	3	-2	71	54
4	3	3	52	37
4	3	-3	27	21
4	3	4	54	36
4	3	-4	152	130
4	3	5	85	89
4	3	-5	63	43
4	3	6	78	92
4	3	-6	113	98
4	3	7	26	33
4	3	-7	51	28
4	4	0	140	137
4	4	1	94	97
4	4	-1	94	85
4	4	2	65	50
4	4	-2	180	205
4	4	3	145	135
4	4	-3	141	152
4	4	4	108	108
4	4	-4	73	72
4	4	5	56	52
4	4	6	27	28
4	5	0	47	48
4	6	0	42	47
-4	0	1	324	315
-4	0	2	20	1
-4	0	3	46	30
-4	0	4	45	31
-4	0	5	218	218
-4	0	6	19	12
-4	0	7	137	139
-4	0	8	34	34
-4	1	0	62	50
-4	1	1	48	30
-4	1	-1	319	275
-4	1	2	110	92
-4	1	-2	40	20
-4	1	3	289	285
-4	1	-4	35	17
-4	1	-5	42	33
-4	1	6	51	51
-4	1	7	25	28
-4	1	-7	50	53
-4	1	8	23	26
-4	1	-8	8	16
-4	1	9	21	50

H	K	L	FOBS	FCAL
-4	2	0	130	109
-4	2	1	221	224
-4	2	-1	52	35
-4	2	2	56	35
-4	2	-2	82	62
-4	2	3	109	99
-4	2	-3	182	184
-4	2	4	51	47
-4	2	5	92	73
-4	2	-5	125	137
-4	2	7	76	75
-4	3	0	25	15
-4	3	1	46	47
-4	3	-1	163	164
-4	3	2	67	53
-4	3	-2	90	77
-4	3	3	112	105
-4	3	-3	56	50
-4	3	4	19	23
-4	3	-4	48	43
-4	3	5	63	89
-4	3	-5	102	90
-4	3	6	20	13
-4	4	0	41	35
-4	4	1	77	69
-4	4	2	23	24
-4	4	3	59	48
-4	4	-4	28	36
5	0	0	173	174
5	0	1	237	236
5	0	2	139	124
5	0	3	131	124
5	0	4	16	10
5	0	5	153	175
5	0	6	14	13
5	0	7	19	26
5	1	1	310	273
5	1	3	260	272
5	1	-4	39	36
5	1	-5	103	126
5	1	6	23	5
5	1	-6	60	61
5	1	-7	47	43
5	1	-8	18	25
5	2	0	55	40
5	2	2	27	30
5	2	-4	154	116
5	2	-4	95	116

H	K	L	FOBS	FCAL
5	2	5	89	80
5	2	-6	20	34
5	2	-7	65	87
5	3	0	82	54
5	3	1	97	75
5	3	-1	188	151
5	3	-2	53	34
5	3	3	114	106
5	3	-3	232	220
5	3	4	68	66
5	3	-4	49	29
5	3	5	21	14
5	3	-5	37	30
5	3	6	33	36
5	3	-6	54	47
5	4	0	25	25
5	4	1	38	72
5	4	-1	102	98
5	4	2	98	100
5	4	-2	43	46
5	4	-3	33	21
5	4	4	63	58
5	4	-4	25	19
5	5	0	88	98
5	6	0	33	39
-5	0	1	380	407
-5	0	2	21	5
-5	0	3	165	160
-5	0	5	83	66
-5	0	6	49	52
-5	0	7	60	63
-5	0	8	12	7
-5	1	0	13	3
-5	1	1	128	102
-5	1	-1	306	279
-5	1	2	106	92
-5	1	-2	101	118
-5	1	3	68	60
-5	1	4	59	58
-5	1	-4	83	86
-5	1	5	42	39
-5	1	-5	63	23
-5	1	6	38	33
-5	1	-6	14	14
-5	1	7	30	26
-5	1	-7	51	71
-5	1	8	33	34
-5	2	0	60	41

H	K	L	FOBS	FCAL
-5	2	1	76	58
-5	2	-1	24	10
-5	2	2	55	46
-5	2	-3	109	107
-5	2	4	61	54
-5	2	-4	58	57
-5	2	5	43	32
-5	2	-5	81	83
-5	2	6	52	43
-5	2	-6	36	39
-5	3	0	58	53
-5	3	-1	45	39
-5	3	2	83	83
-5	3	-2	30	21
-5	3	3	25	23
-5	3	-3	30	25
-5	3	4	50	50
-5	3	5	22	19
-5	3	-5	27	27
-5	4	0	65	77
-5	4	-1	26	32
-5	4	2	46	40
-5	4	-2	34	43
-5	4	3	34	41
6	0	1	102	86
6	0	2	30	29
6	0	3	16	14
6	0	4	37	43
6	0	5	48	58
6	1	3	79	87
6	2	2	55	35
6	2	3	56	55
6	2	4	36	37
6	2	5	107	119
6	3	0	48	32
6	3	1	102	88
6	3	-1	179	150
6	3	2	42	38
6	3	-2	63	46
6	3	3	140	168
6	3	-3	206	185
6	3	4	42	36
6	3	-4	38	38
6	3	5	29	31
6	3	-5	30	24
6	3	-6	46	56
6	3	-7	57	65
6	4	0	34	26

