

The Application of the Structural Correlation  
Method to Determine the Reaction  
Coordinate for a Putative  
Ring Closure Reaction.

by

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To my mother and late father

Abstract

The crystal structure of the compound 2-benzoylethyldene-3-(2,4-dibromophenyl)-2,3-dihydro-5-phenyl-1,3,4-thiadiazole<sup>\*</sup> C<sub>23</sub>H<sub>16</sub>Br<sub>2</sub>N<sub>2</sub>OS (BRMEO) has been determined by using three dimensional x-ray diffraction data. The crystal form is monoclinic, space group P2<sub>1</sub>/c, a = 17.492(4), b = 16.979(1), c = 14.962(1) Å,  $\alpha = \gamma = 90^\circ$ ,  $\beta = 106.46(1)^\circ$ , z = 8, graphite-monochromatized MoK<sub>α</sub> radiation,  $\lambda = 0.71073\text{\AA}$ , D<sub>o</sub> = 1.62g/cc and D<sub>c</sub> = 1.65g/cc. The data were collected on an Nonius CAD-4 diffractometer. The following atoms were made anisotropic: Br, S, N, O, C7, and C14-C16 for each independent molecule; the rest were left isotropic. For 3112 independent reflections with F > 6σ(F), R = 0.057.

The compound has two independent molecules within the asymmetric unit. Two different conformers were observed which pack well together. The S---O interaction distances of 2.493(6) and 2.478(7) Å were observed for molecules A and B respectively. These values are consistent with earlier findings for 2-benzoylmethylene-3-(2,4-dibromophenyl)-2,3-dihydro-5-phenyl-1,3,4-thiadiazole<sup>\*</sup> C<sub>22</sub>H<sub>14</sub>Br<sub>2</sub>N<sub>2</sub>OS (BRPHO) and 2-benzoylpropylidene-3-(2,4-dibromophenyl)-2,3-dihydro-5-phenyl-1,3,4-thiadiazole<sup>\*</sup> C<sub>24</sub>H<sub>18</sub>Br<sub>2</sub>N<sub>2</sub>OS (BRPETO) where S---O distances are less than the van der Waals (3.25Å) but greater than those expected for a single bond (1.50Å). From the results and the literature it appears obvious that the energy/reaction coordinate pathway has a minimum between the end structures (the mono- and bicyclic compounds).

\* See reference (27) for nomenclature.

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## I. Introduction

### A. The Structural Correlation Method

Traditional methods of studying chemical reaction mechanisms have relied on the measurement of reaction rates and the effects of temperature, concentration, solvents etc, on these rates, while the structural aspect has been neglected.

Spectroscopic techniques such as Raman, infra-red, and especially microwave give information regarding the shapes of simple molecules, but are of little help when more complex systems are at hand. Up to now chemists have generally made use of structural information about simple molecules to explain structural features observed in more complex systems. One of the best ways one can provide unambiguous information regarding the arrangement of atoms in molecules is by x-ray crystallography. Currently many thousands of crystal structures are being solved and their data tabulated.

If one considers a simple reaction :



where the structures of A and B are known, it would be useful to follow the structural changes that take place during the reaction. The task of following such changes is beyond any experimental capability. This is exemplified by the great time differences between the very short reaction transformation times ( $10^{-12}$  sec) and the time required to collect crystallographic or spectroscopic data by ordinary techniques.

The structural correlation method<sup>1-5</sup> has been developed so that these structural changes may be followed indirectly. The method makes use of some rather interesting features that have been observed recently. Many cases are known where interatomic distances are longer than the normal single bond, but shorter than the sum of the van der Waals radii<sup>6</sup>. On his review on donor-acceptor interactions<sup>7</sup> Bent commented " Certain kinds of attractive intermolecular interactions may be viewed as incipient valence shell expansions and often as the first stage of bimolecular nucleophilic displacement reactions ". This idea is applicable, of course, to all kinds of reactions, not just bimolecular nucleophilic displacement reactions.

Following are a few examples of structures which exhibit intermolecular contacts shorter than van der Waals.

(a) The donor-acceptor molecular compound formed between trimethylamine and iodine,  $(\text{CH}_3)_3\text{N} \cdots \text{I} \cdots \text{I}$ . The I--I distance is 2.83 Å, about 0.15 Å longer than in gaseous iodine, the N---I--I grouping is linear and the N---I distance is 2.3 Å, about 0.25 Å longer than the value expected for a covalent bond (ca.  $0.7 + 1.33 = 2.03$ ) but much less than the sum of the van

der Waals radii ( $1.5+2.15=3.65$ )<sup>3</sup> (figure 1).

(b) The formation of a distorted  ${}^*\text{Cl}_3\text{SbCl}_3$  octahedron in crystalline compounds containing  $\text{SbCl}_3$ . In some crystalline compounds containing  $\text{SbCl}_3$ , the ligand sphere of antimony is completed by three additional chloride ions  ${}^*\text{Cl}$  to yield  ${}^*\text{Cl}_3\text{SbCl}_3$  which takes the shape of a distorted octahedron<sup>8</sup>. Whereas the Sb-Cl bond distances varied from  $2.351(10)$ - $2.404(9)\text{\AA}$ , the three bridging Sb---Cl interactions are  $3.025(9)$ - $3.307(9)\text{\AA}$  (figure 2).

(c) i. The S--S distance in 6a-thiathiophthene derivatives (X=S).

The S--S distance is  $0.5\text{\AA}$  longer than the covalent bond in dimethyldisulphane ( $2.023\text{\AA}$ ) and about  $1.15\text{\AA}$  shorter than the van der Waals contact ( $3.7\text{\AA}$ )<sup>3</sup> (figure 3).

(c) ii. The S--S distance in dithiafurophthenes (X=O).

Here the S--S distance is  $2.1\text{\AA}$ . The O---S distance lies between  $2.3$ - $2.4\text{\AA}$ , which is clearly less than the sum of the van der Waals radii ( $3.25\text{\AA}$ ), though longer than the length of an S--O single bond ( $1.5\text{\AA}$ )<sup>3</sup> (figure 4).

Basically the structural correlation method has been put forward to bridge the gap that exists between the statics of crystal structures and the dynamics of reaction transformations. While the statics part is known well as a result of the many structures that are solved and stored in data bases each year, the dynamics part requires greater scrutiny. In order to discuss dynamics the concept of potential energy surfaces is highly desirable at this stage.

Figure 1: Donor-acceptor molecular compound formed between trimethylamine and iodine.



Figure 2: The distorted octahedron formed by  $\text{SbCl}_3$  in the crystalline state.

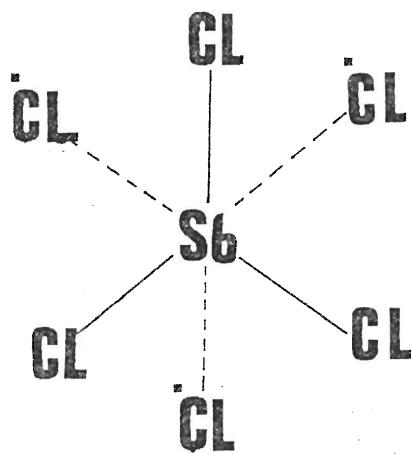


Figure 3: The S---S distance in 6a-thiathiophthene derivative: (X=S).

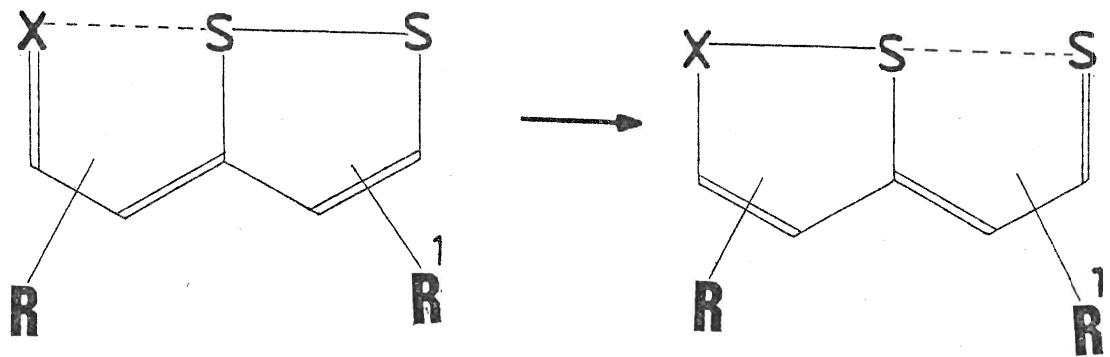
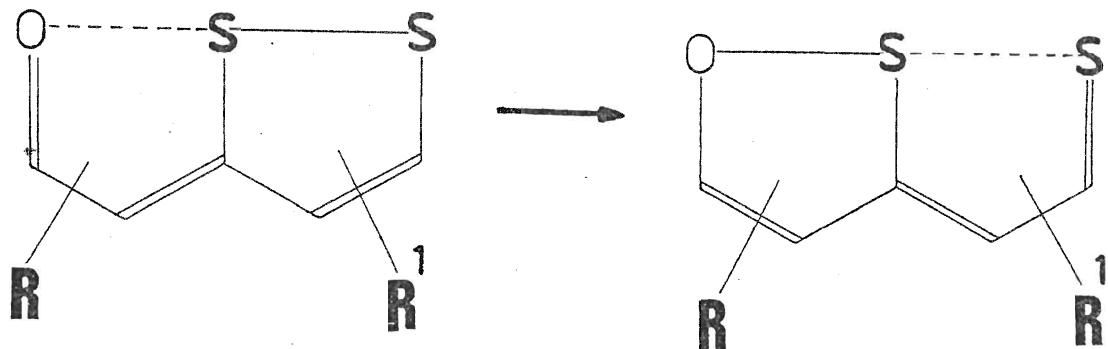
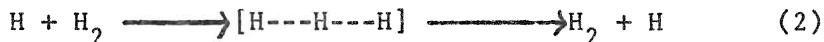


Figure 4: The S---S distance in 6a-dithiafuroporphthenes.



For the hypothetical reaction outlined in equation(1), it is possible to represent the reaction in the form of a potential energy diagram. This particular diagram illustrates how the energy of a system varies along the reaction coordinate pathway (see figure 5). The point of maximum energy is called the transition state (also known as the col). Although this point represents high energy it is useful to think of it as the minimum amount of energy needed for the reaction to take place.

A more detailed way of looking at reaction pathways is shown in figure 6. The potential energy surface shown here is for the simplest of all systems, the hydrogen system:



It was calculated by Liu<sup>9</sup>. This diagram shows how the energy of the system is related to the configuration of the atoms, i.e. to the distances (the H---H---H angle was assumed to be 180°, but of course the energy will vary with the angle as well). Each solid line represents a constant energy contour and each point on the surface portrays a particular arrangement of the 3-dimensional system. The dashed line is the reaction coordinate and it indicates the path of least resistance that an isolated species would follow. In real life we have to contend with many molecules. However, it is assumed that they will all follow paths that are close to the dashed line. A hyperbolic function best

Figure 5: The Energy/Reaction coordinate profile for a hypothetical case.

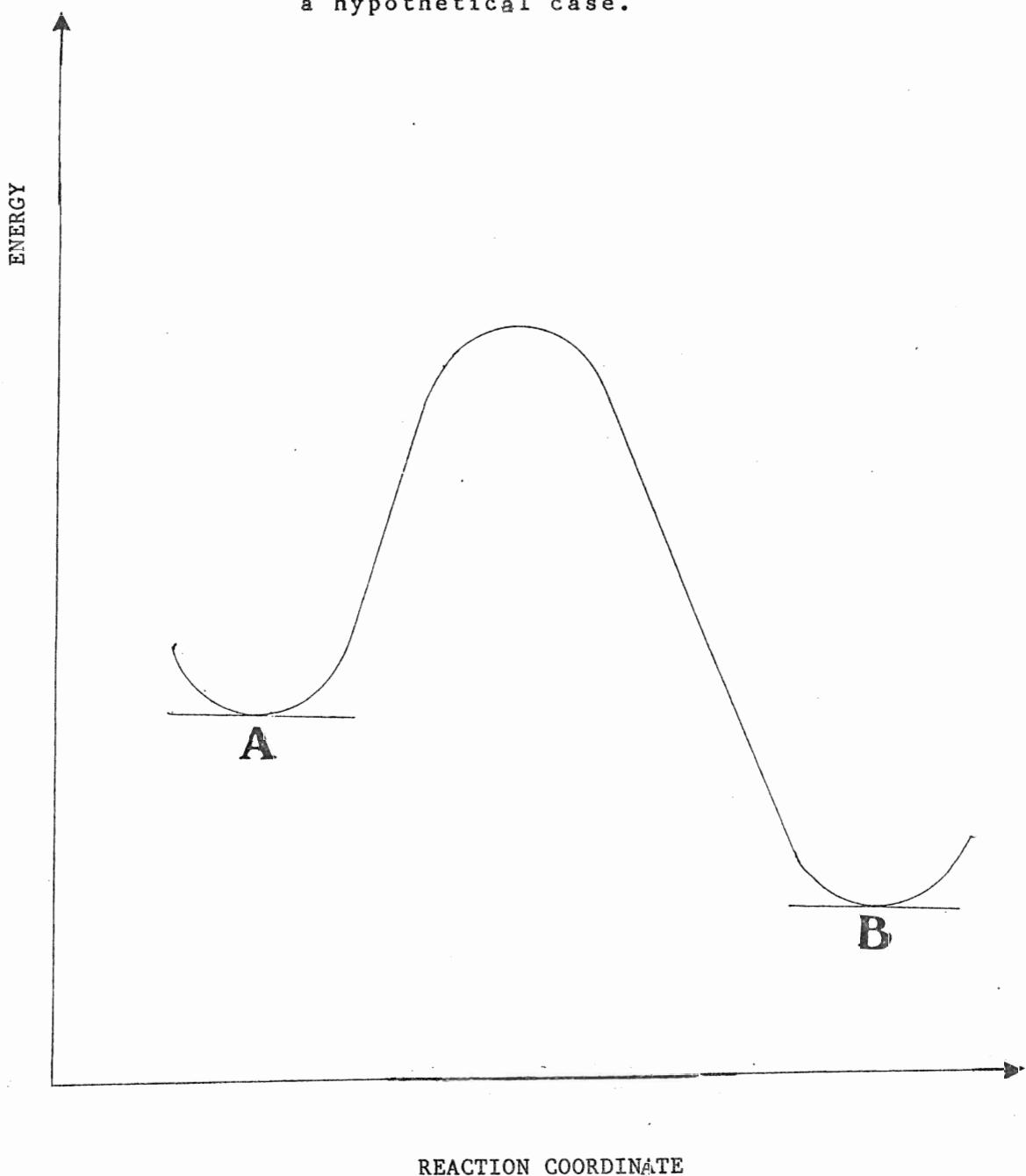
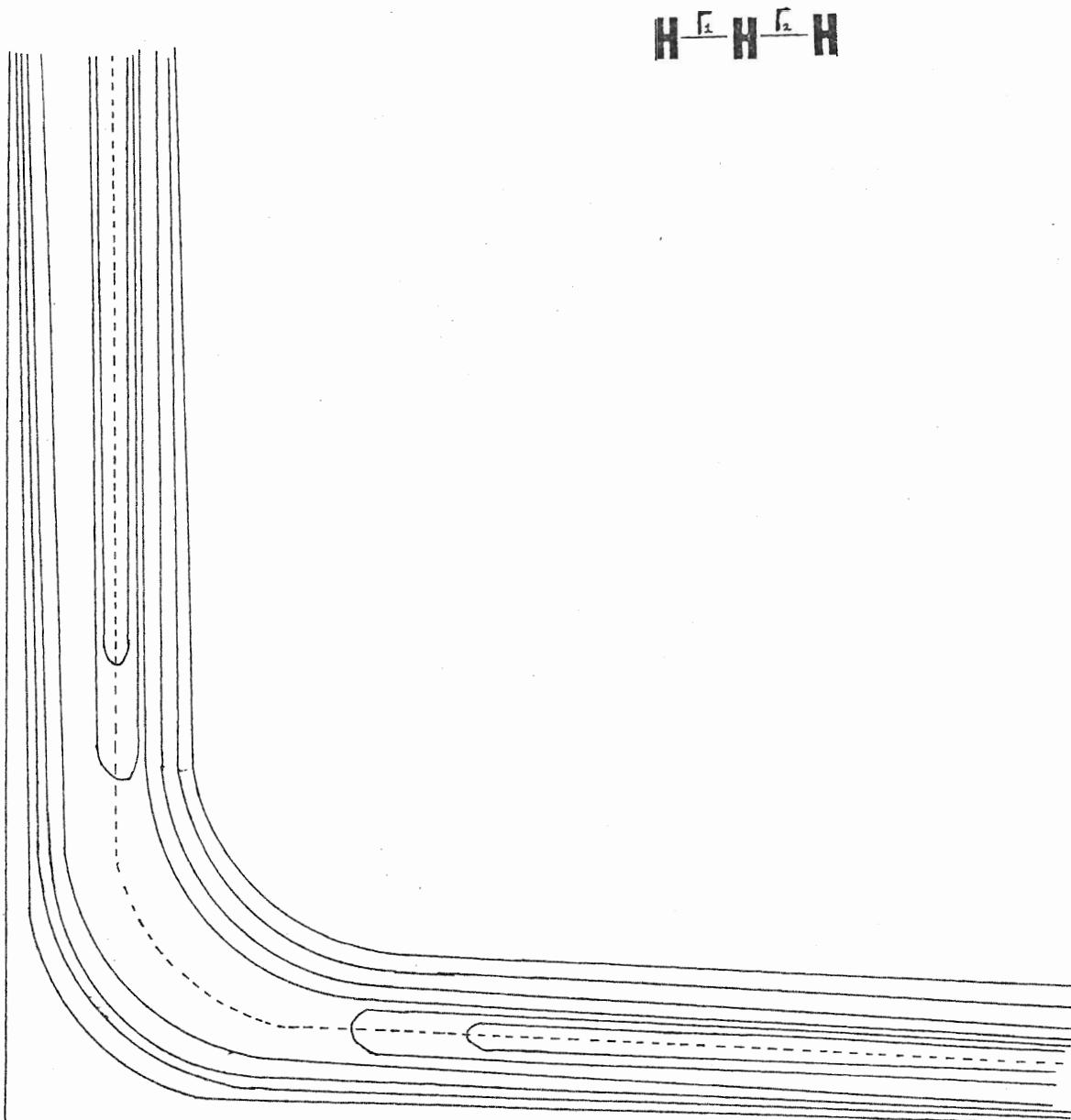


Figure 6: A schematic look at the potential energy surface for the H<sub>3</sub> system.



describes this pathway. In practice these calculations are difficult, if not impossible for all but the simplest systems because of the lengthy nature of the calculations involved.

Crystallography can measure the distances and angles, but not energies. We can infer relative energies of configurations of a given fragment from the relative frequency of occurrence of that fragment in a group of related structures. We can assume a Boltzmann-type distribution:

$$\text{frequency of occurrence} \propto e^{-\Delta E/RT} \quad (3)$$

where  $\Delta E$  is the energy above the lowest energy configurations, and R and T are the gas constant and absolute temperature, respectively. Thus the more frequently a configuration occurs, the lower the energy of the configuration\*.

As a result of his work on bonding in metals, Pauling proposed the following equation that relates the fractional bond formed in these systems, to the length of a single bond:

$$\Delta d = d(n) - d(1) = -c \log [n] \quad (4)$$

\* This relationship of frequency of occurrence to the energy has not been stated before, but it seems obvious as a crude guide to relative energies.

where  $d(n)$ =length of fractional bond,  $d(1)$ =length of single bond,  $c$ =constant (dependent on type of bond)  $c = \Delta d' / \log [2]$ ,  $\Delta d'$  is the bond distance increment in the symmetrical structure [ $\Delta d' = d(0.5) - d(1)$ ], and  $n$ =bond order.

The bond order should not be confused with the bond order of molecular orbital theory since it does not have any theoretical significance. Pauling defined the bond order as follows "n is the number of shared electron pairs involved in the bond. The logarithmic relation is of course, to be expected in consequence of the exponential character of interatomic forces".<sup>10</sup> One can derive the above equation by considering the properties of the Morse function which is a good approximation to the potential energy curves of diatomic molecules,

$$V(R-R_0) = D[1 - \exp(-b[R-R_0])]^2 \quad (5)$$

where  $D$  is the binding energy,  $R_0$  is the equilibrium distance,  $b$  is the Morse constant and  $V(R-R_0)$  is the potential energy at distance  $R$ .

Equation (4) when used in conjunction with the constraint that the sum of the two bond numbers equals unity yields the following equation:

$$10^{-\Delta d_1/c} + 10^{-\Delta d_2/c} = 1 \quad (6)$$

where  $\Delta d_1$  and  $\Delta d_2$  are the relative changes in  $d_1$  and  $d_2$ . When the assumption was first made by Johnson as a result of his work on

gas-phase reactions, he commented " the assumption cannot be purely correct, but it cannot be totally wrong either, and it provides a definite starting point for the discussion ".<sup>11</sup> This type of equation is characteristic of a hyperbolic function and suggests that the dashed curve in figure 6 may be represented by this equation. We therefore calculated the sum of the bond orders in the H<sub>3</sub> system, using data from Liu<sup>9</sup>. As shown in table 1, the sum of the bond numbers does indeed lie between 1.00 and 1.02 along the calculated minimum energy pathway. Thus, detailed quantum mechanical calculations support Johnson's ideas on conservation of bond numbers.

It should be noted that if one considers the formation of an ABC complex, the decrease in the length of the longer intermolecular interaction A---B from the value expected for a normal van der Waals contact is generally much greater than the accompanying increase in the length of the shorter, intramolecular interaction B-C.<sup>2</sup> This can be seen from the data in table 1.

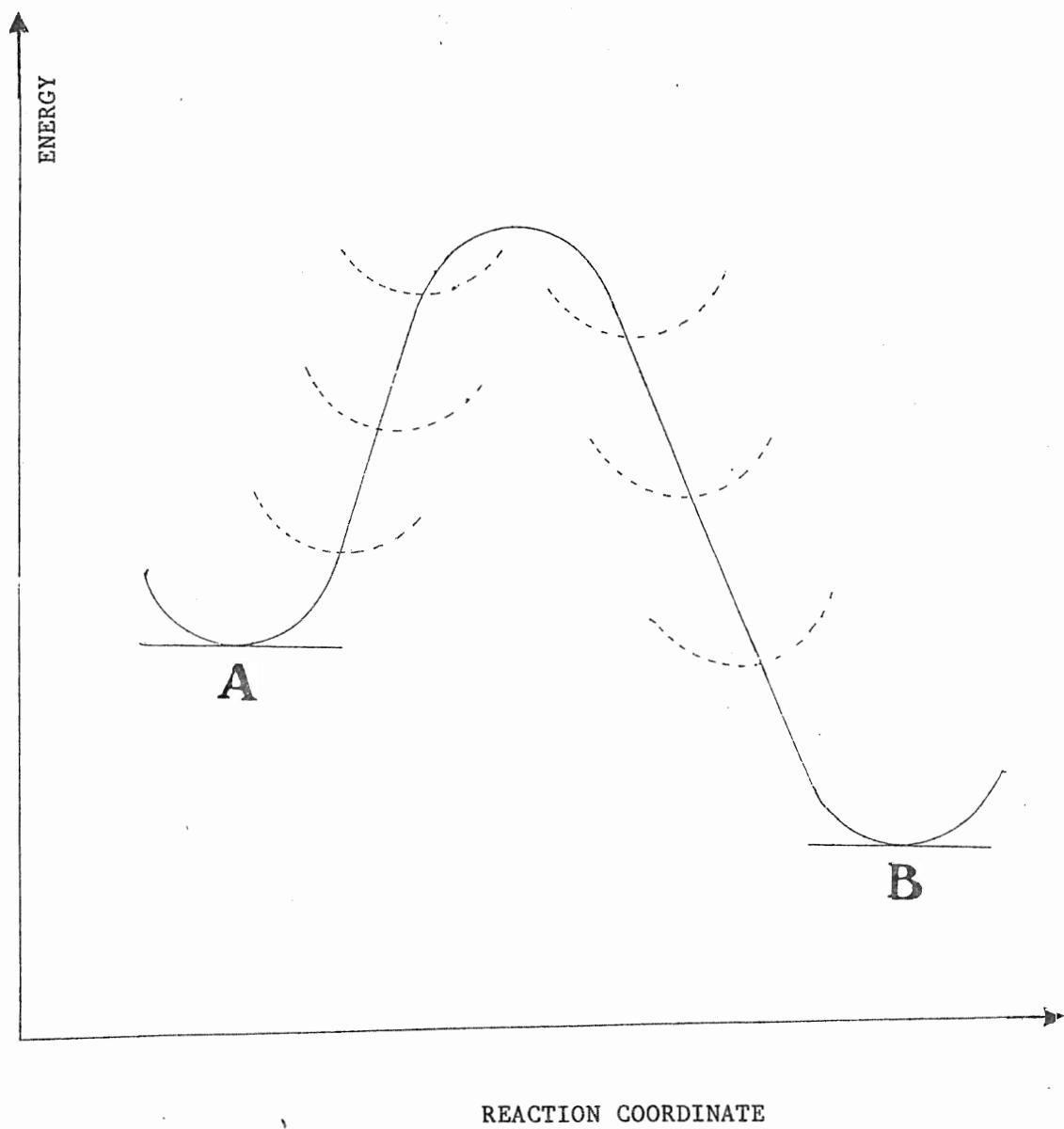
The structural correlation method can best be explained by considering the reaction coordinate/energy diagram shown in figure 7. The reactants and products are assumed to lie at the bottom of the energy curve. Therfore everything else that is above these low energy points represents unstable intermediates. The method stipulates that if one can obtain crystal structures that correspond to these higher-energy points, then these "snapshots" as they are called, when viewed in a synchronised fashion will portray an animated version of the reaction

Table 1. Sum of Bond Numbers Along Calculated Minimum Energy

Pathway for the H<sub>3</sub> System.

R(1)A	R(2)A	Δ d(1)	Δd(2)	n(1)	n(2)	n(1)+n(2)
0.93	0.93	0.19	0.19	0.50	0.50	1
0.96	0.897	0.22	0.16	0.44	0.55	0.99
1.00	0.87	0.26	0.13	0.38	0.62	1
1.05	0.84	0.31	0.10	0.32	0.69	1.01
1.10	0.82	0.36	0.08	0.26	0.74	1.00
1.15	0.80	0.41	0.06	0.22	0.80	1.02
1.20	0.79	0.46	0.05	0.18	0.83	1.01
1.25	0.78	0.51	0.04	0.15	0.86	1.01
1.31	0.77	0.57	0.03	0.12	0.89	1.01
1.36	0.766	0.62	0.03	0.10	0.91	1.01
1.41	0.7612	0.67	0.02	0.08	0.93	1.01
1.46	0.757	0.72	0.02	0.07	0.94	1.01
1.52	0.753	0.78	0.01	0.06	0.96	1.02
1.57	0.7517	0.83	0.01	0.05	0.96	1.01
1.62	0.7496	0.88	0.01	0.04	0.97	1.01
1.68	0.7484	0.94	0.01	0.03	0.97	1.00
1.73	0.746	0.99	0.01	0.03	0.98	1.01
1.78	0.7459	1.04	0.0049	0.02	0.98	1.00
1.84	0.7448	1.10	0.0038	0.02	0.99	1.01
1.89	0.7432	1.15	0.0022	0.01	0.99	1.00

Figure 7: Displacement of equilibrium structure A along reaction coordinate towards B.



pathway. These points represent unstable intermediates, compared to the reactants and products. But as indicated by the dashed curves, it is possible to regard a given system to be stable within its crystalline environment.

The method makes the assumption that although the structure of the molecule in the crystalline environment will not necessarily be the same as the equilibrium structure of the isolated molecule, with respect to the total potential energy of the system the crystal packing energy will play a minor role. Thus, changes that are observed in a given fragment as one goes from structure to structure must provide information about easily deformable coordinates.

In principle there is no reason why one cannot obtain all possible points along the reaction coordinate pathway. However in practice it is not possible to freeze the system at will. One can perturb the system by inflicting on it various strain and steric effects, thereby forcing it to slowly move along the potential curve.

It seems most appropriate to outline some specific cases where the structural correlation method has been applied.

## B. Applications

### 1. Iodine Case

Generally interhalogen compounds have been regarded to have strong intermolecular interactions in their solid phase. This has been shown to be particularly true for compounds that contain iodine. Iodine monochloride crystallizes in two polymorphs where both kinds of atoms are observed to participate in short intermolecular interactions. Although there exist some differences in the structures of the polymorphs, both are quite similar. Whereas the intramolecular distances are longer compared with the internuclear distances observed in gaseous IC<sub>l</sub>, the intermolecular contacts have been found to be 1Å or so shorter than the expected van der Waals distance.

The triiodide system is a classic example, where an iodide ion and a neutral iodine molecule participate in significant donor-acceptor interactions. The triiodide species has been observed to exist essentially in a linear unsymmetrical configuration embedded in a crystalline environment of small cations such as NH<sub>4</sub><sup>+</sup> and Cs<sup>+</sup>. The symmetrical form, where intramolecular and intermolecular distances are equal, is seen when a larger cation like (C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>As<sup>+</sup> is present. This system is able to further its interactions with iodine molecules to form I<sub>5</sub><sup>-</sup>, I<sub>7</sub><sup>-</sup> and I<sub>8</sub><sup>2-</sup> ions.

Table 2 presents the various compounds in which the triiodide

TABLE 2. Interatomic distances [ $\text{\AA}$ ] in polyiodide system,  $\text{I}_3^-$ 

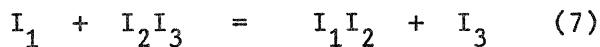
COMPOUND	$d_1(\text{I--I})$	$d_2(\text{I--I})$	$\sigma(d)$
$\text{I}_2(\text{g})$	2.67	4.30	-
$\text{I}_2$	2.70	3.54	-
$[(\text{C}_2\text{H}_5)_4\text{N}]\text{I}_7$	2.735	3.435	0.003
$(\text{NH}_4)\text{I}_3$	2.791	3.113	0.004
$[(\text{CH}_3)_4\text{N}]\text{I}_5$	2.81	3.17	0.015
$\text{Cs}_2\text{I}_8$	2.84	3.00	0.03
$\text{CsI}_3$	2.842	3.038	0.002
$[(\text{C}_5\text{H}_5)_2\text{Fe}]\text{I}_3$	2.85	2.97	0.02
	2.89	2.97	0.02
$[(\text{C}_2\text{H}_5)_4\text{N}]\text{I}_7$	2.892	2.981	0.004
	2.912	2.961	0.004
$[(\text{C}_2\text{H}_5)_4\text{N}]\text{I}_7$	2.904	2.904	0.003
$[(\text{C}_6\text{H}_5)_4\text{As}]\text{I}_3$	2.919	2.919	0.001
$[(\text{C}_5\text{H}_5)_2\text{Fe}]\text{I}_3$	2.93	2.93	0.02
$[(\text{C}_2\text{H}_5)_4\text{N}]\text{I}_3$	2.928	2.928	0.003
	2.943	2.943	0.003

Data taken from reference (3) and references cited therein.

$\sigma(d)$  = standard deviation on d.

fragment was observed, where  $d(1)$  represents the distance between I(1) and I(2) and similarly  $d(2)$  represents the distance between I(2) and I(3). When these values were subsequently used to draw figure 8, a hyperbolic curve as already seen for the hydrogen case is observed.

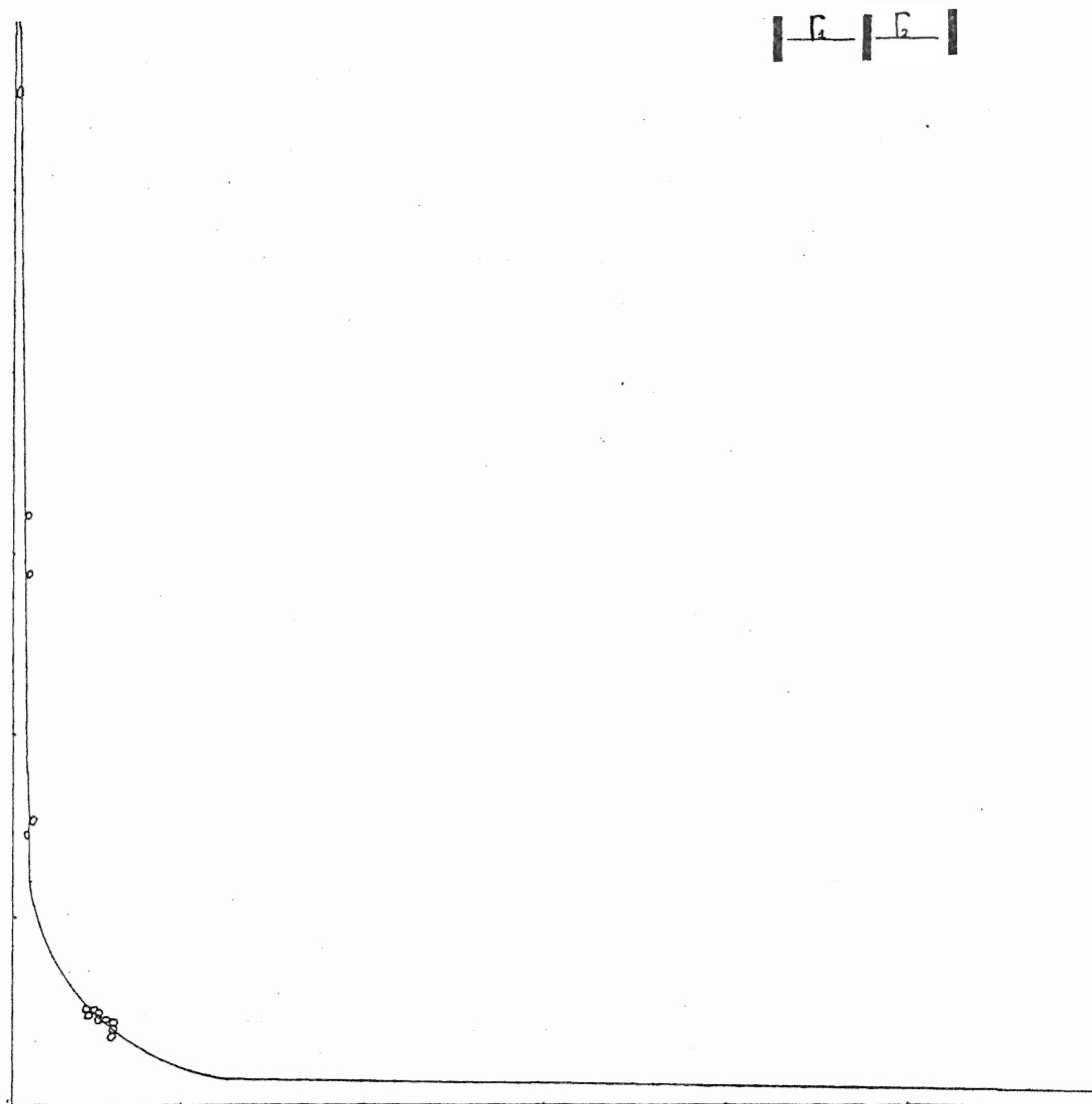
Bent<sup>7</sup> viewed this curve as representing the changes that occur in the distances between nearest neighbors in the following linear exchange reaction:



Since this system has been found to react through a linear intermediate, as shown by the essentially linear values of the fragment in its various environments ( $175-180^\circ$ ), one can regard this pathway to be of lowest resistance. This is conceptually understandable if one gives some thought about the hybridization of the iodine molecule.

Since it is  $sp^3$  hybridized, the only way of maximizing the interaction between the iodine molecule and the iodide ion (also  $sp^3$  hybridized) is if the reaction occurs in an end-on fashion. This way the lone pair of electrons on the iodide ion can be donated into the empty antibonding orbital or a d-orbital of the iodine molecule. All other angles would not facilitate maximum bonding and hence can be regarded as higher energy pathways.

Figure 8: Scatter plot of interatomic distances for  $I_3^-$  system.



5

## 2. Ligand Exchange at Tetrahedral Cadmium

Burgi was the first person to put forward a clear program of how one can go about deriving chemical reaction coordinates from crystal structure data<sup>12</sup>. A tetrahedral cadmium complex was to be used to mimic the reaction pathway of an bimolecular nucleophilic substitution reaction ( $S_N^2$ ). It was based on the reaction outlined in figure 9 for the following cases:

X=Y=I (five cases)

X=Y=S (one case)

X=S, Y=O (three cases)

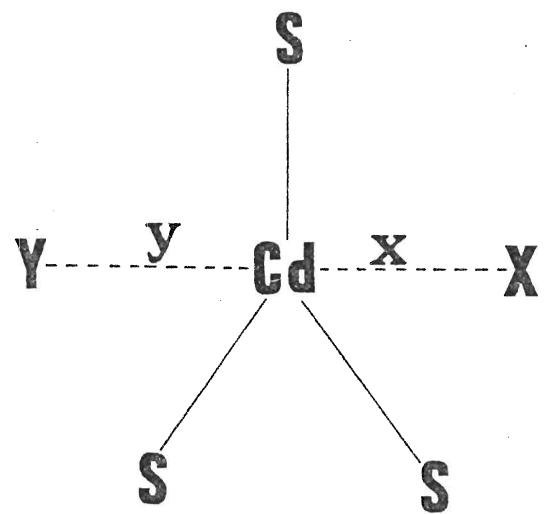
X=I, Y=O (two cases)

Apart from following the usual changes in x with respect to y, a third variable  $\Delta z$  was introduced. This was used to measure the deviation of Cd from the plane defined by the three equatorial sulphurs. If one plots  $\Delta x$  against  $\Delta y$  the hyperbolic curve observed earlier is again seen here (see figure 10). The changes in distance were brought to a common basis by subtracting the relevant sums of covalent radii from the observed changes for each particular ligand. This way the use of different ligands would not affect the correlations.

Figure 11 provides a clearer picture of the reaction, by using z to follow the inversion in configuration.

If we consider the reaction in detail, at the start one has ligand Y infinitely distant from the tetrahedral cadmium complex. As the ligand moves closer to the complex via a backside approach to ligand X, the

Figure 9: Five coordinate intermediate for ligand exchange study.



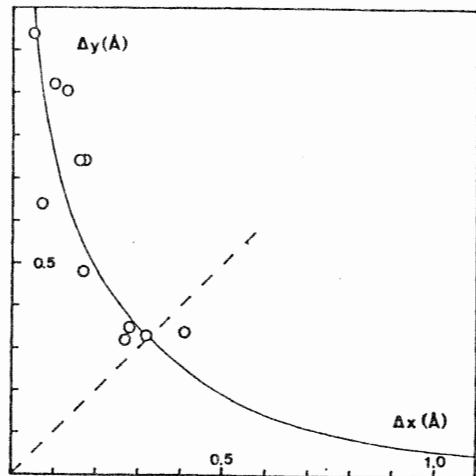


Figure 10: Correlation of the axial distance increment  $\Delta x$  with increment  $\Delta y$ .

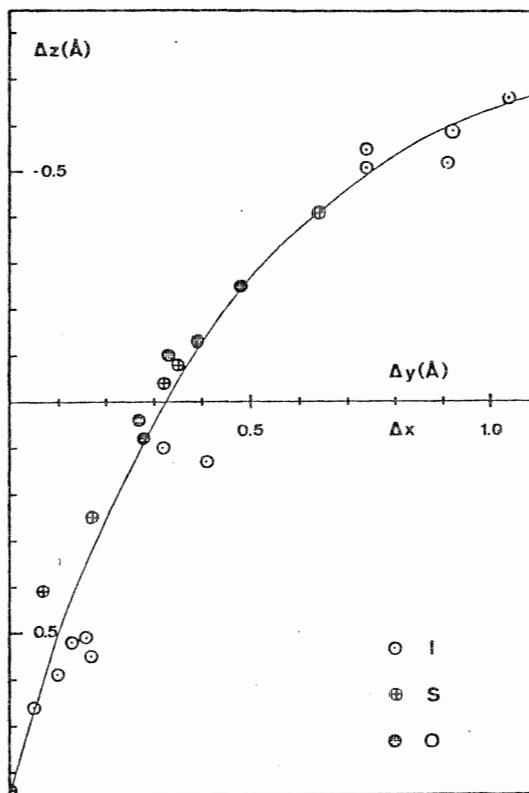


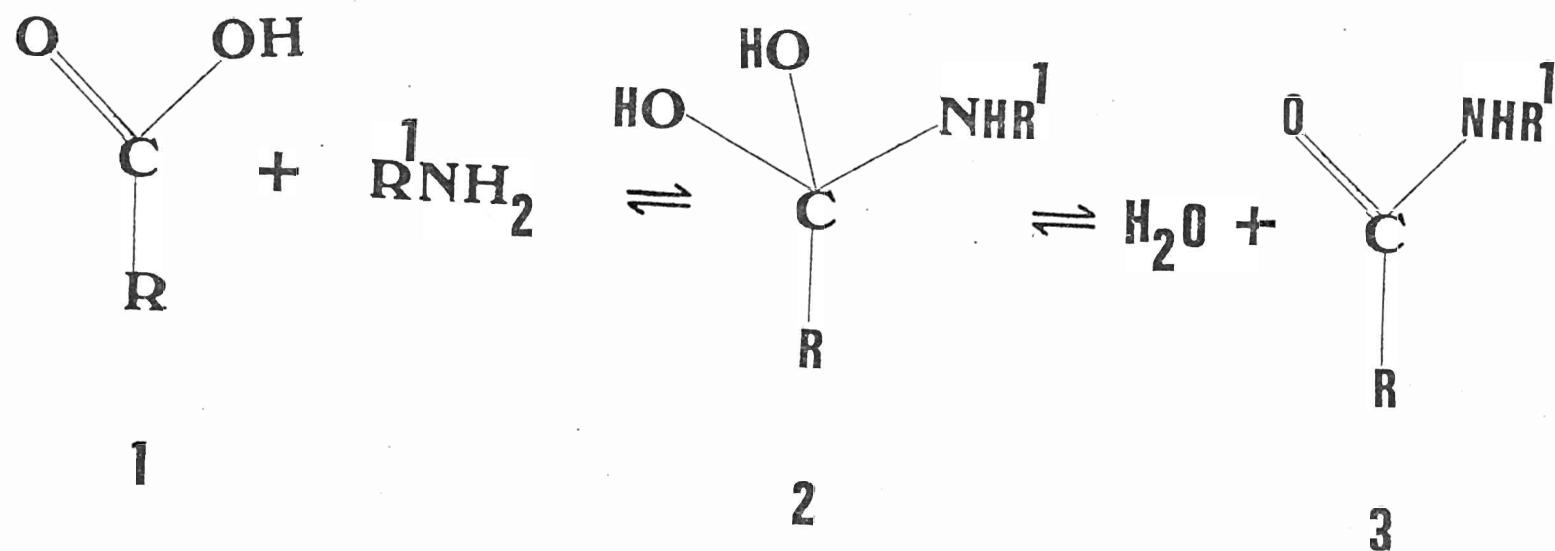
Figure 11: Plot of the distance increments  $\Delta x$  and  $\Delta y$  as a single function of  $\Delta z$ .