H	K	L	FOBS	FCAL
6	4	1	139	159
6	4	-1	103	108
6	4	2	30	23
6	4	-3	75	82
6	4	-4	20	27
-6	0	1	86	77
-6	0	2	49	40
-6	0	3	28	23
-6	0	4	107	96
-6	0	5	49	50
-6	0	6	48	51
-6	0	7	20	14
-6	0	8	21	31
-6	1	0	80	61
-6	1	1	111	100
-6	1	-1	29	25
-6	1	-2	25	20
-6	1	3	68	61
-6	1	-3	33	32
-6	1	4	54	50
-6	1	-4	42	40
-6	1	5	53	43
-6	1	-5	16	7
-6	1	6	52	62
-6	2	0	129	104
-6	2	1	26	22
-6	2	-1	103	87
-6	2	2	13	19
-6	2	-2	65	53
-6	2	3	96	81
-6	2	-3	49	41
-6	2	4	92	88
-6	2	-4	15	15
-6	2	5	71	75
-6	2	6	47	64
-6	3	1	96	115
-6	3	-1	32	23
-6	3	2	84	93
-6	3	-2	63	76
-6	3	3	66	85
-6	3	-3	33	47
-6	3	4	40	61
7	0	0	153	159
7	0	1	101	110
7	0	2	107	124
7	0	3	75	88
7	3	0	96	92
7	3	1	46	38

H	K	L	FOBS	FCAL
7	3	2	94	107
7	3	-2	44	37
7	3	3	57	51
7	3	-3	25	24
7	3	4	39	49
7	3	-4	46	31
7	3	-5	78	68
7	3	-6	81	67
7	3	-7	44	50
7	4	0	76	91
7	4	1	65	60
7	4	-1	52	56
7	4	2	38	46
7	4	-2	53	59
7	4	3	18	24
7	4	-3	19	5
7	4	-4	30	26
7	5	0	32	39
-7	0	1	49	40
-7	0	2	15	8
-7	0	3	165	180
-7	0	4	109	128
-7	0	5	85	106
-7	0	6	41	57
-7	0	7	16	31
-7	1	0	39	35
-7	1	1	153	143
-7	1	-1	43	34
-7	1	2	106	108
-7	1	-2	65	77
-7	1	3	52	65
-7	1	-3	59	80
-7	1	4	22	32
-7	1	-4	46	62
-7	1	5	53	65
-7	1	6	25	34
-7	2	0	78	65
-7	2	-1	67	84
-7	2	3	74	73
-7	2	-3	16	26
-7	2	4	29	26
8	0	0	22	30
8	0	2	8	9
8	0	3	34	57
8	3	0	95	103
8	3	1	99	109
8	3	-1	130	125
8	3	-2	96	99

H	K	L	FOBS	FCAL
8	3	-3	13	12
8	3	-5	69	74
8	3	-6	27	48
8	4	-1	41	54
8	4	-2	64	77
-8	0	1	51	61
-8	0	2	60	68
-8	0	3	15	24
-8	0	4	23	26
-8	0	5	11	5
-8	1	0	47	55
-8	1	3	22	15
9	3	-3	16	35

Table 8

Coordinates and Estimated Standard Deviations II

Atom	Coordinates and ESD in Å					
	x/a	ESD	y/b	ESD	z/c	ESD
Br	.4801	.0022	.4733	.0021	.4431	.0013
O	.3883	.0132	.0980	.0136	.2562	.0100
C(1)	.5276	.0130	.9620	.0126	.2404	.0053
C(2)	.6026	.0087	.0648	.0101	.2865	.0079
C(3)	.7042	.0186	.0020	.0275	.2782	.0140
C(4)	.6583	.0120	.0276	.0186	.4089	.0133
C(5)	.6872	.0151	.1544	.0148	.4861	.0124
C(6)	.6344	.0147	.8835	.0136	.4824	.0145
C(7)	.5573	.0170	.9020	.0165	.5809	.0152
C(8)	.4716	.0089	.9864	.0110	.5380	.0086
C(9)	.3799	.0125	.9710	.0118	.6166	.0111
C(10)	.3646	.0163	.1093	.0180	.7049	.0129
C(11)	.3696	.0201	.8318	.0191	.6882	.0141
C(12)	.3239	.0154	.9701	.0178	.4927	.0152
C(13)	.4159	.0154	.9270	.0119	.4240	.0124
C(14)	.4342	.0110	.9991	.0104	.3012	.0085