X-Cd-S angle is observed to decrease. This results in a concomitant flattening of the  $\text{CdS}_3$  pyramid and also an increase in Cd-X distance. At the transition stage  $\Delta x = \Delta y = 0.32\text{\AA}$ ,  $\Delta z = 0$  and  $\text{CdS}_3$  is coplanar. As the reaction goes to completion one sees a decrease in the Y-Cd bond length with a simultaneous movement of X to infinity. Together with the eleven points obtained from crystal data a twelfth point can be considered for the case  $\Delta x = 0$ ,  $\Delta y = \infty$ ,  $\Delta z = 0.84\text{\AA}$ , corresponding to the geometry of an ideal  $\text{XCdS}_3$  tetrahedron plus an infinitely distant Y atom. During this transformation one has witnessed the probable pathway for the  $\text{S}_{\text{N}}^2$  reaction. Detailed kinetic investigation of these cadmium species has not been possible. So here is a case where the structure correlation method gives a picture of a reaction which has not been studied by classical methods of determining reaction mechanisms.

### 3. Nucleophilic Addition

The reaction shown in figure 12 represents another example in a long line of basic reactions that one meets in organic chemistry. It accounts for the formation and hydrolysis of peptides, carboxylic acid esters and anhydrides, acetals and hemiacetals, ketals and hemiketals. There are basically three stages to this particular reaction. At stage (1) the lone pair on the nitrogen attacks the electrophilic carbon of the carboxylic acid to form a tetrahedral transition state (2). With the elimination of water one observes the transition from (2) to (3) to form the amide. While this particular reaction looks very straight

Figure 12: The general course of reaction in the formation of an amide.



forward in terms of mechanistic information, the structural changes still remain obscure.

In a paper by Burgi et al.<sup>13</sup>, six structures were examined. They dealt with intramolecular N---C=O interactions. The main aim of their paper was to find closely similar subunits from all possible sources and try to correlate these changes in such a way that they can be made to mimic the intuitive pathway concerning the course of reaction. It was observed that as the N---C distance decreased, the carbonyl carbon was seen to be displaced from the plane mapped out by its three ligands. There was a concomitant increase in the C---O distance. It was concluded that since these changes are in line with those expected intuitively for this type of reaction, there is no reason why one cannot make use of the information to map out the reaction pathway.

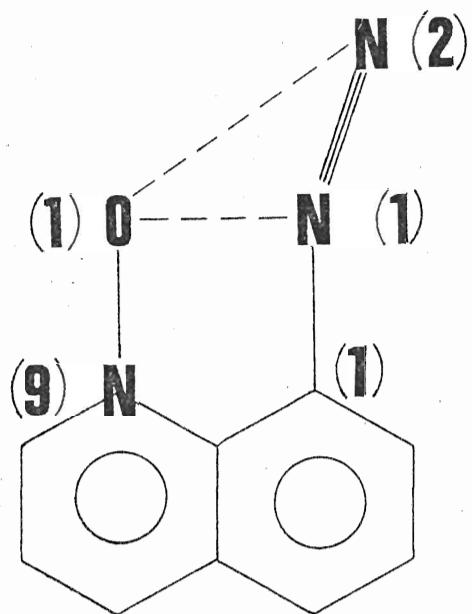
In 1974, the same workers published a paper concerning the above reaction to study the difference in nucleophilic ability of O and N.<sup>14</sup>

#### 4. Recent Examples on The Application of The Structural

##### Correlation Method

Dunitz and Wallis have observed a remarkably short distance between the N,N-triple bond and the nucleophilic oxygen<sup>15</sup> in the compound shown in figure 13. All the evidence points towards the possibility of an incipient nucleophilic attack on a diazonium group. The distance between O(1)---N(1) of 2.44 $\text{\AA}$  is well within the sum of the van der Waals radii (2.9 $\text{\AA}$ ). Although the two atoms are displaced to opposite sides of the

Figure 13: A remarkably short distance between N,N-triple bond and the nucleophilic oxygen.



best plane through the quinoline system, this merely leads to a increase in distance of 0.017 $\text{\AA}$ . The nitrogen (N1) is observed to deviate from linearity by 10.2 degs from the C(1)---N(2) line, and in doing so, it gets closer to O(1). The attractive nature of this interaction is clear, although one may argue that this sort of interaction is expected as a consequence of formal charges being present. The exocyclic angles about the two atoms of interest are seen to change in such a way as to favour this interaction. The possibility of packing forces causing these effects was considered, but was rejected since the closest distance between these atoms and the fluorines on the  $\text{BF}_4^-$  anion is 3.23 $\text{\AA}$  to O(1) and 2.81 $\text{\AA}$  to N(1). An angle of 104.4 $^\circ$  was observed about O(1)-N(1)-N(2) and was concluded to be the preferred angle of attack.

The compound shown in figure 14 undergoes inversion in the solid phase through a transition state which is planar at both nitrogens.<sup>15</sup> Kaftory and Agmon studied this reaction by the structural correlation method<sup>17</sup>. They searched the Cambridge Database for structures containing the 1,2,4-triazolidene ring bonded to a six-membered ring at the 1,4-positions (see figure 15) and selected only structures with fairly accurate parameters (i.e., no heavy-atom structures,  $R < 10\%$ ,  $\sigma^-$  ( $d$ )  $< 0.01\text{\AA}$ ). A total of 27 compounds were obtained by this procedure. Related parameters were averaged by assuming mirror symmetry along the inversion pathway.

In order to follow the transformation, where the N atoms go from

Figure 14: Compound found to undergo inversion in the solid phase.

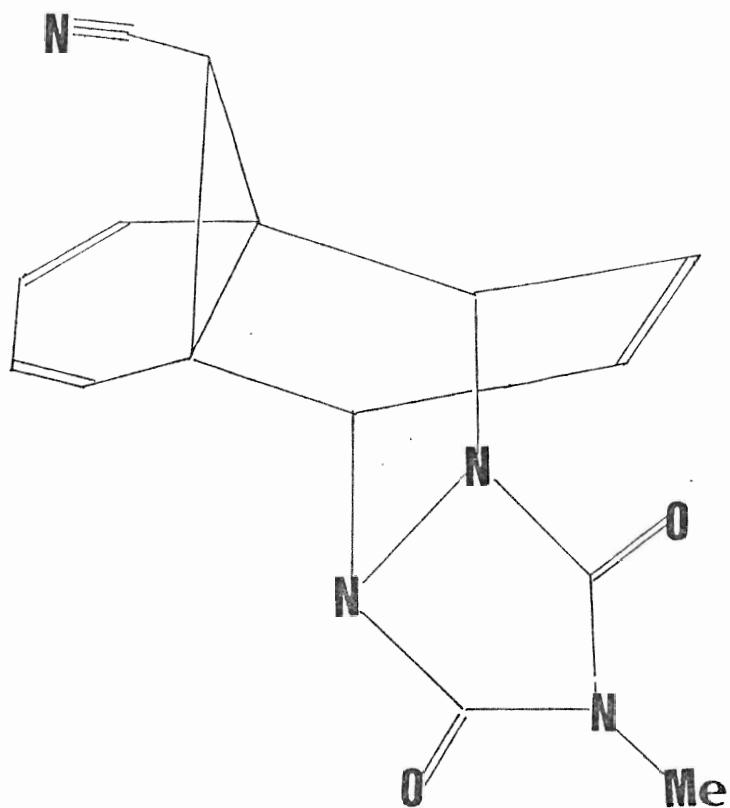


Figure 15: 1,2,4-triazolidine ring bonded to a six-membered ring at the 1,4-positions.

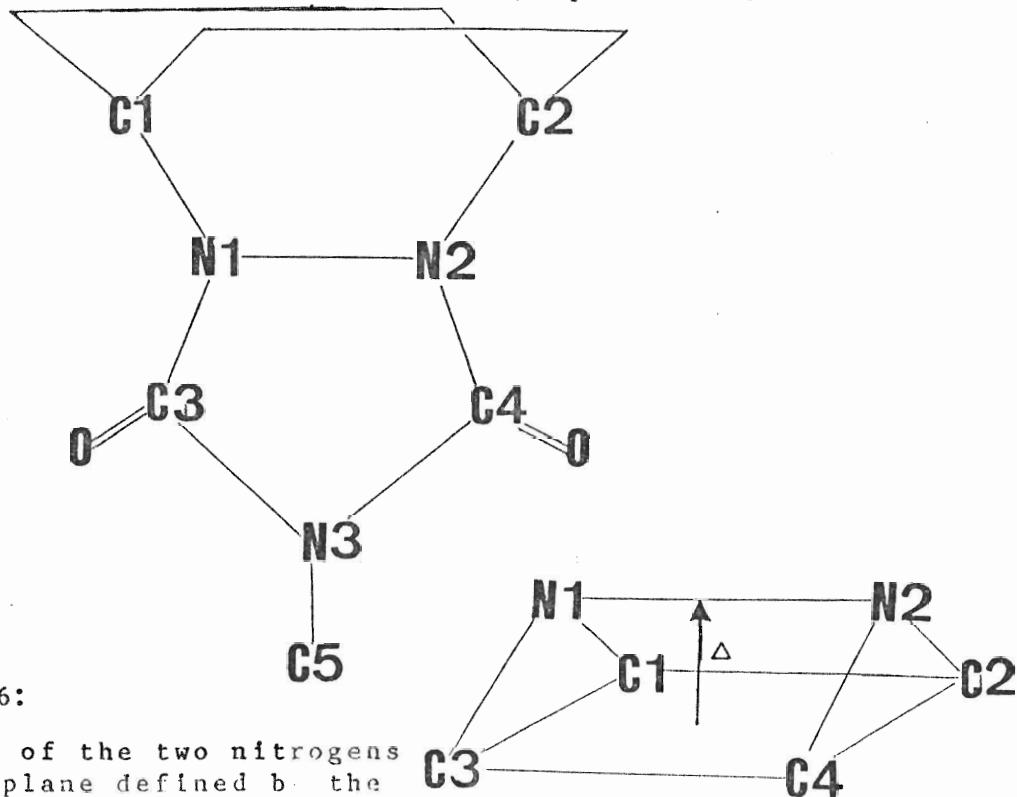


Figure 16:

Deviation of the two nitrogens from the plane defined by the four carbons.

being tetrahedrally hybridized ( $sp^3$ ) to trigonal planar ( $sp^2$ ) at the transition state, two parameters were defined. These were the out-of-plane bending parameter ( $\alpha$ ) and the out-of-plane displacement parameter ( $\Delta$ ). The latter parameter was used to follow the deviation of the two nitrogens from the plane defined by the four carbons (see figure 16). This parameter has been used successfully before for the ligand exchange reaction for the cadmium complexes.<sup>18</sup> An additional parameter  $\alpha_{av}$  was also introduced to follow this change in hybridization at the N(1) centre:

$$\alpha_{av} = (\alpha_1 + \beta_1 + \beta_2)/3$$

where  $\alpha_1 = C1-N1-C3$ ,  $\beta_1 = C1-N1-N2$ ,  $\beta_2 = N2-N1-C3$

Appropriate functions were needed to follow the inversion. They were required to fulfil the criteria of exhibiting mirror symmetry and also being continuous along the inversion pathway. Least-squares procedures were applied in order to obtain the best possible equations to describe the variations in each internal parameter during the inversion. Different effects caused by the introduction of various R-groups were handled separately. Extrapolation of the graphs was able to give the geometrical parameters at the transitions state. These values were found to agree well with theoretical values.

Statistical methods were employed in the final stage to reduce the dimensionality of the problem. This way it is possible to point out parameters that have significant variance during the inversion.

From the results it was evident that a simultaneous rather than a

consecutive double N-inversion reaction takes place. This was exhibited by the general adherence to mirror plane symmetry during the inversion. The parameters of the moiety at the transition state were used to calculate the energy barrier for the inversion. A value of 9 kcal/mol was obtained. This explained why relatively few systems exhibited trigonal planarity about the N-N bond. Also it was believed that the variations in the internal parameters during the inversion were dependent upon the substitution at the N3 position. The value of  $\alpha_{av}$  was observed to change from  $113.2^\circ$  for the amine derivative to a value of  $119.6^\circ$  for the dimethyl-methane derivative.

### C. The S--O Interaction

The S---O interaction was originally noted as a consequence of research being carried out to detect anomalous S---S distances. An S---O distance of  $2.40\text{\AA}^\circ$  was observed by Mammi, Bardi, Traverso and Bezzi<sup>18</sup> (1961). This distance is short with respect to a non-bonding van der Waals distance of  $3.25\text{\AA}^\circ$ , but substantially longer than a single bond ( $1.5\text{\AA}^\circ$ ).

Several dozen structures of the type C-S---Z=Y have subsequently been investigated to see if they also exhibit this phenomenon<sup>19</sup>. Compounds were synthesized with various elements (Z = O, S, N, Se), those with Z=O proving to be the focal point of research. Nyburg summarizes the work in this area up through about 1970<sup>20</sup>. Distances in the range  $2.03\text{\AA}^\circ$  to

$2.93\text{\AA}$  were found between S and O. Typically these systems have a bivalent S atom in a nearly co-planar environment and there is conjugation in a five-membered hetero ring closed by the S---Z interaction:<sup>21</sup> (single bond/no bond resonance; figure 17). Some examples of different types of compounds with short S---O interactions will now be discussed.

Johnson and Paul<sup>22</sup> noted a very short S---O distance when they solved the crystal structure of 3,5 epidithio-2-nitroso-1,5-diphenylpenta-2,4-diene-1-one (figure 18). An S---O distance of  $2.034\text{\AA}$  was observed. This distance was much shorter than the other S---O "nonbonding" contacts that had been observed up until then. The oxygen atom of the nitroso group was in very close contact with S. The N=O bond length of  $1.31\text{\AA}$  was significantly longer than the expected values of  $1.14$ - $1.24\text{\AA}$ . Also the C-N=O angle  $113^\circ$  was much less than the expected ( $125^\circ$ ).

In 1971 Nyburg and coworkers published the crystal structure of the "desaurin from acetophenone"<sup>20</sup> (figure 19). The most striking feature of the desaurin structure was the close planarity of the carbonyl and sulphur systems (central 10 atoms coplanar). An S---O distance of  $2.64\text{\AA}$  was noted.

In recent publications on this interaction, Kalman and Parkayi<sup>21</sup> observed close contacts of  $2.675(5)$  and  $2.655(5)\text{\AA}$  for the two independent molecules that exist in the asymmetric unit for the compound  $\text{C}_{11}\text{H}_{12}\text{N}_2\text{OS}$  (figure 20). With C---S---O angles of  $164.1(4)$  and  $164.9(4)$ ,

Figure 17: Single bond / no bond resonance.

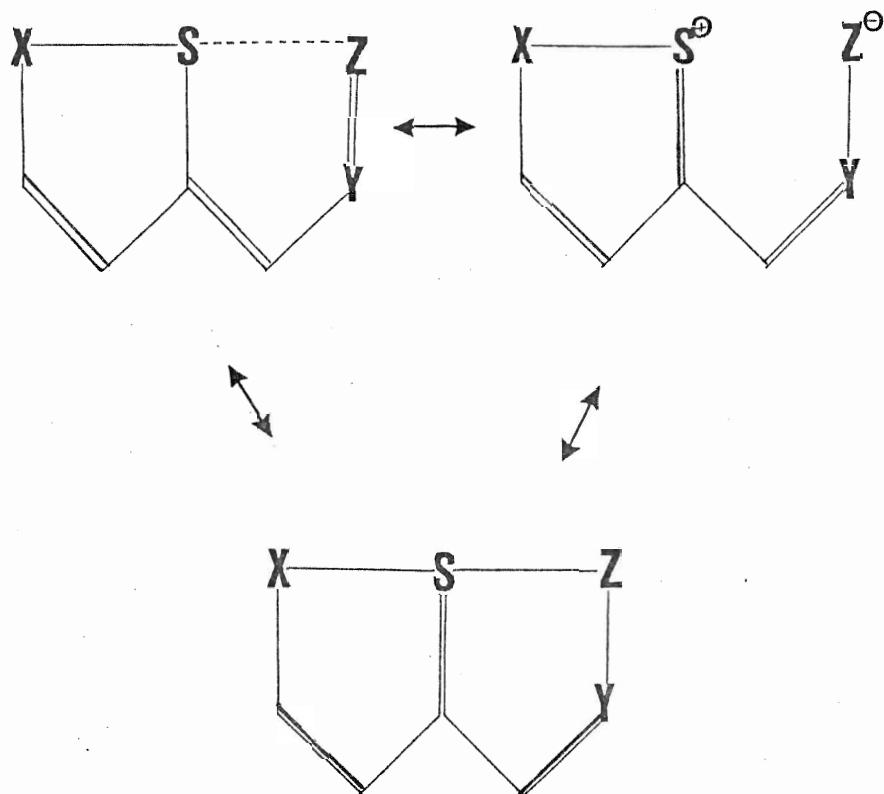
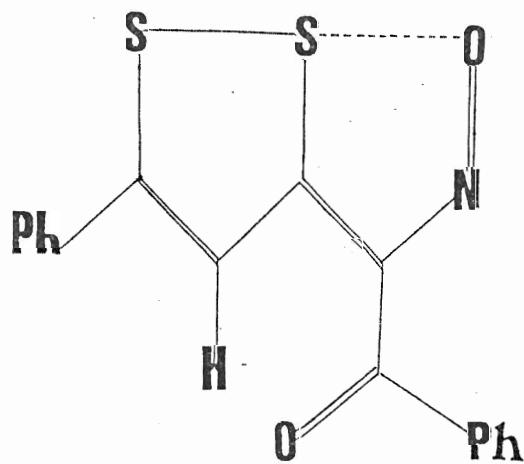


Figure 18: 3,5-epidithio-2-nitroso-1,5-diphenylpenta-2,4-diene-1-one.



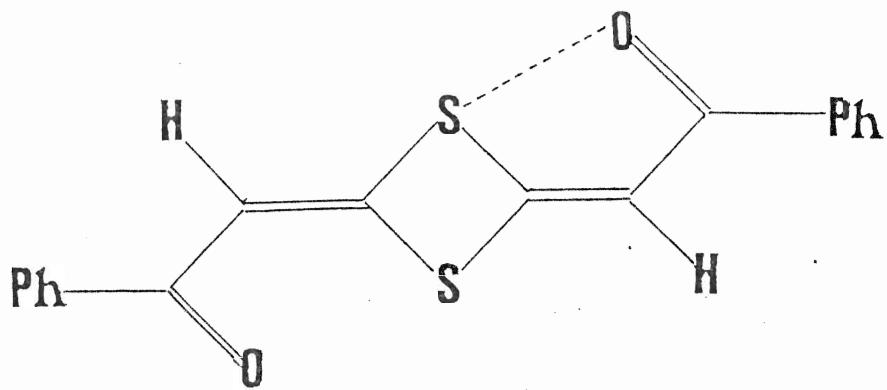


Figure 19: Desaurin from Acetophenone

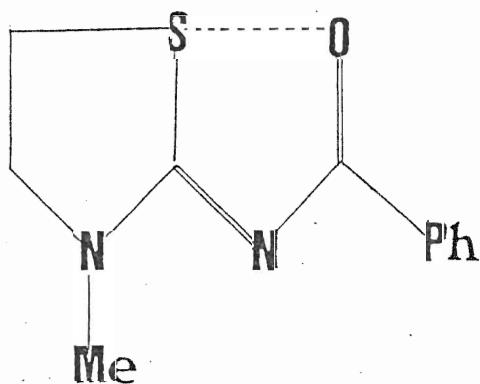


Figure 20: A compound with two independent Molecules.

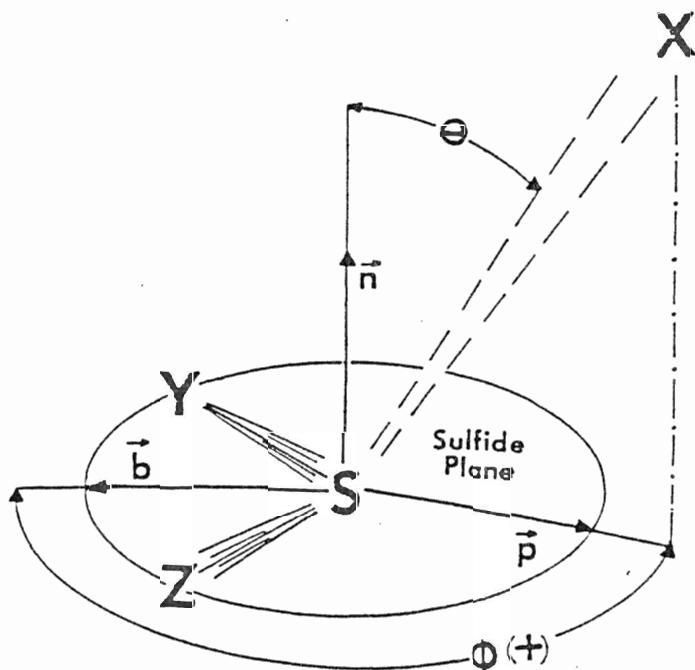
again suggesting a single bond / no-bond resonance.

Although these distances and others in this range appear to reflect considerable interaction between S and O, it has been reported that an S---O distance of  $2.41\text{\AA}$ <sup>o</sup> represents only a very weak, possibly non-existent covalent bond<sup>23</sup>. These conclusions were arrived at when calculations on dissociation energies were carried out using Huggins' equation. A dissociation energy of 2.2kcal/mol was obtained for this bond length, compared to 71.1 kcal/mol for a normal single bond.

In a paper by Dunitz et al., the directional preferences of non-bonded atomic contacts of nucleophiles and electrophiles with divalent sulphur (S bonded to two ligands, neither one being H) was studied.<sup>24</sup> It was found that whereas electrophiles approach S roughly  $20^{\circ}$  from the perpendicular to the plane through atoms Y-S-Z (see figure 21), nucleophiles tend to approach approximately along the extension of one of the covalent bonds to S. With the aid of the "frontier orbital" model it was suggested that electrophiles interact preferentially with highest occupied (HOMO) and nucleophiles with the lowest unoccupied (LUMO) molecular orbital. These findings are further supported by calculations and chemical reaction studies on S systems, and are also consistent with the structural information presented in this section. We have a nucleophile (oxygen) approaching sulphur along the extension of one of the covalent bonds to sulphur.

A very recent paper reports both experimental and theoretical studies, on the charge density about the bond in question<sup>25</sup>. The

Figure 21: Parameters used to study directional preferences of electrophiles and nucleophiles (X) towards sulfide plane.



[The polar angle  $\theta$  ( $0 \leq \theta \leq 180^\circ$ ) and azimuthal angle  $\phi$  ( $-180^\circ < \phi < 180^\circ$ ) are spherical polar coordinates specifying the direction of S---X. Vector  $\vec{n}$  is normal to the sulfide plane,  $\vec{b}$  bisects angle  $\angle(Y-S-Z)$ , and  $\vec{p}$  shows the direction of S---X projected onto the sulfide plane.]

compound 3-benzoylimino-4-methyl-1,2,4-oxathiazane (BMO) was observed to have a S---O distance of  $2.24\text{\AA}$ , when combined x-ray and neutron diffraction studies were carried out (figure 22). This compound was compared with an earlier study, where an S---O distance of  $2.68\text{\AA}$  was observed for 2-(2-chloro-benzoylimino)-1,3-thiazolene (CBT) (see figure 23). It was reasoned that although CBT showed no trace of electrons in the region between S and O, there might exist a better chance for BMO, due to the much shorter S---O interaction distance. In order to obtain greater insight into the interaction, theoretical calculations were carried out on the fragments of interest. It was seen from the energy/distance (S---O) diagrams, that the two distances observed experimentally corresponded to energy minima. The extra bonding present in these species was attributed to the overlap of p-orbitals of oxygen and d-orbitals of sulfur. The difference in bond lengths arises from the nature of the atom adjacent to S. It was stated that charge transfer interactions (and thus very short S---O distances) are possible only when this atom is more electronegative than S.

#### D. Aim of Research

The system being studied is based on a thiadiazolene skeleton . If one looks at the structure of interest, what one can see is a very interesting system which can exhibit various resonance forms. Resonance forms suggest the electronic pathway by which a ring closure reaction

Figure 22: 3-benzoylimino-4-methyl-1,2,4-oxathiazane (BMO).

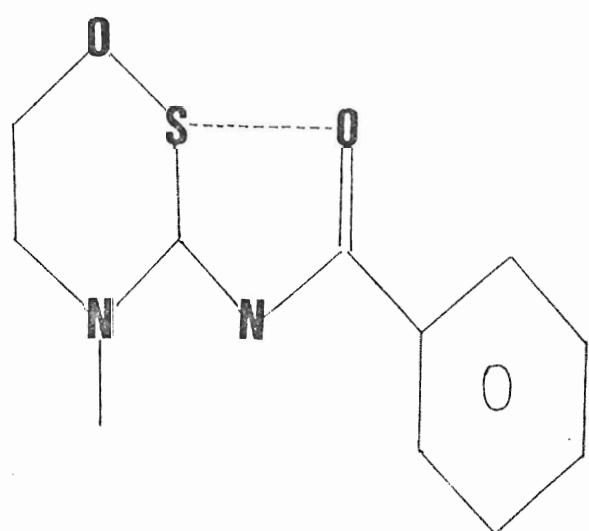
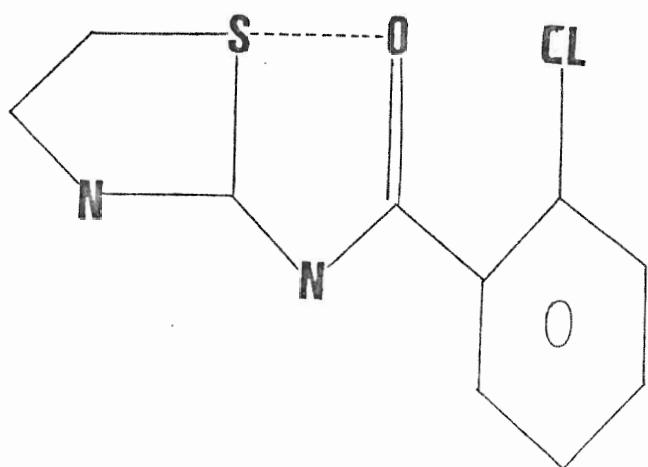


Figure 23: 2-(2-chloro-benzoylimino)-1,3-thiazolene (CBT).



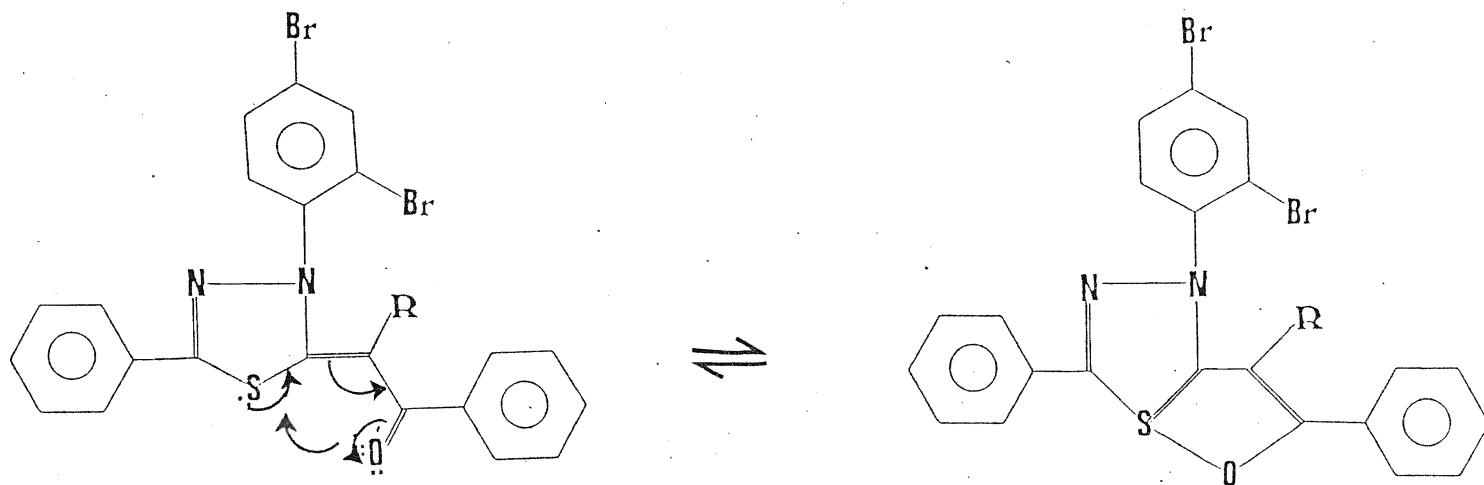
might occur. The actual transformation which is envisaged for our purpose is shown in figure 24. If the lone pair on the oxygen can be brought close enough to the anti-bonding orbital of the S-C bond, then the ring closure reaction can be thought to occur.

Already two other structures have been solved, one with R=H (BRPHO) and the other with R=Et (BRPETO)<sup>26</sup> in our laboratory. The present thesis reports the structure of the methyl derivative BRMEO.

The main aim of this study was to perturb the system in question in such a way as to change the S---O distance from a non-bonding case ( $3.25\text{\AA}$ ) to a bonding one ( $1.5\text{\AA}$ ). It was felt that steric effects play a major role in the activation of this change. This was deduced when a molecular model of the structure was made. If we can make appropriate derivatives of this compound such that the phenyl ring closest to the oxygen can be made to move in a clockwise fashion (forcing the oxygen to move closer to the sulphur), then the desired change in bond distance of approximately  $1\text{\AA}$  can possibly be obtained.

Figure 24:

Envisaged transformation.

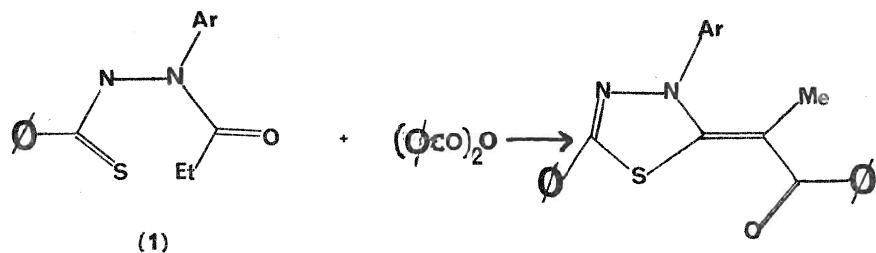


## II. Experimental Section

### A. BRMEO

#### 1. Compound Synthesis and Density Determination.

The compound (BRMEO) used for this particular study was synthesized by Dr Taj Mohammed according to the following scheme:



$\text{Ar} = \text{C}_6\text{H}_3\text{Br}_2$

where compound (1) was previously synthesized by the method outlined in reference 27.

Crystal selection and density determination were carried out by Tony Basile.

This thesis reports the first detailed x-ray study of the compound.

## 2. Preliminary Work

Oscillation and Weissenberg photographs were obtained by Tony Basile using a Weissenberg camera (Charles Supper Co.) with  $\text{CuK}\alpha$  radiation. He determined the space group to be  $P2_1/c$ .

The cell dimensions were obtained from the photographs supplied by Tony Basile. It is noticeable that there exists a significant difference between the  $a$  and  $c$  lengths given by the CAD and those obtained from the Weissenberg photographs.

Weissenberg	CAD
$a_w$ (rotation axis) = $19.60\text{\AA}$	$a_c = 17.492\text{\AA}^\circ$
$b_w = 16.99\text{\AA}^\circ$	$b_c = 16.979\text{\AA}^\circ$
$c_w = 13.67\text{\AA}$	$c_c = 14.962\text{\AA}^\circ$
$c_w \sin \beta_w = 12.85\text{\AA}$	$\beta_c = 106.46^\circ$
$\beta_w = 110^\circ$	
$P2_1/c$	$P2_1/c$

Correct magnitudes of  $a_w$  and  $c_w \sin \beta_w$  are obtained by the vector transformation  $a_w = a_c + c_c$  and  $c_w = a_c + 2c_c$ . This gives  $a_w = 19.53\text{\AA}$ ,  $b_w = 16.99\text{\AA}^\circ$ ,  $c_w = 30.40\text{\AA}^\circ$ ,  $\beta_w = 155^\circ$ , and  $c_w \sin \beta_w = 12.68\text{\AA}^\circ$  (The correct space group is  $P2_1/n$  in this transformation). Obviously, an error was made in assigning the position of the  $1k0$  line on the first layer Weissenberg photograph (from which  $\beta$  was obtained).

The cell volume ( $V = abcsin(\theta)$ ) was calculated to be  $4263\text{\AA}^3$  and consequently the number of molecules per unit cell were calculated as follows:

$$\begin{aligned}\text{unit cell contents} &= d * V * 6.02 * 10^{23} * 10^{-24} \\ &= 1.62 * 4263 * 0.6024 \\ &= 4160.23 \text{ amu}\end{aligned}$$

$$\text{Therefore } \# \text{ molecules/unitcell} = 4160.23 / 528.27 = 7.88 = \text{approx 8.}$$

The existence of 8 molecules per unit cell implies the presence of 2 independent molecules per asymmetric unit for this space group.

### 3. Data Collection

The data collection was carried out by Tony Basile and Roman Guzowski (summer 1983). The intensities were measured on an Enraf-Nonius CAD-4 diffractometer controlled by a PDP-8a computer.

Graphite-monochromatized MoK<sub>α</sub> radiation was used ( $\lambda = 0.71069\text{\AA}$ ). Unit cell parameters were obtained by the collection of 25 centered reflections for  $8.0^\circ < \theta < 16.0^\circ$ . The intensity of a standard reflection was measured every two hours as a check on crystal and electronic stability. The orientation of the crystal was checked every 100 reflections by the recentering of three reflections selected to be nearly orthogonal to each other. The intensities were measured by the  $\omega/2\theta$  scan technique.

Systematic absences in the diffractometer data agreed with those on the Weissenberg photograph, thus confirming space group P2<sub>1</sub>/c.

The various parameters of interest are outlined in Tables 3a and 3b.

Table 3a.

## Crystal Data and Experimental Details of x-ray Diffraction Studies

## Crystal Parameters for BRMEO

Formula  $C_{23}H_{16}Br_2N_2OS$  $a = 17.492(4)\text{\AA}$  $b = 16.979(1)\text{\AA}$  $c = 14.962(1)\text{\AA}$  $\alpha = 90^\circ$  $\beta = 106.46^\circ(1)$  $\gamma = 90^\circ$ Volume =  $4261.55 \text{ \AA}^3$ 

F(000) = 2096.00

Crystal system monoclinic

Absences  $h0\ell$ ,  $\ell$  odd $0k0$ ,  $k$  oddSpace group  $P2_1/c$ 

z 8

 $D_c$  1.65 g/cc $D_o$  1.62 g/cc

Table 3b.

Crystal size	0.34*0.15*0.11mm
Instrument	Nonius CAD-4
Maximum theta	25°
Radiation	MoK $\alpha$ , graphite-monochromatized
Wavelength, Å	0.71073
Scan technique	$\omega/2\theta$
Scan range	0.8 + 0.35tanθ
Scan rate, deg/min	5
Max scan time, Secs	180
Intensity standard variation	289696 $\pm$ 4595 (1.6%)
$\mu$ (cm $^{-1}$ )	40.74
Range of transmission factor	0.25-0.64
Number of reflections collected	8131
Number of independent reflections	7498
Number of reflections with $I > 3\sigma$	3112
R	0.0570
$R_w$	0.0591
Number of parameters refined	343

#### 4. Data Reduction

The program CONVI<sup>28</sup> was utilised to calculate structure factors from the data obtained through the use of the Enraf-Nonius CAD-4 diffractometer. Below the main functions of the program are outlined.

- (1) The data are scaled with reference to the attenuator, so that the intensity of any reflection for which the attenuator is set, is multiplied by 17.0. This value was experimentally determined by Dr M.F. Richardson. None of the BRMEO reflections required the attenuator, however.
- (2) All reflections are normalized to the initial intensity of the standard reflection. For BRMEO the standard showed no systematic change with time, but varied by  $\pm$  1.6% throughout the data collection.
- (3) The diffractometer records each reflection and sub-divides it into 96 divisions, of which the first and last 16 divisions are background counts on the left (BGL) and the right (BGR) of the peak maximum. The following allowance for the background count is made for each reflection:

$$I = INT - 2(BGL + BGR)$$

Where INT is the peak count region. (central 64 divisions)

- (4) Lorentz and polarization corrections are calculated and made on the

intensities. Since the X-ray beam is composed of equal components of parallel and perpendicular vectors one can consider the usual X-ray beam to be unpolarised. The polarization term arises because while the reflection of the electric vector parallel to the reflecting plane is determined by the electron density only, the perpendicular vector is dependent on both the electron density and also  $\cos^2\theta$ . The X-ray beam is partially polarised by the graphite crystal monochromator before being polarized further by the crystal. The following correction is applied to correct for polarization:

$$P = \frac{\cos^2(2\theta_m) + \cos^2(2\theta)}{1 + \cos^2(2\theta_m)}$$

Where  $\theta_m$  is the Bragg angle for graphite monochromator, and  $\theta$  is the Bragg angle for the monochromator graphite crystal. ( $\theta_m$  for Mok $\alpha$  radiation is equal to 6.1).

The Lorentz correction is required since the time required for a reciprocal lattice point to pass through the sphere of reflection is not constant but is related to its position on the reciprocal lattice and the direction of approach. The following correction is applied:

$$L = 1/\sin(2\theta)$$

Where  $\theta$  is the usual Bragg angle for a specific  $hkl$  reflection.

Generally the application of these two corrections is combined and referred to as Lp (Lorentz-polarization) correction.

(5) Both the width and the speed of the scan are corrected for by their respective absolute values, which are stored in the data records.

(6) Finally the standard deviation for the reflection (subsequently used in the refinement procedure to determine the weight of the reflection) is calculated by the following relationship:

$$\text{sigma}(F) = \frac{1}{2\sqrt{\frac{\text{INT} + 4(\text{BGL}+\text{BGR}) * \text{WIDTH} * \text{ATTEN}}{\text{Lp} * \text{FACTOR} * \text{ABS}(\text{NPI}) * \text{RAW}}}},$$

where most of the parameters used are defined as before except the following:

FACTOR is the correction factor for any diminution in the intensity of the standard reflection.

ABS(NPI) is the absolute value of the variable scan speed, read from the original data record.

WIDTH is the scan width.

RAW = INT - 2\*(BGL + BGR)

ATTEN is 17 when set and 1 when not.

## 5. Structure Solution by the Heavy atom Method

The solution of this structure was accomplished by the heavy atom method, using the program SHELX<sup>29</sup>. This method is one of two major techniques utilized to solve the phase problem, the first step in structure solution.

Due to the presence of two independent molecules per asymmetric unit one is faced with the task of calculating 256 possible inter- and intra-set vectors between the two sets of bromines. Once multiplicity of some vectors is taken into account and the self-vector peaks are subtracted 128 peaks are left. Table 4a. lists the peaks expected for both atoms in the same set of positions and also peaks between atoms in different sets.

The Patterson was calculated for one-quarter of the unit cell. The expected height for a Br--Br peak was calculated as follows:

23C---C	23*(6*6)	828
16H---H	16*(1*1)	16
2Br---Br	2*(35*35)	2450
2N---N	2*(7*7)	98
O---O	1*(8*8)	64
S---S	1*(16*16)	<u>256</u>
		<u>3712</u>

Since there are eight molecules within the unit cell, the expected height is  $8 * 3712 = 29696$ . In order to scale everything down to the same height as the origin peak (999), this number needs to be divided by

999;  $29696/999 = 29.73$ . The height of one Br-Br peak is therefore  $(35)^2/29.73 = 41.21$ . Peaks of multiplicity two would be expected to have twice this value.

One would expect each set of bromines to give two peaks, one of type 0,  $2y + 1/2$ ,  $1/2$  and one of type  $2x$ ,  $1/2$ ,  $1/2 + 2z$ . Therefore in the unique quarter of the Patterson one expects  $4 \times 2 = 8$  peaks of multiplicity two. For the interset peaks there are six sets altogether as listed in Table 4a., part B. Here again one expects each set of interset bromines to give 4 peaks, of the type  $x+x'$ ,  $y+y'$ ,  $z+z'$ ;  $x-x'$ ,  $y-y'$ ,  $z-z'$ ;  $x+x'$ ,  $1/2+y-y'$ ,  $1/2+z+z'$ , and  $x-x'$ ,  $1/2+y+y'$ ,  $1/2+z-z'$ . Therefore altogether one expects  $4 \times 6 = 24$  peaks of this type in the unique quarter of the cell. In total 32 peaks of multiplicity two and of the type mentioned above are expected.

In order to obtain three parameters ( $x, y, z$ ) for each of the four independent bromines a trial and error procedure was followed until a consistent set of coordinates could be obtained. Each  $y$ -value was initially matched to a peak of the type  $2x$ ,  $0.5$ ,  $2z + 0.5$ . To test if the parameters obtained were plausible peaks of the type  $2x$ ,  $2y$ ,  $2z$  were looked for. Once a match was found a second bromine position was sought in the same fashion. When two bromine positions were known, they were further checked for consistency by looking for peaks of the type  $x - x'$ ,  $y - y'$ ,  $z - z'$  and  $x + x'$ ,  $0.5 + y - y'$ ,  $0.5 + z + z'$ . Once all four positions were known, to decide which two bromines were situated on the same phenyl ring, interatomic distances were calculated for each set of

Table 4a Patterson Peaks Expected for 4 Sets of Bromine Atoms in  
General Positions, Space Group P2<sub>1</sub>/c.

A. Peaks between atoms in the same set set of positions.

Position.	Multiplicity.
0, 2y + 0.5, 0.5	2
0, -2y + 0.5, 0.5	2
2x, 0.5, 0.5 + 2z	2
-2x, 0.5, 0.5 - 2z	2
2x, 2y, 2z	1
-2x, -2y, -2z	1
2x, -2y, 2z	1
-2x, 2y, -2z	1

Plus three other similar sets for atoms in positions x', y', z'; x'', y'', z''; x''', y''', z'''.

B. Peaks between atoms in different sets.

Position	Multiplicity
x - x', y - y', z - z'	2
x + x', 0.5 + y - y', 0.5 + z + z'	2
x - x', 0.5 + y + y', 0.5 + z - z'	2
x + x', y + y', z + z'	2
-x - x', 0.5 + y - y', 0.5 + z + z'	2
-x + x', y - y', -z + z'	2
-x - x', y + y', -z - z'	2
-x + x', 0.5 + y + y', 0.5 - z + z'	2
x - x', 0.5 - y - y', 0.5 + z - z'	2
x + x', -y - y', z + z'	2
x - x', -y + y', z - z'	2
x + x', 0.5 - y + y', 0.5 + z + z'	2
-x - x', -y - y', -z - z'	2
-x + x', 0.5 - y - y', 0.5 - z + z'	2
-x - x', 0.5 - y + y', 0.5 - z - z'	2
-x + x', -y + y', -z + z'	2

Plus five other similar sets.

Table 4b. Assignment of Patterson Map.

Peak number	HEIGHT	X/A	Y/B	Z/C	Assignment
1	999	0.0000	0.0000	0.0000	origin.
2	999	1.0000	0.0000	-0.0000	sym related to 1.
3	230	0.0000	0.1650	0.5000	0, 1/2 - 2y', y'', y''', 1/2
4	230	1.0000	0.1650	0.5000	sym related to 3.
5	167	0.5622	0.5000	0.3964	x'+x''', 1/2+y'-y''', 1/2+z'+z'''
6	161	0.1155	0.0000	0.2871	+x'-x'', 1/2-y'-y'', 1/2+z'-z''
7	161	0.7222	0.5000	0.2381	2x''', 1/2, 2z'''+1/2
8	137	0.1603	0.0000	0.3416	-x'+x''', y'-y''', -z'+z'''
9	124	0.0000	0.4789	0.5000	0, 1/2+2y, 1/2
10	124	1.0000	0.4789	0.5000	sym related to 9.
11	121	0.7168	0.0000	0.3636	x''-x''', 1/2-y''-y''', 1/2+z''-z'''
12	116	0.3858	0.5000	0.2479	2x, 1/2, 1/2+2z
13	115	0.4439	0.5000	0.1114	x''+x''', y''+y''', z''+z'''
14	101	-0.0033	0.3177	0.1027	x-x', y-y', z-z'
15	101	0.9967	0.3177	0.1027	sym related to 14.
16	101	0.5582	0.1626	0.4866	x+x''', y+y''', z+z''
17	99	0.2729	0.5000	0.2709	2x''', 1/2, 1/2-2z''
18	97	0.0029	0.1560	0.3979	-x+x', 1/2+y+y', 1/2-z+z'
19	97	1.0029	0.1560	0.3979	sym related to 18.
20	92	0.8257	0.1370	0.2597	x-x''', 1/2+y+y''', 1/2+z-z''
21	92	0.1738	0.3368	0.2355	-x+x''', y-y''', -z+z''
22	91	0.8341	0.1773	0.1602	x'-x''', 1/2-y-y''', 1/2+z'-z'''
23	90	0.2746	0.3409	0.3672	x+x''', 1/2-y+y', 1/2+z+z'
24	89	0.4356	0.3229	0.1079	-x'-x''', y'+y''', -z'-z''
25	87	0.7251	0.1834	0.1333	-x-x'', -y-y'', -z-z''
26	86	0.6128	0.3436	0.3536	-x-x', -y-y', -z-z
27	84	0.8890	0.1589	0.1103	x''-x, -y''+y, z''-z
28	84	0.4034	0.5000	0.0512	2x', 1/2, 1/2+2z'
29	82	0.3889	0.1823	0.1486	x+x', 1/2-y+y', 1/2+z+z'
30	82	0.1627	0.5000	0.4768	2x'', 1/2, 1/2+2z''
45	55	0.7279	0.3224	0.2383	2x''', 2y''', 2z'''
52	48	0.6181	0.0323	0.2559	-2x, -2y, -2z
61	44	0.6012	0.3367	0.4538	-2x', 2y', -2z'
78	35	0.8362	0.3418	0.0243	-2x'', -2y'', -2z''

bromine positions. A distance of  $5.6\text{\AA}$  would be indicative that two bromines were substituted on the same phenyl in the 2,4-positions. The addition of these positions led to the eventual elucidation of the rest of the structure. The following set of positional parameters were obtained for the bromines:

Br1A	$x$	$y$	$z$	=	0.1923	0.4870	0.3758
Br2A	$x'$	$y'$	$z'$	=	0.1977	0.1680	0.2757
Br1B	$x''$	$y''$	$z''$	=	0.0804	0.3302	0.4887
Br2B	$x'''$	$y'''$	$z'''$	=	0.3645	0.1549	0.6165

(see Table 4b for assignment of Patterson peaks).

Once the positional parameters for the whole molecule were obtained (except hydrogens, although possible hydrogens were noted about the methyls) least squares refinement led to the drop in residual factor from 55% to 15%. With the introduction of the hydrogen atoms (fixed by calculating angles of  $109.5^\circ$  and C-H bond lengths of  $1.08\text{\AA}$ ), and refinement of anisotropic thermal parameters for Br, S, N, C(7),C(14)-C(16) and O the residual factor was reduced to below 6%.

The structure factors were weighted according to:

$$w = k / (\sigma^2(f) + g F^2)$$

where  $k$  was determined from refined values of  $g$ . The final values were  $g = 0.0005$  and  $k = 1.7137$ . The overall scale factor refined to 1.734(3). The consistency of this weighting scheme was evaluated by an

analysis-of-variance technique. The variance  $\sum(w\Delta^2)$  was checked until it was approximately constant with respect to  $\theta$  and  $|F|$ , where

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

$$\Delta = \left| |F_o| - |F_c| \right|$$

The scattering factors were calculated from the following expression by SHELX:

$$f(\lambda^{-1} \sin \theta) = \sum_i a_i \exp(-b_i \lambda^{-2} \sin^2 \theta) + c$$

The coefficients  $a_i$ ,  $b_i$  and  $c$  for Br with the real and imaginary parts of the corrections for anomalous dispersion were taken from International Tables for X-Ray Crystallography.

The residual fell to a final value of 5.7% when several cycles of blocked least squares refinement were carried out with hydrogen atoms in fixed positions with isotropic temperature factors, 18 anisotropic atoms and the remaining atoms isotropic. Each block contained the overall scale factor and the parameters for one of the independent molecules: 172 variables per block, or 343 in the whole structure.

The final difference map showed peaks  $1.15e/\text{\AA}^3$  high (close to Br1A); the deepest hole was  $-0.32e/\text{\AA}^3$  deep.

## B. BRPETO

The refinement on this structure was completed as a part of the thesis work, since BRPETO was needed to extend the structural correlations in the thiadiazolene compounds.

The structure had already been solved, and everything apart from the three hydrogens on the ethyl portion had already been found from the difference map.

A few peaks were observed close to the methyl carbon (C24)\* on the difference map. Three possible hydrogen peaks were selected and refined to see if they were the correct ones. While two of them behaved, H24C was observed to change in length from 1.20 $\text{\AA}$  to 1.48 $\text{\AA}$ . This implied that this peak was incorrect. Another choice was made, but again this also gave an unusually long distance.

At this point, another problem was noted. The C15-C23 distance was unusually long, while the C23-C24 distance was found to be rather short. For instance a chemically sensible distance for C15-C23 is 1.54 $\text{\AA}$  but it was observed to be 1.69 $\text{\AA}$ . To see if the positions of C15, C23 and C24 represented true minima, the two distances C15-C23 and C23-C24 were fixed to 1.45 $\text{\AA}$  and 1.54 $\text{\AA}$  respectively. The distance C15-C24 was calculated and also fixed by assuming the angle C15-C23-C24 to be 109.28°. This proved to be fruitless, because when the constraints were removed, the atoms were observed to slide back to their original

\*numbering scheme same as BRMEO except extra C methyl is C24; see Fig 27, p. 82

positions.

At this point a difference contour map was calculated to observe the possible disorder that may exist for this group. When the difference map was examined the electron density for C24 was observed to be elongated about a direction not normally expected (see figure 27 BRPET0). Normally one would expect the longest axis of the ellipsoid to be perpendicular to the one observed here, since libration about the C15-C23 bond should be easier than bending of the C15-C23-C24 angle. Static disorder of C24 in such a way as to produce the observed ellipsoid is not really conceivable, as it would result in two different C15-C23-C24 angles.

Once the C24 position was subtracted from the difference map, possible hydrogen positions were looked for and three possible peaks were found, as described previously. Their heights were ca.  $1/3 \text{ e}/\text{\AA}^3$ . Since the hydrogens did not refine well their positions were fixed with C24 allowed to move anisotropically. The final refinement gave a residual factor of 5.8% and the angles about C24 and its three hydrogens were found to lie in the interval  $98.4^\circ$ - $109.2^\circ$  with the C-H distances ranging from  $1.21$ - $1.30\text{\AA}$ . The C15-C24 was longer than expected ( $1.63(1)\text{\AA}$ ) and C23-C24 was  $1.33(2)\text{\AA}$  which is shorter than expected. At this point it was concluded that better data (from a larger crystal or at low temperature) would be needed to complete the refinement.

### III RESULTS

The positional and thermal parameters for BRMEO are listed in Tables 5-10. Important bond distances and angles for BRMEO may be found in Tables 11-12. Bond angles and distances in the phenyl rings for BRMEO may be found in Tables 13-16. The deviations from least squares planes for BRMEO are tabulated in Tables 17-18.

For BRPETO, corresponding data are listed in Tables 19-25.

Table 5. Fractional Coordinates and Equivalent Isotropic Thermal Parameters for Non-Hydrogen Atoms in BRMEO Molecule A.

ATOM	X/A	Y/B	Z/C	Ueq. <sup>a</sup>
BR1A	0.1923(1)	0.4870(1)	0.3758(1)	0.0645
BR2A	0.1977(1)	0.1680(1)	0.2757(1)	0.0700
SA	0.4161(1)	0.5841(1)	0.5586(2)	0.0318
OA	0.4439(3)	0.6619(3)	0.4308(3)	0.0408
N1A	0.3662(4)	0.4508(3)	0.4955(4)	0.0314
N2A	0.3647(4)	0.4462(4)	0.5882(4)	0.0354
C1A	0.3971(4)	0.5275(4)	0.7269(5)	0.0292(2)
C2A	0.4149(5)	0.6018(6)	0.7633(8)	0.0550(3)
C3A	0.4217(6)	0.6166(6)	0.8579(8)	0.0625(3)
C4A	0.4146(6)	0.5573(6)	0.9146(9)	0.0700(3)
C5A	0.3941(8)	0.4821(8)	0.8770(10)	0.0834(4)
C6A	0.3885(6)	0.4668(6)	0.7848(8)	0.0642(3)
C7A	0.3903(4)	0.5132(5)	0.6272(5)	0.0330
C8A	0.3261(4)	0.3858(4)	0.4395(5)	0.0296(2)
C9A	0.2481(5)	0.3913(4)	0.3842(5)	0.0357(2)
C10A	0.2104(5)	0.3266(5)	0.3335(6)	0.0443(2)
C11A	0.2500(5)	0.2572(5)	0.3395(5)	0.0359(2)
C12A	0.3285(5)	0.2496(5)	0.3948(5)	0.0351(2)
C13A	0.3658(5)	0.3152(4)	0.4446(5)	0.0357(2)
C14A	0.3910(4)	0.5189(4)	0.4635(6)	0.0310
C15A	0.3981(5)	0.5364(5)	0.3773(5)	0.0328
C16A	0.4232(4)	0.6149(5)	0.3640(5)	0.0299
C17A	0.4252(4)	0.6437(4)	0.2710(5)	0.0267(2)
C18A	0.4894(5)	0.6880(4)	0.2662(6)	0.0356(2)
C19A	0.4923(5)	0.7203(5)	0.1816(6)	0.0504(2)
C20A	0.4311(5)	0.7068(5)	0.1023(6)	0.0480(2)
C21A	0.3670(5)	0.6639(5)	0.1057(6)	0.0462(2)
C22A	0.3636(5)	0.6300(5)	0.1892(6)	0.0416(2)
C23A	0.3851(5)	0.4750(5)	0.3014(6)	0.0381(2)

$$^{a}U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

Table 6. Fractional Coordinates and Equivalent Isotropic Thermal Parameters for Non-Hydrogen Atoms in BRMEO Molecule B.

ATOM	X/A	Y/B	Z/C	Ueq. <sup>a</sup>
BR1B	0.0804(1)	0.3302(1)	0.4887(1)	0.0786
BR2B	0.3645(1)	0.1549(1)	0.6165(1)	0.0836
SB	-0.1416(2)	0.1348(2)	0.3945(2)	0.0474
OB	-0.1824(3)	0.0902(3)	0.5319(4)	0.0545
N1B	0.0052(4)	0.1648(4)	0.4468(5)	0.0467
N2B	-0.0150(4)	0.1811(4)	0.3529(5)	0.0535
C1B	-0.1301(5)	0.1792(5)	0.2169(6)	0.0421(2)
C2B	-0.2108(5)	0.1950(5)	0.1867(6)	0.0455(2)
C3B	-0.2471(6)	0.2048(5)	0.0910(6)	0.0480(2)
C4B	-0.2037(6)	0.1993(5)	0.0307(8)	0.0546(3)
C5B	-0.1240(6)	0.1832(5)	0.0579(8)	0.0595(3)
C6B	-0.0862(6)	0.1728(5)	0.1535(6)	0.0495(2)
C7B	-0.0894(5)	0.1681(5)	0.3177(6)	0.0451
C8B	0.0899(5)	0.1633(5)	0.4912(6)	0.0464(2)
C9B	0.1331(5)	0.2325(5)	0.5123(6)	0.0433(2)
C10B	0.2153(5)	0.2306(5)	0.5518(6)	0.0502(2)
C11B	0.2520(5)	0.1588(5)	0.5674(6)	0.0500(2)
C12B	0.2113(6)	0.0893(6)	0.5463(6)	0.0569(3)
C13B	0.1286(5)	0.0919(6)	0.5088(6)	0.0527(3)
C14B	-0.0523(5)	0.1410(5)	0.4862(6)	0.0403
C15B	-0.0477(5)	0.1240(5)	0.5762(6)	0.0463
C16B	-0.1178(6)	0.0959(5)	0.5968(8)	0.0485
C17B	-0.1182(5)	0.0720(5)	0.6902(6)	0.0481(2)
C18B	-0.1820(6)	0.0922(6)	0.7235(8)	0.0616(3)
C19B	-0.1850(8)	0.0699(8)	0.8112(9)	0.0832(4)
C20B	-0.1270(8)	0.0248(6)	0.8666(9)	0.0761(3)
C21B	0.0630(6)	0.0013(6)	0.8364(8)	0.0680(3)
C22B	-0.0593(6)	0.0244(6)	0.7482(8)	0.0642(3)
C23B	0.0252(6)	0.1471(6)	0.6559(8)	0.0574(3)

$$^{a}U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

Table 7. Calculated Hydrogen Positions in BRMEO Molecule A.

ATOM	X/A	Y/B	Z/C
HC2A	0.4237	0.6492	0.7191
HC3A	0.4327	0.6758	0.8850
HC4A	0.4246	0.5675	0.9883
HC5A	0.3827	0.4356	0.9207
HC6A	0.3773	0.4076	0.7579
HC10A	0.1499	0.3316	0.2895
HC12A	0.3597	0.1942	0.3989
HC13A	0.4267	0.3105	0.4878
HC18A	0.5380	0.6979	0.3283
HC19A	0.5426	0.7560	0.1784
HC20A	0.4343	0.7308	0.0365
HC21A	0.3182	0.6558	0.0434
HC22A	0.3136	0.5934	0.1911
H23Aa	0.4136	0.4945	0.2501
H23Ab	0.3220	0.4677	0.2687
H23Ac	0.4105	0.4195	0.3304

Table 8. Calculated Hydrogen Positions in BRMEO Molecule B.

ATOM	X/A	Y/B	Z/C
HC2B	-0.2454	0.1995	0.2359
HC3B	-0.3102	0.2168	0.0660
HC4B	-0.2329	0.2080	-0.0425
HC5B	-0.0908	0.1785	0.0073
HC6B	-0.0232	0.1600	0.1774
HC10B	0.2493	0.2843	0.5697
HC12B	0.2424	0.0336	0.5588
HC13B	0.0950	0.0378	0.4943
HC18B	-0.2304	0.1263	0.6796
HC19B	-0.2350	0.0885	0.8357
HC20B	-0.1307	0.0071	0.9345
HC21B	-0.0162	-0.0343	0.8805
HC22B	-0.0100	0.0049	0.7238
H23Ba	0.0073	0.1503	0.7191
H23Bb	0.0474	0.2038	0.6420
H23Bc	0.0716	0.1034	0.6639

Table 9. Anisotropic Thermal Parameters for BRMEO Molecule A.

ATOM	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
Br1A	0.0503(8)	0.0356(6)	0.0982(10)	0.0034(7)	0.0060(7)	0.0109(6)
Br2A	0.0821(10)	0.0465(7)	0.0700(9)	-0.0210(7)	0.0030(7)	-0.0230(7)
SA	0.040(1)	0.033(1)	0.022(1)	-0.005(1)	0.007(1)	-0.005(1)
OA	0.052(4)	0.042(4)	0.025(3)	-0.004(3)	0.006(3)	-0.016(3)
N1A	0.051(5)	0.024(4)	0.018(4)	-0.003(3)	0.008(3)	-0.007(3)
N2A	0.044(5)	0.039(5)	0.024(4)	-0.003(3)	0.008(3)	-0.007(3)
C7A	0.036(5)	0.038(5)	0.022(5)	-0.000(5)	0.003(4)	-0.000(5)
C14A	0.030(5)	0.024(5)	0.037(6)	-0.003(4)	0.006(4)	-0.000(4)
C15A	0.033(5)	0.054(7)	0.008(5)	-0.008(4)	0.001(4)	-0.005(5)
C16A	0.022(5)	0.036(6)	0.030(5)	-0.000(5)	0.006(4)	-0.005(4)

The form of the anisotropic temperature factor is  $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^{*}b^{*}\cos\delta^{*} + 2U_{13}hla^{*}c^{*}\cos\beta^{*} + 2U_{23}klb^{*}c^{*}\cos\alpha^{*})]$ .

Table 10. Anisotropic Thermal Parameters for BRMEO Molecule B.

ATOM	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
Br1B	0.0754(10)	0.0476(8)	0.1233(13)	0.0018(8)	0.0452(9)	0.0015(7)
Br2B	0.0408(7)	0.1109(12)	0.0800(10)	0.0549(9)	-0.0140(7)	-0.0201(8)
S <sub>B</sub>	0.037(1)	0.056(1)	0.043(1)	0.002(1)	0.002(1)	-0.009(1)
O <sub>B</sub>	0.042(4)	0.059(5)	0.055(5)	0.002(4)	0.001(3)	-0.007(3)
N1B	0.026(4)	0.061(6)	0.043(5)	0.005(4)	-0.006(4)	-0.007(4)
N2B	0.045(6)	0.069(7)	0.037(6)	0.007(4)	-0.005(4)	-0.011(5)
C7B	0.046(7)	0.043(6)	0.043(6)	-0.000(5)	0.008(5)	-0.005(5)
C14B	0.032(6)	0.039(6)	0.049(7)	0.001(5)	0.009(5)	-0.007(5)
C15B	0.039(6)	0.046(6)	0.045(7)	0.012(5)	-0.004(5)	-0.012(5)
C16B	0.054(7)	0.038(6)	0.048(7)	0.001(5)	0.006(6)	-0.001(6)

The form of the anisotropic temperature factor is  $\exp[-2\pi^2(U_{11}h^2a^*{}^2 + U_{22}k^2b^*{}^2 + U_{33}l^2c^*{}^2 + 2U_{12}hka^*b^*\cos\delta^* + 2U_{13}hka^*c^*\cos\beta^* + 2U_{23}kbl^*c^*\cos\alpha^*)]$ .

Table 11. Important Bond Distances and Angles in BRMEO, with Estimated Standard Deviations in Parentheses Molecule A.

Bond	Distance, Å	Bond	Distance, Å
SA-C7A	1.723(10)	C14A-SA	1.758(9)
C7A-N1A	1.299(12)	C14A-C15A	1.363(14)
C7A-C1A	1.482(13)	C15A-C16A	1.434(14)
N1A-N2A	1.396(11)	C16A-C17A	1.485(14)
N2A-C8A	1.442(10)	C16A-OA	1.250(11)
N2A-C14A	1.369(12)	OA---SA	2.483(6)

Atoms	Angle, °	Atoms	Angle, °
C7A-SA-C14A	89.7(5)	N2A-C14A-C15A	129.7(9)
SA-C7A-C1A	121.7(7)	SA-C14A-C15A	124.0(7)
SA-C7A-N1A	117.3(7)	C14A-C15A-C16A	116.6(8)
C1A-C7A-N1A	121.0(9)	C15A-C16A-C17A	121.7(8)
C7A-N1A-N2A	107.2(8)	C15A-C16A-OA	120.9(9)
N1A-N2A-C8A	113.1(7)	C17A-C16A-OA	117.4(8)
N1A-N2A-C14A	119.4(7)	C16A-OA---SA	101.5(5)
C8A-N2A-C14A	126.4(7)	OA---SA-C14A	76.8(3)
N2A-C14A-SA	106.3(7)	C7A-SA---OA	166.5(6)

Table 12. Important Bond Distances and Angles in BRMEO, with Estimated Standard Deviations in Parentheses Molecule B.

Bond	Distance, Å	Bond	Distance, Å
SB-C7B	1.752(12)	C14B-SB	1.766(9)
C7B-N1B	1.277(13)	C14B-C15B	1.356(16)
C7B-C1B	1.486(13)	C15B-C16B	1.430(17)
N1B-N2B	1.376(12)	C16B-C17B	1.457(16)
N2B-C8B	1.442(12)	C16B-OB	1.267(12)
N2B-C14B	1.363(15)	OB---SB	2.478(7)
Atoms	Angle, °	Atoms	Angle, °
C7B-SB-C14B	88.8(5)	N2B-C14B-C15B	130.4(9)
SB-C7B-C1B	121.2(8)	SB-C14B-C15B	123.3(8)
SB-C7B-N1B	116.4(8)	C14B-C15B-C16B	118.1(9)
C1B-C7B-N1B	122.4(1.1)	C15B-C16B-C17B	122.5(9)
C7B-N1B-N2B	108.9(9)	C15B-C16B-OB	119.5(1.0)
N1B-N2B-C8B	114.0(9)	C17B-C16B-OB	118.0(1.1)
N1B-N2B-C14B	119.5(7)	C16B-OB---SB	101.9(4)
C8B-N2B-C14B	125.7(8)	OB---SB-C14B	76.9(3)
N2B-C14B-SB	106.3(7)	C7B-SB---OB	165.7(6)

Table 13. Bond Distances in the Phenyl Rings of BRMEO Molecule A.

Bond	Distance, Å	Bond	Distance, Å
C1A-C2A	1.375(14)	C11A-C12A	1.394(12)
C2A-C3A	1.409(17)	C11A-Br2A	1.884(9)
C3A-C4A	1.346(18)	C12A-C13A	1.395(13)
C4A-C5A	1.400(18)	C13A-C8A	1.377(13)
C5A-C6A	1.379(19)	C17A-C18A	1.371(14)
C6A-C1A	1.382(16)	C18A-C19A	1.394(15)
C8A-C9A	1.383(12)	C19A-C20A	1.373(13)
C9A-C10A	1.390(13)	C20A-C21A	1.350(16)
C9A-Br1A	1.882(19)	C21A-C22A	1.393(15)
C10A-C11A	1.357(14)	C22A-C17A	1.401(12)

Table 14. Bond Distances in the Phenyl Rings of BRMEO Molecule B.

Bond	Distance, Å	Bond	Distance, Å
C1B-C2B	1.382(14)	C11B-C12B	1.367(16)
C2B-C3B	1.401(14)	C11B-Br2B	1.898(10)
C3B-C4B	1.338(18)	C12B-C13B	1.396(15)
C4B-C5B	1.363(16)	C13B-C8B	1.377(15)
C5B-C6B	1.407(15)	C17B-C18B	1.388(18)
C6B-C1B	1.382(17)	C18B-C19B	1.383(19)
C8B-C9B	1.384(14)	C19B-C20B	1.354(17)
C9B-C10B	1.392(14)	C20B-C21B	1.380(20)
C9B-Br1B	1.883(10)	C21B-C22B	1.395(18)
C10B-C11B	1.366(15)	C22B-C17B	1.400(15)

Table 15. Bond Angles in the Phenyl Rings of BRMEO Molecule A.

Atoms	Angle, °	Atoms	Angle, °
C1A-C2A-C3A	120.5(1.1)	C9A-C10A-C11A	119.7(0.9)
C2A-C3A-C4A	120.2(1.1)	C10A-C11A-C12A	121.2(0.9)
C3A-C4A-C5A	119.2(1.2)	C10A-C11A-Br2A	120.0(0.7)
C4A-C5A-C6A	120.8(1.3)	C12A-C11A-Br2A	118.8(0.7)
C5A-C6A-C1A	119.7(1.1)	C11A-C12A-C13A	118.4(0.9)
C6A-C1A-C2A	119.3(1.0)	C12A-C13A-C8A	120.8(0.8)
C7A-C1A-C2A	119.6(0.9)	C17A-C18A-C19A	119.8(0.9)
C7A-C1A-C6A	121.1(0.9)	C18A-C19A-C20A	120.3(0.9)
C13A-C8A-C9A	119.3(0.8)	C19A-C20A-C21A	120.6(1.1)
N2A-C8A-C9A	122.1(0.8)	C20A-C21A-C22A	120.0(1.0)
N2A-C8A-C13A	119.3(0.8)	C21A-C22A-C17A	120.3(0.8)
C8A-C9A-C10A	120.5(0.9)	C22A-C17A-C18A	119.0(0.9)
C8A-C9A-Br1A	120.2(0.7)	C16A-C17A-C18A	123.2(0.9)
C10A-C9A-Br1A	119.3(0.7)	C16A-C17A-C22A	117.8(0.7)

Table 16. Bond Angles in the Phenyl Rings of BRMEO Molecule B.

Atoms	Angle, °	Atoms	Angle, °
C1B-C2B-C3B	118.9(1.1)	C9B-C10B-C11B	118.2(1.0)
C2B-C3B-C4B	120.0(1.0)	C10B-C11B-C12B	122.8(1.0)
C3B-C4B-C5B	122.7(1.0)	C10B-C11B-Br2B	118.8(0.8)
C4B-C5B-C6B	118.3(1.2)	C12B-C11B-Br2B	118.3(0.8)
C5B-C6B-C1B	119.7(1.0)	C11B-C12B-C13B	118.5(1.0)
C6B-CB1-CB2	120.3(0.9)	C12B-C13B-C8B	120.1(1.0)
C7B-C1B-C2B	120.5(1.1)	C17B-C18B-C19B	121.7(1.2)
C7B-C1B-C6B	119.2(0.9)	C18B-C19B-C20B	119.1(1.1)
C13B-C8B-C9B	119.8(0.9)	C19B-C20B-C21B	120.3(1.3)
N2B-CB8-CB9	120.9(0.9)	C20B-C21B-C22B	120.4(1.4)
N2B-C8B-C13B	119.3(0.9)	C21B-C22B-C17B	121.9(1.1)
C8B-C9B-C10B	120.6(1.0)	C22B-C17B-C18B	116.4(1.1)
C8B-C9B-Br1B	119.9(0.7)	C16B-C17B-C18B	123.6(1.1)
C10B-C9B-Br1B	119.5(0.8)	C16B-C17B-C22B	119.9(0.9)

Table 17. Deviations from Least Squares Planes for BRMEO Molecule A.

Atom	Deviation, Å	Atom	Deviation, Å
<b>Thiadiazolene ring: <math>0.90368X - 0.34248Y - 0.25703Z = 3.09605</math></b>			
SA*	0.005(2)	C7A*	0.000(7)
N1A*	-0.010(6)	C15A	0.025(8)
N2A*	-0.000(6)	C16A	-0.033(8)
C14A*	-0.002(7)	OA	0.010(5)
<b>C1A-C6A ring: <math>0.95340X - 0.20415Y + 0.22214Z = 4.17641</math></b>			
C1A*	-0.003(9)	C4A*	0.024(11)
C2A*	0.004(10)	C5A*	-0.023(14)
C3A*	-0.014(11)	C6A*	0.013(11)
<b>C8A-C13A ring: <math>-0.57156X - 0.27293Y + 0.77384Z = 0.89821</math></b>			
C8A*	-0.001(7)	C11A*	0.002(8)
C9A*	0.004(8)	C12A*	0.000(8)
C10A*	-0.005(9)	C13A*	-0.001(8)
<b>C17A-C22A ring: <math>-0.53694X + 0.82234Y + 0.18826Z = 6.3365</math></b>			
C17A*	0.006(7)	C20A*	-0.007(9)
C18A*	-0.001(8)	C21A*	0.012(9)
C19A*	0.001(9)	C22A*	-0.011(9)

The equations are of the form  $AX + BY + CZ = 0$ , where X, Y, and Z are orthogonal coordinates in a system with the axes parallel to a\*, b\*, and c\*. Asterisks designate atoms used to calculate the planes.

Table 18. Deviations from Least Squares Planes for BRMEO Molecule B.

Atom	Deviation, Å	Atom	Deviation, Å
Thiadiazolene ring: $-0.27547X + 0.94639Y + 0.16873Z = 4.25648$			
S <sub>B</sub> *	0.007(3)	C <sub>7B</sub> *	0.014(9)
N <sub>1B</sub> *	-0.004(7)	C <sub>15B</sub>	0.032(9)
N <sub>2B</sub> *	-0.029(7)	C <sub>16B</sub>	-0.005(10)
C <sub>14B</sub> *	0.004(9)	O <sub>B</sub>	-0.019(5)
C <sub>1B</sub> -C <sub>6B</sub> ring: $0.17615X + 0.98006Y + 0.09191Z = 2.70109$			
C <sub>1B</sub> *	-0.003(9)	C <sub>4B</sub> *	0.004(10)
C <sub>2B</sub> *	-0.001(9)	C <sub>5B</sub> *	-0.002(10)
C <sub>3B</sub> *	-0.003(9)	C <sub>6B</sub> *	-0.002(9)
C <sub>8B</sub> -C <sub>13B</sub> ring: $-0.39805X - 0.01507Y + 0.91724Z = 6.6277$			
C <sub>8B</sub> *	-0.001(9)	C <sub>11B</sub> *	-0.000(9)
C <sub>9B</sub> *	-0.006(9)	C <sub>12B</sub> *	-0.007(10)
C <sub>10B</sub> *	0.006(9)	C <sub>13B</sub> *	0.007(9)
C <sub>17B</sub> -C <sub>22B</sub> ring: $0.39905X + 0.82570Y + 0.39872Z = 2.94990$			
C <sub>17B</sub> *	0.015(9)	C <sub>20B</sub> *	0.002(13)
C <sub>18B</sub> *	-0.013(11)	C <sub>21B</sub> *	0.000(11)
C <sub>19B</sub> *	0.003(11)	C <sub>22B</sub> *	-0.009(11)

The equations are of the form  $AX + BY + CZ = 0$ , where X, Y, and Z are orthogonal coordinates in a system with the axes parallel to a\*, b\*, and c\*. Asterisks designate atoms used to calculate the planes.

Table 19. Fractional Coordinates and Equivalent Isotropic Thermal Parameters for Non-Hydrogen Atoms in BRPETO.

ATOM	X/A	Y/B	Z/C	U <sub>eq</sub> . <sup>a</sup>
BR1	0.3792(1)	0.2825(1)	0.4434(1)	0.0702
BR2	0.4658(1)	0.9019(1)	0.3827(1)	0.1033
S	0.0274(2)	0.0941(2)	0.3304(1)	0.0459
O	0.0769(4)	-0.0268(6)	0.2394(2)	0.0642
N1	0.0843(5)	0.3275(8)	0.4007(3)	0.0601
N2	0.1492(4)	0.3367(8)	0.3557(3)	0.0595
C1	-0.0591(5)	0.1687(9)	0.4358(3)	0.0461
C2	-0.1362(6)	0.0466(10)	0.4245(3)	0.0643
C3	-0.2078(8)	0.0056(11)	0.4642(3)	0.0747
C4	-0.2010(8)	0.0887(11)	0.5164(3)	0.0745
C5	-0.1255(8)	0.2128(12)	0.5293(3)	0.0739
C6	-0.0539(6)	0.2518(10)	0.4888(3)	0.0626
C7	0.0186(5)	0.2098(9)	0.3939(3)	0.0466
C8	0.2252(5)	0.4669(10)	0.3601(3)	0.0489
C9	0.3286(5)	0.4647(8)	0.3978(3)	0.0443
C10	0.4008(6)	0.5929(8)	0.4049(3)	0.0512
C11	0.3653(8)	0.7252(9)	0.3723(3)	0.0579
C12	0.2608(8)	0.7318(10)	0.3368(3)	0.0629
C13	0.1887(6)	0.6044(11)	0.3298(3)	0.0637
C14	0.1339(5)	0.2212(9)	0.3131(3)	0.0492
C15	0.1879(6)	0.1996(10)	0.2641(3)	0.0563
C16	0.1524(5)	0.0634(8)	0.2280(3)	0.0446
C17	0.2089(5)	0.0239(9)	0.1756(3)	0.0482
C18	0.2883(8)	-0.0952(12)	0.1791(3)	0.0798
C19	0.3366(8)	-0.1390(14)	0.1282(4)	0.0846
C20	0.3068(8)	-0.0609(12)	0.0761(3)	0.0688
C21	0.2270(9)	0.0544(12)	0.0713(3)	0.0787
C22	0.1775(6)	0.0995(11)	0.1202(3)	0.0630
C23	0.2738(11)	0.3278(19)	0.2382(5)	0.1264
C24	0.3740(18)	0.2777(19)	0.2656(5)	0.1827

$$^a U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i * a_j * a_i a_j$$

$$a = 11.749(1) \text{ \AA} \quad b = 8.532(1) \text{ \AA} \quad c = 22.669(4) \text{ \AA}$$

$$\alpha = \gamma = 90^\circ, \beta = 100.17^\circ(1)$$

*z* = 4, Space group = P2<sub>1</sub>/c.

3939 reflections measured on a CAD-4 diffractometer to  $\theta_{max} = 25^\circ$ .

2265 with  $I > 3\sigma(I)$ , R=0.058, 273 parameters refined.

Table 20. Calculated Hydrogen Positions in BRPETO.

ATOM	X/A	Y/B	Z/C
HC2	-0.1409	-0.0187	0.3833
HC3	-0.2692	-0.0891	0.4543
HC4	-0.2540	0.0551	0.5487
HC5	-0.1234	0.2797	0.5699
HC6	0.0067	0.3473	0.4989
HC10	0.4827	0.5901	0.4353
HC12	0.2330	0.8394	0.3135
HC13	0.1052	0.6108	0.3011
HC18	0.3152	-0.1557	0.2211
HC19	0.3972	-0.2351	0.1316
HC20	0.3464	-0.0914	0.0381
HC21	0.2027	0.1146	0.0290
HC22	0.1130	0.1911	0.1149
H23a	0.2594	0.3258	0.1898
H23b	0.2561	0.4439	0.2534
<hr/>			
Positions found on difference map.			
H24a	0.4227	0.2684	0.3183
H24b	0.4233	0.1741	0.2370
H24c	0.4282	0.3993	0.2457
<hr/>			

Table 21. Anisotropic Thermal Parameters for BRPETO.

ATOM	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
Br1	0.088(0.7)	0.046(0.5)	0.081(0.7)	0.014(0.4)	0.028(0.5)	0.003(0.5)
Br2	0.135(1.0)	0.049(0.6)	0.142(1.0)	-0.009(0.6)	0.066(0.9)	-0.029(0.6)
S	0.047(1)	0.055(1)	0.037(1)	-0.003(0.9)	0.011(0.8)	-0.017(3)
O	0.074(3)	0.072(3)	0.052(3)	-0.021(2)	0.025(2)	-0.017(3)
N1	0.059(4)	0.077(4)	0.050(3)	-0.030(3)	0.026(3)	-0.027(3)
N2	0.059(4)	0.073(4)	0.054(3)	-0.026(3)	0.030(3)	-0.025(3)
C1	0.038(4)	0.060(4)	0.040(4)	0.001(3)	0.006(3)	0.002(3)
C2	0.070(5)	0.061(5)	0.068(5)	-0.014(4)	0.029(4)	-0.010(4)
C3	0.089(6)	0.064(6)	0.081(6)	-0.011(4)	0.041(5)	-0.022(5)
C4	0.086(6)	0.072(6)	0.077(6)	0.021(5)	0.046(5)	0.012(5)
C5	0.077(6)	0.094(7)	0.059(5)	0.003(5)	0.034(4)	-0.008(5)
C6	0.069(5)	0.077(6)	0.047(4)	-0.005(3)	0.025(4)	-0.009(4)
C7	0.040(3)	0.070(5)	0.031(3)	-0.003(3)	0.007(3)	-0.001(3)
C8	0.050(4)	0.065(5)	0.035(4)	-0.013(3)	0.018(3)	-0.011(3)
C9	0.059(4)	0.031(3)	0.046(4)	-0.001(3)	0.017(3)	0.006(3)
C10	0.054(4)	0.044(4)	0.059(4)	-0.005(3)	0.017(3)	-0.004(3)
C11	0.073(5)	0.042(4)	0.068(5)	0.002(4)	0.036(4)	-0.005(4)
C12	0.081(6)	0.062(5)	0.053(5)	0.012(4)	0.032(4)	0.014(4)
C13	0.082(5)	0.091(7)	0.037(4)	-0.002(4)	0.010(3)	0.003(5)
C14	0.048(4)	0.056(4)	0.044(4)	-0.004(3)	0.009(3)	-0.008(3)
C15	0.060(5)	0.067(5)	0.048(4)	-0.010(3)	0.025(3)	-0.019(4)
C16	0.039(4)	0.051(4)	0.042(4)	-0.001(3)	0.002(3)	0.005(3)
C17	0.044(4)	0.056(4)	0.048(4)	-0.010(3)	0.015(3)	-0.001(3)
C18	0.074(6)	0.109(8)	0.060(5)	0.008(5)	0.022(4)	0.031(5)
C19	0.064(6)	0.109(8)	0.086(7)	-0.024(6)	0.026(5)	0.020(5)
C20	0.070(5)	0.097(7)	0.043(5)	-0.020(4)	0.018(4)	-0.013(5)
C21	0.105(8)	0.086(7)	0.049(5)	0.011(4)	0.025(5)	0.004(6)
C22	0.070(5)	0.074(5)	0.048(4)	0.004(4)	0.020(4)	0.008(4)
C23	0.113(10)	0.174(13)	0.087(9)	-0.080(9)	0.003(8)	-0.002(9)
C24	0.292(27)	0.172(17)	0.060(8)	-0.036(8)	-0.035(12)	0.036(16)

The form of the anisotropic temperature factor is given in Table 9.

Table 22. Important Bond Distances and Angles in BRPETO, With Estimated Standard Deviations in Parentheses.

Bond	Distance, Å	Bond	Distance, Å
S-C7	1.763(7)	C15-C23	1.631(15)
C7-N1	1.259(9)	C23-C24	1.326(18)
C7-C1	1.472(9)	C14-C15	1.384(10)
N1-N2	1.380(8)	C15-C16	1.441(10)
N2-C8	1.418(9)	C16-C17	1.497(10)
N2-C14	1.369(9)	C16-O	1.235(8)
C14-S	1.752(7)	O---S	2.466(5)

Atoms	Angle, °	Atoms	Angle, °
C7-S-C14	89.0(3)	N2-C14-C15	129.8(7)
S-C7-C1	121.7(6)	S-C14-C15	122.9(6)
S-C7-N1	115.1(5)	C14-C15-C16	115.9(7)
C1-C7-N1	123.2(6)	C15-C16-C17	120.5(7)
C7-N1-N2	111.2(6)	C15-C16-O	121.9(7)
N1-N2-C8	114.5(6)	C17-C16-O	117.5(6)
N1-N2-C14	117.4(6)	C16-O---S	101.5(4)
C8-N2-C14	128.1(6)	O---S-C14	77.7(3)
N2-C14-S	107.3(5)	C7-S---O	166.7(4)

Table 23. Bond Distances in the Phenyl Rings of BRPETO.

Bond	Distance, Å	Bond	Distance, Å
C(1)-C(2)	1.374(10)	C(11)-C(12)	1.345(12)
C(2)-C(3)	1.381(11)	C(11)-Br(2)	1.904( 8)
C(3)-C(4)	1.369(12)	C(12)-C(13)	1.370(12)
C(4)-C(5)	1.379(12)	C(13)-C(8)	1.388(11)
C(5)-C(6)	1.390(11)	C(17)-C(18)	1.372(11)
C(6)-C(1)	1.388(10)	C(18)-C(19)	1.422(12)
C(8)-C(9)	1.355(10)	C(19)-C(20)	1.348(13)
C(9)-C(10)	1.377(10)	C(20)-C(21)	1.350(13)
C(9)-Br(1)	1.903( 7)	C(21)-C(22)	1.393(12)
C(10)-C(11)	1.374(10)	C(22)-C(17)	1.403(10)

Table 24. Bond Angles in the Phenyl Rings of BRPETO.

Atoms	Angle, <sup>o</sup>	Atoms	Angle, <sup>o</sup>
C(1)-C(2)-C(3)	121.9(8)	C(9)-C(10)-C(11)	117.9(7)
C(2)-C(3)-C(4)	118.9(8)	C(10)-C(11)-C(12)	121.0(7)
C(3)-C(4)-C(5)	121.2(8)	C(10)-C(11)-Br(2)	117.4(7)
C(4)-C(5)-C(6)	119.0(9)	C(12)-C(11)-Br(2)	121.5(6)
C(5)-C(6)-C(1)	120.7(8)	C(11)-C(12)-C(13)	121.0(8)
C(6)-C(1)-C(2)	118.3(7)	C(12)-C(13)-C(8)	118.9(8)
C(7)-C(1)-C(2)	121.7(7)	C(17)-C(18)-C(19)	120.8(9)
C(7)-C(1)-C(6)	120.0(7)	C(18)-C(19)-C(20)	120.1(9)
C(13)-C(8)-C(9)	119.2(7)	C(19)-C(20)-C(21)	120.0(8)
N(2)-C(8)-C(9)	121.1(7)	C(20)-C(21)-C(22)	121.2(8)
N(2)-C(8)-C(13)	119.4(7)	C(21)-C(22)-C(17)	120.3(8)
C(8)-C(9)-C(10)	120.9(9)	C(22)-C(17)-C(18)	117.5(7)
C(8)-C(9)-Br(1)	120.9(6)	C(16)-C(17)-C(18)	120.9(7)
C(10)-C(9)-Br(1)	117.3(6)	C(16)-C(17)-C(22)	117.8(7)

Table 25. Deviations From Least Squares Planes for BRPETO.

Atom	Deviation, Å	Atom	Deviation, Å
Thiadiazolene ring: $0.62593X - 0.55218Y + 0.55074Z = 2.99698$			
S*	-0.006(2)	C7*	0.004(7)
N1*	0.000(6)	C15	0.028(7)
N2*	-0.006(6)	C16	0.055(6)
C14*	0.008(7)	O	0.037(5)
C1-C6 ring: $0.62807X - 0.62878Y - 0.45844Z = 2.02022$			
C1*	0.000(7)	C4*	0.004(8)
C2*	-0.000(7)	C5*	-0.004(9)
C3*	-0.002(8)	C6*	0.002(7)
C8-C13 ring: $0.56686X + 0.31699Y + 0.76039Z = 6.71088$			
C8*	-0.020(7)	C11*	-0.021(8)
C9*	-0.009(6)	C12*	0.009(8)
C10*	0.012(7)	C13*	0.011(8)
C17-C22 ring: $0.70087X - 0.66039Y + 0.26963Z = 2.41201$			
C17*	0.007(7)	C20*	0.015(9)
C18*	0.001(9)	C21*	-0.007(9)
C19*	-0.012(10)	C22*	-0.004(8)

The equations are of the form  $AX + BY + CZ = 0$ , where X, Y, and Z are orthogonal coordinates in a system with the axes parallel to a, b, and c. Asterisks designate atoms used to calculate the planes.

#### IV. Discussion

##### A. The Molecular Structures of BRPHO, BRMEO and BRPETO.

The numerical data for BRPHO was obtained through Dr.M.F. Richardson. All the numerical information for BRMEO and BRPETO is listed in Tables 5-25 of the results section.

The molecules were drawn in a specific manner. The five-membered thiadiazolene fragment was considered to lie on the plane of the paper (plane formed by C7-S-C14). This way one can observe the relative deviations of the substituents with respect to this central fragment.

For BRMEO, the molecular structures of the independent molecules A and B, together with the adopted numbering scheme for all three structures are shown in figures 25 and 26 respectively. Figure 27 exhibits the four molecules (for the three structures) that will be described shortly.

Crystals of BRMEO contain two different conformers. This unusual feature, unexpected when the structure was begun, will be discussed further in subsequent sections.