Table 9

Anisotropic Temperature Factors II

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{31}
Br	10.4	6.6	2.7	1.1	0.1	-0.6
O	7.6	8.7	5.1	0.5	3.2	-0.8
C(1)	7.7	4.6	0.6	2.1	0.8	0.2
C(2)	1.3	2.1	2.1	0.2	-1.3	-0.5
C(3)	11.2	16.7	5.1	-2.8	0.8	0.1
C(4)	3.0	8.2	3.6	1.2	-0.2	0.4
C(5)	7.6	4.9	4.4	-0.4	0.1	-1.9
C(6)	7.6	3.4	5.8	0.6	0.4	-1.9
C(7)	11.4	5.1	6.3	-0.7	1.8	-1.9
C(8)	3.0	1.1	1.2	-0.5	-0.6	-0.4
C(9)	4.1	2.9	4.1	-3.9	-0.8	-0.2
C(10)	9.6	5.6	6.3	0.9	-0.5	1.7
C(11)	11.8	7.2	6.1	-2.2	1.6	1.0
C(12)	4.7	6.3	4.6	-0.1	-0.3	0.4
C(13)	7.7	3.0	5.0	-0.8	0.7	-2.0
C(14)	7.4	0.6	2.4	-0.1	-0.3	-2.1

Table 10

Bond Lengths and Estimated Standard Deviations II

Atoms	ESD	Distance
C(1)-Br	0.010	1.96
C(1)-C(2)	0.024	1.51
C(1)-C(14)	0.022	1.49
C(2)-C(3)	0.027	1.57
C(2)-C(4)	0.023	1.57
C(3)-C(4)	0.023	1.55
C(4)-C(5)	0.028	1.54
C(4)-C(6)	0.028	1.53
C(6)-C(7)	0.024	1.53
C(7)-C(8)	0.021	1.53
C(8)-C(9)	0.021	1.57
C(8)-C(13)	0.019	1.55
C(9)-C(10)	0.027	1.58
C(9)-C(11)	0.028	1.50
C(9)-C(12)	0.023	1.54
C(12)-C(13)	0.019	1.56
C(13)-C(14)	0.021	1.49
C(14)-O	0.019	1.22

Table 11

Bond Angles and Estimated Standard Deviations II

Atoms	ESD	Angle (degrees)
C(14)-C(1)-Br	1.00	110.7
C(14)-C(1)-C(2)	1.23	110.5
Br-C(1)-C(2)	1.01	108.0
C(1)-C(2)-C(3)	1.70	114.7
C(1)-C(2)-C(4)	1.50	119.9
C(3)-C(2)-C(4)	1.06	59.6
C(2)-C(3)-C(4)	1.11	60.1
C(2)-C(4)-C(3)	1.18	60.3
C(2)-C(4)-C(5)	1.84	115.9
C(2)-C(4)-C(6)	1.68	119.0
C(3)-C(4)-C(5)	1.91	119.7
C(3)-C(4)-C(6)	1.99	114.5
C(5)-C(4)-C(6)	1.55	116.1
C(4)-C(6)-C(7)	1.48	114.1
C(6)-C(7)-C(8)	1.23	115.7
C(7)-C(8)-C(9)	1.23	118.6
C(7)-C(8)-C(13)	1.35	118.5
C(9)-C(8)-C(13)	1.13	87.2
C(8)-C(9)-C(10)	1.47	111.4
C(8)-C(9)-C(11)	1.68	115.4
C(8)-C(9)-C(12)	1.13	89.2

Atoms	ESD	Angle (degrees)
C(10)-C(9)-C(11)	1.52	111.1
C(12)-C(9)-C(10)	1.68	115.9
C(12)-C(9)-C(11)	1.69	112.3
C(9)-C(12)-C(13)	1.26	87.8
C(12)-C(13)-C(8)	1.22	89.0
C(12)-C(13)-C(14)	1.50	116.8
C(8)-C(13)-C(14)	1.28	113.2
C(13)-C(14)-C(1)	1.37	115.5
C(13)-C(14)-O	1.62	125.5
O-C(14)-C(1)	1.44	119.2