##### 1 Bonds, Angles and Least Squares Planes.

The average C-C distance for the phenyl rings was calculated to be

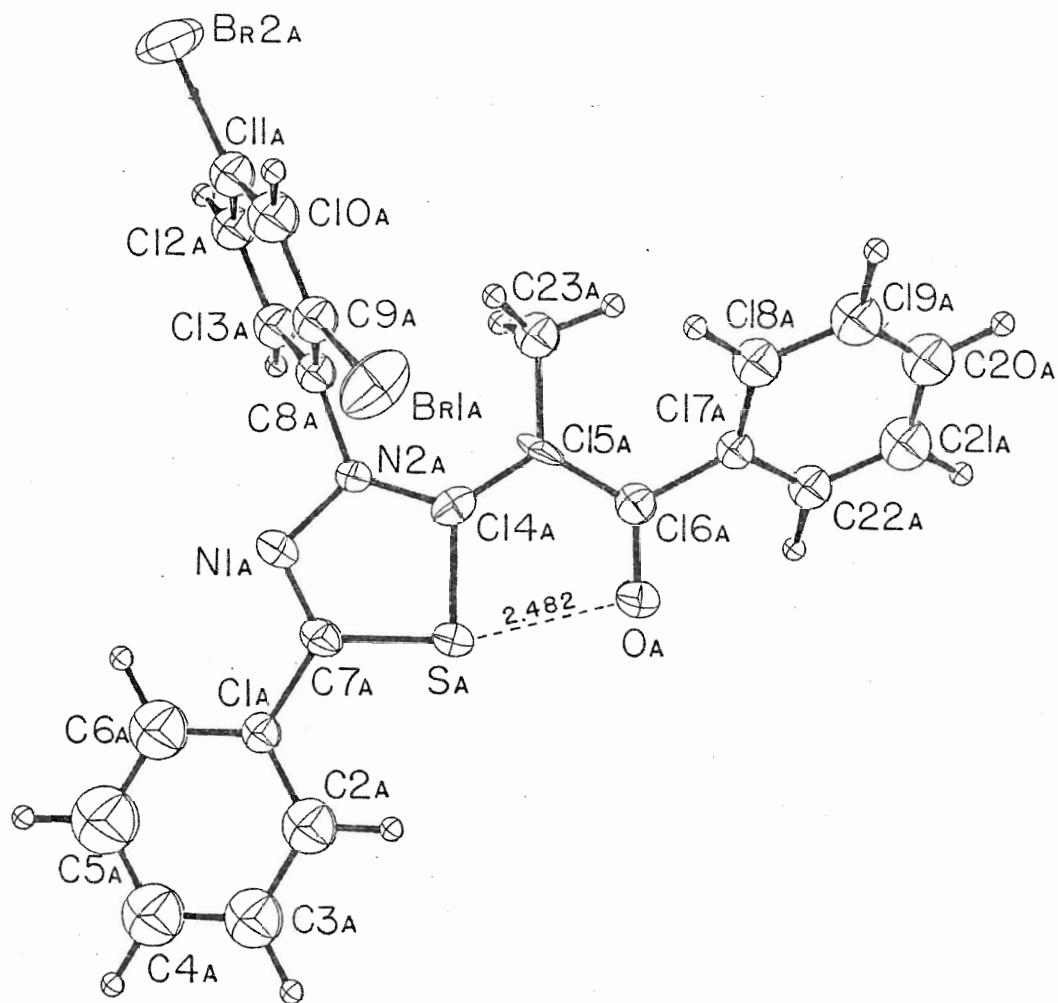
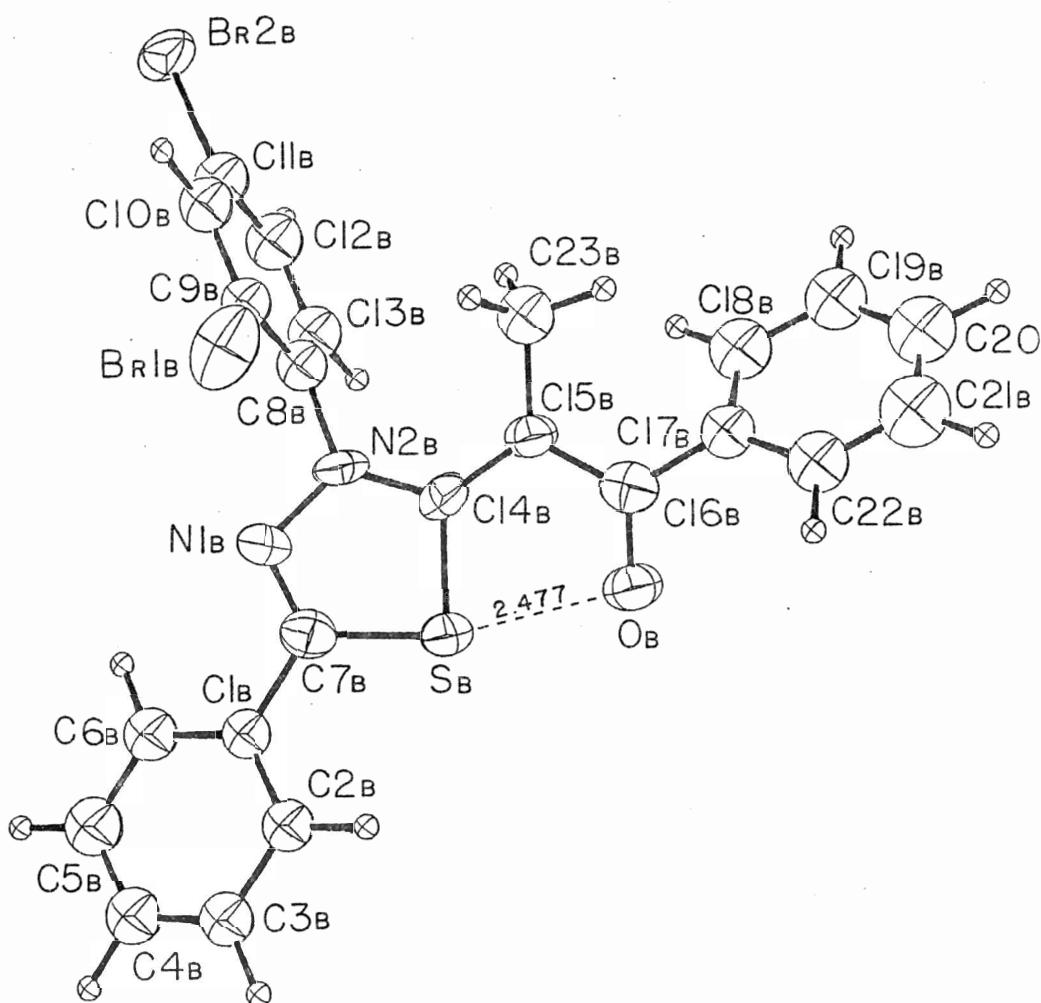


Figure 25. The Molecular Structure of Molecule A, BRMEO.

Figure 26. The Molecular Structure of Molecule B, BRMEO.



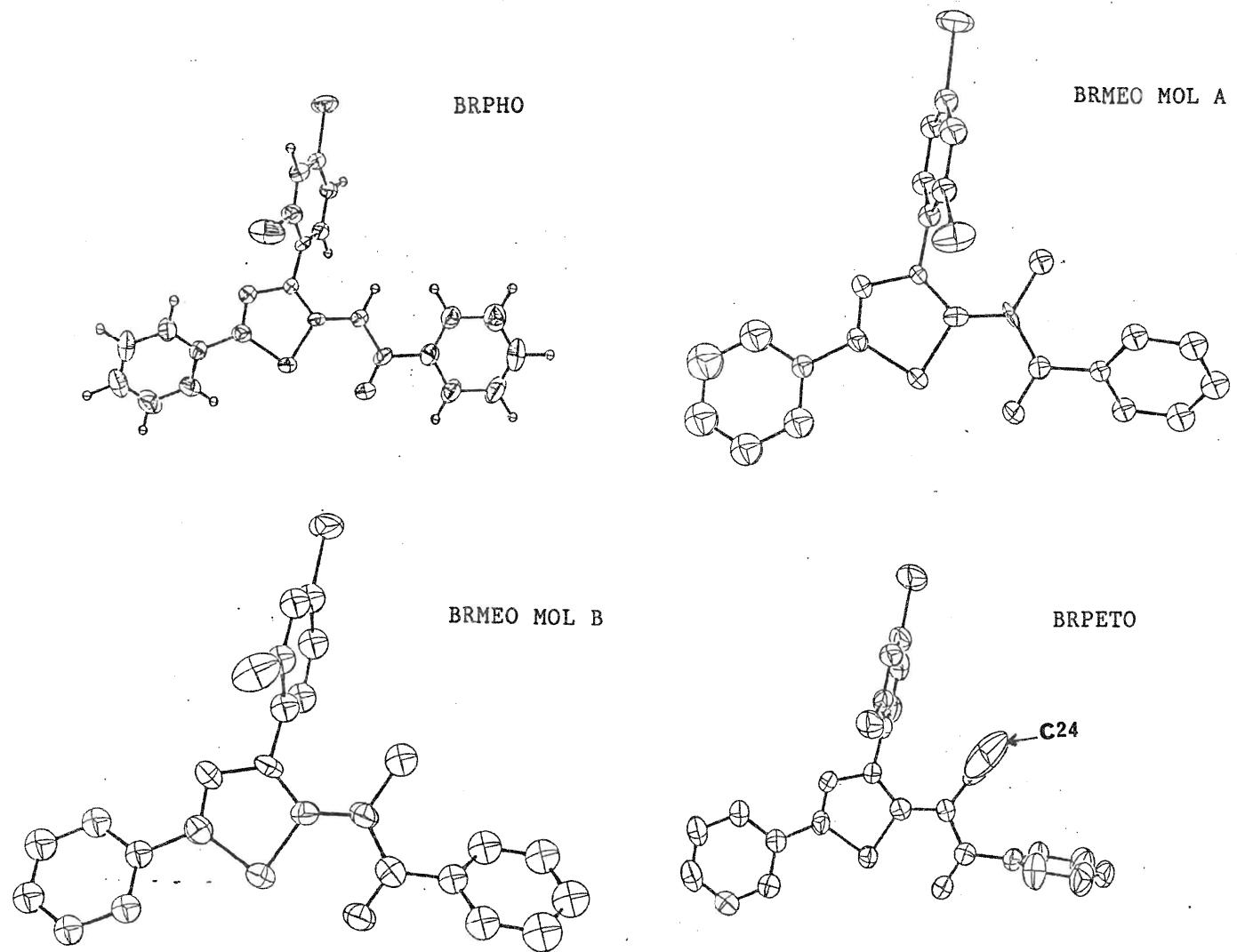


Figure 27. The Molecular Structures of BRPHO, BRMEO and BRPETO.

1.38 $\text{\AA}$  for all three structures. In general the C-C distances for the phenyl rings is 1.39 $\text{\AA}$ , so the average observed value is within experimental error. Librational motions which are a result of collecting structural data at room temperature are responsible for the odd short or long bonds. Domenicano et al. have studied these effects in greater detail (There are other random errors, plus systematic electronic effects as discussed also by Domenicano et al.).<sup>31-32</sup> The bond angles are in general those expected for  $sp^2$  hybridized atoms ( $120^\circ$ ). The phenyl rings are all planar with the largest average deviation being 0.024(11) $\text{\AA}$  for BRMEO (molecule A) (Tables 17, 18 and 25).

The C-Br distances for the 2,4-dibromophenyl rings lie within the expected values of 1.88-1.90 $\text{\AA}$ .<sup>33</sup>

The thiadiazolene fragment for BRMEO was found to be planar within the limits of the analysis for molecules A and B. The largest deviation observed was -0.029(7) $\text{\AA}$  for N2B of molecule B. BRPETO and BRPHO also exhibit planarity of the thiadiazolene fragment with average deviations of 0.006 and 0.016 $\text{\AA}$  respectively. The sum of the angles about N2 were calculated to be  $360^\circ$  indicating its planarity. An expected S-C bond length is 1.80 $\text{\AA}$ <sup>33</sup>, but the observed S-C lengths are 1.723(10) vs 1.758(9) $\text{\AA}$  (S-C7 vs S-C14) and 1.752(12) vs 1.766(9) $\text{\AA}$  for BRMEO molecules A and B, 1.763(7) vs 1.752(7) for BRPETO, and 1.750(6) vs 1.735(6) for BRPHO. These values are less because there exists some double bond character from the delocalization of electrons in the ring system.

The deviation from coplanarity of the carbonyl oxygen from the thiadiazolene plane changes from -0.067 $\text{\AA}$  for BRPHO to a value approximately six times less for BRMEO (molecule A). For all three structures C15 and C16 are significantly deviated from the thiadiazolene plane (Tables 17, 18 and 25).

The benzoyl fragment to the right of the molecules has been mentioned before by Nyburg et al. All three structures have bond distances that lie within the quoted intervals for this fragment (see Tables 11 and 12; intervals quoted by the paper are: C15-C16 1.393-1.49 $\text{\AA}$ , C16-O 1.216-1.306 $\text{\AA}$  and C16-C17 1.44-1.495 $\text{\AA}$ )<sup>20</sup>.

## 2 Torsion angles.

The monosubstituted phenyls C1-C6 are essentially coplanar with the thiadiazolene fragment for BRPETO and BRMEO (molecule A). The larger torsion angles (N1-C7-C1-C6, see table 28) of -37.9 and 24.8° for BRPHO and BRMEO (molecule B), can probably be attributed to the benefit gained by better packing.

The torsion angles (C15-C16-C17-C18), for the phenyl on the benzoyl group go from being essentially zero for BRPHO to a value of 99.6° for BRPETO. This can be due to the steric crowding that exists between the R-group and the phenyl ring: as R becomes larger (H < Me < Et), the phenyl ring C17-C22 twists increasingly out of the plane of the thiadiazolene fragment (see figure 27).

Table 28. Torsion Angles for BRPHO, BRMEO and BRPETO.

	BRPHO (°)	BRMEO MOL A (°)	BRMEO MOL B (°)	BRPETO (°)
N(1)-N(2)-C(8)-C(9)	62.9	103.4	62.0	80.4
N(1)-C(7)-C(1)-C(6)	-37.9	9.1	24.8	-6.6
C(15)-C(16)-C(17)-C(18)	16.5	44.4	-45.1	99.6

The 2,4-dibromophenyl at N2 is in no instance coplanar with the central thiadiazolene fragment (see N1-C7-C1-C6, Table 28). One can explain this in terms of steric crowding between the R-group and the bromine (Br1) at C9, and also to the inability of Br1 to stay coplanar with the thiadiazolene ring because of steric crowding from the lone pair of electrons on N1.

## B. Packing of BRMEO, BRPHO and BRPETO.

Figures 28, 29 and 30 represent the stereoscopic views of the molecular packing for BRMEO, BRPHO and BRPETO respectively, projected onto the ac-plane. Although only the structure of BRMEO was determined for this work, it was thought necessary to include the packing diagrams of the other two structures, in order to find constant features.

It is evident from figure 28 that BRMEO has two independent molecules in the asymmetric unit, arranged with their thiadiazolene rings nearly perpendicular to each other.

Molecule A has its four symmetry related molecules positioned approximately centrally within the unit cell, where molecules A1 and A2 are related by the c-glide, A1 and A3 are related by the  $2_1$  screw axis and A2 and A3 are related by the centre of symmetry. The "double layer" nature of the packing is evident; infinite sheets of molecules are parallel to the bc-plane. Both the thiadiazolene moiety and phenyl ring (C1A-C6A) are almost coplanar and lie approximately parallel to the bc-plane. The C17A-C22A phenyl is twisted such that it lies neither on this plane or the plane perpendicular to it. The 2,4-dibromophenyl substituents lie approximately parallel to the (10 $\bar{1}$ ) plane, coming out or going into the plane of the paper.

Molecule B has its four symmetry related molecules lying parallel to

Figure 28. The Stereoscopic AC-Projection for the Molecular Packing of BRMEO.

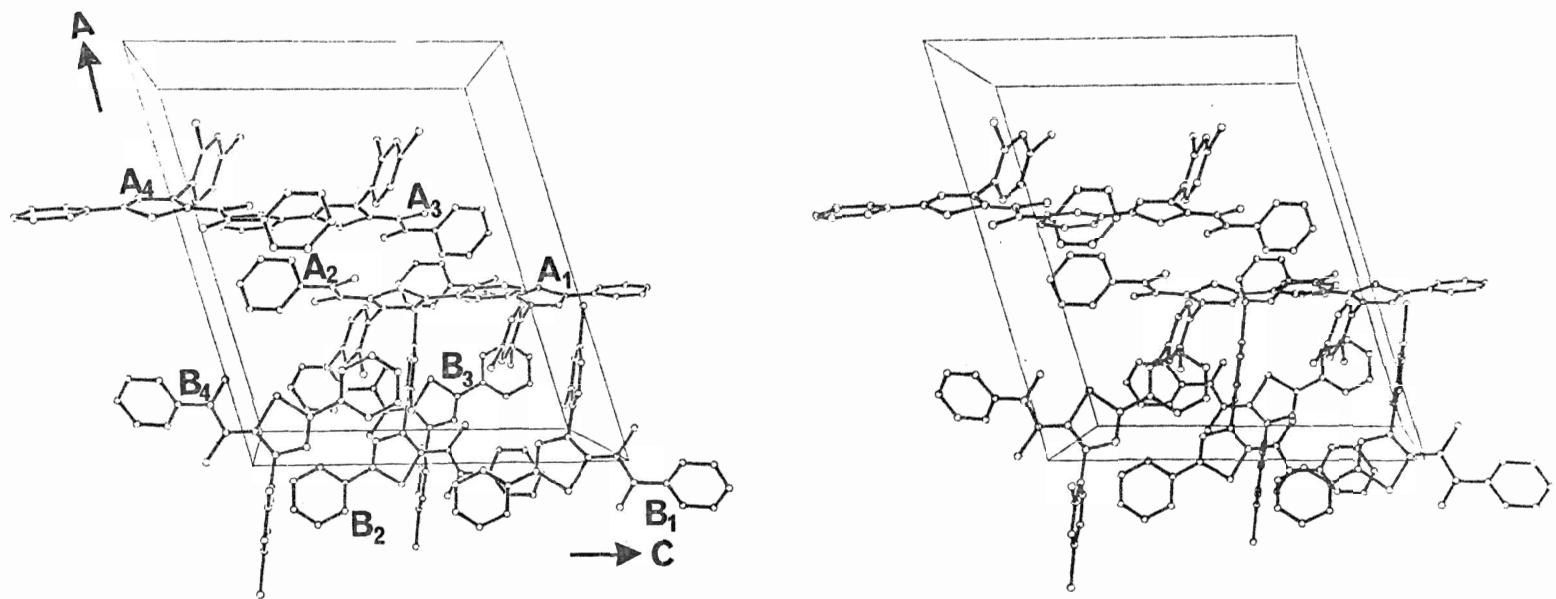


Figure 29. The Stereoscopic AC-Projection for the Molecular Packing of BRPHO.

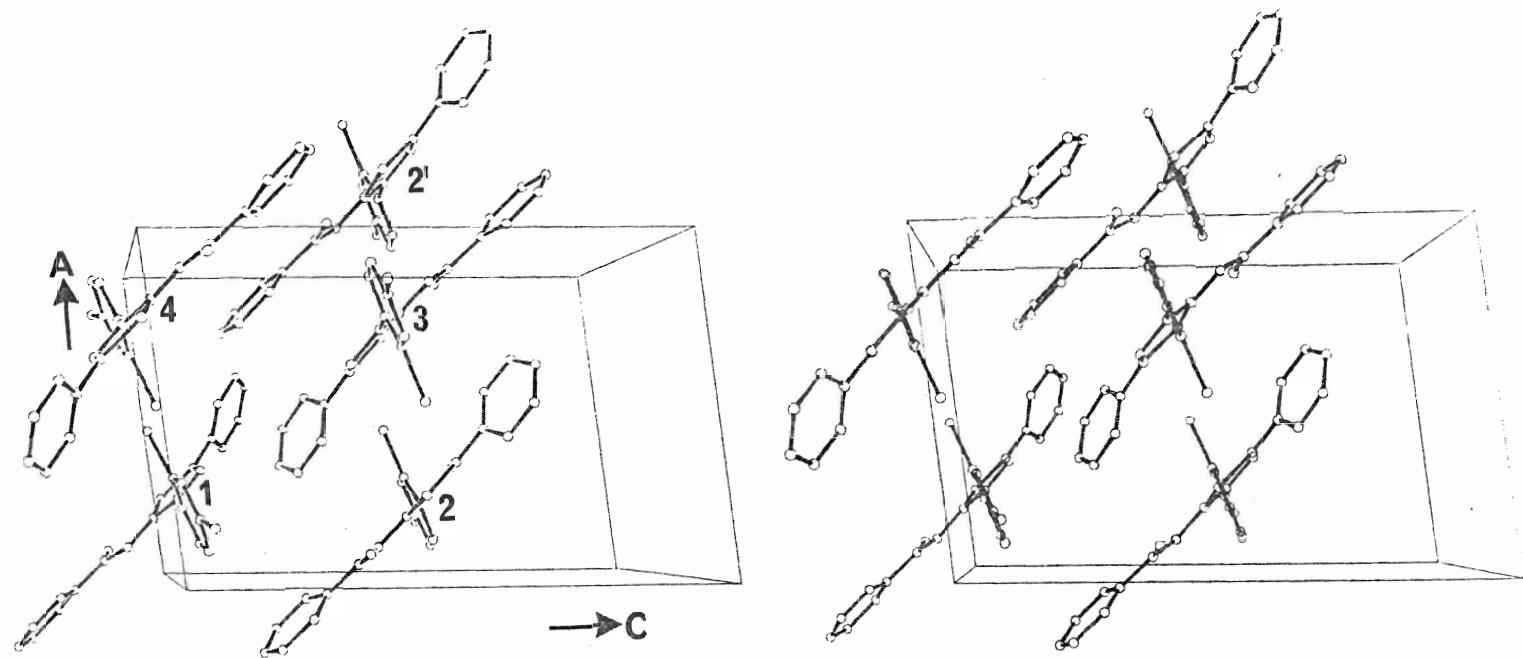
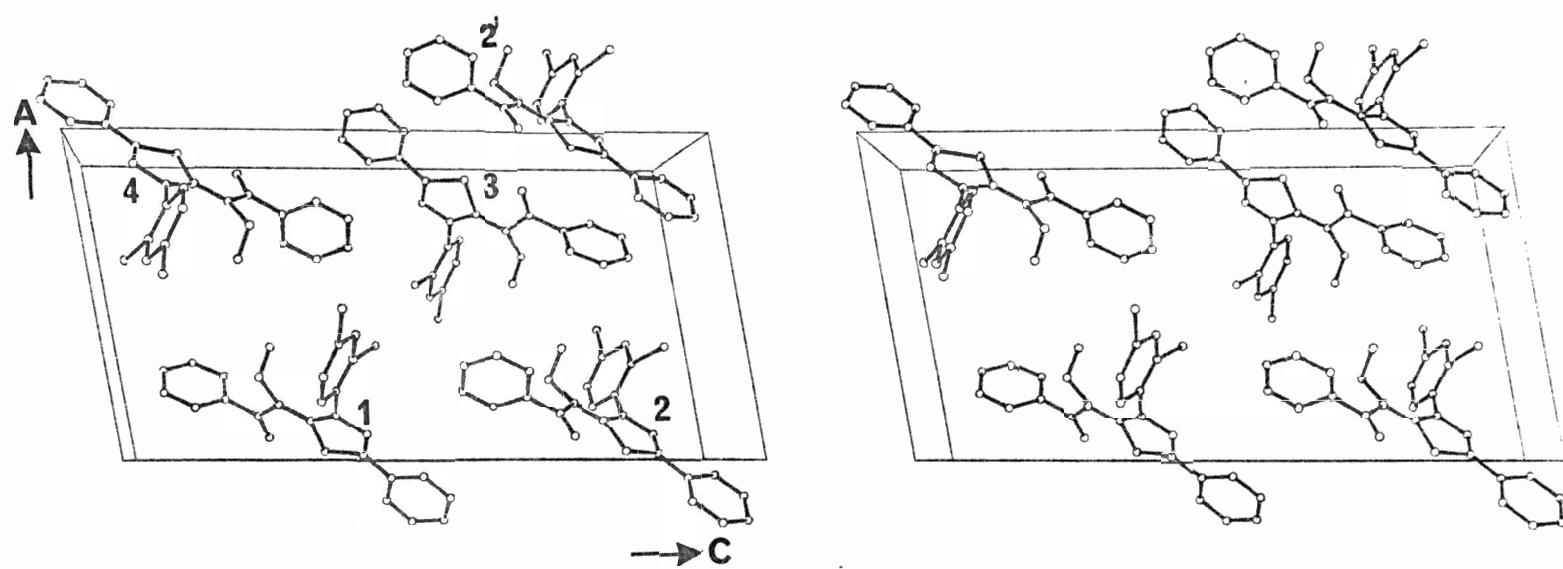


Figure 30. The Stereoscopic AC-Projection for the Molecular Packing of BRPETO.



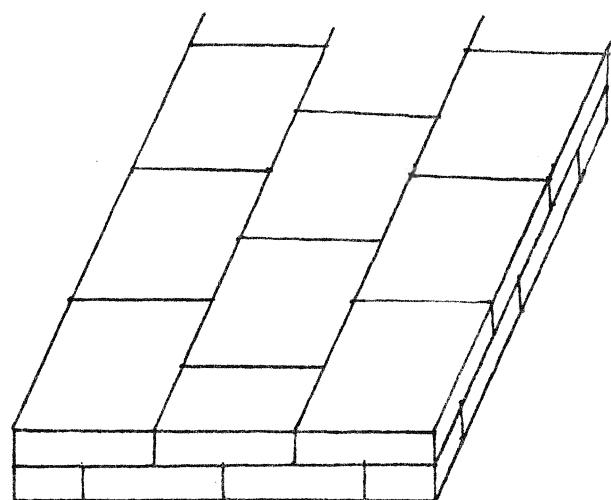
the ac-plane, where B1 and B2 are related by the c-glide, B1 and B3 related by the  $2_1$  screw axis and B2 and B3 related by the centre of symmetry. Here one can see good ring stacking of the molecules. One has corrugated columns parallel to the y-axis interlinked to a thick sheet. The 2,4-dibromophenyl groups are able to efficiently fill the vacant space in between the two different conformers.

The packing diagrams of BRPHO and BRPETO reflect rather different packing arrangements with respect to BRMEO. The packing diagram for BRPHO in figure 29 shows the molecules packing approximately along the the  $(10\bar{1})$  plane. Here molecules 1 and 2 are related by the c-glide, 2 and 3 are related by the centre of symmetry and 1 and 3 related by the  $2_1$  screw axis.

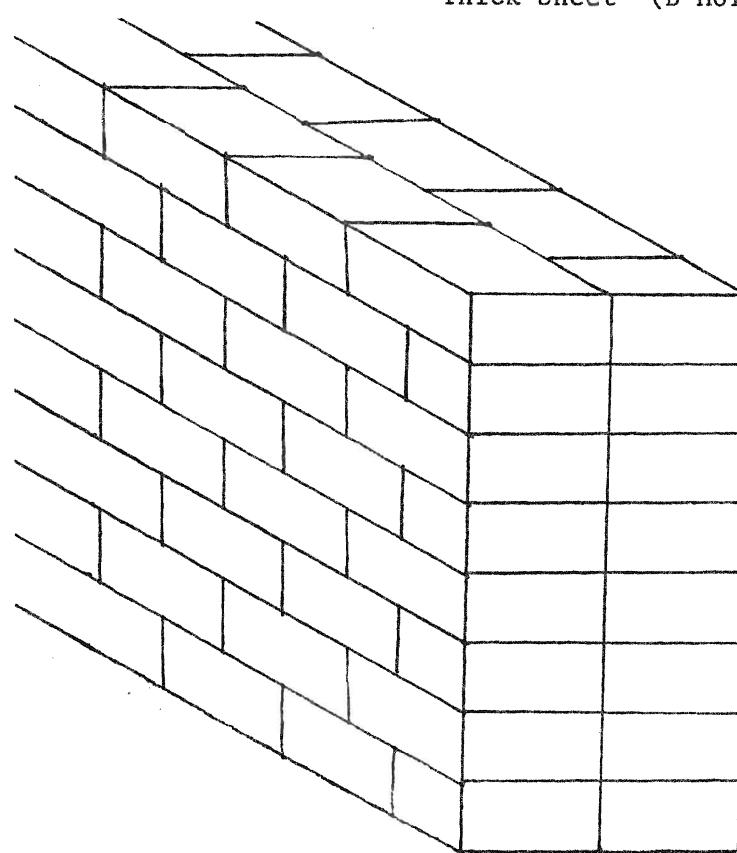
Figure 30 depicts the packing for BRPETO where, molecules 1 and 2 are related by the c-glide, 1 and 3 by the centre of symmetry and 2 and 3 by the  $2_1$  screw axis. The molecules are approximately aligned about the  $(101)$  plane.

In neither BRPHO nor BRPETO are there definable sheets or columns of molecules as there are in BRMEO. However, the B molecules of BRMEO do bear some resemblance to the BRPHO and BRPETO packings, in that there are continuous parallel stacks of thiadiazolene rings. The A molecules of BRMEO are only packed in double layers. The packing in BRMEO is analogous to stacking bricks in two different ways to form walls, as shown in the scheme on the following page.

Double Layer (A Molecules)



Thick Sheet (B Molecules)

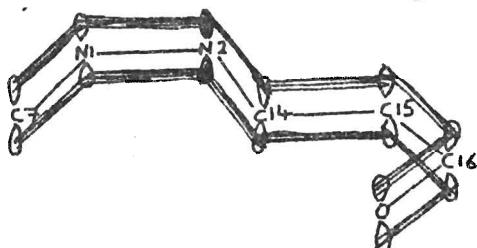


### C. The Putative Ring Closure Reaction

The putative ring closure reaction envisaged to occur, infers changes in certain bond lengths and angles. Figure 24 (p. 39) exhibits the major changes expected from this cyclization process. The position of the present structures along the reaction pathway between the end members can be deduced from the distances and angles.

The S---O distances are 2.629(5), 2.483(6), 2.478(7) and 2.466(5) $\text{\AA}$  for BRPHO, BRMEO (molecules A and B) and BRPETO respectively. There is a decrease in distance as one goes from R=H (BRPHO) to R=Alkyl (BRMEO and BRPETO). These values are indicative of an interaction albeit weak between the atoms. This is further justified by the almost linear bond angles about C7-S-O, which lie in the interval  $163.6^\circ$  (BRPHO) to  $166.7^\circ$  (BRPETO). One interesting feature that stands out from the findings is the fact that the S---O distances for the two different conformers in BRMEO are equal within experimental error.

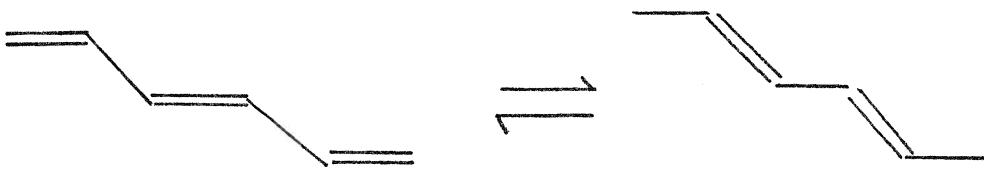
It is difficult to distinguish normal conjugated effects from other effects which result from the ring closure. That is, conjugation as shown, shortens the nominal single bonds and lengthens double bonds.



N<sub>2</sub> planar and can participate in conjugation.

The Cambridge database was used to observe how much of an effect

conjugation has on the bond lengths (see Appendix II). A fragment very similar to the type shown above was searched. A total of 124 hits were observed. The mean distances clearly correspond to bonds that have partial double bond character. It could easily be argued that these distances would lie in between double and single bonds if the data were averaged over two distinct types of structures as shown in the scheme below:



By a visual inspection of the data it was clear that a broad single distribution of the data was present rather than a double hump.

However, there are still some significant distances and angles to look at: the S---O and C=O distances, and the interior angles in the bicyclic system (since angles are not generally affected by conjugation).

If the suggested transformation is the true course of reaction then the bond length of S-C14 should decrease as one goes from a single to a double bond. However this is not what is actually observed for BRMEO. In fact the distances for each conformer are larger than the S-C7 distance (Molecule A 1.723(10) vs 1.756(10) $\text{\AA}$ ; Molecule B 1.752(12) vs 1.766(9) $\text{\AA}$  for S-C7 vs S-C14 in each case).

The C14-C15 and C15-C16 bond lengths do validate the expected

transformation. A decrease in double bond character is expected for C14-C15, and increased bonding is expected for C15-C16. Both bond lengths are intermediate between those expected for single and double bonds. However, these changes could simply be due to the conjugation effects mentioned earlier.

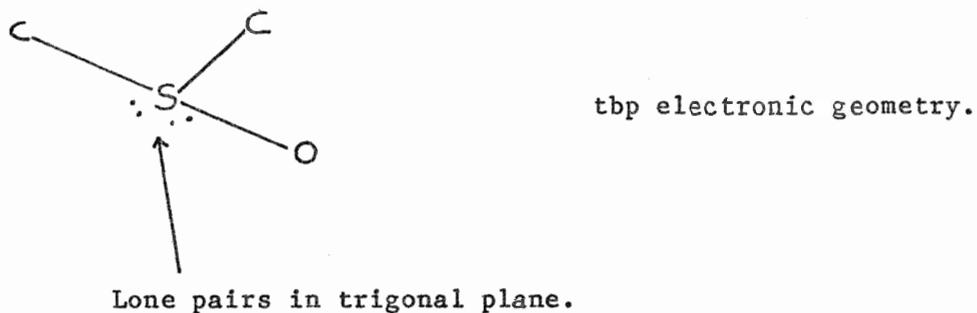
The carbonyl bond would be expected to increase in length as the oxygen tries to bond to the  $sp^3$  hybridized sulphur. The distances observed for molecules A and B are comparable within experimental error (C16=O: Molecule A 1.250(11); Molecule B 1.267(12) $\text{\AA}$ ). These values are only slightly longer than the expected C=O bond length of 1.23 $\text{\AA}$ .<sup>33</sup> This observation is consistent with the statement made in the introduction, the decrease in the length of the longer intermolecular interaction A---B (S---O) from the value expected for a normal van der Waals contact is generally much greater than the accompanying increase in the length of the shorter, intramolecular interaction B-C (C=O).

The bond angles about C14-C15-C16 are  $116^\circ$  and  $118^\circ$  for molecules A and B respectively. Although these values do not provide much information on what kind of changes are actually taking place, their reduction from  $120^\circ$  is consistent with the proposed cyclization.

The angle about S-C14-C15 for the three compounds BRMEO, BRPHO and BRPETO changes from  $125.4^\circ$  for BRPHO to  $122.9^\circ$  for BRPETO as the size of the R-group increases from R=H to R=Alkyl. It appears that this particular change in angle represents a major change and something that could be looked for in subsequent structures.

The fragment comprising S,C7,C14 and O in the bicyclic system would be expected to be nonplanar due to the presence of a lone pair of electrons on the sulphur. However, the fragment is found to be planar for both conformers.

From the envisaged transformation the hybridization of S would be expected to be  $sp^3$ . In fact our data do not indicate this type of hybridization; also the distances are not in general consistent with those expected. The transformation points towards a case where the lone pair of electrons on the oxygen is donated to the sulphur but no subsequent movement of electrons taking place. So there would exist a negative formal charge on sulphur and a positive formal charge on oxygen. This implies an  $sp^3d$  hybridization of sulphur (trigonal bipyramidal geometry):



C-S---O angle expected to be less than  $180^\circ$  from VSEPR considerations.

Now if we turn our attention to how these observed S---O distances for BRMEO plus those for BRPHO and BRPETO fit onto an energy/reaction

coordinate diagram, it is evident that distances between that of the van der Waals ( $3.25\text{\AA}$ ) and that expected for a single bond ( $1.5\text{\AA}$ ) are observed for all three cases. This is also the case for other compounds that were outlined in the introduction on the section entitled "The S---O interaction" and also for numerous compounds found from the Cambridge database search. The S---S bonding appears to resemble that observed for the S---O bonding. This has already been observed and quoted by Nyburg et al.<sup>20</sup>

The structure correlation method implies the obvious assumption that we can infer relative energies from relative frequencies of occurrence, the less frequent, the higher the energy above a reference state.

This has been seen in the examples mentioned in the introduction. For nucleophilic attack at C=O if one looks at Burgi's data one sees a distribution that has no points at the transition state (i.e.  $r_1=r_2$ ). There is a big extrapolation involved to get the transition state, since the C=O bond has not really stretched in any of the structures studied. The points obtained cover only a small portion of the potential energy diagram. (close to starting material minimum).

From the data for  $I_3^-$ , one concludes that  $I_3^-$  is the most stable structure since a cluster of points are observed for  $r_1=r_2$ . This means that the reaction coordinate pathway is represented by a minimum transition state instead of a maximum. So one can infer from the cluster of distances at  $r_1=r_2$  that the "energy of activation" is negative. (i.e. the proposed  $I_3^-$  intermediate is actually more stable

than the reactants).

From our findings an S---O interaction exists without any doubt, albeit a weak one. The envisaged ring closure has not occurred. All that can be said is that a small step along the potential energy surface has been taken. The steric effect due to the R-group, which was pointed out at the onset, has actually been a contributing factor in initiating the ring closure. Electronic effects could also be applied to initiate the ring closure. By replacing the C1-C6 phenyl by an electron withdrawing group (like p-nitrophenyl) it would be possible to pull the electrons on sulphur. This way the sulphur would become slightly electropositive and consequently make the S---O interaction more plausible. On the other hand the C17-C22 phenyl could be replaced by an even stronger electron donating group such as t-butyl and make the carbonyl oxygen more electronegative. This will again aid in forming the bicyclic compound.

V. Conclusion

The crystal structure of  $C_{23}H_{16}Br_2N_2OS$  (BRMEO) has been determined by the heavy atom method. Two different conformers that pack well together have been observed.

Although an S---O interaction is observed, that increases as one goes from R=H (BRPHO) to R=Alky (BRMEO and BRPETO), the envisaged putative ring closure has not occurred. The steric effect played by the R-group has played a role in the activation of the S---O interaction. It seems reasonable to assume that a more bulky group like t-butyl would aid in forming the bicyclic compound.

REFERENCES

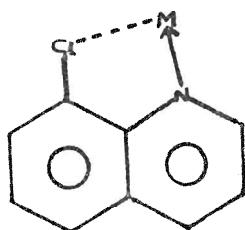
- (1) H.B. Burgi and J.D. Dunitz, Acc. Chem. Res., 16, 153-161 (1983).
- (2) J.D. Dunitz, "X-Ray Analysis and the Structure of Organic Molecules", Cornell University: Ithaca, N.Y., Chapter 9, (1979).
- (3) H.B. Burgi, Angew. Chem., 87, 461 (1975).
- (4) P. Murray-Rust, H.B. Burgi, and J.D. Dunitz, J. Am. Chem. Soc., 97, 921 (1975).
- (5) J.D. Dunitz, Philo. Trans. R. Soc. London, B272, 99-108 (1975).
- (6) N.W. Alcock, Adv. Inorg. Radiochem., 15, 1 (1972).
- (7) H.A. Bent, Chem. Rev., 68, 587 (1968).
- (8) F.W.B. Einstein and A.C. MacGregor, J. C. S. Dalton, 778 (1974).
- (9) B. Liu, J. Chem. Phys., 58, 1925 (1973).
- (10) Ref. (2). P. 347.
- (11) Ref. (2). P. 345.
- (12) H.B. Burgi, Inorg. Chem., 12, 2321 (1973).
- (13) H.B. Burgi, J.D. Dunitz, and E. Shefter, J. Amer. Chem. Soc., 95, 5065-5067 (1973).
- (14) H.B. Burgi, J.D. Dunitz, and E. Shefter, Acta Cryst., B30, 1517 (1974).
- (15) J.D. Wallis and J.D. Dunitz, J. Chem. Soc., Chem. Commun., 671 (1984).
- (16) M. Kaftory and I. Agmon, J. Am. Chem. Soc., 106, 7785-7793 (1984).
- (17) M. Kaftory, J. Am. Chem. Soc., 105, 3832-3836, (1983).
- (18) M. Mammi, R. Bardi, G. Traverso and S. Bezzi, Nature, Lond, 192 1282 (1961).
- (19) N. Lozac'h Adv. Heterocycl. Chem., 13, 161-234 (1971).
- (20) T.R. Lynch, I.P. Mellor, and S.C. Nyburg, Acta Cryst., B27, 1948 (1971).

- (21) A. Kalman and L. Parkanyi, Acta Cryst., B36, 2372 (1980).
- (22) P.L. Johnson and I.C. Paul, Chem. Commun., 1014 (1969).
- (23) F. Leung and S.C. Nyburg, Can. J. Chem., 50, 324 (1972).
- (24) R.E. Rosenfield, Jr., R. Parthasarathy, and J.D. Dunitz, J. Am. Chem. Soc., 99, 4860-4862 (1977).
- (25) C. Cohenaddad, M.S. Lehmann, P. Becker, L. Parkanyl and A. Kalman, J. Chem. Soc. Perkin Trans. 2, 191 (1984).
- (26) M.F. Richardson, Unpublished data.
- (27) P.D. Callaghan, A.J. Elliott, S.S. Gandhi, M.S. Gibson, H. Mastalerz and D.J. Vukov, J. C. S. Perkin 1, 2948 (1981).
- (28) CONVI written by M.F. Richardson and C.R. Paige (1982).
- (29) G.M. Sheldrick, SHELX 83, University Chemical Laboratory, Cambridge, England (1983).
- (30) O. Kennard in International Tables for X-ray Crystallography, Vol IV, The Kynoch Press, Birmingham, England (1968).
- (31) A. Domenicano and P. Murray-Rust, Tetrahedron Letters, 24, 2283-2286 (1979).
- (32) A. Domenicano, P. Murray-Rust and A. Vaciago, Acta Cryst., B39, 457-468 (1983).
- (33) C. R. C. Handbook, 64th ed, Chemical Rubber Company (1983-84).

**Appendix I: Initial Project**

Initial Project: Synthesis of Metal Complexes of 8-Chloroquinoline.

The initial project was also concerned with the structural correlation method. The aim of the project was to make metal complexes of 8-chloroquinoline and determine from their structures the possibility of M-Cl interaction.



When the Cambridge database\* was searched, no compounds of these type were found. However information on metal complexes of 8-hydroxyquinoline was found. Here one observes the ligand bonding through both the deprotonated hydroxyl group and also with the nitrogen in order to form various four and six coordinate complexes.

The procedure used for synthesising metal complexes of pyridine was utilized here, since in both cases one has a nitrogen donor available. Equi-molar solutions of 8-chloroquinoline (ligand) and various metal chlorides and nitrates were mixed and left to evaporate. Solvents that could dissolve both reactants were used when ever possible. Infra-red and melting point analysis were carried out to determine possible product formation. In no case was desired product observed.

The next approach was to first make Metal(acac)<sub>2</sub> (acac = acetyl acetonate) and then react that with the ligand. Since the ligand is

more basic than the acac one would expect the ligand to displace it.  
This again proved to be unfruitful.

Finally to test if the lone pair on the nitrogen is available, the ligand was reacted with hydrochloric acid. When this failed to yield the hydrochloride it was concluded that the lone pair is not available for donation and the project was terminated.

\* CAMBRIDGE DATA CENTRE, UNIVERSITY CHEMICAL LABORATORY,  
LENSFIELD ROAD, CAMBRIDGE, ENGLAND.

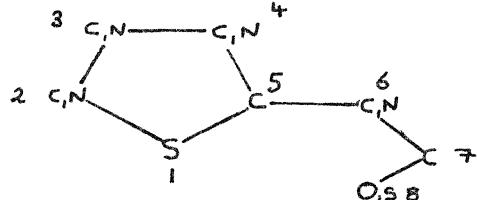
Appendix II<sup>c</sup> Cambridge Database Search

The Cambridge database was utilized to find two types of information that were of importance to my thesis work. The two searches carried out were:

- (1) To find related thiadiazolene type structures.
- (2) To see what effect conjugation has on bond lengths.