Table 12

Out of Plane Displacements for the Four-membered Ring

Atom	Displacement (Å)
C(8)	0.1339
C(9)	-0.1346
C(12)	0.1353
C(13)	-0.1346

Table 13

Calculated and Observed Structure Factors (x10) II

H	K	L	FOBS	FCAL
0	0	2	659	710
0	0	4	899	880
0	0	6	651	611
0	0	8	435	445
0	0	10	222	258
0	0	12	112	115
0	1	1	71	15
0	1	2	742	694
0	1	3	114	470
0	1	4	966	103
0	1	5	131	99
0	1	6	384	428
0	1	7	94	112
0	1	8	229	262
0	1	10	151	125
0	1	11	75	71
0	1	12	278	255
0	2	1	233	218
0	2	2	638	609
0	2	3	425	347
0	2	4	473	381
0	2	5	222	178
0	2	6	547	566
0	2	7	146	163
0	2	8	363	390
0	2	10	228	268
0	2	11	71	95
0	2	12	127	122
0	3	1	104	60
0	3	3	145	115
0	3	4	949	945
0	3	5	135	126
0	3	6	312	341
0	3	7	252	301
0	3	8	229	252
0	3	9	948	977
0	3	11	130	136
0	3	12	215	190
0	4	1	77	91
0	4	2	346	405
0	4	3	253	336
0	4	4	56	15
0	4	5	319	332
0	4	6	190	209
0	4	7	201	224
0	4	8	227	282
0	4	10	182	188
0	4	11	114	147
0	5	1	70	222
0	5	2	138	233
0	5	4	364	442
0	5	6	180	208
0	5	7	262	292
0	5	8	134	140
0	5	9	171	164
0	5	11	128	105
0	6	1	36	73
0	6	2	181	236
0	6	3	185	194
0	6	4	48	60
0	6	5	328	327
0	6	7	75	70
0	6	8	128	140
0	7	2	82	94
0	7	3	159	176
0	7	4	166	178
0	7	7	94	16
0	7	9	107	115
0	8	1	22	48
0	8	2	41	90
0	8	3	57	53
0	8	4	46	64
0	8	5	160	179
1	0	2	421	347
1	0	3	477	420
1	0	4	346	265
1	0	5	1030	1113
1	0	7	206	205
1	0	8	70	78
1	0	9	41	32

H	K	L	FOBS	FCAL
1	0	10	88	87
1	0	11	258	262
1	0	12	78	55
1	1	1	134	80
1	1	2	390	331
1	1	3	606	571
1	1	4	79	104
1	1	5	321	332
1	1	6	145	176
1	1	7	283	321
1	1	8	67	63
1	1	9	429	474
1	1	10	52	33
1	1	11	227	231
1	2	1	975	890
1	2	2	741	681
1	2	3	420	376
1	2	4	190	157
1	2	5	809	795
1	2	6	187	153
1	2	7	239	230
1	2	9	82	107
1	2	10	118	127
1	2	11	220	227
1	3	0	57	16
1	3	1	695	556
1	3	3	375	352
1	3	4	328	326
1	3	5	85	91
1	3	6	263	301
1	3	7	288	310
1	3	8	101	112
1	3	9	322	313
1	3	11	158	156
1	3	12	126	128
1	4	0	701	593
1	4	1	426	374
1	4	2	568	602
1	4	3	287	298
1	4	5	361	356
1	4	6	193	247
1	4	7	172	188
1	4	8	175	184
1	4	9	113	138
1	4	10	163	186
1	4	11	129	141
1	5	1	391	321
1	5	2	247	346
1	5	3	191	193

H	K	L	FOBS	FCAL
1	5	4	300	320
1	5	5	74	122
1	5	6	236	267
1	5	7	187	204
1	5	9	173	149
1	6	0	493	468
1	6	1	217	214
1	6	1	182	214
1	6	2	211	204
1	6	3	141	133
1	6	4	95	108
1	6	5	130	91
1	6	6	157	177
1	6	7	95	94
1	6	8	141	129
1	6	9	104	69
1	6	10	211	185
1	7	2	252	266
1	7	3	83	45
1	7	4	189	213
1	7	5	125	118
1	7	6	191	201
1	8	0	259	272
1	8	1	128	128
1	8	2	48	77
1	8	4	104	111
1	8	6	84	101
2	0	1	332	342
2	0	2	617	620
2	0	3	294	233
2	0	4	38	131
2	0	5	60	57
2	0	6	575	566
2	0	7	108	129
2	0	8	492	517
2	0	9	115	140
2	0	10	350	392
2	0	11	48	71
2	0	12	102	120
2	1	0	61	9
2	1	1	594	445
2	1	2	898	858
2	1	3	154	252
2	1	4	1018	1074
2	1	5	151	156
2	1	6	578	641
2	1	7	88	122
2	1	8	139	166
2	1	9	60	59