1. To find Related Thiadiazolene Type Structures .

In order to carry out the first search the following fragment was introduced using the CONNSER subroutine:



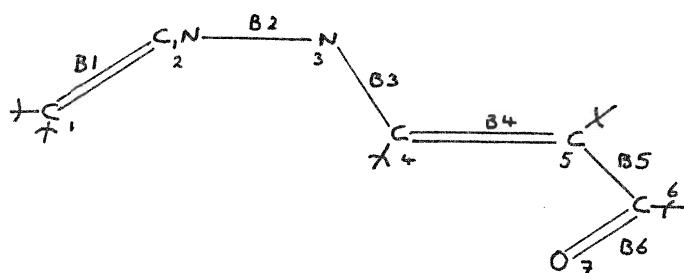
The search gave a total of 89 hits. Bibliographic and geometrical information was obtained by using the BIB and GEOM subroutines respectively. Due to the enormous amount of data presented by this search, it was thought necessary to narrow down the amount of information by applying constraints. The two constraints applied were to firstly accept only those structures whose residual factors were less than or equal to 5% (this would ensure good data) and secondly accept structures where the S---O or S---S interaction distance was less than the sum of the van der Waals radii for the respective bonds. These constraints reduced the number of structures to 11, where 5 structures were for the S---O case and 6 for the S---S.

From a combination of looking at the data presented by the search plus finding the actual structures from the literature, it was possible to make some generalizations as to what factors govern the degree of S---O(S) interactions. It is evident that the two types of interactions (S---O(S)) are very similar. The C,N-S---O,S angle becomes more linear and the coplanarity of the O,S with the five-membered ring increases as the interaction distance deceases. A more electronegative atom than sulphur when placed adjacent to it results in increased interaction.

Also it was noted that different substituents with differing electron donating abilities have an effect on the interaction. These factors are discussed at greater length in the Introduction.

2. To see What Effect Conjugation has on Bond Lengths .

The second search was carried out by introducing the following fragment:



The search yielded 124 hits. Below the mean distances of the 124 structures are presented together with the standard deviations associated with the distances.

B1

B2

B3

B4

B5

B6

Mean(Å)	1.419	1.398	1.382	1.389	1.467	1.264
---------	-------	-------	-------	-------	-------	-------

Std Dev	0.069	0.043	0.043	0.057	0.036	0.068
---------	-------	-------	-------	-------	-------	-------

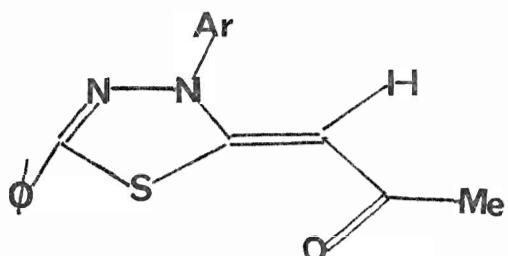
Expected	1.33	1.47(C-N)	1.47	1.37	1.54	1.24
distance.(Å)			1.44(N-N)			

Appendix III: BRMEHO

## BRMEHO

1. Compound Synthesis, Crystal Selection and  
Mounting on CAD-4 Diffractometer

The compound 2-acetylmethyleno-3-(2,4-dibromophenyl)-2,3-dihydro-5-phenyl-1,3,4-thiadiazole (BRMEHO):



used for this particular study was obtained through Dr M.S. Gibson. It was previously synthesized by the method outlined in reference 1. The compound was tan-coloured and the crystals were well shaped prisms.

The criteria used for selecting the crystal were regularity of shape, the absence of visible cracks or surface irregularities and the sharpness of the extinctions when the crystal was rotated in the presence of polarized light under a microscope.

The crystal chosen was centrosymmetric and suggested a centrosymmetric space group. It was mounted on a thin glass fibre using Lepage's Five Minute Epoxy and the fibre was subsequently glued onto a brass pin with household cement such that the glass fibre was approximately 6mm in length from the edge of the pin. Preliminary

<sup>1</sup>P.D. Callaghan, A.J. Elliott, S.S. Gandhi, M.S. Gibson,  
H. Mastalerz and D.J. Vukov, J. C. S. Perkin 1, 2948 (1981).

photographic studies using a Weissenberg camera were not undertaken.

Instead, the pin was mounted directly on a goniometer head and centered on the CAD-4 diffractometer for data collection.

## 2.

## Preliminary Work

The crystal size of  $0.50 \times 0.40 \times 0.25$  mm was measured using the calibrated telescope on the diffractometer. Intensities were measured on an Enraf-Nonius CAD-4 diffractometer controlled by a PDP-8a computer. Graphite monochromatized Mok<sub>2</sub> radiation was used ( $\lambda = 0.71073\text{\AA}$ ). Unit cell parameters were obtained by the measurement of 25 reflections for  $9.8^\circ < \theta < 18.1^\circ$ . The parameters obtained were consistent with a primitive triclinic system, although a transformation could be chosen which gave dimensions consistent with a C-centred monoclinic cell. To determine the correct system a preliminary data collection was undertaken to  $\theta_{\max} = 10^\circ$  for the monoclinic cell. If the system is actually monoclinic, the following intensities will be equivalent:

$$I_{hkl} = I_{\bar{k}\bar{l}\bar{h}} = I_{\bar{h}\bar{k}\bar{l}} = I_{\bar{h}\bar{k}\bar{l}}$$

When the data were examined the only equivalent reflections noted were for  $I_{hkl} = I_{\bar{k}\bar{l}\bar{h}}$ . Thus the system is not monoclinic, but triclinic. To safeguard against the possibility of having missed some reflections a further data collection over  $2^\circ < \theta < 6^\circ$  was carried out with all the cell axes doubled. It was evident from the output that they were missing and so the cell dimensions are correct as shown (see Table 1a) for the triclinic system.

The absorption coefficient and the optimum size were calculated using

the following equations and absorption coefficients for MoK $\alpha$  radiation taken from the International Tables for X-Ray Crystallography<sup>2</sup> :

$$\mu_{pd} = d \sum (f_r)_j (\mu)_j$$

d = density (g/cm<sup>3</sup>)

Here  $(\mu)_j$  absorption coefficient of element j at a given wavelength, and  $(fr)_j$  is the fraction of j in the compound.

The value of  $\mu_{pd}$  was calculated to be 50.47 cm<sup>-1</sup>.

$$\text{optimum size} = 2/\mu_{pd} = 2/50.47 = 0.0396\text{cm}$$

It is common practice to keep the size of the crystal to less than 0.5mm to ensure that it lies within the x-ray beam ( $\sim 0.5\text{mm}$ ).

The density was calculated to be 0.86g/cc for two formula units in the unit cell. Since this value is approximately half that of BRMEO it was concluded that again there exists two molecules in the asymmetric unit, so the density is 1.72g/cc with four formula units/unit cell. The correctness of this deduction was verified when the density was measured to be 1.70g/cc using the flotation method (H<sub>2</sub>O/saturated KI).

<sup>2</sup>O. Kennard in International Tables for X-ray Crystallography, Vol III, The Kynoch Press, Birmingham, England (1968).

3. Data collection.

The data collection was carried out to a  $\Theta_{\max}$  of 25°. A set of three reflections (5 3 3; 3 0 -5 and -1 2 0) were measured every two hours as a check on crystal and electronic stability and no significant variation was observed throughout the data collection. The orientation of the crystal was checked every 100 reflections by the recentering of three reflections selected to be nearly orthogonal to each other. The intensities were measured by the  $\omega/2\theta$  scan technique. Parameters of interest are outlined in Tables 1a and 1b.

Table 1a.

## Crystal Data and Experimental Details of x-ray Diffraction Studies

## Crystal Parameters for BRMEHO

Formula       $C_{17}H_{12}Br_2N_2OS$   
 $a = 12.937(2)\text{\AA}$   
 $b = 13.429(2)\text{\AA}$   
 $c = 13.489(2)\text{\AA}$   
 $\alpha = 60.14^\circ(1)$   
 $\beta = 74.58^\circ(1)$   
 $\gamma = 58.70^\circ(1)$   
Volume =  $1736.52 \text{ \AA}^3$

Crystal system Triclinic

Equivalent Reflections  $I_{hkl} = I_{\bar{h}\bar{k}\bar{l}}$   
Space group       $P\bar{1}$   
 $Z$                 4  
 $D_c$               1.72 g/cc  
 $D_o$               1.70 g/cc

Table 1b.

Crystal size	0.50*0.40*0.25mm
Instrument	Nonius CAD-4
Maximum theta	25°
Radiation	MoK <sub>α</sub> , graphite-monochromatized
Wavelength, Å	0.71073
Scan technique	ω/2θ
Scan range	0.8 + 0.35tanθ
Scan rate, deg/min	4
Max scan time, Secs	180
Intensity standard variation	49865 ± 1166 (2%) (3 0 -5)
μ (cm <sup>-1</sup> )	50.47
Range of transmission factor	0.08-0.28
Number of reflections collected	6565

**Appendix IV: Structure Factor Tables for BRMEO**

## DESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H168R2N2CS

PAGE 1

H	K	L	10FO	10FC	F	K	L	10FC	10FC	H	K	L	10FO	10FC	F	K	L	10FC	10FC
2	0	0	577	-558	(	2	C	441	-419	17	3	C	16	-78	14	5	0	130	161
3	0	0	1375	-1433	1	2	C	1152	-1208	18	3	C	5	151	15	5	0	15	267
4	0	0	3931	-3932	2	2	C	118	-125	19	3	C	138	-95	16	5	0	15	-60
5	0	0	4264	4284	3	2	C	74	104	0	4	C	2081	-2131	17	5	0	16	149
6	0	0	953	-1021	4	2	C	1659	-1655	1	4	C	720	-664	18	5	0	17	53
7	0	0	2665	-2493	5	2	C	2034	2085	2	4	C	885	-917	19	5	0	18	-60
8	0	0	311	-299	6	2	C	110	-87	3	4	C	542	-587	0	6	0	4638	4517
9	0	0	122	-100	7	2	C	717	650	4	4	C	955	951	1	6	0	1780	1754
10	0	0	1141	1159	8	2	C	592	-453	5	4	C	395	406	2	6	0	188	181
11	0	0	959	925	9	2	C	1111	-1116	6	4	C	428	420	3	6	0	9	-111
12	0	0	334	-343	10	2	C	723	810	7	4	C	239	-219	4	6	0	763	-740
13	0	0	781	-733	11	2	C	267	-243	8	4	C	258	-279	5	6	0	1119	1059
14	0	0	732	755	12	2	C	303	-307	9	4	C	471	-510	6	6	0	10	92
15	0	0	360	381	13	2	C	412	-355	10	4	C	779	803	7	6	0	2423	-2397
16	0	0	806	837	14	2	C	650	-751	11	4	C	12	-165	8	6	0	488	-504
17	0	0	91	-182	15	2	C	318	407	12	4	C	88	-118	9	6	0	116	-268
18	0	0	1021	-945	16	2	C	15	117	13	4	C	13	-87	10	6	0	713	708
19	0	0	17	102	17	2	C	207	227	14	4	C	783	-824	11	6	0	1549	1571
2	1	0	196	-214	18	2	C	16	123	15	4	C	486	535	12	6	0	498	-599
3	1	0	1410	1430	19	2	C	17	-182	16	4	C	15	83	13	6	0	135	192
4	1	0	1591	-1584	1	3	C	964	906	17	4	C	55	-67	14	6	0	495	532
5	1	0	874	854	2	3	C	341	379	18	4	C	333	386	15	6	0	202	232
6	1	0	2186	-2193	3	3	C	1672	1609	19	4	C	355	-422	16	6	0	239	354
7	1	0	9	44	4	3	C	987	-986	1	5	C	693	750	17	6	0	325	-401
8	1	0	1798	1795	5	3	C	202	250	2	5	C	197	200	18	6	0	451	-408
9	1	0	3	58	6	3	C	587	-619	3	5	C	259	303	19	6	0	18	-95
10	1	0	1220	1286	7	2	C	59	-106	4	5	C	293	324	1	7	0	1680	-1664
11	1	0	11	-234	8	3	C	966	1003	5	5	C	345	308	2	7	0	545	-555
12	1	0	520	-510	9	3	C	669	-703	6	5	C	1283	1288	3	7	0	667	-641
13	1	0	276	259	10	3	C	520	631	7	5	C	426	-421	4	7	0	723	735
14	1	0	385	-411	11	3	C	396	-463	8	5	C	723	-722	5	7	0	453	443
15	1	0	14	25	12	3	C	12	-201	9	5	C	655	-725	6	7	0	1688	-1636
16	1	0	197	-259	13	3	C	138	220	10	5	C	731	-714	7	7	0	564	-545
17	1	0	244	-257	14	2	O	423	-482	11	5	C	296	252	8	7	0	573	480
18	1	0	16	25	15	3	C	82	165	12	5	C	332	323	9	7	0	1111	1084
19	1	0	17	144	16	3	C	116	-161	13	5	C	154	113	10	7	0	999	1040

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16ER2N2OS

PAGE 2

F	K	L	10FC	10FC	F	K	L	10FC	10FC	H	K	L	10FC	10FC	F	K	L	10FC	10FC
10	9	0	13	30	11	11	C	224	50	14	13	C	17	290	6	16	C	16	295
11	9	0	361	-376	12	11	C	108	-145	15	13	C	99	26	7	16	0	16	49
12	9	0	658	-624	13	11	C	333	-281	0	14	C	486	-518	8	16	C	115	-30
13	9	0	135	-271	14	11	C	76	118	1	14	C	13	-292	9	16	C	96	27
14	9	0	15	126	15	11	C	17	322	2	14	C	13	-31	10	16	0	136	-157
15	9	0	232	116	16	11	C	55	64	3	14	C	179	-106	11	16	0	17	-83
16	9	0	16	160	C	12	C	1948	1975	4	14	C	14	185	12	16	0	99	-56
17	9	0	337	-353	1	12	C	895	884	5	14	C	14	258	1	17	C	206	278
0	10	0	301	-295	2	12	C	389	-383	6	14	C	14	170	2	17	0	87	116
1	10	0	82	94	3	12	C	652	-661	7	14	C	272	271	3	17	0	282	310
2	10	0	361	-356	4	12	C	170	-156	8	14	C	240	92	4	17	C	16	251
3	10	0	1038	-1053	5	12	C	501	466	9	14	C	15	-33	5	17	C	5	92
4	10	0	595	605	6	12	C	287	-235	10	14	C	244	-193	6	17	0	16	76
5	10	0	373	377	7	12	C	811	-888	11	14	C	72	-195	7	17	0	323	-309
6	10	0	12	55	8	12	C	805	-819	12	14	C	296	-290	8	17	C	34	-115
7	10	0	431	428	9	12	C	275	308	13	14	C	5	-110	9	17	C	171	164
8	10	0	667	-653	10	12	C	805	755	14	14	C	221	158	10	17	0	262	104
9	10	0	4	-24	11	12	C	705	684	1	15	C	65	-66	0	18	0	324	310
10	10	0	576	620	12	12	C	16	111	2	15	C	383	-438	1	19	C	352	338
11	10	0	406	-360	13	12	C	251	-257	3	15	C	370	403	2	18	C	147	-125
12	10	0	15	186	14	12	C	17	175	4	15	C	320	274	3	18	0	5	-84
13	10	0	486	-486	15	12	C	203	258	5	15	C	187	96	4	18	0	16	145
14	10	0	242	-354	16	12	C	18	51	6	15	C	298	-260	5	18	0	17	-107
15	10	0	312	300	1	13	C	1284	-1249	7	15	C	275	-233	6	18	C	5	-67
16	10	0	17	75	2	12	C	1154	-1173	8	15	C	108	150	7	18	0	477	-522
17	10	0	170	120	3	13	C	393	352	9	15	C	16	13	8	18	0	18	-235
1	11	0	796	755	4	13	0	406	430	10	15	C	189	33	9	18	0	271	221
2	11	0	70	46	5	13	C	13	-64	11	15	C	389	-398	1	19	C	579	-593
3	11	0	463	440	6	13	C	565	-586	12	15	C	109	-228	2	19	0	514	-529
4	11	0	403	501	7	13	C	550	-611	13	15	C	18	184	3	19	C	5	109
5	11	0	260	242	8	13	C	511	535	0	16	C	326	-317	4	19	C	375	419
6	11	0	567	601	9	13	C	652	613	1	16	C	15	123	5	19	0	220	-56
7	11	0	482	-474	10	13	C	254	252	2	16	C	15	-90	6	19	0	311	-350
8	11	0	397	-469	11	13	C	141	53	3	16	C	124	-14	0	20	0	18	-326
9	11	0	131	187	12	12	0	153	-279	4	16	C	15	118	1	20	C	197	-207
10	11	0	4	-82	13	13	C	16	123	5	16	C	204	-198	2	20	C	223	-103

## OBSERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

PAGE 3

H	K	L	OFC	OFC	F	K	L	OFC	OFC	H	K	L	OFC	OFC	H	K	L	OFC	OFC	F	K	L	OFC	OFC
18	1	1	159	-247	14	2	1	62	-220	10	3	1	133	-105	7	4	1	325	-369	4	5	1	1349	-1310
19	1	1	344	-301	15	2	1	15	-241	11	3	1	557	975	8	4	1	536	-555	5	5	1	134	131
-20	2	1	364	-347	16	2	1	294	-245	12	3	1	465	-423	9	4	1	680	-590	6	5	1	57	138
-19	2	1	17	130	17	2	1	353	377	13	3	1	13	-143	10	4	1	1012	1018	7	5	1	763	-793
-18	2	1	16	34	18	2	1	168	-61	14	3	1	547	-556	11	4	1	442	-463	8	5	1	744	-688
-17	2	1	264	281	19	2	1	18	86	15	3	1	462	-449	12	4	1	528	-557	5	5	1	476	-527
-16	2	1	403	345	-20	3	1	395	386	16	3	1	613	681	13	4	1	13	-193	10	5	1	413	381
-15	2	1	383	-385	-19	3	1	5	262	17	3	1	76	-41	14	4	1	112	-93	11	5	1	584	558
-14	2	1	163	-214	-18	3	1	349	-337	18	3	1	75	-94	15	4	1	764	762	12	5	1	155	-116
-13	2	1	149	159	-17	3	1	120	265	19	3	1	213	55	16	4	1	411	418	13	5	1	123	80
-12	2	1	361	-341	-16	3	1	15	68	-19	4	1	198	-204	17	4	1	241	-228	14	5	1	235	-289
-11	2	1	282	-306	-15	3	1	481	424	-18	4	1	190	-60	18	4	1	17	-45	15	5	1	394	400
-10	2	1	1002	-943	-14	3	1	658	718	-17	4	1	5	45	19	4	1	18	-72	16	5	1	203	134
-9	2	1	753	-788	-13	3	1	794	-870	-16	4	1	85	-32	-19	5	1	94	-187	17	5	1	220	-7
-8	2	1	226	213	-12	3	1	1204	-1315	-15	4	1	185	-49	-18	5	1	219	-95	18	5	1	5	-44
-7	2	1	375	415	-11	3	1	346	-279	-14	4	1	107	-188	-17	5	1	412	-394	-19	6	1	18	16
-6	2	1	548	-603	-10	3	1	80	32	-13	4	1	785	-796	-16	5	1	340	409	-18	6	1	61	-97
-5	2	1	190	191	-9	3	1	1366	1345	-12	4	1	781	811	-15	5	1	15	240	-17	6	1	209	294
-4	2	1	842	885	-8	3	1	957	1004	-11	4	1	283	294	-14	5	1	103	75	-16	6	1	206	-245
-3	2	1	1915	1930	-7	3	1	1473	-1547	-10	4	1	1062	1001	-13	5	1	4	-53	-15	6	1	212	270
-2	2	1	1528	1652	-6	3	1	1303	1367	-9	4	1	1085	1108	-12	5	1	890	-927	-14	6	1	14	151
-1	2	1	308	-350	-5	3	1	2098	2166	-8	4	1	1198	-1216	-11	5	1	12	181	-12	6	1	115	-112
0	2	1	280	-276	-4	3	1	1324	1267	-7	4	1	629	641	-10	5	1	392	382	-12	6	1	543	552
1	2	1	566	-545	-3	3	1	3378	3251	-6	4	1	530	568	-9	5	1	11	-49	-11	6	1	138	34
2	2	1	1641	-1653	-2	3	1	2030	2016	-5	4	1	239	-224	-8	5	1	145	-197	-10	6	1	612	-585
3	2	1	680	640	-1	3	1	550	513	-4	4	1	297	-346	-7	5	1	1066	-1084	-9	6	1	3	-95
4	2	1	1825	-1848	(	3	1	2170	2160	-3	4	1	3926	-3775	-6	5	1	309	-295	-8	6	1	715	-719
5	2	1	168	-134	1	3	1	1158	-1053	-2	4	1	724	-716	-5	5	1	123	39	-7	6	1	383	385
6	2	1	147	-114	2	3	1	724	695	-1	4	1	1818	1779	-4	5	1	9	95	-6	6	1	517	559
7	2	1	580	-635	3	3	1	2316	-2181	0	4	1	3310	3187	-3	5	1	1551	-1491	-5	6	1	10	35
8	2	1	412	452	4	3	1	2675	-2567	1	4	1	213	209	-2	5	1	1028	-992	-4	6	1	653	-600
9	2	1	151	91	5	2	1	2290	2243	2	4	1	66	-17	-1	5	1	678	686	-2	6	1	141	-149
10	2	1	81	-93	6	3	1	2329	2324	3	4	1	1022	-934	0	5	1	2236	2201	-2	6	1	1288	-1262
11	2	1	529	585	7	3	1	1089	1148	4	4	1	1151	1129	1	5	1	2760	2599	-1	6	1	186	198
12	2	1	426	434	8	3	1	745	700	5	4	1	1758	1750	2	5	1	406	-377	0	6	1	261	-241
13	2	1	209	-225	9	3	1	2181	-2170	6	4	1	287	-262	3	5	1	1268	-1185	1	6	1	765	667

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR SRMPC F23H16PR2N20S

PAGE 4

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
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3	6	1	597	-646	1	7	1	1516	1463	0	8	1	410	-436	0	9	1	94	-90
4	6	1	192	-245	2	7	1	322	-314	1	8	1	668	-660	1	9	1	302	247
5	6	1	220	159	3	7	1	634	-576	2	8	1	889	-889	2	9	1	204	-139
6	6	1	503	-486	4	7	1	10	31	3	8	1	527	480	3	9	1	968	-953
7	6	1	10	75	5	7	1	1068	-1084	4	8	1	10	-158	4	9	1	741	-699
8	6	1	11	-51	6	7	1	156	148	5	8	1	515	-612	5	9	1	1055	998
9	6	1	237	221	7	7	1	116	-33	6	8	1	223	200	6	9	1	1175	1204
10	6	1	526	501	8	7	1	809	-854	7	8	1	492	-485	7	9	1	710	667
11	6	1	219	-126	9	7	1	327	329	8	8	1	445	438	8	9	1	28	-56
12	6	1	577	-710	10	7	1	375	424	9	8	1	356	386	9	9	1	698	-681
13	6	1	144	38	11	7	1	4	80	10	8	1	381	-370	10	9	1	13	231
14	6	1	15	-160	12	7	1	574	586	11	8	1	472	452	11	9	1	484	570
15	6	1	15	149	13	7	1	306	-268	12	8	1	14	122	12	9	1	15	176
16	6	1	181	193	14	7	1	266	-279	13	8	1	5	68	13	9	1	15	-302
17	6	1	241	-256	15	7	1	305	306	14	8	1	272	225	14	9	1	521	-632
18	6	1	145	-21	16	7	1	5	-56	15	8	1	542	-494	15	9	1	16	-126
-19	7	1	18	-195	17	7	1	17	218	16	8	1	139	33	16	9	1	354	364
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-17	7	1	481	-480	-18	8	1	150	24	-18	9	1	99	-38	-17	10	1	78	-6
-16	7	1	16	191	-17	8	1	17	167	-17	9	1	267	-40	-16	10	1	130	53
-15	7	1	448	400	-16	8	1	264	254	-16	9	1	5	94	-15	10	1	16	163
-14	7	1	417	-372	-15	8	1	215	101	-15	9	1	221	293	-14	10	1	650	-736
-13	7	1	14	26	-14	8	1	343	-270	-14	9	1	109	185	-13	10	1	256	-253
-12	7	1	424	-413	-13	8	1	14	58	-13	9	1	333	-378	-12	10	1	105	143
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-10	7	1	986	1005	-11	8	1	13	52	-11	9	1	13	-314	-10	10	1	1044	1023
-9	7	1	11	74	-10	8	1	50	-364	-10	9	1	13	110	-9	10	1	13	44
-8	7	1	1170	-1171	-9	8	1	1251	-1228	-9	9	1	733	763	-8	10	1	284	-302
-7	7	1	184	-111	-6	8	1	37	243	-8	9	1	385	372	-7	10	1	179	173
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-4	7	1	781	733	-5	8	1	10	49	-5	9	1	1096	1077	-4	10	1	576	-508
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## RESERVED AND CALCULATED STRUCTURE FACTORS FOR SRMEC C23H16BR2N2OS

PAGE 5

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3	11	1	485	-504	6	12	1	364	-372	11	13	1	16	-140	-10	15	1	156	84
4	11	1	109	-48	7	12	1	361	-406	12	13	1	322	306	-9	15	1	462	494
5	11	1	220	239	8	12	1	4	19	13	13	1	17	-150	-8	15	1	15	130
6	11	1	497	-455	9	12	1	14	166	14	13	1	17	39	-7	15	1	333	-336
7	11	1	256	-251	10	12	1	365	332	-14	14	1	6	-46	-6	15	1	15	107
6	11	1	657	-677	11	12	1	141	-127	-13	14	1	187	78	-5	15	1	505	503
9	11	1	205	-160	12	12	1	403	-407	-12	14	1	117	173	-4	15	1	844	843
10	11	1	112	132	13	12	1	234	-68	-11	14	1	16	109	-3	15	1	147	107
11	11	1	15	-67	14	12	1	17	-63	-10	14	1	394	-454	-2	15	1	707	-736
12	11	1	126	-10	15	12	1	339	248	-9	14	1	711	-706	-1	15	1	582	-566
13	11	1	124	-46	-15	13	1	199	181	-8	14	1	245	-194	0	15	1	205	-121
14	11	1	166	267	-14	13	1	158	-144	-7	14	1	305	312	1	15	1	267	252
15	11	1	292	200	-13	13	1	5	-63	-6	14	1	61	162	2	15	1	131	-182
16	11	1	114	110	-12	13	1	16	20	-5	14	1	388	346	3	15	1	406	-399
-16	12	1	150	21	-11	13	1	85	-13	-4	14	1	14	-216	4	15	1	15	-333
-15	12	1	121	63	-10	13	1	469	457	-3	14	1	437	-459	5	15	1	309	343
-14	12	1	5	40	-9	12	1	15	-155	-2	14	1	747	747	6	15	1	651	695
-13	12	1	16	-64	-8	13	1	621	-592	-1	14	1	13	-171	7	15	1	151	186
-12	12	1	342	407	-7	13	1	167	187	0	14	1	485	517	8	15	1	16	97
-11	12	1	15	24	-6	13	1	401	-423	1	14	1	13	38	9	15	1	15	-126
-10	12	1	4	-111	-5	12	1	13	176	2	14	1	510	-895	10	15	1	217	261
-9	12	1	197	-257	-4	12	1	13	103	3	14	1	307	339	11	15	1	335	352
-8	12	1	646	-645	-3	13	1	368	-384	4	14	1	367	-369	12	15	1	17	-134
-7	12	1	13	306	-2	13	1	469	476	5	14	1	4	55	-12	16	1	6	17
-6	12	1	395	356	-1	12	1	13	71	6	14	1	15	32	-11	16	1	443	439
-5	12	1	108	291	0	13	1	27	-263	7	14	1	291	-299	-10	16	1	568	543
-4	12	1	12	-152	1	13	1	82	51	8	14	1	449	394	-9	16	1	16	-80
-3	12	1	526	-533	2	13	1	160	128	9	14	1	292	276	-8	16	1	266	-254
-2	12	1	4	-8	3	13	1	117	-121	10	14	1	335	306	-7	16	1	176	213
-1	12	1	434	411	4	13	1	43	257	11	14	1	73	42	-6	16	1	119	177
0	12	1	338	343	5	13	1	13	-80	12	14	1	17	-131	-5	16	1	110	231
1	12	1	353	-355	6	13	1	166	-206	13	14	1	192	-307	-4	16	1	548	-542
2	12	1	407	-443	7	13	1	14	251	14	14	1	17	3	-3	16	1	837	-878
3	12	1	69	-53	8	13	1	513	-468	-13	15	1	18	-123	-2	16	1	208	142
4	12	1	90	79	9	13	1	108	EC	-12	15	1	358	-298	-1	16	1	144	190
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## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
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-6	18	1	142	160	-18	C	2	273	-330	18	0	2	157	60	14	1	2	496	-549
-5	18	1	183	259	-17	C	2	5	129	19	0	2	18	126	15	1	2	15	121
-4	18	1	5	29	-16	C	2	528	-545	-20	1	2	18	48	16	1	2	16	-78
-3	18	1	16	-196	-15	C	2	656	654	-19	1	2	17	-22	17	1	2	316	-348
-2	18	1	80	22	-14	C	2	1105	1154	-18	1	2	16	63	18	1	2	17	75
-1	18	1	288	305	-13	C	2	12	-201	-17	1	2	5	-18	19	1	2	268	-281
0	18	1	60	156	-12	C	2	256	285	-16	1	2	48	32	-20	2	2	6	133
1	18	1	16	-284	-11	C	2	161	-126	-15	1	2	971	954	-19	2	2	190	232
2	18	1	355	-356	-10	C	2	967	-929	-14	1	2	399	397	-18	2	2	15	-133
3	18	1	5	-58	-9	C	2	2561	2621	-13	1	2	415	398	-17	2	2	246	-212
4	18	1	112	216	-8	C	2	552	519	-12	1	2	268	262	-16	2	2	58	16
5	18	1	17	154	-7	C	2	828	-857	-11	1	2	846	-849	-15	2	2	159	57
6	18	1	17	-272	-6	C	2	1429	-1411	-10	1	2	3	-57	-14	2	2	205	135
7	18	1	17	-273	-5	C	2	2946	-2905	-9	1	2	10	105	-13	2	2	411	-475
8	18	1	18	-82	-4	C	2	650	555	-8	1	2	1150	-1197	-12	2	2	843	-757
-7	19	1	18	-56	-3	C	2	2653	2773	-7	1	2	598	-643	-11	2	2	697	-654
-6	19	1	18	133	-2	C	2	736	-755	-6	1	2	57	-111	-10	2	2	677	708
-5	19	1	18	-179	-1	C	2	404	-405	-5	1	2	8	-121	-9	2	2	90	397
-4	19	1	17	8	0	C	2	1131	1209	-4	1	2	2521	2573	-8	2	2	55	-84
-3	19	1	193	-96	1	C	2	2346	2342	-3	1	2	1370	1406	-7	2	2	442	-392
-2	19	1	105	57	2	C	2	2394	2388	-2	1	2	199	196	-6	2	2	74	-52
-1	19	1	124	219	3	C	2	215	-206	-1	1	2	792	879	-5	2	2	1335	1359
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2	19	1	297	-166	6	C	2	1065	-982	2	1	2	488	-457	-2	2	2	297	-355
3	19	1	17	44	7	C	2	853	853	3	1	2	2284	-2192	-1	2	2	1591	-1665
4	19	1	5	104	8	C	2	10	-74	4	1	2	1269	-1250	0	2	2	539	-587
5	19	1	178	-63	9	C	2	2033	-1919	5	1	2	96	-155	1	2	2	288	-301
6	19	1	18	49	10	C	2	351	-363	6	1	2	975	996	2	2	2	1011	-915
-2	20	1	6	74	11	C	2	571	552	7	1	2	1464	1464	3	2	2	590	-609
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0	20	1	18	77	13	C	2	1110	1233	9	1	2	178	-171	5	2	2	9	92
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2	20	1	18	-129	15	C	2	793	-783	11	1	2	12	-47	7	2	2	455	465
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## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
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7	3	2	167	203	4	4	2	259	-279	2	5	2	534	-483	0	6	2	663	-591	-2	7	2	553	511
8	3	2	68	141	5	4	2	593	-627	3	5	2	1960	1874	1	6	2	938	952	-1	7	2	540	531
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-17	4	2	557	-453	-15	5	2	17	35	17	5	2	17	270	15	6	2	282	-244	13	7	2	439	-381
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-12	4	2	712	-697	-14	5	2	588	-639	-16	6	2	410	-363	-18	7	2	17	163	-18	8	2	17	118
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-9	4	2	59	104	-11	5	2	1128	1159	-13	6	2	643	619	-15	7	2	534	534	-15	8	2	296	315
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## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMFC C23H16BR2N2OS

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H	K	L	10FC	1CFC	H	K	L	10FC	1CFC	H	K	L	10FC	1CFC	H	K	L	10FC	1CFC
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-3	8	2	240	-260	-3	9	2	1074	-1081	-2	10	2	731	-710	0	11	2	614	595
-2	8	2	207	-246	-2	9	2	1429	-1373	-1	10	2	1032	-1038	1	11	2	338	-357
-1	8	2	147	-124	-1	9	2	899	882	0	10	2	143	-56	2	11	2	12	99
0	8	2	181	-160	0	9	2	729	646	1	10	2	837	849	3	11	2	584	580
1	8	2	660	535	1	9	2	728	649	2	10	2	380	-343	4	11	2	333	364
2	8	2	1195	-1156	2	9	2	591	-520	3	10	2	532	-505	5	11	2	276	278
3	8	2	1217	-1198	3	9	2	1087	-1143	4	10	2	11	-56	6	11	2	169	152
4	8	2	76	-59	4	9	2	101	268	5	10	2	210	-223	7	11	2	686	-749
5	8	2	481	512	5	9	2	11	-52	6	10	2	592	604	8	11	2	13	69
6	8	2	1413	1366	6	9	2	431	455	7	10	2	168	191	9	11	2	14	-50
7	8	2	520	536	7	9	2	96	-93	8	10	2	362	-403	10	11	2	394	-394
8	8	2	12	-151	8	9	2	352	-335	9	10	2	600	572	11	11	2	321	286
9	8	2	499	-566	9	9	2	378	358	10	10	2	14	-110	12	11	2	299	-330
10	8	2	122	192	10	9	2	4	113	11	10	2	15	17	13	11	2	16	-12
11	8	2	242	-246	11	9	2	200	132	12	10	2	15	250	14	11	2	276	223
12	8	2	4	117	12	9	2	15	-373	13	10	2	510	-525	15	11	2	5	-83
13	8	2	15	104	12	9	2	261	-215	14	10	2	89	-57	-16	12	2	425	-445
14	8	2	514	-482	14	9	2	88	-101	15	10	2	92	27	-15	12	2	158	81
15	8	2	115	75	15	9	2	16	342	16	10	2	18	-134	-14	12	2	241	257
16	8	2	5	-38	16	9	2	303	259	-17	11	2	182	-12	-13	12	2	470	446
17	8	2	6	30	17	9	2	252	-228	-16	11	2	17	198	-12	12	2	344	391
18	9	2	6	42	-17	10	2	133	-373	-15	11	2	147	-119	-11	12	2	15	-235
17	9	2	105	-19	-16	10	2	17	57	-14	11	2	349	-415	-10	12	2	4	62
-16	9	2	16	-38	-15	10	2	291	207	-13	11	2	362	-314	-9	12	2	140	165
-15	9	2	425	466	-14	10	2	121	-114	-12	11	2	263	-246	-8	12	2	317	309
-14	9	2	311	-323	-13	10	2	218	113	-11	11	2	610	653	-7	12	2	281	-298
-13	9	2	15	37	-12	10	2	522	-553	-10	11	2	308	280	-6	12	2	674	-671
-12	9	2	14	154	-11	10	2	472	-470	-9	11	2	251	-257	-5	12	2	432	-424
-11	9	2	13	-272	-10	10	2	638	665	-8	11	2	320	293	-4	12	2	4	24
-10	9	2	214	297	-9	10	2	159	-149	-7	11	2	151	226	-3	12	2	626	696
-9	9	2	78	-210	-8	10	2	12	115	-6	11	2	662	683	-2	12	2	79	-64
-8	9	2	77	52	-7	10	2	12	119	-5	11	2	288	276	-1	12	2	390	-340
-7	9	2	91	-50	-6	10	2	589	-639	-4	11	2	1351	-1340	0	12	2	184	207
-6	9	2	519	572	-5	10	2	1320	1283	-3	11	2	801	-713	1	12	2	383	380
-5	9	2	214	-212	-4	10	2	467	425	-2	11	2	493	-573	2	12	2	1710	1686

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR PBMED C23H16BR2N2OS

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H	K	L	10FO	10FC	F	K	L	10FO	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC
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9	13	2	15	-168	-11	15	2	5	-18	0	16	2	170	-73	-6	18	2	17	-171
10	13	2	495	505	-10	15	2	399	401	1	16	2	186	118	-5	18	2	448	-501
11	13	2	587	606	-9	15	2	16	-112	2	16	2	344	-300	-4	18	2	16	109
12	13	2	258	258	-8	15	2	414	-406	3	16	2	15	82	-3	18	2	52	183
13	13	2	76	-249	-7	15	2	15	153	4	16	2	15	22	-2	18	2	191	161
14	13	2	395	-367	-6	15	2	48	-114	5	16	2	16	189	-1	18	2	16	-67
-14	14	2	18	-21	-5	15	2	441	431	6	16	2	16	202	0	18	2	16	22
-13	14	2	107	-146	-4	15	2	149	-150	7	16	2	16	-130	1	18	2	138	221
-12	14	2	159	-296	-3	15	2	914	-858	8	16	2	5	-125	2	18	2	419	373
-11	14	2	257	-287	-2	15	2	262	236	9	16	2	161	-56	3	18	2	310	339
-10	14	2	5	110	-1	15	2	226	-157	10	16	2	17	95	4	18	2	282	-344
-9	14	2	15	-15	0	15	2	410	420	11	16	2	99	-92	5	18	2	132	-221
-8	14	2	15	130	1	15	2	14	132	-11	17	2	344	347	6	18	2	17	-184
-7	14	2	15	-56	2	15	2	214	-324	-10	17	2	182	117	7	18	2	18	-24
-6	14	2	155	182	3	15	2	67	-242	-9	17	2	208	-27	8	18	2	18	221
-5	14	2	478	504	4	15	2	15	-38	-8	17	2	17	-222	-7	19	2	535	-474
-4	14	2	120	140	5	15	2	512	513	-7	17	2	174	236	-6	19	2	113	50
-3	14	2	178	135	6	15	2	15	63	-6	17	2	163	303	-5	19	2	420	444
-2	14	2	580	-510	7	15	2	16	-84	-5	17	2	135	233	-4	19	2	403	455
-1	14	2	13	159	8	15	2	510	-468	-4	17	2	512	-572	-3	19	2	17	34
0	14	2	159	150	9	15	2	51	-157	-3	17	2	501	-552	-2	19	2	108	-125
1	14	2	614	-568	10	15	2	342	403	-2	17	2	277	-261	-1	19	2	17	141
2	14	2	95	-68	11	15	2	17	-70	-1	17	2	132	-46	0	19	2	407	365
3	14	2	654	-703	12	15	2	18	-93	0	17	2	492	507	1	19	2	17	226
4	14	2	14	60	-12	16	2	18	-237	1	17	2	95	-65	2	19	2	370	-401
5	14	2	657	596	-11	16	2	5	-59	2	17	2	16	-163	3	19	2	643	-557
6	14	2	262	235	-10	16	2	69	-23	3	17	2	215	185	4	19	2	309	-284
7	14	2	332	256	-9	16	2	16	11	4	17	2	5	97	5	19	2	38	137
8	14	2	187	-246	-8	16	2	162	68	5	17	2	388	391	-2	20	2	206	-157
9	14	2	382	-305	-7	16	2	146	-57	6	17	2	123	43	-1	20	2	18	-59
10	14	2	16	95	-6	16	2	15	-79	7	17	2	17	-182	0	20	2	12	71
11	14	2	16	126	-5	16	2	15	161	8	17	2	5	-44	-20	1	3	17	-19
12	14	2	17	18	-4	16	2	200	20	9	17	2	122	-46	-19	1	3	91	342
13	14	2	93	-167	-3	16	2	15	99	-9	18	2	83	146	-18	1	3	16	-118
-13	15	2	362	-284	-2	16	2	136	76	-8	18	2	6	36	-17	1	3	302	-354
															-20	2	3	113	-123

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEQ.C23H16BR2N2CS

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	H	K	L	10FO	10FC	F	K	L	10FO	10FC	H	K	L	10FO	10FC	F	K	L	10FC	10FC
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-18	2	3	338	-360	18	2	3	263	-226	15	3	3	15	-209	12	4	3	605	653	
-17	2	3	262	274	-20	3	3	191	209	16	3	3	132	-175	13	4	3	4	-155	
-16	2	3	159	177	-19	3	3	17	37	17	3	3	274	-194	14	4	3	297	-262	
-15	2	3	4	-52	-18	3	3	355	298	18	3	2	520	586	15	4	3	71	172	
-14	2	3	324	350	-17	3	3	468	466	-20	4	3	131	-43	16	4	3	198	-159	
-13	2	3	804	-732	-16	3	3	283	-261	-19	4	2	5	106	17	4	3	365	285	
-12	2	3	12	27	-15	3	3	14	64	-18	4	3	16	52	18	4	3	170	119	
-11	2	3	11	175	-14	3	3	70	112	-17	4	3	16	-237	-20	5	3	18	-110	
-10	2	3	664	-611	-13	3	3	595	-615	-16	4	2	87	-69	-19	5	3	143	183	
-9	2	3	550	-594	-12	3	3	12	292	-15	4	3	14	-127	-18	5	3	16	-120	
-8	2	3	919	-950	-11	3	3	1262	-1318	-14	4	3	4	-138	-17	5	3	16	-247	
-7	2	3	1236	-1308	-10	3	3	679	-639	-13	4	2	206	191	-16	5	3	5	40	
-6	2	3	1203	1132	-9	3	3	1137	1104	-12	4	2	220	250	-15	5	3	94	-154	
-5	2	3	1017	1034	-8	3	3	10	123	-11	4	2	265	-267	-14	5	3	544	548	
-4	2	3	871	839	-7	3	3	1019	1043	-10	4	3	398	404	-13	5	3	265	336	
-3	2	3	793	850	-6	3	3	268	351	-9	4	2	1123	1106	-12	5	3	317	-409	
-2	2	3	1013	-1045	-5	3	3	1337	-1331	-8	4	2	493	479	-11	5	3	215	182	
-1	2	3	85	-88	-4	3	3	973	983	-7	4	2	1757	1793	-10	5	3	130	-36	
0	2	3	2132	2163	-3	2	3	663	689	-6	4	3	994	-1023	-9	5	3	849	869	
1	2	3	1133	-1135	-2	3	3	808	-810	-5	4	3	393	-385	-8	5	3	197	212	
2	2	3	1051	-1000	-1	2	3	1199	1181	-4	4	2	212	-197	-7	5	3	817	-876	
3	2	3	639	-646	0	3	3	3336	-3259	-3	4	2	947	-963	-6	5	3	1304	-1342	
4	2	3	1047	-1040	1	3	3	2851	-2691	-2	4	3	102	-101	-5	5	3	817	-833	
5	2	3	857	884	2	3	3	856	817	-1	4	3	916	-906	-4	5	3	980	1019	
6	2	3	1233	-1220	3	3	3	385	-317	0	4	2	740	-737	-3	5	3	246	270	
7	2	3	1771	-1739	4	3	3	1152	1131	1	4	2	1113	1058	-2	5	3	269	291	
8	2	3	258	285	5	3	3	10	66	2	4	2	1230	1195	-1	5	3	1191	-1180	
9	2	3	220	180	6	3	3	819	-786	3	4	2	677	662	0	5	3	5	-23	
10	2	3	1037	1131	7	3	3	1653	1638	4	4	2	771	757	1	5	3	1509	1455	
11	2	3	509	500	8	3	3	975	1013	5	4	2	176	-175	2	5	3	759	773	
12	2	3	444	-423	9	3	3	466	-530	6	4	2	121	83	3	5	3	685	637	
13	2	3	349	-345	10	3	3	12	-178	7	4	2	1336	1330	4	5	3	1013	-960	
14	2	3	15	-53	11	3	3	654	-712	8	4	2	738	-717	5	5	3	243	-223	
15	2	3	98	-236	12	3	3	130	-57	9	4	2	11	-77	6	5	3	959	868	
16	2	3	303	282	13	3	3	753	820	10	4	3	454	-464	7	5	3	11	154	