H	K	L	FOBS	FCAL
2	1	10	88	81
2	1	11	71	64
2	1	12	244	229
2	2	0	1205	1308
2	2	1	532	467
2	2	2	782	669
2	2	3	431	346
2	2	4	250	254
2	2	5	251	220
2	2	6	372	387
2	2	7	71	80
2	2	8	409	427
2	2	9	82	89
2	2	10	305	327
2	2	11	92	96
2	2	12	124	117
2	3	0	407	154
2	3	1	312	239
2	3	1	256	239
2	3	2	524	513
2	3	3	343	292
2	3	4	694	690
2	3	5	167	192
2	3	6	422	423
2	3	7	155	186
2	3	8	91	114
2	3	9	112	117
2	3	10	94	110
2	3	11	116	119
2	3	12	209	180
2	4	0	783	661
2	4	1	454	401
2	4	2	458	461
2	4	3	360	382
2	4	4	175	172
2	4	5	281	303
2	4	6	194	242
2	4	7	88	82
2	4	8	231	278
2	4	10	201	205
2	4	11	157	141
2	5	1	454	363
2	5	2	164	230
2	5	3	218	272
2	5	4	314	364
2	5	5	130	120
2	5	6	225	234
2	5	7	165	175
2	5	9	122	117

H	K	L	FOBS	FCAL
2	5	10	102	84
2	5	11	144	112
2	6	0	151	195
2	6	1	182	182
2	6	2	135	129
2	6	3	201	228
2	6	4	90	90
2	6	5	278	279
2	6	6	127	141
2	6	7	76	68
2	6	8	123	140
2	6	10	119	99
2	7	0	210	207
2	7	1	189	215
2	7	2	101	82
2	7	3	163	156
2	7	4	180	187
2	7	5	93	72
2	7	6	111	109
2	7	7	145	142
2	8	1	95	100
2	8	2	49	62
2	8	3	107	144
2	8	5	159	172
2	8	6	59	45
3	0	1	447	369
3	0	2	310	256
3	0	3	421	444
3	0	4	398	410
3	0	5	358	380
3	0	6	224	240
3	0	7	220	221
3	0	9	99	89
3	0	10	103	101
3	0	11	251	241
3	0	12	124	95
3	1	0	166	112
3	1	2	553	476
3	1	3	364	336
3	1	4	193	183
3	1	5	97	65
3	1	6	78	109
3	1	7	378	417
3	1	8	93	111
3	1	9	197	203
3	1	10	110	119
3	1	11	144	142
3	2	0	671	582
3	2	1	181	211

H	K	L	FOBS	FCAL
3	2	2	287	214
3	2	3	548	497
3	2	4	156	198
3	2	5	470	463
3	2	6	344	329
3	2	7	194	198
3	2	8	112	98
3	2	9	62	51
3	2	10	122	125
3	2	11	194	202
3	2	12	78	70
3	3	0	141	128
3	3	1	566	541
3	3	2	284	238
3	3	3	237	194
3	3	4	319	316
3	3	5	297	322
3	3	6	234	269
3	3	7	291	307
3	3	8	73	99
3	3	9	228	236
3	3	10	100	105
3	3	11	134	116
3	3	12	107	89
3	4	0	643	532
3	4	1	311	318
3	4	2	53	34
3	4	3	334	328
3	4	4	84	98
3	4	5	305	347
3	4	6	296	321
3	4	7	140	167
3	4	8	173	210
3	4	10	138	150
3	4	11	161	152
3	5	1	235	280
3	5	2	133	182
3	5	3	122	157
3	5	4	240	269
3	5	5	152	198
3	5	6	259	279
3	5	7	122	119
3	5	9	126	143
3	5	10	108	95
3	6	0	322	308
3	6	1	287	298
3	6	2	178	190
3	6	3	119	84

H	K	L	FOBS	FCAL
3	6	4	137	153
3	6	5	113	113
3	6	6	175	196
3	6	7	113	120
3	6	8	147	162
3	6	10	166	141
3	7	1	146	140
3	7	2	95	122
3	7	3	144	144
3	7	4	207	208
3	7	5	84	99
3	7	6	166	161
3	8	0	100	174
3	8	1	87	112
3	8	2	137	167
3	8	4	123	150
3	8	6	90	86
3	8	7	63	43
3	8	8	110	112
4	0	1	711	712
4	0	2	806	781
4	0	3	235	196
4	0	6	265	275
4	0	7	237	273
4	0	8	344	361
4	0	9	132	156
4	0	10	336	361
4	0	11	121	127
4	0	12	82	65
4	1	0	336	269
4	1	1	377	292
4	1	2	620	584
4	1	3	234	240
4	1	4	824	797
4	1	5	255	274
4	1	6	503	556
4	1	7	167	183
4	1	8	74	88
4	1	9	142	165
4	1	10	100	89
4	1	11	65	80
4	1	12	207	184
4	2	0	597	519
4	2	1	599	568
4	2	2	586	511
4	2	3	111	77
4	2	4	300	264
4	2	5	192	205

H	K	L	FOBS	FCAL
4	2	6	198	218
4	2	7	262	293
4	2	8	321	324
4	2	9	122	164
4	2	10	278	294
4	2	11	142	134
4	2	12	108	83
4	3	0	277	269
4	3	1	395	349
4	3	2	441	465
4	3	3	416	388
4	3	4	417	429
4	3	5	286	329
4	3	6	378	425
4	3	7	143	160
4	3	8	81	94
4	3	9	147	186
4	3	10	131	126
4	3	11	75	70
4	4	0	669	656
4	4	1	370	346
4	4	2	247	272
4	4	3	202	130
4	4	4	352	354
4	4	5	239	268
4	4	6	191	223
4	4	7	204	216
4	4	8	172	182
4	4	9	104	100
4	4	11	141	103
4	5	0	142	116
4	5	1	258	242
4	5	2	158	168
4	5	3	217	237
4	5	4	237	247
4	5	5	204	217
4	5	6	187	201
4	5	7	125	118
4	5	9	113	124
4	5	10	88	100
4	5	11	110	86
4	6	0	245	271
4	6	1	227	251
4	6	2	113	122
4	6	3	176	171
4	6	4	166	160
4	6	5	178	194
4	6	6	132	148
4	6	7	91	92

H	K	L	FOBS	FCAL
4	6	8	80	77
4	6	10	125	104
4	7	0	116	143
4	7	1	145	163
4	7	2	98	101
4	7	3	150	121
4	7	4	138	136
4	7	7	145	109
4	7	8	126	95
4	8	0	62	70
4	8	1	100	103
4	8	3	110	133
4	8	5	102	117
4	8	6	70	41
4	8	8	82	49
5	0	1	287	198
5	0	2	816	735
5	0	3	566	591
5	0	4	334	309
5	0	5	330	350
5	0	6	280	289
5	0	7	151	137
5	0	8	37	79
5	0	10	61	67
5	0	11	140	149
5	0	12	84	77
5	1	0	547	469
5	1	1	594	593
5	1	2	108	81
5	1	3	104	43
5	1	4	259	216
5	1	5	208	147
5	1	6	161	171
5	1	7	351	406
5	1	8	101	104
5	1	9	209	201
5	1	10	152	182
5	1	11	117	121
5	2	0	201	202
5	2	1	255	190
5	2	2	550	500
5	2	3	451	452
5	2	4	308	315
5	2	5	357	339
5	2	6	222	238
5	2	7	87	95
5	2	8	85	91
5	2	10	96	97
5	2	11	175	170

H	K	L	FOBS	FCAL
5	2	12	106	87
5	3	0	131	168
5	3	1	387	386
5	3	2	363	341
5	3	3	136	119
5	3	4	299	296
5	3	5	182	201
5	3	6	121	136
5	3	7	174	206
5	3	9	168	174
5	3	10	140	151
5	3	11	103	72
5	4	0	435	416
5	4	1	331	265
5	4	2	136	113
5	4	3	229	224
5	4	4	217	262
5	4	5	280	271
5	4	6	82	85
5	4	7	101	115
5	4	8	80	91
5	4	9	123	113
5	4	11	158	136
5	5	0	83	18
5	5	1	177	155
5	5	2	221	193
5	5	3	118	121
5	5	4	212	228
5	5	5	206	236
5	5	6	143	127
5	5	7	120	119
5	5	9	149	134
5	5	10	118	104
5	6	0	211	237
5	6	1	159	163
5	6	2	167	173
5	6	3	112	118
5	6	4	146	142
5	6	5	157	162
5	6	7	101	109
5	6	8	88	99
5	6	9	126	114
5	7	2	83	65
5	7	3	142	132
5	7	4	185	176
5	7	5	216	194
5	7	6	121	120
5	8	0	97	100

H	K	L	FOBS	FCAL
5	8	1	44	56
5	8	3	76	60
5	8	5	70	52
5	8	7	71	69
5	8	8	100	101
6	0	0	548	568
6	0	1	427	437
6	0	2	705	653
6	0	3	485	416
6	0	4	170	130
6	0	6	76	81
6	0	7	134	189
6	0	8	134	129
6	0	9	181	184
6	0	10	158	159
6	0	11	138	150
6	1	0	63	6
6	1	1	225	167
6	1	2	331	338
6	1	3	89	171
6	1	4	237	242
6	1	5	450	492
6	1	6	327	377
6	1	7	127	168
6	1	8	64	79
6	1	9	111	148
6	1	10	849	732
6	1	11	149	160
6	2	0	444	439
6	2	1	522	490
6	2	2	494	493
6	2	3	257	247
6	2	4	234	204
6	2	5	157	185
6	2	6	191	187
6	2	7	145	208
6	2	8	128	137
6	2	9	158	157
6	2	10	150	163
6	2	11	103	125
6	3	0	165	145
6	3	1	280	246
6	3	2	226	249
6	3	3	163	158
6	3	4	254	258
6	3	5	322	348
6	3	6	281	304
6	3	7	213	209