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2CS

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
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7	6	3	811	662	6	7	3	748	779	5	8	3	429	418	5	9	3	75	-227
8	6	3	362	-396	7	7	3	11	-42	6	8	3	74	-50	6	9	3	291	-282
9	6	3	277	-246	8	7	3	12	245	7	8	3	420	-403	7	9	3	898	891
10	6	3	218	235	5	7	3	4	44	8	8	3	12	-56	8	9	3	595	623
11	6	3	13	85	10	7	3	659	-629	9	8	3	13	-92	9	9	3	182	-113
12	6	3	213	302	11	7	3	313	356	10	8	3	13	159	10	9	3	14	196
13	6	3	15	-98	12	7	3	14	94	11	8	3	708	682	11	9	3	670	-693
14	6	3	49	32	13	7	3	144	-20	12	8	3	211	-214	12	9	3	15	16
15	6	3	16	104	14	7	3	5	27	13	8	3	15	-105	13	9	3	381	378
16	6	3	17	-92	15	7	3	16	-60	14	8	3	16	124	14	9	3	267	-261
17	6	3	17	45	16	7	3	5	-48	15	8	3	344	-340	15	9	3	17	-38
-19	7	3	245	240	17	7	3	257	220	16	8	3	369	341	16	9	3	6	-105
-18	7	3	17	11	-19	8	3	18	-15	17	8	3	89	-147	-18	10	3	18	109
-17	7	3	572	-505	-18	8	3	298	-259	-18	9	3	169	361	-17	10	3	350	-350
-16	7	3	205	-189	-17	8	3	16	146	-17	9	3	17	28	-16	10	3	17	11
-15	7	3	171	-240	-16	8	3	196	253	-16	9	3	301	-259	-15	10	3	106	-198
-14	7	3	445	417	-15	8	3	5	164	-15	9	3	5	-75	-14	10	3	237	-269
-13	7	3	843	767	-14	8	3	219	122	-14	9	3	49	-99	-13	10	3	15	139
-12	7	3	378	-351	-13	8	3	607	-609	-13	9	3	147	-199	-12	10	3	311	-336
-11	7	3	99	-25	-12	8	3	408	-431	-12	9	3	285	293	-11	10	3	363	380
-10	7	3	12	17	-11	8	3	692	645	-11	9	3	595	-588	-10	10	3	625	680
-9	7	3	159	173	-10	8	3	4	60	-10	9	3	537	-523	-9	10	3	544	561
-8	7	3	642	638	-9	8	3	4	-15	-9	9	3	166	174	-8	10	3	747	657
-7	7	3	390	-431	-8	8	3	613	-667	-8	9	3	174	119	-7	10	3	266	-250
-6	7	3	774	-759	-7	8	3	1592	-1543	-7	9	3	981	1014	-6	10	3	145	-202
-5	7	3	419	405	-6	8	3	656	649	-6	9	3	735	707	-5	10	3	11	-26
-4	7	3	280	314	-5	8	3	758	734	-5	9	3	404	-392	-4	10	3	412	404
-3	7	3	37	-105	-4	8	3	482	532	-4	9	3	591	634	-3	10	3	219	205
-2	7	3	10	-50	-3	8	3	629	676	-3	9	3	413	423	-2	10	3	792	-746
-1	7	3	1239	-1303	-2	8	3	973	-951	-2	9	3	240	258	-1	10	3	777	-815
0	7	3	201	225	-1	8	3	943	975	-1	9	3	489	-398	0	10	3	687	-653
1	7	3	1525	1497	0	8	3	551	583	0	9	3	1708	-1703	1	10	3	1032	1038
2	7	3	371	-332	1	8	3	609	-617	1	9	3	1070	-1011	2	10	3	776	774
3	7	3	499	521	2	8	3	884	-846	2	9	3	367	325	3	10	3	37	297
4	7	3	1066	-1070	3	8	3	1377	-1330	3	9	3	11	-63	4	10	3	274	201

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8	11	3	603	-509	12	12	3	226	231	-11	14	3	16	-39	-2	15	3	261	-261
9	11	3	501	-486	13	12	3	151	-201	-10	14	3	16	98	-1	15	3	4	110
10	11	3	287	-276	14	12	3	18	-203	-9	14	3	56	175	0	15	3	421	-429
11	11	3	15	105	-15	13	3	17	-115	-8	14	3	462	-433	1	15	3	455	-494
12	11	3	741	750	-14	13	3	17	50	-7	14	3	445	-365	2	15	3	192	128
13	11	3	16	37	-13	13	3	442	450	-6	14	3	38	83	3	15	3	111	-22
14	11	3	17	-160	-12	13	3	136	-107	-5	14	3	540	575	4	15	3	107	176
15	11	3	18	-181	-11	13	3	271	-318	-4	14	3	292	332	5	15	3	289	-216
-16	12	3	18	162	-10	13	3	339	260	-3	14	3	342	-362	6	15	3	148	-264
-15	12	3	344	339	-9	13	3	115	-151	-2	14	3	447	-534	7	15	3	304	405
-14	12	3	116	-88	-8	13	3	625	619	-1	14	3	107	131	8	15	3	430	525
-13	12	3	16	-132	-7	13	3	264	-315	0	14	3	897	857	9	15	3	166	274
-12	12	3	228	-264	-6	13	3	598	-606	1	14	3	312	337	10	15	3	17	-64
-11	12	3	15	-74	-5	13	3	120	68	2	14	3	611	-679	11	15	3	247	-216
-10	12	3	15	179	-4	13	3	4	-5	3	14	3	773	-765	-12	16	3	18	-127
-9	12	3	192	131	-3	13	3	356	410	4	14	3	505	-482	-11	16	3	17	105
-8	12	3	368	-362	-2	13	3	13	-142	5	14	3	488	488	-10	16	3	342	418
-7	12	3	292	-350	-1	13	3	426	-501	6	14	3	13	61	-9	16	3	405	429
-6	12	3	4	28	0	13	3	13	104	7	14	3	200	-177	-8	16	3	16	131
-5	12	3	13	-77	1	13	3	124	167	8	14	3	154	-81	-7	16	3	16	-16
-4	12	3	473	426	2	13	3	428	489	9	14	3	5	57	-6	16	3	297	-291
-3	12	3	385	-421	3	13	3	241	-275	10	14	3	390	356	-5	16	3	391	367
-2	12	3	317	-340	4	13	3	157	-156	11	14	3	17	192	-4	16	3	15	48
-1	12	3	238	209	5	13	3	243	-180	12	14	3	94	-79	-3	16	3	112	-155
0	12	3	305	328	6	13	3	151	211	13	14	3	96	-211	-2	16	3	158	-102
1	12	3	671	684	7	13	3	15	184	-13	15	3	18	16	-1	16	3	777	-780
2	12	3	304	-372	8	13	3	355	-355	-12	15	3	17	-8	0	16	3	15	-74
3	12	3	869	-863	9	13	3	141	153	-11	15	3	253	-278	1	16	3	49	194
4	12	3	42	-255	10	13	3	195	-174	-10	15	3	312	-317	2	16	3	478	444
5	12	3	13	-68	11	13	3	235	240	-9	15	3	176	-157	3	16	3	317	340
6	12	3	937	1006	12	13	3	17	183	-8	15	3	15	291	4	16	3	248	-227
7	12	3	46	236	13	13	3	274	-350	-7	15	3	586	624	5	16	3	110	68
8	12	3	298	-259	-15	14	3	286	291	-6	15	3	538	545	6	16	3	168	197
9	12	3	150	-180	-14	14	3	346	324	-5	15	3	15	186	7	16	3	181	256
10	12	3	15	-21	-13	14	3	328	-424	-4	15	3	15	-129	8	16	3	17	-233

## DESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

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H	K	L	10FC	1CFC	F	K	L	10FC	1CFC	H	K	L	10FC	1CFC	F	K	L	10FC	1CFC	F	K	L	10FC	1CFC
4	18	3	171	-155	1	C	4	423	398	-2	1	4	3475	3630	-5	2	4	598	-601	-8	3	4	110	-115
5	18	3	153	111	2	C	4	809	808	-1	1	4	549	610	-4	2	4	646	628	-7	3	4	345	377
6	18	3	445	392	2	C	4	345	-363	0	1	4	745	-773	-3	2	4	2926	2966	-6	3	4	1152	-1147
7	18	3	18	-27	4	C	4	2299	2245	1	1	4	560	-500	-2	2	4	147	-99	-5	3	4	626	-599
-6	19	3	6	-17	5	C	4	929	-872	2	1	4	930	-967	-1	2	4	1628	-1685	-4	3	4	811	759
-5	19	3	6	-182	6	C	4	1235	-1293	3	1	4	226	190	0	2	4	1227	-1297	-3	3	4	161	-179
-4	19	3	5	104	7	C	4	300	257	4	1	4	445	-396	1	2	4	871	-853	-2	3	4	110	114
-3	19	3	63	-13	8	C	4	1179	-1111	5	1	4	1175	-1140	2	2	4	1272	1203	-1	3	4	166	-186
-2	19	3	17	84	9	C	4	218	164	6	1	4	492	400	3	2	4	1121	1089	0	3	4	5	62
-1	19	3	274	-56	10	C	4	368	356	7	1	4	779	742	4	2	4	698	-647	1	3	4	169	161
0	19	3	11	140	11	C	4	563	-547	8	1	4	103	34	5	2	4	421	-427	2	3	4	351	296
1	19	3	17	113	12	C	4	356	411	9	1	4	921	956	6	2	4	291	253	3	3	4	562	-530
2	19	3	187	-234	13	C	4	585	612	10	1	4	285	-318	7	2	4	600	623	4	3	4	10	48
3	19	3	205	-102	14	C	4	216	176	11	1	4	301	-335	8	2	4	1034	965	5	3	4	111	-24
4	19	3	18	-118	15	C	4	16	73	12	1	4	636	651	9	2	4	85	-81	6	3	4	91	248
-20	0	4	17	18	16	C	4	140	-185	13	1	4	4	26	10	2	4	951	-999	7	3	4	980	918
-19	0	4	369	-348	17	C	4	142	-222	14	1	4	231	217	11	2	4	13	-88	6	3	4	11	-34
-18	0	4	156	112	18	C	4	521	555	15	1	4	16	-138	12	2	4	13	59	5	3	4	12	-67
-17	0	4	137	90	-20	1	4	17	-408	16	1	4	660	-633	13	2	4	230	269	10	3	4	339	-263
-16	0	4	466	-482	-15	1	4	86	-24	17	1	4	17	124	14	2	4	118	-16	11	3	4	379	-439
-15	0	4	564	550	-18	1	4	16	29	18	1	4	18	-49	15	2	4	480	-544	12	3	4	579	720
-14	0	4	337	-315	-17	1	4	5	151	-20	2	4	17	-30	16	2	4	161	-77	11	3	4	47	-82
-13	0	4	98	-49	-16	1	4	508	464	-19	2	4	118	297	17	2	4	132	50	14	3	4	64	125
-12	0	4	552	519	-15	1	4	426	416	-18	2	4	16	239	18	2	4	42	114	15	3	4	16	-65
-11	0	4	11	16	-14	1	4	304	324	-17	2	4	141	199	-20	3	4	18	-31	16	3	4	16	-200
-10	0	4	1412	1364	-13	1	4	318	313	-16	2	4	15	-301	-19	3	4	17	77	17	3	4	203	189
-9	0	4	346	-313	-12	1	4	268	307	-15	2	4	343	-430	-18	3	4	126	-227	-20	4	4	18	-129
-8	0	4	1093	-1119	-11	1	4	352	-387	-14	2	4	191	212	-17	3	4	15	-53	-15	4	4	278	330
-7	0	4	1239	-1268	-10	1	4	128	-58	-13	2	4	440	454	-16	3	4	15	65	-18	4	4	5	61
-6	0	4	633	-627	-9	1	4	1165	-1200	-12	2	4	12	160	-15	3	4	229	271	-17	4	4	15	-81
-5	0	4	1464	-1570	-8	1	4	347	-344	-11	2	4	443	-391	-14	3	4	4	-11	-16	4	4	129	-59
-4	0	4	1597	-1563	-7	1	4	511	524	-10	2	4	1170	-1171	-13	3	4	284	-321	-15	4	4	669	-723
-3	0	4	1953	-1935	-6	1	4	702	-717	-9	2	4	320	308	-12	3	4	99	152	-14	4	4	727	707
-2	0	4	718	699	-5	1	4	55	56	-8	2	4	1125	1192	-11	3	4	547	-531	-13	4	4	602	593
-1	0	4	2755	2815	-4	1	4	841	-869	-7	2	4	356	381	-10	3	4	639	596	-12	4	4	12	-208
0	0	4	801	-767	-3	1	4	2082	2047	-6	2	4	293	-276	-9	3	4	567	557	-11	4	4	414	-382

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEO C23H16BR2N2OS

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H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC	F	K	L	10FC	10FC
-10	4	4	1282	-1273	-12	5	4	262	-216	-13	6	4	329	331	-14	7	4	604	613
-9	4	4	235	199	-11	5	4	361	385	-12	6	4	775	814	-13	7	4	622	701
-8	4	4	1411	1470	-10	5	4	11	265	-11	6	4	4	12	-12	7	4	135	-152
-7	4	4	822	865	-5	5	4	1813	1864	-10	6	4	114	98	-11	7	4	12	-20
-6	4	4	123	-108	-8	5	4	10	-80	-9	6	4	257	253	-10	7	4	727	-727
-5	4	4	288	-325	-7	5	4	719	-745	-8	6	4	847	-885	-9	7	4	906	-887
-4	4	4	9	33	-6	5	4	104	-27	-7	6	4	408	337	-8	7	4	11	-56
-3	4	4	1129	1176	-5	5	4	571	-487	-6	6	4	910	-970	-7	7	4	592	-652
-2	4	4	814	812	-4	5	4	421	444	-5	6	4	1990	-2061	-6	7	4	476	408
-1	4	4	1176	-1259	-3	5	4	235	-221	-4	6	4	354	314	-5	7	4	32	57
0	4	4	880	-861	-2	5	4	2401	-2451	-3	6	4	1390	-1434	-4	7	4	788	754
1	4	4	403	-426	-1	5	4	866	-983	-2	6	4	1014	1047	-3	7	4	471	545
2	4	4	580	534	0	5	4	298	-301	-1	6	4	960	986	-2	7	4	773	859
3	4	4	1324	1307	1	5	4	628	569	0	6	4	265	-296	-1	7	4	1089	1037
4	4	4	1428	-1397	2	5	4	1604	1589	1	6	4	443	503	0	7	4	596	-601
5	4	4	744	-760	3	5	4	652	-576	2	6	4	148	140	1	7	4	217	176
6	4	4	224	241	4	5	4	272	-260	3	6	4	993	968	2	7	4	10	-189
7	4	4	296	312	5	5	4	822	825	4	6	4	933	924	3	7	4	10	42
8	4	4	1642	1660	6	5	4	92	-117	5	6	4	497	-481	4	7	4	203	-157
9	4	4	305	-279	7	5	4	369	402	6	6	4	1483	-1366	5	7	4	1385	-1302
10	4	4	916	-952	8	5	4	543	-606	7	6	4	553	-538	6	7	4	62	-78
11	4	4	198	-133	9	5	4	958	-973	8	6	4	237	-273	7	7	4	80	-123
12	4	4	342	-347	10	5	4	71	31	9	6	4	94	132	8	7	4	586	595
13	4	4	276	287	11	5	4	110	141	10	6	4	459	490	9	7	4	604	582
14	4	4	15	177	12	5	4	230	-281	11	6	4	588	-630	10	7	4	12	-57
15	4	4	239	-286	13	5	4	15	77	12	6	4	469	470	11	7	4	248	271
16	4	4	17	-104	14	5	4	270	-245	13	6	4	388	360	12	7	4	5	-43
17	4	4	17	145	15	5	4	16	-37	14	6	4	16	-19	13	7	4	256	-232
-20	5	4	353	306	16	5	4	522	514	15	6	4	362	355	14	7	4	16	-49
-19	5	4	259	256	17	5	4	18	16	16	6	4	203	-245	15	7	4	16	-167
-18	5	4	5	-124	-19	6	4	52	-100	17	6	4	180	-131	16	7	4	300	-217
-17	5	4	16	9	-18	6	4	5	59	-19	7	4	18	34	-19	8	4	144	93
-16	5	4	560	-611	-17	6	4	16	-226	-18	7	4	17	-5	-18	8	4	347	343
-15	5	4	114	-218	-16	6	4	5	-129	-17	7	4	5	43	-17	8	4	353	356
-14	5	4	13	104	-15	6	4	106	-72	-16	7	4	384	394	-16	8	4	16	-279
-13	5	4	823	-855	-14	6	4	93	-169	-15	7	4	15	-96	-15	8	4	218	-275

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
-13	9	4	124	-62	-11	10	4	225	-150	-8	11	4	179	82	-3	12	4	850	-914
-12	9	4	393	-431	-10	10	4	250	-255	-7	11	4	586	-518	-2	12	4	12	138
-11	9	4	4	-65	-9	10	4	547	-558	-6	11	4	321	-300	-1	12	4	953	977
-10	9	4	325	292	-8	10	4	938	944	-5	11	4	40	-137	0	12	4	492	469
-9	9	4	500	529	-7	10	4	256	286	-4	11	4	677	714	1	12	4	13	226
-8	9	6	12	-66	-6	10	4	126	167	-3	11	4	198	-220	2	12	4	290	229
-7	9	4	4	-71	-5	10	4	142	118	-2	11	4	1701	-1771	3	12	4	189	-209
-6	9	4	187	-179	-4	10	4	552	-566	-1	11	4	663	-674	4	12	4	347	376
-5	9	4	630	-604	-3	10	4	813	886	0	11	4	178	137	5	12	4	13	-308
-4	9	4	462	426	-2	10	4	128	-56	1	11	4	486	497	6	12	4	710	-658
-3	9	4	426	-419	-1	10	4	1093	-1049	2	11	4	592	620	7	12	4	14	177
-2	9	4	108	-124	0	10	4	311	-282	3	11	4	666	-684	8	12	4	171	-291
-1	9	4	277	269	1	10	4	62	-46	4	11	4	13	-127	9	12	4	69	209
0	9	4	731	-666	2	10	4	246	312	5	11	4	476	491	10	12	4	16	-26
1	9	4	1358	1304	3	10	4	154	192	6	11	4	574	566	11	12	4	248	-245
2	9	4	295	-263	4	10	4	415	-402	7	11	4	14	153	12	12	4	106	161
3	9	4	335	-308	5	10	4	453	-483	8	11	4	635	-577	13	12	4	5	35
4	9	4	421	407	6	10	4	662	707	9	11	4	588	-604	14	12	4	293	332
5	9	4	332	-296	7	10	4	13	122	10	11	4	15	-347	-15	13	4	55	43
6	9	4	1175	1175	8	10	4	430	440	11	11	4	404	403	-14	13	4	447	430
7	9	4	461	505	9	10	4	14	-87	12	11	4	36	-25	-13	13	4	16	-21
8	9	4	356	-394	10	10	4	589	-555	13	11	4	17	48	-12	13	4	128	180
9	9	4	67	-146	11	10	4	40	65	14	11	4	5	-131	-11	13	4	161	95
10	9	4	442	-421	12	10	4	16	9	-16	12	4	160	-166	-10	13	4	280	-304
11	9	4	122	75	13	10	4	204	17	-15	12	4	5	132	-9	13	4	370	-370
12	9	4	479	497	14	10	4	5	65	-14	12	4	147	-194	-8	13	4	14	-265
13	9	4	5	56	15	10	4	306	-352	-13	12	4	276	269	-7	13	4	317	-448
14	9	4	17	-76	-17	11	4	200	-152	-12	12	4	324	358	-6	13	4	108	-73
15	9	4	164	-187	-16	11	4	246	-338	-11	12	4	5	28	-5	13	4	162	168
-16	10	4	74	83	-15	11	4	74	26	-10	12	4	382	432	-4	13	4	465	446
-17	10	4	17	-38	-14	11	4	113	211	-9	12	4	72	-172	-3	13	4	911	925
-16	10	4	5	-76	-13	11	4	381	-358	-8	12	4	131	-272	-2	13	4	566	637
-15	10	4	16	-71	-12	11	4	182	-184	-7	12	4	127	145	-1	13	4	284	-349
-14	10	4	15	61	-11	11	4	14	-134	-6	12	4	214	-250	0	13	4	115	-91
-13	10	4	406	358	-10	11	4	377	400	-5	12	4	245	-215	1	13	4	13	-95
-12	10	4	121	-82	-9	11	4	1132	1255	-4	12	4	560	-539	2	13	4	13	-18
															9	14	4	99	127

## DESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

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H	K	L	10FO	1FCF	H	K	L	10FO	1FCF	H	K	L	1FCF	10FC	H	K	L	1FCF	10FC
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11	14	4	210	-95	-3	16	4	5	179	-7	18	4	17	-17	-8	1	5	1184	1122
12	14	4	18	194	-2	16	4	120	258	-6	18	4	17	160	-7	1	5	494	487
-13	15	4	18	-145	-1	16	4	501	-415	-5	18	4	291	-292	-6	1	5	797	728
-12	15	4	282	-230	0	16	4	15	-314	-4	18	4	203	-140	-5	1	5	830	-866
-11	15	4	184	-177	1	16	4	15	40	-3	18	4	403	-439	-4	1	5	2023	-2070
-10	15	4	369	343	2	16	4	15	-41	-2	18	4	113	39	-3	1	5	1169	1173
-9	15	4	359	413	3	16	4	16	125	-1	18	4	265	329	-2	1	5	1019	1009
-8	15	4	145	-193	4	16	4	16	-142	0	18	4	269	186	-1	1	5	638	674
-7	15	4	15	36	5	16	4	77	-164	1	18	4	17	333	0	1	5	202	-201
-6	15	4	700	-711	6	16	4	351	438	2	18	4	17	-204	1	1	5	1510	-1431
-5	15	4	215	237	7	16	4	227	45	3	18	4	207	150	2	1	5	700	718
-4	15	4	378	351	8	16	4	244	197	4	18	4	5	-25	3	1	5	1012	1013
-3	15	4	15	-199	9	16	4	18	-224	5	18	4	6	28	4	1	5	268	250
-2	15	4	147	71	10	16	4	273	-120	6	18	4	211	-99	5	1	5	517	-541
-1	15	4	423	-450	-11	17	4	204	-277	-6	19	4	13	-18	6	1	5	1324	-1301
0	15	4	10	-97	-10	17	4	18	245	-5	19	4	18	-14	7	1	5	439	-435
1	15	4	580	540	-9	17	4	658	664	-4	19	4	6	175	8	1	5	621	658
2	15	4	316	303	-8	17	4	142	151	-3	19	4	261	402	9	1	5	403	392
3	15	4	5	167	-7	17	4	203	-158	-2	19	4	16	24	10	1	5	463	-487
4	15	4	354	-384	-6	17	4	288	-313	-1	19	4	17	134	11	1	5	267	-289
5	15	4	15	35	-5	17	4	5	-22	0	19	4	207	-142	12	1	5	547	-545
6	15	4	16	376	-4	17	4	16	283	1	19	4	135	31	13	1	5	378	402
7	15	4	579	598	-3	17	4	264	-305	2	19	4	18	-7	14	1	5	654	647
8	15	4	5	-66	-2	17	4	375	-472	3	19	4	6	-133	15	1	5	285	-371
9	15	4	425	-412	-1	17	4	597	-523	-20	1	5	17	-67	16	1	5	5	20
10	15	4	356	-302	0	17	4	249	258	-19	1	5	50	-202	17	1	5	18	-309
11	15	4	18	45	1	17	4	416	444	-18	1	5	16	61	-20	2	5	233	61
-12	16	4	96	-32	2	17	4	16	224	-17	1	5	197	156	-19	2	5	5	63
-11	16	4	144	146	3	17	4	16	-312	-16	1	5	344	-323	-18	2	5	113	-21
-10	16	4	217	-234	4	17	4	253	-189	-15	1	5	229	-156	-17	2	5	15	156
-9	16	4	16	20	5	17	4	175	162	-14	1	5	147	132	-16	2	5	521	-566
-8	16	4	234	267	6	17	4	263	347	-13	1	5	759	805	-15	2	5	248	219
-7	16	4	16	13	7	17	4	5	183	-12	1	5	683	686	-14	2	5	626	561
-6	16	4	174	-113	8	17	4	257	-358	-11	1	5	202	202	-13	2	5	13	259
-5	16	4	49	-150	-9	18	4	128	90	-10	1	5	848	-818	-12	2	5	888	920

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H	K	L	10FO	10FC	F	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC	F	K	L	10FC	10FC
-13	3	5	13	-83	-15	4	5	4	-66	-17	5	5	16	84	-17	6	5	16	-134	-17	7	5	16	163
-12	3	5	203	-134	-14	4	5	114	-219	-16	5	5	15	69	-16	6	5	139	42	-16	7	5	16	205
-11	3	5	941	-933	-13	4	5	575	-650	-15	5	5	333	-298	-15	6	5	15	-38	-15	7	5	299	-289
-10	3	5	11	113	-12	4	5	176	-172	-14	5	5	14	212	-14	6	5	138	163	-14	7	5	359	279
-9	3	5	312	-343	-11	4	5	283	269	-13	5	5	274	231	-13	6	5	443	411	-13	7	5	14	76
-8	3	5	260	-265	-10	4	5	949	951	-12	5	5	867	851	-12	6	5	446	-449	-12	7	5	420	449
-7	3	5	737	763	-9	4	5	70	119	-11	5	5	303	375	-11	6	5	285	-305	-11	7	5	519	490
-6	3	5	779	-782	-8	4	5	228	264	-10	5	5	424	-429	-10	6	5	308	277	-10	7	5	862	-623
-5	3	5	572	560	-7	4	5	656	677	-9	5	5	148	-132	-9	6	5	330	346	-9	7	5	217	-216
-4	3	5	914	1010	-6	4	5	108	147	-8	5	5	479	-482	-8	6	5	359	343	-8	7	5	498	524
-3	3	5	9	-77	-5	4	5	749	820	-7	5	5	678	693	-7	6	5	310	-285	-7	7	5	698	656
-2	3	5	9	-44	-4	4	5	1309	-1331	-6	5	5	31	-90	-6	6	5	1256	-1204	-6	7	5	465	465
-1	3	5	525	-560	-2	4	5	1050	-1110	-5	5	5	757	-819	-5	6	5	10	101	-5	7	5	1110	-1180
0	3	5	1596	-1657	-2	4	5	214	-198	-4	5	5	445	-450	-4	6	5	659	637	-4	7	5	1183	-1154
1	3	5	9	-155	-1	4	5	697	-750	-3	5	5	329	-316	-3	6	5	509	511	-3	7	5	10	-106
2	3	5	255	-280	0	4	5	1409	1379	-2	5	5	1176	1210	-2	6	5	1156	1118	-2	7	5	1267	1280
3	3	5	10	-120	1	4	5	639	-616	-1	5	5	150	-92	-1	6	5	334	-306	-1	7	5	167	214
4	3	5	1347	1342	2	4	5	356	-355	0	5	5	214	175	0	6	5	425	-417	0	7	5	382	368
5	3	5	370	-316	3	4	5	224	226	1	5	5	625	-562	1	6	5	3	93	1	7	5	664	-682
6	3	5	590	529	4	4	5	981	945	2	5	5	62	-52	2	6	5	56	70	2	7	5	346	-257
7	3	5	360	352	5	4	5	1268	1190	3	5	5	1736	1680	3	6	5	546	488	2	7	5	1393	1311
8	3	5	398	-415	6	4	5	255	230	4	5	5	10	-97	4	6	5	11	-141	4	7	5	865	-814
9	3	5	612	541	7	4	5	328	-295	5	5	5	3	-79	5	6	5	312	-302	5	7	5	112	-163
10	3	5	83	226	8	4	5	619	-631	6	5	5	1271	-1200	6	6	5	285	284	6	7	5	643	-663
11	3	5	129	-142	9	4	5	503	417	7	5	5	737	-709	7	6	5	393	330	7	7	5	12	-160
12	3	5	416	355	10	4	5	13	-269	8	5	5	770	723	8	6	5	12	24	8	7	5	1388	1441
13	3	5	118	-162	11	4	5	492	-429	9	5	5	209	265	9	6	5	13	-180	9	7	5	403	-396
14	3	5	132	-204	12	4	5	15	19	10	5	5	13	-34	10	6	5	471	-495	10	7	5	486	-456
15	3	5	260	330	13	4	5	406	-382	11	5	5	496	-462	11	6	5	131	96	11	7	5	566	-523
16	3	5	175	-196	14	4	5	533	463	12	5	5	15	-148	12	6	5	438	437	12	7	5	15	-203
17	3	5	6	36	15	4	5	108	41	13	5	5	244	296	13	6	5	49	97	12	7	5	526	597
-20	4	5	18	33	16	4	5	5	-85	14	5	5	88	180	14	6	5	16	161	14	7	5	253	267
-19	4	5	17	270	17	4	5	61	227	15	5	5	17	-71	15	6	5	224	-199	15	7	5	93	-234
-18	4	5	16	-187	-20	5	5	147	-191	16	5	5	17	-340	16	6	5	278	-303	16	7	5	18	-73
-17	4	5	16	47	-19	5	5	17	-23	-19	6	5	97	33	-19	7	5	80	-72	-19	8	5	57	-53
-16	4	5	15	27	-18	5	5	16	21	-18	6	5	17	94	-18	7	5	220	-218	-18	8	5	5	121

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR SRMEC C23H16BR2N2OS

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	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
-17	8	5	16	-35	-15	9	5	16	-1C3	-13	10	5	15	-144	-9	11	5	523	-539	
-16	8	5	297	-1E7	-14	9	5	377	-377	-12	10	5	345	-375	-8	11	5	13	121	
-15	8	5	129	95	-13	9	5	1C9	33	-11	10	5	255	291	-7	11	5	190	114	
-14	8	5	368	287	-12	9	5	324	-341	-10	10	5	4	-75	-6	11	5	115	-40	
-13	8	5	299	295	-11	9	5	694	-689	-9	10	5	561	553	-5	11	5	785	-784	
-12	8	5	122	212	-1C	9	5	4	4	-8	10	5	318	332	-4	11	5	557	-580	
-11	8	5	409	-4C5	-9	9	5	162	66	-7	10	5	66	-161	-3	11	5	133	120	
-10	8	5	766	-766	-8	9	5	119	79	-6	10	5	798	820	-2	11	5	613	641	
-9	8	5	312	277	-7	9	5	589	-6C5	-5	10	5	12	-227	-1	11	5	639	674	
-8	8	5	12	-24	-6	9	5	4C3	-4C5	-4	10	5	115	-214	0	11	5	402	-421	
-7	8	5	716	-675	-5	9	5	6C3	615	-3	10	5	585	-574	1	11	5	319	-345	
-6	8	5	644	-684	-4	9	5	342	3C7	-2	10	5	808	-797	2	11	5	362	372	
-5	8	5	1149	-1226	-3	9	5	169	-212	-1	10	5	410	-386	3	11	5	612	610	
-4	8	5	1399	1455	-2	9	5	241	280	0	10	5	122	43	4	11	5	902	880	
-3	8	5	1636	1587	-1	9	5	311	-327	1	10	5	160	166	5	11	5	641	-649	
-2	8	5	10	-9C	0	9	5	831	-812	2	10	5	352	346	6	11	5	729	-672	
-1	8	5	295	306	1	9	5	288	284	3	10	5	416	423	7	11	5	447	-486	
0	8	5	1472	-1498	2	9	5	321	-318	4	10	5	264	296	8	11	5	95	160	
1	8	5	400	454	3	9	5	339	-322	5	10	5	13	134	9	11	5	632	607	
2	8	5	864	826	4	9	5	385	362	6	10	5	206	183	10	11	5	226	-136	
3	8	5	371	-404	5	9	5	206	-217	7	10	5	240	-251	11	11	5	128	-189	
4	8	5	888	-892	6	9	5	557	535	8	10	5	14	168	12	11	5	205	-222	
5	8	5	1111	-1137	7	9	5	391	397	9	10	5	82	18	13	11	5	77	98	
6	8	5	313	-302	8	9	5	421	-4C1	10	10	5	15	-82	14	11	5	173	200	
7	8	5	699	753	9	9	5	226	237	11	10	5	298	-336	-16	12	5	18	10	
8	8	5	672	644	10	9	5	134	124	12	10	5	226	-311	-15	12	5	17	124	
9	8	5	790	-753	11	9	5	5	-29	13	10	5	17	-1	-14	12	5	210	201	
10	8	5	4	-171	12	9	5	16	-112	14	10	5	6	135	-13	12	5	260	201	
11	8	5	15	88	13	9	5	74	-122	-17	11	5	113	133	-12	12	5	200	-197	
12	8	5	233	256	14	9	5	5	-17	-16	11	5	264	-222	-11	12	5	315	-298	
13	8	5	634	627	15	9	5	258	222	-15	11	5	16	40	-10	12	5	125	-8	
14	8	5	526	-524	-18	10	5	133	41	-14	11	5	265	-178	-9	12	5	14	-11	
15	8	5	77	-47	-17	10	5	17	-148	-13	11	5	362	320	-8	12	5	212	247	
-18	9	5	18	369	-16	10	5	16	-157	-12	11	5	648	600	-7	12	5	13	-33	
-17	9	5	17	98	-15	10	5	224	-166	-11	11	5	15	-15	-6	12	5	656	-642	
-16	9	5	192	-194	-14	10	5	5	1C9	-10	11	5	14	101	-5	12	5	13	28	

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMED C23H16BR2N2OS

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H	K	L	10FD	10FC	H	K	L	10FD	10FC	H	K	L	10FD	10FC	H	K	L	10FD	10FC
3	13	5	661	600	-13	15	5	113	-183	0	16	5	121	-58	0	18	5	238	-217
4	13	5	159	-72	-12	15	5	205	-192	1	16	5	16	-64	1	18	5	69	148
5	13	5	15	-51	-11	15	5	303	-305	2	16	5	274	198	2	18	5	266	296
6	13	5	15	-231	-10	15	5	97	114	3	16	5	16	95	3	18	5	146	210
7	13	5	102	-25	-5	15	5	55	98	4	16	5	96	37	4	18	5	18	-160
8	13	5	397	426	-8	15	5	16	-95	5	16	5	271	201	5	18	5	351	-384
9	13	5	16	-75	-7	15	5	15	335	6	16	5	17	-43	-5	19	5	18	-145
10	13	5	344	-371	-6	15	5	15	-18	7	16	5	17	171	-4	19	5	18	-93
11	13	5	5	45	-5	15	5	330	364	8	16	5	18	-7	-3	19	5	116	102
12	13	5	207	66	-4	15	5	269	280	9	16	5	92	-122	-2	19	5	80	138
-14	14	5	266	267	-3	15	5	381	-400	-10	17	5	18	-63	-1	19	5	185	-67
-13	14	5	75	100	-2	15	5	117	-89	-9	17	5	17	-28	0	19	5	192	-100
-12	14	5	16	132	-1	15	5	374	-314	-8	17	5	239	-226	1	19	5	18	-47
-11	14	5	619	-629	0	15	5	125	61	-7	17	5	17	69	2	19	5	18	99
-10	14	5	5	-93	1	15	5	15	195	-6	17	5	16	-90	-20	0	6	17	-125
-9	14	5	15	69	2	15	5	15	-266	-5	17	5	289	-238	-19	0	6	467	-436
-8	14	5	15	68	2	15	5	356	-396	-4	17	5	5	-64	-18	0	6	88	100
-7	14	5	15	-28	4	15	5	112	-83	-3	17	5	306	-252	-17	0	6	440	-441
-6	14	5	614	-651	5	15	5	243	193	-2	17	5	324	343	-16	0	6	431	-397
-5	14	5	153	-199	6	15	5	505	452	-1	17	5	164	269	-15	0	6	70	-124
-4	14	5	421	441	7	15	5	16	214	0	17	5	237	-58	-14	0	6	506	-514
-3	14	5	639	563	8	15	5	17	-135	1	17	5	16	106	-13	0	6	1307	1347
-2	14	5	150	94	9	15	5	163	-64	2	17	5	5	-20	-12	0	6	205	229
-1	14	5	14	-198	10	15	5	18	-35	3	17	5	75	331	-11	0	6	275	254
0	14	5	264	-295	-12	16	5	6	-46	4	17	5	17	195	-10	0	6	964	981
1	14	5	15	106	-11	16	5	17	60	5	17	5	328	-234	-9	0	6	1189	-1080
2	14	5	538	540	-10	16	5	142	26	6	17	5	17	-266	-8	0	6	226	218
3	14	5	453	-440	-9	16	5	406	408	7	17	5	18	-122	-7	0	6	929	-896
4	14	5	482	-439	-8	16	5	276	237	-8	18	5	18	62	-6	0	6	1189	-1170
5	14	5	484	-491	-7	16	5	16	20	-7	18	5	18	-176	-5	0	6	182	-189
6	14	5	87	-108	-6	16	5	16	29	-6	18	5	284	-260	-4	0	6	400	-394
7	14	5	495	470	-5	16	5	145	7	-5	18	5	187	-60	-3	0	6	1166	-1178
8	14	5	16	-22	-4	16	5	96	37	-4	18	5	17	187	-2	0	6	741	779
9	14	5	163	-275	-3	16	5	15	-191	-3	18	5	389	428	-1	0	6	846	812
10	14	5	17	-55	-2	16	5	419	-377	-2	18	5	292	181	0	0	6	603	-597
11	14	5	126	22	-1	16	5	86	-266	-1	18	5	17	-192	1	0	6	2722	2644

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

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H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC
1	1	6	607	-508	0	2	6	341	-310	-1	3	€	875	-908	-2	4	6	429	465
2	1	6	191	-228	1	2	6	1617	-1601	0	3	€	415	385	-1	4	6	870	904
3	1	6	191	-162	2	2	6	926	-938	1	3	€	796	-769	0	4	6	592	581
4	1	6	800	-735	3	2	6	10	-146	2	3	€	276	262	1	4	6	1897	-1893
5	1	6	88	36	4	2	6	1139	1053	3	3	€	254	276	2	4	6	1014	-965
6	1	6	832	804	5	2	6	417	366	4	3	€	10	-80	3	4	6	3	81
7	1	6	593	-520	6	2	6	350	-359	5	3	€	107	197	4	4	6	488	434
8	1	6	336	-310	7	2	6	504	-458	6	3	€	339	-295	5	4	6	1071	1067
9	1	6	284	-244	8	2	6	302	228	7	3	€	12	-28	6	4	6	536	-543
10	1	6	13	-32	9	2	6	554	611	8	3	€	251	-257	7	4	6	569	-559
11	1	6	969	1006	10	2	6	13	€1	9	3	€	452	432	8	4	6	780	792
12	1	6	15	207	11	2	6	46	-173	10	3	€	13	-153	9	4	6	130	74
13	1	6	109	-116	12	2	6	702	-764	11	3	€	325	-279	10	4	6	76	88
14	1	6	226	252	13	2	6	193	121	12	3	€	206	191	11	4	6	375	-411
15	1	6	393	-324	14	2	6	16	249	13	3	€	16	-75	12	4	6	565	-593
16	1	6	5	45	15	2	6	17	-117	14	3	€	281	308	13	4	6	16	254
-20	2	6	111	34	16	2	6	17	176	15	3	€	17	-209	14	4	6	237	259
-19	2	6	17	-233	-20	3	6	18	-101	16	3	€	6	-147	15	4	6	17	38
-18	2	6	227	196	-15	3	6	195	198	-20	4	€	6	-43	16	4	6	6	-9
-17	2	6	502	496	-16	3	6	16	37	-19	4	€	17	-60	-20	5	6	369	241
-16	2	6	265	255	-17	2	6	167	-101	-18	4	€	105	-99	-19	5	6	17	-167
-15	2	6	14	51	-16	3	6	169	-130	-17	4	€	416	369	-18	5	6	177	181
-14	2	6	489	-504	-15	3	6	4	181	-16	4	€	454	476	-17	5	6	85	43
-13	2	6	374	-434	-14	3	6	76	-61	-15	4	€	195	-37	-16	5	6	507	-464
-12	2	6	329	321	-13	3	6	13	28	-14	4	€	131	-87	-15	5	6	209	-204
-11	2	6	207	279	-12	3	6	51	-43	-13	4	€	482	-452	-14	5	6	445	-409
-10	2	6	545	-512	-11	3	6	210	-226	-12	4	€	148	153	-13	5	6	62	156
-9	2	6	639	-640	-10	3	6	472	476	-11	4	€	12	122	-12	5	6	423	448
-8	2	6	147	-172	-9	3	6	220	-196	-10	4	€	778	-713	-11	5	6	598	-653
-7	2	6	901	892	-8	3	6	360	355	-9	4	€	278	-287	-10	5	6	261	-252
-6	2	6	1531	1645	-7	3	6	122	198	-8	4	€	11	-38	-9	5	6	86	61
-5	2	6	222	201	-6	3	6	459	-515	-7	4	€	1272	1272	-8	5	6	696	707
-4	2	6	1009	-1101	-5	3	6	359	347	-6	4	€	1150	1192	-7	5	6	1234	1244
-3	2	6	227	-204	-4	3	6	711	-673	-5	4	€	467	-430	-6	5	6	207	149
-2	2	6	9	-141	-3	3	6	1376	1412	-4	4	€	855	-869	-5	5	6	634	-678
-1	2	6	833	839	-2	3	6	536	514	-3	4	€	633	-659	-4	5	6	174	-186

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
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-2	6	6	1398	1469	-1	7	6	277	262	0	8	6	82	-225	2	9	6	457	-402
-1	6	6	537	-508	0	7	6	702	666	1	8	6	1439	-1357	3	9	6	765	668
0	6	6	83	115	1	7	6	356	351	2	8	6	618	-573	4	9	6	191	211
1	6	6	1863	1692	2	7	6	11	15	3	8	6	122	36	5	9	6	13	-69
2	6	6	11	36	3	7	6	259	-273	4	8	6	749	772	6	9	6	4	27
3	6	6	1578	1508	4	7	6	756	-751	5	8	6	430	426	7	9	6	603	-577
4	6	6	295	-296	5	7	6	12	9	6	8	6	592	-561	8	9	6	302	268
5	6	6	737	-717	6	7	6	164	155	7	8	6	164	-224	9	9	6	15	50
6	6	6	170	-228	7	7	6	536	-545	8	8	6	415	358	10	9	6	160	-39
7	6	6	646	-630	8	7	6	398	407	9	8	6	255	251	11	9	6	16	27
8	6	6	554	-499	9	7	6	4	-37	10	8	6	15	227	12	9	6	16	-221
9	6	6	437	419	10	7	6	5	58	11	8	6	306	-271	13	9	6	201	146
10	6	6	344	327	11	7	6	569	546	12	8	6	365	-415	14	9	6	99	44
11	6	6	117	-50	12	7	6	16	19	13	8	6	17	-148	-17	10	6	267	265
12	6	6	362	410	13	7	6	5	145	14	8	6	17	147	-16	10	6	159	-21
13	6	6	277	-223	14	7	6	17	-52	15	8	6	6	69	-15	10	6	63	176
14	6	6	5	130	15	7	6	6	-43	-18	9	6	224	219	-14	10	6	16	153
15	6	6	305	295	-15	8	6	18	-41	-17	9	6	136	-113	-13	10	6	349	-294
-19	7	6	150	110	-18	8	6	17	81	-16	9	6	5	192	-12	10	6	154	41
-18	7	6	169	-158	-17	8	6	478	521	-15	9	6	124	-247	-11	10	6	14	138
-17	7	6	116	-66	-16	8	6	144	155	-14	9	6	15	92	-10	10	6	231	-226
-16	7	6	16	25	-15	8	6	121	-140	-13	9	6	244	174	-9	10	6	13	-33
-15	7	6	185	165	-14	8	6	485	-411	-12	9	6	365	-372	-8	10	6	13	-162
-14	7	6	448	452	-13	8	6	462	-507	-11	9	6	198	171	-7	10	6	13	13
-13	7	6	335	390	-12	8	6	476	518	-10	9	6	13	-46	-6	10	6	814	801
-12	7	6	94	-207	-11	8	6	108	89	-9	9	6	13	71	-5	10	6	160	168
-11	7	6	13	77	-10	8	6	231	-245	-8	9	6	653	602	-4	10	6	600	-588
-10	7	6	376	-438	-9	8	6	453	-454	-7	9	6	94	-87	-3	10	6	213	130
-9	7	6	374	-388	-8	8	6	462	-488	-6	9	6	4	45	-2	10	6	12	-128
-8	7	6	336	-322	-7	8	6	858	827	-5	9	6	12	-39	-1	10	6	404	436
-7	7	6	973	-988	-6	8	6	600	655	-4	9	6	4	-59	0	10	6	88	-25
-6	7	6	109	-45	-5	8	6	415	355	-3	9	6	11	153	1	10	6	992	-950
-5	7	6	482	455	-4	8	6	11	-64	-2	9	6	717	674	2	10	6	12	-130
-4	7	6	420	435	-3	8	6	479	-506	-1	9	6	170	-149	3	10	6	41	-40
-3	7	6	953	987	-2	8	6	424	380	0	9	6	175	-189	4	10	6	333	380
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## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
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11	11	6	511	-554	-11	12	6	88	-136	-1	14	6	247	221	-11	16	6	18	63	6	17	6	129	-186
12	11	6	17	221	-10	13	6	358	-303	0	14	6	221	-159	-10	16	6	17	-160	-8	18	6	205	48
13	11	6	128	266	-9	13	6	15	59	1	14	6	115	-205	-9	16	6	17	-24	-7	18	6	18	-128
-16	12	6	6	63	-8	13	6	208	-240	2	14	6	370	-352	-8	16	6	16	-215	-6	18	6	330	-380
-15	12	6	17	33	-7	13	6	327	-256	3	14	6	15	-33	-7	16	6	37	210	-5	18	6	17	-212
-14	12	6	5	-156	-6	13	6	265	-254	4	14	6	141	314	-6	16	6	119	124	-4	18	6	54	-255
-13	12	6	451	481	-5	13	6	65	-60	5	14	6	16	95	-5	16	6	16	197	-3	18	6	17	-44
-12	12	6	149	68	-4	13	6	14	156	6	14	6	89	56	-4	16	6	5	-35	-2	18	6	244	322
-11	12	6	260	125	-3	13	6	542	622	7	14	6	16	-290	-3	16	6	16	-44	-1	18	6	17	-142
-10	12	6	305	283	-2	13	6	14	167	8	14	6	197	23	-2	16	6	16	-116	0	18	6	264	190
-9	12	6	15	-260	-1	13	6	4	116	9	14	6	197	165	-1	16	6	16	47	1	18	6	5	151
-8	12	6	288	303	0	13	6	9	64	10	14	6	18	221	0	16	6	16	30	2	18	6	289	190
-7	12	6	191	-218	1	13	6	4	24	-13	15	6	79	183	1	16	6	115	-315	3	18	6	6	110
-6	12	6	702	-755	2	13	6	305	314	-12	15	6	144	80	2	16	6	5	19	4	18	6	318	-256
-5	12	6	571	-575	3	13	6	429	-370	-11	15	6	141	-331	3	16	6	5	201	-2	19	6	141	67
-4	12	6	337	-368	4	13	6	467	-471	-10	15	6	52	26	4	16	6	53	154	-20	1	7	18	66
-3	12	6	13	21	5	13	6	15	-153	-9	15	6	200	-107	5	16	6	150	226	-19	1	7	340	-312
-2	12	6	218	290	6	13	6	203	-200	-8	15	6	528	546	6	16	6	411	-471	-18	1	7	16	-97
-1	12	6	426	400	7	13	6	432	353	-7	15	6	68	206	7	16	6	18	-87	-17	1	7	292	235
0	12	6	191	185	8	13	6	116	104	-6	15	6	195	-220	8	16	6	285	216	-16	1	7	5	-32
1	12	6	687	725	9	13	6	5	48	-5	15	6	189	-247	-10	17	6	221	-180	-15	1	7	282	235
2	12	6	197	211	10	13	6	103	97	-4	15	6	222	-166	-9	17	6	18	59	-14	1	7	287	-245
3	12	6	147	241	11	13	6	59	-39	-3	15	6	340	390	-8	17	6	286	295	-12	1	7	125	-88
4	12	6	123	-106	-14	14	6	414	-409	-2	15	6	187	155	-7	17	6	362	324	-12	1	7	294	307
5	12	6	639	-651	-13	14	6	200	-240	-1	15	6	85	-49	-6	17	6	17	-277	-11	1	7	379	329
6	12	6	69	46	-12	14	6	75	-33	0	15	6	204	-219	-5	17	6	17	-214	-10	1	7	434	460
7	12	6	15	-94	-11	14	6	108	-26	1	15	6	195	-250	-4	17	6	137	-69	-5	1	7	62	-67
8	12	6	16	-47	-10	14	6	16	-48	2	15	6	239	305	-3	17	6	235	108	-8	1	7	354	-364
9	12	6	16	20	-9	14	6	252	-313	3	15	6	149	228	-2	17	6	286	260	-7	1	7	408	-481
10	12	6	429	-454	-8	14	6	156	-237	4	15	6	253	261	-1	17	6	16	-230	-6	1	7	10	-71
11	12	6	77	55	-7	14	6	128	277	5	15	6	228	-220	0	17	6	639	-660	-5	1	7	10	-28
12	12	6	245	262	-6	14	6	62	658	6	15	6	17	-136	1	17	6	119	-180	-4	1	7	456	-483
-15	13	6	18	72	-5	14	6	134	85	7	15	6	5	-18	2	17	6	193	241	-3	1	7	1152	-1094
-14	13	6	382	399	-4	14	6	67	102	8	15	6	17	213	3	17	6	504	491	-2	1	7	453	-514
-13	13	6	16	87	-3	14	6	263	-158	9	15	6	346	298	4	17	6	125	232	-1	1	7	1187	1264

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H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC
0	1	7	765	754	-1	2	7	958	1CEO	-1	3	7	403	-419	-1	4	7	926	-961
1	1	7	494	453	0	2	7	389	356	0	3	7	222	226	0	4	7	43	33
2	1	7	821	-759	1	2	7	367	-362	1	3	7	935	-941	1	4	7	284	228
3	1	7	456	-454	2	2	7	841	-766	2	3	7	1257	-1229	2	4	7	1263	1247
4	1	7	810	825	3	2	7	802	-753	3	3	7	1097	1032	3	4	7	423	409
5	1	7	810	787	4	2	7	1056	971	4	3	7	36	-61	4	4	7	603	-569
6	1	7	141	77	5	2	7	772	-653	5	3	7	672	-580	5	4	7	495	485
7	1	7	806	-811	6	2	7	108	-319	6	3	7	12	169	6	4	7	50	29
8	1	7	554	-509	7	2	7	329	338	7	3	7	426	-404	7	4	7	4	151
9	1	7	13	-184	8	2	7	437	-448	8	3	7	761	760	8	4	7	194	199
10	1	7	562	515	9	2	7	713	706	9	3	7	1121	1124	9	4	7	167	-186
11	1	7	367	384	10	2	7	14	62	10	3	7	445	-436	10	4	7	14	114
12	1	7	420	-455	11	2	7	581	-618	11	3	7	15	-223	11	4	7	348	361
13	1	7	42	21	12	2	7	97	132	12	3	7	329	-358	12	4	7	129	-102
14	1	7	16	-27	13	2	7	186	-152	13	3	7	348	-401	13	4	7	150	50
15	1	7	17	172	14	2	7	17	204	14	3	7	328	382	14	4	7	17	23
16	1	7	147	256	15	2	7	17	254	15	3	7	116	-48	15	4	7	284	-282
-20	2	7	18	-133	-20	3	7	18	-52	-20	4	7	18	115	-19	5	7	17	-79
-19	2	7	129	-190	-19	3	7	17	-14	-19	4	7	5	35	-18	5	7	17	-159
-18	2	7	160	-52	-18	3	7	127	144	-18	4	7	254	-27	-17	5	7	16	-77
-17	2	7	141	16	-17	3	7	112	176	-17	4	7	39	42	-16	5	7	15	-181
-16	2	7	127	170	-16	3	7	524	570	-16	4	7	527	-481	-15	5	7	95	219
-15	2	7	512	487	-15	3	7	14	-165	-15	4	7	152	-138	-14	5	7	92	-100
-14	2	7	13	12	-14	3	7	514	-452	-14	4	7	14	112	-13	5	7	13	-126
-13	2	7	55	105	-13	3	7	113	-220	-13	4	7	13	-116	-12	5	7	585	624
-12	2	7	383	270	-12	3	7	115	-177	-12	4	7	4	-54	-11	5	7	180	144
-11	2	7	563	-561	-11	3	7	4	70	-11	4	7	12	26	-10	5	7	717	688
-10	2	7	11	111	-10	3	7	510	-452	-10	4	7	12	-72	-9	5	7	12	49
-9	2	7	862	-918	-9	3	7	1350	-1363	-9	4	7	504	550	-8	5	7	1069	-1168
-8	2	7	773	-748	-8	3	7	107	-124	-8	4	7	796	795	-7	5	7	11	77
-7	2	7	510	548	-7	3	7	1511	1459	-7	4	7	73	-104	-6	5	7	446	-378
-6	2	7	206	217	-6	3	7	965	1035	-6	4	7	462	472	-5	5	7	380	305
-5	2	7	1101	1135	-5	3	7	948	1028	-5	4	7	429	-439	-4	5	7	10	151
-4	2	7	878	-915	-4	3	7	587	-646	-4	4	7	32	-256	-3	5	7	836	-947
-3	2	7	1116	-1160	-3	3	7	442	-418	-3	4	7	434	534	-2	5	7	454	-461
-2	2	7	327	337	-2	3	7	1943	1985	-2	4	7	894	-948	-1	5	7	102	-66

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
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2	6	7	309	-283	3	7	7	345	-350	6	8	7	13	-218	9	9	7	439	443
3	6	7	746	745	4	7	7	363	341	7	8	7	222	-293	10	9	7	16	-297
4	6	7	12	-113	5	7	7	1049	1070	8	8	7	199	214	11	9	7	16	59
5	6	7	196	167	6	7	7	352	-342	9	8	7	438	446	12	9	7	17	-224
6	6	7	310	-310	7	7	7	329	-328	10	8	7	170	39	13	9	7	165	-147
7	6	7	755	-652	8	7	7	14	-42	11	8	7	457	-482	-17	10	7	205	-142
8	6	7	338	305	9	7	7	375	-354	12	8	7	112	-123	-16	10	7	405	-408
9	6	7	93	39	10	7	7	415	512	13	8	7	17	175	-15	10	7	16	-231
10	6	7	181	-149	11	7	7	132	-140	14	8	7	6	159	-14	10	7	143	175
11	6	7	216	265	12	7	7	16	-175	-18	9	7	97	50	-13	10	7	5	132
12	6	7	102	-177	13	7	7	6	232	-17	9	7	339	316	-12	10	7	5	207
13	6	7	5	-92	14	7	7	118	8	-16	9	7	334	371	-11	10	7	185	-193
14	6	7	17	57	-18	8	7	5	106	-15	9	7	102	-263	-10	10	7	522	-574
15	6	7	211	-210	-17	8	7	17	-42	-14	9	7	190	-92	-9	10	7	757	776
-19	7	7	231	-187	-16	8	7	155	166	-13	9	7	145	-242	-8	10	7	359	403
-18	7	7	458	-391	-15	8	7	16	-49	-12	9	7	229	-230	-7	10	7	619	603
-17	7	7	264	290	-14	8	7	15	-104	-11	9	7	14	-103	-6	10	7	88	120
-16	7	7	16	107	-13	8	7	47	117	-10	9	7	476	-501	-5	10	7	819	-848
-15	7	7	372	379	-12	8	7	375	271	-9	9	7	650	-658	-4	10	7	4	73
-14	7	7	5	101	-11	8	7	13	-178	-8	9	7	162	134	-3	10	7	12	-71
-13	7	7	604	-675	-10	8	7	73	-124	-7	9	7	541	509	-2	10	7	210	-202
-12	7	7	312	289	-9	8	7	546	-570	-6	9	7	497	534	-1	10	7	334	-301
-11	7	7	306	268	-8	7	7	334	-357	-5	9	7	643	699	0	10	7	657	-676
-10	7	7	13	214	-7	8	7	578	574	-4	9	7	241	-320	1	10	7	321	315
-9	7	7	412	347	-6	8	7	12	12	-3	9	7	135	24	2	10	7	498	521
-8	7	7	589	-553	-5	8	7	256	-253	-2	9	7	876	936	3	10	7	639	573
-7	7	7	139	-146	-4	8	7	12	-62	-1	9	7	103	-147	4	10	7	175	-240
-6	7	7	400	319	-3	8	7	449	-406	0	9	7	219	-158	5	10	7	14	-116
-5	7	7	404	-394	-2	8	7	909	973	1	9	7	790	-823	6	10	7	14	-85
-4	7	7	206	-253	-1	8	7	819	813	2	9	7	497	-528	7	10	7	536	544
-3	7	7	616	-627	0	8	7	555	-524	3	9	7	447	466	8	10	7	440	461
-2	7	7	405	-357	1	8	7	382	-387	4	9	7	225	182	9	10	7	16	-87
-1	7	7	1060	1030	2	8	7	756	-750	5	9	7	13	-227	10	10	7	16	-83
0	7	7	813	779	3	8	7	12	-68	6	9	7	14	21	11	10	7	17	-142
1	7	7	210	-227	4	8	7	753	723	7	9	7	178	-104	12	10	7	108	-14

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
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-10	12	7	153	-216	-1	13	7	660	678	-13	15	7	6	-81	3	16	7	27	-36
-9	12	7	278	-227	0	13	7	150	143	-12	15	7	18	-285	4	16	7	108	90
-8	12	7	144	151	1	13	7	15	-48	-11	15	7	184	-60	5	16	7	172	-251
-7	12	7	14	-155	2	13	7	176	-204	-10	15	7	17	-155	6	16	7	161	-117
-6	12	7	119	119	3	13	7	351	-365	-9	15	7	16	-203	-9	17	7	6	-15
-5	12	7	147	-224	4	13	7	15	418	-8	15	7	102	180	-8	17	7	18	-207
-4	12	7	275	-183	5	13	7	264	331	-7	15	7	209	208	-7	17	7	17	-67
-3	12	7	263	312	6	13	7	16	-144	-6	15	7	16	124	-6	17	7	347	-384
-2	12	7	563	577	7	13	7	402	-416	-5	15	7	5	32	-5	17	7	17	232
-1	12	7	235	-231	8	13	7	126	46	-4	15	7	306	-363	-4	17	7	17	84
0	12	7	129	-135	5	13	7	17	175	-3	15	7	16	330	-3	17	7	181	-195
1	12	7	478	-426	10	13	7	224	320	-2	15	7	509	536	-2	17	7	255	144
2	12	7	4	-162	-14	14	7	125	-123	-1	15	7	16	40	-1	17	7	145	-198
3	12	7	429	403	-13	14	7	120	127	0	15	7	327	-292	0	17	7	342	340
4	12	7	116	100	-12	14	7	17	154	1	15	7	554	-611	1	17	7	273	263
5	12	7	15	49	-11	14	7	16	-203	2	15	7	276	-240	2	17	7	17	7
6	12	7	15	33	-10	14	7	16	62	3	15	7	16	162	3	17	7	17	44
7	12	7	433	-405	-5	14	7	508	-523	4	15	7	16	-63	4	17	7	18	-106
8	12	7	16	36	-8	14	7	262	-249	5	15	7	40	-129	5	17	7	59	135
9	12	7	16	18	-7	14	7	134	246	6	15	7	17	129	-6	18	7	18	55
10	12	7	5	59	-6	14	7	15	20	7	15	7	17	163	-5	18	7	18	-212
11	12	7	18	23	-5	14	7	337	351	8	15	7	18	269	-4	18	7	6	-175
-15	13	7	18	213	-4	14	7	204	-250	-11	16	7	18	-288	-3	18	7	56	-46
-14	13	7	17	-80	-3	14	7	15	-88	-10	16	7	5	-50	-2	18	7	256	232
-13	13	7	229	-128	-2	14	7	219	226	-9	16	7	17	98	-1	18	7	147	134
-12	13	7	16	167	-1	14	7	525	485	-8	16	7	343	377	0	18	7	18	32
-11	13	7	16	156	0	14	7	199	-201	-7	16	7	540	572	1	18	7	18	-58
-10	13	7	16	113	1	14	7	460	-422	-6	16	7	16	-148	2	18	7	170	-207
-9	13	7	15	42	2	14	7	201	-223	-5	16	7	350	-345	-20	0	8	150	25
-8	13	7	15	-92	3	14	7	16	136	-4	16	7	16	-120	-19	0	8	107	-124
-7	13	7	357	-296	4	14	7	627	629	-3	16	7	146	118	-18	0	8	406	-369
-6	13	7	520	547	5	14	7	197	-261	-2	16	7	5	-35	-17	0	8	203	-254
-5	13	7	373	-362	6	14	7	524	-518	-1	16	7	5	-101	-16	0	8	128	163
-4	13	7	47	123	7	14	7	239	-223	0	16	7	345	-328	-15	0	8	246	-126
-3	13	7	14	-53	8	14	7	17	-53	1	16	7	108	-62	-14	0	8	365	-351

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR SRMEC C23H16BR2N2OS

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
-13	1	8	183	120	-13	2	8	263	-260	-13	3	8	13	-172	-12	4	8	198	-261
-12	1	8	642	674	-12	2	8	373	-428	-12	3	8	176	188	-11	4	8	108	-244
-11	1	8	204	117	-11	2	8	236	-241	-11	3	8	110	144	-10	4	8	12	177
-10	1	8	814	-773	-10	2	8	152	152	-10	3	8	112	-90	-9	4	8	355	381
-9	1	8	469	-454	-9	2	8	240	-235	-9	3	8	148	127	-8	4	8	700	-660
-8	1	8	111	-179	-8	2	8	69	-105	-8	3	8	183	-176	-7	4	8	34	8
-7	1	8	321	-356	-7	2	8	469	-452	-7	3	8	316	-319	-6	4	8	193	190
-6	1	8	334	-340	-6	2	8	174	138	-6	3	8	339	317	-5	4	8	11	32
-5	1	8	822	-812	-5	2	8	976	956	-5	3	8	11	134	-4	4	8	511	592
-4	1	8	488	429	-4	2	8	577	700	-4	3	8	694	-676	-3	4	8	11	186
-3	1	8	1449	1533	-3	2	8	59	-159	-3	3	8	364	416	-2	4	8	424	-382
-2	1	8	924	901	-2	2	8	320	-330	-2	3	8	10	-12	-1	4	8	243	162
-1	1	8	588	581	-1	2	8	10	-68	-1	3	8	103	-30	0	4	8	279	251
0	1	8	93	-62	0	2	8	392	330	0	3	8	120	84	1	4	8	63	-61
1	1	8	864	-888	1	2	8	167	217	1	3	8	362	-374	2	4	8	90	-138
2	1	8	609	644	2	2	8	445	-455	2	3	8	344	328	3	4	8	661	-596
3	1	8	11	90	3	2	8	810	-757	3	3	8	203	207	4	4	8	39	-97
4	1	8	12	-133	4	2	8	12	58	4	3	8	12	-137	5	4	8	989	966
5	1	8	182	-182	5	2	8	246	256	5	3	8	119	-79	6	4	8	292	299
6	1	8	687	-708	6	2	8	330	288	6	3	8	123	-167	7	4	8	248	212
7	1	8	59	125	7	2	8	128	193	7	3	8	4	-31	8	4	8	470	-487
8	1	8	873	891	8	2	8	211	-166	8	3	8	187	-148	9	4	8	15	-279
9	1	8	14	-79	9	2	8	14	-57	9	3	8	174	107	10	4	8	580	606
10	1	8	120	111	10	2	8	242	337	10	3	8	15	65	11	4	8	161	113
11	1	8	15	99	11	2	8	259	205	11	3	8	15	109	12	4	8	16	139
12	1	8	207	-141	12	2	8	5	-74	12	3	8	232	-292	13	4	8	17	-29
13	1	8	285	301	13	2	8	17	24	13	3	8	116	-117	14	4	8	17	-465
14	1	8	5	-53	14	2	8	17	-257	14	3	8	5	66	-19	5	8	5	82
15	1	8	18	-235	15	2	8	6	161	-20	4	8	18	59	-18	5	8	17	-211
-20	2	8	150	239	-20	3	8	63	23	-19	4	8	17	-79	-17	5	8	16	69
-19	2	8	17	-114	-19	3	8	17	46	-18	4	8	136	46	-16	5	8	16	-137
-18	2	8	5	45	-18	3	8	16	82	-17	4	8	134	198	-15	5	8	15	-73
-17	2	8	5	32	-17	3	8	151	243	-16	4	8	147	166	-14	5	8	534	-499
-16	2	8	15	190	-16	2	8	269	-278	-15	4	8	585	517	-13	5	8	14	-76
-15	2	8	315	318	-15	3	8	15	-113	-14	4	8	80	-200	-12	5	8	60	-185
-14	2	8	184	-134	-14	3	8	14	87	-13	4	8	543	-516	-11	5	8	57	-72

## DESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEO C23H16BR2N2OS

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
-6	6	8	445	-363	-6	7	8	119	269	-2	8	8	36	-158	2	9	8	13	-11
-7	6	8	312	-331	-5	7	8	725	-749	-1	8	8	342	-309	3	9	8	494	470
-6	6	8	11	130	-4	7	8	128	-63	0	8	8	410	428	4	9	8	14	-101
-5	6	8	738	723	-3	7	8	324	347	1	8	8	225	224	5	9	8	340	307
-4	6	8	1160	-1216	-2	7	8	850	656	2	8	8	251	-237	6	9	8	115	-222
-3	6	8	793	-783	-1	7	8	807	829	3	8	8	497	-526	7	9	8	638	-590
-2	6	8	134	85	0	7	8	48	-71	4	8	8	582	-537	8	9	8	228	177
-1	6	8	3	99	1	7	8	393	-413	5	8	8	4	113	9	9	8	5	-201
0	6	8	1436	1463	2	7	8	80	-48	6	8	8	14	-120	10	9	8	16	211
1	6	8	66	-139	3	7	8	115	92	7	8	8	313	316	11	9	8	199	226
2	6	8	363	-388	4	7	8	13	164	8	8	8	15	49	12	9	8	420	-309
3	6	8	380	395	5	7	8	120	-301	9	8	8	232	249	-17	10	8	278	148
4	6	8	170	-36	6	7	8	257	-253	10	8	8	16	121	-16	10	8	78	42
5	6	8	399	412	7	7	8	63	-45	11	8	8	17	-61	-15	10	8	221	165
6	6	8	292	-262	8	7	8	641	656	12	8	8	163	21	-14	10	8	153	-93
7	6	8	1154	-1108	9	7	8	461	429	13	8	8	18	-160	-13	10	8	270	-198
8	6	8	350	-286	10	7	8	16	-40	-18	9	8	18	-104	-12	10	8	15	-21
9	6	8	15	-56	11	7	8	116	-80	-17	9	8	170	203	-11	10	8	15	-73
10	6	8	252	285	12	7	8	17	-196	-16	9	8	17	93	-10	10	8	197	-83
11	6	8	5	75	13	7	8	146	310	-15	9	8	422	-374	-9	10	8	616	572
12	6	8	17	-270	-18	8	8	18	-119	-14	9	8	16	251	-8	10	8	285	-286
13	6	8	274	-293	-17	8	8	17	-65	-13	9	8	217	-267	-7	10	8	13	-111
14	6	8	330	301	-16	8	8	5	153	-12	9	8	15	277	-6	10	8	120	39
-19	7	8	233	-122	-15	8	8	65	219	-11	9	8	610	546	-5	10	8	13	-32
-18	7	8	17	-105	-14	8	8	120	59	-10	9	8	608	-602	-4	10	8	837	856
-17	7	8	5	183	-13	8	8	245	-350	-9	9	8	173	202	-3	10	8	244	-339
-16	7	8	72	115	-12	8	8	277	-169	-8	9	8	573	-613	-2	10	8	118	-243
-15	7	8	191	159	-11	8	8	346	-416	-7	9	8	13	101	-1	10	8	295	333
-14	7	8	459	461	-10	8	8	4	59	-6	9	8	352	305	0	10	8	13	-146
-13	7	8	93	37	-5	8	8	167	-182	-5	9	8	246	-195	1	10	8	13	65
-12	7	8	662	681	-8	8	8	13	-253	-4	9	8	12	219	2	10	8	73	-99
-11	7	8	220	-201	-7	8	8	523	587	-3	9	8	179	-269	3	10	8	642	-688
-10	7	8	621	-636	-6	8	8	12	34	-2	9	8	99	-223	4	10	8	14	245
-9	7	8	385	-369	-5	8	8	854	866	-1	9	8	12	-235	5	10	8	410	377
-8	7	8	512	-552	-4	8	8	264	370	0	9	8	136	-144	6	10	8	15	90
-7	7	8	452	433	-3	8	8	313	-357	1	9	8	13	55	7	10	8	447	438

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

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H	K	L	10FO	10FC	H	K	L	10FC	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
-12	12	8	5	-27	-2	13	8	740	717	-10	15	8	74	-32	-6	17	8	18	121
-11	12	8	580	660	-1	13	8	719	746	-9	15	8	190	-128	-5	17	8	5	142
-10	12	8	341	362	0	13	8	185	-235	-8	15	8	16	-51	-4	17	8	17	-138
-9	12	8	141	-156	1	13	8	241	-194	-7	15	8	16	132	-3	17	8	288	-320
-8	12	8	195	-73	2	13	8	15	-121	-6	15	8	16	50	-2	17	8	119	-54
-7	12	8	554	-571	3	13	8	15	-187	-5	15	8	147	-135	-1	17	8	121	94
-6	12	8	335	403	4	13	8	268	309	-4	15	8	387	-368	0	17	8	17	56
-5	12	8	398	-463	5	13	8	389	-363	-3	15	8	225	149	1	17	8	130	-93
-4	12	8	602	-655	6	13	8	5	-193	-2	15	8	16	-204	2	17	8	180	-197
-3	12	8	65	40	7	13	8	17	184	-1	15	8	5	71	3	17	8	18	108
-2	12	8	296	-226	8	13	8	17	187	0	15	8	222	-61	-3	18	8	18	-152
-1	12	8	664	666	9	13	8	442	436	1	15	8	171	-239	-2	18	8	103	80
0	12	8	260	248	-12	14	8	169	120	2	15	8	74	182	-20	1	9	38	60
1	12	8	115	-137	-12	14	8	402	-348	3	15	8	17	-99	-19	1	9	17	-137
2	12	8	306	-324	-11	14	8	237	-140	4	15	8	17	169	-18	1	9	148	13
3	12	8	15	116	-10	14	8	90	-75	5	15	8	17	24	-17	1	9	152	-234
4	12	8	367	350	-9	14	8	169	-174	6	15	8	6	-26	-16	1	9	15	96
5	12	8	232	173	-8	14	8	16	27	7	15	8	18	-11	-15	1	9	28	-24
6	12	8	238	177	-7	14	8	131	-66	-10	16	8	18	43	-14	1	9	108	-76
7	12	8	734	-825	-6	14	8	16	213	-9	16	8	17	54	-13	1	9	141	116
8	12	8	17	-17	-5	14	8	521	487	-8	16	8	200	-115	-12	1	9	13	-79
9	12	8	17	107	-4	14	8	139	197	-7	16	8	278	222	-11	1	9	13	165
10	12	8	193	-98	-3	14	8	5	114	-6	16	8	17	-11	-10	1	9	350	309
-15	13	8	18	162	-2	14	8	104	-66	-5	16	8	5	97	-9	1	9	213	199
-14	13	8	201	45	-1	14	8	15	-160	-4	16	8	397	433	-8	1	9	41	128
-13	13	8	17	172	0	14	8	222	-222	-3	16	8	16	-151	-7	1	9	364	-359
-12	13	8	383	370	1	14	8	16	202	-2	16	8	139	-89	-6	1	9	378	-367
-11	13	8	315	-297	2	14	8	16	-114	-1	16	8	271	-198	-5	1	9	11	-127
-10	13	8	88	-184	3	14	8	16	32	0	16	8	150	21	-4	1	9	725	709
-9	13	8	323	-323	4	14	8	73	-170	1	16	8	105	78	-3	1	9	52	-87
-8	13	8	15	-108	5	14	8	246	-322	2	16	8	166	62	-2	1	9	80	-202
-7	13	8	504	512	6	14	8	17	173	3	16	8	270	-274	-1	1	9	3	-18
-6	13	8	140	-121	7	14	8	5	93	4	16	8	18	43	0	1	9	488	-433
-5	13	8	413	-406	8	14	8	277	62	5	16	8	6	158	1	1	9	692	621
-4	13	8	15	-115	-12	15	8	175	248	-8	17	8	18	61	2	1	9	12	112
-3	13	8	15	65	-11	15	8	17	111	-7	17	8	18	74	3	1	9	12	81
															4	2	9	319	-103

## DESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC	
5	2	9	496	-503	7	3	9	512	-498	10	4	5	16	278	13	5	9	6	-42	
6	2	9	730	773	8	3	9	15	-123	11	4	5	16	-122	-19	6	9	203	172	
7	2	9	14	-51	5	3	9	295	-261	12	4	5	287	-313	-18	6	9	17	-164	
8	2	9	416	-409	10	3	9	5	70	13	4	5	123	104	-17	6	9	210	151	
9	2	9	15	95	11	3	9	413	417	-19	5	5	6	-21	-16	6	9	16	-27	
10	2	9	545	-521	12	3	9	501	-490	-18	5	5	17	-189	-15	6	9	16	-75	
11	2	9	526	555	13	3	9	17	-168	-17	5	5	16	-135	-14	6	9	235	189	
12	2	9	218	204	-15	4	9	18	-85	-16	5	5	151	-149	-13	6	9	305	-319	
13	2	9	133	-159	-18	4	9	17	-80	-15	5	5	120	94	-12	6	9	396	354	
14	2	9	218	183	-17	4	9	217	256	-14	5	5	15	-49	-11	6	9	13	33	
-19	3	9	164	265	-16	4	9	316	-322	-13	5	5	179	-79	-10	6	9	330	-343	
-18	3	9	17	-139	-15	4	9	15	85	-12	5	5	14	177	-9	6	9	202	-295	
-17	3	9	5	-69	-14	4	9	502	-483	-11	5	5	74	-263	-8	6	9	510	-480	
-16	3	9	324	187	-13	4	9	347	-309	-10	5	5	705	731	-7	6	9	28	-67	
-15	3	9	278	262	-12	4	9	276	320	-9	5	5	70	44	-6	6	9	362	339	
-14	3	9	187	223	-11	4	9	252	309	-8	5	5	12	135	-5	6	9	12	-224	
-13	3	9	442	-445	-10	4	9	158	112	-7	5	5	12	19	-4	6	9	4	85	
-12	3	9	1150	-1166	-5	4	9	12	-101	-6	5	5	352	-394	-3	6	9	12	80	
-11	3	9	13	143	-6	4	9	161	-233	-5	5	5	260	242	-2	6	9	430	-398	
-10	3	9	12	-86	-7	4	9	259	264	-4	5	5	267	-248	-1	6	9	45	122	
-9	3	9	140	154	-6	4	9	9	1180	1188	-3	5	5	100	-121	0	6	9	190	195
-8	3	9	117	-111	-5	4	9	11	-150	-2	5	5	329	299	1	6	9	12	-56	
-7	3	9	651	-677	-4	4	9	365	-387	-1	5	5	94	-151	2	6	9	353	317	
-6	3	9	922	866	-3	4	9	441	-484	0	5	5	420	443	3	6	9	13	123	
-5	3	9	1433	1469	-2	4	9	518	-551	1	5	5	89	164	4	6	9	13	-93	
-4	3	9	3	57	-1	4	9	965	978	2	5	5	12	217	5	6	9	88	24	
-3	3	9	11	14	0	4	9	268	-280	3	5	5	121	-188	6	6	9	371	-373	
-2	3	9	906	-1031	1	4	9	507	-523	4	5	5	301	-241	7	6	9	328	-320	
-1	3	9	774	-716	2	4	9	12	66	5	5	5	282	-295	8	6	9	254	284	
0	3	9	1300	1324	3	4	9	12	47	6	5	5	252	240	9	6	9	16	138	
1	3	9	12	-125	4	4	9	1015	579	7	5	5	621	505	10	6	9	103	-29	
2	3	9	955	-914	5	4	9	460	483	8	5	5	128	-86	11	6	9	5	37	
3	3	9	861	-852	6	4	9	293	-336	9	5	5	15	-86	12	6	9	193	-320	
4	3	9	1437	-1410	7	4	9	229	-147	10	5	5	79	-21	13	6	9	18	71	
5	3	9	852	811	8	4	9	5	23	11	5	5	169	-167	-18	7	9	18	-104	
6	3	9	464	468	9	4	9	115	49	12	5	5	348	344	-17	7	9	17	104	
															-18	8	9	18	-54	

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2CS

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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC
-11	8	9	14	E	-5	9	9	611	643	2	10	5	829	-753	10	11	9	308	-225
-10	8	9	285	-268	-4	9	9	599	562	3	10	5	129	143	-15	12	9	167	41
-9	8	9	333	361	-3	9	9	108	13	4	10	5	574	572	-14	12	9	17	-35
-8	8	9	209	-215	-2	9	9	957	-1029	5	10	5	450	385	-13	12	9	17	-154
-7	8	9	111	53	-1	9	9	13	-107	6	10	5	149	-85	-12	12	9	188	270
-6	8	9	544	-519	0	9	9	386	414	7	10	5	16	-250	-11	12	9	16	30
-5	8	9	13	68	1	9	9	242	-294	8	10	5	16	136	-10	12	9	16	-105
-4	8	9	374	358	2	9	9	14	-229	9	10	5	17	40	-9	12	9	57	-164
-3	8	9	140	-65	3	9	9	784	-761	10	10	5	72	295	-8	12	9	396	-429
-2	8	9	653	676	4	9	9	108	-144	11	10	5	18	-258	-7	12	9	15	-131
-1	8	9	337	-355	5	9	9	792	814	-16	11	5	6	32	-6	12	9	15	40
0	8	9	278	304	6	9	9	15	E4	-15	11	5	151	-22	-5	12	9	68	70
1	8	9	641	620	7	9	9	397	-368	-14	11	9	53	-15	-4	12	9	15	-119
2	8	9	585	-553	8	9	9	208	-231	-13	11	5	296	327	-3	12	9	5	-127
3	8	9	13	118	9	9	5	-185	-12	11	5	16	-109	-2	12	9	116	-92	
4	8	9	704	-778	10	9	9	235	300	-11	11	5	16	92	-1	12	9	268	242
5	8	9	14	-65	11	9	9	18	312	-10	11	5	15	-64	0	12	9	120	62
6	8	9	628	653	-17	10	9	18	110	-9	11	5	49	-119	1	12	9	15	-61
7	8	9	400	-398	-16	10	9	54	104	-8	11	5	429	385	2	12	9	15	-53
8	8	9	68	-163	-15	10	9	142	-19	-7	11	5	41	-50	3	12	9	117	-6
9	8	9	102	-192	-14	10	9	147	-257	-6	11	5	258	184	4	12	9	16	40
10	8	9	17	-14	-13	10	9	502	-530	-5	11	5	285	-282	5	12	9	16	31
11	8	9	316	358	-12	10	9	16	233	-4	11	5	14	-97	6	12	9	230	-237
12	8	9	18	20	-11	10	9	15	179	-3	11	5	14	-134	7	12	9	17	57
-17	9	9	6	87	-10	10	9	151	173	-2	11	5	118	-63	8	12	9	164	15
-16	9	9	5	-18	-5	10	9	265	263	-1	11	5	256	266	9	12	9	140	68
-15	9	9	138	98	-8	10	9	157	-195	0	11	5	66	-118	-14	13	9	178	-18
-14	9	9	107	175	-7	10	9	757	745	1	11	5	408	377	-13	13	9	145	147
-13	9	9	340	-355	-6	10	9	501	478	2	11	5	15	-22	-12	13	9	17	-9
-12	9	9	521	-528	-5	10	9	246	-246	3	11	5	116	71	-11	13	9	16	-44
-11	9	9	15	112	-4	10	9	25	-135	4	11	5	15	143	-10	13	9	5	93
-10	9	9	4	-99	-3	10	9	1052	-1114	5	11	5	456	-412	-9	13	9	288	228
-9	9	9	209	162	-2	10	9	411	394	6	11	5	180	148	-8	13	9	16	-109
-8	9	9	279	-311	-1	10	9	426	455	7	11	5	16	-86	-7	13	9	163	40
-7	9	9	756	-747	0	10	9	88	E4	8	11	5	141	108	-6	13	9	15	32
-6	9	9	471	556	1	10	9	137	-107	9	11	5	17	95	-5	13	9	147	-117

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2GS

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H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC
-9	15	9	172	231	-18	C	10	233	-199	-15	1	10	474	421	-12	2	10	550	-548
-8	15	9	160	-197	-17	C	10	597	-593	-14	1	10	5	-97	-11	2	10	85	216
-7	15	9	320	-267	-16	C	10	128	75	-13	1	10	367	431	-10	2	10	134	150
-6	15	9	216	188	-15	C	10	210	-224	-12	1	10	13	57	-9	2	10	12	-23
-5	15	9	145	171	-14	C	10	557	455	-11	1	10	13	-5	-8	2	10	169	302
-4	15	9	155	202	-13	C	10	14	-154	-10	1	10	230	-183	-7	2	10	159	-137
-3	15	9	16	-88	-12	C	10	654	-627	-9	1	10	403	-427	-6	2	10	12	-125
-2	15	9	320	-299	-11	C	10	379	325	-8	1	10	664	-647	-5	2	10	537	458
-1	15	9	48	-56	-10	C	10	52	51	-7	1	10	373	-316	-4	2	10	484	490
0	15	9	221	169	-9	C	10	1170	1157	-6	1	10	267	-283	-3	2	10	12	80
1	15	9	17	-165	-8	C	10	640	645	-5	1	10	12	105	-2	2	10	12	180
2	15	9	248	-185	-7	C	10	973	-929	-4	1	10	543	592	-1	2	10	402	-419
3	15	9	195	-222	-6	C	10	466	-465	-3	1	10	53	308	0	2	10	119	-53
4	15	9	166	-58	-5	C	10	910	-1012	-2	1	10	433	-450	1	2	10	376	372
5	15	9	18	381	-4	C	10	12	-110	-1	1	10	649	682	2	2	10	429	-420
-9	16	9	244	-168	-3	C	10	137	146	0	1	10	166	122	3	2	10	92	124
-8	16	9	56	12	-2	C	10	912	-933	1	1	10	670	639	4	2	10	150	-108
-7	16	9	352	383	-1	C	10	659	-589	2	1	10	12	49	5	2	10	132	18
-6	16	9	263	258	0	C	10	391	359	3	1	10	856	-851	6	2	10	492	515
-5	16	9	5	77	1	C	10	917	857	4	1	10	13	265	7	2	10	15	-42
-4	16	9	5	-218	2	C	10	346	337	5	1	10	49	26	8	2	10	251	287
-3	16	9	308	-368	3	C	10	243	-325	6	1	10	4	-186	9	2	10	5	100
-2	16	9	17	48	4	C	10	1650	-1656	7	1	10	15	73	10	2	10	193	-74
-1	16	9	17	394	5	C	10	13	-108	8	1	10	387	-375	11	2	10	17	135
0	16	9	177	-27	6	C	10	578	617	9	1	10	295	281	12	2	10	5	64
1	16	9	17	-174	7	C	10	145	-55	10	1	10	237	235	13	2	10	18	-72
2	16	9	344	-388	8	C	10	454	438	11	1	10	157	-40	-19	3	10	60	-161
3	16	9	194	-81	9	C	10	1047	-1148	12	1	10	17	-181	-18	3	10	5	-14
-6	17	9	6	-175	10	C	10	421	-436	13	1	10	18	-37	-17	3	10	16	82
-5	17	9	18	-24	11	C	10	318	341	-19	2	10	18	158	-16	3	10	212	-188
-4	17	9	281	-236	12	C	10	275	-212	-18	2	10	59	10	-15	3	10	217	134
-3	17	9	6	-17	13	C	10	479	474	-17	2	10	5	-82	-14	3	10	69	-20
-2	17	9	161	186	-19	1	10	189	-125	-16	2	10	207	194	-13	3	10	14	79
-1	17	9	130	-111	-16	1	10	118	110	-15	2	10	15	61	-12	3	10	14	-139
0	17	9	141	137	-17	1	10	16	-66	-14	2	10	106	-60	-11	3	10	206	-146
-19	0	10	17	-63	-16	1	10	150	9	-13	2	10	80	120	-10	3	10	286	-276