H	K	L	FOBS	FCAL
6	3	8	118	118
6	3	9	94	105
6	3	10	73	72
6	3	11	97	100
6	4	0	375	320
6	4	1	335	310
6	4	2	266	282
6	4	3	83	88
6	4	4	204	221
6	4	5	137	159
6	4	6	192	194
6	4	7	176	179
6	4	8	99	91
6	4	9	106	102
6	4	10	150	131
6	5	0	324	322
6	5	1	174	138
6	5	2	185	149
6	5	3	144	127
6	5	4	144	146
6	5	5	231	231
6	5	6	162	161
6	5	7	181	163
6	5	8	101	87
6	5	9	116	104
6	6	0	237	227
6	6	1	96	106
6	6	2	179	192
6	6	3	58	73
6	6	4	93	100
6	6	5	85	78
6	6	6	91	91
6	6	7	85	60
6	7	1	71	88
6	8	0	72	75
6	8	2	77	97
6	8	6	70	48
7	0	1	85	84
7	0	2	331	293
7	0	3	393	372
7	0	4	148	129
7	0	5	288	272
7	0	6	195	217
7	0	11	173	150
7	1	0	563	529
7	1	1	240	228
7	1	2	75	123
7	1	3	57	83

H	K	L	FOBS	FCAL
7	1	5	54	57
7	1	6	286	308
7	1	7	225	245
7	1	8	110	111
7	1	9	143	149
7	1	10	154	169
7	1	11	67	63
7	2	0	39	78
7	2	1	155	190
7	2	2	198	235
7	2	3	344	340
7	2	4	169	168
7	2	5	206	215
7	2	6	163	183
7	2	7	51	36
7	2	10	51	38
7	2	11	143	126
7	3	0	117	142
7	3	1	200	199
7	3	2	104	133
7	3	3	91	74
7	3	4	153	169
7	3	5	178	186
7	3	6	172	193
7	3	7	189	210
7	3	8	95	92
7	3	9	102	106
7	3	10	133	123
7	3	11	72	50
7	4	1	130	147
7	4	2	131	123
7	4	3	189	207
7	4	4	96	101
7	4	5	100	119
7	4	9	123	119
7	5	1	149	147
7	5	2	136	135
7	5	3	70	63
7	5	4	189	171
7	5	5	264	236
7	5	6	92	70
7	5	7	132	135
7	6	0	95	115
7	6	1	144	133
7	6	3	165	170
7	6	6	73	55
7	6	7	90	65
7	6	9	135	105

H	K	L	FOBS	FCAL
7	7	0	72	12
7	7	5	91	150
7	8	0	126	125
7	8	1	136	132
7	8	3	120	121
8	0	0	261	288
8	0	1	298	314
8	0	3	123	153
8	0	5	173	182
8	0	6	99	144
8	0	7	166	188
8	0	8	82	70
8	0	9	136	122
8	0	10	109	105
8	0	11	206	184
8	1	0	135	100
8	1	1	214	207
8	1	2	245	272
8	1	3	354	376
8	1	5	326	370
8	1	6	142	133
8	1	7	133	157
8	1	11	129	117
8	2	0	135	173
8	2	1	264	296
8	2	2	141	142
8	2	3	208	203
8	2	4	79	79
8	2	5	141	147
8	2	6	118	125
8	2	7	136	160
8	2	8	54	45
8	2	9	93	99
8	2	10	109	96
8	2	11	166	156
8	3	0	231	230
8	3	1	109	109
8	3	2	186	187
8	3	3	263	274
8	3	4	124	152
8	3	5	212	245
8	3	6	122	117
8	3	7	130	148
8	3	8	101	94
8	3	10	57	43
8	4	1	206	213
8	4	2	218	214