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR ERMEC C23H16BR2N2OS

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H	K	L	10FO	10FC	F	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC	F	K	L	10FC	10FC
-5	4	10	691	702	-1	5	10	415	-454	4	6	10	499	-486	5	7	10	16	-70	-13	9	10	381	-357
-4	4	10	232	198	0	5	10	136	-145	5	6	10	392	401	10	7	10	409	451	-12	9	10	15	109
-3	4	10	39	118	1	5	10	577	-577	6	6	10	218	-248	11	7	10	6	-15	-11	9	10	15	84
-2	4	10	310	247	2	5	10	153	166	7	6	10	15	157	-17	9	10	153	-20	-10	9	10	15	-80
-1	4	10	526	-523	3	5	10	426	432	8	6	10	16	-347	-16	8	10	156	135	-5	9	10	4	-63
0	4	10	242	-261	4	5	10	493	-467	9	6	10	573	-625	-15	8	10	162	37	-8	9	10	621	-581
1	4	10	182	-320	5	5	10	163	284	10	6	10	119	21	-14	8	10	206	119	-7	9	10	14	-47
2	4	10	41	159	6	5	10	269	257	11	6	10	144	180	-13	8	10	151	31	-6	9	10	456	494
3	4	10	13	183	7	5	10	69	-204	12	6	10	18	88	-12	8	10	390	-412	-5	9	10	13	-16
4	4	10	449	466	8	5	10	16	84	-18	7	10	18	-127	-11	8	10	15	-95	-4	9	10	176	42
5	4	10	44	-150	9	5	10	16	-170	-17	7	10	109	-60	-10	8	10	253	-220	-3	9	10	272	-356
6	4	10	14	146	10	5	10	16	-273	-16	7	10	314	347	-9	8	10	14	-18	-2	9	10	378	-343
7	4	10	597	570	11	5	10	252	264	-15	7	10	103	209	-8	8	10	14	297	-1	9	10	203	242
8	4	10	442	-450	12	5	10	157	124	-14	7	10	128	157	-7	8	10	4	81	C	9	10	404	382
9	4	10	371	363	-18	6	10	274	-284	-13	7	10	5	-124	-6	8	10	378	310	1	9	10	135	-137
10	4	10	16	62	-17	6	10	238	-202	-12	7	10	51	79	-5	8	10	121	173	2	9	10	266	207
11	4	10	17	-18	-16	6	10	519	-570	-11	7	10	261	262	-4	8	10	4	37	3	9	10	15	-203
12	4	10	113	341	-15	6	10	16	-7	-10	7	10	121	83	-3	8	10	4	129	4	9	10	15	-69
-19	5	10	18	145	-14	6	10	429	436	-9	7	10	121	26	-2	8	10	207	194	5	9	10	385	390
-18	5	10	17	-33	-13	6	10	15	-51	-8	7	10	895	-941	-1	8	10	13	27	6	9	10	16	-225
-17	5	10	5	91	-12	6	10	247	266	-7	7	10	43	-136	0	8	10	207	234	7	9	10	16	21
-16	5	10	16	67	-11	6	10	152	-185	-6	7	10	94	-145	1	8	10	99	-58	8	9	10	146	149
-15	5	10	330	-330	-10	6	10	289	235	-5	7	10	540	468	2	8	10	273	-277	5	9	10	246	-130
-14	5	10	15	-98	-5	6	10	465	471	-4	7	10	363	406	3	8	10	14	-140	10	9	10	352	330
-13	5	10	237	-260	-8	6	10	4	-25	-3	7	10	284	-344	4	8	10	208	-235	-16	10	10	EC	149
-12	5	10	327	-364	-7	6	10	458	-443	-2	7	10	13	126	5	8	10	15	151	-15	10	10	100	113
-11	5	10	125	211	-6	6	10	650	-626	-1	7	10	192	256	6	8	10	345	348	-14	10	10	16	-101
-10	5	10	181	151	-5	6	10	13	-103	0	7	10	806	713	7	8	10	42	15	-13	10	10	16	68
-9	5	10	409	451	-4	6	10	220	-122	1	7	10	13	113	8	8	10	16	75	-12	10	10	16	-74
-8	5	10	913	900	-3	6	10	297	289	2	7	10	582	-555	9	8	10	119	-33	-11	10	10	15	-57
-7	5	10	130	65	-2	6	10	1065	-1156	3	7	10	580	-656	10	8	10	165	8	-10	10	10	15	122
-6	5	10	270	306	-1	6	10	598	-522	4	7	10	272	-233	11	8	10	90	-15	-5	10	10	171	135
-5	5	10	345	-341	0	6	10	261	274	5	7	10	478	453	-17	9	10	148	-26	-8	10	10	15	-77
-4	5	10	572	-562	1	6	10	161	110	6	7	10	97	-46	-16	9	10	5	-9	-7	10	10	14	170
-3	5	10	624	661	2	6	10	919	925	7	7	10	16	58	-15	9	10	103	-69	-6	10	10	14	-197
-2	5	10	156	-172	3	6	10	446	-513	8	7	10	456	-371	-14	9	10	16	64	-5	10	10	130	-125

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	H	K	L	10FD	10FC	H	K	L	10FD	10FC	H	K	L	10FD	10FC	H	K	L	10FD	10FC
	-4	10	10	870	802	7	11	10	5	71	-3	13	10	16	41	-3	15	10	140	-84
	-3	10	10	324	-280	8	11	10	17	45	-2	13	10	5	40	-2	15	10	282	-278
	-2	10	10	91	51	9	11	10	164	-212	-1	13	10	125	228	-1	15	10	17	236
	-1	10	10	132	55	-14	12	10	18	112	0	13	10	313	249	0	15	10	17	3
	0	10	10	628	-616	-13	12	10	161	-94	1	13	10	73	52	1	15	10	17	-81
	1	10	10	454	375	-12	12	10	5	50	2	13	10	340	-375	2	15	10	18	34
	2	10	10	15	-61	-11	12	10	16	58	3	13	10	565	-566	3	15	10	18	-196
	3	10	10	15	-78	-10	12	10	16	109	4	13	10	17	135	-8	16	10	18	10
	4	10	10	16	316	-9	12	10	625	641	5	13	10	159	100	-7	16	10	222	128
	5	10	10	216	-279	-8	12	10	16	-190	6	13	10	198	236	-6	16	10	18	-28
	6	10	10	172	-38	-7	12	10	477	-448	7	13	10	6	-123	-5	16	10	200	199
	7	10	10	302	320	-6	12	10	204	-102	-12	14	10	18	-198	-4	16	10	5	-32
	8	10	10	54	33	-5	12	10	580	-619	-11	14	10	18	-2	-3	16	10	18	-50
	9	10	10	18	177	-4	12	10	49	199	-10	14	10	273	-245	-2	16	10	246	157
	-15	11	10	149	-214	-3	12	10	5	87	-9	14	10	119	-23	-1	16	10	18	-15
	-14	11	10	5	-143	-2	12	10	550	-628	-8	14	10	139	122	0	16	10	12	-53
	-13	11	10	16	-59	-1	12	10	15	-51	-7	14	10	16	-87	1	16	10	164	-65
	-12	11	10	16	-175	1	12	10	229	-260	-6	14	10	16	193	-19	1	11	276	194
	-11	11	10	187	231	1	12	10	494	529	-5	14	10	127	125	-18	1	11	5	-14
	-10	11	10	84	-70	2	12	10	517	534	-4	14	10	113	-11	-17	1	11	313	-287
	-9	11	10	129	32	3	12	10	16	-219	-3	14	10	16	33	-16	1	11	16	49
	-8	11	10	523	523	4	12	10	239	-236	-2	14	10	16	42	-15	1	11	15	-249
	-7	11	10	15	-39	5	12	10	17	-137	-1	14	10	16	66	-14	1	11	15	269
	-6	11	10	15	136	6	12	10	174	202	0	14	10	283	268	-13	1	11	450	416
	-5	11	10	260	276	7	12	10	5	115	1	14	10	17	-109	-12	1	11	152	43
	-4	11	10	15	-293	8	12	10	93	-148	2	14	10	221	-225	-11	1	11	586	583
	-3	11	10	133	20	-12	13	10	242	133	3	14	10	120	-83	-10	1	11	150	55
	-2	11	10	15	-73	-12	13	10	121	-137	4	14	10	74	-64	-9	1	11	13	116
	-1	11	10	262	-248	-11	12	10	5	45	5	14	10	210	175	-8	1	11	299	-293
	0	11	10	15	85	-10	12	10	403	420	-10	15	10	18	72	-7	1	11	337	-332
	1	11	10	15	58	-9	13	10	16	-252	-9	15	10	44	-45	-6	1	11	206	-140
	2	11	10	164	-81	-8	13	10	16	-199	-8	15	10	17	-26	-5	1	11	213	176
	3	11	10	134	247	-7	13	10	231	-232	-7	15	10	17	-91	-4	1	11	553	540
	4	11	10	16	-164	-6	13	10	112	-183	-6	15	10	17	189	-3	1	11	325	-323
	5	11	10	5	43	-5	13	10	384	387	-5	15	10	17	-56	-2	1	11	113	-60
	6	11	10	17	50	-4	13	10	152	133	-4	15	10	194	-52	-1	1	11	114	-310

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H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC
4	2	11	234	205	9	3	11	147	-503	-15	5	11	135	125	-9	6	11	4	17
5	2	11	67	-56	10	3	11	302	-267	-14	5	11	308	193	-8	6	11	218	-173
6	2	11	15	-65	11	3	11	179	-73	-13	5	11	84	98	-7	6	11	101	-121
7	2	11	15	-224	-18	4	11	18	83	-12	5	11	15	-46	-6	6	11	402	406
8	2	11	50	271	-17	4	11	230	-253	-11	5	11	149	-130	-5	6	11	337	-339
9	2	11	16	147	-16	4	11	21	13	-10	5	11	14	138	-4	6	11	111	-36
10	2	11	17	-157	-15	4	11	88	-80	-9	5	11	485	504	-3	6	11	65	84
11	2	11	402	433	-14	4	11	168	-122	-8	5	11	205	305	-2	6	11	462	-441
-19	3	11	127	100	-13	4	11	117	-84	-7	5	11	358	-322	-1	6	11	484	466
-18	3	11	17	28	-12	4	11	493	-531	-6	5	11	381	-427	0	6	11	13	155
-17	3	11	17	160	-11	4	11	14	47	-5	5	11	211	239	1	6	11	14	179
-16	3	11	16	-27	-10	4	11	211	226	-4	5	11	86	-128	2	6	11	403	390
-15	3	11	132	-132	-5	4	11	423	388	-3	5	11	466	430	3	6	11	456	-422
-14	3	11	376	306	-8	4	11	522	493	-2	5	11	350	-332	4	6	11	186	-11
-13	3	11	291	-288	-7	4	11	593	-586	-1	5	11	4	-33	5	6	11	5	84
-12	3	11	206	-197	-6	4	11	288	273	0	5	11	304	294	6	6	11	141	33
-11	3	11	517	-515	-5	4	11	123	93	1	5	11	13	173	7	6	11	241	277
-10	3	11	780	-739	-4	4	11	348	383	2	5	11	315	250	8	6	11	5	-54
-9	3	11	95	296	-3	4	11	13	-130	3	5	11	23	11	9	6	11	17	-37
-8	3	11	490	464	-2	4	11	1197	-1236	4	5	11	15	-67	10	6	11	17	177
-7	3	11	13	-29	-1	4	11	13	-57	5	5	11	49	-55	-17	7	11	18	-73
-6	3	11	13	-51	0	4	11	252	-226	6	5	11	5	104	-16	7	11	91	-45
-5	3	11	1075	-1096	1	4	11	600	608	7	5	11	232	274	-15	7	11	163	144
-4	3	11	4	68	2	4	11	87	129	8	5	11	16	-171	-14	7	11	5	-3
-3	3	11	722	643	3	4	11	586	-586	9	5	11	5	19	-13	7	11	208	193
-2	3	11	90	260	4	4	11	4	35	10	5	11	308	-287	-12	7	11	15	167
-1	3	11	278	-288	5	4	11	68	-96	11	5	11	147	105	-11	7	11	234	-208
0	3	11	410	-456	6	4	11	325	329	-18	6	11	162	-18	-10	7	11	636	584
1	3	11	329	-301	7	4	11	256	331	-17	6	11	17	-106	-9	7	11	4	-181
2	3	11	580	625	8	4	11	263	-305	-16	6	11	5	12	-8	7	11	14	121
3	3	11	136	-103	9	4	11	149	-134	-15	6	11	5	66	-7	7	11	14	101
4	3	11	539	-586	10	4	11	17	-160	-14	6	11	123	44	-6	7	11	459	-443
5	3	11	307	-269	11	4	11	6	19	-13	6	11	158	27	-5	7	11	248	209
6	3	11	119	-126	-18	5	11	154	-168	-12	6	11	15	-66	-4	7	11	13	-93
7	3	11	471	498	-17	5	11	17	52	-11	6	11	15	41	-3	7	11	13	-124
8	3	11	16	237	-16	5	11	347	-223	-10	6	11	14	-224	-2	7	11	133	59

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2CS

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H	K	L	10FO	10FC	F	K	L	10FO	10FC	H	K	L	10FO	10FC	F	K	L	10FC	10FC
7	8	11	16	-43	-6	10	11	301	329	5	11	11	411	383	1	13	11	98	61
8	8	11	17	47	-7	10	11	15	-45	6	11	11	17	187	2	13	11	314	-277
9	8	11	18	-11	-6	10	11	15	-181	7	11	11	18	-41	3	13	11	109	196
-16	9	11	191	-191	-5	10	11	217	353	-13	12	11	149	53	4	13	11	18	169
-15	9	11	17	56	-4	10	11	163	175	-12	12	11	121	-11	5	13	11	145	-171
-14	9	11	17	12	-3	10	11	131	-65	-11	12	11	17	58	-11	14	11	221	241
-13	9	11	16	69	-2	10	11	438	-434	-10	12	11	16	19	-10	14	11	18	-44
-12	9	11	134	54	-1	10	11	560	-617	-9	12	11	5	-37	-9	14	11	5	-129
-11	9	11	400	-387	C	10	11	129	-154	-8	12	11	29	-92	-8	14	11	17	-96
-10	9	11	326	-361	1	10	11	115	175	-7	12	11	16	-136	-7	14	11	238	-275
-9	9	11	5	107	2	10	11	5	17	-6	12	11	100	-78	-6	14	11	301	351
-8	9	11	146	-46	3	10	11	163	-167	-5	12	11	71	-13	-5	14	11	5	65
-7	9	11	262	258	4	10	11	16	-102	-4	12	11	343	286	-4	14	11	17	-222
-6	9	11	132	-169	5	10	11	16	123	-3	12	11	16	-176	-3	14	11	17	55
-5	9	11	659	-628	E	10	11	196	151	-2	12	11	184	-194	-2	14	11	108	-98
-4	9	11	423	503	7	10	11	206	137	-1	12	11	168	214	-1	14	11	268	262
-3	9	11	246	214	E	10	11	356	-305	0	12	11	156	116	0	14	11	263	168
-2	9	11	14	-131	-14	11	11	262	200	1	12	11	362	325	1	14	11	17	-83
-1	9	11	15	-124	-13	11	11	117	-116	2	12	11	102	-59	2	14	11	6	110
0	9	11	678	-704	-12	11	11	17	7	3	12	11	449	-412	3	14	11	18	-37
1	9	11	140	103	-11	11	11	207	155	4	12	11	17	174	-8	15	11	154	130
2	9	11	513	525	-10	11	11	152	-120	5	12	11	17	100	-7	15	11	18	89
3	9	11	5	-100	-9	11	11	342	316	6	12	11	158	56	-6	15	11	18	-98
4	9	11	206	-193	-8	11	11	16	-127	-12	13	11	18	-63	-5	15	11	18	-263
5	9	11	206	-151	-7	11	11	15	-132	-11	13	11	154	45	-4	15	11	90	189
6	9	11	329	-312	-6	11	11	5	75	-10	13	11	17	77	-3	15	11	168	184
7	9	11	324	359	-5	11	11	15	-176	-9	13	11	17	9	-2	15	11	208	-26
8	9	11	17	96	-4	11	11	49	163	-8	13	11	16	12	-1	15	11	141	-146
9	9	11	59	-173	-3	11	11	5	50	-7	13	11	16	-97	0	15	11	369	-329
-15	10	11	6	-122	-2	11	11	15	-104	-6	13	11	5	22	1	15	11	18	80
-14	10	11	108	15	-1	11	11	113	137	-5	13	11	158	196	-18	0	12	291	-253
-13	10	11	182	-152	C	11	11	122	223	-4	13	11	16	-15	-17	0	12	17	200
-12	10	11	308	-313	1	11	11	16	123	-3	13	11	16	-226	-16	0	12	264	-241
-11	10	11	5	-14	2	11	11	16	-46	-2	13	11	73	159	-15	0	12	254	-266
-10	10	11	16	275	3	11	11	16	-178	-1	13	11	5	16	-14	0	12	234	131
-9	10	11	330	283	4	11	11	17	-114	0	13	11	140	-9	-13	0	12	344	-337

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2CS

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F	K	L	10FO	10FC	F	K	L	10FO	10FC	H	K	L	10FC	10FC	H	K	L	10FO	10FC	F	K	L	10FC	10FC
-5	1	12	559	-574	2	2	12	113	-16	9	3	12	122	-41	-12	5	12	215	-133	-3	6	12	129	91
-4	1	12	88	-82	3	2	12	546	586	10	3	12	38	-136	-11	5	12	343	264	-2	6	12	153	149
-3	1	12	4	33	4	2	12	15	-393	-18	4	12	6	35	-10	5	12	165	98	-1	6	12	689	668
-2	1	12	675	647	5	2	12	15	-252	-17	4	12	145	136	-9	5	12	443	446	0	6	12	298	-360
-1	1	12	13	-71	6	2	12	16	205	-16	4	12	158	13	-8	5	12	14	-63	1	6	12	238	-204
0	1	12	115	74	7	2	12	187	-206	-15	4	12	130	-131	-7	5	12	497	-538	2	6	12	312	-152
1	1	12	14	57	8	2	12	5	210	-14	4	12	16	212	-6	5	12	774	828	3	6	12	233	-255
2	1	12	113	132	9	2	12	54	66	-13	4	12	273	209	-5	5	12	44	63	4	6	12	711	675
3	1	12	5	-140	10	2	12	81	-288	-12	4	12	15	154	-4	5	12	298	314	5	6	12	171	-245
4	1	12	135	-55	-18	3	12	18	-86	-11	4	12	5	-84	-3	5	12	14	304	6	6	12	423	-380
5	1	12	526	-560	-17	3	12	5	-36	-10	4	12	327	-396	-2	5	12	691	-793	7	6	12	17	127
6	1	12	16	67	-16	3	12	17	-40	-9	4	12	80	-13	-1	5	12	230	172	8	6	12	77	-200
7	1	12	136	292	-15	3	12	16	84	-8	4	12	262	179	0	5	12	304	-285	5	6	12	18	207
8	1	12	17	-169	-14	3	12	5	81	-7	4	12	129	-81	1	5	12	323	-322	-17	7	12	18	-167
9	1	12	17	78	-13	3	12	147	106	-6	4	12	13	-176	2	5	12	15	25	-18	7	12	173	158
10	1	12	324	-351	-12	3	12	15	-94	-5	4	12	270	285	3	5	12	5	22	-15	7	12	17	-13
-18	2	12	18	15	-11	3	12	133	150	-4	4	12	256	259	4	5	12	121	243	-14	7	12	148	113
-17	2	12	17	65	-10	3	12	271	-283	-3	4	12	181	-161	5	5	12	450	453	-12	7	12	166	152
-16	2	12	16	107	-5	3	12	14	-55	-2	4	12	13	125	6	5	12	16	160	-12	7	12	16	-3
-15	2	12	80	-141	-8	3	12	130	88	-1	4	12	632	-627	7	5	12	299	-325	-11	7	12	15	118
-14	2	12	454	457	-7	3	12	83	-71	0	4	12	136	-66	8	5	12	132	31	-10	7	12	15	-51
-13	2	12	15	15	-6	3	12	13	92	1	4	12	391	391	9	5	12	18	-157	-9	7	12	118	-44
-12	2	12	170	-194	-5	3	12	152	-129	2	4	12	280	-261	-17	6	12	18	8	-8	7	12	5	-22
-11	2	12	15	-102	-4	3	12	167	158	3	4	12	463	331	-16	6	12	372	-420	-7	7	12	203	-166
-10	2	12	392	-424	-3	3	12	13	65	4	4	12	15	-56	-15	6	12	17	-13	-6	7	12	386	-401
-9	2	12	86	156	-2	3	12	346	-409	5	4	12	498	-416	-14	6	12	16	-104	-5	7	12	393	-439
-8	2	12	13	-29	-1	3	12	305	360	6	4	12	218	281	-13	6	12	145	-40	-4	7	12	14	-11
-7	2	12	164	131	0	3	12	187	-184	7	4	12	30	70	-12	6	12	554	549	-3	7	12	66	110
-6	2	12	265	289	1	3	12	14	302	8	4	12	17	77	-11	6	12	15	-51	-2	7	12	416	408
-5	2	12	155	-125	2	3	12	14	95	9	4	12	195	221	-10	6	12	15	188	-1	7	12	167	-183
-4	2	12	427	330	1	3	12	347	-344	10	4	12	313	-240	-9	6	12	15	29	0	7	12	162	-95
-3	2	12	103	-47	4	3	12	15	255	-17	5	12	101	31	-8	6	12	431	-442	1	7	12	15	78
-2	2	12	231	158	5	3	12	15	123	-16	5	12	82	-99	-7	6	12	14	149	2	7	12	114	162
-1	2	12	282	-298	6	3	12	5	56	-15	5	12	16	-117	-6	6	12	273	-324	3	7	12	16	88
0	2	12	334	-349	7	3	12	155	105	-14	5	12	16	-100	-5	6	12	585	-584	4	7	12	344	-340
1	2	12	301	294	8	3	12	75	-94	-13	5	12	197	-210	-4	6	12	14	-113	5	7	12	533	-506

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR ERMEC C23H16BR2N2CS

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H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC
6	7	12	5	-115	-8	9	12	180	-166	6	10	12	180	266	4	12	12	18	179
7	7	12	147	187	-7	9	12	15	64	-13	11	12	397	-362	-11	13	12	18	122
8	7	12	152	56	-6	9	12	173	-228	-12	11	12	17	-59	-10	13	12	67	33
9	7	12	18	6	-5	9	12	195	156	-11	11	12	17	35	-9	13	12	187	-47
-16	8	12	18	-36	-4	9	12	145	122	-10	11	12	16	76	-6	13	12	17	151
-15	8	12	5	172	-3	9	12	243	-139	-9	11	12	16	58	-7	13	12	425	-399
-14	8	12	5	-19	-2	9	12	15	-184	-8	11	12	16	92	-6	13	12	210	-285
-13	8	12	16	-156	-1	9	12	15	-53	-7	11	12	149	91	-5	13	12	5	-94
-12	8	12	81	-129	0	9	12	201	301	-6	11	12	16	119	-4	13	12	17	37
-11	8	12	383	-329	1	9	12	308	236	-5	11	12	124	82	-3	13	12	345	280
-10	8	12	15	-83	2	9	12	134	200	-4	11	12	5	-63	-2	13	12	5	122
-9	8	12	15	82	3	9	12	16	-203	-3	11	12	16	6	-1	13	12	150	-138
-8	8	12	219	253	4	9	12	16	-167	-2	11	12	217	-271	0	13	12	75	-189
-7	8	12	94	-23	5	9	12	17	105	-1	11	12	200	-72	1	13	12	159	77
-6	8	12	15	42	6	9	12	5	70	0	11	12	90	103	2	13	12	6	-25
-5	8	12	230	-150	7	9	12	354	466	1	11	12	139	-104	3	13	12	6	170
-4	8	12	139	134	-14	10	12	138	26	2	11	12	198	211	-9	14	12	18	117
-3	8	12	489	464	-13	10	12	259	245	3	11	12	310	-219	-8	14	12	6	76
-2	8	12	15	-27	-12	10	12	5	-116	4	11	12	17	97	-7	14	12	170	56
-1	8	12	189	203	-11	10	12	157	-116	5	11	12	326	356	-6	14	12	60	47
0	8	12	259	-228	-10	10	12	16	-25	-12	12	12	18	295	-5	14	12	126	-84
1	8	12	5	-31	-9	10	12	161	-72	-11	12	12	5	129	-4	14	12	202	128
2	8	12	288	249	-8	10	12	16	50	-10	12	12	168	-108	-3	14	12	130	211
3	8	12	16	-147	-7	10	12	170	111	-9	12	12	17	117	-2	14	12	18	-50
4	8	12	16	-60	-6	10	12	16	-117	-8	12	12	242	-217	-1	14	12	98	-92
5	8	12	154	-121	-5	10	12	15	-48	-7	12	12	148	267	0	14	12	48	-29
6	8	12	17	87	-4	10	12	223	228	-6	12	12	5	-124	1	14	12	18	-52
7	8	12	5	205	-3	10	12	15	-45	-5	12	12	364	-351	-17	1	13	18	-2
8	8	12	18	-61	-2	10	12	149	71	-4	12	12	16	-44	-16	1	13	17	-38
-15	9	12	18	102	-1	10	12	70	-254	-3	12	12	369	-425	-15	1	13	413	-386
-14	9	12	17	195	0	10	12	10	-153	-2	12	12	329	329	-14	1	13	401	316
-13	9	12	17	-51	1	10	12	51	274	-1	12	12	165	112	-13	1	13	137	75
-12	9	12	5	-10	2	10	12	16	-47	0	12	12	17	-232	-12	1	13	5	159
-11	9	12	304	-281	3	10	12	17	132	1	12	12	152	133	-11	1	13	15	135
-10	9	12	16	29	4	10	12	151	-157	2	12	12	17	-55	-10	1	13	345	-359
-9	9	12	16	348	5	10	12	196	-104	3	12	12	225	249	-9	1	13	208	170

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMOC C23H16ER2N2OS

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H	K	L	10FC	10FC	F	K	L	10FC	10FC	H	K	L	10FC	10FC	F	K	L	10FC	10FC
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2	2	13	400	406	-16	4	13	17	-18	-5	5	13	449	-497	6	6	13	5	65
3	2	13	110	-184	-15	4	13	17	-168	-4	5	13	4	-30	7	6	13	18	287
4	2	13	16	-123	-14	4	13	16	-106	-3	5	13	267	364	-16	7	13	163	-145
5	2	13	136	112	-13	4	13	16	50	-2	5	13	199	178	-15	7	13	65	-56
6	2	13	223	-228	-12	4	13	49	-84	-1	5	13	15	-9	-14	7	13	17	58
7	2	13	17	16	-11	4	13	129	59	0	5	13	195	-166	-13	7	13	.312	269
8	2	13	5	19	-10	4	13	5	-236	1	5	13	15	-200	-12	7	13	137	68
9	2	13	18	-56	-9	4	13	15	-168	2	5	13	15	371	-11	7	13	135	-96
-17	3	13	285	228	-8	4	13	265	255	3	5	13	5	-31	-10	7	13	188	-42
-16	3	13	17	48	-7	4	13	57	-100	4	5	13	114	167	-9	7	13	246	-177
-15	3	13	16	216	-6	4	13	14	105	5	5	13	16	-216	-8	7	13	108	185
-14	3	13	133	-227	-5	4	13	14	-126	6	5	13	232	-249	-7	7	13	15	90
-13	3	13	225	-219	-4	4	13	319	-258	7	5	13	5	63	-6	7	13	119	-135
-12	3	13	15	-114	-3	4	13	233	163	8	5	13	198	25	-5	7	13	253	198
-11	3	13	283	-187	-2	4	13	33	-101	-16	6	13	147	22	-4	7	13	485	-464
-10	3	13	230	-149	-1	4	13	81	71	-15	6	13	274	259	-3	7	13	15	143
-9	3	13	211	-196	0	4	13	15	-161	-14	6	13	17	22	-2	7	13	15	-91
-8	3	13	358	-281	1	4	13	229	-218	-13	6	13	215	42	-1	7	13	15	-173
-7	3	13	228	252	2	4	13	5	-86	-12	6	13	222	32	0	7	13	45	42
-6	3	13	253	333	3	4	13	16	76	-11	6	13	217	-245	1	7	13	5	-32
-5	3	13	14	229	4	4	13	16	145	-10	6	13	15	10	2	7	13	16	213
-4	3	13	345	340	5	4	13	235	-254	-9	6	13	135	74	3	7	13	5	99
-3	3	13	14	-125	6	4	13	17	70	-8	6	13	5	10	4	7	13	16	-160
-2	3	13	191	246	7	4	13	17	-233	-7	6	13	15	100	5	7	13	17	-287
-1	3	13	558	575	8	4	13	195	171	-6	6	13	15	27	6	7	13	179	174
0	3	13	344	-369	-16	5	13	277	-163	-5	6	13	185	-245	7	7	13	6	54
1	3	13	5	78	-15	5	13	17	-101	-4	6	13	5	-96	-15	8	13	18	60
2	3	13	447	-513	-14	5	13	105	-61	-3	6	13	48	12	-14	8	13	79	196
3	3	13	15	25	-13	5	13	103	-65	-2	6	13	123	68	-13	8	13	17	-74
4	3	13	572	559	-12	5	13	336	345	-1	6	13	236	280	-12	8	13	16	126
5	3	13	148	-21	-11	5	13	15	-93	0	6	13	32	-134	-11	8	13	243	-167
6	3	13	248	-179	-10	5	13	49	-171	1	6	13	217	135	-10	8	13	5	48
7	3	13	17	-105	-5	5	13	37	52	2	6	13	5	76	-9	8	13	350	369
8	3	13	5	-192	-8	5	13	228	255	3	6	13	16	14	-8	8	13	238	-298
9	3	13	147	212	-7	5	13	243	55	4	6	13	16	-1	-7	8	13	260	-233
															-12	10	13	17	71

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEC C23H16BR2N2OS

H	K	L	10FC0	10FC	H	K	L	10FC0	10FC	H	K	L	10FC0	10FC	H	K	L	10FC0	10FC
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-10	10	13	148	-67	-7	12	13	186	108	1	0	14	148	255	-11	2	14	16	-6
-9	10	13	74	-175	-6	12	13	245	-265	2	0	14	16	80	-10	2	14	333	-258
-8	10	13	159	51	-5	12	13	52	-56	3	0	14	16	74	-9	2	14	103	-82
-7	10	13	16	27	-4	12	13	17	24	4	0	14	5	-54	-8	2	14	83	-173
-6	10	13	16	-157	-3	12	13	17	42	5	0	14	17	233	-7	2	14	15	-50
-5	10	13	180	213	-2	12	13	47	188	6	0	14	153	-70	-6	2	14	345	369
-4	10	13	205	-218	-1	12	13	17	-25	7	0	14	18	48	-5	2	14	15	.64
-3	10	13	315	284	6	12	13	122	-134	-16	1	14	18	73	-4	2	14	214	-135
-2	10	13	80	140	1	12	13	328	257	-15	1	14	17	147	-3	2	14	15	-33
-1	10	13	51	-286	2	12	13	18	72	-14	1	14	195	73	-2	2	14	5	78
0	10	13	34	-51	-8	12	13	6	-77	-13	1	14	16	78	-1	2	14	120	100
1	10	13	390	-387	-7	13	13	18	22	-12	1	14	113	-194	0	2	14	15	49
2	10	13	17	58	-6	13	13	134	-116	-11	1	14	219	232	1	2	14	525	-539
3	10	13	234	249	-5	13	13	175	114	-10	1	14	15	-141	2	2	14	16	-68
4	10	13	18	-91	-4	13	13	18	-30	-9	1	14	15	39	3	2	14	204	190
5	10	13	18	-34	-3	13	13	158	-71	-8	1	14	278	-254	4	2	14	16	-15
-12	11	13	255	187	-2	13	13	18	126	-7	1	14	344	-345	5	2	14	17	48
-11	11	13	5	-27	-1	13	13	6	-236	-6	1	14	15	-7	6	2	14	122	-219
-10	11	13	17	-34	0	13	13	186	160	-5	1	14	118	-80	7	2	14	6	-53
-9	11	13	386	361	-16	0	14	18	122	-4	1	14	15	-37	-16	3	14	52	137
-8	11	13	5	-139	-15	0	14	311	-318	-3	1	14	236	-174	-15	3	14	5	-84
-7	11	13	57	120	-14	0	14	16	-22	-2	1	14	95	107	-14	3	14	5	27
-6	11	13	16	-161	-13	0	14	206	-151	-1	1	14	287	323	-13	3	14	179	139
-5	11	13	532	-553	-12	0	14	16	75	0	1	14	5	88	-12	3	14	16	-226
-4	11	13	52	214	-11	0	14	15	238	1	1	14	49	-26	-11	3	14	5	125
-3	11	13	247	27	-10	0	14	15	163	2	1	14	509	-536	-10	3	14	235	-90
-2	11	13	360	351	-9	0	14	498	432	3	1	14	145	40	-9	3	14	15	107
-1	11	13	280	193	-8	0	14	84	-46	4	1	14	16	79	-8	3	14	115	78
0	11	13	255	-268	-7	0	14	340	327	5	1	14	243	193	-7	3	14	5	-24
1	11	13	5	-42	-6	0	14	363	-358	6	1	14	17	54	-6	3	14	105	69
2	11	13	69	83	-5	0	14	15	-127	7	1	14	18	-236	-5	3	14	520	-555
3	11	13	57	103	-4	0	14	360	250	-16	2	14	253	85	-4	3	14	247	261
-11	12	13	154	-131	-3	0	14	164	-54	-15	2	14	47	-133	-3	3	14	48	-78
-10	12	13	341	162	-2	0	14	107	258	-14	2	14	16	64	-2	3	14	258	231
-9	12	13	18	-72	-1	0	14	287	-263	-13	2	14	211	15	-1	3	14	15	200
															-12	5	14	162	156

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR SRMEC C23H16BR2N2CS

PAGE 40

H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC	H	K	L	10FC	10FC
-11	5	14	114	-69	3	6	14	17	-170	-2	8	14	260	150	1	10	14	67	-146
-10	5	14	16	-110	4	6	14	17	360	-1	8	14	309	272	2	10	14	18	33
-9	5	14	242	243	5	6	14	18	-210	0	8	14	268	-273	-10	11	14	6	117
-8	5	14	15	173	6	6	14	18	93	1	8	14	5	-190	-9	11	14	18	57
-7	5	14	15	125	-14	7	14	18	78	2	8	14	17	-91	-8	11	14	18	-60
-6	5	14	73	72	-13	7	14	17	29	3	8	14	17	-54	-7	11	14	293	351
-5	5	14	15	-111	-12	7	14	131	77	4	8	14	405	364	-6	11	14	52	-119
-4	5	14	271	241	-11	7	14	17	-50	-13	9	14	18	14	-5	11	14	28	-64
-3	5	14	276	291	-10	7	14	141	-61	-12	9	14	6	78	-4	11	14	17	34
-2	5	14	184	-198	-5	7	14	193	-147	-11	9	14	235	-168	-3	11	14	5	135
-1	5	14	16	-102	-8	7	14	16	-207	-10	9	14	17	51	-2	11	14	171	-14
0	5	14	326	-295	-7	7	14	50	-103	-9	9	14	17	-75	-1	11	14	6	-50
1	5	14	16	86	-6	7	14	16	196	-8	9	14	266	256	0	11	14	295	-287
2	5	14	408	374	-5	7	14	150	236	-7	9	14	5	52	1	11	14	6	-22
3	5	14	117	-52	-4	7	14	16	-242	-6	9	14	16	-266	-7	12	14	18	221
4	5	14	198	96	-3	7	14	16	32	-5	9	14	16	25	-6	12	14	18	-261
5	5	14	17	-148	-2	7	14	95	-186	-4	9	14	5	-128	-5	12	14	6	35
6	5	14	210	-140	-1	7	14	240	133	-3	9	14	136	76	-4	12	14	183	136
-15	6	14	237	21	0	7	14	364	343	-2	9	14	139	237	-3	12	14	324	-405
-14	6	14	300	-277	1	7	14	133	-237	-1	9	14	5	-177	-2	12	14	162	175
-13	6	14	17	135	2	7	14	75	69	0	9	14	176	98	-15	1	15	41	-59
-12	6	14	76	31	2	7	14	17	-7	1	9	14	123	-210	-14	1	15	17	74
-11	6	14	164	105	4	7	14	5	-187	2	9	14	17	-31	-13	1	15	17	-148
-10	6	14	338	293	5	7	14	18	56	3	9	14	18	53	-12	1	15	5	67
-9	6	14	188	8	-14	8	14	18	-184	-11	10	14	18	-124	-11	1	15	129	129
-8	6	14	16	231	-13	8	14	18	-7	-10	10	14	207	-166	-10	1	15	136	-76
-7	6	14	16	-17	-12	8	14	17	20	-9	10	14	125	-37	-9	1	15	5	80
-6	6	14	158	29	-11	8	14	5	-52	-8	10	14	5	-76	-8	1	15	211	-257
-5	6	14	16	74	-10	8	14	70	43	-7	10	14	17	123	-7	1	15	87	137
-4	6	14	246	-58	-9	8	14	16	-218	-6	10	14	182	200	-6	1	15	122	254
-3	6	14	16	-135	-8	8	14	203	-243	-5	10	14	17	-124	-5	1	15	5	30
-2	6	14	183	-181	-7	8	14	16	155	-4	10	14	5	-154	-4	1	15	342	-345
-1	6	14	16	117	-6	8	14	5	63	-3	10	14	17	67	-3	1	15	16	-251
0	6	14	153	84	-5	8	14	45	51	-2	10	14	5	95	-2	1	15	16	-75
1	6	14	184	229	-4	8	14	16	109	-1	10	14	17	37	-1	1	15	16	128
2	6	14	16	22	-3	8	14	16	-49	0	10	14	17	-52	0	1	15	362	388

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMEO C23H16BR2N2OS

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H	K	L	10FO	10FC	F	K	L	10FO	10FC	H	K	L	10FO	10FC	F	K	L	10FC	10FC
-5	3	15	111	249	-5	5	15	146	-180	-8	7	15	17	175	-1	9	15	18	-134
-4	3	15	16	165	-8	5	15	87	-21	-7	7	15	16	-157	0	9	15	172	15
-3	3	15	167	161	-7	5	15	5	-56	-6	7	15	342	272	1	9	15	18	58
-2	3	15	128	57	-6	5	15	202	179	-5	7	15	306	-268	-9	10	15	18	-44
-1	3	15	188	-212	-5	5	15	16	-55	-4	7	15	171	-155	-8	10	15	168	140
0	3	15	200	-67	-4	5	15	16	-174	-3	7	15	16	-10	-7	10	15	18	36
1	3	15	179	141	-3	5	15	121	49	-2	7	15	5	-146	-6	10	15	98	162
2	3	15	17	-56	-2	5	15	16	-185	-1	7	15	17	375	-5	10	15	18	108
3	3	15	116	56	-1	5	15	158	210	0	7	15	99	39	-4	10	15	18	-170
4	3	15	93	99	0	5	15	200	-166	1	7	15	106	-143	-3	10	15	18	125
5	3	15	221	-184	1	5	15	164	43	2	7	15	18	-72	-2	10	15	341	-264
-14	4	15	6	72	2	5	15	127	57	3	7	15	172	-145	-1	10	15	18	-18
-13	4	15	17	-154	3	5	15	5	-151	-12	8	15	321	279	-14	0	16	18	-74
-12	4	15	5	-148	4	5	15	222	265	-11	8	15	6	-146	-13	0	16	18	38
-11	4	15	16	83	-13	6	15	6	24	-10	8	15	154	92	-12	0	16	108	141
-10	4	15	16	-64	-12	6	15	17	45	-9	8	15	17	-213	-11	0	16	17	75
-9	4	15	16	233	-11	6	15	17	-28	-8	8	15	17	103	-10	0	16	17	126
-8	4	15	114	-33	-10	6	15	17	30	-7	8	15	5	163	-9	0	16	16	81
-7	4	15	16	-91	-5	6	15	16	14	-6	8	15	107	-130	-8	0	16	16	52
-6	4	15	71	70	-8	6	15	104	19	-5	8	15	17	44	-7	0	16	208	140
-5	4	15	16	-15	-7	6	15	5	-38	-4	8	15	5	-139	-6	0	16	16	-166
-4	4	15	16	116	-6	6	15	16	-146	-3	8	15	17	222	-5	0	16	16	59
-3	4	15	151	-8	-5	6	15	224	-24	-2	8	15	17	50	-4	0	16	143	-207
-2	4	15	16	-165	-4	6	15	16	-121	-1	8	15	17	72	-3	0	16	315	-145
-1	4	15	16	-74	-2	6	15	16	-14	0	8	15	11	-93	-2	0	16	230	250
0	4	15	5	36	-2	6	15	146	186	1	8	15	111	-155	-1	0	16	363	-339
1	4	15	159	63	-1	6	15	141	-80	2	8	15	18	175	0	0	16	17	274
2	4	15	17	-46	0	6	15	11	-25	-10	9	15	37	108	1	0	16	108	-233
3	4	15	5	60	1	6	15	17	-228	-9	9	15	18	-23	2	0	16	169	58
4	4	15	177	-206	2	6	15	17	-168	-8	9	15	112	35	3	0	16	364	418
5	4	15	181	161	3	6	15	167	76	-7	9	15	77	133	-13	1	16	18	45
-14	5	15	145	-171	-13	7	15	6	79	-6	9	15	65	-92	-12	1	16	144	-6
-13	5	15	160	29	-12	7	15	18	-110	-5	9	15	17	121	-11	1	16	5	39
-12	5	15	5	-108	-11	7	15	17	68	-4	9	15	17	28	-10	1	16	5	26
-11	5	15	149	294	-10	7	15	17	-62	-3	9	15	94	-29	-9	1	16	157	72
-10	5	15	74	21	-9	7	15	17	40	-2	9	15	184	162	-8	1	16	5	-56

## RESERVED AND CALCULATED STRUCTURE FACTORS FOR BRMPC C23H16BR2N2CS

PAGE 42

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC					
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-4	3	16	16	117	-11	5	16	18	121	0	6	16	261	-42	-8	1	17	134	-83	-7	3	17	18	-22
-3	3	16	61	-175	-10	5	16	150	-44	1	6	16	18	22	-7	1	17	158	95	-6	3	17	122	23
-2	3	16	17	118	-5	5	16	17	-49	-10	7	16	18	-108	-6	1	17	198	-242	-5	3	17	153	197
-1	3	16	17	-40	-6	5	16	17	-65	-9	7	16	235	25	-5	1	17	17	144	-4	3	17	57	-121
0	3	16	108	-17	-7	5	16	17	259	-8	7	16	148	103	-4	1	17	17	24	-2	3	17	139	-72
1	3	16	5	3	-6	5	16	17	140	-7	7	16	17	-33	-3	1	17	17	7	-2	3	17	89	-92
2	3	16	135	-236	-5	5	16	148	120	-6	7	16	5	5	-2	1	17	95	124	-1	3	17	6	19
-13	4	16	171	-76	-4	5	16	140	-65	-5	7	16	17	-152	-1	1	17	168	-202	-10	4	17	6	-63
-12	4	16	18	-206	-3	5	16	225	-312	-4	7	16	17	-5	0	1	17	18	196	-5	4	17	25	167
-11	4	16	17	-30	-2	5	16	17	63	-3	7	16	18	182	-11	2	17	18	-78	-6	4	17	98	-105
-10	4	16	5	160	-1	5	16	17	41	-2	7	16	18	-92	-10	2	17	18	-113	-7	4	17	18	137
-9	4	16	17	-21	0	5	16	104	12	-1	7	16	6	10	-9	2	17	128	90	-6	4	17	18	-21
-8	4	16	5	-17	1	5	16	18	148	0	7	16	12	77	-8	2	17	17	-85	-5	4	17	112	-83
-7	4	16	226	-95	-11	6	16	293	357	-9	8	16	163	-109	-7	2	17	136	127	-4	4	17	18	105
-6	4	16	16	121	-10	6	16	18	6	-8	9	16	150	-90	-6	2	17	17	23	-3	4	17	121	-120
-5	4	16	5	-48	-5	6	16	150	-150	-7	8	16	18	-130	-5	2	17	17	-94	-2	4	17	18	141
-4	4	16	5	13	-8	6	16	17	185	-6	8	16	6	16	-4	2	17	17	117	-6	5	17	18	109
-3	4	16	17	-35	-7	6	16	150	-122	-5	8	16	256	128	-3	2	17	315	-311	-7	5	17	18	-112
-2	4	16	75	-92	-6	6	16	17	189	-4	8	16	6	189	-2	2	17	18	111	-6	5	17	158	72
-1	4	16	321	289	-5	6	16	17	6	-3	8	16	18	-57	-1	2	17	6	34	-5	5	17	167	-54
0	4	16	11	-12	-4	6	16	17	-226	-2	8	16	174	-131	-10	3	17	6	16	-4	5	17	17	40
1	4	16	167	16	-3	6	16	5	16	-11	1	17	18	-151	-9	3	17	18	-169	-3	5	17	150	26
2	4	16	18	-40	-2	6	16	31	-78	-10	1	17	297	208										