H	K	L	FOBS	FCAL
8	4	3	164	174
8	4	4	147	180
8	4	6	96	89
8	4	7	84	77
8	4	9	100	88
8	4	10	96	70
8	5	0	222	198
8	5	1	144	115
8	5	2	149	135
8	5	3	140	137
8	5	4	79	81
8	5	6	137	128
8	5	7	81	76
8	5	8	113	71
8	6	1	104	113
8	6	2	123	131
8	6	3	74	106
8	6	4	156	163
8	6	6	95	63
8	6	8	86	55
8	8	2	94	99
8	8	4	100	63
8	8	5	64	56
9	0	1	98	98
9	0	3	45	45
9	0	4	136	129
9	0	5	180	179
9	0	6	135	139
9	1	0	226	241
9	1	1	192	203
9	1	2	228	228
9	1	3	143	145
9	1	4	41	40
9	1	5	99	106
9	1	6	115	134
9	1	7	79	77
9	1	8	107	107
9	1	9	82	82
9	1	10	105	94
9	2	0	60	54
9	2	2	95	126
9	2	4	90	108
9	2	5	114	141
9	2	6	109	130
9	2	7	54	68
9	2	9	62	72
9	3	0	197	224

H	K	L	FOBS	FCAL
9	3	1	153	168
9	3	2	106	96
9	3	3	129	129
9	3	4	43	52
9	3	5	155	163
9	3	6	48	73
9	3	7	89	104
9	3	8	97	96
9	3	9	55	47
9	3	10	65	55
9	4	1	140	126
9	4	2	78	87
9	4	3	93	110
9	4	4	80	118
9	4	6	116	84
9	5	0	74	55
9	5	1	135	131
9	5	3	85	90
9	5	5	152	143
9	5	8	96	57
9	6	1	170	169
9	6	3	91	111
9	6	4	88	85
9	8	0	93	74
9	8	1	111	108
9	8	3	72	69
10	0	0	106	120
10	0	1	245	262
10	0	2	55	51
10	0	3	54	65
10	0	4	65	86
10	0	5	58	74
10	0	6	64	44
10	0	7	130	134
10	0	9	108	107
10	1	0	121	111
10	1	1	199	197
10	1	2	58	65
10	1	3	194	204
10	1	4	106	133
10	1	5	184	224
10	1	7	112	125
10	1	10	59	48
10	2	0	61	70
10	2	1	146	217
10	2	3	110	127
10	2	5	97	113
10	2	6	50	44

H	K	L	FOBS	FCAL
10	2	7	89	105
10	2	9	104	106
10	3	0	180	189
10	3	1	114	116
10	3	2	91	99
10	3	3	113	138
10	3	4	102	102
10	3	5	140	170
10	3	7	77	92
10	3	9	67	51
10	4	1	131	162
10	4	3	136	138
10	4	5	87	79
10	4	6	104	82
10	5	0	82	96
10	5	1	86	78
10	5	2	91	82
10	5	3	82	68
10	5	6	124	79
10	6	1	77	101
10	6	2	68	82
10	6	6	92	67
10	8	0	64	63
11	0	1	106	115
11	0	3	73	50
11	0	4	163	167
11	0	5	47	50
11	0	9	66	40
11	1	0	126	122
11	1	1	68	42
11	1	3	46	37
11	1	4	90	100
11	1	5	69	69
11	1	8	103	93
11	2	0	55	73
11	2	1	43	80
11	2	3	43	64
11	2	4	138	154
11	2	5	46	43
11	2	7	59	57
11	2	8	49	21
11	2	9	63	33
11	3	0	94	91
11	3	1	73	65
11	3	2	46	36
11	3	3	75	105
11	3	4	96	92
11	3	5	93	91

H	K	L	FOBS	FCAL
11	3	6	50	46
11	3	8	95	77
11	4	1	83	80
11	4	3	71	73
11	4	4	125	97
11	4	7	95	79
11	5	3	133	121
11	5	5	113	84
11	6	1	88	83
12	0	0	75	32
12	0	1	166	182
12	0	2	86	55
12	0	3	131	145
12	0	4	60	41
12	0	5	110	98
12	0	7	56	22
12	0	9	101	95
12	1	1	93	108
12	1	2	82	92
12	1	3	66	56
12	1	4	46	43
12	1	5	12	13
12	1	7	74	72
12	2	1	13	18
12	2	2	44	42
12	2	3	95	131
12	3	1	84	88
12	3	4	52	42
12	3	5	73	98
12	3	6	65	41
12	3	7	52	67
12	4	1	89	115
12	4	3	86	69
12	4	6	87	79
12	5	3	91	52
12	5	5	90	65
13	0	6	53	4
13	1	0	56	98
13	1	2	62	60
13	1	5	44	37
13	1	6	58	69
13	1	8	63	59
13	2	1	73	83
13	2	3	72	104
13	4	1	111	92
13	4	4	87	44
14	1	1	55	52
14	1	3	50	41

H	K	L	FOBS	FCAL
14	1	4	44	57
14	1	5	76	82
14	2	1	55	60
14	3	5	51	74
14	4	0	79	30
15	1	6	54	38
15	3	0	65	61
15	3	1	50	43
16	2	4	59	26

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